Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #1

Field Safety for Water Quality Sampling

Version 1.0 (04/10/2007)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

This Standard Operating Procedure (SOP) describes procedures for conducting field and laboratory aspects of water quality monitoring in a safe manner, recognizing that certain hazards are inherent in both field and laboratory environments. This SOP both (1) identifies hazards associated with field and laboratory settings and (2) outlines approaches to reduce or mitigate those hazards.

The primary tool used to promote safe conduct is the Job Hazard Analysis. This approach is consistent with NPS Director's Order 50 and Reference Manual 50B for Occupational Heath and Safety.

Table of Contents

1. Introduction and Objectives 3
2. Risks Inherent in Water Quality Monitoring 3
2.1. Working alone 3
2.2. Long road trips 3
2.3. Backcountry roads and trails 3
2.4. Activities in and on the water 4
2.5. Varied weather conditions 4
2.6. Use of hazardous materials 4
3. Job Hazard Analysis 4
4. Field Site Description 7
5. Routine and Emergency Communication 7
5.1. Safety contact numbers 9
5.1.1. Arches National Park 9
5.1.2. Black Canyon of the Gunnison National Park 10
5.1.3. Bryce Canyon National Park 10
5.1.4. Canyonlands National Park 11
5.1.5. Capitol Reef National Park 11
5.1.6. Curecanti National Recreation Area 12
5.1.7. Zion National Park 13
5.2. Park radio 13
6. Vehicle Accident Procedure 15
7. Chemical Storage and Use 15
8. Appendix A: Blank Job Hazard Analysis Form 17
9. Appendix B: Suggested topics to be addressed in a Job Hazard Analysis 18
10. Appendix C: Example NCPN Job Hazard Analysis for Field Activities 19
11. Appendix D: NCPN Job Hazard Analysis for Laboratory Activities 20
12. Appendix E: Safety Considerations in Field Activities 21
13. Appendix F: Vehicle Accident Form 34

List of Tables

Table 1. Suggested list of park contacts and information for field operations. 9
Table 2. List of park radio contacts for BRCA, ZION, and CARE. 14
Table 3. Chemical list to be posted on chemical cabinet at BRCA. 16

List of Figures

Figure 1. Example e-mail to ZION staff informing them of water quality monitoring activities. 8
Figure 2. Example of emergency contact form to be completed and taken on field trips. 23

1. Introduction and Objectives

Encountering hazardous situations is inherent in water quality monitoring activities. Thus, staff members need to be aware of possible hazards and be able to adjust approaches accordingly so that high-quality monitoring can be conducted without injury or property damage.

The objectives for the Northern Colorado Plateau Network (NCPN) water quality monitoring program, and the specific objectives for operations under this SOP, are:

- To monitor water quality conditions, including accessing monitoring sites, making field measurements, gathering, processing, and analyzing water quality samples in a manner that recognizes hazardous conditions and takes measures to minimize the risk of personnel injury.

- To maintain a high-quality monitoring effort, avoiding missed data collection and undue expense due to personnel injury or property damage.

Some hazardous conditions are inherent to all monitoring activities being undertaken by NCPN staff; others are specific to individual sites. Examples of the former include exposure to winter and summer weather, highway driving, and using hazardous chemicals. Site-specific hazards might include a specific four-wheel drive road or access through a particular slot canyon. To minimize the possibility of accident or injury, a Job Hazard Analysis will be conducted for each employee engaged in field water quality monitoring. Second, hazards associated with specific sampling sites will be identified in the Field Site Description for each site. These procedures both improve operational safety by identifying hazardous conditions to NPS staff and supervisors and develop approaches to avoid or mitigate the hazards.

Safety always comes first, and because the logistics of field work can require much of a field supervisor's attention, it is the responsibility of everyone in the field to point out safety issues or concerns that otherwise could be overlooked in efforts to complete the field tasks at hand or when distractions occur from problems that may arise.

2. Risks Inherent in Water Quality Monitoring

Risks inherent in water quality monitoring in general, and specifically to monitoring the sites selected in NCPN parks, include the following:

2.1. Working alone

Field staff often will work alone when traveling to and from remote sites and when making measurements and collecting samples in the field. As a result, particular attention must be given to safety.

2.2. Long road trips

Travel between parks will require long road trips at all times of the year and under varied weather and road conditions.

2.3. Backcountry roads and trails

Accessing water quality sample sites requires travel on secondary roads that are gravel or dirt, and some sites require hikes of up to three hours each way. The unsurfaced roads can become

impassible during wet weather, and may remain muddy for several days after snowmelt. Hiking is generally done on established trails that vary in difficulty and condition. Some are steep, rocky, or sandy when dry but become muddy in wet weather.

2.4. Activities in and on the water

Sample collection and flow measurements will require entering the main current in streams and boating to sites on lakes and large rivers. Extra care is necessary when wading because many streams are swift, stream bottoms can be loose or slippery, and water temperatures can be near freezing in winter. Precautions are needed to (1) recognize and avoid unacceptable risks, (2) use proper techniques to avoid accidents, and (3) be properly equipped for self -rescue. It is recognized that some sites will not be accessible during periods of bad weather. Similarly, it will not be possible to get a wading flow measurement during periods of flood**. If there is any doubt in regard to whether conditions are safe, the correct decision is to forgo sampling until another day.** When discharge is too high for wading, use an estimated discharge (see the STORET Flow Severity Index and drop-down menus for hydrograph limb in NPSTORET Templates for "qualitative flow" estimates). These are found in the Field_forms.xls spreadsheet page "field codes" on the hard drive of the hydrologist's computer. A discharge also may be obtained from a bridge location if the appropriate equipment is available.

Excerpts from USGS Field Manual section 9.4 in Appendix E give excellent guidance for safety precautions working on, in, and near water.

2.5. Varied weather conditions

The Colorado Plateau region is characterized by weather conditions that vary considerably through the seasons, day-to-day, and hour-by-hour. Snow, wind, and temperatures below 0°F occur in the winter, and summer temperatures are often well above 100°F. Lightning is common during the summer monsoon storms. Hypothermia and heat-related injury are possible, and occasional discomfort is certain. Successful field work requires preparation for this wide range of conditions.

2.6. Use of hazardous materials

Some of the reagents used are hazardous and require proper storage, handling, and personal protection, including eye and skin protection.

3. Job Hazard Analysis

Each National Park Service employee performing field inventory and monitoring activities in the NCPN will, in conjunction with his/her supervisor (and, if appropriate, other knowledgeable persons), develop a Job Hazard Analysis (JHA) of the task to be performed. The procedure to be used is presented in NPS Reference Manual #50B, Occupational Safety and Health Program. Section 13 of that document, a step-by-step procedure for performing a JHA, is presented below. In addition:

- A form for completing a JHA is presented in Appendix A, and a list of suggested hazards (to be used in STEP 3) that are known to be associated with field water quality monitoring are presented in Appendix B.
- An example JHA for Water Quality Field Work is presented in Appendix C.
- An example JHA for Water Quality Laboratory Sample Processing is presented in Appendix D.

- The USGS National Field Manual includes a Chapter for Safety in Field Activities. Pertinent sections are included as a useful reference in Appendix E.

Excerpt: Section 13 of NPS Reference Manual 50B. Section numbers are maintained as per the original document.

13. JOB HAZARD ANALYSIS (JHA)

13.1 REFERENCE

A. Departmental Manual (DM), Part 485, Chapter 14.

13.2 REQUIREMENTS

A. Each operating unit will develop and implement a system by which major activities and individual jobs are analyzed. The Job Hazard Analysis (JHA) system will be used in the planning and completion of work assignments and projects as to:

- The sequence of work,
- The potential hazards associated with the sequence (action), and
- The methods, procedures, and safeguards to prevent, reduce and or control at-risk behaviors and reinforce safe behaviors.

B. When work is performed by others (e.g., contractors) the contracting officer (CO) will require that the contractor develops appropriate Job Hazard Analyses (JHAs) for the tasks to be performed. This information will be provided to the Contracting Officer (CO) or Contracting Officer's Representative (COR) as part of the accident prevention plan, and will be approved/accepted by the CO/COR prior to permitting the contractor to proceed with the work. The written JHA will be retained at the job site by the contractor, and made available, upon request, to persons evaluating/monitoring the activity.

13.3 PROCEDURES:
A Job Hazard Analysis (JHA) is a procedure used to review job methods and uncover hazards. Employees and supervisors should work together in the development of the JHA to assure that all characteristics of the job are examined, and the most efficient means of addressing the safety factors can be accomplished. A JHA should be used for job clarification, hazard awareness, training, and as an accident investigation tool. The following steps are involved in the development of a JHA:

STEP 1
Identify jobs posing the greatest accident risk, and prioritize selected jobs into three main areas:

- Jobs with highest rates of accidents and disabling injuries.
- Jobs where "close calls" (serious accident potential) have occurred.
- New jobs or jobs where changes have been made.

STEP 2
Targeting the top-priority jobs, break the selected job down into successive steps, actions, or activities and observe how these actions are performed. This is best done by jointly analyzing each step in order of occurrence. Each step should accomplish some major task. The task will consist of a set of behaviors/actions; list all the steps in a job in logical succession.

STEP 3
Identify the hazards, unsafe behaviors, and potential accidents for the job. Identify all hazards produced by the environment, equipment, materials, and those connected with the job procedure, as well as hazards that "might result."

Close observation and knowledge of the particular job, as well as discussion of the job, will help you to develop a reliable list of hazards, unsafe behaviors, and potential accidents. The job observation should be repeated as often as necessary until all hazards have been identified.

STEP 4

Develop safe job procedures to eliminate the hazards and prevent the potential accidents. Produce a written description of how to do each step safely and avoid the identified hazards.

The principal solutions may include:

1. Find a new way to do the job. Determine the work goal and analyze the various ways of reaching this goal to see which way is safest.

2. Change the physical conditions that create the hazard. Ask the question: "What change in physical condition will eliminate the hazard or prevent the accident?" When a change is found, analyze it for other benefits that might also accrue, i.e., cost/time savings.

3. Change the work procedure. Ask, "What should the employee do or not do to eliminate this hazard or prevent this potential accident?" Answers should precisely state what to do and how to do it; general precautions such as "be alert" or "use caution" are useless.

4. Try to reduce the necessity of doing a job, or at least the frequency with which it must be performed. However, reducing the frequency of the job ONLY limits the exposure; it does not eliminate the hazard.

STEP 5
Established JHAs should be reevaluated periodically to ensure that they reflect the latest, safest, and most efficient way to perform the task. New equipment, tools, methods, and safety standards can require changes in JHAs. All employees concerned with the job should be informed of the changes and instructed in any new procedures. All new employees should be trained on applicable JHAs.

STEP 6
If an accident or injury occurs on a specific job, the JHA should be reviewed immediately to determine whether changes are needed in the job procedure. If the accident results from failure to follow JHA procedures, the facts should be discussed with all those who do the job.

STEP 7
Supervisors are responsible for maintaining JHA records within their work group.

4. Field Site Description

A record of site locations and specific hazards that might be present will be developed in Field Site Descriptions that are kept as a reference for field staff in the field folder that accompanies staff in the field vehicle. This will serve as a reference and reminder for existing and new staff. The procedure for completing field site descriptions is presented in SOP 4: Site Selection, Conditions, and Documentation.

5. Routine and Emergency Communication

Using established lines of communication and a standardized Check-in/Check-out procedure are essential to ensuring that assistance can be provided in case of a mishap or delay. This is particularly important because NCPN field staff often work alone. A routine will be established wherein NCPN field staff contact park staff to notify them of the time and location of work in each park (Figure 5-1).

To: Ray O'Neil/ZION/NPS, Kodi Schoppmann/ZION/NPS, Zion Dispatch
Subject Line: Water Quality Sampling Itinerary

I will be in Zion for monthly water quality sampling as part of the Northern Colorado Plateau NPS inventory and monitoring project.

My vehicle is a white Jeep Cherokee 4 door with government plates.

I will be parked at the NF Virgin River Bridge on the North Fork Road 6/8/06.

I will access North Creek from the pull out near the park boundary in the afternoon on 6/8/06.

I will park at the Lee Pass Trailhead parking area for the day to access LaVerkin Creek on 6/9/06.

I am traveling solo and have a radio with Zion frequencies.

Thanks,

David

David Thoma
Hydrologist
National Park Service
Northern Colorado Plateau Network
P.O. Box 640201
Bryce Canyon, UT 84764
Voice: 435-834-4905
http://www.nature.nps.gov/im/units/ncpn/

Figure 1. Example e-mail to ZION staff informing them of water quality monitoring activities.

For travel in the backcountry and off of surfaced roads, park notification will also include an expected time of return. In periods of inclement weather this notification and check-in procedure may be extended to hard -surfaced roads.

A contacts list will be developed and attached to the Field Site Description described in SOP 4, which will be carried in the field vehicle. A suggested list of contacts is presented below. Where their radio coverage is superior to that of the NPS, this list may be expanded to include the BLM or other agencies.

Table 1. Suggested list of park contacts and information for field operations.

Park	Contact type	Name	Radio call number	Telephone (office and home)	Cell phone	Hours of availability
	Primary contact					
	Secondary contact					
	Backcountry contact					
	Park dispatch					
	Other contacts					

5.1. Safety contact numbers

The regional 24-7 emergency number is 435-772-3322. Use this number after midnight when park dispatch offices close. They can then phone a designated "on-duty" ranger at any regional park.

5.1.1. Arches National Park

Contact	Radio	Telephone	Comments
Duty ranger			
Law enforcement dispatch			
Outside the park			
County/City Dispatch			
Nearest emergency facility:			
Regional 24-7 emergency number		435-772-3322	Use this number after midnight when park dispatch offices close. They can then phone a designated "on-duty" ranger at any regional park.

5.1.2. Black Canyon of the Gunnison National Park

Contact	Radio	Telephone	Comments
Duty ranger	Call “Duty ranger”, then say “Bryce Canyon employee ‘your name’”		Radio channel 4 works best in most places.
Law enforcement dispatch	Use radio channel 4 to hit repeater and channel 1 for direct contact. Radio coverage is spotty below the rim.	NA	
Outside the park		911	
Montrose County Dispatch		970-252-4023	Cell phone coverage is reliable in BLCA.
Nearest emergency facility: Montrose Memorial Hospital (Montrose)		970-249-2211	Cell phone coverage is reliable in BLCA.
Regional 24-7 emergency number		435-772-3322	Use this number after midnight when park dispatch offices close. They can then phone a designated “on-duty” ranger at any regional park.

5.1.3. Bryce Canyon National Park

Contact	Radio	Telephone	Comments
Duty ranger	851; radio channel 13 works best in most places.		
Law enforcement dispatch	Use radio channel 14 to hit repeater and channel 13 for direct contact. Radio coverage is spotty below the rim.	800-582-4351	
Outside the park		911	
Garfield County Sheriff		435-676-2411	
Nearest emergency facility: Garfield County Clinic (Panguitch)		435-676-8842	
Garfield Memorial Hospital (Panguitch)		435-676-8811	
Regional 24-7 emergency number		435-772-3322	Use this number after midnight when park dispatch offices close. They can then phone a designated “on-duty” ranger at any regional park.

5.1.4. Canyonlands National Park

Contact	Radio	Telephone	Comments
Duty ranger			
Law enforcement dispatch			
Outside the park			
County/City Dispatch			
Nearest emergency facility:			
Regional 24-7 emergency number		435-772-3322	Use this number after midnight when park dispatch offices close. They can then phone a designated “on-duty” ranger at any regional park.

5.1.5. Capitol Reef National Park

Contact	Radio	Telephone	Comments
Duty ranger	Call “Patrol Ranger”, then say “Bryce Canyon employee ‘your name’”		
Law enforcement dispatch	Use radio channel 5 to hit repeater and channel 9 for direct contact. This is radio-specific but programmed this way on the NCPN Hydrologist Radio	435-425-3791 x111	Staffed only between 8am and 6pm
Outside the park		911	
Wayne County Sheriff		435-836-2789	
Nearest emergency facility: County Clinic (location)		435-425-3744	
Sevier Valley Hospital (Richfield)		435-869-8271 or 800-356-8757	
Regional 24-7 emergency number		435-772-3322	Use this number after midnight when park dispatch offices close. They can then phone a designated “on-duty” ranger at any regional park.

5.1.6. Curecanti National Recreation Area

Contact	Radio	Telephone	Comments
Duty ranger	Call #NA		Radio channel 2 works best in most places.
Law enforcement dispatch	Use radio channel 2 to hit repeater and channel 1 for direct contact. Radio coverage is spotty below the rim and on lower reservoirs.	NA	
Outside the park		911	Cell phone coverage is reliable in CURE.
Gunnison County/City Dispatch		970-641-8000	Cell phone coverage is reliable in CURE.
Nearest emergency facility: Gunnison Valley Hospital (Gunnison)		970-641-1456	Cell phone coverage is reliable in CURE.
Regional 24-7 emergency number		435-772-3322	Use this number after midnight when park dispatch offices close. They can then phone a designated “on-duty” ranger at any regional park.

5.1.7. Zion National Park

Contact	Radio	Telephone	Comments
Duty ranger	Call “Zion Dispatch” then say “Bryce Canyon employee ‘your name’”		
Law enforcement dispatch	Use radio channel 10, 11, or 12 to hit repeater and channel 9 for direct contact. This is radio-specific but programmed this way on the NCPN Hydrologist Radio. Radio coverage is spotty below the rim.	435-772-0178	
Outside the park		911	
Washington County Sheriff		435-634-5730	
Nearest emergency facility: Zion Clinic (Springdale)		435-772-3226	Open Tuesdays only in winter
Hurricane Clinic		435-635-4485	
Dixie Regional Hospital (St. George)		435-688-4000	
Regional 24-7 emergency number		435-772-3322	Use this number after midnight when park dispatch offices close. They can then phone a designated “on-duty” ranger at any regional park.

5.2. Park radio

A personal two-way radio programmed with NPS frequencies is usually the best method for routine field and emergency communication in and near parks.

A radio has been made available on long-term loan to the NCPN hydrologist from Bryce Canyon National Park (BRCA). The radio has 13 programmed channels corresponding to the repeaters and direct channels used in BRCA, Zion National Park (ZION), and Capitol Reef National Park (CARE). BRCA staff helped program radio frequencies used by each district where the hydrologist typically works (**Error! Reference source not found.**). In some cases, reliable cell phone coverage exists and can be used. When practical, NCPN field staff will carry back-up means of emergency communication, including a cellular phone and a signal mirror.

Table 2. List of park radio contacts for BRCA, ZION, and CARE.

Radio channel	Frequencies
1	^DNF Simplex
	Rx/Tx 171.550
2	DNF Repeater (Tone 123.0)
	Rx 171.550 Tx 172.350
3	BLM Escalante Simplex
	Rx/Tx 163.0375
4	BLM Escalante Repeater (Tone 131.8)
	Rx 163.0375 Tx 166.6375
5	CARE
6	Air to Ground 1
	Rx/Tx 172.325
7	Air to Ground 2
	Rx/Tx 171.575
9	Common Use
	Rx/Tx 168.350
10	Zion Direct
	Rx/Tx 166.325
11	Zion Lava PT (Tone 107.2)
	Rx 166.325 TX 166.925
12	Zion West Temple (Tone 114.8)
	Rx 166.325 TX 166.925
13	Bryce Simplex
	Rx/Tx 172.500 AW
14	Bryce Repeater (Tone 123.0)
	Rx 172.500 TX 170.100

6. Vehicle Accident Procedure

In the event of a vehicle accident, ensure the safety of all passengers and move the vehicle from traveled lanes if possible. Contact emergency personnel and seek medical attention. Accident reporting forms can be found in Appendix F.

7. Chemical Storage and Use

Chemicals must be stored according to their Material Safety Data Sheet (MSDS) information. The MSDS sheets for all chemicals are kept in a clearly marked folder in the lab.

A list of all chemicals (Table 3.) must be posted on the outside of the cabinet door (or wherever they are stored). This is done so that: (1) emergency personnel are able to identify the contents in case of an emergency and (2) people who are sensitive to certain materials are able to avoid exposure. The list of chemicals should include the name of the chemical, the quantity of that chemical in storage, and the quantity of that chemical that has been used (this includes anything that has been opened).

One list must be in the lab and copies of this list must be given to both the hazardous materials coordinator (Tom Dearden) and the operations ranger (Dave Fox) at BRCA.

Table 3. Chemical list to be posted on chemical cabinet at BRCA.

Water Quality Lab Chemical List		
Solution	**Quantity stored**	**Quantity used**
pH Buffer Solution 4	500ml-3	500ml-1 Lg-1
pH Buffer Solution 7	500ml-2	Lg-2
pH Buffer Solution 10	500ml-1	500ml-2 Lg-1
In Situ Quick Cal Solution	Sm-3 Lg-3	Sm-1 Lg-1
Sodium Sulfite Solution	0	1
Deionized Water	0	2
Conductivity Standard 147	0	1
Conductivity Standard 1413*	0	2
Conductivity Standard 12880	0	1
DO Electrode Filling Solution	0	1
pH Electrode	0	2
Stablcal Formazin Standard 800 NTU	0	2 (1 exp)
Stablcal Formazin Standard <0.1 NTU	0	2 (1 exp)
Stablcal Formazin Standard 20 NTU	0	2 (1 exp)
Stablcal Formazin Standard 100 NTU	0	2 (1 exp)
Colilert	0	1 (Box of 200 Packets)
Colilert Comparator	0	1 (In Freezer)
High Vacuum Grease	1	2
Sodium Sulfite	0	1
Cobalt (II) Chloride	0	1
Potassium Chloride	0	1
10% NH3 Cleaning Solution	0	1
Clorox Bleach	0	1
Arm and Hammer Baking Soda	0	0
Unless otherwise noted all chemicals are in main cabinet for storage.		
pH-Cabinet, Under Sink, Refrigerator, Box on Shelf		
Deionized Water- On Shelf		
Conductivity Standard 147- Refrigerator		
Conductivity Standard 1413- Refrigerator		
Conductivity Standard 12880- Refrigerator,		
Colilert- In Office		
Colilert Comparator- Freezer		

* 1413 is normally the single point calibrant, with the others used to check linearity of calibration.

8. Appendix A: Blank Job Hazard Analysis Form

JOB HAZARD ANALYSIS **(CERTIFICATION OF HAZARD ASSESSMENT - 29 CFR 1910.133)** **U.S. National Park Service**	**JOB ACTIVITY:** **DATE:** **PREPARED BY:** **CERTIFIED BY:**	
PERSONAL PROTECTIVE EQUIPMENT REQUIRED:		**QUALIFICATIONS, EXPERIENCE, OR TRAINING REQUIRED:**
BASIC JOB STEPS	**HAZARDS**	**SAFE JOB PROCEDURE**
Break work down to basic elements (such as remove, lift, carry, stop, start, apply, return, squeeze, weld, saw, walk, hold, grind, place, etc.). Describe **what** is done, not how it is done.	For each job step, state what accident could occur and/or what hazard is present. To determine this, ask yourself if the person could fall; overexert; be exposed to burns, fumes, rays, gas, etc.; hit against, be struck by, come in contact with, be caught in, on, or between something?	State how each element of work should be performed to prevent the accident or avoid the hazard. What should the person do or not do? Be specific. What precautions should be taken? Ask yourself what you can do to eliminate, modify, guard, identify, or protect against the potential hazard or accident, including such things as how the worker stands, holds, uses, carries, dresses, etc.

Add additional rows as needed.

9. Appendix B: Suggested topics to be addressed in a Job Hazard Analysis

See Appendix E for more background on hazards around water identified by the USGS.

The following list of topics represents a suggested minimum that should be expanded with knowledge of specific sites and through experience by the NCPN water quality field staff.

General job activity	Basic job step	Potential hazards
All field activities	Environmental conditions	Temperature and sun exposure
		Adverse weather (rain, snow, lightning and wind)
		Hazardous animals and plants
Accessing sites	Highway driving	Varied road conditions Fatigue Behavior of other drivers Animals and other obstructions
	Driving on unimproved roads	Rough and rutted roads
		Muddy roads
		Narrow roads with poor site distances
	Winter driving	Slippery roads and poor visibility
	Hiking	
Flow measurement and Sample collection	Wading	Swift currents and deep water
		Flood flows
		Cold water and ice
		Contaminated water
	Walking on stream banks	Slick footing
		Adjacent swift currents and deep water
Sample processing	Acidifying samples	Exposure to acid
	Filtering samples	Mechanical pumps

It is assumed that qualified field staff members are proficient in the safe use of water quality monitoring equipment. If, in the judgment of the staff or supervisor, this is not the case, training will be arranged to ensure that the equipment can be used, and the tasks accomplished, safely.

10. Appendix C: Example NCPN Job Hazard Analysis for Field Activities

JOB HAZARD ANALYSIS (CERTIFICATION OF HAZARD ASSESSMENT - 29 CFR 1910.133) **U.S. National Park Service**	**JOB ACTIVITY: Water Quality Field Work** **DATE: 5/8/06** **PREPARED BY: David Thoma** **CERTIFIED BY: Jeff Stock BRCA safety officer / signature on file**

PERSONAL PROTECTIVE EQUIPMENT REQUIRED: Warm clothes, sun and rain protection, insect repellent, life vest, waders, communication equipment, shovel, and jack.	**QUALIFICATIONS, EXPERIENCE, OR TRAINING REQUIRED:** Basic backcountry competence

BASIC JOB STEPS	**HAZARDS**	**SAFE JOB PROCEDURE**
Break work down to basic elements (such as remove, lift, carry, stop, start, apply, return, squeeze, weld, saw, walk, hold, grind, place, etc.). Describe **what** is done, not how it is done.	For each job step, state what accident could occur and/or what hazard is present. To determine this, ask yourself if the person could fall; overexert; be exposed to burns, fumes, rays, gas, etc.; hit against, be struck by, come in contact with, be caught in, on, or between something?	State how each element of work should be performed to prevent the accident or avoid the hazard. What should the person do or not do? Be specific. What precautions should be taken? Ask yourself what you can do to eliminate, modify, guard, identify, or protect against the potential hazard or accident, including such things as how the worker stands, holds, uses, carries, dresses, etc.
1. Highway driving	a) Varied road conditions and road hazards such as animals and other obstructions b) Fatigue c) Behavior of other drivers	Be well rested before road trips and constantly alert to obstructions and behavior of other drivers. Sleep if over tired on the way home after long days.
2. Dirt road driving	a) Rough and rutted roads, high-clearance driving b) Muddy roads c) Narrow roads with short sight distances d) Animals and other obstructions	Be well-rested before road trips and constantly alert to obstructions and behavior of other drivers. If overtired, stop to sleep on the way home after long days.
3. Inclement weather driving	a) Slippery roads and poor visibility	Drive slowly. Watch for snow, ice, ruts, washed-out roads, washboard surfaces, loose gravel, and flash floods. Be well-rested before road trips and constantly alert to obstructions and behavior of other drivers. If overtired, stop to sleep on the way home after long days.
4. Hiking	a) Steep, loose terrain b) Uneven footing c) Long distances	Wear sturdy shoes and don't rush on uneven terrain. Distribute weight low and close to back in backpack. Rest frequently and drink lots of water. Avoid hiking during mid-day in summer. Adjust work schedule accordingly. Be wary of dangerous animals.
5. Stream access	a) Swift currents, deep water b) Flood flows c) Cold water and ice d) Contaminated water	Approach streams cautiously. Don't enter stream if visual estimate of product of depth and velocity is >10 (depth in ft and velocity in ft/s). Be wary of slippery and loose stream beds and dangerous animals.
6. Deal with environmental conditions	a) Temperature and sun exposure b) Adverse weather (rain, snow, lightning, and wind)	Wear sun protection and have appropriate extra clothing in case weather changes. Keep to low areas during lightning. Watch for flash floods.

11. Appendix D: NCPN Job Hazard Analysis for Laboratory Activities

JOB HAZARD ANALYSIS (CERTIFICATION OF HAZARD ASSESSMENT - 29 CFR 1910.133) **U.S. National Park Service**	**JOB ACTIVITY: Water Quality Laboratory Sample Processing** **DATE: 5/8/06** **PREPARED BY: David Thoma** **CERTIFIED BY: Jeff Stock BRCA safety officer / signature on file**

PERSONAL PROTECTIVE EQUIPMENT REQUIRED: Rubber gloves, cloth apron, ventilated room, eye protection, wipes for acid and solvent spills, chemical storage cabinets, appropriately labeled bottles and containers, pressure cooker and hotplate for disposal of bacteria cultures.	**QUALIFICATIONS, EXPERIENCE, OR TRAINING REQUIRED:** Basic laboratory experience

BASIC JOB STEPS	**HAZARDS**	**SAFE JOB PROCEDURE**
Break work down to basic elements (such as remove, lift, carry, stop, start, apply, return, squeeze, weld, saw, walk, hold, grind, place, etc.). Describe **what** is done, not how it is done.	For each job step, state what accident could occur and/or what hazard is present. To determine this, ask yourself if the person could fall; overexert; be exposed to burns, fumes, rays, gas, etc.; hit against, be struck by, come in contact with, be caught in, on, or between something?	State how each element of work should be performed to prevent the accident or avoid the hazard. What should the person do or not do? Be specific. What precautions should be taken? Ask yourself what you can do to eliminate, modify, guard, identify, or protect against the potential hazard or accident, including such things as how the worker stands, holds, uses, carries, dresses, etc.
1. Wash and prepare field sampling equipment	a) Eye and skin irritation from detergent b) Standing one place for long periods of time	Wear eye protection, rubber gloves, apron, know how to clean spills, neutralize acid in baking soda before disposal, work with exhaust fan running, take frequent breaks, comfortable shoes for standing, wash hands after processing
2. Calibrate multiparameter probe	a) Standing one place for long periods of time b) Eye and skin irritation from Cobaltus Chloride, 10%NO3 cleaning solution, Sodium Sulfite, pH and conductivity standards c) Cobaltus Chloride is a possible human carcinogen d) Inhalation of acid fumes	Wear eye protection, rubber gloves, apron, know clean up acid spills, work with exhaust fan running, take frequent breaks, comfortable shoes for standing, wash hands after processing. Do not allow cobaltus chloride or sodium sulfite to become airborne
3. Process field surface water samples	a) Back strain from lifting heavy cooler with samples and ice	Lift with legs, not back.
4. Process bacterial samples	a) Danger of ingesting bacterial media b) Potential inoculation or ingestion of bacterial colonies after incubation	Wear rubber gloves, eye protection, and apron. Do not puncture bacteria containing plates after incubation, remove flammables from work area, do not let pressure cooker run dry, run exhaust fan during sterilization procedure. Keep bacteria plates in biohazard bags after counting colonies. Wash hands vigorously after handling bacteria plates.

12. Appendix E: Safety Considerations in Field Activities

Excerpts from *USGS National Field Manual*, Chapter A9, "Safety in field activities," by Susan L. Lane and Ronald G. Fay (10/1997), are presented below as a useful reference when planning for field safety in the collection and processing of water quality samples. The intent of adding this section as an appendix is to help with development of JHAs.

Some sections are omitted, for instance, where the activities described will not occur in NCPN parks, or where the hazards described are so site-specific (such as hazardous plants or animals) that the general information provided is of little utility.

9.1 SAFETY POLICIES, REGULATIONS, AND REQUIREMENTS (omitted)

9.1.2 JOB HAZARD ANALYSIS

Many accidents and injuries can be prevented by analyzing and communicating hazards. A job hazard analysis (JHA) identifies hazards and describes actions to avoid mishaps. For example, JHAs are used to determine and document the proper personal protective equipment required for the job. JHA program requirements are given in DOI Departmental Manual 485, chap. 14, and in USGS Handbook 445-2-H, chap. 2.

JHA components include:

- The sequence of steps associated with an activity.
- Identification of hazards involved for each step.
- Determination of controls needed to eliminate or reduce the hazard.

Visit the field site, if possible, to be sure that you and your equipment can get to, sample at, and leave the site safely. Talk to employees who may have visited the site—you might receive your most valuable safety information from them. **Always check the study workplan, field folder and site records, and other available information before departure.**

9.1.3 PERSONAL PROTECTIVE EQUIPMENT

Personal protective equipment (PPE) is defined as safety equipment for your skin, eyes, ears, face, head, extremities, and respiration (see USGS Handbook 445-1-H, chap. 9 and OSHA 29 CFR 1910, Subpart I). **The USGS is required to supply PPE, and you are required to use it!**

Proper PPE selection is based on the hazards likely to be encountered, as well as compliance with regulatory safety requirements. Examples of PPE are cited throughout this chapter (see sections 9.4, 9.6, and 9.11.1).

9.1.4 TRAINING

Field personnel must be aware of safety training requirements and certification or recertification needed to perform specific tasks. For example,

- Personnel who operate government-owned or leased vehicles must be certified and periodically recertified in defensive driving.
- Cardiopulmonary resuscitation (CPR) and other first-aid certification and recertification are mandatory for field personnel.

Safety training and certification requirements, recommendations, and opportunities are subject to change—check with your safety officer periodically to keep current with prevailing requirements.

9.2 FIELD TRIP PREPARATIONS AND EMERGENCY CONTACTS

Before leaving for the field, obtain the information shown on figures 9-1 and 9-2, and carry this with you in the field—either at the front of Chapter A9 or in the field folder. Put Chapter A9, or the sections of it that are applicable to your work, in a binder and keep it with you. **Become familiar with the information provided here before you are in the field and confronted with an emergency.**

Certain types of data-collection activities require a field team of two or more people (Horowitz and others, 1994). While the multi-person field team also might be important to carrying out technical procedures, the field team or buddy system can be a lifesaving practice. Mobile telephones provide a minimum precaution if personnel will be unaccompanied. Satellite communication services might be available for remote areas where cellular service is not available.

Basic preparations that should become routine before every sampling activity:

- Use safety checklists when preparing for a field trip. Examples of checklists are in section 9.11. Develop your checklists from existing site safety information and site reconnaissance (job hazard analysis or site safety plan).
- Keep a field folder for each surface-water and ground-water site at which water-quality data will be collected. The safety-related contents of a field folder include:
    - Copies of the checklists mentioned above.
    - Site type (hazardous waste, confined space, cableway, wading site, bridge site, boat site) and site description.
    - Site location (include map, site sketch, and description).
    - Locations and phone numbers of emergency facilities, such as a hospital or first aid station, police and fire departments, utility companies.
    - Additional information specific to the site: for example, if it is open to hunting, and season dates; appropriate clothing (such as orange safety vests).
- Make an itinerary for every field trip and leave a copy at the office and with family or colleagues. Schedule times to check in at work and with family or colleagues when field trips require overnight stays. Follow the established schedule. Notify all concerned parties if your schedule changes.
- Obtain or reserve communication equipment, such as a cellular phone or two-way radio.

Emergency Contacts

Personal contacts

Name: _____ Phone: (home) _____(work) _____

Name: _____ Phone: (home) _____(work) _____

U.S. Geological Survey contacts

District Office: _____

Safety Officer: _____

National Water Quality Laboratory, Arvada, CO.

Emergency coordinator (303) 467-8000

Local emergency contacts (or call 911)

Hospital _____ Phone: _____

Address: _____

Other medical facility (24-hour care)__ Phone: _____

Address: _____

Police _____

Fire _____

Utility _____

Health Information Centers

Center for Disease Control

Information Hotline: (404) 329-3311

Fax: Disease Directory: (404)-332-4565

Other

_____

_____

Figure 2. Example of emergency contact form to be completed and taken on field trips.

9.3 TRANSPORTATION

Various modes of transportation are used to reach field sites. Each type of transportation comes with specific guidelines for safe operation, for which appropriate training must be completed before leaving for the field. Training requirements are described in the USGS Handbook 445-2-H and are provided by office or regional safety officials. Perform a safety inspection on all transportation equipment before using. Safety inspection checklists are found in USGS Handbook 445-1-H, chap. 12.

9.3.1 ROAD VEHICLES AND TRAILERS

Passenger cars, vans, and 4-wheel-drive vehicles can differ in operation and in safety features, depending on the vehicle's age, make, and model. Become familiar with all operating systems of the vehicle before you leave for the field—test operation of lights, locks, seat belts, windows, 4-wheel drive and winch, and hood release.

"Musts" that apply to all USGS employees driving government vehicles:

- Have a valid U.S. State driver's license.
- Take a course in driver safety—the current requirement is to take the course every 3 years.
- Inspect all vehicles before use.
- Tie down or otherwise secure all cargo.
- Wear a seat belt.
- Obey all traffic regulations and operate your vehicle safely.

Inspect each vehicle thoroughly before departing from the office (USGS Handbook 445-I-H, chap. 12): discovering that the 4-wheel drive is not working when you are in the backwoods can result in considerable inconvenience and place you in a dangerous situation.

- Check that your vehicle is equipped with the proper safety equipment (see 9.11.2).
- Create checklists specific to each vehicle and (or) each type of sampling trip to make the inspection process routine.
    - Towing a trailer or equipment with your vehicle requires additional planning, caution, and proficiency in driving.
    - Vehicles and trailers vary in maximum load capacity.
    - Vehicles and trailers must be compatible and have a matching hitch ball and coupling.
    - Trailers are required to have standard safety equipment such as safety chains (placed in an "x" under the coupling), lights, brakes (if applicable), and load-securing devices.
    - Be aware that air turbulence from passing vehicles can cause your trailer to sway.
    - Before towing, consult USGS Handbook 445-1-H, chap. 14, p. 14-5, for additional guidance.
- Drive more slowly.
- Disengage the vehicle's automatic "overdrive gear" option when towing.
- Maintain extra following and stopping distance to allow for the increased load. Loads must be balanced.
- **Do not exceed the maximum recommended load capacity.** (Check owner's manuals or contact vehicle or trailer manufacturers for this information).

Obtain the training that will help you handle potentially dangerous driving conditions; for example, wet, icy, or snow-filled roads; high wind velocity; glare from bright sunshine; poor visibility from dust storms, fog, or heavy precipitation. In very hot climates, keep the windows slightly open when the vehicle is parked to avoid shattering windows from the heat. **To avoid asphyxiation from carbon monoxide, never sit or work in a closed vehicle with the engine running for more than a few minutes.**

9.3.2 WATERCRAFT

Boats are used extensively by USGS personnel for sampling the quality of water in rivers and lakes. All boats must carry equipment as required by the U.S. Coast Guard. Checklists are useful for ensuring that all the proper equipment is in place (see 9.11.3). WRD Memorandum No. 96.25 and the DOI Departmental Manual 485 provide regulations for watercraft safety.

Head back to harbor in the event of a storm. You are required to follow U.S. Coast Guard regulations for waterway safety and etiquette. Carry extra lines, foul-weather gear, and backup equipment. Remember, all cargo must be secured. For additional information see USGS Handbook 445-1-H, chap. 17, and USGS Handbook 445-2-H, chap. 16.

9.3.3 AIRCRAFT

Airplanes and helicopters are sometimes used for sampling trips to remote areas. Pilots and passengers must follow stringent guidelines when using these means of transportation. Any sampling activity involving the use of aircraft must be carefully planned and coordinated with USGS safety officials, the DOI Office of Aircraft Services, and appropriate local and regional safety officials, as described in USGS Handbook 445-1-H, chap. 16, and USGS Handbook 445-2-H, chap. 13. WRD Memorandum No. 95.49 provides an example of a safety plan for aircraft use.

9.3.4 OTHER METHODS OF TRANSPORTATION

Additional methods of transportation include snowmobiles, all terrain vehicles, horses or mules, and backpacking. Evaluate and practice these methods before use, taking care to consider the measures necessary for a safe trip and making sure to obtain proper training. USGS Handbook 445-1-H, chap. 14, and USGS Handbook 445-2-H, chap. 14, provide more detailed information.

9.4 SURFACE-WATER ACTIVITIES

Collection of water-quality samples can be as simple as hand dipping water from a slow moving stream to as complex as using heavy power equipment while working from a cableway. Know the safety steps needed for any sampling method used.

9.4.1 WADING

Examine the section of a stream or river you plan to wade. Check the field folder for information relating to safety, including maximum depths in relation to stage, wading-section anomalies such as slippery conditions and drop-offs or holes (a wading rod can be used to help assess streambed conditions), and velocity curves for determining wadable stages. **Do not attempt to wade a stream for which values of depth multiplied by velocity equal or exceed 10 ft^2/s.** For example, a stream only 2 ft deep but with velocities of 5 ft/s or more can be dangerous to wade.

While wading to collect a water sample:

- Wear a personal flotation device (PFD) during wading activities.
  - Approved PFDs for wading include the standard jacket type and the suspender type. The PFD must fit properly, be rated for your weight, and be in good condition.
  - The PFD should be dried and kept indoors between trips.
- Wear hip boots or chest waders. Boots and waders provide protection from cold and pollutants, as well as from underwater objects. Be aware of the possibility of slipping and going underwater (feet up, head down) while wearing them. Practice wearing hip boots and waders in a controlled, group-training situation before using for field work. The following recommendations are the result of experiments with boots (Joseph, 1957) and field experience.
  - Hip boots with a strap at the top are better than boots that are open. The strap should be pulled closed. This allows air to be trapped in the boot in case you are submerged. The air cushion can be used as a partial mechanism for flotation until you reach shore or are rescued.

- Avoid hip boots with tight ankles. These are difficult to remove in an emergency situation.
- Avoid chest waders that are tight fitting at the top. Like tight-ankled hip boots, they are difficult to remove in an emergency situation. Whenever chest waders are worn, a PFD also must be worn.

- Be aware of surrounding conditions.
  - Watch for debris floating downstream, such as logs, aquatic vegetation, or "rafts" of animals seeking higher ground.
  - Watch for sand channels that can shift under foot and become quicksand.
  - Watch the stream stage, especially when it could rise rapidly.
  - When wading below a dam or control structure, contact the gate operator before entering the stream.

9.4.2 WORKING ON BRIDGES

A bridge safety plan is required by WRD Memorandum No. 95.17. For every bridge site, develop and diagram a detailed procedure that conforms to State and Federal regulations. Keep this procedure and diagram in the field folder. The bridge procedure includes plans for safety cones and signs, lane blockage, and traffic control.

Equipment used for sampling from a bridge can be heavy and unwieldy. Practice assembling and using the equipment before starting field activities, and make sure the equipment is operational before leaving the office. When using a bridge crane, keep a pair of heavy-duty wire cutters readily available for cutting the cable in case debris snags the sounding line.

Be aware of boat traffic. The bridge crane cable should have strips of bright plastic flagging attached in intervals to make it easily visible to all boat traffic.

- **A PFD must be worn when working on bridges.** The suspender type PFD is approved for bridge work.
- Workers involved in peripheral activities should wear high-visibility vests with reflective tape.

9.4.3 WORKING FROM BOATS

Safety regulations for the various types of boats used by the USGS when obtaining water-quality samples are comprised of U.S. Coast Guard (USCG) and OSHA rules, and are supplemented by USGS requirements.

Before working from a boat, obtain the appropriate training for the vessel being used, as specified in WRD Memorandum No. 96.25. This training should cover all the specifics regarding boat operation as per USCG regulations. Before taking a boat on the water, ensure that the vessel is in operating condition. Boats are to be inspected annually. If a vehicle is being used to trailer the boat to the site, the vehicle and trailer are to be included in the preliminary inspection. Equip the boat with all USCG-required equipment (see the checklist in 9.11.3) as well as items that the study team deems appropriate for emergencies or equipment failures. Some Districts[1] keep a duffel bag on each boat stocked with standard emergency supplies such as flashlights, air horns, and whistles.

A float plan for each field trip must be left with the supervisor or other designee. The plan should include, at a minimum:

- Date and purpose of trip.
- Name(s) of operator(s) and name(s) of passengers.
- Destination and route to be taken.

- Time of departure and estimated time of return.
- Radio frequency or cellular designation, if applicable.
- Type of watercraft, and its length, color, identification number, and other unique features.

[1]The term “District” as used in this report refers to an organizational unit of the USGS located in any of the States or Territories of the United States.

Each person on the boat must wear an orange USCG-approved PFD, which should have at least 200 cm2 of retroreflective material on the front, back, and reversible sides. Inspect all PFDs for damage before and after each use. Store PFDs properly and discard damaged PFDs.

- Do not wear hip boots or waders in a boat. Wearing boots or waders could be a safety hazard if the boat should tip or you are thrown out.
- **Don’t panic if you fall out of a boat!** Think through and execute the basic safety procedures that you have been taught.

Attach bright colored plastic flagging in intervals to the tag line used to delineate the transect along which samples will be collected, so as to make it easily visible to boat traffic. In areas of frequent boat traffic, station a member of the field team on shore who can drop the tag line rapidly, should it present a hazard to boats.

All boat passengers should be practiced in emergency procedures and the location of emergency equipment. Boat operators are required to have current training in first aid and CPR. They must also know how to use the type(s) of fire extinguisher(s) on the watercraft.

9.4.4 WORKING FROM CABLEWAYS (omitted)

9.4.5 SCUBA DIVING (omitted)

9.4.6 ELECTROFISHING (omitted)

9.5 GROUNDWATER ACTIVITIES (omitted)

9.6 CHEMICALS

USGS employees are routinely exposed to chemicals during the water-quality sampling process. Chemicals—as solids, liquids, or gases—range from dilute salt solutions to strong acids, bases, dyes, and organic compounds. Field measurements and the processing of sample water can cause chemical reactions that generate dangerous fumes and by-products.

Be cognizant of the regulations that govern the use, transportation, and disposal of chemicals and wastes. Because regulations vary greatly from state to state, contact your safety officer or state agency for the proper procedures in your locality.

9.6.1 USE AND HANDLING

For safe use of chemicals, follow the guidelines given below, communicate hazards to all members of the field team, use proper personal protective equipment, and apply common sense when working with dangerous substances.

- **Obtain information about the chemical or compound.**
  - Material Safety Data Sheets (MSDS) supply material characteristics such as chemical description, fire and explosion data, chemical compatibility and reactivity, protection precautions, and spill procedures. These documents are required by OSHA 29 CFR 1910.1200 (hazard communication) and usually are shipped with chemicals when purchased. Your safety officer or chemical supplier also can provide the MSDS needed.
  - Chemical safety information is available from many Federal and private sources. For example, the Permissible Exposure Limit (PEL) of a given chemical is listed by OSHA

29 CFR 1910.1000, subpart Z, and in the Pocket Guide to Chemical Hazards published by the National Institute for Occupational Safety and Health (June 1994).

- DOI and USGS internal memorandums describe chemical usage and handling policies of our most commonly used chemicals. For example, refer to:

  DOI Departmental Manual 485, chap. 29, for general guidelines on chemical safety in laboratories.

  USGS Handbook 445-2-H, chap. 7, for Hazard Communication Program goals.

  WRD Memorandums No. 93.44, 94.06, and 94.07 for storage, transportation, handling, and disposal of formaldehyde, hydrochloric acid, and methyl alcohol, respectively.

  Office of Water Quality Technical Memorandums 94.02 and 94.16 for information on discontinuing field use of mercury-filled thermometers and mercury-based sample preservative, respectively.

- **Use Personal Protective Equipment (PPE) (see sections 9.1.3 and 9.11.1).**

  - Wear protective clothing, including gloves, glasses, lab coat, and if necessary, use a respirator. Remember that you must be certified before using a respirator (see USGS Handbook 445-2-H, chap. 5).

  - Select PPE appropriate to the chemical(s) to be contacted. PPE can be designed for specific chemicals. For example:

    Respirator cartridges are ineffective if they fail to filter the chemical hazard encountered.

    Wear gloves that will provide adequate protection. Gloves become ineffective if they are dissolved by the chemical you are using. Vinyl gloves are used for handling inorganic acids and bases. Latex gloves are used for handling relatively mild organic solvents, such as methanol and acetone; nitrile gloves can be used for some stronger organic solvents.

- **Use the following standard procedures.**

  - Avoid unnecessary exposures and spills. Never place chemical containers where they can be knocked over. For example:

    Clean up chemical residues or spills immediately and appropriately.

    Keep chemical spill kits near the work area.

  - Work with adequate ventilation or under a hood when working with hazardous or reactive chemicals and gases.

  - Keep eye wash kits readily accessible while working with chemicals.

  - Handle and mix chemicals and compounds appropriately (check the MSDS). For example:

    When transferring flammable liquids, all metal containers must be grounded to eliminate igniting the liquid with an electrical spark.

    When preparing a hydrochloric or nitric acid cleaning solution, the sequence is to put water in the vessel first and then add the acid.

  - Open chemical containers slowly and carefully, wearing proper PPE. Allow hot containers to cool before opening. Open frozen or encrusted lids with caution. For example:

To open fused-glass ampules, break the ampule at the base of neck, in a direction away from you and others. Use an ampule breaker if it is safer for you, and wear gloves.

Check containers and ampules for contents lodged near the container top or neck. Dislodge trapped material by gently tapping the container at the top.

- Properly dispose of all parts of the spent ampule (Office of Water Quality Technical Memorandum 92.11). Temporarily store used ampules in an appropriate container. Do not let these wastes accumulate in your vehicle or work area—they produce corrosive and potentially explosive fumes.

9.6.2 TRANSPORT

The USGS must follow applicable Federal, DOT, EPA, and supplemental State guidelines when transporting chemicals and chemical wastes. References include the *Emergency Response Guidebook* (U.S. Department of Transportation, 1990) and the *Driver's Pocket Guide to Hazardous Materials* (J.J. Keller & Associates, 1993). USGS chemical transportation guidelines are summarized below.

The USGS transports small quantities of chemicals for use in field and office-laboratory procedures. The USGS is not considered a commercial carrier which, by definition, transports materials in commerce or in the furtherance of a commercial enterprise (transporting for profit).

Because the USGS transports small quantities of chemicals and wastes, the Federal EPA and DOT small-quantity exemption rules apply. According to these guidelines, the USGS is exempt from many industry handling and shipping regulations and training requirements. Check your State's laws on intrastate transport of small quantities of chemicals in a Federal government vehicle.

It is not necessary to placard your vehicle when transporting small quantities of chemicals. Placarding a vehicle informs enforcement and emergency response personnel that the vehicle contains large amounts of the placarded material. For their own safety, first-response teams might not immediately approach a wrecked vehicle that is placarded for a highly dangerous or reactive material.

For safe containment and transport of chemicals:

- Protect yourself and passengers in the vehicle by stowing and securing chemicals away from the passenger compartment and behind a safety screen or barrier.
- Stow compressed gas cylinders securely and in a vertical position, if possible. Never transport cylinders with regulators attached, or with safety caps missing.
- Try to transport chemicals in their original DOT-approved shipping containers with the package correctly sealed. If the original packaging is not available, use a container that will resist and contain the material in the event of an accident. Use of an overpack container, such as an ice chest, is recommended to further protect against breakage and spills.
- Carry spill kit(s) appropriate for the chemicals being transported.
- Label packages as to their contents, and remove old, inappropriate labels from containers that are being reused.
- Keep a chemical inventory list in the glove box or side door pocket of the vehicle. This list should also have an emergency contact name and phone number. Keep a copy of each chemical's Material Safety Data Sheet (MSDS) here, as well.
- Waste products should be transported in the same safe and approved manner as other hazardous materials. The container must be of adequate composition and strength and must be labeled with the type and concentration(s) of chemical(s).

9.6.3 STORAGE AND DISPOSAL

Chemicals require strict storage procedures for safety reasons, as well as to avoid chemical degradation, volatilization, and contamination. Specific storage areas should be designated in your vehicle and office laboratory. Use appropriate, approved chemical storage cabinets. Never store incompatible materials together. Refer to USGS Handbook 445-1-H, chap. 8, for chemical compatibility and storage.

- Separate storage is needed for acids, corrosives, and flammables.
- High purity, laboratory-grade flammables should be stored apart from other flammables, such as gasoline and solvents.

Chemical and waste disposal requirements, and exemptions, are regulated federally by the EPA. Supplemental State guidelines must also be followed. Contact your safety officer or waste disposal representative for details, and follow these general disposal guidelines:

- Do not indiscriminately dump chemicals or pathogens down sinks, toilets, or drains.
- Do not let chemical wastes accumulate in your vehicle, office, or laboratory. Waste disposal storage areas should be established and used.
- Do not mix incompatible wastes. Waste disposal companies have strict and costly requirements for accepting mixed wastes.
- Always label the storage container of a waste product to indicate its contents.
- Put chemical containers, such as spent ampules containing chemical preservative, in chemically separated storage containers and dispose of properly. Instructions are given in Office of Water Quality Technical Memorandum 92.11, and in Horowitz and others (1994).
- The dilute acid solution used for equipment cleaning can be prepared for safe disposal by following procedures described by Horowitz and others (1994, p. 12).

9.7 CONTAMINATED WATER

Water being sampled could be contaminated with pathogens and hazardous chemicals. Use caution and extra protection when working with water known or suspected to contain high concentrations of pathogens. Sample containers, shipping containers, and paperwork must indicate the type and severity of the contamination. This alerts personnel to the appropriate personal protective equipment and procedures needed. **Communicate known or suspected contamination to all personnel who could come in contact with the sample.**

Waterborne, disease-causing organisms (pathogens) are found in nearly all surface-water systems, and occur in some ground-water systems as well. Most pathogens originate from the body fluids and feces of animals and humans. Pathogens enter surface-water resources primarily through sewage discharges and spills, storm and agricultural runoff, and direct contact. Microorganisms also are transported on small particles such as dust or aerosols (gaseous suspension of very fine particles). Pathogens enter ground water through infiltration from septic tank effluent, leachate from fields and ponds, and from faulty well seals and casings. Bacteria, viruses, and other pathogenic organisms can occur in the most pristine environments. **Never drink sample water, no matter how pristine the environment appears.**

To minimize exposures to and effects from contaminated water:

- Receive required inoculations. The USGS immunization program requires inoculations for field personnel working around polluted water (WRD Memorandum 96.29). Employees receive inoculations for waterborne pathogens such as typhoid, tetanus, hepatitis, polio, and rabies at USGS expense. Contact your safety officer about receiving appropriate inoculations before you sample.
- Use personal protective equipment, including respiratory equipment (certification required) when working over turbulent, polluted flows, and in shelters containing evidence

of excrement (see WRD Memorandums 94.30 and 95.06). Pathogens can enter your body through many openings such as your mouth, eyes, nose, cuts, scrapes, or chapped skin.

- Wear rubber boots, coveralls or aprons, gloves, and splash protection (a disposable dust mask offers splash and dust protection at a very low cost).
- Do not ingest pathogens or other contaminants. Never eat or drink while sampling or put pencils or other items in your mouth, and do not store food or drink in sample coolers.
- Carry antibacterial soap; wash before leaving the site. Remember to wash again after unloading supplies.
- Disinfect all contaminated surfaces as soon as possible.
- Handle bacteria plates carefully and autoclave them before disposal. An aseptic technique for bacteria enumeration is described in Chapter A7.1 of this *National Field Manual*.

9.8 ENVIRONMENTAL CONDITIONS (omitted)

9.9 ANIMALS (omitted)

9.10 PLANTS (omitted)

9.11 CHECKLISTS FOR STANDARD SAFETY EQUIPMENT

Checklists are helpful for ensuring that personnel have the appropriate safety equipment available during field trips. Each study team needs to consider the specific needs for their work and should customize these checklists as necessary.

9.11.1 CHECKLIST FOR PERSONAL PROTECTIVE EQUIPMENT

✓	Personal Protective Equipment[1]
	Chemical and disease protection
	Aprons
	Eye/Face splash guards
	Gloves (vinyl and/or latex or nitrile). Sizes: S M L XL
	Protective suits. Sizes: S M L XL
	Respirators (certification required for use)
	Climatic and UV protection
	Boots
	Fluids (for example, water and sports drinks)
	Hat, wide-brimmed
	Insect repellent (unscented)
	Rain gear
	Sunglasses
	Sunscreen
	Temperature-modifying clothing
	Flotation and reflective protection
	Orange flotation vests and jackets
	Safety harness
	Protection for working around heavy objects and machinery
	Back belt
	Hardhat
	Hearing protection
	Safety glasses
	Steel-toed safety boots
	Work gloves

[1] PPE must be selected based on the hazards likely to be encountered. The USGS is required to supply appropriate PPE, and field personnel are required to use it.

9.11.2 CHECKLIST FOR VEHICLES AND VEHICULAR LABORATORIES

✓	Items for Field Vehicles
	Chemical protection and storage
	Chemical spill kit
	Eye wash kit (replace old or expired wash solution)
	Material Safety Data Sheets (MSDS)
	Chemical reagents (stored in appropriate area)
	Flammable solvents (stored in appropriate dedicated area)
	Pressurized gases (stored in appropriate area)
	Communications and instructions
	Field folder (including maps, emergency phone numbers for medical facilities, office contacts, family contacts)
	Cellular phone/communication equipment (check that the service is operational for the area to be traveled)
	First aid and protective equipment
	Complete change of clothes (stored in dry area)
	Fire extinguisher (safely secured)
	First aid kit and manual (check for missing or old, expired items and replace if necessary)
	Orange reflective vest
	Miscellaneous equipment
	Bungee cords (to secure loose articles)
	District flood plan (most current version)
	Flagging
	Flares
	Flashlight (including fresh batteries)
	Flexible hose (to vent exhaust away from vehicle)
	Safety cones
	Tool kit

13. Appendix F: Vehicle Accident Form

MOTOR VEHICLE ACCIDENT REPORT	Please read the Privacy Act Statement on Page 3.	INSTRUCTIONS: Sections I thru IX are filled out by the vehicle operator. Section X, Items 72 thru 82c are filled out by the operator's supervisor. Sections XI thru XIII are filled out by an accident investigator for bodily injury, fatality, and/or damage exceeding $500.

SECTION I - FEDERAL VEHICLE DATA

1. DRIVER'S NAME *(Last, first, middle)*	2. DRIVER'S LICENSE NO./STATE/LIMITATIONS	3. DATE OF ACCIDENT

4a. DEPARTMENT/FEDERAL AGENCY PERMANENT OFFICE ADDRESS	4b. WORK TELEPHONE NUMBER
	( )

5. TAG OR IDENTIFICATION NUMBER	6. EST. REPAIR COST	7. YEAR OF VEHICLE	8. MAKE	9. MODEL	10. SEAT BELTS USED
	$				☐ YES ☐ NO

11. DESCRIBE VEHICLE DAMAGE	

SECTION II - OTHER VEHICLE DATA *(Use Section VIII if additional space is needed.)*

12. DRIVER'S NAME *(Last, first, middle)*	13. DRIVER'S LICENSE NUMBER/STATE/LIMITATIONS

14a. DRIVER'S WORK ADDRESS	14b. WORK TELEPHONE NUMBER
	( )

15a. DRIVER'S HOME ADDRESS	15b. HOME TELEPHONE NUMBER
	( )

16. DESCRIBE VEHICLE DAMAGE	17. ESTIMATED REPAIR COST
	$

18. YEAR OF VEHICLE	19. MAKE OF VEHICLE	20. MODEL OF VEHICLE	21. TAG NUMBER AND STATE

22a. DRIVER'S INSURANCE COMPANY NAME AND ADDRESS	22b. POLICY NUMBER
	22c. TELEPHONE NUMBER ( )

23. VEHICLE IS	24a. OWNER'S NAME(S) *(Last, first, middle)*	24b. TELEPHONE NUMBER
☐ CO-OWNED ☐ RENTAL ☐ LEASED ☐ PRIVATELY OWNED		( )

25. OWNER'S ADDRESS(ES)

SECTION III - KILLED OR INJURED *(Use Section VIII if additional space is needed.)*

A	26. NAME *(Last, first, middle)*			27. SEX	28. DATE OF BIRTH
	29. ADDRESS				
	30. MARK "X" IN TWO APPROPRIATE BOXES ☐ KILLED ☐ INJURED ☐ DRIVER ☐ HELPER ☐ PASSENGER ☐ PEDESTRIAN	31. IN WHICH VEHICLE ☐ FED ☐ OTHER (2)	32. LOCATION IN VEHICLE	33. FIRST AID GIVEN BY	
	34. TRANSPORTED BY	35. TRANSPORTED TO			
B	36. NAME *(Last, first, middle)*			37. SEX	38. DATE OF BIRTH
	39. ADDRESS				
	40. MARK "X" IN TWO APPROPRIATE BOXES ☐ KILLED ☐ INJURED ☐ DRIVER ☐ HELPER ☐ PASSENGER ☐ PEDESTRIAN	41. IN WHICH VEHICLE ☐ FED ☐ OTHER (2)	42. LOCATION IN VEHICLE	43. FIRST AID GIVEN BY	
	44. TRANSPORTED BY	45. TRANSPORTED TO			

46. Pedestrian	a. NAME OF STREET OR HIGHWAY	b. DIRECTION OF PEDESTRIAN *(SW corner to NE corner, etc.)* FROM	TO
	c. DESCRIBE WHAT PEDESTRIAN WAS DOING AT TIME OF ACCIDENT *(Crossing intersection with signal, against signal, diagonally; in roadway playing, walking, hitchhiking, etc.)*		

NSN 7540-00-634-4041
Previous edition not usable

91-109

STANDARD FORM 91 PAGE 1 (REV. 2-93)
Prescribed by GSA - FPMR 101-38.6

SECTION IV - ACCIDENT TIME AND LOCATION *(Use Section VIII if additional space is needed.)*

47. DATE OF ACCIDENT	48. PLACE OF ACCIDENT *(Street address, city, state, ZIP Code; Nearest landmark; Distance nearest intersection; Kind of locality (industrial, business, residential, open country, etc.); Road description).*
49. TIME OF ACCIDENT AM PM	

50. INDICATE ON THIS DIAGRAM HOW THE ACCIDENT HAPPENED

Use one of these outlines to sketch the scene. Write in street or highway names or numbers.

a. Number Federal vehicle as 1, other vehicle as 2, additional vehicle as 3 and show direction of travel with arrow.

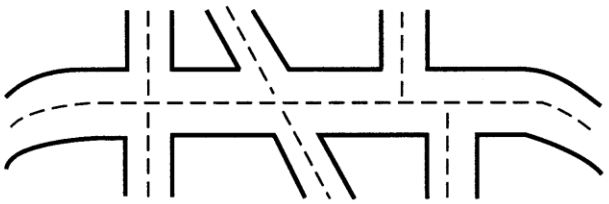
Example: 1 2

b. Use solid line to show path before accident 2 and broken line after the accident 2

c. Show pedestrian by

d. Show railroad by

e. Place arrow in this circle to indicate *NORTH*



51. POINT OF IMPACT *(Check one for each vehicle)*

FED	2	AREA
		a. FRONT
		b. R. FRONT
		c. L. FRONT
		d. REAR
		e. R. REAR
		f. L. REAR
		g. R. SIDE
		h. L. SIDE

52. DESCRIBE WHAT HAPPENED *(Refer to vehicles as "Fed", "2", "3", etc. Please include information on posted speed limit, approximate speed of the vehicles, road conditions, weather conditions, driver visibility, condition of accident vehicles, traffic controls (warning light, stop signal, etc.) condition of light (daylight, dusk, night, dawn, artificial light, etc.), and driver actions (making U-turn, passing, stopped in traffic, etc.).*

SECTION V - WITNESS/PASSENGER *(Witness must fill out SF 94, Statement of Witness) (Continue in Section VIII.)*

A	53. NAME *(Last, first, middle)*	54. WORK TELEPHONE NUMBER ( )	55. HOME TELEPHONE NUMBER ( )
	56. BUSINESS ADDRESS	57. HOME ADDRESS	
B	58. NAME *(Last, first, middle)*	59. WORK TELEPHONE NUMBER ( )	60. HOME TELEPHONE NUMBER ( )
	61. BUSINESS ADDRESS	62. HOME ADDRESS	

SECTION VI - PROPERTY DAMAGE *(Use Section VIII if additional space is needed.)*

63a. NAME OF OWNER	63b. OFFICE TELEPHONE NUMBER ( )	63c. HOME TELEPHONE NUMBER ( )
63d. BUSINESS ADDRESS	63e. HOME ADDRESS	
64a. NAME OF INSURANCE COMPANY	64b. TELEPHONE NUMBER ( )	64c. POLICY NUMBER ( )

65. ITEM DAMAGED	66. LOCATION OF DAMAGED ITEM	67. ESTIMATED COST $

SECTION VII - POLICE INFORMATION

68a. NAME OF POLICE OFFICER	68b. BADGE NUMBER	68c. TELEPHONE NUMBER
69. PRECINCT OR HEADQUARTERS	70a. PERSON CHARGED WITH ACCIDENT	70b. VIOLATION(S)

STANDARD FORM 91 PAGE 2 (REV. 2–93)

SECTION VIII - EXTRA DETAILS

SPACE FOR DETAILED ANSWERS. INDICATE SECTION AND ITEM NUMBER FOR EACH ANSWER. IF MORE SPACE IS NEEDED, CONTINUE ITEMS ON PLAIN BOND PAPER.

SECTION IX - FEDERAL DRIVER CERTIFICATION

In compliance with the Privacy Act of 1974, solicitation of the information requested on this form is authorized by Title 40 U.S.C. Section 491. Disclosure of the information by a Federal employee is mandatory as the first step in the Government's investigation of a motor vehicle accident. The principal purposes for using this information is to provide necessary data for legal counsel in legal actions resulting from the accident and to provide accident information/statistics in analyzing accident causes and developing methods of reducing accidents. Routine use of information may be by Federal, State or local governments, or agencies, when relevant to civil, criminal, or regulatory investigations or prosecutions. An employee of a Federal agency who fails to report accurately a motor vehicle accident involving a Federal vehicle or who refuses to cooperate in the investigation of an accident may be subject to administrative sanctions.

I certify that the information on this form *(Sections I thru VIII)* is correct to the best of my knowledge and belief.

71a. NAME AND TITLE OF DRIVER	71b. DRIVER'S SIGNATURE AND DATE

SECTION X - DETAILS OF TRIP DURING WHICH ACCIDENT OCCURRED

72. ORIGIN	73. DESTINATION

74. EXACT PURPOSE OF TRIP

	DATE	TIME *(Circle one)*		DATE	TIME *(Circle one)*
75. TRIP BEGAN		a.m. p.m.	76. ACCIDENT OCCURRED		a.m. p.m.

77. AUTHORITY FOR THE TRIP WAS GIVEN TO THE OPERATOR	78. WAS THERE ANY DEVIATION FROM DIRECT ROUTE
☐ ORALLY ☐ IN WRITING *(Explain)*	☐ NO ☐ YES *(Explain)*
79. WAS THE TRIP MADE WITHIN ESTABLISHED WORKING HOURS	80. DID THE OPERATOR, WHILE ENROUTE, ENGAGE IN ANY ACTIVITY OTHER THAN THAT FOR WHICH THE TRIP WAS AUTHORIZED.
☐ YES ☐ NO *(Explain)*	☐ NO ☐ YES *(Explain)*

81. COMPLETED BY DRIVER'S SUPERVISOR	a. DID THIS ACCIDENT OCCUR WITHIN THE EMPLOYEE'S SCOPE OF DUTY	b. COMMENTS
	☐ YES ☐ NO	

82a. NAME AND TITLE OF SUPERVISOR	82b. SUPERVISOR'S SIGNATURE AND DATE	82c. TELEPHONE NUMBER
		( )

STANDARD FORM 91 PAGE 3 (REV. 2-93)

SECTION XI - ACCIDENT INVESTIGATION DATA

83. DID THE INVESTIGATION DISCLOSE CONFLICTING INFORMATION. ☐ YES ☐ NO *(If "Yes", explain below.)*

84. PERSONS INTERVIEWED

NAME	DATE	NAME	DATE
a.		c.	
b.		d.	

85. ADDITIONAL COMMENTS *(Indicate section and item number for each comment.)*

SECTION XII - ATTACHMENTS

LIST ALL ATTACHMENTS TO THIS REPORT

SECTION XIII - COMMENTS/APPROVALS

86. REVIEWING OFFICIAL'S COMMENTS

87. ACCIDENT INVESTIGATOR	88. ACCIDENT REVIEWING OFFICIAL
a. SIGNATURE AND DATE	a. SIGNATURE AND DATE
b. NAME *(First, middle, last)*	b. NAME *(First, middle, last)*
c. TITLE	c. TITLE
d. OFFICE	d. OFFICE
e. OFFICE TELEPHONE NUMBER ( )	e. OFFICE TELEPHONE NUMBER ( )

*U.S. Government Printing Office: 1993 — 342-199/60088

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #2

Staff Training, NCPN Units in Colorado

Version 1.0 (4/10/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

This Water Quality Monitoring Standard Operating Procedure (SOP) describes the level of training for field and laboratory personnel conducting water quality sampling in NCPN Units in the state of Colorado. Laboratory analysis for Colorado parks is performed by the National Water Quality Laboratory operated by the USGS. All field measurements and sampling in these units are conducted using procedures and protocols consistent with those of the U.S. Geological Survey (USGS). Therefore, training will be in USGS methods.

The current staffing structure, and that for the foreseeable future, includes two levels of professional expertise and, therefore, two levels of training. The monitoring program is lead by a professional aquatic biologist or hydrologist at CURE/BLCA who is responsible for program management and quality control, in addition to field measurements and sampling and laboratory management. In addition, seasonal or temporary staff is hired each year under the supervision of the lead aquatic biologist/hydrologist to participate in field measurements and sampling, and laboratory support activities.

Table of Contents

1. Training Requirements 3
1.1. Lead aquatic biologist/hydrologist 3
1.2. Seasonal technicians 3
2. Appendix A: USGS Training Topics 4

1. Training Requirements

1.1. Lead aquatic biologist/hydrologist

The primary standard for training for the lead aquatic biologist/hydrologist is the two-week USGS training course entitled **Field Water-Quality Methods for Ground Water and Surface Water** (QW1028TC) or an equivalent. This course is generally offered twice per year in Lakewood, Colorado. Attendance by NPS staff is permitted on a space available basis. The course includes classroom and field demonstration of all aspects of surface and groundwater sampling. A complete listing of the topics covered and the typical amount of time devoted to each is shown in Appendix A.

In addition, this training will be supplemented with regular reference to this set of SOPs and the USGS National Field Manual for Water Data Collection, communication with the USGS office in Grand Junction. When partnering with USGS, they will provide 2 quality reviews annually of NPS field and laboratory techniques where USGS field monitoring staff accompany NPS staff on monitoring trips to observe, discuss and report on supplies, equipment and techniques used by the NPS staff. These must be found to be acceptable to the USGS in order for the data to be acceptable to the USGS as described in the NCPN Water Quality Monitoring Protocol.

Training in the Data Management needs specific to the NCPN will be acquired from existing NCPN staff including the NCPN hydrologist and Data Manager.

In the event of staff turnover in the lead position, replacement personnel will obtain the required USGS training as soon as practical. In the interim, training will be obtained through USGS field sampling personnel stationed in Grand Junction, Colorado, and other trained staff performing water quality monitoring for the NCPN. It is expected that new hires will arrive with basic knowledge of the principles of water quality monitoring, and field and laboratory skills in the operation of common water quality equipment. These elements will be included in the advertisement of vacancies and ranking of applicants.

1.2. Seasonal technicians

Seasonal technicians will be trained by the lead aquatic biologist/hydrologist at the beginning of each field season. This training will include, at a minimum, the following topics:

- Purpose and Objectives of Monitoring
- Field Safety (Travel, River, Lake and Boat)
- Lab Safety
- Preparing for the Field
- Preparing/Handling of Sample Bottles
- Field Measurements (Discharge, Turbidity, Secchi Depth)
- Multi-parameter Meters (pH, Alkalinity, Temp, DO, SC)
- Equipment Care, Cleaning & Calibration
- Sampling Theory
- Surface-Water Sample Collection
- Samplers and Splitters

- Data Entry and Sample Tracking
- Field and Laboratory Demonstrations
- Troubleshooting and Contingencies
- Reference Materials (USGS National Field Manual, NCPN Water Quality SOPs)

Oversight and monitoring by the lead aquatic biologist/hydrologist will continue through the season. Seasonal hires will be made with the expectation that new staff enter on duty with basic skills in field and laboratory procedures and safety, at a minimum, and some knowledge or experience in water quality sampling will be desirable.

2. Appendix A: USGS Training Topics

Field Water Quality Methods for Ground Water and Surface Water (QW1028TC). (As of August 2005 and subject to revision). Duration is 2-Weeks.

Training topic	Time devoted (Typical)
INTRODUCTION AND OVERVIEW	
Introduction	½ hour
Purpose and History of this Course	½ hour
Field Safety	1½ hours
Lab Safety (NWQL)	¼ hour
Water-Quality Information Sources & Public Relations	1 hour
FIELD MEASUREMENTS -- THEORY/DEMO/LAB EXERCISE/TROUBLE-SHOOTING/DISCUSSION	
Microbiology 1, 2 and 3	8 ½ hours
Preparing for the Field	1½ hours
pH, Alkalinity	4 hours
Temp, DO, SC, Turbidity	4 hours
Multi-parameter Meters	1½ hours
PREPARING FOR THE FIELD -- PAPERWORK, SAMPLE PROCESSING, SAFETY, LOGISTICS	
Tour of the USGS National Water Quality Laboratory	3½ hours
ASRs and Preparing Sample Bottle Sets	1½ hours
Surface-Water Field Folders	½ hour
Ground-Water Field Folders	½ hour
Water-Quality “Jeopardy” (quiz)	1¼ hours
EQUIPMENT CLEANING/BLANK PROCESSING	
Equipment Cleaning/Blanks-GW	4 hours
Equipment Cleaning-SW	1¾ hours
Processing Equipment/Field Blanks-SW	1¾ hours
SAMPLING THEORY	
Ground-Water Sampling	
Orientation, Concepts, Objectives, and Planning	1 hour
Well Construction and Development	1 hour
Well Sampling Equipment	1¼ hours
Solid Phase Extraction/Spikes/Sampling for Emerging Contaminants	2 hours
Field Activities and Preparations	1 hour
Sample Collection and Preservation	1 hour

Training topic	Time devoted (Typical)
Gas Sampling for Age Dating	1 hour
Surface-Water Sampling	
Sampling Methods	1 hour
Samplers and Splitters	¾ hour
How Samplers Work	1 hour
Site Selection	½ hour
Solid Phase Extraction/Spikes/Sampling for Emerging Contaminants	2 hours
Turbidity	1 hour
EWI/EDI Exercise	1 hour
Personal Field Computer Software (PCFF)	1 hour
Sampling Protocol - Equipment Demo/Hands-on	1 hour
FIELD TRIPS	
Field Trip Logistics	½ hour
Ground Water	8 hours
Surface Water	8 hours
Course Wrap-up	1 hour

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #3

Staff Training, Utah Units

Version 1.0 (4/10/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

The purpose of this SOP is to identify the primary areas of training required of NCPN personnel by the state of Utah for cooperation in the Utah water quality program. It is not intended as a substitute for training. It lists the general aspects of the UT training course and a brief description of each as well as the agreement between NCPN and the state.

The basic elements of training include a one-day course where sample collection and handling are covered by Department of Water Quality (DWQ) personnel. Training is also provided in filling out field forms that accompany samples to the laboratory.

Table of Contents

1. Introduction **3**
2. NPS/State of Utah Agreement **3**
3. Training Schedule **3**
4. Information Received at the Training **3**
4.1. Sampling schedule 3
4.2. Analysis schedules 3
4.3. Data form instruction 3
4.4. Sample collection and handling 4
5. Instrument Care/Calibration **4**
6. Field Safety **4**
7. Data Review **5**
8. Contacts **5**
9. Appendix A: Written Agreement between NCPN and State of Utah **5**
10. Appendix B: Annual Workplan for NPS Cooperation with State of Utah **15**

1. Introduction

The Environmental Protection Agency gives authority and resources to the states to implement the intent of the Clean Water Act. The State of Utah works with agency cooperators like the National Park Service to collect and analyze water to determine if water bodies are in compliance with their designated use as classified by the State.

2. NPS/State of Utah Agreement

An informal written agreement was requested from UTDWQ water quality monitoring director Richard Denton that describes the cooperative agreement between the State of Utah and NCPN for water quality monitoring. The agreement (Appendix A) mentions that NCPN agrees to attempt 12 site visits per year, and lists the names and locations of sampling sites.

The location of sampling sites can change, but they must be approved by UTDWQ personnel (Richard Denton). He will also request an annual work schedule by the end of March that lists the sites, their location and sampling frequency (Appendix B). This table is updated annually as per Tables 9 and 10 in the Protocol narrative which outlines the long term rotating panel design.

3. Training Schedule

The primary training for collection of water samples for UT park units is covered in a one day annual cooperators meeting held in the spring in various locations throughout the state. Persons not attending this training cannot submit water samples for analysis. If staff are available, back-up staff will also attend this training. If new staff comes on board outside of the regular training season (spring) arrangements will be made with UTDWQ for a special training session. Contact state water quality monitoring director, Richard Denton 801-538-6055 to arrange a one-day training session.

4. Information Received at the Training

4.1. Sampling schedule

The UTDWQ requests 12 samples per year from each site as weather, flow and road conditions permit. Cooperators are also asked to attempt at least one winter sample collection. This allows the state to meet their mandated reporting requirements.

4.2. Analysis schedules

Samples are analyzed for metals quarterly, preferably; January, April, July and October. The same sampling protocol is used for quarterly samples as for monthly samples.

4.3. Data form instruction

Instructions are provided for filling out the data forms prior to shipping samples to the lab. Samples for ‘quarterly’ and ‘monthly’ have different lab and run sheets. See SOP 8 Appendices D through G for printable hard copy data forms corresponding to monthly or quarterly samples. Note that all hard copy data forms that accompany samples to the

laboratory are generated automatically and can be printed from Field_forms.xls described in SOP 8 section 11.

4.4. Sample collection and handling

Detailed instruction for collecting, filtering and shipping samples is provided in SOP 10.

5. Instrument Care/Calibration

For NCPN protocols see SOP 5 Preparations and Equipment Calibration, Handling, & Storage, All Units. Following are notes and recommendations from UT staff.

1. Calibrate daily (NCPN will usually re-calibrate pH and conductivity at each site unless time restrictions prohibit. DO will always be recalibrated if elevation between sites is more than 800ft.)
2. Keep a calibration log with each instrument.
    a. For the Troll this will be generated automatically and will be incorporated into the Field_forms.xls data sheet (electronic version)
    b. Record spin test time for pygmy / price meter in log book and Field_forms.xls.
3. Calibrating with individual standards involves several steps, but is relatively easy and worth the time investment. For detailed discussion about calibration frequency and methodology see SOP 5 section 6.
4. Calibrate conductivity at 1413 us/cm.
5. If numbers in the field appear flaky, recalibrate. This is based on professional judgment relative to instrument history and expected range in conductivity for the water being sampled.
6. UDEQ makes up 2 liters of buffer and uses/reuses it all week. They claim to have found little or no drift before and after each week.
7. DO Calibration
    a. Visually inspect DO membrane for tears or bubbles under the membrane.
    b. They use DI water for calibration and compare it weekly with Winkler method in the lab.
    c. Do not screw calibration cup tightly onto the instrument as the O-ring seal will pressurize the cup slightly and change the O_2 concentration.
8. Get absolutely NO water in the cable connection of the TROLL.

6. Field Safety

For thorough coverage of NCPN specific safety protocols refer to SOP 1 Field Safety. Some discussion of field safety occurs at the UT sponsored training course including:

1. Job Hazard Analysis completed at entry onto the job, and annually
2. First Aid
    a. Basic Survival
        i. Space Blanket
        ii. Signal Mirror
        iii. Knife

iv. Waterproof Matches
v. Water Supply

b. Safety Equipment
   i. Basic Tool Kit
   ii. Emergency reflectors
   iii. Shovel
   iv. Communications
   v. Radio set to all park and other important frequencies

7. Data Review

The UTDWQ lab will flag data as questionable if it arrives warm, is past holding time or there are other problems. Since the data is going into STORET as NPS data, NPS will have final say on whether the data is tossed or not.

8. Contacts

Director: Richard Denton 801-538-6055; rdenton@utah.gov

Data: Arne Hultquist; ahultquist@utah.gov

Laboratory: Sanwat Chandhuri; SCHAUDHU@utah.go

Assistant: Jack Oman; 801-584-8400 ask for "sample receiving" or "mailroom" for bottles

46 North Medical Dr., Salt Lake City, UT 84113

9. Appendix A: Written Agreement between NCPN and State of Utah

Why is the state of Utah participating with the National Park Service in the Northern Colorado Plateau Inventory and Monitoring water quality program?

The purpose of the Inventory and Monitoring program of the Northern Colorado Plateau Network (NCPN) of the National Park Service is to develop a stronger scientific basis for stewardship and management of natural resources. This includes evaluation of waters in Utah National Parks for status and trends in water quality in relation to Federal and State standards. This goal is compatible with a public policy objective of the state of UT to "conserve the waters of the state and to protect, maintain and improve the quality thereof for public water supplies, for the propagation of wildlife, fish and aquatic life, and for domestic, agricultural, industrial, recreational and other legitimate beneficial uses…. to cooperate with other agencies of the state, agencies of other states and the federal government in carrying out these objectives." (Rule R317-2-1A).

NCPN water quality monitoring will support UDEQ in the evaluation of protected uses through water quality standards by expanding the number of water sources that can be monitored. The broader objectives of NCPN water quality monitoring are to:

- collect, analyze and interpret data to support management in relation to 303(d) listing of waters.
- collect, analyze and interpret data to support management of threatened or otherwise special waters, using state standards developed under the Clean Water Act.

Because the State has the expertise and existing laboratory facilities for quantitative analysis of water quality parameters, and a vested interest in the quality of waters within the state, the NCPN requests to enter a cooperative agreement with the State of Utah for the mutually beneficial purpose of monitoring park waters.

Agreement to sample monthly.

An important goal of the NCPN water quality monitoring program is to capture a long-term record of representative water quality conditions across the parks, and to track the status of individual sites of great interest. This requires a consistent methodology and monthly sampling, such that in the long-run, significant trends in water quality parameters will be apparent in spite of variability. The sampling will be conducted by NPS personnel in most cases or by State trained cooperators using State of Utah methodology (learned via annual training provided by Utah Water Quality Laboratory).

Site locations

Site locations and sampling frequencies are highlighted in the attached table.

Table A1. NCPN Utah sites Selected for 2005–2006 water quality monitoring.

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
ARCH	Courthouse Wash near Moab, UT	M-0	225	599524	383646/ 1093445	L	1	NPS Coop w/ UT – Rotating	X		12/yr	CP, MI, N, T, TSS, FIB	Bact, Status and trends. Expand from rotation to continuous long-term
ARCH	Salt Wash at Wolfe Ranch Road	M-1	263	599522	384414/ 1093109	L	1	NPS Coop w/ UT – Rotating	**X**		12/yr	CP, MI, N, T, TSS, FIB	Status and trends. Expand from rotation to continuous long-term
ARCH	Freshwater Spring			599520	384429/ 1093141	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	
ARCH	Sleepy Hollow			599525	384024/ 1093821	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	
ARCH	Willow Spring			599527	384160/ 1093743	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	
ARCH	Lost Spring			599529	384813/ 1093050	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
ARCH	Upper Courthouse Wash	M-2		5995245	384117/ 1093915	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	
BRCA	Sheep Creek below Spring	M-5			373417/ 1121201	L	1	None	**X**		8/yr	CP, MI, N, T, FIB, Tu	Water source for hikers, grazing/wildlife
BRCA	Yellow Creek below Spring	M-6			373428/ 1120825	L	1	None	**X**		8/yr	CP, MI, N, T, FIB, Tu	Water source for hikers, grazing/wildlife
CANY	Colorado River above Green River	M-7	1039	495240	381133/ 1095302	L	1	NPS Coop w/ UT		09180500	10-12/yr	MI, N, T, TSS, FIB	Se, P, Status and trends
CANY	Green River above Colorado River	M-8	1055	493001	381124/ 1095321	L	1	NPS Coop w/ UT		09315000	10-12/yr	MI, N, T, TSS, FIB	Se,Status and trends
CANY	Green river at Mineral Bottom			495242	383119/ 1095955	L	1	NPS Coop w/ UT		09315000	10-12/yr	MI, N, T, TSS, FIB	Status and trends

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
CANY	Colorado River at Potash Boat Ramp			495629	383020/ 1093932	L	1	NPS Coop w/ UT		09180 500	10-12/yr	MI, N, T, TSS, FIB	Status and trends
CANY	Colorado River at Big Drop 3			495238	380829/ 1095544	L	1	NPS Coop w/ UT		09180 500 + 09315 000	10-12/yr	MI, N, T, TSS, FIB	Status and trends
CANY	Salt Creek at Crescent Arch	M-9	108 3	599515	380506/ 1094559	R	1	NPS Coop w/ UT	**X**	09187 650 (upstr eam)	12/yr	MI, N, T, TSS, FIB	Status and trends
CANY	Salt Creek - Peekaboo			599516	380708/ 1094501	R	2	NPS Coop w/ UT			12/yr	MI, N, T, TSS, FIB	Status and trends
CANY	Little Spring Canyon	M-10	107 2	599512	381103/ 1094759	R	2	NPS Coop w/ UT			12/yr	MI, N, T, TSS, FIB	Se, Status and trends
CANY	Cave Spring			599505	380924/ 1095412	R	2	NPS Coop w/ UT			12/yr	MI, N, T, TSS, FIB	Status and trends
CANY	2.4 mile Loop Spring			599502	380832/ 1094845	R	2	NPS Coop w/ UT			12/yr	MI, N, T, TSS, FIB	Status and trends
CANY	Maze Overlook			599550	381342/ 1095943	R	2	NPS Coop w/ UT			12/yr	MI, N, T, TSS, FIB	Status and trends

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
CANY	Chocolate Drops			599542	381329/ 1100009	R	2	NPS Coop w/ UT			12/yr	MI, N, T, TSS, FIB	Status and trends
CANY	Horseshoe Canyon			599544	382742/ 1101157	R	2	NPS Coop w/ UT			12/yr	MI, N, T, TSS, FIB	Status and trends
CARE	Fremont River at Hickman Bridge	M-11	1423	495433	381719/ 1111403	L	1	UTDEQ			12/yr	MI, N, T	Phosphorus
CARE	Halls Creek below the narrows	M-12	1450		373654/ 1105215	R	1	None	X		12/yr	CP, MI, N, T, TSS, FIB	few data, BACT., hiker impact, nutrients
CARE	Oak Creek above Diversion	M-13	1461	495497	380457/ 1110812	R	1	None			12/yr	MI, N, T, TSS, FIB	Bact, few data, status and trends
CARE	Pleasant Creek at South of Sleeping Rainbow Ranch	M-14	1473	495478	381048/ 1111050	R	2	None			12/yr	MI, N, T, TSS, FIB	Total Phosphorus, ammonia, pH, BACT.

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
CARE	Sulphur Creek at Mouth at Fruita, UT	M-15	1481	495477	381733/ 1111543	R	2	None			12/yr	MI, N, T, FIB	Bact. & Nutrients
DINO	Green River near Jensen	M-29	3341	493790	402434/ 1091405	L	1	USGS & UTDEQ		09261000	8-12/yr	MI, N, T	Status and trends
DINO	Yampa River at Deerlodge Park	M-30	3478		402706/ 1083128	L	1	USGS 4x/year only	X	09260050	8-12/yr	CP, MI, N, T, TSS	Increasing ph & nutrients effects on biota. NCPN expand frequency to 8/yr by USGS
DINO	Green River above Gates of Lodore	M-31	3369		404417/ 1085249	R	1	USGS (sediment only)	X		8-12/yr	CP, MI, N, T	Status and trends
DINO	Jones Hole Creek below Fish hatchery	M-32	3416		403515/ 1090324	R	1	None	X	09260500	8/12/yr	CP, MI, N, T, FIB, Tu	Effects of fish hatchery upstream on creek, nutrients.
DINO	Cub Creek at Mouth	M-33	3326		402513/ 1091428	R	2	None			8-12/yr	MI, N, T	Status and trends
HOVE	Cajon Spring			599571	371758/ 1091101	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
HOVE	Square Tower Sp.			599570	372309/ 1090452	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
HOVE	Hackberry Spring 1			599572	372428/ 1090130	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
HOVE	Hackberry Spring 2				372428/ 1090129	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
HOVE	Horseshoe Spring				372431/ 1090148	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
HOVE	Goodman Point					R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
HOVE	Sipapu Bridge Spring			599533	383109/ 1095942	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
NABR	Armstrong Canyon near Kachina Bridge			599531	373549/ 1100149	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
NABR	Owachomo Bridge Sp.			599532	373454/ 1100048	R	2	NPS Coop w/ UT – Rotating			12/yr	MI, N, T, TSS, FIB	Springs are priority water source
ZION	North Fork Virgin River at Confluence	M-35	4493	495095	370948/ 1130044	L	1	UTDEQ		09405500	12/yr	MI, N, T, Tu	Total Phosphorus

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
ZION	North Fork Virgin River at North Fork Road (narrows trailhead)	M-36	4511	9405450	372325/ 1124929	L	1	None	X		12/yr	CP, MI, N, T, Tu	BACT., source tracking, trends
ZION	East Fork Virgin River at Confluence	M-37	4454	495140	370947/ 1130038	R	1	UTDEQ		09404900	12/yr	MI, N, T, Tu	Total phosphorus, source tracking, trends
ZION	La Verkin Creek at Lee Pass Trail	M-38	4478	ZION_USU_LVC	372426/ 1131030	R	1	None	X		12/yr	CP, MI, N, T	Total Phosphorus, Ammonia
ZION	North Creek at planned gage in Park	M-39	4486		371540/ 1130620	R	1	None	X	Planned NPS/ UDWR	12/yr	CP, MI, N, T	Total Phosphorus
ZION	East Fork Virgin River, Upper Park Boundary	M-40			371054/ 1125148	R	2	None			12/yr	MI, N, T, Tu	Source tracking, trends. Access very difficult.

Park	Site name	NCPN site ID	NCPN database site no	STORET site ID	Latitude/ Longitude	Panel: long-term continuous or rotation	Priority*	Current monitoring	Planned NCPN supported site	Streamflow gage	Sample frequency	Sample parameter groups**	Considerations
ZION	North Creek above confluence	M-41	4485	495089	371209/ 1131031	R	2	UTDEQ			12/yr	MI, N, T	TDS TMDL

Water-quality vital signs monitoring sites for NCPN with other significant monitored sites. NCPN sites for monitoring in 2005 are highlighted.

10. Appendix B: Annual Workplan for NPS Cooperation with State of Utah

Table 10.1. National Park Service water quality sites in the state of Utah, 2006–2007.

Park unit	Location	STORET code	Latitude	Longitude	Sampling frequency
NATURAL BRIDGES NATIONAL MONUMENT	Sipapu Bridge Spring	5995330	37.6197222	-110.0058333	12/year
	Armstrong Canyon near Kachina Bridge	5995310	37.6002778	-110.0302778	12/year
	Owachomo Bridge Spring -	5995320	37.5816667	110.0102778	12/year
HOVENWEEP NATIONAL MONUMENT	Hackberry Spring Pool	5995720	37.4083333	-109.025	12/year
	Square Tower Spring	5995700	37.3855556	-109.0805556	12/year
	Cajon Spring	5995710	37.2972222	-109.1833333	12/year
ARCHES NATIONAL PARK	Lower Courthouse Wash	5995240	38.6130556	-109.5791667	12/year
	Salt Wash near Wolfe Ranch	5995220	38.7352778	-109.5188889	12/year
CANYONLANDS NATIONAL PARK	Salt Creek near Crescent Arch	5995150	38.0852778	-109.7663889	12/year
	Colorado River at Potash Boat Ramp	4956290	38.4669444	-109.6655556	5-7/year
	Green River at Mineral Bottom	4930150	38.5266667	-109.9927778	2-3/year
	Green River above confluence	4930010	38.1902778	-109.8891667	7-9/year
	Colorado River above confluence	4952400	38.1925	-109.8838889	7-9/year
	Colorado River below Big Drop 3	4952380	38.0708333	-110.0463889	5-7/year
ZION NATIONAL PARK	North Fork Virgin River	4951260	37.39048	-112.82504	12/year
	North Creek at park boundary	4950920	37.26646	-113.09659	12/year
	La Verkin Creek at Lee Pass Trail	4950800	37.40563	-113.17983	12/year
BRYCE CANYON NATIONAL PARK	Sheep Creek at park boundary	4951855	37.56584	-112.19881	12/year
	Yellow Creek at park boundary	4951857	37.57349	-112.12945	12/year
CAPITOL REEF NATIONAL PARK	Halls Creek below narrows	4954812	37.61495	-110.87123	12/year

Note: The Latitude and Longitude of sites in Zion, Bryce and Capitol Reef do not match the coordinates in EPA STORET. The correct site coordinates are listed on this page.

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #4

Site Selection, Conditions, and Documentation

Version 1.0 (04/10/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

The criteria and rationale used to select which water bodies to sample in NCPN parks, the sampling interval, and the sampling duration (panel) are outlined in the protocol narrative. This SOP presents a description of the criteria used to select sample sites in streams, how to develop a representative sampling technique for the site, and an example of documentation that will be made for each site as part of the permanent record.

The content in this SOP is modified from USGS National Field Manual, specifically: Wilde (2005) section A.1.2.1.A, which can be accessed via http://water.usgs.gov/owq/FieldManual/chapter1/Ch1_contents.html, and Wilde and Radtke (2005) section A.6.0.2.A, which can be accessed via http://water.usgs.gov/owq/FieldManual/Chapter6/final508Chapter6.0.pdf.

Table of Contents

1. Site selection objectives 3
2. Prior to site selection 3
3. Field reconnaissance for representative flowing-water sites 3
4. Preliminary selection of representative flowing-water site 5
5. Determining representative site and appropriate sampling technique 5
6. Site documentation 8
7. Field folders 10
7.1. Field folder checklist 10
8. Appendix A: Blank Field Site Description 12
9. Appendix B: Example of Completed Field Site Description 15
10. Citations 20

List of Figures

Figure 1. Example of base flow over-sampling to determine degree of stream mixing on Sheep Creek, Bryce Canyon National Park, UT. Sheep Creek has a high gradient and base flow less than 1.0 ft^3/s. 6

List of Tables

Table 1. Descriptive statistics used to interpret degree of stream mixing. 7
Table 2. Calibration acceptance criteria that can be used to determine if a stream is well mixed.. 7

1. Site Selection Objectives

Objectives are to collect baseline data that accurately represent condition of the water resource by sampling at sites and times that are spatially and temporally representative of flow and concentration of analytes. This requires carefully locating the sampling site and collecting samples in a repeatable and representative manner. A good location for collecting moving water samples will often coincide with a good location for determining discharge. However, this may not be the case in low-flow situations where flow is measured at a channel constriction and samples are collected from pools. In cases such as this, flow and sample collection should be made as close together as possible.

Following are guidelines that may be useful in selecting sampling sites. These guidelines are written to be as broadly applicable as possible to all manner of flowing waters, and give field personnel guidance in selecting appropriate sites. For this reason the guidelines can be followed verbatim, or they can be used with professional judgment to streamline the site selection process.

2. Prior to Site Selection

Some effort is required prior to selecting sites to ensure sites and data collected will meet the objectives of the monitoring effort.

1. Understand the purpose for which the various types of data will be collected and the aqueous system that each sample should represent.
2. Review the protocol narrative, especially types of measurements and samples needed.
3. Review site files and field folders of sites currently being sampled. Note site location, description and access, and review any previously collected physical, chemical, and biological data.
4. Obtain and keep current with UTDWQ or USGS training, equipment use and the laboratory requirements associated with data-collection activities.

3. Field Reconnaissance for Representative Flowing-Water Sites

1. For sites in Utah, ensure perennial surface flow is greater than 1 mile to meet UTDWQ requirement. Record the date and method of determining flow length on the site description form (Appendix A). Keep a record of the following in a field notebook.
    a. During base flow conditions, hike the length of flow upstream and/or downstream from the sampling point to be certain surface flow occurs continuously for at least 1 mile.
        i. Use a GPS, and 7.5 minute topographic map to make sure a mile has been traversed
        ii. Ask local residents or park staff about the perennial nature of the water body to be certain it flows continuously.

b. If the stream does not flow more than a mile or does not flow perennially, a different water body must be selected if UTDWQ will perform laboratory analysis.

2. When hiking the stream reach carry a GPS, map, digital camera and field note book.
3. Make field notes of and photograph potential sampling locations.
    a. Note sites that can be most easily accessed at all times of the year keeping in mind some times of year may make access impractical.
    b. Note cross sections where samples can be collected at any stage throughout the period of study.
    c. Note cross sections that are least likely to change in shape due to erosion or deposition.
    d. Note locations where staff gages could be installed to develop rating curves
        i. These locations will have a stable channel and a will allow observation of extreme high and low stages.
        ii. The gage can be securely attached to an immovable object
        iii. Reference bench marks can be established
    e. Note potential for vegetation encroachment and any other factors that would force moving to a different location for sampling at a future time.
    f. Note potential sources of contamination at the site, based on target analytes to be collected.
    g. Note proximity to gauging station.
        i. If proximal then it may be possible to use continuous flow measurements from station.
        ii. If a stream-gauging station is not at or near the sampling site, or if discharge cannot be rated or estimated with sufficient accuracy it may need to be measured at time of sampling.
    h. Note uniformity of flow
        i. An ideal sampling site will have uniform flow, and stable bottom contour, and will be well-mixed along the cross section.
        ii. An ideal site has unidirectional flow that does not include eddies.
    i. Note proximity to factors that make flow or concentrations non-homogenous
        i. Consider upstream confluences of streamflow or point sources of contamination to avoid sampling a cross section where flows are poorly mixed or not unidirectional.
        ii. It may be necessary to sample in reaches upstream from bridges or other structures, to avoid contamination from the structure or from a road surface.

4. Preliminary Selection of Representative Flowing-Water Site

After noting the potential sampling sites review notes and photographs and rank them based on:

1. Qualitative determination of representativeness of the system
2. Proximity to a sampling site near a reach where other data are collected (such as data for suspended sediment, bedload, bottom material, or biological material) and (or) for which historical data are available.
3. Access issues related to safety
4. Distance from vehicle
5. Potential for staff gage installation

5. Determining Representative Site and Appropriate Sampling Technique

To obtain data representative of the section, the variability of discharge and field measurements across the stream must be known. This information is used to determine if the equal-discharge-increment (EDI) or equal-width-increment (EWI) method of locating field-measurement points should be used (SOP 10 Appendix A). Typically water in small steep gradient streams will be well mixed. Laminar flow and the possibility for poorly mixed discharge are most likely in the large rivers.

1. After tentative selection of a sampling site, make measurements of pH, specific conductance, temperature, dissolved oxygen and turbidity at a number of equally-spaced verticals along the cross section and at multiple depths within each vertical.
2. Evaluate the cross-sectional profile data for variability in discharge and field-measurement values.
    a. Cross sectional profiles can be constructed in Excel or other plotting software (Figure 1).
    b. Descriptive statistics for cross sections should also be computed (Table 1).

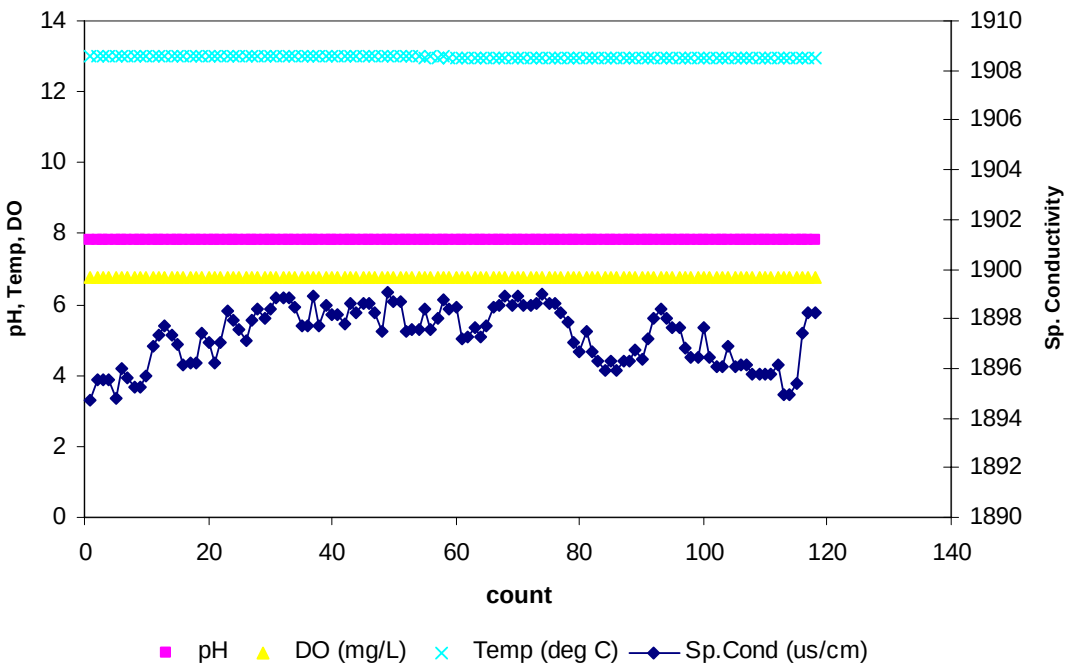


Figure 1. Example of base flow over-sampling to determine degree of stream mixing on Sheep Creek, Bryce Canyon National Park, UT. Sheep Creek has a high gradient and base flow less than 1.0 ft^3/s.

Table 1. Descriptive statistics used to interpret degree of stream mixing.

	Temp (deg C)	Pressure (mm Hg)	Clark DO (mg/L)	pH	Spc. Cond (us/cm)
min	12.93	588	6.76	7.79	1895
max	13.03	588	6.79	7.80	1899
mean	12.98	588	6.78	7.79	1897
median	12.97	588	6.78	7.79	1898
range	0.10	0	0.02	0.01	4
stdev	0.03	0.02	0.01	0.00	1.15

3. Is the stream well mixed or not?
   For well calibrated sensors, if the range in core parameter values is outside of the acceptable accuracy criteria for the individual sensor the stream is probably not well mixed. The descriptive statistics from the Sheep Creek example indicate that the measured variability in situ is less than the calibration acceptance criteria in Table 2, and thus would be considered well mixed.

Table 2. Calibration acceptance criteria that can be used to determine if a stream is well mixed.

CORE PARAMETER	Sensor Measurement (acceptance/rejection criteria)	Optimum Goal/Target (Range) (USGS criteria)
Temperature	± 0.3 º C	± 0.2 º C
[1]Specific Conductance[2]	± 5 µS/cm or ± 3%	± 5 µS/cm or ±3%
pH[3]	± 0.3 S.U.	± 0.2 S.U.
Dissolved Oxygen[2]	± 0.4 mg/L, ± 5% saturation	± 0.3 mg/L, ± 3% saturation

1Whichever is greater

2 Parameter values (SC and DO) may be affected by 2 to 3% per degree Celsius change in temperature

3 If after a 2–point calibration and performance of any necessary pH sensor maintenance, error criteria are still not met, a 3-point calibration should be performed before sensor is rejected and replaced.

4. If results indicate the stream is not well-mixed and it should be based on its gradient and turbulence move up or down reach and re-sample. Select the EDI or EWI method to locate points of measurement (SOP 10 Appendix A; for more information refer to Wilde et al., 1999).
5. If stream depth and velocities along the cross section are relatively uniform and evaluation of oversampling indicates the stream is well mixed, use the EWI method for future monitoring.
6. If stream depth and velocities along the cross section are highly variable, use the EDI method for future monitoring.
7. In a small and well-mixed stream, a single point at the centroid of flow may be used to represent the cross section. The centroid of flow is defined as the point in

the increment at which discharge in that increment is equal on both sides of the point.

8. Field-measurement profiles of stream variability are needed for low- and high-flow conditions and should be verified at the beginning of each panel rotation (i.e. at the start of each two year sampling interval).
9. After deciding on an appropriate sampling site and methodology a formal Field Site Description (FDS) will be written.

6. Site Documentation

Each site will have a Field Site Description (FSD) that provides information on its location, condition and the record of data collection planned or obtained historically from the site. A blank FSD to fill out in the field and a completed example are presented in Appendix A and B respectively. The FSD is carried in the field folder and updated regularly to help establish a comprehensive and complete record. The components of a complete FSD include:

1. *Site identification.* Includes the agency, unit and address of the organization in charge of sampling and data management at this site, and the official name of the site along with any identifying codes.

2. *Location of sample collection.* Includes latitude and longitude, 7.5 minute quadrangle name, county name, state, hydrologic unit and a brief description of the location relative to significant landmarks. A more detailed description of navigating to the site is presented in the roadlog.

3. *Land ownership.* Who owns the land at the site and the land that must be crossed to reach the site. Where are vehicles parked while sampling.

4. *Contact information.* Who should be notified before traveling to the site. Provide names, phone numbers and radio call numbers.

5. *Safety contacts.* Who to call in an emergency. Include radio call numbers, phone numbers and phone number and location of nearest hospital or emergency facility.

6. *Establishment and history.* List when the sampling site was established, frequency of visits and availability of rating curve.

7. *Parameters.* List what data are collected at the site and how (e.g. Grab sample, continuous?). List what laboratory analyses are conducted on samples collected from the site, and what laboratories perform the analysis.

8. *Drainage area.* List the drainage area and how the number was obtained.

9. *Gage.* If a gage is present describe its shelter, and operational equipment such as bubbler, strip chart, staff gage, and what organization maintains it. Also list web site if the gage records real time flow.

10. Assessment of Gage Adequacy: Is this gage located close to the sampling site, and are there considerations that should be taken into account when using this gage to estimate flow at the sampling site?

11. *Sampling frequency.* How often is data collected? Is data collected continuously via automated sensors at this site?

12. *Pertinent data.* Include elevation of the site and descriptions of instrumentation installed at the site and date it was installed.

13. *Reference and benchmarks.* Provide a careful description of bench marks, their elevation and location in reference to the sampling site.

14. *Channel and control.* Give a complete description of channel characteristics that control the water elevation at different stages of flow. Include descriptions of vegetation.

15. *Discharge measurements.* Give a description of techniques used to measure discharge at low, medium and high stages. Also indicate where discharge measurements are made at these stages if they are not all made at the same location. How is discharge measurement made when ice is present?

16. *Floods.* When was the maximum flow recorded and what was the stage height then?

17. *Point of zero flow.* What is the elevation of no flow, and has it ever been observed?

18. *Winter flow.* Does the channel stay open during winter?

19. *Water quality monitor extremes.* Provide the range in water quality data observed over the life of the sampling site and when the maxima and minima occurred.

20. *Regulation and diversion.* Is it an unregulated and undiverted stream?

21. *Accuracy / representativeness.* How accurate are the records? Have some been estimated? Is the gage considered representative of the sampling location?

22. *Purpose and cooperation.* Who conducts the sampling and who are the cooperating entities?

23. *Special equipment needs.* What special equipment might be necessary for measurements and monitoring at different flow levels?

24. *Sampling methodology.* What type of sampling is used to account for variability in the system?

25. *Road log.* A detailed description of starting location and distance traveled on road and trail segments as well as mileage to intersections, junctions and turn-offs. For walking distances estimate travel time.

26. *Access hazards.* Each site will have an analysis of potential safety considerations that includes recommended clothing and equipment necessary for safe site visits. It will also include potential dangers associated with travel to the site and hazards associated with work at the site. Specifically included are: general access issues, wet weather access issues, winter access and hot weather issues.

27. *Regional and local maps.* To aid navigation to and provide a general sense of site location in broad context a regional and local map (could be original, scanned, photocopied, GIS product, etc.) with annotation are included.

7. Field Folders

The field folder is a working set of documents that contain all the information needed to sample at field sites. It serves as a reference folder containing information needed by trained personnel to locate and safely collect and process water samples. A copy of the field folder is taken along on each sampling trip, with an original maintained in office files. General contents of the field folder are listed below.

7.1. Field folder checklist

1. Station description and the following in hard copy format:
    a. Conditions
        i. Hydrologic and geologic sections
        ii. Photographs to document site conditions and sampling location or change in stream channel morphology
        iii. GIS hardcopy of physical data layers to aid understanding relationships
        iv. Profiles of cross section of stream channel at sampling location(s)
        v. Stream-bottom geometry
    b. Statistical Data Summary from field forms as spreadsheet graphics
        i. Plots of field-measured data (last 5-10 years of record); if there is a good enough relation to show outliers, include:
            1) Conductivity versus streamflow.
            2) Conductivity versus alkalinity.
            3) Temperature versus time.
            4) Statistical summary of historical water data:
            5) Cumulative-discharge curves at about 10-, 50-, and 90-percent duration.
            6) Velocity cross sections at about 10-, 50-, and 90-percent duration.
            7) Equipment to use at various flows.
            8) Results from representative sample size determination

9) Flow-duration curve
10) Discharge-related maximum-minimum values
11) Seasonal, maximum-minimum values
12) Discharge rating curves and (or) tables

2. Sampling instructions
    a. When to collect samples (high or low discharge).
    b. Target sampling window (time of day)
    c. Bottle types and number needed for each sampling site
    d. A list of sample handling and treatments (i.e. filtration, acidification, chilling)
    e. Other observations to make including time since high flow, vegetation density or encroachment, weather and any noticeable changes that could affect data.
3. Reference materials
    a. Laminated equipment manuals or documentation for
        i. flow meter/wading rod with rating charts
        ii. weir with rating chart
        iii. troll and probes
        iv. rugged reader
        v. peristaltic pump
        vi. digital camera
        vii. GPS manual
        viii. Short-cut reference cards for equipment – these should also be in the field pack
4. Laboratory data forms
    a. Laboratory codes for site conditions and analyses to be requested
    b. UTDWQ surface-water field form
    c. Site condition codes and an example of a completed form.
5. Write in rain field notebook
6. Shipping instructions
    a. Days to ship and what days to avoid
    b. Amount of ice to use
    c. Mailing labels to and from laboratory.
    d. Location of nearest post office or shipping agent.
7. Log recording visits, attempted visits, samples collected and other activities

8. Appendix A: Blank Field Site Description

Modified from Typical USGS Site Description (with assistance from J. Brown USGS Lakewood, CO; personal communication)

Field Site Description Form

To be filled in after selection of field site

Site identification

Agency:
Unit and address:
Official name of the site along with any identifying codes:

Location of sample collection

Latitude:
Longitude:
7.5 minute quadrangle name:
County name, state:
Hydrologic unit:
Brief description of the location relative to significant landmarks:

Land ownership

Getting to site:
At the site:
Where are vehicles parked while sampling?:

Contact information

Who to contact prior to site visit
Name:
Phone number:
Radio call number :

Safety contacts

Radio call numbers:
Phone numbers:
Phone number and location of nearest hospital or emergency facility:

Establishment and history

Establishment date:
Location of historic data:
Availability of rating curve y / n?

Parameters

List what data are collected at the site and how: (e.g. Grab sample, flume or meter?).
List what laboratory analyses are conducted:

Drainage area

Determine drainage area from GIS

Gage

If a gage is present describe its shelter, and operational equipment such as bubbler, strip chart, staff gage, and what organization maintains it:
web site:
Assessment Gage Adequacy. Is this gage located close to the sampling site, and are there considerations that should be taken into account when using this gage to estimate flow at the sampling site?:

Sampling frequency

Frequency of visits and rotation panel or continuous? Is data collected continuously via automated sensors at this site?:

Pertinent data

Elevation of the site:
descriptions of instrumentation installed at the site and date it was installed:

Reference and benchmarks

Carefully describe bench marks, their elevation and location in reference to the sampling site:

Channel and control

Completely describe channel characteristics that control the water elevation at different stages of flow. Include descriptions of vegetation.:

Discharge measurements

Describe techniques used to measure discharge at low, medium and high stages. Also indicate where discharge measurements are made at these stages if they are not all made at the same location. How is discharge measurement made when ice is present?:

Floods

Max flow date and stage height?

Point of zero flow

What is the elevation of no flow, and has it ever been observed?

Winter flow

Does the channel stay open during winter? If not what techniques are used in sampling?

Water quality monitor extremes

Dates and range of max flow:
Dates and range of DO:
Dates and range of pH:
Dates and range of temp:
Dates and range of cond:

Regulation and diversion

Is this water body regulated? y / n?

Accuracy / representativeness

How accurate are the records? Have some been estimated? Is the gage considered representative of the sampling location?

Purpose and cooperation

Who conducts the sampling and who are the cooperating entities?

Special equipment needs

What special equipment might be necessary for measurements and monitoring at different flow levels?

Sampling methodology

What type of sampling is used to account for variability in the system?

Road log

A detailed description of starting location and distance traveled on road and trail segments as well as mileage to intersections, junctions and turn-offs.

Access hazards

general access issues: wet weather access issues: winter access: hot weather issues:

Regional and local maps

Append scanned and annotated maps of region and local area around site

9. Appendix B: Example of Completed Field Site Description

United States Department of the Interior

National Park Service
Northern Colorado Plateau Inventory and Monitoring Program
2282 SW Resource Blvd.
Moab, Utah 84532

WATER QUALITY FIELD STATION DESCRIPTION

Sheep Creek, Bryce Canyon National Park

LOCATION OF SAMPLE COLLECTION
Lat 373417, long 1121201, (Bryce Point Quad., scale. 1:24,000), Garfield County, UT
Approximately 2.5 miles downstream from bench mark 7998 at Swamp Canyon Overlook which is approximately 5 miles south of Bryce Canyon National Park visitor center on Hwy 63, the main park road.
Hazards Associated with Site: Rock surfaces are often slippery with algae. Ponderosa snag will fall someday.

LAND OWNERSHIP
Access to sampling site involves parking at Swamp Canyon Overlook in Bryce Canyon National Park, or driving through BLM administered lands and private land on the Sheep Creek road a western spur of the Cotton Wood road. Parking is at the remote and infrequently used Sheep Creek Dixie National Forest Trailhead. Sampling site is entirely within Bryce Canyon National Park 20 upstream from park boundary.

CONTACT INFORMATION
Before traveling to site, contact Chief of Resource Management and Chief Ranger.
Chief of Resource Management (435-834-5632, Kristin_Legg@NPS.gov , Radio call # 900).
Chief Ranger (435-834-5647, Brent_McGinn@nps.gov Radio call # 800)

SAFETY CONTACTS
Duty Ranger call # 851, BRCA law enforcement dispatch 800-582-4351
Use radio channel 14 to hit repeater and channel 13 for direct contact. Radio coverage is spotty below the rim.
Outside park dial 911 or 435-676-2411 Garfield County Sheriff.
Nearest emergency facility: Garfield County Clinic 435-676-8842
Garfield Memorial Hospital 435-676-8811

ESTABLISHMENT AND HISTORY

Established October, 2005. This site is on a monthly sampling schedule in a bi-annual sampling rotation with other Northern Colorado Plateau Network sites. It will be monitored monthly for two years then not at all for two years.

PARAMETERS
Core parameters measured in situ include: discharge, dissolved oxygen concentration and percent saturation, specific conductance, pH and temperature. Grab samples are collected for metals, nutrients, turbidity and fecal coliform indicators. Metals and nutrients are analyzed by UTDWQ while turbidity and fecal coliform indicators are measured at BRCA.

DRAINAGE AREA
To be determined

GAGE
No permanent installations at this site.

CONTINUOUS WATER-QUALITY MONITOR
No continuous measures of water quality are made at this site.

PERTINENT DATA
Elevation 7149 ft

REFERENCE AND BENCH MARKS
The site is 20 ft upstream from park boundary fence above a 90 degree bend in the stream.

CHANNEL AND CONTROL
Streambed consists of gravels and cobbles. Vertical bank on west side of stream is 1-2 ft high then forest vegetation. Bank on east is low gradient sand and gravel grading into forest vegetation. Channel is tortuous and entrenched upto 6 feet along most of its length. The stream is high gradient and well mixed. Cattle trespass frequently at this site as the boundary fence is in disrepair.

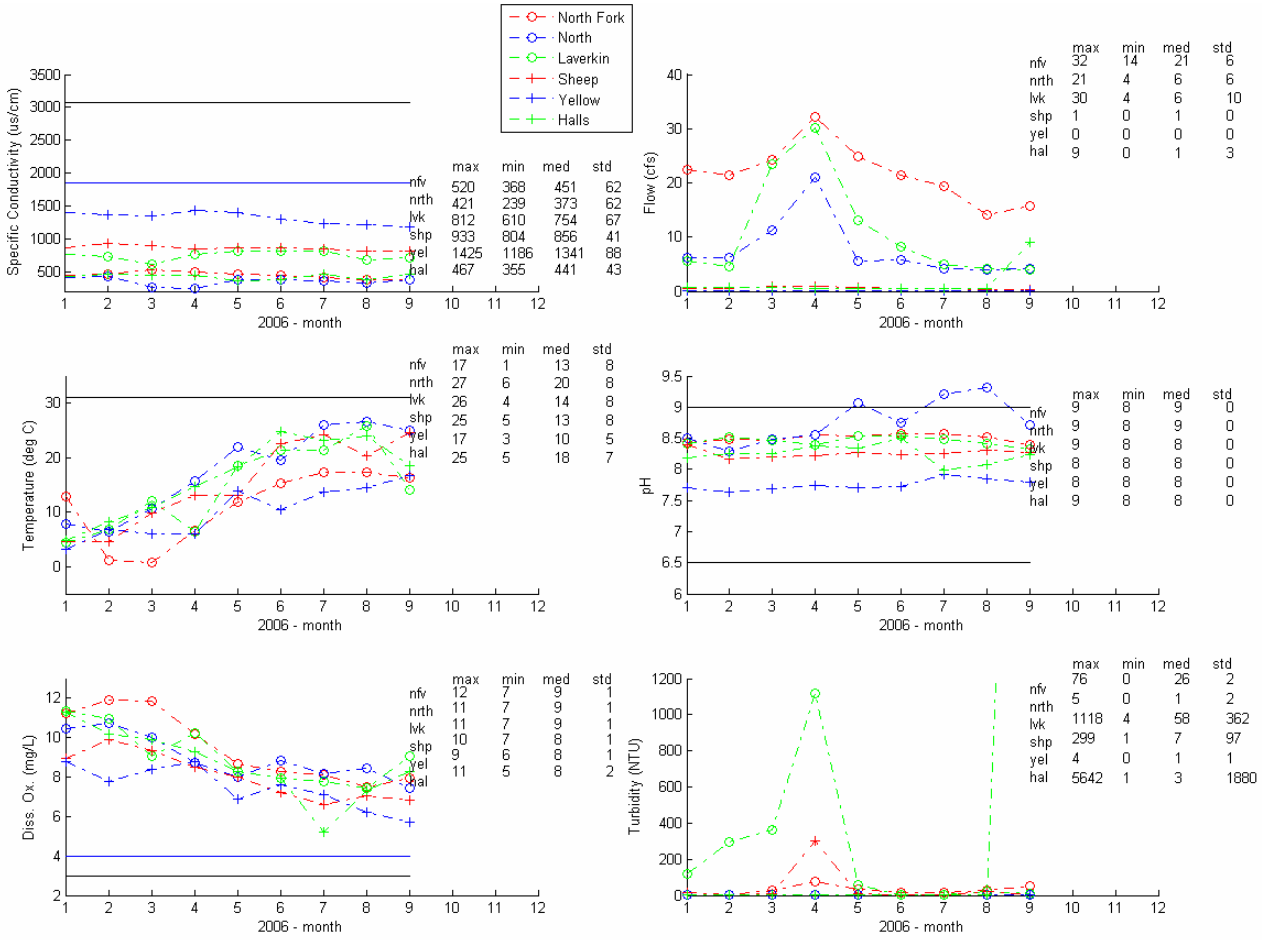
DISCHARGE MEASUREMENTS
Flow is measured with either the 1or 8 inch Baski flume, or V-notch weir. High-water Flow Measurement has not been made at this site, but the maximum stage based on high water marks could be waded and measured with a pygmy meter. Discharge will be estimated if too high for wading.
This site is cold in winter and may have ice on stream margins.

FLOODS
Maximum discharge observed during period of record, 1 ft^3/s

POINT OF ZERO FLOW
Not yet observed.

WINTER FLOW
Flows periennially through winter

WATER-QUALITY MONITOR EXTREMES



REGULATION AND DIVERSION
This is an unregulated and undiverted stream.

ACCURACY / REPRESENTATIVENESS
Records usually good.

PURPOSE AND COOPERATION
Station is operated to collect basic surface-water records as part of the National Park Service Northern Colorado Plateau Inventory and Monitoring Program – Water Quality. Water quality is monitored in cooperation with Utah Department of Water Quality, and Bryce Canyon National Park.

SPECIAL EQUIPMENT NEEDS

Low flow at Sheep creek is typically best measured with the 1 inch Baski cut throat flume. Higher flows are measured with the pygmy meter. A life jacket is not necessary at this site under normal circumstances.

SAMPLING METHODOLOGY

This site is sampled using centroid of flow for core parameters. Water quality samples for laboratory analysis are collected from the stream centroid using a single dunk technique.

ROAD LOG

Hiking option

0.0 (mi) Head south from Bryce Canyon National Park Visitor Center
1.0 Turn off to Bryce Point (continue past this intersection)
5.0 Swamp Canyon Overlook parking area on east side of road.
7.0 Park here and walk down Swamp Canyon Connector trail to junction with Under the Rim Trail, then continue down stream to sampling site.

Drive / Hike option

0.0 (mi) Bryce Canyon National Park Visitor Center
15 Cannonivlle on Hwy 12 turn south on Cottonwood road
20 Turn west on Sheep Ck.
27 USFS Sheep Creek Trail head
28 Follow Grandview Trail to Noon Canyon
29.5 Follow Sheep Creek upstream to park boundary

ACCESS HAZARDS

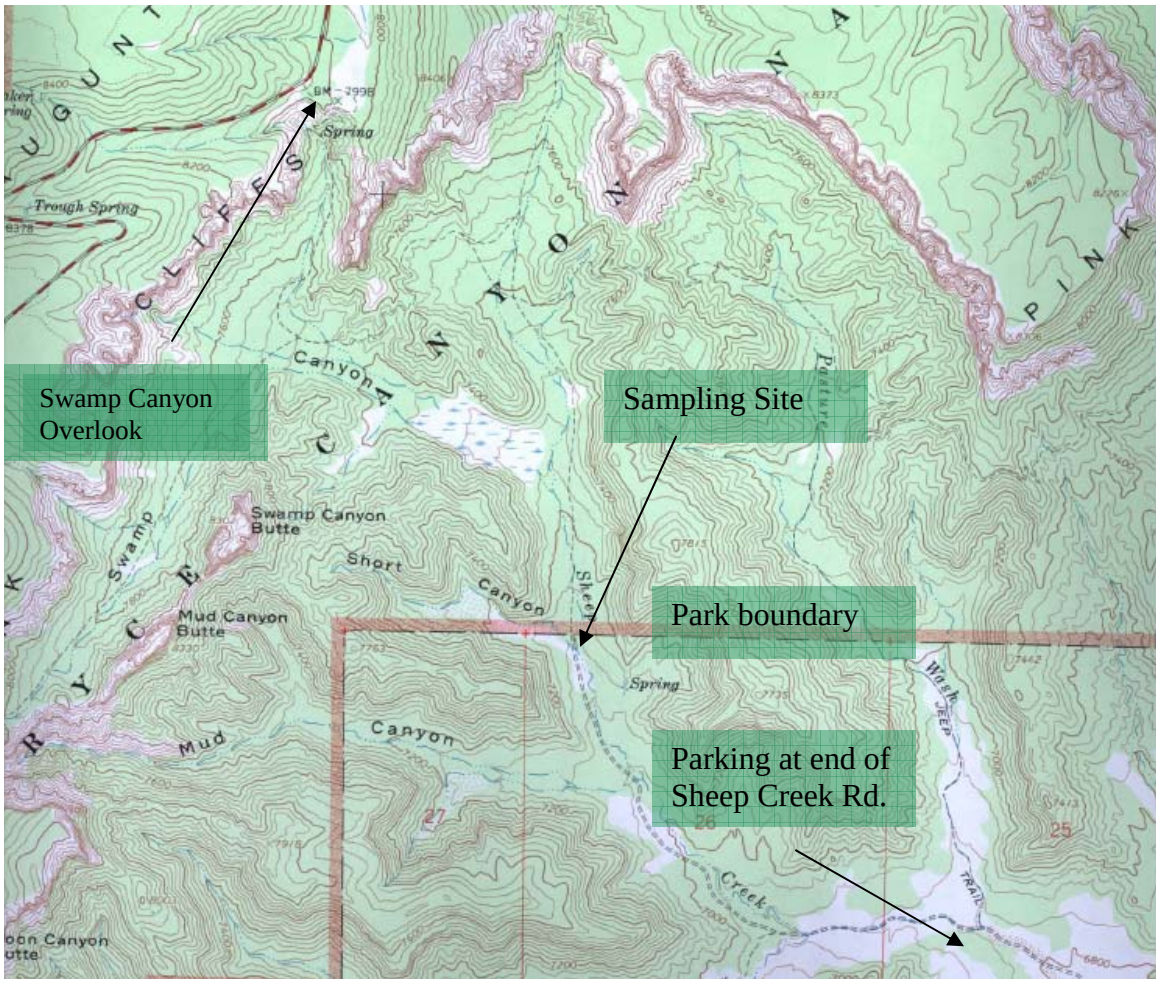
General Access – The 2.5 miles of dirt road from State route 9 is often rough and rutted. A high clearance vehicle is needed.

Wet-weather Access – Road is often impassible in wet weather. Sampling can be delayed up to 3 weeks for improved conditions.

Winter Access – Road is often impassible when wet in winter. Conditions vary year-to-year.

Hot-weather Access – No hot weather considerations.

An enormous Ponderosa snag is close enough to be a widow maker. Be wary of this on windy days.



Location of Sheep Creek field site, Bryce Canyon National Park.

10. Citations

Edwards, T.K., and Glysson, D.G., 1999, Field methods for measurement of fluvial sediment: Techniques of Water-Resources Investigations of the United States Geological Survey, book 3, chap. C2, 80 p.

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T., eds., September 1999, Collection of water samples: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. Section A4.1.1, accessed 8/30/2005 at http://water.usgs.gov/owq/FieldManual/chapter4/html/4.1_contents.html

Wilde, F.D., Radtke, D.B., eds., August 2005, Allocating points of measurement U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6., section 6.0.2.A, accessed 8/29/2005 from http://water.usgs.gov/owq/FieldManual/

Wilde, F.D., January 2005, Preparations for water sampling: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A1, accessed __*date*__ at http://pubs.water.usgs.gov/twri9A1/.

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #5

Preparations, Equipment Calibration,

Handling and Storage

Version 1.01 (2/4/08)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.0	2/4/08	D. Thoma	Add quick start guide to premob, update UT equip checklist	Make premob faster	1.01

This SOP provides guidelines for equipment handling, calibration and maintenance, to ensure the highest standard in quality assurance. Data quality begins before the first sample is collected by taking care to use equipment properly, being aware of data-quality requirements, and being alert to potential sources of equipment malfunction. This SOP guides (in part) preparation and use of the In-Situ Troll 9000 and 9500 series sonde and Rugged Reader display instruments in the measurement of core field parameters.

Table of Contents

1. Presampling checklist **4**
1.1. 2-4 weeks before trip 4
1.2. 1-2 weeks before trip 4
1.3. 1-7 days before trip 4
2. Field Trip Preparations **4**
2.1. Field checklist, Utah parks 4
2.2. Hiking checklist, Utah parks 7
2.3. Field checklist, CO parks 7
3. Equipment Log **8**
4. Premobilization checks **8**
4.1. Utah parks 8
4.2. Colorado parks 8
5. Data quality objective for core parameters **9**
6. Equipment Calibration **9**
6.1. Flow meters 9
6.2. Multiparameter probes 9
7. Equipment handling and calibration **10**
7.1. Colorado Units - Hydrolab 10
7.1.1. DO calibration *10*
7.1.2. pH calibration *10*
7.1.3. Conductivity /SC calibration *10*
7.1.4. General care and maintenance *10*
7.2. Utah Units - Troll 9000/9500 10
7.2.1. DO calibration *10*
7.2.2. pH calibration *11*
7.2.3. Conductivity calibration *11*
7.2.4. Temperature *11*
7.2.5. Multiparameter probe storage *11*
7.2.6. Turbidity *11*
8. Troubleshooting **12**
8.1. Colorado Units -Refer to the user manual Appendix E 12
8.2. Utah Units 12
8.2.1. Troll 9000 Elastomer *12*
8.2.2. Conductivity sensor *12*
8.2.3. pH sensor *12*
8.2.4. DO sensor (Clark cell) *12*
8.2.5. Top setting wading rod *13*
9. General care and maintenance **13**
10. Software updates **13**
11. Vehicle equipment **13**
12. Post sampling activities, UT **14**
13. Post sampling activities, CO **14**
14. Citations **14**
15. Appendix A: Multiparameter probe premobilization check UT parks except DINO **16**
15.1. Terminology 16
15.2. Rinsing 16
15.3. Premobilization Quick Start Guide: 17
15.4. Long Version of Premobilization procedures 18
16. Field_forms.xls premobilization worksheet metadata **26**
17. Appendix B. Original Premobilization Guidance from WRD that was modified above to Work with Troll 9000 units **30**
18. Appendix C: In-situ Inc. Troll 9000 Profiler Users Guide **46**

19. Appendix D: In-situ Inc. WinSitu 4.0 Software Manual.....47
20. Appendix E: In-situ Inc. WinSitu 2000 Software Manual.....48
21. Appendix F: Hydrolab Users Manual.....49
22. Appendix G: Hydrolab Display Unit Manual.....50
23. Appendix H DO calibration CO Units.....51
24. Appendix I: pH calibration CO Units.....53
25. Appendix J: Conductivity calibration CO Units.....59
26. Appendix K: Top Setting Wading Rod.....64
27. Appendix L: AquaCalc Pro Users Manual.....66
28. Appendix M: Pygmy meter.....82
28.1. English Rating Chart.....98
28.2. Metric Rating Chart.....99
29. Appendix N: Hach 2100P Turbidimeter Manual.....100
30. Appendix O: NCPN Water Quality Equipment and Supplies.....101
30.1. Equipment.....101
30.2. Supplies.....105
31. Appendix P: Equipment Inventory and Ownership.....107
32. Appendix Q: Troll 9000 Dissolved Oxygen (amperiometric) service instructions.....108
33. Appendix R: USGS Memo on Turbidity Units.....109
34. Appendix S: USGS National Field Manual Section 6.7 – Turbidity.....114

List of Tables

Table 1. Recommended MP Instrument Post-Calibration Check Error Limits (NPS Core Parameters). These are also the Data Quality Objectives (DQO's) discussed in the Quality Assurance standard operating procedure (SOP 7 section 7.2)..... 9

1. Presampling checklist

1.1. 2-4 weeks before trip

1. Obtain permission to use a GOV vehicle if available
2. Obtain travel authorization. NCPN staff can arrange a blanket travel authorization.
3. Check quantity and expiration dates of deionized water, calibration solutions for conductivity, pH, DO, and turbidity. Order more if necessary.

1.2. 1-2 weeks before trip

1. Contact park staff and private land owners prior to sampling and obtain permission for site access. Even if we have blanket permission they still like to know when we're there. Don't contact too far in advance or they'll forget.
2. Fill deionized rinse bottle and calibration solution bottles
3. Check supply of filters, pump tubing. Replace pump tubing if worn
4. Check supply of sample bottles from UTDWQ and request more if necessary.
    a. Contact Andy Christiansen 801-584-8204

1.3. 1-7 days before trip

1. Check availability of coolers for mailing.
2. Check that equipment batteries are sufficiently charged in
    a. Troll probe (4 D cell alkaline 1.5v)
    b. Rugged reader (charge the battery pack)
    c. Aquacalc (2 - 9v batteries)
    d. GPS (2 AA alkaline)
    e. Digital camera (2 AA NiMH)
3. Give new DO membranes at least 24 hours to condition
4. Make sure all data from previous sampling trips has been downloaded, saved and then deleted from above equipment.
5. Update field folder with new safety and access information and results from previous site visits, sampling and analysis of data.
6. Conduct premobilization equipment calibration and error check (Appendix A)

2. Field Trip Preparations

2.1. Field checklist, Utah parks

Checklists help ensure that equipment and supplies will be ordered on time, that data-collection activities will be completed appropriately, and that data-quality requirements will be met.

1. Field forms hard copy and pencil for recording data
2. MP Troll 9000/9500
    a. Troll body with calibrated SC, pH, DO sensors installed
    b. Calibration cup with sponge
    c. Stainless steel sensor cover
    d. Sensor insertion / extraction tools with grease
    e. Spare sensors including SC, pH, and DO

    f. Equipment log book
    g. Rugged Reader with charged battery (bring spare for long trips)
    h. Comit2 cable for Rugged Reader
    i. Down hole poly cable –pre connected to Comit2 cable
    j. 2 D cell spare batteries
    k. Rugged Reader battery car charger
    l. Rugged Reader USB cable
3. Calibration standards in mesh bag
    a. pH 7 buffer
    b. pH 10 buffer
    c. 1413 µs/cm conductivity standard
4. 6 Sample bottles in carry bag with sharpie for labeling
    a. 1 L carry bottle
    b. Total Chemistry bottle
    c. Nutrients bottle with 2ml acid
    d. Filtered metals with 2ml acid
    e. Bacteria bottle
    f. Turbidity vial
5. Coozy with ice for bacteria
6. Pygmy meter
    a. Oil
    b. Spare parts including extra pivot and lock bolt
    c. Screw drivers
    d. Cables for connecting to Aquacalc Pro
    e. 2 spare 9v batteries
    f. Stop watch for spin test
7. Turbidity meter with manuals
    a. Blank vials
    b. Standard gelex vials for calibration check
8. Digital camera
    a. spare batteries
    b. USB cable
    c. Backup replacement memory stick
    d. Spare alkaline AA batteries
9. Aquacalc Pro
    a. Spare batteries (two 9v)
    b. Connect cable
    c. Wading rod
    d. Breakdown parts stored in pygmy meter box
    e. Tape reel
    f. Tape clamps
10. Baskie flume
    a. Spare parts bags (screws, level, screw driver)
    b. Large 8 inch base and small 2 inch base with support bars
    c. Rating charts are in field folder equipment manuals
11. Bacteria

    a. Sealer, rubber sealer mold and power cord
    b. Incubator and power cords (both wall and car)
    c. Bacteria trays
    d. Bacteria food
    e. Bacteria bottles
    f. Rubber gloves
    g. Hand sanitizer
    h. Coozy and 1 L bottle with ice to cool samples
12. Glass alcohol filled thermometer
13. Geopump
    a. DI water squirt bottle
    b. Rubber gloves
    c. Spare tubing
    d. Charged battery
    e. 4um filters
14. Cooler
    a. Ice
    b. Packing tape
    c. Bubble wrap
    d. Sharpie
15. Laptop with Active sync, Winsitu and Aquacalc software loaded
16. GPS unit and manuals
    a. Spare batteries
17. Backup copy of SOPs on CD
18. Price current meter
    a. Meter
    b. Fins
    c. Oil
    d. Screw driver
    e. Rating charts
    f. Miscellaneous spare parts in vial
    g. Magnifying glass to inspect whiskers
    h. Connect cable for Aquacalc Pro (only have one for both meters – resides with Pygmy)
19. Field folder
    a. Maps
    b. Data forms
        i. Field sheets
        ii. Site description forms
    c. Safety information
    d. Copies of equipment manuals
    e. Software CD's
    f. Manuals on CD
    g. Field note book
20. Life vest
21. Waders

22. Boots
23. Warm gloves
24. Radio and emergency contacts
    a. Spare radio battery
25. Shovel and jack

2.2. Hiking checklist, Utah parks

When hiking in to a sampling site it is important to go over a checklist to ensure all the equipment necessary to complete the job makes it to the site.

1. Data forms, field notebook, clip board, pencil and sharpie
2. Radio and emergency contact numbers
    a. Spare radio battery, personal locator beacon, cell phone
3. 6 Sample bottles in carry bag with sharpie for labeling
    a. 1 L carry bottle
    b. Total Chemistry bottle
    c. Nutrients bottle with 2ml acid
    d. Filtered metals with 2ml acid
    e. Bacteria bottle
    f. Turbidity vial
4. Coozy with ice for bacteria
5. Flow
    a. Wading rod or calibrated bucket and baggie
    b. Survey tape and clamps
    c. AquaCalc and spare parts including spare 9v batteries
    d. Flume, spare parts, nut driver, tables and level
    e. Mini shovel
6. Troll with sensors calibrated and installed
    a. Spare sensors
    b. Connect cable
    c. Cal cup with sponge
    d. Rugged Reader with stylus
    e. Protective stainless steel cap for submerging in stream
7. conductivity standards in mesh bag
8. Thermometer
9. Clothing (winter)
    a. Chest waders, life vest, gloves, hat, matches

2.3. Field checklist, CO parks

See USGS National Field Manual procedures outlined in Chapter A1 section 1.1.1 pgs 12-14. This is the check list used by hydrologists in CO network parks.

http://water.usgs.gov/owq/FieldManual/

Items specific to bacteria sample collection are listed in SOP 11 section 3.1

3. Equipment Log

An equipment log book will be kept with each piece of sampling equipment that records the following:

1. Property number and ownership
2. Help line numbers and contact information for support in case equipment problems arise that you cannot solve. This includes official tech support and others you know who have experience with the equipment.
3. Date of fresh battery installation
4. Date when problems with operation of the instrument occurred and description
5. Date when maintenance was performed and by whom
6. Date when parts were replaced and by whom
7. Date and results from every calibration

The **logbook** should be kept in the equipment case so that it can be readily accessed in the field or lab. Much of the above information is stored in the **premobilization notebook page in Field_forms.xls**.

4. Premobilization checks

4.1. Utah parks

For UT park units using the **Troll 9000** and **Troll 9500** sondes and **Field_forms.xls** spreadsheet, the WRD recommended **premobilization equipment check** is designed to minimize trouble with multiparameter probes in the field (Appendix A). Calibrations using high quality **Standards** are made for conductivity, and pH, whereas dissolved oxygen (Clark cell technology) is calibrated to water saturated air. Then the instrument is checked for accuracy against solutions of known conductivity (specific conductance), pH, dissolved oxygen and temperature (measured with NIST-traceable liquid-in-glass thermometer(s) or lab digital thermometer). Precalibration readings (optional) indicate the tendency and degree of a particular instrument and/or sensor to go out of calibration between regular field use and can in be instructive of the remaining sensor life and if short term storage measures are adequate. All of these data are recorded in **Field_forms.xls** premobilization page which automatically compares calibration results and measurements against allowable ranges. A cell in the spreadsheet then reports if the calibration and accuracy are acceptable. If the calibration and accuracy check do not meet the WRD recommended guidelines, the user must consult the appropriate chapter in the user manual and take action to fix the problem.

4.2. Colorado parks

Hydrologists in Colorado park units perform instrument checks and premobilization tasks as per USGS National Field Manual procedures outlined in Chapter A1 pgs 9-16. http://water.usgs.gov/owq/FieldManual/

5. Data quality objective for core parameters

Quantitative ranges in allowable variation have been established to ensure data collected using the multiparameter probes are representative (Table 1). The following table lists the allowable range in measurement values. This is a measure of accuracy, or repeatability in field measurements that will help guard against collection of spurious data either due to improper equipment handling, or in sufficient time for probe equilibration with ambient water. The allowable ranges are drawn from USGS field manual and NPS-WRD guidance. These are the values against which premobilization error checks are compared.

Table 1. Recommended MP Instrument Post-Calibration Check Error Limits (NPS Core Parameters). These are also the Data Quality Objectives (DQO's) discussed in the Quality Assurance standard operating procedure (SOP 7 section 7.2).

CORE PARAMETER	Sensor Measurement (acceptance/rejection criteria)	Optimum Goal/Target (Range) (USGS criteria)
Temperature	± 0.3 º C	± 0.2 º C
[1]Specific Conductance[2]	± 5 µS/cm or ± 3%	± 5 µS/cm or ±3%
pH[3]	± 0.3 S.U.	± 0.2 S.U.
Dissolved Oxygen[2]	± 0.4 mg/L, ± 5% saturation	± 0.3 mg/L, ± 3% saturation

[1] Whichever is greater
[2] Parameter values (SC and DO) may be affected by 2 to 3% per degree Celsius change in temperature
[3] If after a 2–point calibration and performance of any necessary pH sensor maintenance, error criteria are still not met, a 3-point calibration should be performed before sensor is rejected and replaced.

6. Equipment Calibration

Refer directly to equipment manual (Appendix E-M) for calibration instructions and troubleshooting suggestions.

6.1. Flow meters

1. Spin test flow meters before every site visit
2. Greater than 45 second spin required before measurements are made.
3. If this is not achieved, refer to Appendix L and M for maintenance instructions.

6.2. Multiparameter probes

1. Calibrate pH, DO, and conductivity with standards in temperature equilibrium with ambient water before taking measurements at each site.
    a. Note: Clark cell DO sensors must be re-calibrated at each new site and pH should at least be checked against a standard buffer (e.g. pH=7)!
    b. Optical DO may not require re-calibration at every site but that decision should be based on experience gained with use over time. Check with instrument manufacturer.
2. Calibration results are copied and pasted directly into the Field_forms.xls spreadsheet (SOP 8 section 11), or recorded manually on hard copy and entered into personal computer field forms (PCFF) in CO units.

7. Equipment handling and calibration

7.1. Colorado Units - Hydrolab

7.1.1. DO calibration

Hydrologists in the CO units of the NCPN use Hydrolab multiparameter probes and follow the user handbook for operation and the USGS National Field Manual for calibration procedures (Appendix G).

7.1.2. pH calibration

Hydrologists in the CO units of the NCPN use Hydrolab multiparameter probes and follow the user handbook for operation and the USGS National Field Manual for calibration procedures (Appendix H).

7.1.3. Conductivity /SC calibration

Hydrologists in the CO units of the NCPN use Hydrolab multiparameter probes and follow the user handbook for operation and the USGS National Field Manual for calibration procedure (Appendix I). The only exception being that for calibration of conductivity use air (0 µs/cm) as the first calibration point instead of a low standard.

Note: Most MP instruments/SC cells I believe take a 1 pt. cond/SC calibration so it is usually best to first check (not calibrate) the SC sensor in air (for zero reading), then make a 1 pt. calibration with the with 1413 uS/cm std. and then if desired check the linearity of the sensor at high and low standard values across a broad range (e.g. 100 to 10,000 uS/cm. This is best done in the office but may be done occasionally in the field when desired.

7.1.4. General care and maintenance

Follow guidelines in operator manual

7.2. Utah Units - Troll 9000/9500

7.2.1. DO calibration

Hydrologists in the UT units of the NCPN use In-Situ multiparameter probes and follow the user handbook for operation and calibration. A two point calibration for zero DO and 100% DO is performed in the lab. In the field a 100% DO calibration is made with water saturated air. Appendix B.

Note: from chapter 6.2.1 NFM USGS

1. Test the instrument to ensure that it will read zero in a DO-free solution. See Appendix A.
2. If the instrument reading exceeds 0.2 mg/L, then the sensor membrane and electrolyte (if present) need to be replaced or the sensor needs to be repaired

7.2.2. pH calibration

Hydrologists in the UT units of the NCPN use Instu multiparameter probes and follow the user handbook for operation and use a 2 point calibration in the lab and in the field (pH 7.0 and 10.01)) meant to bracket typical water body pH. Appendix B.

7.2.3. Conductivity calibration

Hydrologists in the UT units of the NCPN use In-Situ multiparameter probes and follow the user handbook for operation and calibration. A one point calibration with KCl is made at 1413 us/cm

Note: use only KCl conductivity standards. That is the type for which internal temperature calibration works (Appendix B).

7.2.4. Temperature

A temperature check is made in the lab prior to field mobilization. (Appendix A) by comparing thermistor reading to that of an NIST certified liquid thermometer or less expensive NIST traceable thermometer.

7.2.5. Multiparameter probe storage

After calibration and during a field run soak a sponge in ambient water and place in the calibration cup to maintain 100% humidity. Attach the calibration cup to the probe body with all sensors installed and store the probe in an upright position whenever possible. For long term storage between field runs remove sensors from the probe body.

1. Conductivity sensor – store dry
2. DO sensor – clean as per user manual Appendix B and store dry.
3. pH sensor – to preserve the sensor's reference solution, store the sensor in the electrode storage bottle in the solution in which it was shipped. A good alternative is a strong potassium chloride (KCl) solution, such as In-Situ's 58,670 µS/cm conductivity calibration solution. Insure the bulb is immersed to keep it hydrated (Appendix B).

7.2.6. Turbidity

Hydrologists in CARE, BRCA, and ZION measure turbidity with a Hach 2100P Turbidimeter (Appendix M).

It is calibrated with primary turbidity standards whenever the secondary standard values begin to drift. The degree of unacceptable drift is defined as +/- 3% in the Field_forms.xls spreadsheet. Follow the calibration procedures outlined in the user manual (Appendix M).

Note: There are now 10 (NTU, NTRU, BU, AU, NTMU, FNU, FNRU, FBU, FAU and FNMU) reporting units for turbidity being adopted by USGS, ASTM, ISO 7027 and EPA (STORET) based on the light source (white or broadband), the degree of the detection angle (90, 180, or 30 to 15 degrees), use of single or multiple detector units, with or without ratio compensation and if a backscatter configuration is used in the sensor. See

USGS Office of Water Quality Technical Memorandum 2004.03 and NFM Chapter 6.7 in Appendices Q and R. The Hach 2100P employs a white or broadband (400-680 nanometers) light source, a 90 degree detection angle and multiple detectors with ratio compensation so the reporting units are NTRU.

8. Troubleshooting

8.1. Colorado Units -Refer to the user manual Appendix E

8.2. Utah Units

8.2.1. Troll 9000 Elastomer

Frequently the Troll Probe will not make an electronic connection to the Rugged Reader through the cable. Most often this can be corrected by disassembling the backshell and crushing the elastomer flat to make a better electrical connection. Occasionally the wires on the elastomer must be sanded to achieve a connection. This is not a problem on Troll 9500 units which have a different connector.

8.2.2. Conductivity sensor

Rarely have any issues occurred with the conductivity sensor. Be sure to never use partially frozen conductivity solution. Freezing will un-equally distribute the salts in solution and that which can be poured into the calibration cup will not have the conductivity stated on the bottle.

8.2.3. pH sensor

The pH sensor is generally reliable as long as it is stored in a high conductivity solution to prevent diffusion and loss of filling solution through the glass bulb. Also, store the sensor in an upright position to prevent gas bubbles getting into the bulb.

Of the core parameters measured when using optical DO, the pH sensor is now the least reliable/robust sensor of the 4 (Temperature, SC, pH, DO) so requires the most care during use and storage. This is critical to be aware of and each manufactures short and long term storage recommendations should be followed.

8.2.4. DO sensor (Clark cell)

- The DO sensor gradually looses sensitivity and electrodes must be cleaned as per instructions in Appendix P. Every other month, or when sonde batteries die is a good time for cleaning.
- The DO calibration in the field can be accelerated considerably if the probe is placed in the stream for 5 min prior to calibration.
- If calibration doesn't move past nominal after 20 minutes accept nominal. The reason for this is to prevent water in the cal cup from warming to air temperature during calibration.
- Wrapping the sonde/sensor guard in a wet towel (white) that has been dipped in the water body for temperature equilibration is also a useful technique to calibrate the DO sensor in the field as this will also provide a 100% water saturated air local environment for calibration.

- The most effective solution is to submerge the sonde in the water body right up to the air vent. This can be difficult to manage and may be impossible in shallow water, but it is by far the quickest method to achieve calibration stability when water temperature and air temperature are very different.

8.2.5. Top setting wading rod

- The top setting wading rod is maintenance free and durable but not indestructible
- Do not bend the rod
- When transporting ensure all screws are tightened so they do not get lost
- Ensure the wire assemblies to not get caught on brush or rocks. These are likely the weakest part of the wading rod
- If the wire breaks loose, scrape loose ends with a sharp knife to 'brighten' and remove corrosion then wrap tightly around the connector at base of wading rod to ensure a good connection is made.

9. General care and maintenance

Refer to equipment manuals for care and handling of equipment. Each instrument is unique and requires a thorough knowledge of its operation for safe and trouble free calibration and measurements.

1. Avoid jarring blows
2. Keep dry
3. Maintain fresh batteries.
4. Do not transport while attached to cables
5. Ensure the wire connectors are firmly attached or contact will not be established (a common issue)
6. Protect the LCD screen from scratches by transporting with a secure cover
7. Press the keys firmly to ensure data entry is successful
8. All field equipment that comes in contact with native water should be cleaned by rinsing with clean tap water.

10. Software updates

Latest Software versions for the Troll 9000/9500 and Rugged Reader can be downloaded free from the In-Situ website after the original software has been installed. Updates should be downloaded at least once per year from http://www.in-situ.com/In-Situ/Downloads/Downloads.html (accessed 9/8/05)

11. Vehicle equipment

1. Check vehicle for safety equipment and supplies including
    a. Jack
    b. Shovel
    c. Jumper cables
    d. Flashlight

    e. Warning cones
2. Material safety data sheets
3. Flares, and remote communications system

12. Post sampling activities, UT

1. Download data from camera (hot swappable USB port copy and paste), Aquacalc (Appendix E), Rugged Reader (see SOP 8 pg 15 'download to PC')
2. Fill out Field_forms.xls and check column J for completeness
3. Make hard copy archive of completed Field_forms.xls pages
4. Update field folders and computer files.
5. Log samples for preparation to ship to laboratory (Lab sheets and Run sheets)
    a. E-mail *.csv file runsheet in Field_forms.xls to Arne Hultquist arnehultquist@utah.gov.
    b. Email North Creek flow data to Amos Rehm
        i. arehm@utah.gov
6. UPS water samples to laboratory
    a. Record UPS tracking number in Field_forms.xls
7. Store standards in refrigerator.
8. Check that all equipment is clean and properly stored.
9. Prepare a list of items to be ordered.

13. Post sampling activities, CO

1. Download data from Aquacalc (Appendix E)
2. Update PCFF forms for each site visit
3. Print archival copies of completed forms
4. Prepare samples for shipping as per Wilde et al., 2004 in Chapter 5 in USGS National Field Manual http://water.usgs.gov/owq/FieldManual/

14. Citations

Wilde, F.D., January 2005, Preparations for water sampling (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A1, accessed 8/7/06, from

http://pubs.water.usgs.gov/twri9A/.

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T., eds., April 2004, Processing of water samples (version 2.1): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A5, accessed __*date*__ at http://pubs.water.usgs.gov/twri9A5/ (Separate updates for 5.6.1.F, "Wastewater, pharmaceutical, and antibiotic compounds," 5.6.4.A, "Arsenic speciation," and 5.6.4.B, "Low-level Mercury" are provided on the Web page.)

15. Appendix A: Multiparameter probe premobilization check UT parks except DINO

The purpose of a premobilization procedure is to ensure the multi-parameter probe is calibrated, operating within acceptance criteria and this is documented prior to field work. The procedure involves the following:

- Making parameter readings prior to instrument calibration
- Calibration of the instrument sensors with known standards or by following established procedures
- Making parameter measurements on known standards and reference solutions
- Comparing values with established acceptance criteria and determining sensor error

Following this customized premobilization procedure is the latest revision (March 2007) of NPS – WRD guidance (personal communication, Peter Penoyer) on premobilization instrument checks. The concepts and intent of this document have been incorporated into the "premob" worksheet of Field_forms.xls specifically to work with the In-Situ Troll 9000/9500 multiparameter sonde. The metadata for "premob" worksheet follows these instructions and can also be found in SOP 8 Appendix B where other meta data for Field_forms.xls exists.

15.1. Terminology

The terms "standards" and "solutions" (reference or check solutions) will have restricted use in these procedures. "**Standards"** will refer to a high quality reference preparation of some documented accuracy provided by a reputable vendor. "**Solutions"** as in reference or check solutions as used herein will refer to those relatively simple preparations made by monitoring staff whose accuracy may not be readily documented but nonetheless serve an important purpose for field checking sensor measurement and linearity (e.g. zero DO solutions). In general, **readings** will be the term used for instrument measurements made on standards and solutions before calibration of the instrument and **measurements** will be the term used for post calibration values collected. Likewise, **results** are the EPA STORET terminology used for measurement values obtained from environmental media made with calibrated instruments.

15.2. Rinsing

It is exceedingly important that sensors be rinsed thoroughly between readings and measurements of conductivity and pH calibration standards. Thorough rinsing will reduce the possibility of error caused by carry over of droplets of calibration standards.

After removing a sensor from a calibration standard and before placing it in the next standard, rinsing should include at a minimum two DI water rinses of the sensors and cal cup, a larger quantity rinse of the calcup inverted and sloshed over the sensors, then 2 rinses with the subsequent calibration standard.

Things to do the day before premobilization when using a Clark cell DO sensor (at the start of the field season and one or more times thereafter during the field season). Performing precalibration readings of the DO sensor prior to each field mobilization over the course of the field season is optional but often informative:

- Check to see if the dissolved oxygen (D.O.) sensor needs to be changed.(If it does, see the powerpoint file titled " D.O. cleaning and maintenance" in Appendix P of this SOP. Note that new membranes may require 12 or more hours to "relax" before measurements are most accurate. Check for updates at
- http://www.in-situ.com/In-Situ/Downloads/Downloads_Presentations.php)
- If cleaned let the DO sensor condition overnight by installing and taking 1 reading
- Make 0% dissolved oxygen reference solution (instructions below)
- Make 100% dissolved oxygen reference solution using aquarium bubbler
- Pour one tray of water deep enough to fully submerge the sonde sensors and run the aquarium bubbler overnight to saturate and equilibrate with room temperature.
- Alternatively use a flow cell and "bubble box" arrangement to saturate a water sample quickly to perform the saturated DO solution measurement.

If not using the Thoma / Malick DO calculator at (http://www.nature.nps.gov/im/units/ncpn/Tools.cfm) which is built into the premobilization worksheet in Field_forms.xls look up and calculate theoretical equilibrium DO saturation (for comparison with your readings) from the USGS National Field Manual DO tables and local station pressure (uncorrected to sea level) or hand held display/built in barometer.

15.3. Premobilization Quick Start Guide:

1) Set up tap water bath overnight for temperature and DO equilibration

2) Take precalibration readings in water bath with sonde and thermometer

3) Take SC precal readings in 146, 1413, 12900

4) Calibrate SC in 1413

5) Take postcal SC readings in 1413, 146, 12900

6) Take pH precal readings 4,7,10

7) Calibrate pH in 7, 10

8) Take postcal pH readings in 10, 7, 4

9) Take precal zero DO reading

10) Take precal 100% DO reading

11) Calibrate 100% DO

12) Take postcal 100% DO reading

13) Take postcal zero DO reading

14) Get pressure reading from airport barometer on-line

15) Take post calibration water bath sonde reading with sonde and thermometer

16) Optional: take readings cal solutions used last month in the field

15.4. Long Version of Premobilization procedures

Step 1: Print out the premobilization sheet

1. This file can be found in the first tab for the "Field Forms" file for the current month
2. The shaded areas are the places that will be filled in. All other cells are formulas that will be filled in automatically.
3. All pre- and post-calibration readings are entered on this sheet
4. Before printing the file, type in the operator name and verify that the equipment specifications have not changed since last calibration.
5. Be sure to note any equipment maintenance including
    a. New batteries
    b. New sensors
    c. Cleaned sensors

Step 2: Before beginning the premobilization procedure

1. Locate all of the equipment that will be needed (see list below)
2. Make sure the batteries for the probe and Rugged Reader have sufficient power
3. Check to see if any of the sensors need to be replaced (this is especially true for the dissolved oxygen (DO) sensor, which has to be conditioned for 24 hours before you can continue with the procedure).

Equipment

1. Premobilization sheet
2. Sonde
3. Probes (Sensors) for sonde
    a. pH
    b. dissolved oxygen
    c. conductivity
4. Rugged Reader
5. Yellow cord to attach sonde to Rugged Reader

6. 0% dissolved oxygen solution made with sodium sulfite and cobaltus chloride (instructions below)
7. 100% dissolved oxygen solution (white plastic tray with aquarium bubbler)
    a. Sodium sulfite (Na_2SO_3) crystals
    b. Cobaltous (Cobalt (II)) chloride ($CoCl_2$) crystals
    c. Small bucket to collect waste solutions recovered from sensor rinses
8. Conductivity/SC standards at room temperature, 1413, 147, 12,880
9. pH standards at room temperature, pH 4, pH 7, pH 10
10. Beakers for solutions (probe needs to fit into beaker)
11. Lab stand (to hold sonde)
12. Deionized water

Step 3: Setting up the MP Troll 9000/9500 and Rugged Reader

1. Carefully insert the sensors in the correct ports of the sonde (please see p.12-13 of MP Troll 9000/9500 Manual in Appendix B of SOP 5) and connect the sonde to the Rugged Reader with the yellow cord. (Always handle the sensors, particularly the DO sensor, gently and never touch the tips).
2. After turning on the Rugged Reader (power button is on the lower right of the key pad and is encircled). The "My Info" page will appear.
3. Touch the screen with a stylus (a pen or pencil) and a new screen will appear. Touch "Start" in the upper left of the new screen and a menu will appear.
4. From the menu, select "Pocket-situ 4". Next, double-click (touch twice) on the "COM1-19200" option. After a few seconds, double-click on the "Parameters" option that will appear.
5. Select the desired parameter by touching it. Upon testing, the information and readings will appear on the screen below the menu portion. Notice the "Read" and "Calibrate" commands at the bottom of the screen.
6. To exit, touch the "X" in the upper right of the screen. Select "Yes" to exit Pocket- situ. You are back at the screen with "Start" in the upper left corner. Turn off the Rugged Reader with the power button.

Step 4: Premobilization pre-calibration water bath or sink water readings

1. Repeat steps b through d above.
2. Touch "parameters" and the "profiler" tab should appear in the lower right of the screen
3. Touch "profiler" to open a window that allows naming a file.
4. Touch the keyboard icon to allow typing and enter the premobilization profiler file name according to the following convention, "pm_**pre**_month_day_year"
5. Place the sonde in the tray of water that stood overnight *with* the bubbler
6. Place the NIST mercury thermometer in the tray next to the sonde
7. Only the bulb need be immersed. This type of thermometer is calibrated to read accurately with just the bulb in the water.

8. Allow both the sonde and thermometer to equilibrate for 5 minutes
9. Record the NIST thermometer reading.
10. Be sure to turn off bubbler to keep bubbles from collecting on the DO sensor and make sure to stir the solution if using a Clark cell that consumes oxygen at the membrane tip.
11. Record 7 sonde readings by touching "snapshot" 7 times in succession
12. Touch "close" to store the data and exit profiler mode
13. This file will be downloaded and pasted into Field_forms.xls
14. Note: This step will be repeated in Step 8 after calibrating sensors

Step 5: Pre-calibration readings (optional) and calibration of specific conductance (SC)

Note: this tells you how much the calibration may have shifted since the last use of the sonde. You gain insight into the stability of your sensor this way, but its not mandatory.

1. Pre-calibration measurements
    a. Take pre-calibration readings of specific conductance (SC) for three standards (SC 147, 1413 and 12,880 uS/cm).
        i. As a general rule with conductivity start with lowest standard and work up in conductivity when taking readings or when calibrating to minimize solution carry-over error. Also, rinse copiously with tap water, then DI water and several times with the next solution in which the sensor will be submerged to eliminate potential for dilution or carry over concentration effects. Copious rinsing means lots of small volume rinses, **NOT** one large volume rinse. Use of a heavy mist spray bottle for initial rinses and blotting dry with Kimwipes before final rinse with the standard also works well.
        ii. For example when going from 147 to 1413 uS/cm standard rinse well with DI water, blot dry with Kimwipes and then rinse with 1413 before loading cal cup with the 1413 uS/cm standard. Likewise after a precal measurement of the 12,880 uS/cm standard, rinse copiously with DI water, then rinse with 1413 before loading cal cup with 1413 uS/cm standard.
    b. Standards readings
        i. Screw the bottom onto the Cal Cup, fill to fill lines with lowest conductance solution (here 147 uS/cm) and attach to sonde. Make sure both the conductance sensor and the metallic temperature sensor are adequately immersed in solution. Rotate entire apparatus back and forth to ensure mixing.
        ii. On Rugged Reader, choose "Conductivity" from "Parameters" (please see "Step 3" above). Touch "Read" and record reading on pre-mobilization sheet. Repeat for temperature.
    c. Prep for next standard
        i. Before reading the next higher conductivity standard (here 1413), rinse the sensor(s) well. First, fill the Cal Cap with tap water, attach to the sonde, and rotate back and forth on lab stand

to assure a complete rinse. Repeat with deionized (DI) water. Then gently shake off the excess water. Finally, rinse sensor with a small amount of the solution that will be tested next (here 1413).

ii. Repeat the above procedure for the next two solutions, making sure to go in ascending order of conductivity standard (here 1413 then 12,880).

2. Conductivity Calibration - One step calibration using 1413 uS/cm standard
    a. Fill Cal Cup as above with 1413 standard and touch "Calibrate" at the bottom of the screen. On the next screen (where 1413 is set as the default), select "Next" then "Run". The sensor is now calibrating
    b. The screen first reads "Unstable", then "Nominal", then "Stable". When it reads "Stable", touch "Finish".
    c. You will be given the option of viewing the calibration reports. It is not necessary to view them as the information is later downloaded from the Rugged Reader to a computer then pasted onto the Excel sheet with other pre-mobilization data.
    d. After selecting "No" or viewing the calibration reports then touching "X", the screen will return to the menu from which more readings can be taken.
3. Post-calibration Measurements
    a. Repeat measurement of the same three standards as in the pre-calibration readings.
    b. As in the pre-calibration readings, measure the conductivities of the three standards in order of ascending conductivity, remembering to rinse well between the measurements

Step 6: pH Calibration

1. Pre-calibration sensor readings of pH
    a. Take pre-calibration readings of pH for three standards pH 4.0, 7.0 and 10.01.
        i. As with conductivity standards readings and measurements always start with the lowest standard and rinse thoroughly with water and the next standard before taking the next reading or measurement.
    b. Start with the lowest pH (here pH 4.0) standard in the Cal Cup
    c. On the Rugged Reader, choose "Parameters" then "pH". Touch "Read" then record the data on the pre-mobilization sheet. After proper rinsing read pH of subsequent solutions in ascending value of pH (here 7.0 then 10.0).
2. Two Point Calibration with pH 7.0 and 10.0 or 4.0 and 7.0
    a. Note: bracket anticipated environmental waters with 4.0 and 7.0 or 7.0 and 10.0 in a two point calibration when feasible and follow directions below. These are room temperature calibrations.

b. Pour lower pH standard (here pH 7.0) into Cal Cup and attach to sonde.
c. From the "Parameters" menu, touch "pH" then "Calibrate". On the next screen (where 7.0 and 10.0 are the default settings), select "Next" then "Run". Wait for the screen to make the progression from "Unstable" to "Nominal" to "Stable". Screen will show that it's ready for next pH solution to be tested.
d. Rinse the sensor(s) thoroughly with water and pH 10 standard before loading pH 10.01 standard in the cal cup.
e. When "Stable" appears, touch "Finish". It is unnecessary to record the calibration report by hand as the information is later downloaded from the Rugged Reader to the Field_forms.xls spread sheet with other pre-mobilization data.
f. After selecting "No" (to view the calibration reports) or viewing them and touching "X" in the upper right-hand corner of the screen, you return to the screen which allows more readings to be taken. Rinse the sensor(s) thoroughly and perform the post-mobilization step described below.

3. Post Calibration Measurements
   a. Repeat measurement of the three solutions measured in the pre-mobilization
      i. As in the pre-calibration readings, measure the pH and temperature of the three solutions in ascending order of pH (here 4.0, then 7.0, then 10.0), making sure to rinse properly between different solutions.

Step 7: Calibration of dissolved oxygen

The calibration of dissolved oxygen (DO) differs from the other calibrations because there is not a pre-made calibration standard solution. There are two solutions that need to be prepared; one for 0% DO (required) and another for 100% DO (recommended).

1. Preparation of 0% DO Reference solution
   a. Add ~1 gram of reagent grade sodium sulfite crystals and a few crystals of cobaltus (II) chloride to 1 liter of deionized water, leaving minimum headspace.
   b. Invert the container several times to dissolve the crystals into solution and allow it to stabilize for a few minutes.
      i. For checks in the field prepare the two chemicals in the proportions above in a dry 1 liter bottle, take to the field and just add DI water when needed
   c. The resulting DO measurement performed on this solution should be less than or equal to 0.2 mg/L. If this is not achieved, allow the solution to sit overnight with no air in the bottle by squeezing the bottle until the meniscus is at the bottle rim then cap tightly. This gives the sodium sulfite a chance to consume oxygen. If the bottle is stored

with out headspace it can be used many times. It can also be recharged by adding more sodium sulfite and cobaltus chloride.

2. Preparation of the 100% DO Reference Solution from Tap Water (optional)
    a. Make a water bath using a plastic container and an aquarium bubbler. Place the bubbler no more then 6 inches under the water surface to avoid over-saturation. The water should reach saturation overnight (~12 hours).
    b. Operation of the bubbler overnight at a high rate can lead to over saturation of the water bath (from stone bubbler). In general it is best to use an intermediate / moderate bubble generation setting which should work best for overnight applications. In-Situ makes a flow cell with an inexpensive bubbler attachment ("bubble box") to achieve a more rapid saturation of a smaller volume flow cell (for use in field or lab) quickly (several minutes – usually 10 to 20) than waiting overnight depending on adjustable bubbler setting.
    c. In a 5 to 8 gallon capacity lab sink saturation will be achieved overnight. Smaller containers will achieve saturation much quicker and a flow cell with "bubble box" attachment can achieve saturation in 10 to 15 minutes. That is the least time consuming alternative.

3. Pre-calibration DO Sensor Readings
    a. Take pre-calibration readings for
        i. 100% DO reference solution
            1) Lower the sonde directly into the bubbling water. Position the sonde at a slight angle from vertical and away from the bubbler to avoid having a bubble on the sensor tip, or better yet turn off the bubbler and stir or wave the sensor through the solution.
            2) To read the DO, select the "dissolved oxygen" option from the "parameters" menu. Then touch "read".
            3) Record the readings in the appropriate shaded area of the pre-mobilization sheet.
            4) Record reading on the pre-mobilization sheet.
        ii. 0% DO reference solution
            1) Lower the sonde directly into the bottle and try to avoid allowing air into the bottle. Having used a bottle with an appropriately sized mouth, you can seal most gaps with Para-Film.
            2) Perform the 0% DO reading in "Profiler" mode. This allows you to track change in DO with time as it may take several minutes or even hours to get the lowest DO reading.
            3) Record reading on the pre-mobilization sheet.
            4) (Note- the 0% DO step can be time-consuming and often gives wide-ranging readings, so it may be skipped

in the pre-calibration. It should be performed in the post-calibration, however).

iii. Water Saturated Air
    1) Fill and attach the CalCup with water dipped from the bubbler tray (This will ensure a fast calibration when water is equilibrated to room temperature. It has nothing to do with using DO saturated water from the bubbler) and invert the sonde on the lab stand.
    2) The top of the DO sensor should be above the water level and free of moisture, but the temperature sensor should be beneath the water surface.
    3) Loosen the CalCup so the vent in the threads is visible, thus ensuring the water is at ambient pressure.
    4) Record reading on the pre-mobilization sheet.
    5) Proceed to the calibration step

4. DO Calibration- air saturated with water (DI or tap, doesn't matter)
    a. If water saturated air reading was the most recent reading made the instrument is already setup to calibrate.
    b. Otherwise, repeat the set-up (inverted sonde) described above for water saturated air
    c. From the bottom right of the screen select "Calibrate", then select "Next", then "Run".
    d. The probe will at first read "unstable", then "nominal", then "stable". This process takes longer then the other calibrations (12-20minutes). If it is still not reading stable after 30 minutes, it may be necessary to cancel the current calibration and restart the procedure.
    e. After it reads stable, select "finish" and a screen will pop up giving the option to view the calibration reports. Viewing the calibration reports is not necessary; the information is later downloaded and pasted into Field_forms.xls spread sheet with the other pre-mobilization information.
    f. After viewing the calibration reports and touching "X" or selecting "no" (to view the calibration reports), you return to the screen from which more readings can be taken.
    g. Record a post calibration measurement on the pre-mobilization sheet

5. Post-calibration Measurements
    a. Take readings for
        - 100% DO reference solution (optional),
        - 0% DO reference solution and
        - water saturated air
            i. Repeat the measurements for water saturated air, 100% DO and 0% DO solutions as described in the "pre-mobilization readings".

ii. Be sure to turn off bubbler to keep bubbles from collecting on the DO sensor and make sure to stir the solution if using a Clark cell that consumes oxygen at the membrane tip.
iii. Record a post calibration measurement on the pre-mobilization sheet

Step 8: Premobilization post calibration water bath or sink water readings

1. Repeat Step 4 except this time name the file "pm_**post**_month_day_year"
2. This file will be downloaded and pasted into Field_forms.xls

Step 9: Complete Premobilization Instrument Check

1. Download calibration reports and premob_pre and premob_post data files
2. Place them in the appropriate locations in the premob worksheet in Field_forms.xls.
3. Calculations for error and sensor constants will be made automatically.
4. Review the results and if any problems are apparent resolve them before leaving for the field.

16. Field_forms.xls premobilization worksheet metadata

item 1	item 2	cell range	description
	Date	B3	date of premob test
	Start time	B4	start time of testing
	End time	D4	finish time of testing
	Operator	C5	name of person conducting test
	Inst. Mfr.	C6	maker of multiparameter probe
	Sensor serial number	C7	serial number of probe body
	Model	C8	model of probe body
	Display	C9	unit that allows viewing parameter data
	Display voltage (%)	C10	voltage of display unit
	Sonde (v)	C11	voltage of probe
	Sondepower (%)	C12	power remaining in probe
	Notes:	C13	any notes specific to this calibration and error check procedure
	Sensor	H4:H9	type of sensor attached to probe body
	Sensor ID	H4:H9	identification number of sensor attached to probe body
Error Check Solution: Sink water, and sodium sulfite solution			
Pre-Cal. Measurements	MP temp ^{O}F	C18	multiparameter probe temp (degrees F)
Pre-Cal. Measurements	NIST Thermometer ^{O}C	C19	NITST thermometer (degrees C)
Pre-Cal. Measurements	SC (us/cm)	C22	multiparameter probe specific conductivity (uc/cm)
Pre-Cal. Measurements	pH	C23	multiparameter probe pH
Pre-Cal. Measurements	DO (mg/L)	C24	multiparameter probe DO (mg.L)
Pre-Cal. Measurements	BP (mmHg)	C25	multiparameter probe barometric pressure (mmHg)
Post-Cal. Measurements	MP temp ^{O}F	J18	multiparameter probe temp (degrees F)
Post-Cal. Measurements	NIST Thermometer ^{O}C	J19	NITST thermometer (degrees C)
Post-Cal. Measurements	SC (us/cm)	J22	multiparameter probe specific conductivity (uc/cm)
Post-Cal. Measurements	pH	J23	multiparameter probe pH
Post-Cal. Measurements	DO (mg/L)	J24	multiparameter probe DO (mg.L)
Post-Cal. Measurements	BP (mmHg)	J25	multiparameter probe barometric pressure (mmHg)

Temperature error check	%Error	I28	automatically calculated percent error
Temperature error check	Allowable?	J28	answers question is this level of erorr OK?
	pre-mob_pre file name	A31	name of data file where premobilization sink water data is stored
	pre_mob_post file name	G31	name of data file where premobilization post-calibration sink water data is stored

Specific Conductance Calibration (1413 µS/cm std.) & Error Check @ 100 and 10,000 µS/cm

Pre-Cal. Reading	reading	B35:B37	multiparameter probe precalibration specific conductivity readings in conductivity standards
Pre-Cal. Reading	lot #	C35:C37	lot number of the indicated conductivity solution
Pre-Cal. Reading	Exp	D35:D37	expiration date of the indicated pH buffer solution
Pre-Cal. Reading	maker	E35:E37	manufacturer of the indicated pH buffer solution
Pre-Cal. Reading	Cat #	F35:F37	catalog number of the indicated pH buffer solution
Post-Cal. Reading		H35:H37	Specific conductivity reading after calibrating at 1413
Post-Cal. Reading	%Error	I35:I37	computes percent error
	Allowable?	J35:J37	answers is this error level allowable?

pH Calibration @ pH 7 & pH 10 su and Error Check @ pH 4

Pre-Cal. Reading	reading	B52:B54	reading in the indicated pH buffer solution
Pre-Cal. Reading	lot #	C52:C55	lot number of the indicated pH buffer solution
Pre-Cal. Reading	Exp	D52:D56	expiration date of the indicated pH buffer solution
Pre-Cal. Reading	maker	E52:E57	manufacturer of the indicated pH buffer solution
Pre-Cal. Reading	Cat #	F52:F58	catalog number of the indicated pH buffer solution
Post-Cal. Reading	%Error	I52:I54	percent error
Post-Cal. Reading	Allowable?	J52:J54	answers is this error level allowable?
	voltage pH 4 std	C57	sensor voltage (mV)
	voltage pH 7 std	C58	sensor voltage (mV)
	voltage pH 10 std	C59	sensor voltage (mV)
	temp deg C	F57:F59	temperature of corresponding buffer solution
For 2 point calibration	Slope 1 mV/pH	E62	slope of calibration curve (mV/pH), 2pt calib
For 2 point calibration	Offset 1 mV	E63	offset of calibration curve (mV), 2 pt calib
If making a 3 point calibration	Slope 2 mV/pH	I62	slope of calibration curve (mV/pH), 3 pt calib
If making a 3 point calibration	Offset 2 mV	I63	offset of calibration curve (mV), 3 pt calib

	offset check	D66	check for offset within acceptable range
	slope check	D67	checl for slope within acceptable range
	maintenance needed?	D68	answers is maintenance needed?
Dissolved Oxygen (DO):			
	Membrane thickness (mil)	C74	thickness of DO membrane (mil)
	Calib Method	C75	method used for calibration
	Edit baro. setting?	C76	was the barometric pressure value set by user?
	stimulus	C77	the maximum DO concentration for the measured temperature and pressure at time of calibration 9mg/L)
	Pre-Cal. Reading	C78	reading of DO prior to calibration with sensor in solution indicated in C75 (mg/L)
	Slope	C79	calibration slope (nA/mg/L)
	Offset	C80	calibration offset (nA)
	temp. during cal	C81	temperature during calibration (degrees C)
	baro. during cal	C82	barometric pressure during calibration (degrees C)
	Manuf. Date	E74	manufacture date of membrane cap
Error check1	Method	G75	method used for error check
Error check1	Edit baro. setting?	G76	was the barometric pressure value set by user?
Error check1	stimulus	G77	the maximum DO concentration for the measured temperature and pressure at time of calibration 9mg/L)
Error check1	Pre-Cal. Reading	G78	reading of DO prior to calibration with sensor in solution indicated in C75 (mg/L)
Error check2	Method	K75	method used for error check
Error check2	Edit baro. setting?	K76	was the barometric pressure value set by user?
Error check2	stimulus	K77	the maximum DO concentration for the measured temperature and pressure at time of calibration 9mg/L)
Error check2	Pre-Cal. Reading	K78	reading of DO prior to calibration with sensor in solution indicated in C75 (mg/L)
	Post-Cal. Reading1	C86	post calibration reading with sensor in C75 (mg/L)
	Post-Cal. Reading2	G86	post calibration reading with sensor in G75 (mg/L)
	Post-Cal. Reading3	K86	post calibration reading with sensor in K75 (mg/L)
	%Error	I89:I90	percent error between stimulus and reading in error check solutions
	Allowable?	J89:J91	is this error allowable based on WRD guidance
	Notes:	B99	notes realted to DO calibration
	maintenance needed?	C94	based on acceptable range of calibration curve slope and offset is maintenance needed?
	Bryce Airport BP	C86	sea level pressure at Bryce airport found at www.noaa.gov enter local zip code and scroll to bottom of page to find current

			weather conditions at local airport
	Pressure %error	B90	percent difference between airport pressure adjusted to local altitude and Troll reading at office

Turbidity Calibration: with primary formazin standard (Minimum once every 3 months)

Gelex	Measured value	C107:G107	value of currently measured gelex standard
Gelex	% rel. diff.	C108:G108	difference between assigned value and current reading
	± 3% Y/N	C109:G109	is the relative difference greater than 3%?
	Recalibration necessary?	G110	is a formazin recalibration necessary?
	Standard Value	B112:E112	formazin standard value (NTU)
	Lot Number	B113:E113	lot number of formazin standard
	Exp. Date	B114:E114	expiration date of formazin standard
	Assigned value to Gelex	C116:E116	expected range of gelex standard (NTU)
	Secondary standards	C117:E117	assigned value based on most current calibration (NTU)
	Date of Latest calibration and assignment of Gelex values	G119	most recent calibration and assignment of values to gelex standards

17. Appendix B. Original Premobilization Guidance from WRD that was modified above to Work with Troll 9000 units

DRAFT (work in progress)

WRD Recommended

Pre-mobilization Multi-parameter Instrument Checks (Freshwater)

(For the Core Required Parameters of Temperature, Sp. Cond., pH, and DO)

Ensuring that multi-parameter (MP) instrument sensors (Temp., Sp. Cond., pH, and DO) for NPS core water quality parameters operate well and meet QC standards/error check limits before field mobilization is critical. **These pre-mobilization MP instrument checks are recommended as a means to ensure Servicewide consistency in QA/QC in MP instrument use and sonde/sensor measurement accuracy after performing a calibration in the office or lab under controlled or optimum conditions.** This should permit your on site field calibrations to run smoothly and quickly. Our goal is to present a methodology that is done in the lab/office, is relatively simple to perform and can become routine, while minimizing demands on monitoring staff. This approach should ensure core parameter data is measured with instruments that meet common performance and acceptance/rejection criteria Servicewide.

These instrument checks compare a calibrated instrument parameter measurement against reference standards or reference solutions across a broad range that are either purchased or prepared and provide a reference value of high confidence. These pre-mob instrument error checks should be done in addition to other regular (quarterly, annual etc.) instrument checks proscribed by network QA/QC protocols and SOPs. An informed and safe operation of these instruments requires more than knowing which buttons to push. An understanding of the principles of operation, calibration techniques and system setup (software and hardware) is necessary to obtain meaningful results and requires that the user read the instrument manufacturers operations manual as the first step.

Table 1 lists the recommended allowable MP instrument post-calibration error limits that instrument sensors should achieve prior to performing core parameter measurements at NPS monitoring sites. Also provided are slightly more stringent criteria of the USGS that should be a goal or target to be adopted within 5 years from the time network monitoring programs are up and running.

Table 1. Recommended MP Instrument Post-Calibration Check Error Limits (NPS Core Parameters)

CORE PARAMETER	Sensor Measurement (acceptance/rejection criteria)	Optimum Goal/Target (Range) (USGS criteria)
Temperature	± 0.3 º C	± 0.2 º C
[1]Specific Conductance[2]	± 5 µS/cm or ± 3%	± 5 µS/cm or ±3%
pH[3]	± 0.3 S.U.	± 0.2 S.U.
Dissolved Oxygen[2]	± 0.4 mg/L, ± 5% saturation	± 0.3 mg/L, ± 3% saturation

[1]Whichever is greater
[2] Parameter values (SC and DO) may be affected by 2 to 3% with each degree change in temperature
[3] If after a 2–point calibration and performance of any necessary pH sensor maintenance, error criteria are still not met, a 3-point calibration should be performed before sensor is rejected and replaced.

Recommended Calibration (pre-error check for freshwater use) of each MP instrument sensor as follows:

Temperature: N/A – calibration of thermistor is done by manufacturer (should be a one-time event by manufacturer unless sensor fails error criteria above)

Specific Conductance: 1- pt. calibration using a 1413 µS/cm standard (for subsequent error check use 100 µS/cm followed by 10,000 µS/cm standards). Specific conductance sensors should be very linear over this range. (time required ~ 5 min.)

pH: 2-pt. calibration using pH 4 & 7 or pH 7 & 10 buffers that best bracket anticipated environmental waters to be monitored (for subsequent error check test with the third buffer not used in the calibration). Note that one Nernst slope is obtained from a 2-pt. calibration. A 3-pt. calibration determines two slopes (i.e. one for the 7 to 10 pH range and one for the 4 to 7 pH range). A 3-pt. calibration may be necessary if error criteria are not met based on a 2-pt. calibration and sensor maintenance is performed or environmental waters to be monitored extend into both the higher and lower pH calibration ranges above and below a pH of 7. (time required ~ 10 min.)

Dissolved Oxygen: A single point, water-saturated air calibration (for subsequent error check test with a prepared zero DO solution or a DO saturated solution achieved with a portable bubbler at some true/station barometric pressure for your location). Available bubbler systems (portable) also now make it possible to readily perform calibrations in oxygen saturated water. Oxygen saturated water calibrations may be slightly more accurate with some MP instrument systems. (time required ~20 – 30 minutes)

Note: All sensors degrade with use and over time. Strict adherence to recommended sensor maintenance and storage conditions between use and during transport of sondes between sites will greatly maximize sensor longevity and minimize field problems.

Recommended Post-Calibration Error Check Procedures:

These error checks should be performed after MP instrument calibration is done per above and consistent with manufacturer's instructions and network protocols. These are best performed connected to the appropriate vendor's handheld display (e.g. 650 YSI

display for use with Series 6 datasonde or minisonde; In-Situ RuggedReader with MP9000 data sonde; Hach-Hydrolab Surveyor Display with Series 5 mini/datasondes or laptop computers with appropriate vendor software). Several other vendors MP instruments are also available (Eureka Environmental Engineering, Global Water Instrumentation Inc., Stevens Water Monitoring Systems Inc., Horiba Instruments Inc. etc.) but will not be discussed here. A **ring stand and clamp** to stabilize position of sonde and avoid damage from falling over or dropping is recommended in conducting error check procedures.

No more than 48 hours prior to field mobilization, instrument sensors should be calibrated and a measurement error check performed. These calibrations are not a substitute for calibrations that should be performed daily in the field or at each monitoring site but are intended to ensure instruments work properly with a high degree of confidence in their measurement accuracy and any sensor problems are found before field work begins. Instrument/sensor checks may be performed as described below for measurements to be made in fresh environmental waters. Slight modifications to these procedures may be appropriate prior to marine monitoring (e.g. salinity measurement checks/using different standards for specific conductance/salinity calibrations in error check measurements and use of salt water baths in sensor checks more representative of ambient saltwater monitoring conditions etc.).

All post-calibration measurements of standards or reference solutions should fall within the NPS post-calibration, measurement check error limits specified in Table 1 (above) prior to use of that sensor in the field. Examples showing actual results of these measurement error checks made by WRD in the lab with a Series 6 YSI instrument are provided below for comparison and reference along with calibration/error check tips/insights gained from MP instrument testing of Hach-Hydrolab and In-Situ sondes by WRD and information reported by other organizations. A completed "Pre-Mobilization MP Instrument Example Log Book Entry" for instrument calibration and error checking is provided at the end of this procedure.

With the exception of conductivity sensors (specific conductance), slightly more stringent acceptance criteria is being adopted by the USGS for calibration check error limits (also provided in Table 1) of selected sensors in their continuous monitoring guidance and these criteria should also be a "goal or target" in NPS core parameter monitoring. These more stringent acceptance/rejection criteria for selected sensors (temperature, pH, DO) should generally be achievable in the lab/office with appropriate calibration technique, using standards of good quality, and with MP equipment/sensors in good working order. Less experienced users of these instruments or instruments with older sensors or those having experienced heavy use may find the USGS acceptance criteria difficult to achieve. Some basic or limited measure of sensor trouble-shooting is recommended if these more stringent USGS criteria are not achieved in the office/laboratory prior to field mobilization [e.g. redo a multi-point calibration curve and/or compare millivolt measurements (e.g. pH sensor) or charge (DO sensor) to the acceptable range suggested by manufacturer, perform sensor servicing and maintenance (cleaning), or other sensor troubleshooting proscribed by manufacturer]. It is recommended that MP instrument sensors be replaced as soon as possible, if (after servicing) they are still unable to meet

the more stringent USGS acceptance/rejection criteria or in the case of temperature sensors, the instrument/sonde should be returned to the manufacturer for recalibration or replacement of the thermistor. If the slightly less stringent NPS error check criteria are not met for sensors measuring the core parameters, network staff should replace sensors before proceeding to the field or if that is not possible, note in the instrument log book that sensor acceptance criteria were not met and document the sensor failure and amount of error.

Sensors degrade with time/use and algorithms used to compute a temperature or salinity compensated measurement can vary slightly among instrument manufacturers. Thus, where two or more instruments are available for measurement comparisons, field measurements should not be expected to agree precisely. However, measurement values of two or more instruments should not disagree by a value more than twice that of the error criteria (i.e. the range indicated by the +/- value). By performing these pre-mobilization calibrations and error checks, field instrument measurement discrepancies should be minimized and greater Servicewide consistency in quality control of core parameter measurements should be realized.

Temperature Error Check (~10 min to 1 hr.): Compare temperature of MP thermistor against that of either a NIST-certified or NIST-traceable digital or liquid thermometer in a stable water bath(s) at one or more points within the range of anticipated water body temperatures to be monitored. As long as the thermometer is in some way referenced to NIST standards (i.e. at least NIST-traceable), has a range from at least -1 ºC to 45 ºC with 0.1 ºC divisions, it should be acceptable. A single point in the 0 to 20 degree Celsius range, preferably near anticipated water body temperature(s) should be sufficient, but multiple points may be chosen based upon any wider range of anticipated temperature conditions of the water body(s) for that monitoring event. Measurements made by the MP thermistor and NIST-certified or NIST-traceable thermistor or liquid-in-glass thermometer for determining error should be made to the nearest 0.1 ºC and agree within 0.2 ºC (i.e. the post-calibration check error limit specified by the USGS). However, measurement agreements within ± 0.3 degree Celsius will be acceptable but thermistors should be replaced or repaired before additional mobilizations occur with this (or greater) amount of temperature error. Note that these single point, pre-mobilization error checks are in addition to the quarterly or annual multi-point calibration/error checks required in maintaining an MP instrument and its log book. These NIST-traceable reference thermometers are typically mercury-filled thermometers and should not be used in the field.

Table 2. Example: Single point error check for MP temperature (thermistor)

Perform after some selected water volume mixture of hot and cold tap water (e.g. sink water bath, bucket, flow cell) is drawn so as to approximate room temperature (typically in range of 18 to 20 °C). Using this well-mixed, room temperature water bath as a stable system (e.g. 18.8 ºC in example below), submerge MP instrument temperature sensor and reference thermometer adjacent to each other in water bath and compare stabilized measurements after a few minutes. (Note: if water bath is significantly different than room temperature, it may not reach stability quickly, may display a vertical/horizontal

temperature gradient to container sides or slowly change in the direction of the room temperature with time). If desired, a two point error check across a temperature range may be performed by running a selected container or sink full from the cold water tap or preparation of an ice water bath, comparing the MP instrument measurement with NIST-traceable thermometer of the cold/ice water bath and then allowing the water to approach room temperature overnight. A second error check at the higher temperature would similarly be performed by comparing the MP instrument (thermistor) and NIST-traceable thermometer measurements of the room temperature equilibrated water bath on the following day.

Table 2. Single point MP instrument (thermistor) error check

NIST-Certified or NIST-Traceable Thermometer*	MP Measurement Result (Thermistor)	Measurement Error
Room Temperature (air) @ 20.8 ºC	20.94 ºC (in air)	~ + 0.14 ºC → acceptable
Water Bath Temp. @ 18.8 ºC	18.94 ºC (water)	~ + 0.14 ºC → acceptable

*May substitute a lower cost NIST-Traceable thermometer if graduated in 0.1 ºC units and calibrated to NIST standards. [e.g. Fisherbrand Thermometer (Fisher Scientific Cat. No. 15-000A) or equivalent]

Table 3. Two point MP instrument (thermistor) error check done overnight in deep lab sink water bath

2 NIST-Certified or NIST-Traceable Thermometer(s)	MP Measurement Result[1] (Thermistor)	Measurement Error
Cold (tap) water bath (day 1) Thermometer 1 14.8 ºC Thermometer 2 14.7 ºC	14.63 °C	- 0.17 °C - 0.10 °C
Ambient/near room temperature water bath (day 2) Thermometer 1 18.8 °C Thermometer 2 18.7 °C	18.67 ºC	-0.05 °C -0.13 ºC

Note that the smaller the water bath/container used, the greater potential for a temperature gradient to occur between the walls of the container and the location of each temperature sensor within the water bath. This can give rise to several hundredths of a degree difference in measurements between sensors for water baths more that a few degrees different from ambient (room) temperature.

[1]A magnifying glass was used to aid estimation of the NIST-traceable thermometer reading to the nearest hundredth degree centigrade so values could be rounded to the nearest tenth of a degree.

Specific Conductance Error Check (~10 min.): Having calibrated the specific conductance sensor of the MP instrument with a standard in the 1000 µS/cm to 1500 µS/cm range per above (**a 1413 µS/cm KCl standard** is commonly used and recommended here), record the conductivity cell constant (e.g. for YSI 6 series sondes go to "**Advanced Menu**" under "**Cal Constants**"). Confirm that the conductivity cell constant reads within the range 5.0 +/- 0.45 for YSI conductivity cells used in 6 Series sondes for freshwater monitoring.

Begin by measuring the lower conductivity check standard first (to minimize carry-over potential from the high conductivity standard) by filling calibration cup to appropriate level. For each check standard measurement, be sure to triple rinse with standard after blotting sensors dry (before first rinse) with Kimwipes™ EX-L (or equivalent). It is very important to avoid any carry over of salts from a higher ionic strength standard by rinsing the sensor with DI water and blotting dry the sensor(s) and inside of calibration cup with Kimwipes followed by triple rinsing sensor(s) with the subsequent standard to be measured. Record measurement of this low conductivity standard after filling calibration cup to appropriate level, inverting cup and sonde at lease twice for solution to contact all surfaces, tapping calibration cup if necessary to dislodge any entrapped bubbles in the conductivity probe's opening and allowing measurement to stabilize. Make the measurement and record the stabilized value (**Measurement Result)** in example table below or on the in the log book entry example provided. Discard low conductivity standard and rinse sensors and inside of cal cup with DI water, blot sensors and interior of cal cup dry and triple rinse with small amounts of the high conductivity standard (one in the 5000 µS/cm to **10,000 µS/cm** range). Record the **Measurement Result** of the second (high conductivity) standard after going through the same pre-measurement steps for the low conductivity standard (above).

Both the high range and low range calibration standards should be checked against the post-calibration check error limits specified in Table 1 (i.e. sensor measurements should agree within ± 3% or 5 µS/cm reported for the standards, whichever is greater) for this sensor to be acceptable for field use (in this case the same as the USGS criteria). These criteria should generally be achievable with good technique and sensors in good working condition given the high linearity of today's quality conductivity sensors. If the error limit is not achieved, it may indicate some troubleshooting and/or maintenance of the sensor is needed or the sensor needs replacement.

Also at this time, an additional test may be performed with DI water to check quality of the DI water itself which should measure ≤ 2 µS/cm. The **stability of the pH sensor in measuring low ionic strength water** may also be checked at this time, particularly if such waters are anticipated in field monitoring. The pH sensor measurement should be considered stable when no more than a 0.05 pH change occurs per 30 seconds in DI water. (Also note: pH sensors should not be left in calibration cups filled with DI water for extended lengths of time as this will deplete the internal buffer solution of the pH sensor/probe).

After confirming that the **Post-calibration Conductivity Cell Constant** of 5.06472 (in this case meets YSI criteria of 5.0 ±0.45, for other instruments see appropriate

manufacturers recommendations), complete Table 3 below or the instrument log book entry example provided at the end of this document.

Table 3. Example: Specific conductance error check results after 1-pt. calibration of MP instrument Specific Conductance sensor with **1413 µS/cm standard** (YSI series 6 instrument used in this example) and post-calibration measurement of 100 µS/cm and 10,000 µS/cm reference standards.

Check Standard/Soln.	MP Measurement Result	MP Measurement Error*
100 µS/cm standard	102 µS/cm	(2% or 2µS/cm is < the greater of 3% or 5 µS/cm so meets criteria)
10,000 µS/cm standard	10,068 µS/cm	(0.68% is < 3% so meets criteria)
DI Water (≤ 2 µS/cm)[1]	2 µS/cm	≤ 2 µS/cm (meets criteria)
pH (DI Water)	5.28 su (stable @ 3 min.)	Reached Stability w/lest than a 0.05 pH change/30 sec (so acceptable for low ionic strength pH measurements)

* Note – conductivity sensors should be highly linear and greater error is potentially introduced when using low specific conductance standards in a single point calibration (e.g. < 1000 µS/cm).
1 Various types/grades/sources of DI water are available and some may have specific conductance's that exceed the desired ≤ 2 µS/cm criteria. Distilled/deionized water from grocery stores generally does not meet the criteria. DI water from a dual ion exchange column procured for your office/lab, generally should meet these criteria as should DI water of good quality purchased through a vendor (see USGS discussion of DI and distilled water in Technical Memorandum 92.01 and ASTM for discussion of reagent grade water types (Type I, II, III and IV).

pH Error Check (10 – 20 min.): Perform a 2-point calibration with pH standards that bracket the anticipated water of environmental interest (e.g. using pH buffers of 4 & 7 or 7 & 10 std. units). Record the millivolt readings of the first two pH calibration standards. Make a pH measurement of the third calibration buffer (4 or 10) not used in the 2-point calibration and record pH and millivolt readings. Check the pH measurement of the third buffer (4 or 10) against the post-calibration check error limit specified for pH in Table 4 (± 0.3 std. units) and check to see that it falls within pH millivolt reading and Span/Slope Range specified by the manufacturer (YSI in this example). Normally, (± 0.2 std. units) should be achievable with sensors in good working order and good technique. Some trouble shooting or further check of the instrument/pH sensor is warranted if more than a 0.2 pH unit difference occurs and the operators manual should be consulted (e.g. perform a 3-pt. calibration, record and plot pH vs millivolt readings showing the two Nernst slopes between pH 7 and 10 and pH 4 and 7) and then rerun the error check procedure. If the sensor still does not meet error check criteria, troubleshoot and/or perform some maintenance on the pH electrode or its reference electrode per manufacturer's guidance. The pH probe or its reference should be serviced, refilled, or replaced if a post-calibration error check still yields an error check measurement greater than ± 0.3 su after a 3-point calibration or the pH sensor falls outside the millivolt measurement or span/slope specifications of the manufacturer (YSI in example provided below). Note that pH standards are highly buffered and provide optimal pH measurement (stability) conditions, unlike low ionic strength or poorly buffered environmental waters where pH measurements may drift slowly one way or the other by up to several hundredths of a pH standard unit per minute.

Table 4A. Example: Post-calibration (**pH 7 and pH 10 buffers)** error check of MP instrument pH sensor using a pH 4 standard reference (YSI series 6 or 600XL minisonde used in this example)**.**

pH 7 calib. buffer - millivolt reading -11.8 mv (0 +/- 50 mv; sensor meets YSI criteria)

pH 10 calib. buffer - millivolt reading -172.7 mv (-180 +/- 50 mv; sensor meets YSI criteria)

Compute Span/slope = $|pH\ 7_{mv} + pH\ 10_{mv}|$ = 184.5*

where 165 ≤ Span ≤ 180 is an acceptable range; Span/slope out of acceptable range so does not meet YSI criteria*

Check Standard	pH Measurement Result	pH Measurement Error	pH in Millivolts
pH 4 Buffer	**3.71**	**0.29** (within acceptable range)	**+ 164.6**
DI Water			

* perform troubleshooting/maintenance as both pH measurement error is near failure of acceptance criteria and calculated span/slope exceed YSI acceptance/rejection criteria

OR

Table 4B. Example: Post-calibration (**pH 7 and pH 4 buffers)** error check of MP instrument pH sensors using a pH 10 reference standard (YSI Series 6 or 600XL sonde used in this case)**.**

pH 7 calibration buffer - millivolt reading -10.8 mv (0 +/- 50 mv; meets YSI criteria)

pH 4 calibration buffer - millivolt reading + 168.4 mv (180 + / - 50 mv; meets YSI criteria)

Compute Span/slope = $|pH\ 7_{mv} + pH\ 4_{mv}|$ = +157.6

where 165 ≤ Span ≤ 180 is the acceptable range; Span/slope out of acceptable range so does not meet YSI criteria*

Check Standard	pH Measurement Result	pH Measurement Error	pH in Millivolts
pH 10 Buffer	**9.69**	**0.31**	**- 171.4**
DI Water	**7.10**	**N/A**	**N/A**

In this example, the pH sensor fails slightly both the NPS/USGS error acceptance criteria (±0.3 and ±0.2, respectively) and the acceptable span/slope criteria recommended by YSI. Therefore, a 3-pt. calibration (pH 4, 7 & 10 buffers) was run, the error checks were performed and pH millivolt response was checked against YSI span/slope a second time (Table 5 below).

Table 5. pH post-calibration (3-pt.) measurements

Buffer	Measured pH	Millivolt Response
pH 4	4.00	162.5
pH 7	7.11	-12.8
pH 10	9.95	-175.9

Span/slope 1 (pH 4 to pH 7) interval: **Span/slope = | 162.5 + (-12..8) |= 149.7 mv**

Span/slope 2 (pH 7 to pH 10) interval: **Span/slope = | (-12.8) + (-175.9) |= 188.7 mv**

While meeting the individual pH millivolt response criteria of YSI for the pH 4 (180 +/- 50 mv), pH 7 (0 +/- 50 mv), and pH 10 (0 +/- 50 mv), the span/slope determinations fall outside the YSI criteria and indicate the pH sensor should be serviced or replaced.

Also note that some vendor's standard pH sensors (e.g. Hydrolab and In-Situ) may not perform well in low ionic strength waters less than 50 uS/cm conductivity and require use of their low ionic strength pH sensors in those waters to reach a stable pH reading. This should be discussed with the instrument vendor to determine if a low ionic strength pH sensor should be purchased in place of or in addition too the standard pH sensor for the range of environmental monitoring conditions anticipated (based on the low ionic strength sensor's applicable range and the ionic strength of network waters). You may also check the pH of the DI water (~ 2 µS/cm) to see if stability in the pH measurement is achievable. It is rare for environmental waters to reach a specific conductance value below 10 µS with possible exception of those in which rainwater is a significant component.

Dissolved Oxygen (DO): Most MP instrument manufacturers recommend a water-saturated air calibration (easiest) for DO sensors that measure DO based on the amperometric method using Clark cell/membrane technology. After performing the single point water-saturated air calibration in units of percent saturation at 100%, a DO sensor measurement error check (that of a YSI Series 6 Rapid Pulse sensor in this example) may be performed in one of several ways. Three (3) error check alternative methods for DO are provided. **Method 1** (low DO solution check) is relatively quick and may be done to establish measurements are linear through low DO conditions but may not identify a low measurement bias because DO sensors do not measure below zero or

negative DO values. Thus Method 1 may be combined readily with **Methods 2 or 3** for the most comprehensive error checking.

A new optical or Luminescent DO technology is beginning to replace the amperometric (Clark cell) membrane technology. One or more of the QC methods below may also be used with this new technology until/unless better measurement confirmation methods are developed from additional experience with the new LDO sensor.

Method 1: Zero DO Reference Solution Check

1) Compare the calibrated MP instrument measured value with that of a prepared low DO reference solution. The low DO reference solution is prepared by adding ~1 gram of reagent grade sodium sulfite (crystals) and a few crystals (<0.001 milligram) of cobaltous chloride to 1 liter of DI water leaving minimum headspace. Multiple 1-liter poly bottles may be prepared in advance by just adding the reagents to the 1- liter bottles (dry) and later adding DI water (should these solutions be desired for later field checks). Invert the container several times to achieve dissolution of the solid/crystal reagents and allow it to stabilize for a few minutes. The sodium sulfite is an oxygen scavenger and the resulting DO measurement performed on this solution should be ≤ 0.2 mg/L. The stability of this low DO solution may not last for more than a day if headspace air is present so the container should be maintained with little or no headspace to maximize this stability period (can return the measured low DO solution back to the 1 liter storage container to keep headspace at minimum). It is usually best to prepare fresh daily for each low DO instrument check and smaller volume, 1-liter solutions with significant headspace should not be expected to retain their low DO character more than a day or two. For solutions stored with little or no headspace, we have observed that the low/near zero DO solutions may be maintained for a week or more. Always provide the date of the prepared zero DO solution on the bottle if retained for subsequent use.

Table 6. Example: Error check against low DO solution after calibration of MP DO sensor in water saturated air (YSI Series 6 Rapid Pulse DO sensor in this case).

Check Solution	MP Measurement Result (reject if meas. > 0.2 mg/L)	Measurement Criteria (≤ 0.2 mg/L)
Low DO Solution[1]	0.18 mg/L @ 5 min.	(meets criteria)
Office/lab Tap Water (compare w/ historic)	7.72 mg/L	(reasonable result based on previous testing)

[1]This is a one-sided (high) check for error (linearity) as a DO sensor will not read a negative DO (i.e. reads to zero but not below). If the DO sensor is reading low (systematic low measurement error) but still linear, this error check method will not discover this low bias.

DO charge 41.0 (found in YSI display by Report → DO Charge → Discrete Run @ 4 second rate). Is the DO charge 50 +/- 25? **Yes,** so DO charge is acceptable

Method 2:

2) Compare the calibrated MP instrument measured value to that DO value determined from allowing a water bath (sink), small bucket, or other water filled container (e.g. flow cell with bubbler set up) with an air pump & stone bubbler arrangement to reach equilibrium saturation at true barometric (station pressure) of your location. In-Situ Inc. also has a portable bubbler arrangement for a 2 point calibration of their new RDO sensor (w/MP9000 instrument) that will work for any vendors, smaller diameter minisonde. Use DI water or reasonably low ionic strength tap water of < 2000 µS/cm to avoid the need for salinity correction (use a salinity correction on water > 2000 uS/cm specific conductance per NFM table 6.2-7). With the stone bubbler (in a large lab sink), this water should reach DO saturation and equilibrium with surrounding atmospheric conditions in approximately 12 hours (overnight) or less. Do not submerge bubbler stone more than 6 inches in water bath to avoid potential for over-saturation. For the small portable bubblers (e.g. In-Situ's), oxygen saturation of the small volume of water used in calibration can be reached in 10 to 15 minutes. Saturation may be determined in the portable (flow cell) bubblers when there is no change/increase in DO but care should be taken to avoid supersaturation. At equilibrium, the MP measured DO value should compare closely to the saturation values provided by DO solubility tables based on uncorrected (to sea level) or barometric pressure (i.e. station pressure) for your location and the air/room temperature (no salinity correction should be necessary below 2000 µS/cm so go to values in NFM Table 6.2-6). For example, at a temperature of 20 degrees Celsius and 755 mm Hg uncorrected (to sea level)/station barometric pressure (i.e. includes affects of both the current weather-related pressure system and altitude at your location), DO at 100% saturation should be 9.0 mg/L (NFM Table 6.2-6). For salinities greater than 2000 µS/cm specific conductance, apply the salinity correction factor found in Table 6.2-7 of the NFM. These tables or individual entries may also be calculated online at **http://water.usgs.gov/software/dotables.html**.

Table 7. Example: Error check against equilibrium DO saturation (lab sink, flow cell bubbler and small pail) at Fort Collins office/ lab location (elev. ≈ 5,000 feet a.m.s.l.) based on uncorrected (to sea level) barometric pressure after MP instrument DO sensor calibration in water saturated air.

Pre-calibration DO charge 49.2 mv

Post-calibration DO charge 49.2 mv (found by Report → DO Charge → Discrete Run @ 4 second rate for YSI series 6 instruments)

Is the DO charge 50 +/- 25 (**yes**, acceptable; meets YSI criteria) @ Calibration: Temp. 20.11 ºC; BP 636 mm Hg; 100% saturation from DO tables is 7.52 mg/L

Check Solution	Result from Table*	MP Measurement Result	DO Meas. Error (or other assessment)
Saturated Solution for Oxygen Solubility (overnight lab sink water bath w/stone bubbler)	**7.52 mg/L** (@ 100 % sat.) (82.6%$_{YSI}$ sat.)	**7.72 mg/L** (83.9% saturation[1])	+0.20 mg/l or 2.4% (meets criteria)
Saturated Solution for Oxygen Solubility (flow cell filled with tap water and bubbler attachment run for 15 minutes)	**7.52 mg/L** (@ 100% sat.) (82.6 %$_{YSI}$ sat.)	**7.40 mg/L** (81.9% saturation[1])	-0.12 mg/L or 1.8% (meets criteria)
Saturated Solution for Oxygen Solubility (small pail filled with tap water and bubbler attachment run for 30 minutes)	TBD	TBD	TBD
Low DO Solution	N/A	0.18 mg/L	< 0.2 mg/L (meets criteria)
Labs Tap Water Sample (compare with historic)	N/A	8.06 mg/L	w/in historic meas. DO range of 6.5 to 8.5 mg/L (as function of temp. and BP)

* DO saturated water from USGS NFM Table 6.2-6 based on overnight (stabilized) water bath temperature (20.1 ºC) and atmospheric pressure. Pressure using uncorrected barometric or station pressure for office located at Fort Collins, CO. (in this example station pressure of 636 mm Hg was web accessed from local airport at http://www.crh.noaa.gov/den/awebshtml/webpres.shtml to confirm/recalibrate YSI built-in barometer of handheld display).

[1] **Note:** The YSI Series 6 instrument/display above reported an 82% to 84% saturation value which is relative to the 100% saturation value for fresh water at sea level at this temperature. This 100% saturation value at sea level (760 mm Hg @ 20 ºC) is 9.1 mg/L (note that Hach-Hydrolab and In-Situ instruments will report saturation (100%) based on the elevation of the location (i.e. not relative to a higher equilibrium saturation at the lower sea level elevation where pressure is greater and dissolved oxygen at saturation is higher). Therefore, while measurements should agree closely in mg/L (DO) among YSI, In-Situ and Hach-Hydrolab instruments, percent saturation values using YSI instruments will nearly always be less, unless measurements are made at sea level where DO in mg/L and % saturation should all agree among vendor instruments. This is also why the YSI instrument during calibration at 100% saturation (in air), actually reads a % saturation less than 100% when the calibration is performed above sea level (e.g. in this case at Fort Collins 7.54 mg/L / 9.3 mg/L ≈ 82.4% DO saturation (or relative to 100% saturation at sea level) for the YSI instrument even though 7.64 mg/L is 100% DO saturation at the altitude of WRD office in Fort Collins).

Method 3:

3) Compare the calibrated MP instrument DO measurement on a known saturated water sample to that value determined by Winkler titration (Idometric titration) or Azide Modification of Winkler Method (Hach Method 8215/8332 for 1 mg/L to > 10 mg/L DO range) using the recommended procedure of the test kit manufacturer. Alternatively, you may compare the MP DO sensor measurement to a second calibrated lab DO instrument of known, high accuracy, if available and accuracy has also been confirmed recently by Winkler titration (if a lab DO instrument is available, it may be best to confirm both instrument measurements at this time with a Winkler titration of a common sample). A Hach test kit (OX-DT) should work well for the Winkler titration but some practice in the lab is usually necessary to achieve good precision (personal communication, Richard Wagner, USGS). The Azide Modification of the Winkler Method (wet chemical) takes 15 to 25 minutes to perform depending on how routine the procedure has become for the analyst.

DO charge 48.2 (found by Report → DO Charge → Discrete Run @ 4 second rate)

Is the DO charge 50 +/- 25? **yes / no**

Check Solution	Result from Winkler Titration	MP DO Meas. Result	MP DO Meas. Error (or other assessment)
Tap Water Bath brought to saturation w/bubbler (BP 638 mm Hg & 19.1ºC); should be: 7.75 mg/l DO from Table 6.2-6 NFM	Avg. from 2 titrations: 7.41 mg/L	7.65 mg/l	(-0.15 mg/L rel. to NFM saturation table) (+0.24 mg/L rel. to Winkler titration)
Low DO Solution (fresh)	N/A		(meets ≤0.2 mg/L criteria?)
Lowered DO soln. (meas. of 2 month old low DO Solution w/headspace)	6.46 mg/L	6.60 mg/L	+0.14 mg/L difference so consistent with above results

Note, you may wish to combine the water bath for a temperature error check (overnight to achieve water bath stabilized to/near room temperature) with this DO error check method using an aquarium air pump and air stone bubbler in the same overnight water bath to achieve oxygen saturation. This DO error check could also be easily coupled with measurement of a freshly prepared low DO solution to confirm linearity of the DO sensor from saturation to zero DO.

Once these pre-mob sensor error checks become routine, you should arrive at the field monitoring site with a high confidence that the core parameter sensors will calibrate and function correctly, that the data collected will be representative of environmental conditions, and that minimal on site calibration time and little or no trouble shooting will be necessary.

Finally, calibrated and error checked MP instruments should be transported to the monitoring site in shock resistant cases and protected against temperature extremes, particularly freezing conditions. Chemical hand warmers placed in carrying case or other means to keep instrument above freezing may work well when transporting instruments under very cold conditions.

Recommended Equipment/Supplies List to Perform Error Checks.

In addition to the standard equipment, supplies and accessories for the MP Instrument suggested by the manufacturer, the following equipment (or some equivalent), chemicals, or standards is recommended for performing these error checks of core parameters in the lab/office.*

(Vendor product references are provided as examples only and do not reflect an endorsement of their product)

- NIST-Certified, NIST-Traceable or a thermometer "calibrated against NIST-traceable standards" (range -1 to 51 ºC or greater) with 0.1 ºC divisions (see Fisher Scientific Cat No. 15-000A @ $28.49 for less expensive thermometer w/0.1 ºC divisions calibrated against "NIST-Traceable standards"). Hach has a NIST-Certified Thermometer for $633.45 if you have additional temperature QC needs/concerns.
- Dissolved Oxygen Single Parameter Test Kit OX-DT (Hach Cat No. 2063100 at $207.40) for Winkler titrations includes a digital titrator. The test kit could be used as a backup method to MP instruments for field DO measurements also. Will perform ~ 50 tests with replacement chemicals/reagent set (Cat no. 22722-00 at $57.90)
- Conductivity Calibration standards of 100, 1413, and 10,000 µS/cm at minimum (5000 and 500 µS/cm or other standards are optional or purchase as needed – note bad batches of standards are rare but do happen; one possible less expensive source for water quality monitoring needs at http://www.inmtn.com/index.htm)
- pH buffer solutions of 4, 7, and 10 standard units.
- Sodium Sulfite, Anhydrous 454 g (Hach @ $20.20 – CAS No. 7757-83-7)
- Cobalt Chloride, ACS 113g (Hach @ $43.20 – CAS No. 7791-13-1)
- Aquarium Air Pump - 5 gal (Top Fin Brand avail. at Petsmart for $5.79)
- Air tubing ($2) and air stone ($1.50) to oxygenate water bath in sink or other measurement container
- Alternatively, an inexpensive complete portable bubbler system for lab or field calibration of the DO sensor (2 ½" or less diameter sondes) is available through In-Situ Inc. (flow cell and portable air pump "bubble box").

*Always review Material Safety Data Sheets (MSDS) that accompany chemical shipments for handling hazards before use of chemicals or calibration standards.

Pre-Mobilization MP Instrument (Calibration) Log Book Example Entry:

Date: 8-12-05 Sensor ID #'s Temp. _____

Time: 2:30 PM End Time 5:10 PM Cond./SC _____

Person: PEP pH _____

Instr. Mfr. Yellow Springs Instruments DO _____

Serial # _____ Model # _____

Display: Handheld _____ Batt. V._____(display)

Laptop _____ Batt. V. _____(sonde)

Barometric Pressure _____ mm Hg

Ref. Source _____

Error Check Solution: Sink Water Bath w/bubbler

Pre-Cal. Instrument Measurements	**Post-Cal. Instrument Measurements**
MP Temp. 18.67 ºC	MP Temp. 18.80 ºC
NIST Thermometer 1 18.72 °C	NIST Thermometer 1 ____ ºC
NIST Thermometer 2 18.80 °C	NIST Thermometer 2 ____ ºC
SC 114 µS/cm Cond. 100 µS/cm	SC 117 Cond. 103
pH 7.74 su	pH 7.92
DO 7.97 mg/L	DO
BP 636.8 mm Hg	BP 636.0

Specific Conductance Calibration (1413 µS/cm std.) & Error Check @ 100 and 10,000 µS/cm

Pre-cal measurement of 1413 standard was 1374 µS/cm; post-cal measurement was 1413 µS/cm

100 µS/cm reference std. 102 µS/cm; ERROR 2 µS/cm or 2%

10,000 µS/cm reference std. 10,066 µS/cm; ERROR 66 µS/cm or 0.66%

pH Calibration @ pH 7 & pH 10 su and Error Check @ pH 4

Pre-cal pH measurement of pH 7 std. was 6.96 su; measurement of pH 10 std. was 9.87 su

pH 7 standard -11.8 mv; pH 10 standard -172.7 mv: Span/Slope = +184.5

pH 4 reference std. 3.71 su ; +164.6 mv; ERROR -0.29 su

pH of DI water (consider stable when less than 0.05 su/30 sec. change)

Dissolved Oxygen Calibration in 100% Water Saturated Air

Pre-Cal DO Chg. 49.2 mv; Post-Cal DO Chg. 49.2 mv; (meets 50 +/- 25 mv criteria)

Temp. 20.11 ºC; BP 636.0 mm Hg; DO Table (100% sat.) 7.52 mg/L (82.6%$_{YSI}$)

A. Zero DO Ref. Solution (fresh) MP Meas. 0.18 mg/L; 2.1 % sat.

B. Sat. DO Water Bath (bubbler 12 hrs.) MP Meas. 7.72 mg/L 83.9 % sat.

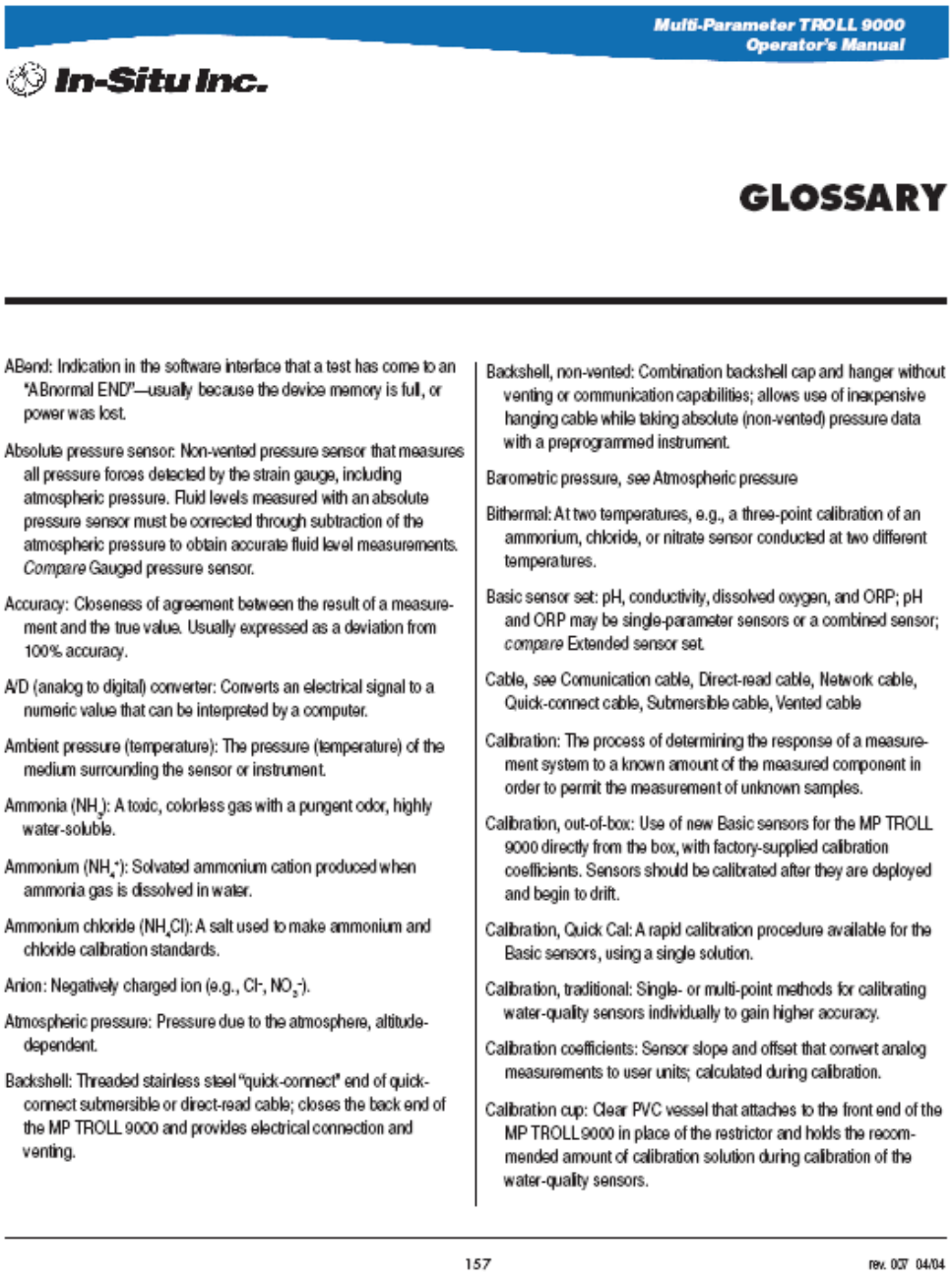
Flow Cell (bubbler 30 min.) MP Meas. 7.40 mg/L 81.9 % sat.

C. Sat. DO Water Bath from Mod. Winkler titration 7.35 mg/L 98 % sat. (calc. rel. to table)

Water Bath SC 117 µS/cm; Cond. 103 µS/cm; pH 7.92 su; End Time 5:10 PM

18. Appendix C: In-situ Inc. Troll 9000 Profiler Users Guide

Double-click picture to open pdf file in Adobe Acrobat Reader.



In-Situ Inc.

GLOSSARY

ABend: Indication in the software interface that a test has come to an "ABnormal END"—usually because the device memory is full, or power was lost.

Absolute pressure sensor: Non-vented pressure sensor that measures all pressure forces detected by the strain gauge, including atmospheric pressure. Fluid levels measured with an absolute pressure sensor must be corrected through subtraction of the atmospheric pressure to obtain accurate fluid level measurements. *Compare* Gauged pressure sensor.

Accuracy: Closeness of agreement between the result of a measurement and the true value. Usually expressed as a deviation from 100% accuracy.

A/D (analog to digital) converter: Converts an electrical signal to a numeric value that can be interpreted by a computer.

Ambient pressure (temperature): The pressure (temperature) of the medium surrounding the sensor or instrument.

Ammonia (NH_3): A toxic, colorless gas with a pungent odor, highly water-soluble.

Ammonium (NH_4^+): Solvated ammonium cation produced when ammonia gas is dissolved in water.

Ammonium chloride (NH_4Cl): A salt used to make ammonium and chloride calibration standards.

Anion: Negatively charged ion (e.g., Cl^-, NO_3^-).

Atmospheric pressure: Pressure due to the atmosphere, altitude-dependent.

Backshell: Threaded stainless steel "quick-connect" end of quick-connect submersible or direct-read cable; closes the back end of the MP TROLL 9000 and provides electrical connection and venting.

Backshell, non-vented: Combination backshell cap and hanger without venting or communication capabilities; allows use of inexpensive hanging cable while taking absolute (non-vented) pressure data with a preprogrammed instrument.

Barometric pressure, *see* Atmospheric pressure

Bithermal: At two temperatures, e.g., a three-point calibration of an ammonium, chloride, or nitrate sensor conducted at two different temperatures.

Basic sensor set: pH, conductivity, dissolved oxygen, and ORP; pH and ORP may be single-parameter sensors or a combined sensor; *compare* Extended sensor set.

Cable, *see* Comunication cable, Direct-read cable, Network cable, Quick-connect cable, Submersible cable, Vented cable

Calibration: The process of determining the response of a measurement system to a known amount of the measured component in order to permit the measurement of unknown samples.

Calibration, out-of-box: Use of new Basic sensors for the MP TROLL 9000 directly from the box, with factory-supplied calibration coefficients. Sensors should be calibrated after they are deployed and begin to drift.

Calibration, Quick Cal: A rapid calibration procedure available for the Basic sensors, using a single solution.

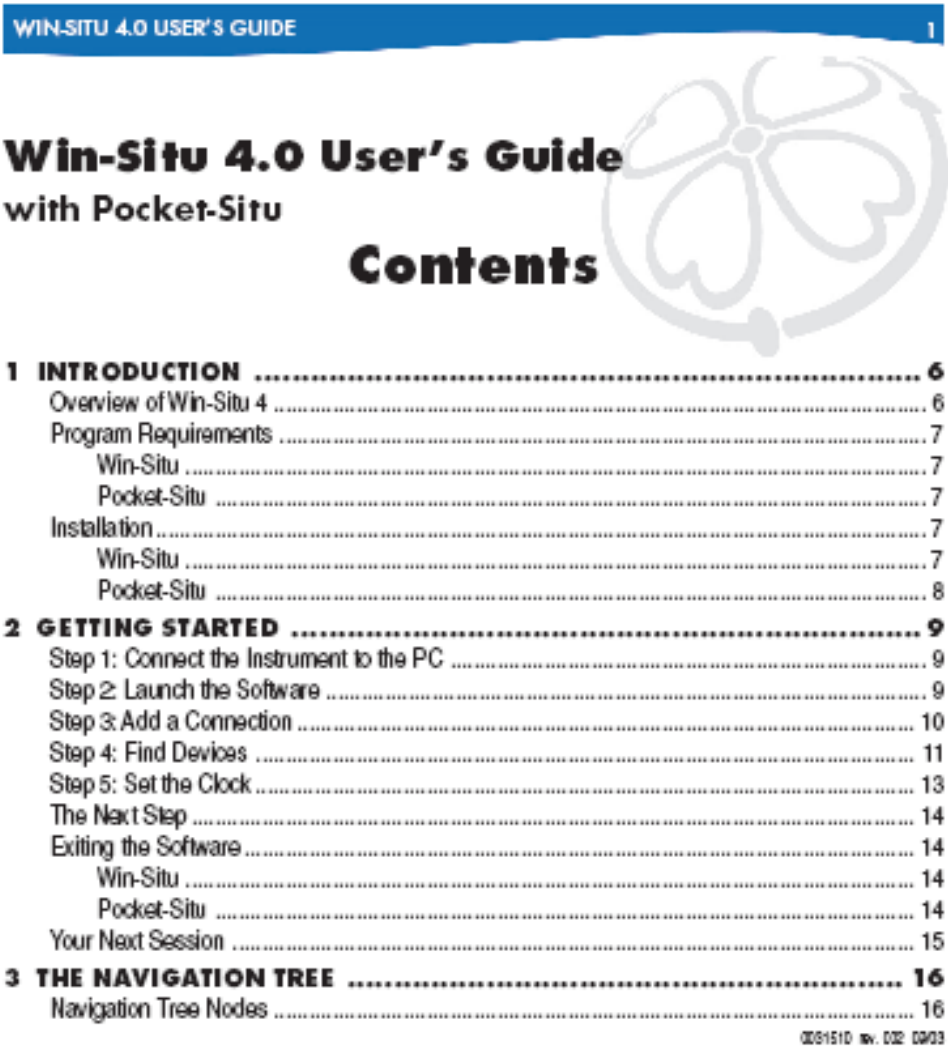
Calibration, traditional: Single- or multi-point methods for calibrating water-quality sensors individually to gain higher accuracy.

Calibration coefficients: Sensor slope and offset that convert analog measurements to user units; calculated during calibration.

Calibration cup: Clear PVC vessel that attaches to the front end of the MP TROLL 9000 in place of the restrictor and holds the recommended amount of calibration solution during calibration of the water-quality sensors.

19. Appendix D: In-situ Inc. WinSitu 4.0 Software Manual

Double-click picture to open pdf file in Adobe Acrobat Reader.



Win-Situ 4.0 User's Guide

with Pocket-Situ

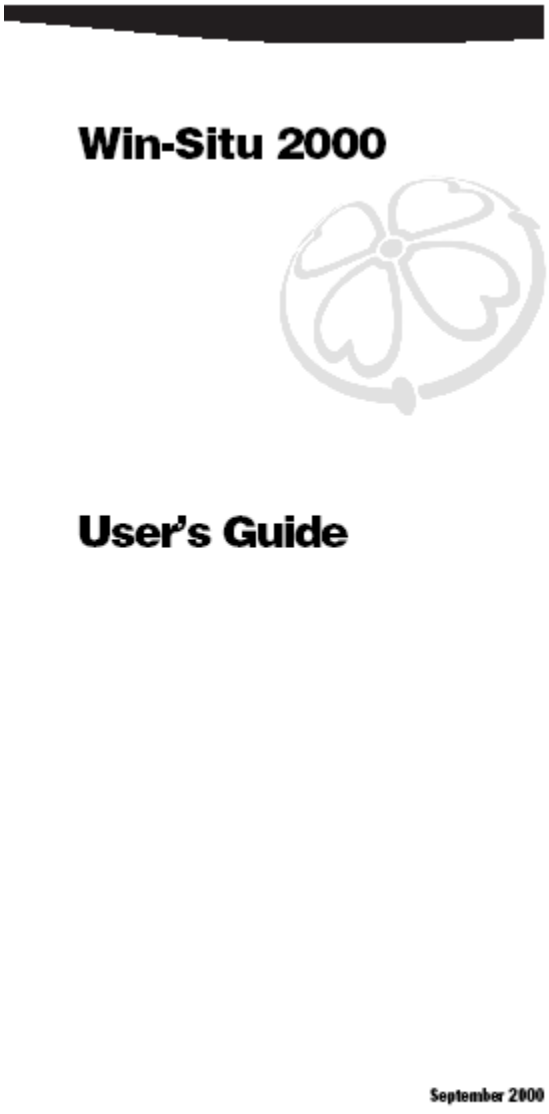
Contents

1 INTRODUCTION 6
Overview of Win-Situ 4 6
Program Requirements 7
Win-Situ 7
Pocket-Situ 7
Installation 7
Win-Situ 7
Pocket-Situ 8
2 GETTING STARTED 9
Step 1: Connect the Instrument to the PC 9
Step 2: Launch the Software 9
Step 3: Add a Connection 10
Step 4: Find Devices 11
Step 5: Set the Clock 13
The Next Step 14
Exiting the Software 14
Win-Situ 14
Pocket-Situ 14
Your Next Session 15
3 THE NAVIGATION TREE 16
Navigation Tree Nodes 16

0061510 rev. 002 09/03

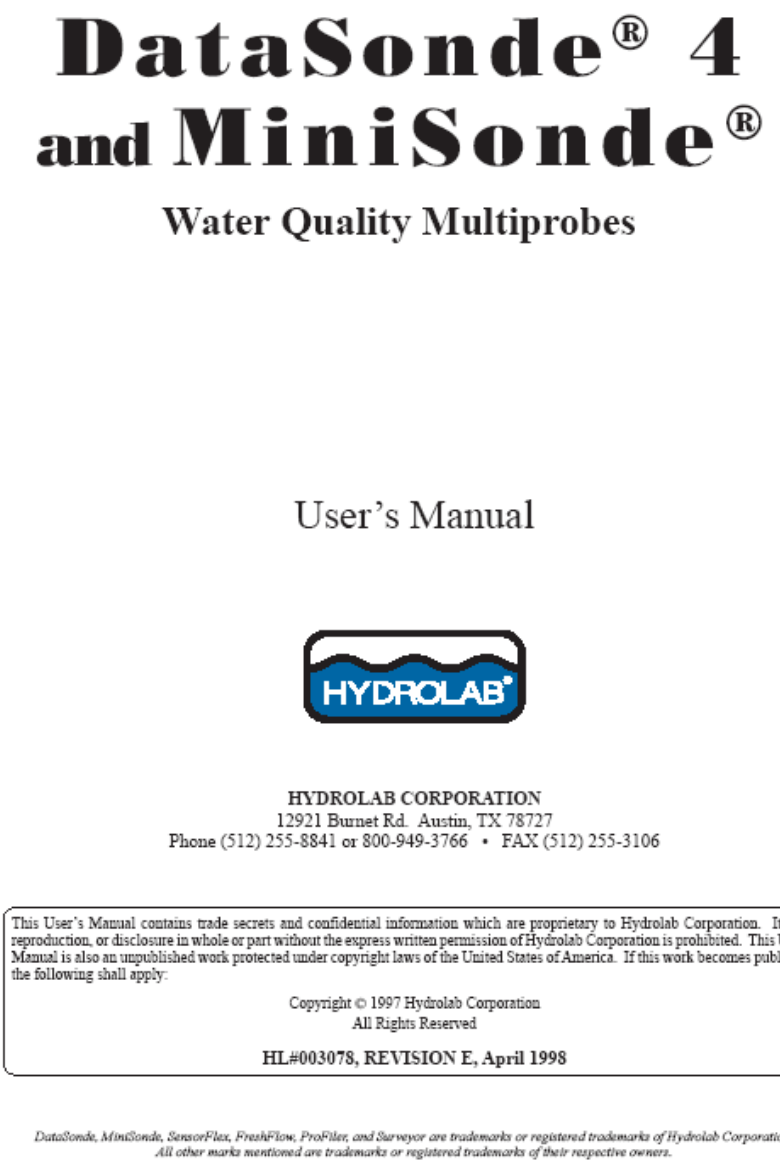
20. Appendix E: In-situ Inc. WinSitu 2000 Software Manual

Double-click picture to open pdf file in Adobe Acrobat Reader.



21. Appendix F: Hydrolab Users Manual

Double-click picture to open pdf file in Adobe Acrobat Reader.



DataSonde® 4
and MiniSonde®

Water Quality Multiprobes

User's Manual

HYDROLAB

HYDROLAB CORPORATION
12921 Burnet Rd. Austin, TX 78727
Phone (512) 255-8841 or 800-949-3766 • FAX (512) 255-3106

This User's Manual contains trade secrets and confidential information which are proprietary to Hydrolab Corporation. Its use, reproduction, or disclosure in whole or part without the express written permission of Hydrolab Corporation is prohibited. This User's Manual is also an unpublished work protected under copyright laws of the United States of America. If this work becomes published, the following shall apply:

Copyright © 1997 Hydrolab Corporation
All Rights Reserved

HL#003078, REVISION E, April 1998

DataSonde, MiniSonde, SensorFlex, FreshFlow, ProFiler, and Surveyor are trademarks or registered trademarks of Hydrolab Corporation. All other marks mentioned are trademarks or registered trademarks of their respective owners.

22. Appendix G: Hydrolab Display Unit Manual

Double-click picture to open pdf file in Adobe Acrobat Reader, then advance to page 2 to view file. For some reason the first page is left blank).

23. Appendix H DO calibration CO Units

From USGS National Field Manual

Radtke,D.B., A.F. White, J.V. Davis, and F.D. Wilde eds., April 1998, Dissolved Oxygen, Air calibration chamber in air. U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6., section 6.2, accessed _3/9/2006 from http://pubs.water.usgs.gov/twri9A6/

Procedure 3--Air-calibration chamber in air

This procedure is similar to the procedure for air-calibration chamber in water, except that the calibration chamber is in air rather than in water.

- The air-calibration-chamber-in-air procedure requires sensors in which the temperature-sensing thermometer is adjacent to the membrane.

- The DO instrument used must be able to automatically compensate for temperature changes. The YSI analog and digital DO instruments are automatically temperature compensating for changes in the solubility of oxygen in water and in the permeability of the sensor membrane.

1. Wet the inside of the calibration chamber with water--pour out the excess water (leave a few drops) and insert the sensor into the chamber (this ensures 100-percent humidity).
2. Allow 10 to 15 minutes for the DO sensor and the air inside the calibration chamber to equilibrate.
3. Read the ambient atmospheric pressure (from the pocket altimeter-barometer) to the nearest 1 mm of mercury.
4. Check mechanical zero. Adjust if necessary.
5. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes. Read the temperature to the nearest 0.5°C.
    - The temperature inside the chamber should approximate the water temperature, measured with a calibrated thermometer.
    - If the two temperatures do not match, allow additional time for the chamber and the water temperature to equilibrate.
    - If the temperature in the chamber still does not approximate the water temperature, the thermistor in the DO sensor might be malfunctioning. Measure the water temperature with a calibrated field thermometer.

6. Use the oxygen-solubility table 6.2-6 to determine the DO saturation at the measured temperature and atmospheric pressure. (Refer to section 6.2.4 and table 6.2-7 for salinity corrections.)
7. Select a proper scale:
    - Analog YSI instruments--0 to 10 or 0 to 20 mg/L.
    - Digital YSI instruments--0.1 or 0.01 mg/L.
8. Adjust the calibration control until the instrument reads the DO saturation value determined from the oxygen-solubility table. The instrument is now calibrated and ready for use.

Do not use the air-calibration-in-air procedure if the calibration chamber temperature differs from the temperature of the water to be measured.

24. Appendix I: pH calibration CO Units

From USGS National Field Manual

Wilde, F.D., Eurybiades Busenberg, and D.B. Radtke eds., January 2006, pH, calibration. U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6., section 6.4.2, accessed _3/9/2006 from http://pubs.water.usgs.gov/twri9A6/

6.4.2 CALIBRATION

Calibrate and check the operation of a pH instrument system at the field site. Two pH buffers are needed to properly calibrate the pH instrument system (pH 7 buffer and either the pH 4 or 10 buffer, depending on the anticipated sample pH). A third buffer can be used to check instrument system performance over a larger range. The pH of the buffer solution is temperature dependent: pH 10 buffers change more per unit change in temperature than do pH 4 buffers. The temperature of buffer solutions must be known, and temperature-correction factors must be applied before calibration adjustments are made. Calibration and operating procedures differ with instrument systems—check the manufacturer's instructions.

Meters with microprocessors have reliable autocalibration functions that will automatically compensate for buffer temperatures and indicate Nernst slope. For such meters, follow the manufacturer's calibration instructions precisely—do not take shortcuts.

 Check the records of electrode performance before each calibration and field trip (see 6.4.1). Electrode response is optimal between approximately 98 percent and 99.5 percent. A slope of 94 percent indicates possible electrode deterioration.

At 90 percent slope, the electrode cannot be used.

Chapter A6. Field Measurements pH, Version 1.3 (1/2006)

pH — 13

 Calibrate or check the temperature sensor calibration at least annually, and tag the sensor with the date of in-house certification. Do not use the automatic temperature compensating function of a pH meter if it has not been certified within the past 12 months.

 Record calibration in the instrument log book and on field forms at the time of instrument calibration.

To calibrate the pH instrument system, follow the 10 steps listed below:

1. Temperature equilibration of equipment (this is recommended, even if using an automatic compensating meter).

a. Bring the pH buffers, thermometer (if necessary), container, and electrode to the temperature of the sample.

b. Allow 15 to 30 minutes for the buffers to adjust to the sample temperature. When making temperature corrections, use the correction factors provided by the buffer manufacturer (temperature coefficients can vary with buffer manufacturer).

● To equilibrate to stream temperature, place the buffer bottles in a minnow bucket or mesh bag and suspend them in the stream.

● To equilibrate to ground-water temperature, place the buffer bottles in a mesh bag and suspend them in a bucket or other large container (an ice chest works well) overflowing with water being pumped from the well.

2. Inspect the pH electrode.

a. Check for damage to the electrode bulb, body, or cables.

b. Rinse any precipitate off of the electrode with deionized water (the measurement can be affected if precipitate falls into the buffer or sample).

c. Slide the protective sleeve up or down to uncover the filling hole.

d. Gently shake or tap the electrode to dislodge and remove air bubbles trapped in the sensing tip of the electrode and to remove excess deionized water. Do not wipe the electrode.

3. Calibration rinse.

a. Rinse (with pH 7 buffer) the electrode, thermometer or automatic temperature compensating (ATC) sensor, and a container large enough to hold the sensors and buffer. Discard the used pH buffer into a waste container.

b. Pour fresh pH 7 buffer into the buffer-rinsed container that holds the electrode and thermometer. Allow the instruments to equilibrate for 1 minute (if necessary), then discard the buffer into a waste container.

4. Calibration.

a. Pour fresh pH 7 buffer into the container that holds the electrode and thermometer or ATC sensor.

● The bulb of the pH electrode must not touch the bottom or side of the container.

● Add enough pH buffer to cover the reference junction.

b. Swirl the sample gently or stir carefully with the electrode. If using a magnetic stirrer, stir slowly enough so that a vortex is not created. Place a thin piece of insulating material (styrofoam or cardboard) between the magnetic stirrer and beaker to prevent transfer of heat to the buffer solution.

c. Measure the temperature of the buffer solution; remove the thermometer (it is not necessary to remove the ATC sensor).

d. Determine the theoretical pH of the buffer from the temperature-correction tables.

e. Note and record the pH temperature readings. Adjust the meter reading to the pH value using the "standardize" function on the meter (usually a knob or pressure pad). Record the adjusted pH value for the 7.0 buffer and associated millivolt reading.

f. Remove the electrode and ATC sensor (some instruments require that the meter be switched to the standby or off position before removing the electrode from the solution).

● Repeat the calibration steps using fresh portions of reference buffer solution until two successive readings are obtained at the adjusted pH value for pH 7 buffer without further adjustment to the system.

● Discard the used pH 7 buffer into a waste container.

Chapter A6. Field Measurements pH, Version 1.3 (1/2006)

pH — 15

5. Slope adjustment rinse.

a. Rinse the electrode and thermometer or ATC sensor thoroughly with deionized water.

b. Rinse a clean container, electrode, and thermometer with a second buffer (usually pH 4 or 10) that brackets the expected pH value of the sample; discard the used buffer into a waste container.

c. Pour the second buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.

6. Slope adjustment. **For modern meters, this step is automated— skip to step 7.**

a. Pour a fresh portion of the second pH buffer into a container holding the electrode and thermometer or ATC sensor.

b. Stir slowly (no vortex) or swirl manually. Follow the directions in 4b, above.

c. Measure the temperature and pH of the buffer solution and check the pH value of the buffer on temperature coefficient tables. Record the pH and temperature readings.

d. Adjust the slope to the value of the second pH buffer at known temperature. (Some meters have separate slope-adjustment knobs, pressure pads, or other devices, whereas others have to be adjusted by use of a temperature knob.) Record the adjusted pH value and associated millivolt reading.

e. Discard the used buffer into a waste container.

f. Repeat steps 6(a) through 6(e) using successive portions of the buffer solution until two successive readings are obtained without further adjustment.

7. Rinse the electrode and thermometer or ATC sensor thoroughly with deionized water.

8. If using a noncompensating or nonautomated meter, repeat the calibration rinse (step 3) and calibration procedures [steps 4(a) through 4(d)] to ensure that the slope adjustments did not affect the calibration adjustment.

● This step is a check only; no adjustment should be needed, but the result should be recorded. If adjustment is needed, repeat the entire calibration procedure.

● If adjustment is still needed, a systematic problem is likely (see 6.4.4). Inspect the instrument system, clean the electrode or add filling solution, or use a spare electrode or meter.

9. Calibration check rinse.

a. Rinse the electrode and thermometer or ATC sensor with deionized water.

b. Rinse another clean container, electrode, and thermometer with a third buffer (pH 4 or 10) and discard the used buffer into a waste container.

c. Pour the third buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.

10. Calibration range check.

a. Pour a fresh portion of third pH buffer into a container holding the electrode and thermometer or ATC sensor.

b. Stir without forming a vortex or swirl slowly (see step 4b).

c. Measure the temperature of the buffer solution (remove the liquid-filled thermometer and check the temperature-adjusted pH value), if necessary for the meter being used.

d. The pH instrument system should read the value of the third buffer at a known temperature within ±0.1 pH units.

● Meters reading to three or more places to the right of the decimal may not provide better accuracy than ±0.05 units, and their accuracy must be verified.

● If it checks, the instrument system is calibrated over a range of pH 4 to 10 and is ready for ANC or alkalinity titrations as well as pH measurement.

● If the instrument system does not check over the entire range, recalibrate before measuring the sample pH. Recalibrate before an alkalinity/ANC titration if the sample has a pH greater than 7.0.

e. Discard the used buffer into a waste container.

f. Rinse the electrode and thermometer (or ATC sensor) with deionized water.

Calibration for low-conductivity water

Proper calibration of pH instrument systems with standard buffers does not guarantee accurate pH measurement in water with conductivity less than 100 µS/cm. The following recommendations for pH measurement in low-conductivity water are taken from Busenberg and Plummer (1987).

1. After calibration with pH 4, 7, and 10 buffers, check electrode performance daily in appropriate sulfuric acid standard solution with conductivity less than 20 µS/cm. (For solution preparation and handling, refer to Busenberg and Plummer, 1987).

● Before using the sulfuric acid standard solution, check for contamination by measuring conductivity.

2. Check electrode performance with deionized water saturated with an analyzed nitrogen-carbon dioxide gas mixture having a carbon dioxide mole fraction of less than 0.5 percent.

● Addition of KCl is not recommended because of the potential for contamination and other complications.

3. Rinse the electrode at least three times, preferably with a portion of the sample to be measured.

4. Calibrate and measure pH in quiescent (unstirred) solutions after the sample has been homogenized by stirring.

5. Check the electrode performance (slope) before using the percent Nernst slope and (or) millivolt readings at pH 7 and pH 4. Keep a record of the electrode slope and millivolt readings—they can signal electrode deterioration.

25. Appendix J: Conductivity calibration CO Units

From USGS National Field Manual

Radtke, D.B, J.V. Davis, and F.D. Wilde, eds., August 2005, Specific Electrical Conductance. U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6., section 6.3.2, accessed _3/9/2006 from http://pubs.water.usgs.gov/twri9A6/

Conductivity systems must be calibrated before every water-quality field trip and again at each site before samples are measured. Calibration readings are recorded in the instrument log book and on field forms at the time the instrument is calibrated. Remember, the temperature sensor on the conductivity sensor must be District certified within the past 4 months.

Calibration and operating procedures differ, depending on instrument and sensor type.

CALIBRATION 6.3.2

- Some conductivity sensors may need to be soaked overnight in deionized water before use—Check the manufacturer's instructions.
- Some analog instruments require an initial mechanical zero adjustment of the indicator needle.
- For a cup-type cell, calibration and measurement procedures described for the dip-type cell apply; the only difference is that standards are poured directly into the cup-type cell.
- When using a dip-type cell, do not let the cell rest on the bottom or sides of the measuring container.

Calibrate at your field site—bring standards to sample temperature.

Conductivity systems normally are calibrated with at least two standards. Calibrate sensors against a standard that approximates sample conductivity and use the second standard as a calibration check. The general procedures described in steps 1 through 15 below apply to most instruments used for field measurements—check the instrument manual for specific instructions.

1. Inspect the instrument and the conductivity sensor for damage. Check the battery voltage. Make sure that all cables are clean and connected properly.
2. Turn the instrument on and allow sufficient time for electronic stabilization.

3.Select the correct instrument calibration scale for expected conductivity.

4.Select the sensor type and the cell constant that will most accurately measure expected conductivity.

5.Select two conductivity standards that will bracket the expected sample conductivity. Verify that the date on the standards has not expired.

6.Equilibrate the standards and the conductivity sensor to the temperature of the sample.

- Put bottles of standards in a minnow bucket, cooler, or large water bath that is being filled with ambient water.
- Allow 15 to 30 minutes for thermal equilibration. Do not allow water to dilute the standard.

7.Rinse the conductivity sensor, the thermometer (liquid-in-glass or thermistor), and a container large enough to hold the dip-type sensor and the thermometer.

- **First**, rinse the sensor, the thermometer, and the container three times with deionized water.
- **Next**, rinse the sensor, the thermometer, and the container three times with the standard to be used.

8.Put the sensor and the thermometer into the rinsed container and pour in fresh calibration standard.

9.Measure water temperature. **Accurate conductivity measurements depend on accurate temperature measurements or accurate temperature compensation.**

- If the sensor contains a calibrated thermistor, use this thermistor to measure water temperature.
- If using a manual instrument without a temperature display or temperature compensation, adjust the instrument to the temperature of the standard using a calibrated liquid-in-glass or a thermistor thermometer.

10.Agitate a submersible-type conductivity sensor up and down under the solution surface to expel air trapped in the sensor. Read the instrument display. Agitate the sensor up and down under the solution surface again, and read the display. Repeat the procedure until consecutive readings are the same.

11.Record the instrument reading and adjust the instrument to the known standard value.

- For nontemperature-compensating conductivity instruments, apply a temperature-correction factor to convert the instrument reading to conductivity at 25□C.
- The correction factor depends to some degree on the specific instrument used—use the temperature-correction factor recommended by the manufacturer. If this is not available, use correction factors from table 6.3–3.
- If an instrument cannot be adjusted to a known calibration standard value, develop a calibration curve. After temperature compensation, if the percentage difference from the standard exceeds 5 percent, refer to the troubleshooting guide (section 6.3.4).

12.Record in the instrument log book and on field forms:

- The temperature of the standard solution.
- The known and the measured conductivity of the standard solution (including ± variation).
- The temperature-correction factor (if necessary).

13.Discard the used standard into a waste container. Thoroughly rinse the sensor, thermometer, and container with deionized water.

14.Repeat steps 7 through 13 with the second conductivity standard.

- The purpose for measuring a second standard is to check instrument calibration over the range of the two standards.
- The difference from the standard value should not exceed 5 percent.
- If the difference is greater than 5 percent, repeat the entire calibration procedure. If the second reading still does not come within 5 percent of standard value, refer to the troubleshooting guide in section 6.3.4 or calibrate a backup instrument.
- **Switching instrument calibration scales could require re-calibration**.

15.Record in the instrument log book and on field forms the calibration data for the second standard.

Do not use expired standards. Never reuse standards. Specific Electrical Conductance, Version 1.2 (8/2005) U.S. Geological Survey TWRI Book 9 **10—SC**

Table 6.3–3. Correction factors for converting non-temperature-compensated values to conductivity at 25 degrees Celsius, based on 1,000 microsiemens potassium chloride solution

[Use of potassium-based constants on non-potassium-based waters generally does not introduce significant errors for general purpose instruments used to measure conductivity]

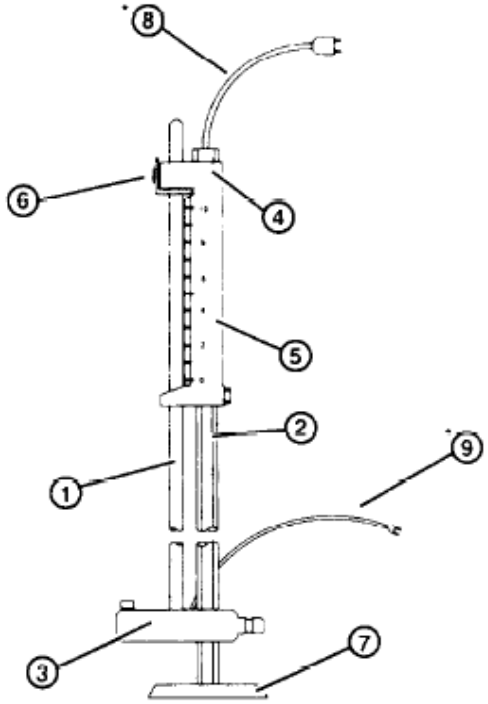
Temperature (degrees Celsius)	Correction factor	Temperature (degrees Celsius)	Correction factor	Temperature (degrees Celsius)	Correction factor
0.5	1.87	10.5	1.39	20.5	1.09
1.0	1.84	11.0	1.37	21.0	1.08
1.5	1.81	11.5	1.35	21.5	1.07
2.0	1.78	12.0	1.33	22.0	1.06
2.5	1.76	12.5	1.32	22.5	1.05
3.0	1.73	13.0	1.30	23.0	1.04
3.5	1.70	13.5	1.28	23.5	1.03
4.0	1.68	14.0	1.27	24.0	1.02
4.5	1.66	14.5	1.26	24.5	1.01
5.0	1.63	15.0	1.24	25.0	1.00
5.5	1.60	15.5	1.22	25.5	0.99
6.0	1.58	16.0	1.21	26.0	0.98
6.5	1.56	16.5	1.19	26.5	0.97
7.0	1.54	17.0	1.18	27.0	0.96
7.5	1.52	17.5	1.16	27.5	0.95
8.0	1.49	18.0	1.15	28.0	0.94
8.5	1.47	18.5	1.14	28.5	0.93
9.0	1.45	19.0	1.13	29.0	0.92
9.5	1.43	19.5	1.12	29.5	0.91
10.0	1.41	20.0	1.11	30.0	0.90

To extend the temperature range shown in table 6.3–3, consult the manufacturer's guidelines. If these are unavailable, use the following equation:

where,
C*25*= corrected conductivity value adjusted to 25°C;
C*m* = actual conductivity measured before correction; and
t*m* = water temperature at time of C*m* measurement.

26. Appendix K: Top Setting Wading Rod

Use of Top Setting Wading Rods



(1) The 3/8" diameter Aluminum rod.
(2) The 1/2" Hexagon Stainless Steel rod.
(3) The Sliding Support.
(4) The Handle.
(5) The Vernier.
(6) The Trigger.
(7) The Base.
(8) The Wire Assy. - Handle.*
(9) The Wire Assy. - Meter.*

The above numbers are used for purposes of description only, and do not reflect part numbers for ordering purposes.

*Wading Rods with an E suffix are designed for use with Direct Reading Current Meters and do not have Items (8) and (9).

SET-UP AND USE

The base(7) is installed by threading it and its accompanying lock washer on to the threaded lower end of the Hexagon Rod(2). The Current Meter is then mounted on to the Sliding Support(3) and is held in place by tightening the set screw, (the type of screw whether a thumb screw or a slotted screw will vary depending on the type of Meter you are using). If you are using a Price Type Current Meter, the Wire Assy.-Meter(9) is attached to the binding post of the Meter and the Wire Assy.-Handle(8) is attached to the headset connector. If you are using a Direct Reading type Current Meter, the wiring goes directly to the Current Meter and no other connections need be made. In the latter case you can use a Wading Rod with the Suffix E which does not have the #(8) or #(9) Wire Assys. The Current Meter and Wading Rod are now ready for use.

As the name implies, Top Setting Wading Rods are designed for use in waters shallow and safe enough to be waded. Once at a point where readings are to be taken, the Wading Rod with the Meter mounted, is placed vertically into the water until the Base(7) rests on the bottom. The water depth is now read on the Hexagon Rod(2). An example is given on back of card, for both English and Metric Rods.

EXAMPLES

English

Models: 1246, 1247, 1248, 1249, 1266-E, 1267-E.

The Hexagon Rod(2) is marked in graduations of .1 ft with a single line every .1 ft, a double line every .5 ft and a triple line every 1 ft. The Wading Rod is placed vertically into the water and the depth is read on the Hexagon Rod(2). In this example the water depth is read as 2.2 ft. To position the Current Meter at .6 of the 2.2 ft depth, squeeze the Trigger(6) and slide the Aluminum Rod(1) until its graduation marked 2(corresponding to the water depth in feet) is in line with the 2 on the Vernier(5) (corresponding to the number of .1 ft in the water depth). This will position the Current Meter at .6 of the water depth .4 up from the bottom. This procedure applies to any water depth. The number on the Aluminum Rod(1) corresponding to the water depth in whole feet is brought even with the Vernier(5) graduation corresponding to the number of .1 ft in the water depth. This method complies with the .6 method of taking velocity measurements. By doubling the water depth and using the resulting numbers to position the Aluminum Rod(1) the Meter is set at .2 of the water depth .8 up from the bottom. Conversely, by positioning the Aluminum Rod(1) to the numbers corresponding to 1/2 the water depth, the Meter is placed at .8 of the water depth .2 up from the bottom. The latter two positions correspond to the .2 .8 method for taking velocity measurements.

Metric

Models: 1256, 1257, 1276-E, 1277-E.

The Hex Rod (2) is marked in .2 decimeter graduations with a single line every .2 dm, a double line every dm and a triple line every 5 dm (1/2 meter). In this example the water depth is read as 5.2dm on the Hex Rod (2). To position the Meter at .6 of the 5.2dm depth, squeeze the Trigger (6) and slide the Aluminum Rod (1) up until its graduation marked 5 (corresponding to the number of whole decimeters in the water depth) is in line with the 2 on the Vernier (5) (corresponding to the number of .1 dm in the water depth). This will position the Meter at .6 of the water depth, .4 up from the bottom. This procedure applies to any water depth. The number on the Aluminum Rod (1) corresponding to the water depth in whole decimeters in the water depth is brought to the number on the Vernier (5) corresponding to the number of .1 dm in the depth. This method complies with the .6 method of taking velocity measurements. By doubling the water depth and using the resulting numbers the Meter can be set at .2 of the water depth, .8 up from the bottom. Conversely, by using the numbers corresponding to 1/2 the water depth the Meter can be set at .8 of the water depth .2 up from the bottom. The latter two positions correspond to the .2 .8 depth method for taking velocity measurements.

SCIENTIFIC INSTRUMENTS, INC.
SURVEYING & HYDROLOGICAL INST
518 WEST CHERRY STREET
MILWAUKEE, WISCONSIN 53212
PHONE (414) 263-1600
FAX NUMBER (414) 263-5506

27. Appendix L: AquaCalc Pro Users Manual

Getting Started

We encourage you to contact JBS Instruments with questions and comments.

All of your comments about the functionality and ease of use of the AquaCalc Pro are very important to us, no matter whether they are good or bad, or seem significant or trivial, are very important to us. Your comments will help us make the AquaCalc Pro a better product and easier to use.

Important Terms

There are a few basic terms that are important to working with the AquaCalc Pro. Some of these terms have changed from their use in the AquaCalc 5000.

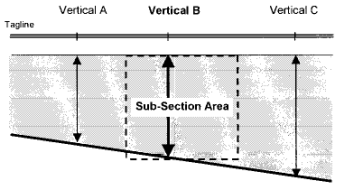
Section - Measurement section from beginning edge to ending edge. Formerly known as a Transect.

Vertical -- A location in the section identified by a tag-line distance where depth and velocity measurements are performed.

Observation (often abbreviated as Observe or Obs.) -- An individual velocity measurement performed in a Vertical.

Observation Depth -- A location in the vertical as measured from the surface, .2, .6, or .8.

Sub-Section -- Also known as a "panel". The area of a stream related to a vertical, extending halfway to each of the adjacent verticals and from surface to bottom as shown in the following figure.



GID - Gage ID / Station or Section Identifier

UID - User ID / Users name or identifier

Soft-Key - The three keys that are located directly below the display. The function of these keys are redefined by the AquaCalc firmware as needed.

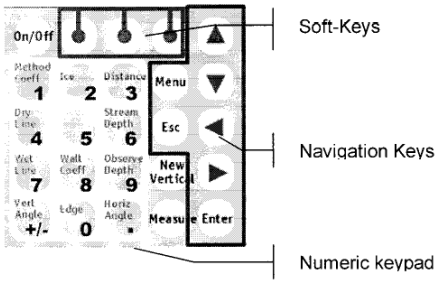
Firmware - The program that is loaded into the flash memory of the AquaCalc.

Warning - Opening the case will void warranty, there are no user serviceable components in the Pro.

Warranty - limited three-year parts and labor.

Keypad Descriptions

The keypad is a five key by five key membrane switch.



On/ Off

Press the On/Off key until the beep changes tones. The power down screen will then appear. Pressing any key will resume the Pro to the Main Menu screen. To save batteries, the AquaCalc will power down at the default setting of 10 minutes. This setting can be changed in the System Preferences.

Soft-Keys



The functions of the three keys centered directly under the display are changed depending upon the screen. The function of each key is displayed on the bottom rows of the display.

Navigation Keys



Up and Down Arrow Keys – While in the Measure screen, these keys are used to change the Observation Depth. When in the Main Menu they are used to adjust the display contrast. Also used to edit alphanumeric entries.

Left and Right Arrow keys – While in the Measure screen the Left and Right Arrow keys are used to move between verticals.

Enter – The Enter key completes an entry, and exits out of a menu.

Menu – This allows the user to access the Main Menu or a menu associated with the current screen.

Esc – Escape key allows the user to cancel or back out of a data entry screen without making changes.

New Vertical – While in the Measure screen, pressing this key will create a new vertical and request the location of that vertical requiring the user to enter the distance.

Measure – Starts the stopwatch and starts counting the clicks. Pressing Measure from the Main Menu screen

The 10 numeric keys are used to enter parameters such as Distance and Depth. The decimal key and plus minus key are used in conjunction with the Numeric keys.

1/Method Coeff - User enters a numeric value of 1 or allows the user to edit the Method Coeff

2/Ice - User enters a numeric value of 2 or allows the user to enter an Ice Draft

3/Distance - User enters a numeric value of 3 or allows the user to edit the Distance

4/Dry Line - User enters a numeric value of 4 or Dry Line not yet implemented

5 - User enters a numeric value of five. (This key can be used to test the AquaCalc by filling up a complete section with test data. Open a new section and press and hold the 5 key)

6/Stream Depth - User enters a numeric value of 6 or allows the user to enter the Depth

7/Wet Line - User enters a numeric value of 7 or Wet Line not yet implemented

8/Wall Coeff - User enters a numeric value of 8 or allows the user to edit the wall coeff

9/Observe Depth - User enters a numeric value of 9 or Observe Depth not yet implemented (nonstandard)

0/Edge - User enters a numeric value of 0 or sets a vertical to edge of water

+/-/Vertical Angle - User enters a numeric value of +/- or Vertical Angle not yet implemented

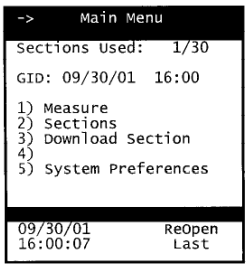
./Horizontal Angle - User enters a decimal point or allows the user to edit the horizontal angle coefficient

Start It Up

Turn on the AquaCalc by pressing the On/Off key. (You may turn off the AquaCalc by pressing and *holding* the On/Off key.) As the AquaCalc starts it will briefly display an opening screen and then will display the Main Menu:

Main Menu

The third line of the Main Menu shows the Section Identifier (GID) for the current section. When you turn your AquaCalc on it creates a new Section, and automatically names the new section with the date and time until changed by the user.



The Main Menu contains several items that can be selected by pressing the number on the keypad that corresponds to the number at the left of the menu item. For example, to go to the Measure screen, press the 1 key while in the Main Menu (You may also press the Measure key to go to the Measure screen). The Main Menu Items are as follows:

While in the Main Menu, the contrast can be adjusted using the Up and Down Arrow keys.

1) **Measure** - This takes you to the Measure screen where a majority of the tasks are performed. Pressing the **Measure** key will also take you to the Measure Screen
2) **Sections** - This is where a section can be Opened, Deleted or a New one made. The sections screen is made up of three screens with 10 sections per screen. These different screens are accessed form the soft keys.
3) **Download Section**- Selecting this option will send the current section information and observations to your computer.
4) **Reserved for adding other types of nonstandard meters in the future.**

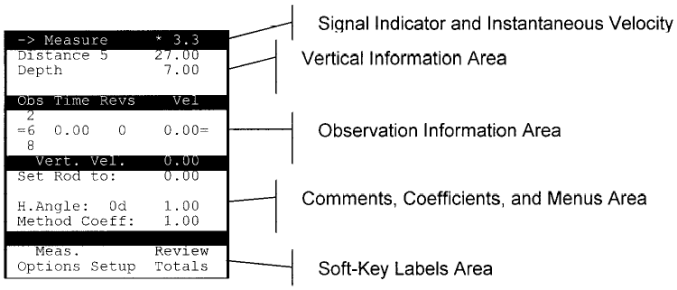
5) **System Preferences** – Allows the user to set time and date, power control, Baud rate, turn on and off beep tones for the keypad and meter, adjust the contrast of the display, and adjust the units (SAE/English or Metric). The **Date** and **Time** soft keys will also take you to System Preferences.

   When displaying the System Preferences three new soft keys appear. These keys are used to set or change the Owner ID, Reset the entire System to default settings and erasing all data, and the About screen which provides information about the AquaCalc Pro such as User ID, Serial Number, and Firmware version.

Soft-Keys – Date and Time and ReOpen Last, by selecting the Date and Time soft-key you will be placed in the Preferences menu where the Date and Time can be edited. The ReOpen Last soft-key will open the last section you were in when the Pro powered down.

Important Note: If you turn off the AquaCalc, or if the AquaCalc turns itself off automatically, you will need to use the "ReOpen Last" Soft-Key on the Main Menu to load and return to your last section.

The Measure Screen



The most common screen or display the user will see when using the AquaCalc is the Measure screen.

When connected to a spinning current meter, the top line will show an instantaneous velocity along with a flashing asterisk (*) that corresponds to the current meter "clicks".

The Vertical Velocity (Vert. Vel.) line shows the calculated mean velocity for the vertical.

In the center of the screen are three lines that identify the 2, 6, and 8 observation locations.

Navigating in the Measure Screen

Experiment with the keys in the Measure screen, each is designed to provide a special function. Pay special attention to the white navigation keys that surround the numeric keypad.

Changing Verticals

Use the Left and Right Arrow keys to move between verticals in a section. The right arrow moves you to the next vertical and the left arrow to the previous. A warning message will appear when you try to move past the last vertical.

Changing the Observation Depth
Use the Up and Down Arrows to move between the Observation Depths.

Returning to the Main Menu
Use the Menu key to return to the Main Menu from within the Measure screen.

Measure Screen Soft keys

Measure Options
Pressing the Measure Options Soft-Key opens up a menu that allows access to functions that apply to the current Vertical and the Observations in that Vertical

These include:

1) **Velocity: Meas./Est.** – Defines how the velocity at the observation will be obtained either measured or estimated.
2) **V. Depth: Meas./Est.** – Defines how the stream depth at the vertical will be obtained either measured or estimated.
3) **Flow Dir.: Normal/Reverse** – Defines the direction of flow in the vertical.
4) **Reserved for future use.**
5) **Reserved for future use.**
6) **Erase Obs Data** – Allows the user to erase the selected observation.
7) **Delete Vertical** - Allows the user to delete the current vertical.

Setup
Pressing the Setup Soft-Key opens up a menu that allows access to Section Setup and Vertical Setup.

Section Setup Menu Page 1 of 2
1) **GID:** - The gage ID can be edited here using alphanumeric characters.
2) **UID:** - The user ID can be edited here using alphanumeric characters.
3) **Meter**: - Selection of current meter (defaults to Price AA ST2).
4) **Reserved for future use.**
5) **Suspension:** - Selection of rod or cable.
6) **Sounding Wt:** - Select a value of a Columbus weight including hanger bar position.
7) **Ice Draft:** No/Yes selection for measuring ICE sections.
8) **Meas.:** Selection of measurement type.
9) **Measure Time(S):** Stopwatch time for monitoring clicks.
0) **Pct. Q Limit**: - Enter a value of maximum percent of estimated Q per panel.

Section Setup Menu Page 2 of 2
1) **Water Edge:** - Right/Left.
2) **Slope:** - Enter the slope of the stream as a percent.

3) **Gage Ht:** - Beginning primary gage height.

4) **Staff Ht:** - Beginning staff gage height.

5) **Estimated Q: -** Value as estimated from the rating table.

6) **Gage Ht:** - Ending primary gage height.

7) **Staff Ht:** - Ending staff gage height.

8) **Adj. Est. Q:** - An estimated value of Q that be changed after the start of the measurement.

Soft-Keys – Adjust Contrast Darken and Lighten – When in the field quite often you will be standing directly in the sun and your display will darken, you can access the contrast adjustment while in the measure screen by selecting the Setup Soft-Key then the Darken / Lighten Soft-Keys.

Review Totals
Selecting the Review Totals key in the Measure Menu opens up the Subsection Totals Screen and provides another Soft-Key that allows the user to review the Section Totals

Data Entry Methods

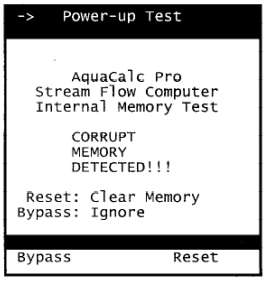
There are three ways the user can enter information when requested by the AquaCalc:

Toggle between predetermined values: -- Pressing the appropriate key toggles (or cycles) through predefined values. For example: Pressing the 4) Baud Rate key in the System Preferences Menu cycles through the available communication baud rates.

Selection of Menu Items -- When presented with a list of choices, such as a list of available meters, an item may be selected by pressing the indicated key on the numeric keypad.

Value Entry: When text and numeric values are entered by the user, such as Gage ID and Distance, they are displayed at the bottom of the screen during the entry process. Pressing **Enter** accepts the entry, while pressing **Esc** discards the value entered. The Left Arrow (**<**) key deletes the last key entered and the Right Arrow (**>**) key adds a space to a text entry.

Internal Memory Test Failure:



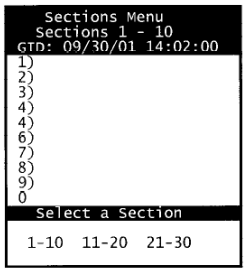
During the power-up initialization of the AquaCalc Pro the section data is tested for data integrity. If the section data is improperly formatted or a firmware upgrade requires the section database to be reset, the AquaCalc Pro will display a Corrupt Memory Detected screen and request the user to reset the section and meter databases. Pressing the Right Soft Key will continue the Reset operation **all data will be lost during a reset**. If you have data in the AquaCalc then press the bypass key and upload the data to your PC.

Performing a Measurement

Turn the AquaCalc On.

The main Menu screen appears.

Create a New Section



There are two ways to create a new section:

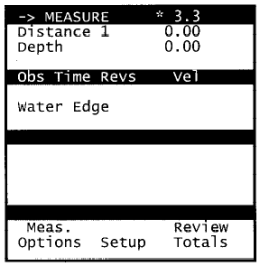
Select Measure from the Main Menu and go directly to the Measure screen. This will create a new section and give it a gage ID consisting of the date and time. You will then be placed directly into the measurement screen where you can directly start to measure or select from the Soft-Keys and set up the section.

Select Sections from the Main Menu and create a new section in the Sections screen by first selecting an empty section and pressing New on the Soft-Keys. This selection will place you directly into the Section Setup menu to setup the section. Setup the section and when completed you will be in the Measure screen with the first vertical defined as Water Edge.

In the Measure Screen

After setting up a section press the Esc key to return to the Measure screen.

1) The Display will now show Water Edge in the center of the display.



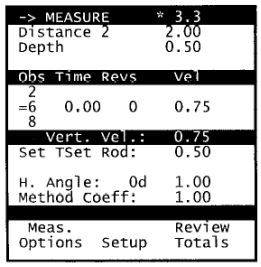
2) Connect your wading rod and current meter up the AquaCalc using the provided cable.

3) Enter a tag line distance for the Water Edge by pressing the Distance/3 key and entering a number using the numeric keypad and then pressing the Enter key. A measurement may not be performed at the Water Edge.

4) In the stream, move to the next tag line distance where you wish to perform a measurement.

5) Press the New Vertical key on the AquaCalc to move to the next vertical.

6) Enter the tag line distance as requested followed by the Enter key.



7) Press the Stream Depth/6 key and enter a depth for the stream, pressing the Enter key to complete the entry.

8) Select Observation Depth (.2 .6 .8) using the Up and Down arrow keys, noticing that the active line changes (identified by the equal signs in each margin).

9) Notice that the "Set Rod to:" line changes as different observation depths are chosen.

10) Press the Measure key to begin measuring, press the Halt key to stop a measurement before the time is complete and record a partial measurement. Press the "Esc" key or the "Abort" Soft-Key to cancel the measurement and not record a measurement.

Erratic Flow Reset
The AquaCalc Pro will restart the count and the measurement time if the time between "clicks" becomes erratic to fast or to slow. This condition is generally caused by a bad connection, or an improperly adjusted current meter.

11) Enter Horizontal Angle, Method Coefficient, and any other items that are necessary to define the conditions at the vertical by pressing the appropriate keys on the numeric keypad.

12) Select a new Observation Depth in the existing Vertical if desired by using the scroll keys, and perform another measurement. All observation velocities must be either a single point, two point, or three point. All other combinations are illegal and the vertical velocity will equal zero.

13) When all of the measurements are complete in the Vertical, press the "Next Vertical" key

14) Enter a Distance and a Stream Depth for the new vertical.

15) Perform a measurement in the new vertical.

16) Continue across the stream until finished.

17) When the section is complete, Mark the last vertical as the edge of water by pressing the "Edge/0" key.

18) Use the "Display Total" Soft-Key to review the Section summary.

Downloading a Measurement

1) Connect the AquaCalc Pro to your computer using the provided data cable.

2) The AquaCalc DataLink software provided with the Pro or any other serial communications package can be used to download a section from the AquaCalc to your computer.

3) Install the provided DataLink application on your PC.

4) Start the DataLink program. Click the Settings button to bring up the Settings dialog box. Choose the appropriate communications port and baud rate. The baud rate you choose must match the baud rate you set in the AquaCalc. The default baud rate is 9600. Select the New button from the tool bar. The Create New Capture Dialogue box will appear on the screen requesting that you create a new file or overwrite an existing file; you can also append data to an existing file. A dialog box will appear telling you to press Enter on the AquaCalc ignore this dialogue box.

5) Select and load the Section you wish to download in the Section Menu

6) In the Main menu, select the Section Download option by pressing the 3 key to begin the transfer.

7) The file will appear on your screen and will automatically be saved.

Using Non-Standard Current Meters

An addition to the built-in current meter rating curves for the default Price AA and Pygmy meters, the AquaCalc can store the rating curves for additional user defined "non-standard" current meters.

Non standard current meter ratings can be defined in several segments, one of which is used for lower velocities, the other segments for progressively higher velocities. Each segment is represented by an equation. A crossover or "breakpoint" velocity value is also specified that indicates the velocity at which the next equation is used.

Each equation takes the form:

$y = m * x + b$

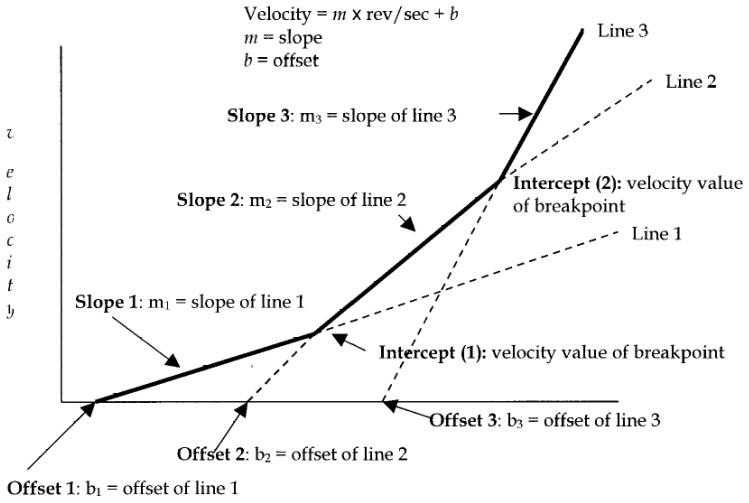
or

Velocity = *m* * (revolutions/second) + *b*

Where m is the slope of the line and b is the value where the line intercepts the *rev/sec* axis (which in the AquaCalc Pro is identified as the "Offset").

The AquaCalc Pro can handle non-standard meter rating curves with up to three line segments representing three velocity ranges. The "Intercept" value in the AquaCalc Pro represents the crossover or breakpoint velocity value at which second (or third) segment is used.

Figure 1: Current Meter Definition in the AquaCalc Pro



So for a sample Type AA Standard No. 1 two segment equation example:

EQUATIONS: V=2.18R + .020(2.200) 2.17R + .030

Slope 1:	2.18
Offset 1:	.020
Intercept (1):	2.20
Slope 2:	2.170
Offset 2	.030
Intercept (2):	0.0
Slope 3:	0.0
Offset 3:	0.0

A single segment rating curve equation (such as the new Type AA Standard No. 2) would be entered as:

EQUATION: V = 2.2048R + 0.0178

Slope 1:	2.2048
Offset 1:	0.0178
Intercept (1):	0
Slope 2:	0.0
Offset 2:	0.0
Intercept (2):	0.0
Slope 3:	0.0
Offset 3:	0.0

Step-by-step procedure to add a non-standard current meter

1) From the main menu, select number 4 "Meter Setup"
2) Use the right arrow navigation key to select a not yet identified meter (usually number 4 meter)
3) Press number 1 to name this meter
4) Press number 2 to name the meter ID number
5) Press number 3 to select the type of meter
6) Press number 4 to change to metric if required.
7) Press number 5 to change revolutions/pulses
8) Using the soft key, go to page 2of2
9) Utilizing keypad number 3 through 0, enter meter constants
10) Press Save

Upgrading the AquaCalc Pro firmware

When you received your AquaCalc it contained the latest firmware version. As JBS continues to improve the AquaCalc Pro, we will make firmware updates available to our customers. To obtain updates please contact JBS Instruments or check our website. You will then need to install the update by sending the update file from your computer to the AquaCalc using the AquaCalc Pro Firmware Updater from JBS.

The firmware in the AquaCalc Pro can be updated by the user. You need to perform the following steps:

1. Copy all data from the AquaCalc Pro to your computer using the DataLink program.

   WARNING: All Sections and observations in the AquaCalc Pro may be lost when updating the firmware!

2. Download the AquaCalc Pro Firmware Updater Installation Program from the JBS Energy website: http:// jbsenergy.com/Instruments/Downloads/ . Follow the links on the page to the AquaCalc Pro Download page.

   You may either choose to run the Installation program from the website or or to first save it to your computer and then run it.

3. If you chose the "Save" option, run the Updater Installation Program to install the Updater software on your computer. Otherwise go to the next step.

4. Connect the AquaCalc Pro to your computer using the data communications cable provided with your Pro.

5. Run the AquaCalc Pro Firmware Updater by selecting **AQ Pro Firmware Update** from the Windows Start Menu. Follow the on-screen directions in the Updater.

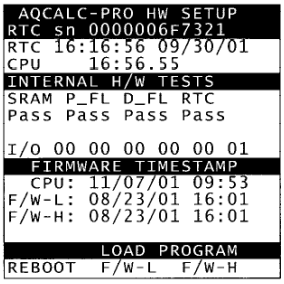
6. After your AquaCalc Pro firmware has been updated, you may remove the Updater software from your computer using the Windows Add/Remove Software function found in the Control Panel.

The AquaCalc Firmware Update Screen

The following provides an explanation of the items on the Firmware screen.

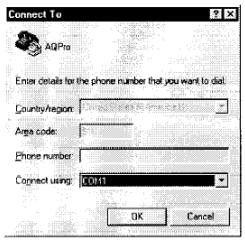
1. Turn off the AquaCalc Pro, if it is on.

2. While holding down the Enter key, press the On/Off key. Hold the Enter key until the Diagnostics Screen appears.
3. The AquaCalc will display the following Diagnostics Screen:



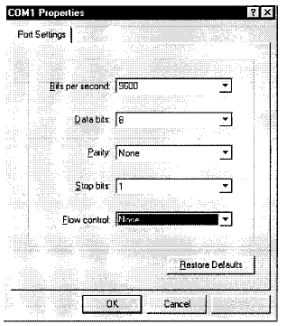
4. The serial number for your AquaCalc is displayed on the second line
5. RTC: Real Time Clock time and date
6. CPU Clock Time
7. Internal Hardware Tests for SRAM, Program Flash, Data Flash and Real Time Clock with Pass/Fail test results. If fail is reported please contact JBS.
8. I/O - Confirmation of data Input/Output
9. Firmware Timestamp – Version information for CPU and Firmware version time stamps
10. Program loading Status Line
11. The bottom line contains 3 soft keys – Reboot, F/W-L (Load Low) and F/WH (Load High)

1. The Connect To dialog box will now appear.



In the **Connect To** screen select an available communications port in the **Connect Using** drop-down list. Select COM1 or another available port. Click the OK button.

2. In the COM Properties dialog box: Select Bits per Second: 9600, Data bits: 8, Parity: None, bits: None, Flow Control, None. Press OK.



3. You are now at the main screen and have now completed the set up of HyperTerminal
4. Next you should save the set up by selecting File then Save
5. HyperTerminal is now setup

Loading the update into the AquaCalc

Loading to the Low Memory Location
With the AquaCalc connected to your computer and showing the Diagnostics screen, The HyperTerminal program setup and running and the update program stored on the computer's hard disk, you are now ready to update the firmware.

1. In the HyperTerminal program select "Send Text File..." from the Transfer menu. A file selection dialog box will open.
2. Select the folder where you stored the AquaCalc Pro firmware upgrade and select and highlight the latest version. **Do not double click on the file!** Double-clicking will open the file immediately. Wait.
3. On the AquaCalc Pro, use the center soft key to select F/W-L. A flashing message "Send Text File NOW" will appear.
4. In HyperTerminal, press the Open button, to begin the Transfer

5. The file is now loading this can take from 3 to 7 minutes depending on your operating system. The AquaCalc Pro will indicate that it is receiving the file by displaying the message "Uploading". When this message is no longer displayed and the firmware date and time changes, the transfer is complete.
6. Now you must load the update into High memory.

Loading to the High Memory Location
Repeat the above steps for loading into the low memory location, but at Step 4, select the "F/W-H" Soft-Key on the AquaCalc to load the same program into the AquaCalc's High memory location.

Once you have completed these steps and the AquaCalc, insure that the dates in the Low and High locations have changed and are the same. This indicates that you have successfully updated you firmware.

Reboot the AquaCalc by pressing the "REBOOT" Soft-Key.

You have successfully upgraded your AquaCalc Pro.

Angle Coefficient Protractor

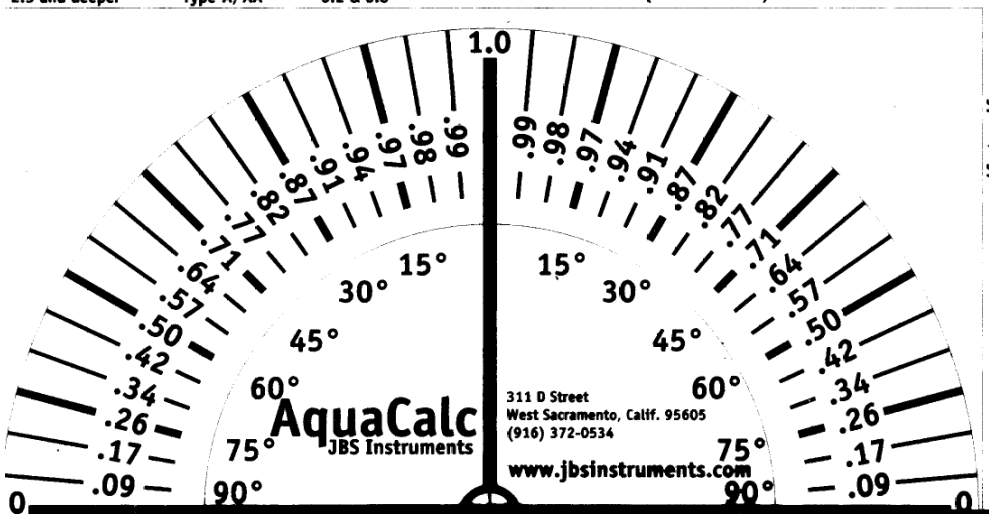
Current-Meter & Velocity-Measurement Method for Var. Depths

Water Depth in Feet	Use This Meter	Use This Velocity Method
0.0 to 0.3	*Surface float, Volumetric, Weir, Flume, Dye*	
0.3 to 1.5	Pygmy	0.6
1.5 to 2.5	Type-A, AA	0.6
2.5 and deeper	Type-A, AA	0.2 & 0.8

Minimum Depths for Weights & Velocity Measurement Methods

Suspension	Min. Depth (ft) 0.6 Method	Min Depth (ft.) 0.2 & 0.8 Method
15 C .5, 30 C .5	1.2	2.5
50 C .55	1.4	2.8
50 C .9	2.2	4.5
75 C 1.0, 100 C 1.0, 150 C 1.0	2.5	5.0
200 C 1.5, 300 C 1.5	3.8	7.5

Note - Use 0.2 method for depths 2.5-3.7 ft. with coefficient (estimated 0.88)



Acceptable Spin Test Limits, in Minutes

Meters	Normal	Min for Field Operation
Pygmy	1.5	0.5
Ice, Vane	3	1.5
Type A, Type AA	4	1.5

28. Appendix M: Pygmy meter

Pygmy Current Meters

The Geological Survey designed the first of its pygmy current meters (see fig. 1) in 1936. The pygmy current meter is of the Price type in that it contains a cup-type bucket wheel mounted on a vertical shaft having bearings that operate in air pockets. The bucket wheel is 2 inches in diameter (two-fifths the size of that in the small Price current meter). The pygmy meter is designed particularly for the measurement of discharges of those streams that are so shallow that the small Price current meter fails to perform accurately, but which have too great a flow to be measured conveniently by either volumetric means or with small weirs.

The pygmy meter differs from the type-AA small Price current meter in respects other than size (see fig. 4). The contact chamber is an integral part of the yoke and contains a single-revolution contact only. The meter has no tailpiece nor has it any provision for suspension from a cable. There is no bucket-wheel raising nut on the pygmy meter, but a small brass plug is provided to replace the pivot when the meter is stored or transported.

USGS Pygmy Meter Model 6205



The bucket wheel revolves about 2¼ times as fast as that of the small Price current meter. This relatively high speed, combined with the fact that no multiple-contact arrangement is provided, limits its use to conditions where the revolutions are counted aurally to velocities not exceeding 3 feet per second.

The Survey's pygmy current meters are constructed so that the bucket-wheel-and-hub assembly may be removed from the yoke as a unit for convenience in cleaning and oiling. Instructions for removing and replacing such assemblies follow:

To remove the bucket-wheel-and-hub assembly from the yoke:

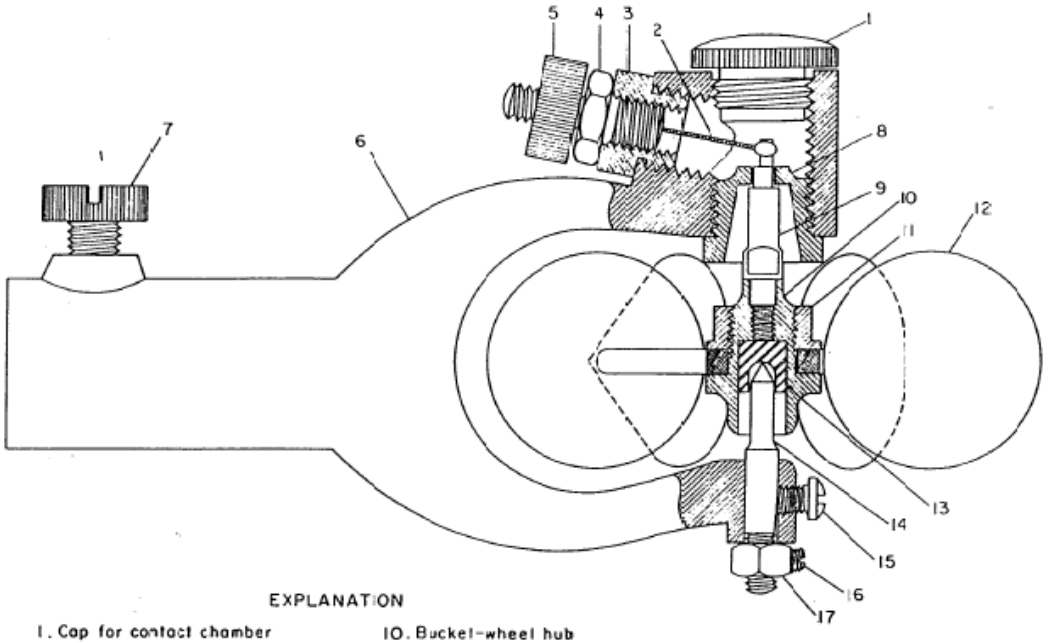
1. Remove the cap.
2. Release the set screw holding the pivot in the yoke.
3. Remove the pivot.
4. Tighten the set screw into the yoke (otherwise, it may offer difficulties in removing the bucket wheel).
5. Lower the bucket wheel to the lowest position in the yoke and carefully slide it forward and outward. If it is found that the bucket-wheel-and-hub assembly does not come out freely, return it to its original position and rotate it one-sixth of a turn. Repeat this operation until successful. Never apply force in removing the bucket-wheel-and hub assembly because the shaft and eccentric may become bent.

To insert the bucket-wheel-and-hub assembly into the yoke:

1. With the pivot removed, set screw tightened, cap removed, and yoke and shaft held upside down, direct the upper end of the shaft into the hole of the upper bearing, and carefully adjust the bucket wheel into position within the arms of the yoke. Do not apply force. If the bucket wheel cannot be placed within the yoke without forcing, remove it, turn it one-sixth of a revolution, and repeat until successful.
2. Unscrew the set screw to a position that will permit the pivot to be inserted.
3. Insert the pivot.
4. Tighten the set screw and turn the yoke right side up.
5. Replace the cap.

RICKLY HYDROLOGICAL CO.

1700 Joyce Avenue Columbus, Ohio 43219 U.S.A.
PHONE: (614) 297-9877 http://www.rickly.com
FAX: (614) 297-9878 E-mail: sales@rickly.com



EXPLANATION

1. Cap for contact chamber
2. Binding-post beaded wire
3. Binding-post insulating bushing
4. Binding-post body
5. Binding-post nut
6. Yoke
7. Yoke set screw
8. Upper bearing
9. Shaft
10. Bucket-wheel hub
11. Bucket-wheel hub nut
12. Bucket wheel
13. Pivot bearing
14. Pivot
15. Pivot set screw
16. Pivot-adjusting nut keeper screw
17. Pivot-adjusting nut

Figure 4.—Assembly diagram of pygmy current meter.

RICKLY HYDROLOGICAL CO.
1700 Joyce Avenue
Columbus, Ohio 43219 U.S.A.
PHONE: (614) 297-9877

CARE OF THE CURRENT METER

Rinse Current Meter in clear water as soon as possible after use, and dry using a soft cloth. Never place a wet Current Meter in its case. Lubricate, using lubricant supplied after approximately each 8 hours of use, or at least once a week if used infrequently.

To avoid damage to Pivot & Pivot Bearing, be sure to replace Pivot with brass Shipping Pivot supplied with Current Meter when Current Meter is not in use.

1700 Joyce Avenue · Columbus, Ohio 43219 · USA
Phone: (614) 297-9877 www.rickly.com
Fax: (614) 297-9878 E-mail: sales@rickly.com

RICKLY HYDROLOGICAL CO.

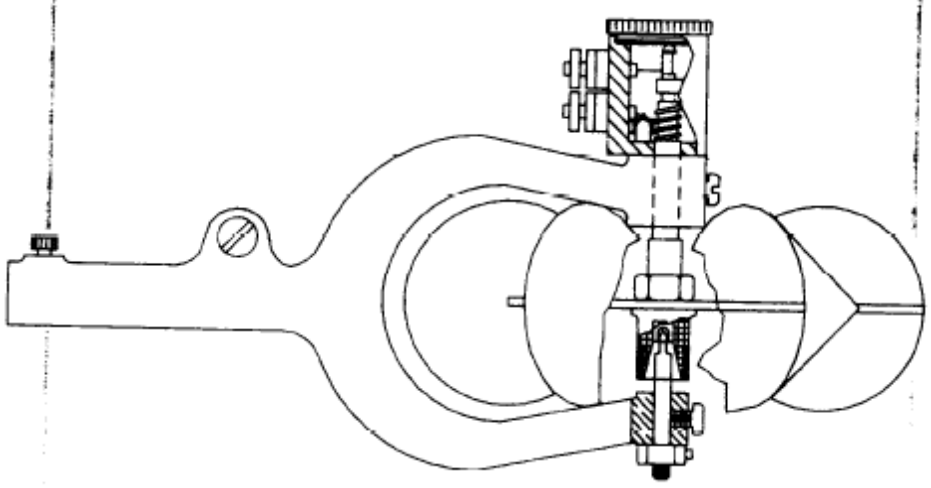
1700 Joyce Avenue · Columbus, Ohio 43219 · USA
Phone: (614) 297-9877 www.rickly.com
Fax: (614) 297-9878 E-mail: sales@rickly.com

STANDARD RATING TABLE NO. 2 FOR PYGMY CURRENT METER (6/99)
EQUATION: V = 0.9604 R+ 0.0312 (R=revolutions per second)

Seconds	VELOCITY IN FEET PER SECOND Revolutions														
	3	5	7	10	15	20	25	30	40	50	60	80	100	150	200
40	0.103	0.151	0.199	0.271	0.391	0.511	0.631	0.752	0.992	1.23	1.47	1.95	2.43	3.63	4.83
41	0.101	0.148	0.195	0.265	0.383	0.500	0.617	0.734	0.968	1.20	1.44	1.91	2.37	3.54	4.72
42	0.100	0.146	0.191	0.260	0.374	0.489	0.603	0.717	0.946	1.17	1.40	1.86	2.32	3.46	4.60
43	0.098	0.143	0.188	0.255	0.366	0.478	0.590	0.701	0.925	1.15	1.37	1.82	2.26	3.38	4.50
44	0.097	0.140	0.184	0.249	0.359	0.468	0.577	0.686	0.904	1.12	1.34	1.78	2.21	3.31	4.40
45	0.095	0.138	0.181	0.245	0.351	0.458	0.565	0.671	0.885	1.10	1.31	1.74	2.17	3.23	4.30
46	0.094	0.136	0.177	0.240	0.344	0.449	0.553	0.658	0.866	1.08	1.28	1.70	2.12	3.16	4.21
47	0.093	0.133	0.174	0.236	0.338	0.440	0.542	0.644	0.849	1.05	1.26	1.67	2.07	3.10	4.12
48	0.091	0.131	0.171	0.231	0.331	0.431	0.531	0.631	0.832	1.03	1.23	1.63	2.03	3.03	4.03
49	0.090	0.129	0.168	0.227	0.325	0.423	0.521	0.619	0.815	1.01	1.21	1.60	1.99	2.97	3.95
50	0.089	0.127	0.166	0.223	0.319	0.415	0.511	0.607	0.800	0.992	1.18	1.57	1.95	2.91	3.87
51	0.088	0.125	0.163	0.220	0.314	0.408	0.502	0.596	0.784	0.973	1.16	1.54	1.91	2.86	3.80
52	0.087	0.124	0.160	0.216	0.308	0.401	0.493	0.585	0.770	0.955	1.14	1.51	1.88	2.80	3.73
53	0.086	0.122	0.158	0.212	0.303	0.394	0.484	0.575	0.756	0.937	1.12	1.48	1.84	2.75	3.66
54	0.085	0.120	0.156	0.209	0.298	0.387	0.476	0.565	0.743	0.920	1.10	1.45	1.81	2.70	3.59
55	0.084	0.119	0.153	0.206	0.293	0.380	0.468	0.555	0.730	0.904	1.08	1.43	1.78	2.65	3.52
56	0.083	0.117	0.151	0.203	0.288	0.374	0.460	0.546	0.717	0.889	1.06	1.40	1.75	2.60	3.46
57	0.082	0.115	0.149	0.200	0.284	0.368	0.452	0.537	0.705	0.874	1.04	1.38	1.72	2.56	3.40
58	0.081	0.114	0.147	0.197	0.280	0.362	0.445	0.528	0.694	0.859	1.02	1.36	1.69	2.51	3.34
59	0.080	0.113	0.145	0.194	0.275	0.357	0.438	0.520	0.682	0.845	1.01	1.33	1.66	2.47	3.29
60	0.079	0.111	0.143	0.191	0.271	0.351	0.431	0.511	0.671	0.832	0.992	1.31	1.63	2.43	3.23
61	0.078	0.110	0.141	0.189	0.267	0.346	0.425	0.504	0.661	0.818	0.976	1.29	1.61	2.39	3.18
62	0.078	0.109	0.140	0.186	0.264	0.341	0.418	0.496	0.651	0.806	0.961	1.27	1.58	2.35	3.13
63	0.077	0.107	0.138	0.184	0.260	0.336	0.412	0.489	0.641	0.793	0.946	1.25	1.56	2.32	3.08
64	0.076	0.106	0.136	0.181	0.256	0.331	0.406	0.481	0.631	0.782	0.932	1.23	1.53	2.28	3.03
65	0.076	0.105	0.135	0.179	0.253	0.327	0.401	0.474	0.622	0.770	0.918	1.21	1.51	2.25	2.99
66	0.075	0.104	0.133	0.177	0.249	0.322	0.395	0.468	0.613	0.759	0.904	1.20	1.49	2.21	2.94
67	0.074	0.103	0.132	0.175	0.246	0.318	0.390	0.461	0.605	0.748	0.891	1.18	1.46	2.18	2.90
68	0.074	0.102	0.130	0.172	0.243	0.314	0.384	0.455	0.596	0.737	0.879	1.16	1.44	2.15	2.86
69	0.073	0.101	0.129	0.170	0.240	0.310	0.379	0.449	0.588	0.727	0.866	1.14	1.42	2.12	2.81
70	0.072	0.100	0.127	0.168	0.237	0.306	0.374	0.443	0.580	0.717	0.854	1.13	1.40	2.09	2.78
	3	5	7	10	15	20	25	30	40	50	60	80	100	150	200

MODEL'S 1210, 1215 AND 1220 PRICE TYPE "AA" CURRENT METERS

INSTRUCTION AND CARE MANUAL



SCIENTIFIC INSTRUMENTS, INC.
518 W. Cherry Street, Milwaukee, WI 53212
Phone: (414) 263-1600 • Fax: (414) 263-5506

MODELS 1210, 1215 AND 1220 CURRENT METERS

TABLE OF CONTENTS

Introduction **1**
General 1
Theory of Operation 1
Specifications 1

Using the Current Meter **5**
Preparing the Current Meter 5
Collecting Data 8

Total Discharge Volume **9**
Theory 9
Total Discharge Calculation Methods 12
General 12
Velocity Curve Method 12
Two-Point Method 12
Six-Tenths Method 13
Converting Current Meter Data to Flow Velocity 13

Maintenance and Servicing **15**
Cleaning and Inspection 15
Pivot Assembly Adjustment 16
Lubrication 17
Spin Test 17
Service Parts 18
Model 1215 18
Model 1210 19
Model 1220 20

Accessories **21**
Wading Rods 21
Sounding Reels 21
Cranes 21
Sounding Weights 21

Bibliography **21**

INTRODUCTION

GENERAL

Scientific Instruments Models 1210, 1215 and 1220 Price-type Current Meters, in combination with other equipment, are used for measuring flow velocity in bodies of water. The meters are rated for measuring velocity from .25 fps to 8 fps (.08 mps to 2.44 mps).

Price-type meters have been in use in the U.S. since the early part of the 20th century. Due to their relatively low cost, ease of use, accuracy in the presence of sediment-laden water, reliability and serviceability, they continue to be an industry standard.

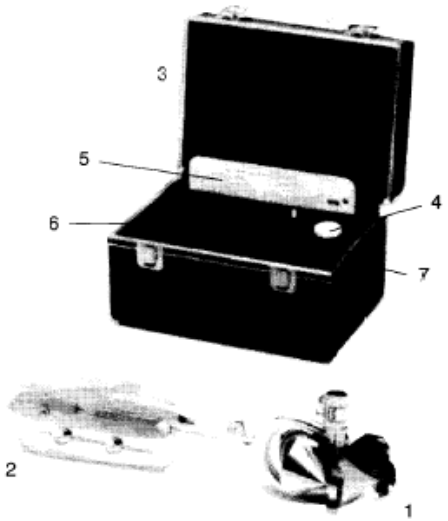
THEORY OF OPERATION

Price-type current meters (also referred to in this manual as instruments or meters) operate essentially the same way regardless of size, method of measurement, suspension means or data collection methods. A precisely balanced "Bucket Wheel" is mounted on a vertical pivot and is turned by flowing water. Water velocity is determined by counting the number of revolutions of the "Bucket Wheel" over a given period of time. A "Cat Whisker Switch" (or magnetic pick-up in the Model 1215) produces a signal that is either manually counted or recorded by a counting instrument. Magnetic pick-up meters are more accurate for measuring very low water velocities. This value, or count, is then compared on a cross-reference chart, or rating chart, to yield a water flow velocity.

SPECIFICATIONS

MODELS 1210, 1215 and 1220

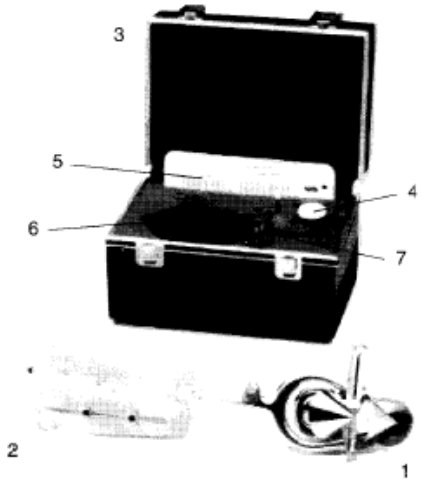
FLOW VELOCITY – feet per second (meters per second)	
– Minimum	.25 fps (.08 mps)
– Maximum	8.00 fps (2.44 mps)
WEIGHT – pounds (kilogram)	Approximately 1.8 lbs (.81 kg)
SUSPENSION MEANS	Models 1210, 1215 Wading Rod or Cable
	Model 1220 Wading Rod Only



1 Current Meter
2 Tail Fin Assembly
3 Carrying Case
4 Spare Parts Container including:
- Pivot Assembly
- Binding Post Assembly
- Pivot Set Screw
- Thumb Screw
- Hanger Screw

5 Rating Charts
6 Instrument Oil (2 Oz.)
7 Screwdriver

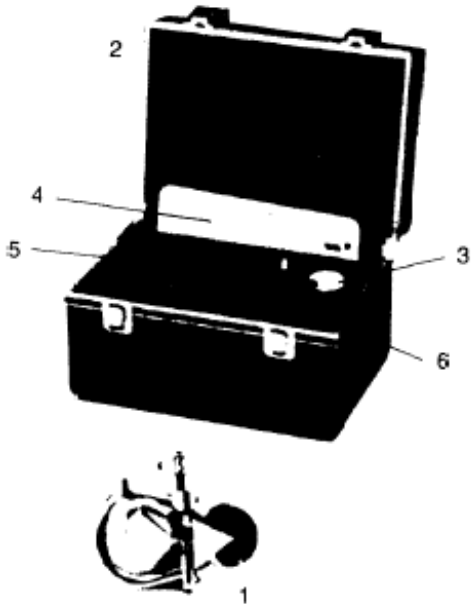
Figure 1. Model 1210 Current Meter



1 Current Meter
2 Tail Fin Assembly
3 Carrying Case
4 Spare Parts Container Including:
- Pivot Assembly
- Pivot Set Screw
- Thumb Screw
- Hanger Screw

5 Rating Charts
6 Instrument Oil (2 Oz.)
7 Screwdriver

Figure 2. Model 1215 Current Meter



1 Current Meter
2 Carrying Case
3 Spare Parts Container Including:
  - Pivot Assembly
  - Binding Post Assembly
  - Pivot Set Screw
4 Rating Charts
5 Instrument Oil (2 Cz.)
6 Screwdriver

Figure 3. Model 1220 Current Meter

USING THE CURRENT METER

PREPARING THE CURRENT METER

Inspect the Current Meter to ensure that it is in good operating condition. Refer to Figure 4 and check the following:

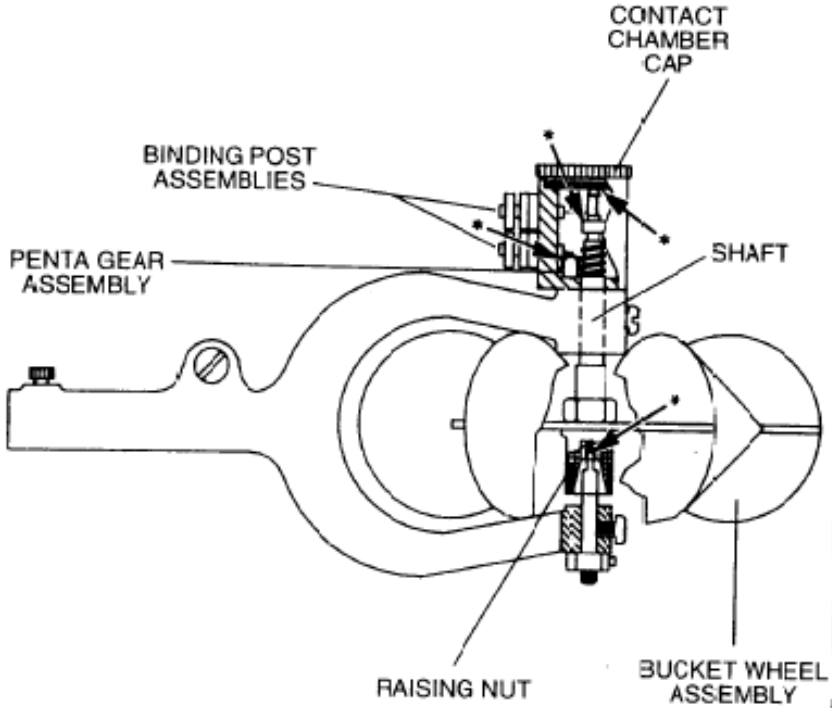
- Turn the Knurled Raising Nut clockwise, when viewed from above, until it is in the up position. This brings the Pivot into contact with the Pivot Bearing allowing the Bucket Wheel to spin freely. (Note: The Raising Nut is a left handed thread and turns in the opposite direction of a standard thread.)
- Check that the Bucket Wheel spins freely and does not bind or wobble and is defect free.
- Remove the Contact Chamber Cap and inspect the upper and lower Binding Post Contact Wires. Wires should be corrosion free and show no sign of excessive bends. (Does not apply to Model 1215)
- Ensure that the Contact Wires make contact with the Shaft and Penta Gear for approximately 1/6 of a revolution when the Bucket Wheel is turned.
- Add a drop of instrument oil to bearing surfaces. (Marked by arrows on Figure 4)
- Perform a spin test. (See MAINTENANCE AND SERVICE section).

CAUTION

Do not turn the "Bucket Wheel" in the wrong direction as this can damage the contact wires and cause inaccurate readings. The correct direction of rotation is counter-clockwise as viewed from above.

Note: The contact unit on the Model 1215 meter is sealed and cannot be visually checked.

The Current Meter is now ready for mounting on a wading rod or cable suspension system. Please refer to instructions supplied with the suspension system being used.



* Arrows indicate bearing surfaces to be oiled

Figure 4. Preparing Meter for Use

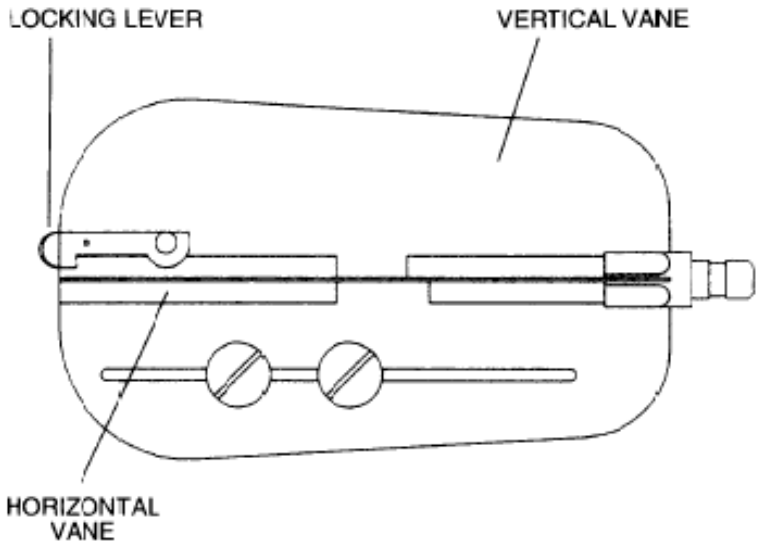


Figure 5.

- Assemble the Tail Fins as shown in Figure 5. Fins should have no overhang on either end when assembled properly. Make sure Locking Lever is fully locked.
- Install the Tail Fin assembly in accordance with instructions included with the suspension system being used.
- Attach the Current Meter to the applicable suspension system. Follow the instructions included with the suspension system (wading rod, sounding reel, etc.).
- Refer to Figure 4 and connect the monitoring instrument lead to the Meter as follows:
  - To obtain a pulse every revolution, connect the lead to the Upper Binding Post stamped "1". Position the lead under the Knurled Nut and tighten the nut.
  - To obtain a pulse every fifth revolution, connect the lead to the Lower Binding Post stamped '5". Position the lead under the Knurled Nut and tighten the nut.

COLLECTING DATA

1. Lower the Current Meter into the water. Position it for the method to be used (see TOTAL DISCHARGE CALCULATION METHODS).
2. Ensure that the Meter is positioned so the "Bucket Wheel" is facing into the current or into the direction of water flow.
3. Collect and record data. Listen for the pulses from the Meter through the headset and, using a stopwatch, count the pulses over a period of 40 to 70 seconds.

Note: Before and after each measurement, examine the meter for damage, wear or faulty alignment. Check meter balance if a hanger is used.

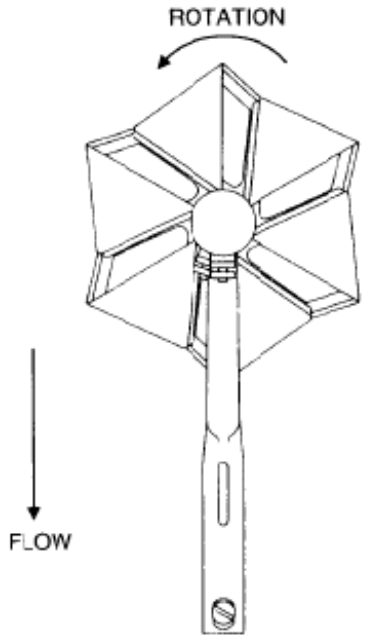


Figure 6. Direction of Rotation

TOTAL DISCHARGE VOLUME

THEORY

The data collected with the Current Meter is used to determine the total discharge volume of a stream or a river.

Total discharge is the sum of the products of the partial areas (vertical cross-sections) and their respective average velocities. As such, total discharge of a river can be expressed by the following formula:

Total Discharge Formula

$$Q = \Sigma\ (a\ v)$$

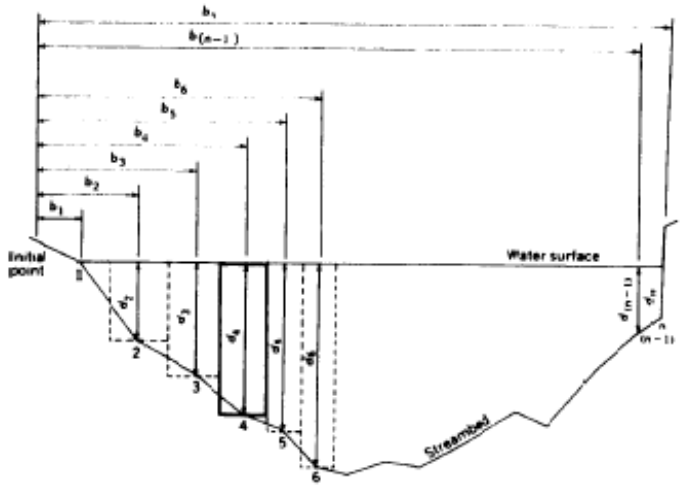
Q = total discharge

a = individual partial cross-sectional area

v = corresponding mean velocity of the flow normal to the partial area

Velocity measurements can be taken at many points in the cross-section of a river or stream and totaled to equal the total discharge. Each measured point represents a vertical cross-section. The flow of water in any vertical cross-section can be described as a point on a non-linear curve. That is, the water moves fastest at the surface and slowest at the bottom.

It can be assumed that the velocity sample at each point represents the mean velocity in a partial rectangular area. This rectangular area extends laterally halfway the distance from the preceding sample location and halfway the distance to the next sample location. It extends vertically from the water surface to the bottom. The cross-section is defined by depths at locations 1, 2, 3,... n. Refer to Figure 7.



EXPLANATION

1, 2, 3, ... n	Observation points
$b_1, b_2, b_3, \ldots\ldots b_n$	Distance, in feet, from the initial point to the observation point
$d_1, d_2, d_3, \ldots\ldots d_n$	Depth of water, in feet, at the observation point
Dashec lines	Boundary of partial sections; one heavily outlined discussed in text

Figure 7. Cross-Sectional Area of a River or Stream

The velocities are sampled at each point to obtain the mean of the velocity distribution. To compute the partial discharge for any partial section at any given location, use the follcwirg formula:

Partial Discharge Formula

$$q_x = v_x \left[\frac{(b_x - b_{(x-1)})}{2} + \frac{(b_{(x+1)} - b_x)}{2} \right] d_x$$

$$= v_x \left[\frac{b_{(x+1)} - b_{(x-1)}}{2} \right] d_x$$

where

q_x = discharge through partial section x,
v_x = mean velocity at location x,
b_x = distance from initial point to location x,
$b_{(x-1)}$ = distance from initial point to preceding location,
$b_{(x+1)}$ = distance from initial point to next location,
d_x = depth of water at location x.

Using the example shown in Figure 7, the discharge through the partial section 4 would be expressed as follows:

Partial section 4 discharge

$$q_4 = v_4 \left[\frac{b_5 - b_3}{2} \right] d_4$$

The procedure is similar when X is at an end section. The preceding location at the beginning of the cross-section is designated Location 1; the next location at the end cf the cross-section is designated Location n. Discharge through the end section would be expressed as follows:

End Section Discharge

$$q_1 = v_1 \left[\frac{b_2 - b_1}{2} \right] d_1 \quad \textit{and}$$

$$q_n = v_n \left[\frac{b_n - b_{(n-1)}}{2} \right] d_n$$

TOTAL DISCHARGE CALCULATION METHODS

General

There are three commonly used methods of calculating total discharge. The Velocity Curve method is the most accurate, the slowest and the most complicated. The Two-Point and the Six-Tenths methods are the most commonly used. Of the three, the Two-Point method should be used wherever possible because of its favorable balance of accuracy and speed.

Velocity Curve Method

This method involves taking as many readings as possible at as many different depths as possible along the entire vertical cross-section and averaging all the readings to determine the mean velocity. This method can be quite complex and can require literally hundreds of readings for larger bodies of water. The Velocity Curve method should only be used when absolute accuracy is required and time is of no concern.

Two-Point Method

When using the Two-Point method, measurements are taken at two pre-determined points to find the mean velocity. Measurements are taken at 20% and 80% of the water surface-to-bottom distance. The average of these two readings is taken as the mean velocity of the vertical cross-section. Experience has shown that this method renders more accurate results than any other method except the Velocity Curve method.

Note: The Two-Point method should not be used in water less than 2.5 feet (.762 meter) deep.

Six-Tenths Method

The velocity of water at a point six-tenths below the surface very closely approximates the mean velocity of the entire vertical cross-section, making it possible to gather relatively accurate data by taking only one measurement in a particular vertical cross-section.

Position the Current Meter at six-tenths of the total water depth (four tenths up from the bottom) below the water's surface. For a stream 2.2 feet (.67 meter) deep, this position would be 1.32 feet (.40 meter) below the surface of the water.

The Six-Tenths method gives reliable results under several conditions:

- When the water depth is between .3 and 2.5 feet (.091 and .762 meter)
- When large volumes of slush, ice, debris or flotsam make the Two-Point method impossible
- When the requirement for a sounding weight precludes the use of the Two-Point method
- When quick measurements are required

CONVERTING CURRENT METER DATA TO FLOW VELOCITY

Use the English or metric chart furnished with the Meter to convert raw data to water velocity. As an example:

- 100 pulses (revolutions of the "Bucket Wheel") are counted in 60 seconds.
- Using the English rating chart, locate 100 (revolutions) on the top line and 60 (seconds) in the vertical column.
- These two values intersect at a water velocity of 3.65 fps.
- See charts on page 14.

93

English Rating Chart

ENGLISH RATING TABLE FOR PRICE AA CURRENT METER

VELOCITY IN FEET PER SECOND — STANDARD RATING NO. 1

TIME IN SECS	REVOLUTIONS 2	5	7	10	15	20	25	30	40	REVOLUTIONS 50	60	80	100	150	200	250	300	360	TIME IN SECS
40	163	282	401	561	837	1.11	1.38	1.66	2.20	2.74	3.28	4.37	5.45	8.17	10.88	13.59	16.30	19.02	40
41	160	280	397	551	818	1.08	1.35	1.62	2.15	2.68	3.21	4.28	5.32	7.97	10.62	13.28	15.91	18.55	41
42	158	273	383	536	799	1.06	1.32	1.58	2.10	2.61	3.13	4.18	5.20	7.78	10.34	12.95	15.53	18.11	42
43	152	270	379	521	780	1.03	1.28	1.54	2.05	2.55	3.06	4.07	5.08	7.60	10.12	12.65	15.17	17.69	43
44	149	266	361	514	760	1.01	1.26	1.51	2.00	2.50	2.99	3.98	4.96	7.43	9.86	12.36	14.83	17.29	44
45	145	260	358	501	747	998	1.23	1.47	1.96	2.44	2.92	3.89	4.85	7.26	9.67	12.08	14.40	16.81	45
46	142	257	350	484	731	968	1.20	1.44	1.92	2.39	2.86	3.80	4.75	7.11	9.48	11.83	14.18	16.54	46
47	139	252	343	484	716	948	1.18	1.41	1.88	2.34	2.80	3.72	4.65	6.96	9.28	11.57	13.86	16.19	47
48	135	247	336	474	701	928	1.16	1.38	1.84	2.29	2.74	3.65	4.55	6.81	9.07	11.33	13.58	15.85	48
49	133	242	330	465	687	910	1.13	1.35	1.80	2.24	2.68	3.57	4.46	6.67	8.89	11.10	13.32	15.53	49
50	131	238	325	456	674	892	1.11	1.32	1.76	2.20	2.63	3.50	4.37	6.54	8.71	10.88	13.05	15.22	50
51	128	234	319	447	661	876	1.09	1.30	1.73	2.16	2.58	3.43	4.29	6.41	8.54	10.67	12.79	14.92	51
52	126	230	313	438	649	858	1.07	1.28	1.70	2.12	2.53	3.37	4.20	6.28	8.36	10.46	12.55	14.64	52
53	124	226	308	431	637	843	1.05	1.25	1.67	2.08	2.48	3.31	4.12	6.17	8.23	10.31	12.37	14.28	53
54	121	222	303	424	626	827	1.03	1.23	1.63	2.04	2.44	3.24	4.05	6.06	8.07	10.08	12.08	14.09	54
55	119	218	297	418	615	813	1.01	1.21	1.61	2.00	2.40	3.19	3.98	5.95	7.92	9.89	11.87	13.84	55
56	117	215	293	408	604	798	993	1.19	1.58	1.97	2.35	3.13	3.90	5.84	7.78	9.72	11.66	13.59	56
57	115	211	288	403	594	785	976	1.17	1.55	1.93	2.31	3.08	3.84	5.74	7.64	9.55	11.45	13.35	57
58	113	208	284	396	584	772	960	1.15	1.52	1.90	2.27	3.02	3.77	5.64	7.51	9.38	11.25	13.12	58
59	111	205	279	388	574	759	944	1.13	1.50	1.87	2.24	2.97	3.71	5.55	7.39	9.23	11.08	12.92	59
60	109	202	274	383	565	747	929	1.11	1.47	1.84	2.20	2.92	3.65	5.46	7.26	9.07	10.88	12.69	60
61	107	198	270	377	556	735	913	1.09	1.45	1.81	2.16	2.88	3.59	5.37	7.14	8.92	10.70	12.48	61
62	105	196	266	373	547	723	899	1.07	1.43	1.78	2.13	2.83	3.53	5.28	7.02	8.76	10.53	12.28	62
63	104	193	262	366	539	712	885	1.06	1.40	1.75	2.09	2.79	3.47	5.20	6.92	8.64	10.36	12.09	63
64	102	190	258	361	531	701	872	1.04	1.38	1.72	2.06	2.74	3.42	5.12	6.81	8.51	10.20	11.90	64
65	101	188	255	355	523	691	858	1.03	1.36	1.70	2.03	2.70	3.37	5.04	6.71	8.38	10.05	11.71	65
66	099	185	251	350	515	681	846	1.01	1.34	1.67	2.00	2.66	3.32	4.97	6.61	8.26	9.90	11.54	66
67	098	183	248	345	508	671	833	996	1.32	1.65	1.97	2.62	3.27	4.89	6.51	8.13	9.75	11.37	67
68	096	180	244	341	501	661	821	983	1.30	1.62	1.94	2.58	3.22	4.82	6.41	8.01	9.60	11.20	68
69	095	178	241	336	494	652	810	968	1.28	1.60	1.91	2.54	3.17	4.75	6.32	7.89	9.46	11.04	69
70	093	176	238	331	487	643	798	954	1.27	1.58	1.89	2.51	3.13	4.68	6.23	7.78	9.33	10.88	70

EQUATIONS — LESS THAN 40 REVS: $\frac{REV}{TIME} \times 2.180 + .020 = F.P.S.$ MORE THAN 40 REVS: $\frac{REV}{TIME} \times 2.170 + .030 = F.P.S.$

RATING LIMITS — .25 to 8.0 F.P.S.

Metric Rating Chart

METRIC RATING TABLE FOR PRICE AA CURRENT METER

VELOCITY IN METERS PER SECOND — STANDARD RATING NO. 1

TIME IN SECS	REVOLUTIONS 2	3	5	7	10	15	20	25	30	40	REVOLUTIONS 50	60	80	100	150	200	250	300	360	TIME IN SECS
40	089	089	122	172	255	338	421	503	671	835	1.00	1.33	1.66	2.48	3.32	4.14	4.97	5.80	40
41	085	087	119	168	248	329	411	493	655	817	978	1.30	1.62	2.43	3.24	4.04	4.85	5.66	41
42	084	085	117	164	243	323	402	482	640	796	954	1.27	1.59	2.37	3.16	3.95	4.73	5.52	42
43	082	083	114	161	238	314	392	469	625	777	933	1.24	1.55	2.32	3.08	3.86	4.62	5.39	43
44	081	082	112	157	233	308	384	460	610	762	911	1.21	1.51	2.26	3.01	3.77	4.52	5.27	44
45	079	080	109	154	228	301	374	448	587	744	890	1.19	1.48	2.21	2.95	3.69	4.42	5.15	45
46	078	078	107	151	223	295	366	439	569	729	871	1.16	1.45	2.17	2.88	3.60	4.32	5.04	46
47	076	077	105	148	218	289	360	430	573	713	853	1.13	1.42	2.12	2.82	3.53	4.23	4.93	47
48	075	075	103	144	214	283	354	421	561	698	835	1.11	1.39	2.08	2.78	3.48	4.14	4.83	48
49	073	074	101	142	209	277	344	411	548	683	820	1.09	1.36	2.03	2.71	3.38	4.06	4.73	49
50	072	073	099	139	205	272	338	405	536	671	802	1.07	1.33	1.99	2.65	3.32	3.98	4.64	50
51	070	071	097	136	201	267	332	396	527	656	786	1.05	1.30	1.95	2.60	3.25	3.90	4.55	51
52	069	070	095	134	196	262	326	390	516	646	771	1.03	1.28	1.92	2.55	3.19	3.83	4.46	52
53	068	069	094	131	194	257	320	381	509	634	759	1.01	1.26	1.88	2.51	3.13	3.75	4.38	53
54	067	068	092	129	191	252	314	375	497	622	744	988	1.23	1.85	2.46	3.07	3.69	4.29	54
55	065	066	091	127	187	248	308	369	491	610	732	972	1.21	1.81	2.41	3.01	3.62	4.22	55
56	064	065	089	125	184	244	303	363	482	601	718	954	1.19	1.78	2.37	2.96	3.55	4.14	56
57	063	064	088	123	181	239	297	357	472	588	704	938	1.17	1.75	2.33	2.91	3.49	4.07	57
58	061	063	086	121	178	235	293	351	463	578	692	921	1.15	1.72	2.29	2.86	3.43	4.00	58
59	060	062	085	119	175	231	288	344	457	570	683	909	1.13	1.69	2.25	2.81	3.37	3.93	59
60	059	062	084	117	172	226	283	338	448	561	671	890	1.11	1.66	2.21	2.76	3.32	3.87	60
61	058	061	082	115	169	224	278	332	442	552	658	878	1.09	1.64	2.18	2.72	3.26	3.80	61
62	056	060	081	113	167	220	274	326	436	543	648	863	1.08	1.61	2.14	2.68	3.21	3.74	62
63	055	059	080	112	164	217	270	323	427	533	640	850	1.06	1.58	2.11	2.63	3.16	3.69	63
64	054	058	078	110	162	214	266	317	421	524	626	838	1.04	1.56	2.08	2.59	3.1	3.63	64
65	053	057	076	108	159	211	262	314	415	518	618	823	1.03	1.54	2.05	2.55	3.06	3.57	65
66	052	056	075	107	157	208	258	308	408	509	610	811	1.01	1.51	2.01	2.51	3.01	3.52	66
67	051	056	074	105	155	205	254	304	402	503	601	799	999	1.48	1.98	2.46	2.97	3.47	67
68	050	055	073	104	153	202	250	299	396	494	591	786	983	1.47	1.95	2.44	2.93	3.41	68
69	049	054	072	102	151	199	247	295	390	486	585	777	966	1.45	1.93	2.40	2.88	3.36	69
70	048	054	071	101	148	196	244	291	387	482	578	768	954	1.43	1.90	2.37	2.84	3.32	70

EQUATION — LESS THAN 40 REVS: $\frac{REV}{TIME} \times 2.180 + .020 = .3048 = MPS$ MORE THAN 40 REVS: $\frac{REV}{TIME} \times 2.170 + .030 = .3048$

RATINGS LIMITS .076 TO 2.44 MPS

MAINTENANCE AND SERVICING

CLEANING AND INSPECTION

CAUTION

Maintenance and adjustments as outlined in this manual are intended as routine maintenance. Always contact manufacturer before attempting any major repairs or replacement of parts. Damage caused by service other than that outlined here may void manufacturers warranty.

- Wash the Current Meter in clean water after each use and dry with a soft cloth.
- Lubricate all bearing surfaces (see Figure 4) after each use, or at least weekly when not in use. Use the instrument oil supplied with the Meter.
- Remove the Contact Chamber Cap after each use and allow the inside of the Chamber to dry thoroughly.
- Turn the Knurled Raising Nut counterclockwise, when viewed from above, as far as it will travel. This separates the Pivot and Pivot Bearing, and prevents the "Bucket Wheel" from spinning.

CAUTION

Do not use excessive force to disassemble the Current Meter. Excessive force could damage some parts.

- Replace Binding Posts if Contact Wires are broken or bent excessively or will no longer make contact with the Gears or Shaft.

Note: Model 1215 Meters have magnetic pick-ups in place of contact wires. The Chamber is sealed and requires no field service.

- Replace any screws that have excessively worn heads or threads.
- Replace Pivot Assembly if it shows any signs of wear or damage.

PIVOT ASSEMBLY & ADJUSTMENT

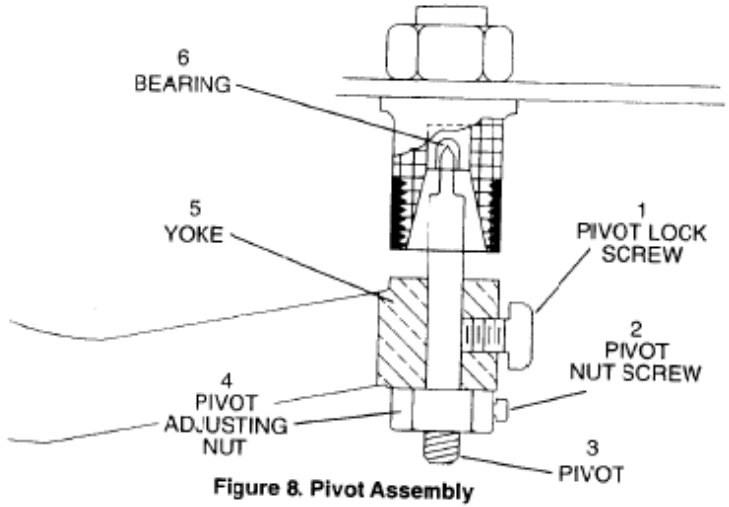


Figure 8. Pivot Assembly

Note: The Contact Chamber cap must be in place when adjusting the Pivot Assembly.

1. See Figure 8. Loosen the #2 Pivot Nut Screw and turn the #4 Pivot Adjusting Nut clockwise approximately two turns.
2. Slightly loosen the #1 Pivot Lock Screw. The #3 Pivot has a flat area under this screw. Make sure the flat area of the Pivot remains under this screw.
3. With the Meter in a vertical position (its normal operating position), push the #3 Pivot upward as far as it will go. While keeping the #3 Pivot in this position tighten the #1 Pivot Lock Screw.
4. Turn the #4 Pivot Adjusting Nut counterclockwise, as viewed from above, until it touches the #5 Yoke, then slightly loosen the #1 Pivot Lock Screw.
5. Turn the #4 Pivot Adjusting Nut 1/8 turn counterclockwise, as viewed from above, and tighten the #1 Pivot Lock Screw. This will position the #3 Pivot correctly in relation to the #6 Bearing.
6. Tighten the #1 Pivot Lock Screw.

Note: There will be a small amount of lateral play in the "Bucket Wheel." This ensures that the wheel is not impaired by the pivot while taking measurements.

SPIN TEST

CAUTION

Do not turn the "Bucket Wheel" in the wrong direction as this can damage the contact wires and cause inaccurate readings. The correct direction of rotation is counter-clockwise as viewed from above (see Figure 5).

A spin test should be performed after adjustment and lubrication to ensure that the Current Meter is in good working order.

Turn the Raising Nut clockwise, when viewed from above, until it is in the up position to place the pivot in contact with the Pivot Bearing. Hold or mount the Current Meter in a horizontal position.

Spin the "Bucket Wheel" briskly, counterclockwise as viewed from above, by snapping it with a thumb or finger. The "Bucket Wheel" should spin freely for a minimum of 3-1/2 minutes and come slowly to a stop. (The "Bucket Wheel" may reverse direction after stopping. This is normal and does not affect the Current Meter accuracy.)

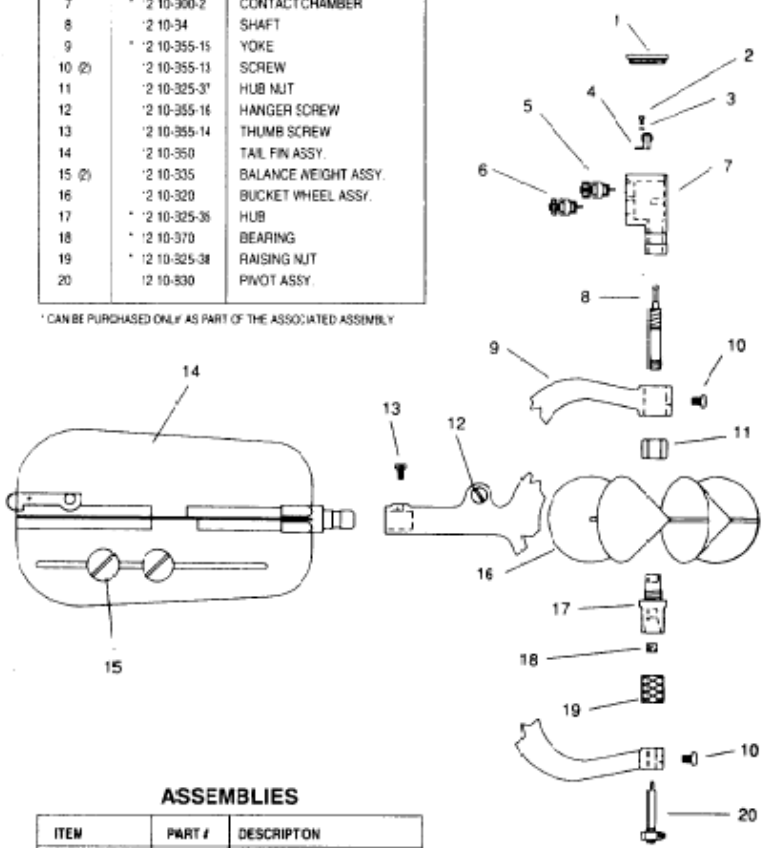
If the "Bucket Wheel" does not spin for a minimum of 3-1/2 minutes or if it comes to an abrupt stop, refer to PIVOT ASSEMBLY ADJUSTMENT in this section for proper adjustment procedures. If, after adjustment, the Current Meter still will not pass the spin test, additional factory service may be required.

IT IS RECOMMENDED YOUR METER BE FACTORY SERVICED ON A PERIODIC BASIS

ITEM		PART #	DESCRIPTION
1		12 10-300-1	CONTACT CHAMBER CAP
2	*	12 10-11	SCREW
3	*	12 10-12	WASHER
4	*	12 10-305	PENTA GEAR ASSY.
5		12 10-310	BINDING POST ASSY.-SINGLE
6		12 10-315	BINDING POST ASSY.-PENTA
7	*	12 10-300-2	CONTACT CHAMBER
8		12 10-34	SHAFT
9	*	12 10-355-15	YOKE
10	(2)	12 10-355-13	SCREW
11		12 10-325-37	HUB NUT
12		12 10-355-16	HANGER SCREW
13		12 10-355-14	THUMB SCREW
14		12 10-350	TAIL FIN ASSY.
15	(2)	12 10-335	BALANCE WEIGHT ASSY.
16		12 10-320	BUCKET WHEEL ASSY.
17	*	12 10-325-35	HUB
18	*	12 10-370	BEARING
19	*	12 10-325-38	RAISING NUT
20		12 10-330	PIVOT ASSY.

* CAN BE PURCHASED ONLY AS PART OF THE ASSOCIATED ASSEMBLY

MODEL 1210 SERVICE PARTS



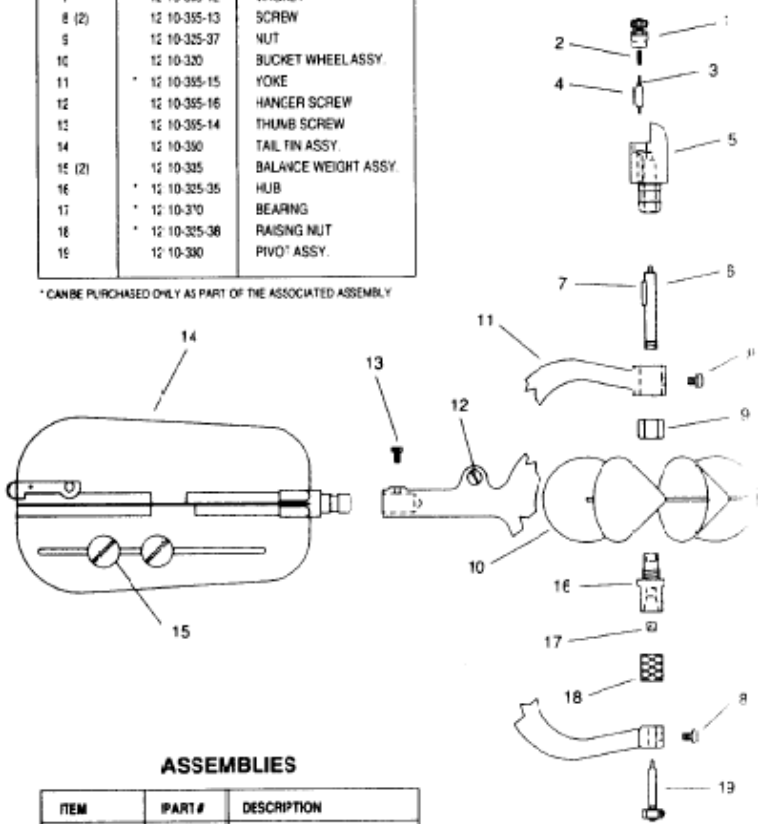
ASSEMBLIES

ITEM	PART #	DESCRIPTON
1, 2, 3, 4, 5, 6, 7	12 10-300	CONTACT CHAMBER ASSY.
11, 17, 18, 19	12 10-325	HUB ASSY.
14, 15	12 10-350	TAIL FIN ASSY.
9, 10 12, 13	12 10-355	YOKE ASSY.

ITEM		PART #	DESCRIPTION
1	*	12 15-315	BINDING POST
2	*	12 15-310-7	SPRING
3	*	12 15-310-6	SLEEVE
4	*	12 15-310-8	REED SWITCH
5	*	12 15-300-2	CONTACT CHAMBER
6	*	12 15-305-11	SHAFT
7	*	12 15-305-12	MAGNET
8	(2)	12 10-355-13	SCREW
9		12 10-325-37	NUT
10		12 10-320	BUCKET WHEEL ASSY.
11	*	12 10-355-15	YOKE
12		12 10-355-16	HANGER SCREW
13		12 10-355-14	THUMB SCREW
14		12 10-350	TAIL FIN ASSY.
15	(2)	12 10-335	BALANCE WEIGHT ASSY.
16	*	12 10-325-35	HUB
17	*	12 10-370	BEARING
18	*	12 10-325-38	RAISING NUT
19		12 10-330	PIVOT ASSY.

* CAN BE PURCHASED ONLY AS PART OF THE ASSOCIATED ASSEMBLY

MODEL 1215 SERVICE PARTS



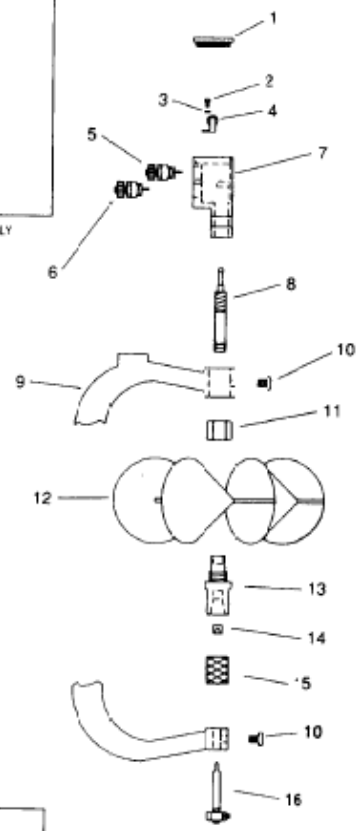
ASSEMBLIES

ITEM	PART #	DESCRIPTION
1, 2, 3, 4, 5	12 15-300	CONTACT CHAMBER ASSY.
6, 7	12 15-305	SHAFT ASSY.
1, 2, 3, 4	12 15-310	BINDING POST ASSY.
9, 16, 17, 18	12 10-325	HUB ASSY.
14, 15	12 10-350	TAIL FIN ASSY.
8, 11, 12, 13	12 10-355	YOKE ASSY.

ITEM	PART #	DESCRIPTION
1	12 10-300-1	CONTACT CHAMBER CAP
2	* 12 10-11	SCREW
3	* 12 10-12	WASHER
4	* 12 10-305	PENTA GEAR ASSY.
5	12 10-310	BINDING POST ASSY.-SINGLE
6	12 10-315	BINDING POST ASSY.-PENTA
7	* 12 10-300-2	CONTACT CHAMBER
8	12 10-34	SHAFT
9	* 12 20-355-15	YOKE
10 (2)	12 10-355-13	SCREW
11	12 10-325-37	HUB NUT
12	12 10-320	BUCKET WHEEL ASSY.
13	* 12 10-325-35	HUB
14	* 12 10-370	BEARING
15	* 12 10-325-36	RAISING NUT
16	12 10-330	PIVOT ASSY.

* CAN BE PURCHASED ONLY AS PART OF THE ASSOCIATED ASSEMBLY

MODEL 1220
SERVICE PARTS



ASSEMBLIES

ITEM	PART #	DESCRIPTION
1, 2, 3, 4, 5, 6, 7	12 10-300	CONTACT CHAMBER ASSY.
11, 13, 14, 15	12 10-325	HUB ASSY.
9, 10.	12 20-355	YOKE ASSY.

ACCESSORIES

WADING RODS

A Wading Rod is used to position a Current Meter at a desired depth in water that is shallow enough for wading. Refer to the instructions supplied with the Wading Rod.

SOUNDING REELS

A Sounding Reel is used to support and raise or lower a Current Meter from an overhead structure or boat. It is usually used in conjunction with a crane or boom. Refer to the instructions supplied with the Sounding Reel.

CRANES

A Crane provides the mount for a Sounding Reel and, with its boom, permits extension beyond a bridge railing or other obstruction. Wheels allow for easy movement to another location. Refer to the instructions supplied with the Crane.

SOUNDING WEIGHTS

Sounding Weights are used to maintain the position of a Current Meter in flowing water. They range in weight from 15 to 300 pounds. Refer to the instructions supplied with the Sounding Weight.

BIBLIOGRAPHY

Thomas J. Buchanan and William P. Sommers; "Techniques of Water-Resources Investigations of the United States Geological Survey, Chapter A8, Discharge Measurements at Gaging Stations, Book 3 Applications of Hydraulics." 1969 by the U.S. Government Printing Office, Washington, D.C.

28.1. English Rating Chart

English Rating Chart

ENGLISH RATING TABLE FOR PRICE AA CURRENT METER

VELOCITY IN FEET PER SECOND — STANDARD RATING NO 1

TIME IN SECS	REVOLUTIONS 3	5	7	10	15	20	25	30	40	50	60	80	100	150	200	250	300	350	TIME IN SECS
40	183	292	401	565	837	1 11	1 38	1 65	2 20	2 74	3 28	4 37	5 45	8 17	10 88	13 59	16 30	19 02	40
41	180	286	392	552	818	1 08	1 35	1 62	2 15	2 66	3 21	4 26	5 32	7 97	10 62	13 26	15 91	18 55	41
42	176	280	383	539	799	1 06	1 32	1 58	2 10	2 61	3 13	4 16	5 20	7 78	10 36	12 95	15 53	18 11	42
43	172	273	375	527	780	1 03	1 29	1 54	2 05	2 55	3 06	4 07	5 08	7 60	10 12	12 65	15 17	17 69	43
44	169	266	367	515	763	1 01	1 26	1 51	2 00	2 50	2 99	3 98	4 96	7 43	9 89	12 36	14 83	17 29	44
45	165	262	359	504	747	989	1 23	1 47	1 96	2 44	2 92	3 89	4 85	7 26	9 67	12 09	14 50	16 91	45
46	162	257	352	494	731	968	1 20	1 44	1 92	2 39	2 86	3 80	4 75	7 11	9 46	11 82	14 18	16 54	46
47	159	252	345	484	716	948	1 18	1 41	1 88	2 34	2 80	3 72	4 65	6 96	9 26	11 57	13 88	16 19	47
48	156	247	338	474	701	928	1 16	1 38	1 84	2 29	2 74	3 65	4 55	6 81	9 07	11 33	13 59	15 85	48
49	153	242	331	465	687	910	1 13	1 35	1 80	2 24	2 69	3 57	4 46	6 67	8 89	11 10	13 32	15 53	49
50	151	238	325	456	674	892	1 11	1 33	1 76	2 20	2 63	3 50	4 37	6 54	8 71	10 88	13 05	15 22	50
51	148	234	319	447	661	875	1 09	1 30	1 73	2 16	2 58	3 43	4 28	6 41	8 54	10 67	12 79	14 92	51
52	146	230	313	439	649	858	1 07	1 28	1 70	2 12	2 53	3 37	4 20	6 29	8 38	10 46	12 55	14 64	52
53	143	226	308	431	637	843	1 05	1 25	1 67	2 08	2 49	3 31	4 12	6 17	8 22	10 27	12 31	14 38	53
54	141	222	303	424	626	827	1 03	1 23	1 63	2 04	2 44	3 24	4 05	6 06	8 07	10 08	12 09	14 09	54
55	139	218	297	416	615	813	1 01	1 21	1 61	2 00	2 40	3 19	3 98	5 95	7 92	9 89	11 87	13 84	55
56	137	215	292	409	604	799	993	1 19	1 58	1 97	2 35	3 13	3 90	5 84	7 78	9 72	11 65	13 59	56
57	135	211	286	402	594	785	978	1 17	1 55	1 93	2 31	3 08	3 84	5 74	7 64	9 55	11 45	13 35	57
58	133	208	283	396	584	772	960	1 15	1 52	1 90	2 27	3 02	3 77	5 64	7 51	9 38	11 25	13 12	58
59	131	205	279	389	574	759	944	1 13	1 50	1 87	2 24	2 97	3 71	5 55	7 39	9 22	11 06	12 90	59
60	129	202	274	383	565	747	928	1 11	1 47	1 84	2 20	2 92	3 65	5 45	7 26	9 07	10 88	12 69	60
61	127	199	270	377	556	735	913	1 09	1 45	1 81	2 16	2 88	3 59	5 37	7 14	8 92	10 70	12 48	61
62	125	196	266	372	547	723	899	1 07	1 43	1 78	2 13	2 83	3 53	5 28	7 03	8 78	10 53	12 28	62
63	124	193	262	366	539	712	885	1 06	1 40	1 75	2 10	2 79	3 47	5 20	6 92	8 64	10 36	12 09	63
64	122	190	258	361	531	701	872	1 04	1 38	1 72	2 06	2 74	3 42	5 12	6 81	8 51	10 20	11 90	64
65	121	188	255	355	523	691	858	1 03	1 36	1 70	2 03	2 70	3 37	5 04	6 71	8 38	10 05	11 71	65
66	119	185	251	350	515	681	846	1 01	1 34	1 67	2 00	2 66	3 32	4 96	6 61	8 25	9 89	11 54	66
67	118	183	248	345	508	671	833	996	1 32	1 65	1 97	2 62	3 27	4 89	6 51	8 13	9 75	11 37	67
68	116	180	244	341	501	661	821	982	1 30	1 62	1 94	2 58	3 22	4 82	6 41	8 01	9 60	11 20	68
69	115	178	241	336	494	652	810	968	1 28	1 60	1 92	2 55	3 17	4 75	6 32	7 89	9 48	11 04	69
70	113	176	238	331	487	643	799	954	1 27	1 58	1 89	2 51	3 13	4 68	6 23	7 78	9 33	10 88	70

EQUATIONS — LESS THAN 40 REVS: $\frac{\text{REV}}{\text{TIME}} \times 2\ 180 + 020 = \text{F P S}$

MORE THAN 40 REVS: $\frac{\text{REV}}{\text{TIME}} \times 2\ 170 + 030 = \text{F P S}$

RATING LIMITS — 25 to 8 0 F P S

28.2. Metric Rating Chart

Metric Rating Chart

METRIC RATING TABLE FOR PRICE AA CURRENT METER

VELOCITY IN METERS PER SECOND — STANDARD RATING NO 1

TIME IN SECS	REVOLUTIONS 3	5	7	10	15	20	25	30	40	REVOLUTIONS 50	60	80	100	150	200	250	300	350	TIME IN SECS
40	055	089	122	172	255	338	421	503	671	835	1 00	1 33	1 66	2 49	3 32	4 14	4 97	5 80	40
41	055	087	119	168	249	329	411	493	655	817	978	1 30	1 62	2 43	3 24	4 04	4 85	5 65	41
42	054	085	117	164	245	323	402	482	640	798	954	1 27	1 59	2 37	3 18	3 95	4 73	5 52	42
43	052	083	114	161	238	314	393	469	625	777	933	1 24	1 55	2 32	3 08	3 86	4 62	5 39	43
44	051	082	112	157	233	308	384	460	610	762	911	1 21	1 51	2 26	3 01	3 77	4 52	5 27	44
45	050	079	109	154	228	301	374	448	597	744	890	1 19	1 48	2 21	2 95	3 69	4 42	5 15	45
46	049	078	107	151	223	295	366	439	585	729	871	1 16	1 45	2 17	2 88	3 60	4 32	5 04	46
47	048	077	105	148	218	289	360	430	573	713	853	1 13	1 42	2 12	2 82	3 53	4 23	4 93	47
48	048	075	103	144	214	283	354	421	561	698	835	1 11	1 39	2 08	2 76	3 45	4 14	4 83	48
49	047	074	101	142	209	277	344	411	549	683	820	1 09	1 36	2 03	2 71	3 38	4 06	4 73	49
50	046	073	099	139	205	272	338	405	536	671	802	1 07	1 33	1 99	2 65	3 32	3 98	4 64	50
51	045	071	097	136	201	267	332	396	527	658	786	1 05	1 30	1 95	2 60	3 25	3 90	4 55	51
52	045	070	095	134	196	262	326	390	518	646	771	1 03	1 28	1 92	2 55	3 19	3 83	4 46	52
53	044	069	094	131	194	257	320	381	509	634	759	1 01	1 26	1 88	2 51	3 13	3 75	4 38	53
54	043	068	092	129	191	252	314	375	497	622	744	986	1 23	1 85	2 46	3 07	3 69	4 29	54
55	042	066	091	127	187	248	308	369	491	610	732	972	1 21	1 81	2 41	3 01	3 62	4 22	55
56	042	066	089	125	184	244	303	363	482	601	716	954	1 19	1 78	2 37	2 96	3 55	4 14	56
57	041	064	088	123	181	239	297	357	472	588	704	939	1 17	1 75	2 33	2 91	3 49	4 07	57
58	041	063	086	121	178	235	293	351	463	579	692	921	1 15	1 72	2 29	2 86	3 43	4 00	58
59	040	062	085	119	175	231	288	344	457	570	683	905	1 13	1 69	2 25	2 81	3 37	3 93	59
60	039	062	084	117	172	228	283	338	448	561	671	890	1 11	1 66	2 21	2 76	3 32	3 87	60
61	038	061	082	115	169	224	278	332	442	552	658	878	1 09	1 64	2 18	2 72	3 26	3 80	61
62	038	060	081	113	167	220	274	326	436	543	649	863	1 08	1 61	2 14	2 68	3 21	3 74	62
63	038	059	080	112	164	217	270	323	427	533	640	850	1 06	1 59	2 11	2 63	3 16	3 69	63
64	037	058	079	110	162	214	266	317	421	524	628	835	1 04	1 56	2 08	2 59	3 11	3 63	64
65	037	057	078	108	159	211	262	314	415	518	619	823	1 03	1 54	2 05	2 55	3 08	3 57	65
66	036	056	076	107	157	208	258	308	408	509	610	811	1 01	1 51	2 01	2 51	3 01	3 52	66
67	036	056	076	105	155	205	254	304	402	503	601	799	999	1 49	1 98	2 48	2 97	3 47	67
68	035	055	074	104	153	202	250	299	396	494	591	786	982	1 47	1 95	2 44	2 93	3 41	68
69	035	054	074	102	151	199	247	295	390	488	585	777	966	1 45	1 93	2 40	2 88	3 36	69
70	034	054	073	101	148	196	244	291	387	482	578	765	954	1 43	1 90	2 37	2 84	3 32	70

EQUATION — $\frac{REV}{TIME}$ × 2 180 + 020 × 3048 = MPS

LESS THAN 40 REVS

MORE THAN 40 REVS

$\frac{REV}{TIME}$ × 2 170 + 030 × 3048

RATINGS LIMITS · 078 TO 2 44 MPS

29. Appendix N: Hach 2100P Turbidimeter Manual

Double-click picture to open pdf file in Adobe Acrobat Reader.



CAT. NO. 46500-88

PORTABLE TURBIDIMETER

Model 2100P

Instrument and Procedure Manual

© Hach Company, 1991-2004.
All rights reserved. Printed in the U.S.A.

te/dk 3/04 8ed

30. Appendix O: NCPN Water Quality Equipment and Supplies

Updated 8/7/06

30.1. Equipment

Task	Item	Number Needed	Number on Hand	Need a Backup?	Additional Needed	Likely Vender
Field Access	Vehicle	1	1	No		GSA until November 2006
	Vehicle Safety First Aid Shovel Road Hazard Tool Kit	1 set	1	No		
Field Safety	Radio $3,400	1	1	No	0	On loan from BRCA fire (Bruce Fields)
	Waders Hip Boots Personnel Flotation Device Gloves Digital Camera	1 1 1 1 1	1 1 1 1 1	No “ “ “ “	1@ ~ $90 1@ ~ $70 1@ ~ $100 1@ ~ $30 1@ ~ $300	
Flow Measurement	Aquacalc Pro Current and Flow Calculator With Adaptor, Rod Mount, Carrying case	1	1	No, rely on Battery with headset as backup.	0	
	Wading Rod	1	1	No	1	Collapsing rod for hiking
	Pigmy Meter	1	1	Consider Price AA as backup	0	

Task	Item	Number Needed	Number on Hand	Need a Backup?	Additional Needed	Likely Vender
	Magnetic pickup head for Pigmy	1	0	No	1	Rickly Hydrologic 101-015 Kit, Conversion, PMH $210.00 Presently on-hold, not urgent
	Price AA Meter	1	1 from ZION	Consider Pigmy as Backup	0	
	Magnetic pickup head for Price	1	0	No	1	Rickly Hydrologic 101-004 Kit, Conversion, AA MH $167.00 Presently on-hold, not urgent
	Stopwatch	1	1	No	0	
	50 Meter Tape	1	1	No	0	
	Plastic Squeeze Clamps to Anchor Tape	2	2	No	0	
	Portable Flume	1 from ZION	1	No	0	
	Weir Plates Ruler (for measuring depth of water surface in weir notch. this should probably be metal. A measuring device may be provide with weir.)	1	1	No	0	
	Staff Gage	1	0	no	None at present	Rickly Hydrologic Style C are $28.00 ea. Two per site will cover water depths up to 6.7 feet. See cat no. 801-050 & 801-051.
Lab	CYLINDER BEAKER		1-100ml 3-10ml 6	No	0	
	Carboy for DI Water		4-9 liter 2-20 liter	No	No	

Task	Item	Number Needed	Number on Hand	Need a Backup?	Additional Needed	Likely Vender
	Need source of DI Water. We were going to get it from Glen Canyon NRA, but they are switching to buying commercially prepared DI water.	5 gal DI water	1	0	0	Order from fisher scientific
Field Measurement	HACH Turbidimeter, with standards	1	1	No	0	
	MP Troll 9000E, 2 DR Version, Base Unit pH Sensor (Uninstalled), for TROLL 9000 Dissolved Oxygen Sensor (Uninstalled) Conductivity Sensor, Low Range (Uninstalled) Rugged reader System 10' T9K TROLL Quick Connect Cable & ACC, Elastomer, SSP Pelican Hard Shell Carrying Case	1 2 2 1 1 1 1	2 1 1 1 2 1 1	No	 0 0 0 0 0	GSA 0032000 pH Sensor (Uninstalled), for TROLL 9000, $133.82 0032870 Dissolved Oxygen Sensor (Uninstalled) $301.10 0033210 Conductivity Sensor, Low Range (Uninstalled) $167.28 0021450-01-01 10' T9K TROLL Quick Connect Cable, $154.56 0026470 Pelican Hard Shell Carrying Case, $295 This will provide a fully equipped backup unit.
Sample Collection						
Sample Processing	Geopump with easy load head	1	1	no	0	Pump on loan from Zion
	Masterflex® C-FLEX® tubing, L/S® 15, 25 ft	1 foot per sample (60'/year)	50 ft	no	0	Cole-Parmer Instrument Company
Sample Shipment	Coolers	2	2	no	0	Local

Task	Item	Number Needed	Number on Hand	Need a Backup?	Additional Needed	Likely Vender
Office						

30.2. Supplies

(for SW Utah Parks figure 60samples/year. This is based on 7 sample sites x 12 months, but some sites not accessible year-round)

Task	Item	Number Needed per Sample	Number Needed per Year	Number on Hand	Additional and Cost	Likely Vender
Field Access	Radio Battery			0	1	August 10 order to Dale Sorrels in CANY (435-719-2323)
Flow Measurement	None					
Field Measurement	For TROLL DO Membrane for short-flat sensor MP TROLL 9000 Maintenance Kit Comprehensive 1-mil DO Sensor Service Kit Dissolved Oxygen Calibration Kit		 1 1 1	 1 1 1	 0 0 0 0 0	
	Field Notebooks		3	3	0	
	Instrument Log Notebooks			3-6	0	
	Pocket thermometer		1	1	0	
	For Turbidimeter Sample Cells		1 pk of 6		1 pk of 6	HACH Cat. No. 24347-06 Turbidimeter Sample Cell, $20.00
Sample Collection	Sample bottles	Supplied by Utah Lab			0	
	DI-Water	Small quantity	10 gal	5 gal	1	89.00 Fisher Sci
	BUFFER SOLUTION, PH 4.01 BUFFER SOLUTION, PH 10.00 BUFFER SOLUTION, PH 7.00	100 ml/sampling day (~50/yr)	1.5 gal “ “	2+ 2+ 2+	1 1 1	Purchase as needed

Task	Item	Number Needed per Sample	Number Needed per Year	Number on Hand	Additional and Cost	Likely Vender
	Conductivity standard 180µS/cm Conductivity standard 1000µS/cm	100ml/sampling day (~50/yr)	1.5 gal “	0 0		HACH sells these for ~$10/100ml. Daigger in ~$16/16 oz. VWR ~$20/pint YSI (in VWR catalog) $64/qt VWR or GSA
	N-DEX NITRILE GLOVES,	100 large 100 med			0	
Sample Processing	FILTER ELEMENT	1	60	30 pk of 10 = 300	0	None needed now, but this will be a considerable annual cost.
Sample Shipment	Shipping Tape				None needed now	Local
	Shipping				$11.00 per cooler	UPS from BRCA to Salt Lake over night
General Supplies	Zip-Lock Bags Markers Aluminum Foil					Local
Office						

31. Appendix P: Equipment Inventory and Ownership

Water Quality Property on loan from ZION to NCPN Hydrologist in BRCA

August 12, 2005

Item	NPS Prop. #	Quantity	Serial #	Condition	Price	Purchase Year	Location
Baski Portable 18” long Cutthroat Flume 8” and 1” throat widths	none	1	none	Good	$600	1998	BRCA
Price Type AA Current Meter, Model 1210, Scientific Instruments Inc., w/earphones, case, tools	none	1	S00890	New	$800	1998	BRCA
Mini Current Meter, Model 6205	none	1	None	New	$800	2002	BRCA
Rickley Hydrological Co., w/ case, tools Turbidimeter, HACH 2100P (Catalog No. 46500-00) w/ sample cells and standards & case	none	1	4000024884	Good	$775	2000	BRCA
Peristolic Pump, Solinst, 12V	none	1	1518	Good	$700	2002	BRCA

32. Appendix Q: Troll 9000 Dissolved Oxygen (amperiometric) service instructions

Double-click picture to open pdf file in Adobe Acrobat Reader.



33. Appendix R: USGS Memo on Turbidity Units

In Reply Refer To: September 24, 2004
Mail Stop 412

Office of Water Quality Technical Memorandum 2004.03

Subject: Revision of NFM Chapter 6, Section 6.7—Turbidity

Purpose of Memorandum

This memorandum announces a rewrite of "Chapter 6, Section 6.7—Turbidity" of the U.S. Geological Survey (USGS) National Field Manual for the Collection of Water-Quality Data (NFM), and the implementation of new reporting units and parameter codes for storing and reporting turbidity data in USGS databases and products (Attachments 1 and 2). Information sources supporting these changes are included. This method will be effective October 1, 2004. The revised NFM Section 6.7 is available online at http://water.usgs.gov/owq/FieldManual/Chapter6/6.7_contents.html.

Problem

The NFM documents USGS protocols for the collection, quality assurance, storage, and publication of water-quality data. NFM Section 6.7—"Turbidity" documents methods for equipment selection, calibration, measurement, and data storage. Use of the revised method will help improve the quality and comparability of reported data, and will reduce the variability associated with such data.

Turbidity is a useful measurement that is growing in popularity and importance in scientific and resource monitoring programs, both within and outside of the USGS. Uses of turbidity data include measuring water clarity for drinking water and ecological applications, indicating visual impairment in water, real-time monitoring of conditions in watersheds, use as a surrogate for suspended-sediment concentration (SSC) and other constituents in water, and estimating transport of contaminants associated with suspended materials (Gray and Glysson, 2003).

However, turbidity itself is not an inherent physical property of water (as is, for example, temperature), but rather is a measure of light scattering through a liquid as measured by detectors with known geometry. The configuration of detectors and the source of light are important factors in the response of the turbidity instrument. Although comparisons among instruments with differing designs are often robust, they can also vary according to the character of the sample's matrix and particulates. Results from an interagency workshop held in 2002 demonstrated that turbidity data from different sources and instrumentation can be highly variable and are often in disagreement with each other, even when calibration methods are similar (Gray and Glysson, 2003). In effect,

instruments with different detector geometries and light sources often do not make equivalent measurements.

The U.S. Environmental Protection Agency (USEPA) method 180.1 established specific requirements for instrument configuration for the measurement of turbidity in drinking water within the United States (U.S. Environmental Protection Agency, 1993). The International Organization for Standardization (ISO) method 7027 established instrument requirements for measurement of drinking water in Europe (International Organization for Standardization, 1999), but uses a different light source than USEPA Method 180.1. Measurements complying with USEPA Method 180.1 are reported in Nephelometric Turbidity Units (NTU), whereas those complying with ISO 7027 are reported in Formazin Nephelometric Units (FNU) in Europe. In practice, during the past two decades most turbidity data in the U.S. have been reported in NTUs, regardless of their light source or detector geometry. Data stored in the USGS's National Water Information System (NWIS) and in USEPA's STORET database have been stored under parameter code 00076, and have been reported in NTUs; however, use of that parameter code has not been restricted to instruments that comply with USEPA Method 180.1.

Resolution

In order to reduce the variability associated with turbidity data, the USGS has created new reporting units for turbidity that are based on the instrument design (Attachment 1). These reporting units correspond to new parameter and method codes for storing turbidity data in the National Water Information System (NWIS). The use of NTU and FNU will be retained, but will be restricted to data from instruments that comply with the specific designs defined in USEPA Method 180.1 and ISO 7027, respectively. Eight new reporting units have been promulgated that correspond to existing and anticipated instrument designs that do not strictly conform to USEPA Method 180.1 or ISO 7027. Finally, the protocol discontinues the use within USGS of Formazin Turbidity Units (FTU), a reporting unit that has been commonly used, but which lacks specificity to any particular measurement technology.

Along with the new reporting units as specified in Attachment 1, users are now required to record the specific instrument make and model using the method code field in NWIS. Table A-1 in NFM Section 6.7 (Attachment 2) provides a current (August 2004) list of each instrument make and model according to its detector configuration and light source as specified for each new reporting unit and shows the appropriate parameter and method codes for each. Method codes are required when storing data in NWIS in order to provide traceability and allow for more informed comparisons of data in the future.

As the USGS is changing its reporting units for turbidity, other organizations are making the same changes. The American Society for Testing and Materials (ASTM) is in the process of making these changes and will use the same new reporting units. The USGS and ASTM have been working together in order to ensure traceability and improve comparability among turbidimeters.

The revised protocol also clarifies the use of appropriate calibration standards, in accordance with ASTM provisions. Although "scratch" formazin is recognized as the primary calibration standard, commercially-prepared solutions of stabilized formazin and styrene divinylbenzene polymer beads are also accepted as calibrants. However, consistent use of one type of calibrant is required to minimize errors introduced during calibration.

The revised turbidity protocol in Section 6.7 provides additional tools for USGS users. A decision tree will help the user determine the most appropriate instrument type for a given application. An appendix lists specific turbidity instruments, including manufacturer and model, so that users can look up the appropriate NWIS parameter code and method code for data entry.

Implementation and Data Handling

The revised turbidity protocol, including the use of new parameter codes and method codes, will be implemented on October 1, 2004. Concurrently with implementation of the new parameter codes, parameter code 00076 is no longer to be used for storing new data. This code will eventually be locked so that it will not accept new data. Historical data, stored previously under NWIS parameter code 00076, may be migrated to the new parameter codes on a case by case basis, only where knowledge of specific instruments and their instrument configuration is sufficient to allow selection of an appropriate reporting unit, parameter code, and method code according to Table A-1. These changes should be done with the guidance of the District Water-Quality Specialist. Attachment 3 provides information on a program that is available to aid in migrating historical data to the new parameter and method codes.

Training on the new data reporting and storage requirements will take place through cyber seminars initially to be held on September 22 and 28, 2004. Information will also be provided in the Field Water-Quality Methods for Ground Water and Surface Water training course (QW1028TC) and the NFM.

NWISWeb (real-time)

Currently, NWISWeb is incapable of displaying or retrieving method codes with real-time continuous data. Efforts are underway through the USGS Surface Water Users Group (SWUG) to add database functionality so that method codes would be displayed along with real-time data. As an interim measure, station descriptions for sites that serve continuous turbidity data to the web must be updated to include a description of the turbidity instrumentation used at the site, along with the appropriate method code.

Publications

Districts may need documentation or references in report products to cooperators to support the use of the new reporting units. Some appropriate documents are listed in the 'Reference' section of this memo and in NFM Section 6.7. All products should use the

new reporting limits and list the instruments used for the study. Guidelines for rounding data for reports are provided in NFM Section 6.7 (Table 6.7-6).

Future modifications and maintenance

New parameter and method codes will be added as the technology for measuring turbidity evolves. These will be included in, and announced with, the quarterly NWIS updates and in the "Comments and Errata" page of the online NFM.

Sources of information

Approved Methods

NFM Section 6.7 (Anderson, Chauncey W., 2004)

ASTM – Low-Level Static Method (ASTM International, 2003)

ISO 7027 (International Organization for Standardization, 1999)

Guidelines and Standard Procedures for Continuous Water-Quality Monitors: Site Selection, Field Operation, Calibration, Record Computation, and Reporting, Water-Resources Investigations Report 00-4252 (Wagner and others, 2000)

USEPA 180.1 (U.S. Environmental Protection Agency 1993)

References

Anderson, C.W., 2004, Turbidity, (version 2): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A6, section 6.7, accessed September 24, 2004 from http://water.usgs.gov/owq/FieldManual/Chapter6/6.7_contents.html.

ASTM International, 2003, D6855-03 Standard test method for determination of turbidity below 5 NTU in static mode: ASTM International, Annual Book of Standards, Water and Environmental Technology, 2003, vol. 11.01, West Conshohocken, Pennsylvania, 13 p. [http://www.astm.org/cgibin/SoftCart.exe/DATABASE.CART/REDLINE_PAGES/D6855.htm?L+mystore+flap7535+1094151215]

Davies-Colley, R.J., and Smith, D.G., 2001, Turbidity, suspended sediment, and water clarity— A review: Journal of the American Water Resources Association, v. 37, no. 5, p. 1085–1101.

Gray, J.R., and Glysson, G.D., 2003, Proceedings of the Federal Interagency Workshop on turbidity and other sediment surrogates, April 30-May 2, 2002, Reno, Nevada: U.S. Geological Survey, Circular 1250, 56 p. [http://pubs.water.usgs.gov/circ1250]

International Organization for Standardization, 1999, Water quality — Determination of turbidity: Geneva, Switzerland, International Organization for Standardization, ISO 7027, 10 p.

U.S. Environmental Protection Agency 1993, Methods for the determination of inorganic substances in environmental samples: Cincinnati, OH, U.S. Environmental Protection Agency EPA/600/R-93/100, 178 p.

Wagner, R.J, Mattraw, H.C., Ritz, G.F., and Smith, B.A., 2000, Guidelines and Standard Procedures for Continuous Water-Quality Monitors: Site Selection, Field Operation, Calibration, Record Computation, and Reporting, U.S. Geological Survey Water-Resources Investigations Report 00-4252, 53 p. [http://pubs.usgs.gov/wri/wri004252/]

Timothy L. Miller /s/
Chief, Office of Water Quality

This memorandum does not supersede any other Office of Water Quality Technical Memorandum.

Distribution: All WRD Employees

Attachments (in attached file)

34. Appendix S: USGS National Field Manual Section 6.7 – Turbidity

TURBIDITY 6.7

By Chauncey W. Anderson

	Page
Turbidity	TBY-3
6.7.1 Equipment	5
6.7.1.A Interferences and instrument design	5
6.7.1.B Data storage	9
6.7.1.C Instrument selection and maintenance	11
Decision considerations for instrument selection	13
Signal-processing options	18
Maintenance of turbidity instruments	18
6.7.2 Calibration	21
6.7.2.A Calibration solution: use, preparation, and dilution	24
6.7.2.B Calibration procedures	26
Benchtop (static) turbidimeter calibration	27
Submersible (dynamic) turbidity sensor calibration	29
Spectrophotometric turbidimeter calibration	32
6.7.3 Measurement	33
6.7.3.A Static (benchtop) determination	34
6.7.3.B Dynamic (submersible-sensor) determination	42
6.7.3.C Spectrophotometric determination	45

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #6

Cumulative bias control

Version 1.00 (4/10/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

Table of Contents

1. Introduction 3
1.1. Observer 3
1.2. Instruments 3
1.3. Methods and indicators 3
2. Guidelines 3
2.1. Duration of measurement overlap 4
2.1.1. Personnel 4
2.1.2. Instrument 4
2.1.3. Method or indicator 4
3. Record of Bias 4
3.1. Precision 5
3.1.1. Standard deviation 5
3.1.2. Coefficient of variation, or RSD 5
3.1.3. Standard error 5
3.2. Sensitivity 5
3.3. Bias 6
3.3.1. Percent difference 6
3.3.2. Confidence interval around mean PD 6
3.4. Significance test 6
4. Interpretation 6
4.1. Precision 7
4.2. Sensitivity (MPEU) 7
4.3. Bias 7
4.4. Significance test (for $n > 30$) 7
4.5. Considerations 8
4.6. Making adjustments 9
4.7. Documentation 9
5. References 9
6. Appendix A: Standard Deviation 11
7. Appendix B: Standard error 13
8. Appendix C: Slope 15
9. Appendix D: Confidence 17
10. Appendix E: T-test 19
11. Appendix F: Record_of_Bias.xls spreadsheet 21

1. Introduction

This is a long term monitoring program and as such it is expected that staff, equipment and even indicators and measured parameters will change over time. It is extremely important to quantify the effect that change in these factors may have on data so to ensure that data comparability proceeds through the life of the monitoring program and beyond. This SOP outlines the major types of change, and how changes can be mitigated or reported to ensure change detection is due to environmental conditions rather than observer, sensor or methodology.

It should be noted that data adjustments are never made before reporting values to long term storage databases. Adjustments are only made on data when doing long-term trend analysis. The utility of tracking cumulative bias change is to try to make it easier for future data users to get all the data into comparable units for long term trend analysis.

1.1. Observer

Observer variability needs to be controlled because although most of the water quality measurements are made with machines or instruments, there is the potential for bias in selecting where measurements are made. The exact location of a sampling site is somewhat subjective (SOP 4) as is the location where flow and core parameters are measured and where sample bottles are dipped.

1.2. Instruments

Changing instruments and sensors over time introduces the potential for bias. For instance core parameter sondes may have different algorithms for temperature compensation. Thus even if calibrated at room temperature results may vary in cold field conditions.

1.3. Methods and indicators

Changes in methods and indicators may be necessary as new techniques are developed that prove to be superior to older techniques. Rather than stick stubbornly to older techniques this monitoring program should evolve and adopt new methods but have a clear understanding of how results from older methods can be used with new methods. This requires carefully tracking changes due to methods or indicators so that future data users will not misinterpret a change in trend.

2. Guidelines

This SOP is a guide based on understanding the types of change that are likely to occur. It is based in “best case scenario” circumstances recognizing that not all of the conditions outlined below can be met. For instance a basic premise when instruments change is that both the old and new instruments are available to make simultaneous measurements to evaluate bias. As has already happened in the first year of monitoring a core parameter sonde failed completely making a side by side comparison with a new sonde impossible. In this case other aspects of quality assurance will be the only recourse for proving data

are temporally comparable. In this case the calibration records and instrument log books provide some assurance of consistency between sensors.

2.1. Duration of measurement overlap

The number of paried measurements necessary to understand bias and develop good ways to correct it will vary depending on the type of measurement, its variability and the variability of the environment in which the measurements are being made. Generally, the bigger the change, the more measurements will be necessary to demonstrate how the change might affect the data and its interpretation. The following numbers of paired measurements are a minimum, but more is always better.

2.1.1. Personnel

When the only change is in personnel a minimum of 7 measurements will be made on the same object or sample by each observer. Flow is the field measurement most likely to exhibit significant user deviation. This is due to user variability in how the instrument is held and where it is placed during the 20 + readings needed for each cross section, or how well the flume or weir is sealed against leaks. Take readings to ensure the highest possible degree of repeatability by making measurements close in space and time.

2.1.2. Instrument

When the only change is in instrument or sensor a minimum of 30 measurements will be made on the same object or sample with each instrument. Sonde measurements should be made in containerized sample to avoid natural variation. DO measurements should be made in an agitated sample to avoid oxygen consumption at the sensor tip if using a ploarographic type sensor. The same instrument and calibration constants should be used by both observers to limit instrument variability.

2.1.3. Method or indicator

When a change in method or indicator occurs at least 50 paired measurements will be made. More measurements are necessary when indicators change because very few indicators respond in exactly the same way to environmental stimuli. The more complex the relationship between the measured value and what it is intended to represent the more measurements need to be made to ensure data from the two indicators or methods can be compared through time.

3. Record of Bias

To aid evaluation of potential changes an Excel spreadsheet (Record_of_Bias.xls Appendix F) has been constructed that determines measurement precision, sensitivity and bias from multiple measurements of the same parameter. It is designed to help users determine when a difference is significant and how to correct it. The data recorded in the spreadsheet will be a part of the permanent meta data record and can be used in other more sophisticated applications if warranted. Each time a new test is performed to evaluate bias the Record_of_Bias.xls spreadsheet should be saved as a new file with a date extension added to the name (eg Record_of_Bias_1-26-07.xls). Following are

explanations of the metrics and methods used to evaluate results computed by entering readings in the Record_of_bias.xls spreadsheet.

3.1. Precision

Precision refers to how closely individual measurements agree with one another. For one observer it measures how tightly repeated measures cluster. Ideally repeated measures made by the same observer on the same medium will be very similar.

3.1.1. Standard deviation

Standard deviation will be used to assess precision. It is the average amount by which estimates in a distribution differ from the mean (not to be confused with the correct value) of measured values, ignoring the sign of the difference. Note that units of standard deviation are the same as the units of measure. It does not compare one observer versus the other. A formal definition including calculation of standard deviation is provided in Appendix A.

3.1.2. Coefficient of variation, or RSD

Normalizes the standard deviation by the mean. It is a unitless value that can be thought of as each observer's percentage difference from their mean. It does not compare one observer versus the other. Coefficient of variation is,

CV = stdev/mean.

Relative Standard Deviation (RSD) which provides a more intuitive comparison is CV*100

3.1.3. Standard error

Is the average amount of error in the prediction of y for an individual x, or the average deviation around the best fit line in a regression context. Whereas standard deviation is the spread of each individual observer's results, standard error is the spread around the 'prediction'. In bias correction, standard error indicates the average error in prediction based on variability of observations. Standard error is defined in Appendix B.

3.2. Sensitivity

We measure sensitivity via an estimate of measurement precision expanded uncertainty (MPEU). See section 7.1.6.3 in SOP 7 for more information. Measurement sensitivity will be evaluated by analysis of repeated measures of a normal environmental sample. This is essentially an estimate of precision but in a more rigorous sense based on a confidence interval. It differs from AMS+ (section 7.1.5.3) in that MPEU samples are made in a bucket, while AMS+ measurements are made insitu.

MPEU is calculated as,

$MPEU = t * sd_n$

where t = the 99% confidence middle t value for sample size n,

sd_n = standard deviation of n replicate samples on a normal environmental sample.

3.3. Bias

Bias (slope) is the rise over run for the best fit regression line through the data pairs. Offset is the y-intercept for the equation that describes the best fit regression line. Bias and slope are used to correct a known and persistent error that cannot be corrected by changing sensors or re-calibrating. Slope is defined in Appendix C.

3.3.1. Percent difference

Percent difference is a measure of how different two measurements are expressed relative to the first measurement.

$PD = ((q_2-q_1)/q_1)*100$,

where, q_1 = observer 1 measurement value, and

q_2 = observer 2 measurement value

The sign of the average PD across many measurement pairs indicates the direction of bias.

3.3.2. Confidence interval around mean PD

Confidence interval is interpreted as the range in values in which future measurements are likely to occur. Construct a 95% confidence interval around the mean for the average bias change expressed as a percent difference (PD). Confidence interval is explained in Appendix D.

3.4. Significance test

A significance test helps determine if a new observer, sensor, or method causes a change in measured values that are not caused by ‘true’ change in the parameter of interest.

Once the slope and intercept are determined a statistical t-test is performed to determine if the two sets of measurements (eg. those of sensor 1 vs. sensor 2) came from the same population with the same mean. T-test is defined in Appendix D.

4. Interpretation

After the measurements have been made and the metrics calculated they need to be interpreted for the presence of bias and action must be taken to correct bias if found. In all cases the first choice is to eliminate bias by trying to determine what caused the change then fixing it if possible. This may involve changing sensors, methods or adjusting techniques.

If no solution exists for eliminating bias between old and new methods and the data meet the measurement quality objectives outlined in SOP 7 section 7 the biased measurements must be reported without adjustment. It will be the responsibility of the present

hydrologist (you) to ensure a record of this bias is available for future data users so they can make corrections before doing long-term trend analysis.

4.1. Precision

Precision needs to be controlled in order to maintain confidence in the interpretation of bias calculations. If precision is low as evidenced by large standard deviation, coefficient of variation and standard error more measurements are necessary to 'see' a bias and have a high degree of confidence in the interpretation of slope and t-test results. If precision is low increase sample size or determine the source of variability and eliminate it before proceeding to evaluate other calculations.

4.2. Sensitivity (MPEU)

This value gives an indication of how different two values can be that is a true difference rather than a difference due to measurement process noise (Irwin 2007). In other words how strong does the signal have to be for our measurements to detect a true difference?

When changing instruments or sensors the sensitivity should be maintained in the same range, or improved. Sensitivity is analogous to resolution in that it is the finest amount of stimulus that induces a response in the sensor. There is no meaningful way to estimate sensitivity of human observers in the context of monitoring. It is more meaningful in reference to machine readings and is a value typically provided by the instrument or sensor manufacturer. This value should be compared to the calculated value of MPEU. Ideally they will be in the same order of magnitude, but the value reported by the manufacturer will likely be lower.

4.3. Bias

A persistent or average change in one direction constitutes a bias that is evaluated by the slope of the regression line, the average percent difference and the confidence interval around the average percent difference. If the confidence interval around the average percent difference includes 1 it is unlikely that measurements from the two different sensors, observers or methods are significantly different. To answer that question with a higher level of confidence drop the alpha level from 0.05 to 0.01. If the confidence interval still includes 1 then it is probably safe to say no bias is present and thus no correction needs to be made, but be sure to evaluate all of the other metrics to further increase confidence via convergence of evidence.

4.4. Significance test (for $n > 30$)

In Excel TTEST is used to determine whether two samples are likely to have come from the same two underlying populations that have the same mean. If the true means are the same their difference = zero. So a hypothesis test can be written two ways.

Ho: $U_1 - U_2 = 0$

Ha: $U_1 - U_2 <> 0$

or

Ho: $U_1 = U_2$

Ha: U_1 not = U_2

One way to evaluate the comparability of two instruments would be to test them side-by-side under varying conditions in the field. If testing a sensor or sonde be sure to take readings from a bucket to eliminate natural variability or poor mixing as factors. Such a comparison is called a *paired* t-test. In Excel run the t-test by clicking tools, data analysis and select t-Test: Paired Two Sample for Means

Another more controlled method is to collect many data points using each instrument in the same water sample. This method will help account for instrument sensitivity and instrument variability. This is called a *two-sample* t-test. In the Record_of_bias.xls spreadsheet the t-test is defined as a two tailed, paired test. In Excel parlance tails = 2 and type = 1.

Regardless of test method chosen, the output is interpreted the same way. Determine if the t-test result (this is not the Student's t value but rather the area under the curve corresponding to a p-value) is greater than the chosen level of significance, usually α = 0.05 or less. If the t-test value is greater than the significance level then accept the null hypothesis that the means are equal. If the t-test value is less than the level of significance then reject the null hypothesis that the means are equal.

If the t-test result is significant (that is, we reject the null that the means are equal) then the source of the problem causing the means to be unequal should be identified. If the source cannot be identified and corrected then mathematical adjustment of observer 2 results are justified. This is only plausible when there is no other way to determine or correct for the difference in observed values. Use the equation of the regression line to adjust observer 2 results to be comparable with observer 1 results.

4.5. Considerations

Before making adjustments to raw data a careful evaluation of the statistical results needs to be made in light of how the test was conducted. Did the test consist of multiple replicated paired measurements in a bucket from one stream, or was it set up as paired measurements from a bucket at multiple streams? The statistics will be interpreted differently depending on how the test was conducted. For instance in the first case, the standard deviation, coefficient of variation and confidence intervals should all be small values. In the second case these values will be much higher due to natural variability across the different streams.

The offset, bias, slope, t-test, bias, percent difference and sample size calculations are more meaningful when the test includes multiple streams. This type of test is more meaningful because it provides information about how the two observers, instruments or techniques compare across the expected range in field conditions.

Insufficient sample sizes result in data that are difficult to interpret. A way to ensure sufficient sample size is to conduct paired evaluations until the 95% confidence value

(expressed as a percent difference) drops below 20% of either the old or new mean. In other words, if the 95% confidence value (1/2 the CI range) is greater than 20% of either the old or new mean then the number of paired measurements should be increased after all other sources of variation have been controlled.

In all cases it is wise to carefully evaluate statistics in light of the way the data were collected, the sample size, standard deviation and above all common sense. This type of data analysis should be exploratory and these and other statistical tests can be performed, but before adjustments are made there should be a convergence of evidence where several or all of the statistics point toward the same conclusion.

4.6. Making adjustments

Note: Adjustments will NEVER be made to data uploaded and stored in long term storage data bases. Adjustments are only made during interpretation by future users who might be trying to make long term data temporally comparable for trend analysis.

After determining that the means are not equal it is possible to adjust new results to correspond to old results such that measurement effects are subtracted from the new results. After adjustment temporal differences should only result from natural effects.

1. In Excel plot new method results on X-axis and old method results on Y-axis.
2. Determine the equation for the regression line.
3. Right click on a data point and click add trend line.
4. Make type linear and under options select display equation and R-square value on chart.
5. Use the equation of the regression line to adjust new method values to correspond with old method. If X-axis was the old method the regression equation is already set up this way.

4.7. Documentation

If adjustments are made they need to be carefully documented. Each time a new test is performed to evaluate bias the Record_of_Bias.xls spreadsheet should be saved as a new file with a date extension added to the name (eg Record_of_Bias_1-26-07.xls). It is exceedingly important that the original raw data and ad hoc test data be retained so that in the future evaluations can be made using other techniques. For this reason NEVER alter data saved in long-term storage data bases if the data meet the measurement quality objectives (SOP 7 section 7). Records of adjustments used in data interpretation should be noted in the data management and data analysis SOP's 14 and 15 as well.

5. References

Irwin, R.J. 2007. Draft Part B lite (Just the Basics) QA/QC Review Checklist for Aquatic Vital Sign Monitoring Protocols and SOPs, National Park Service, Water Resources Division. Fort Collins, Colorado, distributed on Internet only at

http://www.nature.nps.gov/water/Vital_Signs_Guidance/Guidance_Documents/PartBLite.pdf

6. Appendix A: Standard Deviation

From the Excel 2003 help file

Estimates standard deviation based on a sample. The standard deviation is a measure of how widely values are dispersed from the average value (the mean).

Syntax

STDEV(**number1**,number2,...)

Number1, number2, ... are 1 to 30 number arguments corresponding to a sample of a population. You can also use a single array or a reference to an array instead of arguments separated by commas.

Remarks

STDEV assumes that its arguments are a sample of the population. If your data represents the entire population, then compute the standard deviation using STDEVP.

The standard deviation is calculated using the "n-1" method.

Arguments can either be numbers or names, arrays, or references that contain numbers.

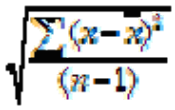
Logical values and text representations of numbers that you type directly into the list of arguments are counted.

If an argument is an array or reference, only numbers in that array or reference are counted. Empty cells, logical values, text, or error values in the array or reference are ignored.

Arguments that are error values or text that cannot be translated into numbers cause errors.

If you want to include logical values and text representations of numbers in a reference as part of the calculation, use the STDEVA function.

STDEV uses the following formula:



where x is the sample mean AVERAGE(number1,number2,...) and n is the sample size.

Example

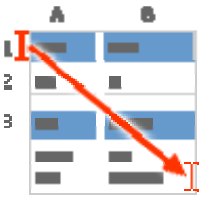
Suppose 10 tools stamped from the same machine during a production run are collected as a random sample and measured for breaking strength.

The example may be easier to understand if you copy it to a blank worksheet.

How to copy an example

1. Create a blank workbook or worksheet.
2. Select the example in the Help topic.

NOTE Do not select the row or column headers.



Selecting an example from Help

3. Press CTRL+C.
4. In the worksheet, select cell A1, and press CTRL+V.
5. To switch between viewing the results and viewing the formulas that return the results, press CTRL+` (grave accent), or on the **Formulas** tab, in the **Formula Auditing** group, click the **Show Formulas** button.

	A	
1	**Strength**	
2	1345	
3	1301	
4	1368	
5	1322	
6	1310	
7	1370	
8	1318	
9	1350	
10	1303	
11	1299	
	Formula	**Description (Result)**
	=STDEV(A2:A11)	Standard deviation of breaking strength (27.46391572)

7. Appendix B: Standard error

From the Excel 2003 help file

Returns the standard error of the predicted y-value for each x in the regression. The standard error is a measure of the amount of error in the prediction of y for an individual x.

Syntax

STEYX(**known_y's**,**known_x's**)

Known_y's is an array or range of dependent data points.

Known_x's is an array or range of independent data points.

Remarks

Arguments can either be numbers or names, arrays, or references that contain numbers.
Logical values and text representations of numbers that you type directly into the list of arguments are counted.
If an array or reference argument contains text, logical values, or empty cells, those values are ignored; however, cells with the value zero are included.
Arguments that are error values or text that cannot be translated into numbers cause errors.
If known_y's and known_x's have a different number of data points, STEYX returns the #N/A error value.
If known_y's and known_x's are empty or have less than three data points, STEYX returns the #DIV/0! error value.
The equation for the standard error of the predicted y is:

$$\sqrt{\frac{1}{(n-2)}\left[\sum(y-\bar{y})^2-\frac{\left[\sum(x-\bar{x})(y-\bar{y})\right]^2}{\sum(x-\bar{x})^2}\right]}$$

where x and y are the sample means AVERAGE(known_x's) and AVERAGE(known_y's), and n is the sample size.

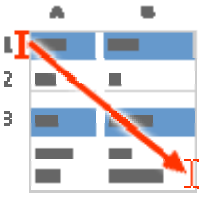
Example

The example may be easier to understand if you copy it to a blank worksheet.

How to copy an example

1. Create a blank workbook or worksheet.
2. Select the example in the Help topic.

   Note Do not select the row or column headers.



   Selecting an example from Help

3. Press CTRL+C.
4. In the worksheet, select cell A1, and press CTRL+V.
5. To switch between viewing the results and viewing the formulas that return the results, press CTRL+` (grave accent), or on the **Tools** menu, point to **Formula Auditing**, and then click **Formula Auditing Mode**.

	A	B
	Known y	**Known x**
1	2	6
2	3	5
3	9	11
4	1	7
5	8	5
6	7	4
7	5	4

8	**Formula**	**Description (Result)**
	=STEYX(A2:A8,B2:B8)	Standard error of the predicted y-value for each x in the regression (3.305719)

8. Appendix C: Slope

Returns the slope of the linear regression line through data points in known_y's and known_x's. The slope is the vertical distance divided by the horizontal distance between any two points on the line, which is the rate of change along the regression line.

Syntax

SLOPE(**known_y's**,**known_x's**)

Known_y's is an array or cell range of numeric dependent data points.

Known_x's is the set of independent data points.

Remarks

The arguments must be either numbers or names, arrays, or references that contain numbers.
If an array or reference argument contains text, logical values, or empty cells, those values are ignored; however, cells with the value zero are included.
If known_y's and known_x's are empty or have a different number of data points, SLOPE returns the #N/A error value.
The equation for the slope of the regression line is:

$$b = \frac{\sum (x - \bar{x})(y - \bar{y})}{\sum (x - \bar{x})^2}$$

where x and y are the sample means AVERAGE(known_x's) and AVERAGE(known_y's).

The underlying algorithm used in the SLOPE and INTERCEPT functions is different than the underlying algorithm used in the LINEST function. The difference between these algorithms can lead to different results when data is undetermined and collinear. For example, if the data points of the known_y's argument are 0 and the data points of the known_x's argument are 1:

SLOPE and INTERCEPT return a #DIV/0! error. The SLOPE and INTERCEPT algorithm is designed to look for one and only one answer, and in this case there can be more than one answer.
LINEST returns a value of 0. The LINEST algorithm is designed to return reasonable results for collinear data, and in this case at least one answer can be found.

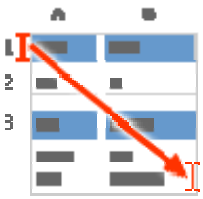
Example

The example may be easier to understand if you copy it to a blank worksheet.

How to copy an example

1. Create a blank workbook or worksheet.
2. Select the example in the Help topic.

   Note Do not select the row or column headers.



   Selecting an example from Help

3. Press CTRL+C.

4. In the worksheet, select cell A1, and press CTRL+V.
5. To switch between viewing the results and viewing the formulas that return the results, press CTRL+` (grave accent), or on the **Tools** menu, point to **Formula Auditing**, and then click **Formula Auditing Mode**.

	A	B
	Known y	**Known x**
1	2	6
2	3	5
3	9	11
4	1	7
5	8	5
6	7	4
7	5	4

	Formula	**Description (Result)**
8	=SLOPE(A2:A8,B2:B8)	Slope of the linear regression line through the data points above (0.305556)

9. Appendix D: Confidence

From the Excel 2003 help file

Returns a value that you can use to construct a confidence interval for a population mean. The confidence interval is a range of values. Your sample mean, x, is at the center of this range and the range is x ± CONFIDENCE. For example, if x is the sample mean of delivery times for products ordered through the mail, x ± CONFIDENCE is a range of population means. For any population mean, μ_0, in this range, the probability of obtaining a sample mean further from μ_0 than x is greater than alpha; for any population mean, μ_0, not in this range, the probability of obtaining a sample mean further from μ_0 than x is less than alpha. In other words, assume that we use x, standard_dev, and size to construct a two-tailed test at significance level alpha of the hypothesis that the population mean is μ_0. Then we will not reject that hypothesis if μ_0 is in the confidence interval and will reject that hypothesis if μ_0 is not in the confidence interval. The confidence interval does not allow us to infer that there is probability 1 – alpha that our next package will take a delivery time that is in the confidence interval.

Syntax

CONFIDENCE(**alpha**,**standard_dev**,**size**)

Alpha is the significance level used to compute the confidence level. The confidence level equals 100*(1 - alpha)%, or in other words, an alpha of 0.05 indicates a 95 percent confidence level.

Standard_dev is the population standard deviation for the data range and is assumed to be known.

Size is the sample size.

Remarks

If any argument is nonnumeric, CONFIDENCE returns the #VALUE! error value.
If alpha ≤ 0 or alpha ≥ 1, CONFIDENCE returns the #NUM! error value.
If standard_dev ≤ 0, CONFIDENCE returns the #NUM! error value.
If size is not an integer, it is truncated.
If size < 1, CONFIDENCE returns the #NUM! error value.
If we assume alpha equals 0.05, we need to calculate the area under the standard normal curve that equals (1 - alpha), or 95 percent. This value is ± 1.96. The confidence interval is therefore:

$$\bar{x} \pm 1.96\left(\frac{\sigma}{\sqrt{n}}\right)$$

Example

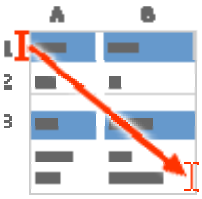
Suppose we observe that, in our sample of 50 commuters, the average length of travel to work is 30 minutes with a population standard deviation of 2.5. With alpha = .05, CONFIDENCE(.05, 2.5, 50) returns 0.692952. The corresponding confidence interval is then 30 ± 0.692952 = approximately [29.3, 30.7]. For any population mean, μ_0, in this interval, the probability of obtaining a sample mean further from μ_0 than 30 is more than 0.05. Likewise, for any population mean, μ_0, outside this interval, the probability of obtaining a sample mean further from μ_0 than 30 is less than 0.05.

The example may be easier to understand if you copy it to a blank worksheet.

How to copy an example

1. Create a blank workbook or worksheet.
2. Select the example in the Help topic.

Note Do not select the row or column headers.



Selecting an example from Help

3. Press CTRL+C.
4. In the worksheet, select cell A1, and press CTRL+V.
5. To switch between viewing the results and viewing the formulas that return the results, press CTRL+` (grave accent), or on the **Tools** menu, point to **Formula Auditing**, and then click **Formula Auditing Mode**.

	A	B
	Data	**Description**
1	0.05	Significance level
2	2.5	Standard deviation of the population
3	50	Sample size
4	**Formula**	**Description (Result)**
	=CONFIDENCE(A2,A3,A4)	Confidence interval for a population mean. In other words, the confidence interval for the underlying population mean for travel to work equals 30 ± 0.692952 minutes, or 29.3 to 30.7 minutes. (0.692952)

10. Appendix E: T-test

From the Excel 2003 help file

Returns the probability associated with a Student's t-Test. Use TTEST to determine whether two samples are likely to have come from the same two underlying populations that have the same mean.

Syntax

TTEST(**array1**,**array2**,**tails**,**type**)

Array1 is the first data set.

Array2 is the second data set.

Tails specifies the number of distribution tails. If tails = 1, TTEST uses the one-tailed distribution. If tails = 2, TTEST uses the two-tailed distribution.

Type is the kind of t-Test to perform.

If type equals	This test is performed
1	Paired
2	Two-sample equal variance (homoscedastic)
3	Two-sample unequal variance (heteroscedastic)

Remarks

If array1 and array2 have a different number of data points, and type = 1 (paired), TTEST returns the #N/A error value.
The tails and type arguments are truncated to integers.
If tails or type is nonnumeric, TTEST returns the #VALUE! error value.
If tails is any value other than 1 or 2, TTEST returns the #NUM! error value.
TTEST uses the data in array1 and array2 to compute a non-negative t-statistic. If tails=1, TTEST returns the probability of a higher value of the t-statistic under the assumption that array1 and array2 are samples from populations with the same mean. The value returned by TTEST when tails=2 is double that returned when tails=1 and corresponds to the probability of a higher absolute value of the t-statistic under the "same population means" assumption.

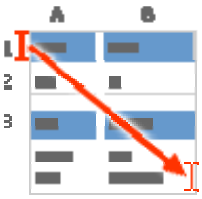
Example

The example may be easier to understand if you copy it to a blank worksheet.

How to copy an example

1. Create a blank workbook or worksheet.
2. Select the example in the Help topic.

Note Do not select the row or column headers.



Selecting an example from Help

3. Press CTRL+C.
4. In the worksheet, select cell A1, and press CTRL+V.
5. To switch between viewing the results and viewing the formulas that return the results, press CTRL+` (grave accent), or on the **Tools** menu, point to **Formula Auditing**, and then click **Formula Auditing Mode**.

	A	B
	Data 1	**Data 2**
1	3	6
2	4	19
3	5	3
4	8	2
5	9	14
6	1	4
7	2	5
8	4	17
9	5	1
10	**Formula**	**Description (Result)**
	=TTEST(A2:A10,B2:B10,2,1)	Probability associated with a Student's paired t-Test, with a two-tailed distribution (0.196016)

11. Appendix F: Record_of_Bias.xls spreadsheet

Double click on image to open in Excel

Record of Cumulative Bias Control — **Example worksheet - Bogus Data**

Change in Staff
When change in staff occurs 7 measurements of the following on the same sample will be recorded
Preferrably this should be done over time at different sampling sites. The sample should be containerized to avoid natural variability.
The goal is to replicate observer 1 results thus observer 2 is the independent variable (x) used to predict the dependent variable (y)

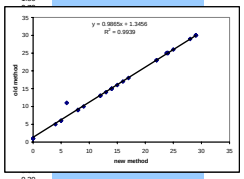
observation	observer 1 (old = y) pH	observer 2 (new = x) pH	percent difference	fraction of change old to new	observer 1 (old = y) specific conductance (us/cm)	observer 2 (new = x) specific conductance (us/cm)	percent difference	fraction of change old to new	observer 1 (old = y) temperature (deg C)	observer 2 (new = x) temperature (deg C)	percent difference	fraction of change old to new	observer 1 (old = y) dissolved oxygen (mg/L)	observer 2 (new = x) dissolved oxygen (mg/L)	percent difference	fraction of change old to new	observer 1 (old = y) flow (cfs)	observer 2 (new = x) flow (cfs)	percent difference	fraction of change old to new	date (mm/dd/yyyy)	time	location
1	7.10	7.09	-0.14	1.00	533.00	523.00	-1.88	1.02	32.00	35.20	10.00	0.91	8.30	6.30	-24.10	1.32	10.00	12.00	20.00	0.83			
2	7.13	7.13	0.00	1.00	534.00	524.00	-1.87	1.02	33.00	36.30	10.00	0.91	8.40	6.40	-23.81	1.31	8.00	10.00	25.00	0.80			
3	7.11	7.15	0.56	0.99	535.00	526.00	-1.68	1.02	34.00	37.40	10.00	0.91	8.50	6.50	-23.53	1.31	7.00	9.00	28.57	0.78			
4	7.12	7.12	0.00	1.00	536.00	526.00	-1.87	1.02	35.00	38.50	10.00	0.91	8.60	6.60	-23.26	1.30	12.00	14.00	16.67	0.86			
5	7.15	7.11	-0.56	1.01	537.00	527.00	-1.86	1.02	36.00	32.40	-10.00	1.11	8.70	6.70	-22.99	1.30	10.00	12.00	20.00	0.83			
6	7.09	7.09	0.00	1.00	538.00	528.00	-1.86	1.02	37.00	33.30	-10.00	1.11	8.80	6.80	-22.73	1.29	5.00	7.00	40.00	0.71			
7	7.17	7.10	-0.98	1.01	539.00	529.00	-1.86	1.02	38.00	24.20	-10.00	1.11	8.90	6.90	-22.47	1.29	2.00	4.00	100.00	0.50			
mean	7.12	7.11	-0.16	1.00	536.00	526.14	-1.84	1.02	35.00	35.33	1.43	1.00	8.60	6.60	-23.27	1.30	7.71	9.71	35.75	0.76			
std dev	0.03	0.02	0.49	0.00	2.16	2.12	0.07	0.00	2.16	2.21	10.69	0.11	0.22	0.22	0.58	0.01	3.40	3.40	29.36	0.12			
offset	6.61	******	******	******	7.06	******	******	******	51.90	******	******	******	2.00	******	******	******	-2.00	******	******	******			
bias (slope)	0.07	******	******	******	1.01	******	******	******	-0.48	******	******	******	1.00	******	******	******	1.00	******	******	******			
bias direction	******	******	**negative bias**	**-0.01**	******	******	**negative bias**	**-9.86**	******	******	**positive bias**	**0.33**	******	******	**negative bias**	**-2.00**	******	******	**positive bias**	**2.00**			
coeff of var.	0.00	0.00	******	******	0.00	0.00	******	******	0.06	0.06	******	******	0.03	0.03	******	******	0.44	0.35	******	******			
std. err.	0.03	******	******	******	0.41	******	******	******	2.06	******	******	******	0.00	******	******	******	0.00	******	******	******			
t-test	0.42	******	******	******	0.00	******	******	******	0.83	******	******	******	#DIV/0!	******	******	******	0.00	******	******	******			
count (n)	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00	7.00			
95% conf +/-	0.02	0.02	0.36		1.60	1.57	0.05		1.60	1.64	7.92		0.16	0.16	0.43		2.52	2.52	21.75				
95% conf. upper	7.15	7.13	0.20	1.01	537.60	527.71	-1.79	1.02	36.60	36.96	9.35	1.08	8.76	6.76	-22.84	1.31	10.23	12.23	57.50	0.85			
95% conf. lower	7.10	7.10	-0.52	1.00	534.40	524.58	-1.89	1.02	33.40	33.69	-6.49	0.92	8.44	6.44	-23.70	1.30	5.19	7.19	14.00	0.67			
MPEU (99%)	0.09	0.07			6.79	6.65			6.79	6.94			0.68	0.68			10.69	10.69					
sufficient n?	sufficient n	sufficient n			sufficient n	sufficient n			sufficient n	sufficient n			sufficient n	sufficient n			sufficient n	sufficient n					
				↑	If the 95% CI range includes 1 not sig different			↑	If the 95% CI range includes 1 not sig different			↑	If the 95% CI range includes 1 not sig different			↑	If the 95% CI range includes 1 not sig different			↑	If the 95% CI range includes 1 not sig different		

notes: how was the test conducted in as much detail as you can provide
what instruments and serial numbers were used
what was the type of each sensor
who were the observers and what were their experience levels
was sampling done from a bucket
were flows constant during the period of the test

Change in Meters, methods or indicators
When change in meters, instruments, or methods occurs 30 measurements of the following on the same sample will be recorded
Preferred method: The data format below is used in a 2-tailed paired t-test with equal variances assumed
Paired means a data point using each method is collected at each different site, so they must be paired site-by-site for comparison
In Excel terminology for TTEST tails = 2 type = 1

If we wanted to compare results of each method at one site the sample number from each instrument doesn't need to be the same and we would call that test a two sample equal variance test.
In Excel terminology for TTEST tails = 2 type = 2

site visit	Old method (units?)	New Method (units?)	percent difference	fraction of change old to new	date (mm/dd/yyyy)	time	location
1	25	23.75	-5.00	-4.75			
2	26	25	-3.85	-6.50			
3	30	29	-3.33	-8.70			
4	10	9	-10.00	-0.90			
5	23	22	-4.35	-5.06			
6	13	12	-7.69	-1.56			
7	9	8	-11.11				
8	1	0	-100.00				
9	29	28	-3.45				
10	25	24	-4.00				
11	30	29	-3.33				
12	25	24	-4.00				
13	30	29	-3.33				
14	15	14	-6.67				
15	14	13	-7.14				
16	25	24	-4.00				
17	15	14	-6.67				
18	6	5	-16.67				
19	17	16	-5.88				
20	5	4	-20.00				
21	13	12	-7.69				
22	16	15	-6.25				
23	9	8	-11.11				
24	1	0	-100.00				
25	23	22	-4.35				
26	6	5	-16.67	-0.30			
27	15	14	-6.67	-2.10			
28	1	0	-100.00	0.00			
29	18	17	-5.56	-3.06			
30	11	6	-45.45	-0.13			
mean	10.71	9.14	-39.81	-1.52			
std dev	8.50	8.61	43.48	1.97			
offset	1.83	******	******	******			
bias (slope)	0.97	******	******	******			
bias direction	******	******	**negative bias**	**-1.57**			
coeff of var.	0.79	0.94	******	******			
std. err.	1.63	******	******	******			
t-test	0.03	******	******	******			
count (n)	7.00	7.00	7.00	7.00			
95% conf +/-	6.30	6.38	32.21				
95% conf. upper	13.42	13.49	-7.60	-0.06			
95% conf. lower	0.83	0.73	-72.02	-2.98			
MPEU (99%)	26.71	27.06		↑			
sufficient n?	need more n	need more n		If the 95% CI range includes 1 then the results are likely not different			



See this web site for more explanation and an example you can run in Excel.
http://helios.bto.ed.ac.uk/bto/statistics/tress4a.html
Test for difference in Means

run the t-test by clicking tools, data analysis and select
t-Test: Paired Two Sample for Means

	Variable 1	*Variable 2*
Mean	16.2	15.05833333
Variance	84.92413793	86.74001437
Observations	30	30
Pearson Correlation	0.996950193	
Hypothesized Mean Diffe	0	
df	29	
t Stat	8.564230555	
P(T<=t) one-tail	9.81006E-10	
t Critical one-tail	1.699126996	
P(T<=t) two-tail	1.96E-09	
t Critical two-tail	2.045229611	

(The test will "ask" *what is the probability of obtaining our given results by chance if there is no difference between the means?*)

(This shows the t value calculated from the data)

(This shows the probability of getting our calculated t value by chance alone. If it is extremely low the means are significantly different) this is the same result of t-test above
(This shows the t value that we would need to exceed in order for the difference between the means to be significant)

compare P(T<=t) two-tail to the selected confidence level. If this value is < than the significance level then reject the null hypothesis that the means are equal
compare P(T<=t) two-tail to the selected confidence level. If this value is > than the significance level then accept the null hypothesis that the means are equal

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #7

Quality Assurance Project Plan (QAPP) for

Water Quality Monitoring

Version 1.0 (4/10/2007)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

Approvals

_____ Date _____
Dustin Perkins
NCPN Network Coordinator, Inventory and Monitoring Program

_____ Date _____
David Thoma
NCPN Hydrologist, Water Quality Monitoring Program

_____ Date _____
Dave Sharrow
Hydrologist, Zion National Park

_____ Date _____
Charlie Schelz
Biologist, Southeast Utah Group

_____ Date _____
Matt Malick
Hydrologist, Curecanti National Recreation Area

_____ Date _____
Kirby Wynn
Program Manager, USGS Western Slope

_____ Date _____
Roy Irwin
Water Quality Section, Water Resources Division, National Park Service

Table of Contents

1. Introduction 5
1.1. Utah Laboratory Analysis 5
1.2. Colorado Laboratory Analysis 6
2. Park Acronyms 6
3. Distribution list and contact information 7
4. Project Management 7
5. Problem Definition/Background 8
5.1. Monitoring for Clean Water Act compliance and intent 8
5.2. Water quality standards 8
5.3. Intended uses 9
5.4. Objectives 9
5.4.1. Collect, analyze and interpret data to support management in relation to 303(d) listings of waters. 9
5.4.2. Collect, analyze and interpret data to support management of threatened or otherwise special waters, using state standards developed under the Clean Water Act (CWA). 9
5.4.3. Evaluate data for early warning signs to provide resource managers with the ability to mitigate problems and improve park resources. 10
5.4.4. Evaluate data to ensure the intent of CWA anti degradation policy is met 10
5.5. Monitoring Questions 10
5.5.1. What are the chemical and biological ranges in water quality parameters within selected waters of NCPN parks? 10
5.5.2. What are the long-term status and trends in selected water quality parameters at selected sites on selected NCPN water bodies? 11
5.5.3. Are selected NCPN water bodies in compliance with designated beneficial uses? 11
5.5.4. Are the existing chemical and biological ranges at selected times and sites consistent with establishment of Outstanding Natural Resource Waters designation (ONRW)? 11
5.5.5. What are likely point and non-point pollution sources within the watersheds? 11
5.5.6. Are specific management actions reducing pollution loads? 12
6. Project/Task Description Synopsis 12
7. Measurement Quality Objectives 13
7.1. Data Quality Objectives (DQOs) 13
7.1.1. Target population 14
7.1.2. Representativeness 14
7.1.3. Completeness 14
7.1.4. Data Comparability (internal/NPS and external) 15
7.1.5. Measurement Sensitivity and detection limits 16
7.1.6. Measurement Precision / Repeatability 19
7.1.7. Measurement Systematic Error/Bias/Percent Recovery 20
7.2. Decision Rules 26
7.3. Handling non-detects 27
7.4. Violation of Data Quality Objectives 28
8. Training Requirements / Certification 28
9. Documentation and Records 28
9.1. Utah parks 28
9.1.1. Field data 28
9.1.2. Laboratory data 29
9.2. Colorado parks 29
9.2.1. Field data 29
9.2.2. Laboratory data 29
9.3. Data management (SOP 14) 29
9.4. Evaluation (SOP 15) 30
9.5. Reporting (SOP 15) 30
10. Sampling Process Design 30
10.1. Program design 30
10.2. Type, frequency and timing of sample collection 30

10.3. Sample site selection....30
10.4. Site identification....30
10.5. Sampling constraints....30
11. Sampling Methods Requirements....31
11.1. Parameters....31
11.2. Equipment care and maintenance....31
12. Sample Handling and Custody Requirements....32
12.1. Utah Park sample handling and custody....32
12.2. Colorado Park sample handling and custody....32
13. Analytical Methods Requirements....32
14. Quality Control Requirements....32
15. Instrument/Equipment Testing, Inspection, and Maintenance Requirements....33
16. Instrument Calibration and Frequency....33
17. Inspection/Acceptance Requirements for Supplies....33
18. Data Acquisition Requirements....33
19. Data Management....34
20. Assessment and Response Actions....34
21. Reports....34
22. Data Review, Validation, and Verification Requirements....35
23. Validation and Verification Methods....36
24. Reconciliation with Data Quality Objectives....36
24.1. Effectiveness....36
24.2. Completeness....36
25. Citations....37
26. Appendix A: State of Utah Department of Environmental Quality Division of Water Quality Quality Assurance Program Plan for Water Monitoring Programs....38
27. Appendix B: Utah Environmental Testing Laboratory Certification Bulletin....58
28. Appendix C: Quality Control at United States Geological Survey National Water Quality Laboratory....63
28.1. INTRODUCTION....63
28.2. QUALITY-CONTROL PROCESS....63
28.3. INORGANIC ANALYSIS....64
28.4. ORGANIC ANALYSIS....66
28.5. SELECTED REFERENCES....69
29. Appendix D: Example AMS+ and AMS calculations....72

List of Tables

Table 1. Northern Colorado Plateau Network units....6
Table 2. People who will be aware of the project goals, tasks, and products....7
Table 3. Project tasks and responsibilities....8
Table 4. Project task extended timetable....13
Table 5. Measurement quality objectives as rejection criteria....21
Table 6. Recommended MP instrument post-calibration check-error limits....26
Table 7. Equipment characteristics for NCPN water quality monitoring....31

1. Introduction

The Quality Assurance Project Plan (QAPP) is the written record of the QA/QC program.

Quality assurance (QA) refers to the overall *management system* which includes the organization, planning, data collection, quality control, documentation, evaluation, and reporting activities of the group. QA provides the information needed to ascertain the quality of data and whether it meets the requirements of the program. QA ensures that data will meet defined standards of quality with a stated level of confidence.

Quality control (QC) refers to the routine *technical activities* whose purpose is, essentially, error control. Since errors can occur in either the field, the laboratory or in the office, QC must be part of each of these functions. QC should include both internal and external measures.

The format of this QAPP is from the EPA document,

EPA 2001. Environmental Protection Agency Requirements for Quality Assurance Project Plans, EPA QA/R-5. EPA Office of Environmental Information, Washington, D.C.

http://www.epa.gov/quality/qs-docs/r5-final.pdf Accessed online 1/30/06

Additional information used to guide development of this QAPP was drawn from The Volunteer Monitor's Guide To Quality Assurance Project Plans United States Environmental Protection, Agency Office of Wetlands Oceans and Watersheds, 4503F EPA 841-B-96-003, September 1996,
http://www.epa.gov/owow/monitoring/volunteer/qappcovr.htm , Accessed 1/30/06.

It has been modified to meet NCPN monitoring activities as directed by the NPS-WRD guidance document, Part B lite (Just the Basics) Review Checklist for Protocols and SOPs, May 20, 2005 (Draft).
http://www.nature.nps.gov/water/VitalSigns_index/VitalSignsdocuments.htm Accessed 1/30/06

1.1. Utah Laboratory Analysis

As an Agency Cooperator, the NCPN partners with the State of Utah, Division of Water Quality (UTDWQ) to monitor water quality conditions in NPS units in Utah. Staff members from NCPN and cooperating parks are responsible for taking field measurements, collecting and processing samples, delivering them to the state laboratory, and upon receipt of results verifying the quality of the result. The state is responsible for providing sample containers, conducting laboratory analysis, providing agency cooperators with timely laboratory results to review, and entering data into STORET. The UTDEQ is also responsible for program management and quality assurance and quality control that are acceptable to the Environmental Protection Agency (EPA).

1.2. Colorado Laboratory Analysis

As an Agency Cooperator, the NCPN partners with the USGS to monitor water quality conditions in NPS units in Colorado. For most CO park units, staff from NCPN is responsible for taking field measurements, collecting and processing samples, delivering them to the National Water Quality Laboratory (NWQL), and upon receipt of results verifying the quality of the results. The exception to the aforementioned is that USGS staff takes field measurements, collects sample and delivers samples to the National Water Quality Laboratory on the Green and Yampa rivers. The USGS provides laboratory analysis, and timely results to review. Data from the USGS laboratory are entered into the National Water Information System (NWIS) data base, while NPS staff enters data into STORET. The National Water Quality Laboratory is responsible for laboratory management, quality assurance and quality control that is acceptable to the EPA.

2. Park Acronyms

Table 1. Northern Colorado Plateau Network units.

Unit Name	Park Code	State	Hectares
Arches National Park *	ARCH	UT	30,966
Black Canyon of the Gunnison National Park	BLCA	CO	12,239
Bryce Canyon National Park	BRCA	UT	14,502
Canyonlands National Park *	CANY	UT	136,610
Capitol Reef National Park	CARE	UT	97,895
Cedar Breaks National Monument	CEBR	UT	2,491
Colorado National Monument	COLM	CO	8,310
Curecanti National Recreation Area	CURE	CO	17,433
Dinosaur National Monument	DINO	CO/UT	85,097
Fossil Butte National Monument	FOBU	WY	3,318
Golden Spike National Historic Site	GOSP	UT	1,107
Hovenweep National Monument*	HOVE	UT/CO	318
Natural Bridges National Monument *	NABR	UT	3,009
Pipe Spring National Monument	PISP	AZ	16
Timpanogos Cave National Monument	TICA	UT	101
Zion National Park	ZION	UT	59,900
Total Network Area			**473,312**

*Parks in the Southeast Utah Group (SEUG)

3. Distribution list and contact information

Table 2. People who will be aware of the project goals, tasks, and products.

Name/Title/Agency	Contact
Dustin Perkins/Program Coordinator/NCPN	435-719-2358, dusty_perkins@nps.gov
David Thoma/Hydrologist/NCPN	435-834-4905, **dave_thoma@nps.gov**
Dave Sharrow/Hydrologist/ZION	970-240-5431, dave_sharrow@nps.gov
Matt Malick/Hydrologist/CURE	970-641-2337 x-248, **matt_malick@nps.gov**
Ken Stahlnecker/Chief resource mgmt./CURE	970-641-2337 x-225, **ken_stahlnecker@nps.gov**
Dave Worthington / CARE biologist	435-425-3791 x-145, dave_worthington@nps.gov
Charlie Schelz / SEUG biologist	435 719-2135 Charlie_Schelz@nps.gov
Mary Moran / SEUG biotech	435 719-2139 Mary_Moran@nps.gov

4. Project Management

This project is intended to be a long-term (30+ years) monitoring plan. As such, consideration in this QAPP is given to potential change in equipment, laboratory method, and staff. The plan will be managed and largely implemented by network (NCPN) personnel. Some sites will be monitored in cooperation with USGS and technical assistance will be provided by USGS and NPS Water Resources Division (WRD). Decisions regarding the I&M Water Quality Program development and long term monitoring plan have been and will continue to be made by consensus of the NCPN aquatic professionals team consisting of key network staff including the water quality specialist, network coordinator, park hydrologists, and biologists. The I&M Water Quality Program will remain adaptive to changing park and network needs. Broader-based, long-term decisions are approved by the NPS-WRD with additional technical guidance from USGS and the NCPN technical committee. These include approval of WRD-funded staff work plans and approval of the overall monitoring plan.

Table 3. Project tasks and responsibilities.

Name (Affiliation)	Title/Responsibility
David Thoma (NCPN)	Hydrologist Technical Lead - oversee collection of monitoring data and data management, coordinate report writing and protocol revision
Dave Sharrow (ZION), Charlie Schelz (SEUG), Matt Malick (CURE)	Aquatic Professionals Team - technical working group
Dustin Perkins (NCPN)	NCPN I&M Coordinator - Coordinate with water quality technical lead on reporting requirements, assist with peer review process, program oversight
Helen Thomas / Russ DenBleyker (NCPN)	NCPN Data Management Staff - assist with water quality database management, archive, and uploading to NPS and national data bases
Kirby Wynn (USGS)	USGS Western Slope water quality program manager - provide technical oversight and coordination with cooperative agreements.
Pete Penoyer / Roy Irwin (WRD)	WRD Hydrologists - technical oversight, peer review, and formal program approval

5. Problem Definition/Background

5.1. Monitoring for Clean Water Act compliance and intent

General objectives of the Water Quality monitoring program are intended to meet the overarching purpose of the I&M program to understand status and trends of key resources, increase understanding of natural variation, and provide managers with early warning of abnormal change. More specifically, the water quality program is designed to generate the information needed to evaluate status and trends in selected water bodies and to determine if water quality in selected sites in NCPN parks is sufficient to meet a protected class of uses established by states under the Clean Water Act (CWA). Furthermore, given the intrinsic value of waters wholly encompassed in, or originating in National Parks it is critical that they be managed for anti-degradation, an important provision in the CWA, to protect the good quality of waters that meet or exceeded their standards.

5.2. Water quality standards

Water quality standards are established to protect the beneficial uses of the streams, rivers, lakes and reservoirs within the states (Appendix A of the Protocol Narrative). Beneficial uses include source of drinking water (Class 1C), and recreation (Classes 2A and 2B). Fishing and other aquatic life classifications include cold water game fish (3A), warm water game fish (3B), non-game fish (3C) and other aquatic life such as waterfowl and shore birds (3D). Waters used for irrigation and stock watering are classified as Class 4. A special class, 5 has been established for the Great Salt Lake in UT. Streams and lakes of the state are classified as one of the above or a combination of them. Future

changes to this list can be verified by checking current web links to state water quality guidelines listed in the Protocol narrative Table 1. The information generated in this monitoring effort will be used to direct management actions that maintain high quality status of waters and improve them as needed.

5.3. Intended uses

The quantitative information generated by evaluating NCPN waters for Clean Water Act compliance and intent will be used by managers who strive to achieve the following for selected waters in the network:

1. Maintain waters that vary within their natural chemical and biological ranges and meet applicable federal and state water quality criteria. Justification. Waters that vary within their natural ranges can typically support healthy aquatic ecosystems and most beneficial uses.

2. Improve water quality of impaired waters. Justification. The NPS GPRA goal is that 99.3% of streams and rivers managed by NPS will meet State and Federal water quality standards.

3. Demonstrate and maintain high water quality where it exists. Justification. National Parks should be leading the way in high water quality regardless of beneficial uses (i.e., consideration should be given to water resources for their intrinsic value alone).

5.4. Objectives

5.4.1. Collect, analyze and interpret data to support management in relation to 303(d) listings of waters.

1. Data collected monthly are evaluated to determine if they exceed state criteria and thus fail to meet minimum required conditions for intended use.

2. The quality of water is assessed as “fully supporting” (good to excellent water quality), “partially supporting” (meets the standards most of the time), and “not supporting” (frequently the water quality standards are not met). Definitions for these categories can be found in state 305(b) reports to Congress. (Appendix B in Utah’s 2006 report to Congress)

5.4.2. Collect, analyze and interpret data to support management of threatened or otherwise special waters, using state standards developed under the Clean Water Act (CWA).

1. Data are evaluated to determine if acute or chronic conditions exist that do not place a water body on the 303(d) list, but could still have short term or longer term detrimental effects on species of interest. These are spikes or long term near-exceedance conditions that will be apparent from monthly and longer term analysis of results. When a water body parameter exhibits chronic or acute conditions further investigation (e.g. literature review and perhaps more detailed study) into the potential detrimental effects will be required.

2. Determine the cause(s) of non-attainment of standards. Initially this will be done by association where cause and effect relationships are obvious, such as selenium from geologic deposits in the watershed. If no obvious association is apparent further investigation will be required.

   Determining the cause of water quality impairment will allow managers to develop a plan for bringing the problem water body into compliance. These plans analyze the Total Maximum Daily Loading of contaminants to the water and are therefore referred to as "TMDL" plans. States can consider a wide variety of remedies including reducing the contaminant loading, changing the designated protected uses (E.g. from Cold Water Fishery to Warm Water Fishery) to more accurately reflect ambient conditions, or documenting that the "poor" quality of the water is due to natural causes and is not correctable.

5.4.3. Evaluate data for early warning signs to provide resource managers with the ability to mitigate problems and improve park resources.

1. Short term trends that may not be statistically significant and occasional acute conditions that cannot be used as conclusive indicators of long term water quality degradation may still serve as early warning signs of system degradation that could signal the need for more intensive study of a potential problem.

2. Water quality data will be coupled with upland monitoring or riparian monitoring data when available to strengthen conclusions about sources and extent of degradation.

3. In the course of evaluating objectives 1-3 and with attention to early warning signs, data gaps will become apparent where insufficient knowledge exists to inform management. Such knowledge gaps will become focal points for projects, collaborative research or expansion of the monitoring program.

5.4.4. Evaluate data to ensure the intent of CWA anti degradation policy is met

Long term analysis of data trends will allow managers to determine if the intent of the anti degradation concept of the CWA is being met.

5.5. Monitoring Questions

The data analysis SOP (SOP 15) outlines the specific procedures for answering the monitoring questions. The following specific questions will be answered to inform adaptive management of water resources:

5.5.1. What are the chemical and biological ranges in water quality parameters within selected waters of NCPN parks?

1. This will be determined over time as data accumulate under different flow, seasonal and climatic conditions. Characteristic range and variability will be determined for the water year and for segments of the water year.

2. Characteristic conditions of water bodies will be determined in the shortest period of time by analyzing data from base flow conditions, because this is when most sampling will occur.

5.5.2. What are the long-term status and trends in selected water quality parameters at selected sites on selected NCPN water bodies?

1. This will be determined over time as a function of flow, season and climatic conditions. Trend analysis of historic data (section 2.0 of the Protocol Narrative) provides an indication of durations required to detect differences over time. The relatively long durations are due to high natural variability in parameter values, and climate and the monthly sampling period and the bi-annual rotating panel design of the monitoring program.

2. This question will be confounded if drivers of natural variation change during early stages of monitoring (eg, fire, extreme climate events, or even human influences). For this reason, where possible water bodies of similar character can be compared to determine if a trajectory occurs that may be due to impairment. It should be noted that the monitoring plan was not designed for such inter-site comparisons or extrapolation to other water bodies, but when reasonable this possibility will be considered to strengthen arguments for changes in condition that may be due to relatively obvious factors (ie. July 2006 fire in North Creek watershed).

5.5.3. Are selected NCPN water bodies in compliance with designated beneficial uses?

1. This will be determined on a monthly basis via comparison to state standards.

2. This information will become part of the State 303(d) report.

5.5.4. Are the existing chemical and biological ranges at selected times and sites consistent with establishment of Outstanding Natural Resource Waters designation (ONRW)?

1. This will only be possible after many years of monitoring and through cooperation with the state Water Board, but should be a long term goal of this program.

2. It is a consensus decision based on data that shows a water body consistently exceeds its designated use criteria.

3. The CWA specifically mentions waters in National Parks as prime examples of potential ONRW designation.

5.5.5. What are likely point and non-point pollution sources within the watersheds?

1. This question will be addressed where possible through association. That is, many pollutants have unique or typical sources such as selenium from geologic deposits and nutrients from agricultural activities.

2. Follow-up studies will be required to determine causality in cases that are not obvious.

5.5.6. Are specific management actions reducing pollution loads?

1. This will be addressed by tracking long term trends and by follow-on study after causality has been determined. Monitoring alone without additional study related to cause and effect cannot alone determine if management actions have the intended consequence.

2. This may require cooperation with States to establish total maximum daily loads (TMDLs) and best management practices (BMPs) in watersheds.

6. Project/Task Description Synopsis

An historic analysis of existing data conducted through a cooperative agreement with USGS and discussion with park resource managers and water quality specialists narrowed the field of potential sampling sites to those that are currently impacted or threatened. Those waters on the 303d list are listed in the protocol narrative section 1.3.3. Water bodies exceeding state standards for intended uses are presented in section 1.4. Rationale for sampling design and site selection is presented in section 2.0 and sampling sites and panel rotation for the sites are given in Table 2-3.

Site visits are coordinated to occur during periods of low flow when possible, but sampling during higher flow will provide information on the natural range and variability of water quality parameters under variable flow regimes.

For each monthly site visit flow and core parameters of temperature, pH, conductivity and dissolved oxygen are measured (SOP 8) and samples are collected for chemical analysis (SOP's 9 and 10). Samples from CO parks are analyzed by the USGS National Water Quality Laboratory in Lakewood, CO, while samples from UT parks are analyzed by the UT Department of Environmental Quality Water Quality Laboratory in Salt Lake City, UT. The analytes and methods of analysis for each analyte can be found in SOP's 12 and 13 that describe laboratory methods.

Field parameters and calibration data are evaluated in the field before leaving the site and laboratory data are evaluated in blocks when they become available from the cooperating laboratories. Repeated measures are made in the field to minimize error, and when possible laboratory samples are re-run if necessary.

Bias and precision in field measured core parameters are minimized by careful calibration and equipment maintenance (SOP 5). Bias and precision in laboratory analysis is minimized by laboratory specific quality control procedures that result in EPA certification. Error due to sloppy field technique in sample collection is minimized by formal training in sample handling and monitoring trip replicates and field blanks (SOP 2, 9, and 12 for CO and 3, 10 and 13 for UT Parks respectively).

Core parameters, flow, calibration data and laboratory data are stored electronically both in Excel (printer-friendly) and ascii formats. Ascii formatted data are imported to the MS Access historic data base, NCPN-water for archive and data analysis (SOP 14).

Data analysis and reporting will be made on an annual basis to track condition and trends in data range and variability. Emphasis will be placed on comparison with state water quality standards. The standards vary by state and intended use of the water body. Links to state standards can be found in Table 1-1 of the protocol narrative.

Table 4. Project task extended timetable.

Deliverables	FY06	FY07	FY08	FY09	FY10
Develop QAPP and SOP's	X				
Coordinate monitoring with state and USGS	X				
Review QAPP and SOP's	X	X	X	X	X
Attend USGS WQ training	X				
Collect monitoring data	X	X	X	X	X
Compile, QC and archive data	X	X	X	X	X
Annual summary report		X	X	X	X
Comprehensive data analysis and synthesis					X

7. Measurement Quality Objectives

This section is intended to demonstrate how this project generates data of known and documented quality, resulting in complete, accurate and transferable information. Data credibility necessary for the intended uses will be achieved when it is:

1. consistent over time and consistent between staff members
2. collected and analyzed using standardized and acceptable techniques
3. comparable to data collected in other assessments using the same methods
4. used appropriately to make decisions based on sound statistics

7.1. Data Quality Objectives (DQOs)

These are the quantitative and qualitative terms that describe how good data need to be in order to meet project objectives. DQOs for *measurement data* (referred to here as data quality indicators) are discussed in detail below but are listed by NPS WRD as:

1. Target population (section 7.1.1)
2. Representativeness (section 7.1.2)
3. Completeness (section 7.1.3)
4. Data comparability (internal/NPS and external section 7.1.4)
5. Measurement sensitivity and detection limits (section 7.1.5)
6. Measurement precision as repeatability (section 7.1.6)
7. Measurement systematic error/bias (section 7.1.7)

7.1.1. Target population

The target population to be monitored for water quality is all potential values that might be measured at a group of selected sites on perennial waters monitored monthly during daylight hours chosen through a judgmental process involving the participation of park resource managers, NCPN staff, and USGS and other water quality specialists. The water bodies to be sampled and the sampling design for the network are justified in the protocol narrative (section 2.0).

7.1.2. Representativeness

Representativeness for targeted sites is ensured by selecting spatially and temporally representative sampling times and exact places at each targeted site being monitored. Methods to ensure representative site selection are found in SOP 4, but the sites selected will only give qualitative hints of what might be going on the larger water body, not definitive quantitative evidence. Methods used to ensure representativeness based on flow conditions are presented in Appendix A of SOP 8.

Good QA/QC practices and calibration practices will help insure that the measured values are indeed representative of magnitude of the signal being measured at the particular time and place of measurement. For example, it is up to the hydrologist collecting field data to verify good calibration of sensors, that core parameter data are within a reasonable range of historic values while at the site and that samples are collected in a manner that best represents water flowing past the cross section. The completed site description hard copies reside in the Field Folder and include an annually updated range in parameter values. If core parameter values are well outside the normal range, documentation explaining why will be included on the field forms that will accompany the data through to final archive.

7.1.3. Completeness

Given the goals of assessing attainment of beneficial use criteria and assessing range and variability in all seasons it is extremely important that samples be collected throughout the year. However, it is not reasonable to expect that all sites will be accessible all year given remote locations and access issues. Given our minimum detectable differences and calculated statistical power, 90% completeness should insure large enough sample sizes to meet project goals outlined in the protocol narrative (sections 2.5 thru 2.9). That said, every effort will be made to achieve 100% completion of data collection and analysis, but realistically some sites will be missed in the monthly sampling runs. Some error in core parameters and laboratory analysis is also expected that will result in un-usable data. The state of UT requires 10 monthly site visits for 305(b) reporting.

7.1.3.1. Compliance

The completeness of sampling on an annual basis is related to the number of monthly samples necessary to make compliance assessments relative to beneficial use criteria. From a compliance standpoint 4 or more samples are required to assess violations of acute standards, while 10 or more samples are required to assess violations of chronic standards (UT DWQ 2006).

The sampling frequency for most network parks is monthly, which is cost driven for CO parks. For UT parks, it is driven by cooperator requirements established by UT DEQ laboratory to make 305(b) reports. The exceptions to monthly sampling in CO parks are the Green and Yampa Rivers, which are sampled 6 and 4 times annually in FY 06 due to cost constraints.

7.1.3.2. Statistics

The completeness of data required for long term trend analysis is related to parameter variability. Durations necessary to detect statistically significant trends have been discussed in the protocol narrative sections 2.6 through 2.9. In summary, changes greater than 30% of the historic mean can be detected in as little as 2 years of monthly monitoring for pH, dissolved oxygen, specific conductivity but not for different forms of P.

It may take longer than 2 years to detect significant trends (>30% change in mean parameter value) even at 100% completeness (see figure 3 in protocol narrative). Likewise more subtle change detection will require longer monitoring durations.

Costs of sampling the Yampa and Green exempt these sites from this standard for completeness in FYO6. If funding allows, sampling on these rivers will increase in the future.

7.1.4. Data Comparability (internal/NPS and external)

7.1.4.1. Internal

Data comparability through time across network parks even when sensors, instruments, and staff change occur is ensured by following guidelines for staff training (SOP's 2 and 3) site selection (SOP 4), equipment handling and maintenance (SOP 5) and sample processing (SOP's 9 and 10). When methods or equipment change that may result in bias a record will be made in equipment log books and the appropriate SOP. See the protocol revision (SOP 16) for more information on how changes in SOP's are made and archived. These changes and maintenance fixes are also recorded in the premobilization worksheet in Field_forms.xls (SOP 5 Appendix A). If older equipment is still operable, it will be used in conjunction with newer equipment to determine if a bias exists that can be corrected (SOP 6).

An important component of this program involves storing calibration reports for DO, pH and SC in the Field_forms.xls spreadsheets. Both calibration reports from monthly pre-mobilization exercises (SOP5 Appendix A) and field calibrations (SOP 8) are stored for each site visit. Field_forms.xls has built in evaluation routines that compare calibration results to expected values. Evaluation of temporal change in sensor constants, slopes and offsets provides an excellent way to predict when sensors may be wearing out.

7.1.4.2. External

Core parameters. Data comparability in core parameters is maximized by employing USGS standard operating procedures for measurement (SOP 8) and NPS WRD guidelines for calibration frequency and methodology (SOP 5 Appendix A). Additionally, careful attention is paid to equipment manufacturer guidelines to maintain equipment in

optimum working condition (SOP 5). Further assurance of data comparability in core parameter data is achieved by using National Institutes of Standards and Technology (NIST) traceable standards for calibration. The data quality objectives DQO's for core parameters are summarized in SOP 5 Table 1. These quality goals are ensured primarily through careful equipment calibration and repeated measures.

Built into the Field_forms.xls spread sheet, used for recording field data in UT parks, are quality control checks that calculate and report whether or not the core parameter data meet data quality objectives defined in SOP 5. Failure of core parameter data to meet the DQO's specified in well mixed streams outlined in SOP 5 should result in data rejection. This is the standard outlined by NPS WRD (Irwin 2004) as the criteria for "reproducibility plus", where "reproducibility" is related to instrument noise and "plus" is change in values due to slight heterogeneity in a medium like a well mixed flowing stream.

Laboratory analysis. Similarly, laboratory data created by this monitoring program has comparability assured by using USGS standard methods (SOP 12) for CO parks and EPA standard methods or methods outlined in Standard Methods for Analysis of Water and Wastewater (SOP 13) for UT parks. Systematic error or bias is controlled in the laboratories by running standards and blanks (SOP 12 and 13). Error incurred by poor techniques in sample collection and handling are minimized by following sample collection and handling standard operating procedures (SOP's 9 and 10), and by running replicates and trip blanks [still working on this with UT lab, i.e. what level of QC they will support for free for a relatively small operation like NCPN called and e-mailed again late Oct 2006].

Further assurance of data comparability and data credibility comes from laboratory certification by EPA every three years for the UT lab. More detail regarding the quality control process for the Utah Environmental Laboratory which includes the Water Quality Laboratory is provided in Appendix A. Updates of audits for the National Water Quality Laboratory can be found at http://nwql.usgs.gov/Public/perf_hdr.html. More detail regarding the quality control process for NWQL which analyzes samples from the CO park units is provided in Appendix B.

7.1.5. Measurement Sensitivity and detection limits

The low-level method detection limits (MDL) and minimum level of quantitation (ML) are the terms used to describe the smallest presence that can be observed and the accurate quantity of an analyte of interest. They are important concepts in water quality data reporting and analysis for many reasons especially when considering chronic low level pollution that is near the detection limits of analysis methods (Irwin 2006). These values will be updated / reported at least once per year or when methods change and become part of the permanent data record. Values below the MDL and ML will be reported as per NPS guidance (Irwin 2006) which follow Helsel's recommendations (Helsel, 2005. Nondetects and Data Analysis: Statistics for Censored Environmental Data. Wiley. 288 pp.**,** http://www.wiley.com/WileyCDA/WileyTitle/productCd-0471671738.html**)**

7.1.5.1. Method Detection Limit (MDL)

The method detection limit (MDL) is the lowest value that can be distinguished from zero.

Colorado park units. Analytes, method, and reporting limits for CO parks are listed in SOP 12 Table 12-1. The reporting limits are the minimum detectable or quantifiable concentration reported by the USGS NWQL laboratory. These can change a little year-to-year based on laboratory performance checks. The reporting limit types include Lab Reporting Limit (2 x the Long-Term Minimum Detection Limit (LTMDL)) and Minimum Reporting Limit. Reporting limits for the National Water Quality Laboratory will be checked and reported (as an appendix in SOP 6 – cumulative bias) at least prior to each annual STORET data up-load. A description of the LTMDL approach can be found at http://nwql.usgs.gov/Public/ltmdl/ltmdlsplash.html. The following information related to method detection level (MDL) is taken from the USGS open file report 99-193. It follows the procedure used by EPA found at http://water.usgs.gov/owq/OFR_99-193/detection.html#detection.

In summary, given the assumptions of constant standard deviation at low concentration, normal distribution, and minimal matrix interferences, the USEPA MDL is the concentration at which no more than 1 percent of the blank measurements result in a false positive detection. Therefore, detections at concentrations greater than or equal to the MDL concentration should be true detections 99 percent of the time.

The USEPA MDL is calculated as
$MDL = s * t_{(n-1,\ 1-\alpha = 0.99),}$
where
n = number of replicate spike determinations at 1 to 5 times the estimated MDL,
s = standard deviation of measured concentrations of n spike determinations,
t = Student's t value at n-1 degrees of freedom and 1-α (99percent) confidence level.
When n=7 and α=0.01, then t=3.14 and
α = level of significance.

Bias and precision are also evaluated by collecting 1 field blank and one field replicate per 21 one samples. (1 blank, 1 rep per field event). USGS requires 1 blank and 1 rep per 20 sites. They recommend 1 blank and 1 rep per 10 sites (Malick personal Comm..)

Utah park units. Analytes, method, and minimum reporting levels (RL's) for UT parks are listed in SOP 13 Appendix C. [Waiting for reply [e-mailed and called 10-30-06] to answer questions about MRL, ML and blank controls ie section 7.5.2 below] Need to coordinate with Arne and Sanwat on a laboratory blank methodology Text in blue is from Part B lite.

For each chemical measurement done in the lab are the following adequately covered?

1. A blank control measurement quality objective (MQO), if applicable.

2. What types of blanks will be controlled (trip blanks, lab blanks, etc.)

3. Will the MQO be used as a data acceptance performance standard?

4. Will data reported be adjusted by adding concentrations found in blanks? If not, how will blank control be accomplished (reduce contamination and re-run the samples?).]

7.1.5.2. Minimum Level of Quantitation (ML)

The minimum level of quantitation (ML) is the lowest value that can be reliably measured. It is generally the MDL multiplied by 3.18. This is a value widely accepted to be sufficiently high (99% confidence) to adequately protect against reporting false negatives. Typically the ML is only pertinent to laboratory data as field core parameters are well into the quantitative range. See http://www.epa.gov/waterscience/methods/det/rad/rad.pdf

There are two exceptions where terms other than the ML can be used (Irwin 2006):

1. When dealing with STORET, the phrase "lower quantitation limit" (LQL) can be considered a synonym for a ML.

2. If a USGS lab is used, the USGS alternative to the ML, the laboratory reporting level (LRL) may be used instead of the ML. The LRL is defined as two times the USGS LT–MDL (http://water.usgs.gov/owq/OFR_99-193/level.html). If the network is going to use USGS LRLs, explain how and how often they will be calculated and reported.

7.1.5.3. Core parameter sensitivity UT park units

Manufacturer specifications. Manufacturer specified measurement sensitivity for multi parameter probes used to measure core parameters in UT Park units is provided on pg 10-11 in the MP Troll 9000 Operators manual (SOP 5 Appendix B) and in SOP 5 Appendix E for CO Park units. The only case where parameter value is likely to approach detection level in core parameters is the case for DO when measuring ground water or deep lakes. Achieving accurate low DO calibrations are discussed in the premobilization procedure SOP 5 Appendix A. However, since manufacturer's claims for resolution specifications are not consistent and not necessarily good representations of instrument measurement sensitivity in harsh outdoor environments, AMS (see next section) will be documented for each field measurement instrument no less than once a season (usually at the beginning and end of the field seasons).

Alternative Measurement Sensitivity Plus (AMS+). In the range of quantitative values where low level detection limits are not an issue measurement sensitivity is determined by evaluating the variability of repeated measurements on an environmental sample. Over time, AMS+ can be used to bound measurement uncertainty on each single data point (when 7 measurements are made on the same environmental sample). It includes instrument noise and natural heterogeneity.

AMS+ is calculated as
t*sd,
where

s = sample standard deviation of measurement values obtained in situ
t = 3.708, the 99% confidence middle (two-sided) t-value for sample size 7

The result is functionally analogous to a ML but uses the two-sided (middle) t-value because we are not only interested in one side (distinguishing a value from zero) but in the two-sided issue of how large of a difference between two individual values we can justify as actually being a true difference (from one another) rather than being due to measurement noise.

AMS+ is a concept used to evaluate measurement precision in an ambient condition such as a well mixed flowing stream. It is calculated the same way as AMS, but rather than sampling a stream sample from a bottle 7 repeated measurements are made in rapid succession in situ. Because 7 measurement of each core parameter are measured at each site visit AMS+ can be determined for each site visit. The long term average AMS+ will be reported to STORET.

AMS+ is recorded in the STORET metadata "analytical procedure description" text box. Like MDLs, AMS should be re-calculated when something significant in the measurement process changes or every sampling season at a minimum. For example, if the person measuring, the measuring instrument, or the methods or SOPs change, recalculate AMS+ to see if measurement sensitivity has changed. (Irwin 2006). An example of AMS+ calculation for core parameters is presented in Appendix D.

7.1.6. Measurement Precision / Repeatability

7.1.6.1. Laboratory analysis

The precision of laboratory analysis is monitored by the laboratory performing the analysis. The most important measures of precision are the MDL and LTMDL which includes both a measure of sensitivity and precision. The details of laboratory performance are presented in SOP 12 table 12-1 for CO parks and SOP 13 Appendix C for UT parks.

7.1.6.2. Relative Percent Difference (RPD)

Precision is the variability of repeated measures of the same thing (duplicate). It is a less rigorous measure of sensitivity than AMS (sample size 2 rather than 7), but is used more frequently to ensure measurements are in 'control' especially in laboratory analysis where 7 duplicates are not always practical.

RPD is calculated as

$$\text{RPD} = \left[\frac{S_1 - S_2}{(S_1 + S_2)/2} \right] \times 100$$

where

S_1 = the larger test result value,
S_2 = the smaller test result value.

7.1.6.3. Core parameters

Because the field measured values of core parameters are always expected to be in the "quantitative" range or well above the detection limits of the sensor, detection limits are not reported. However, measurement sensitivity will be evaluated by analysis of repeated (7) measures of a normal environmental sample. This is essentially an estimate of precision but in a more rigorous sense and is termed an Alternative Measurement Sensitivity. The results will be placed in the STORET metadata "analytical procedure description" text box.

AMS is calculated as
$AMS = 3.708 \times sd_7$
where 3.708 = the 99% confidence middle t value for sample size 7,
sd_7 = standard deviation of 7 replicate samples on a normal environmental sample.

The AMS calculation uses the two sided t value because we are not only interested in one side (distinguishing a value from zero) but in the two-sided issue of how large of a difference between two individual values we can justify as actually being a true difference from one another and not due to measurement process noise (Irwin 2004).

The AMS is calculated using data collected during premobilization with measurements made in tap water (well water from BRCA). In this case it would be inappropriate to use in situ repeated measures (as we do for AMS+) because the intent is to evaluate instrument noise alone with out confounding influence of stream variability. However, it will be calculated at least once per season and will be reported in the instrument log book. It can easily be calculated from data stored in Field_forms.xls as a measure of precision calculated for each core parameter prior to field runs (Appendix D).

7.1.7. Measurement Systematic Error/Bias/Percent Recovery

Systematic error/bias is the systematic or persistent distortion of a measurement process that causes errors in only one direction (usually high or low). For historical comparisons, results for systematic error/bias is usually expressed as a as % recovery with the correct or expected answer being considered 100% with an acceptable range between 90-110% for core parameters (Irwin 2004). To meet latest interagency guidelines and recent revisions in Part B lite, QC bias results will also be expressed as percent difference (In 2006, the Environmental Data Standards Council (EDSC, an interagency group including EPA, States and Tribes standardized the basic calculation and terminology definitions for bias using percent differences (PDs, rather than % recovery, see document at http://www.envdatastandards.net/content/article/detail/646). In this monitoring effort the systematic error/bias measurement quality objective (MQO) will be used as a data acceptance performance standard for core parameters (Table 5).

Table 5. Measurement quality objectives as rejection criteria.

Parameter	Instrument or method	Relative percent difference (RPD of duplicates)1	Blanks (field)	Measurement systematic error (% recovery or PD)2	Alternative measurement sensitivity (AMS+)3	Method detection limit (MDL)4	Minimum level of quantitation (ML)5
Colorado laboratory parameters	See SOP 12 table 12-1						
Utah laboratory parameters							
pH	Troll 9000	+/- 30%	…..	95-105%	0.010	na	na
Dissolved Oxygen (mg/L)	Troll 9000 Amperiometric	+/- 30%	…..	95-105%	0.148	na	0.02
Temperature (oC)	Troll 9000 Thermistor	+/- 30%	…..	95-105%	0.042	na	na
Specific Conductance (µs/cm)	Troll 9000 Low range	+/- 30%	…..	95-105%	6.155	na	na
E. Coli. (MPN6)	IDEXX Quantitray/2000	+/- 60%	…..			0	0.99
Turbidity	Hach P2100	+/- 3%	…..	95-105%	0.5	0.01	3.18 * MDL
Utah laboratory parameters							
Alkalinity: Method SM2320B SOP 13 Appendix E	titration	+/- 30%	na	90-110%	na	na	na
Ammonia: Method EPA 350.1 SOP 13 Appendix F	Automated Colorimetry	+/- 30%	<MDL	90-110%	na	0.1	3.18 * MDL
Bromide and Chloride: Method EPA 300.0 SOP 13 Appendix G	Ion Chromatography	+/- 30%	< MDL	90-110%	na	0.1 and 0.2 mg/L respectively	3.18 * MDL
Bromate: Method EPA 300.0 SOP 13 Appendix G	Ion Chromatography	+/- 30%	< MDL	90-110%	na	0.2 mg/L	3.18 * MDL
Chemical Oxygen Demand – COD: Method EPA 410.4 SOP 13 Appendix I	Chemical Oxygen Demand by Colorimetry	+/- 30%	< MDL	90-110%	na	3 mg/L	3.18 * MDL

Parameter	Instrument or method	Relative percent difference (RPD of duplicates)1	Blanks (field)	Measurement systematic error (% recovery or PD)2	Alternative measurement sensitivity (AMS+)3	Method detection limit (MDL)4	Minimum level of quantitation (ML)5
Chloride: Method EPA 323.3 SOP 13 Appendix J	Not available on-line	+/- 30%	< MDL	90-110%	na		3.18 * MDL
Chromium-VI: Method SM3500CD SOP 13 Appendix L	Colorimetric	+/- 30%	< MDL	90-110%	na	na	3.18 * MDL
Conductivity: Method EPA 120.1 SOP 13 Appendix M	Conductance by Conductivity Meter	+/- 30%	< MDL	90-110%	na	na	3.18 * MDL
Cryptosporidium and Giardia: Method EPA 1623 SOP 13 Appendix N	Cryptosporidium and giardia by filtration/IMS/FA microscopy	+/- 30%	< MDL	90-110%	na	40 ocysts / 10 L	3.18 * MDL
Cyanide:(Water) Method EPA 335.4 SOP 13 Appendix O	Cyanide, total, by Colorimetry	+/- 30%	< MDL	90-110%	na	.005 mg/L	3.18 * MDL
Fluoride: Method SM4500C SOP 13 Appendix P	Ion selective electrode	+/- 30%	< MDL	90-110%	na	na	3.18 * MDL
Lead and Copper: Method EPA 200.8 SOP 13 Appendix Q	Metals in Waters by ICP/MS	+/- 30%	< MDL	90-110%	na	0.6 and 0.5 mg/L respectively	3.18 * MDL
Metals, Total: Methods EPA 200.7, EPA 200.8, EPA 200.9 Appendices R,S,T,U	Metals in Water by ICP-AES, Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption,	+/- 30%	< MDL	90-110%	na	Variable, see appendicies SOP 13 for specific values by metal	3.18 * MDL
Metals, Dissolved: *(Filtered in the Field) Methods EPA 200.7, EPA 200.8, EPA 200.9 Appendices R,S,T,U	Same as above						
Mercury: Method	Mercury	+/- 30%	< MDL	90-110%	na	0.2 mg/L	3.18 * MDL

Parameter	Instrument or method	Relative percent difference (RPD of duplicates)1	Blanks (field)	Measurement systematic error (% recovery or PD)2	Alternative measurement sensitivity (AMS+)3	Method detection limit (MDL)4	Minimum level of quantitation (ML)5
EPA 245.1 SOP 13 Appendix T	(Manual Cold Vapor Technique)						
Nitrate and Nitrite: Method EPA 353.2 SOP 13 Appendix U	Nitrate-Nitrite Nitrogen by Colorimetry	+/- 30%	< MDL	90-110%	na	0.05 mg/L	3.18 * MDL
Nitrite: Method EPA 353.2 SOP 13 Appendix U	Nitrate-Nitrite Nitrogen by Colorimetry	+/- 30%	< MDL	90-110%	na	na	3.18 * MDL
Nutrients, Total: Methods 365.1 Phosphate SOP 13 Appendix V	Phosphorus (all forms) by Semi-Automated Colorimetry	+/- 30%	< MDL	90-110%	na	0.01 mg/L for both phosphate and phosphorus	3.18 * MDL
Nutrients, Dissolved: *(Filtered in the Field) Methods 365.1 SOP 13 Appendix V (Phosphate), EPA 353.2 (Nitrate/Nitrite) SOP 13 Appendix U	Same as above						
pH: Method EPA 150.1 SOP 13 Appendix V	pH in Water by Electrometric Method	+/- 30%	< MDL		na	na	3.18 * MDL
Phosphate: Method EPA 365.1 SOP 13 Appendix V	Same as above						
Silica: Method EPA 370.1 SOP 13 Appendix W	Silica by Colorimetry	+/- 30%	< MDL	90-110%	na	2 mg/L	3.18 * MDL
Solids, Total Dissolved – TDS: (Filterable residues) Method SM2540C, EPA 160.1	Filterable Residue by	+/- 30%	< MDL	90-110%	na	10 mg/L	3.18 * MDL

Parameter	Instrument or method	Relative percent difference (RPD of duplicates)1	Blanks (field)	Measurement systematic error (% recovery or PD)2	Alternative measurement sensitivity (AMS+)3	Method detection limit (MDL)4	Minimum level of quantitation (ML)5
SOP 13 Appendix W Solids, Total Suspended – TSS: (Non-filterable residues) Method EPA 160.2	Drying Oven Non-filterable Residue by Drying Oven	+/- 30%	< MDL	90-110%	na	4 mg/L	3.18 * MDL
SOP 13 Appendix X Solids, Total Volatile – TVS: Method EPA 160.4	Volatile Residue by Muffle Furnace	+/- 30%	< MDL	90-110%	na	na	3.18 * MDL
SOP 13 Appendix Y Solids, Total Settable – RSS: Method EPA 160.5	Determination Settleable Matter in Water Using an Imhoff Cone	+/- 30%	< MDL	90-110%	na	na	3.18 * MDL
SOP 13 Appendix Z Sulfate: (Surface Water) Method EPA 375.2	Sulfate by Colorimetry	+/- 30%	< MDL	90-110%	na	0.5 mg/L	3.18 * MDL
SOP 13 Appendix AA Sulfide: (Water) Method EPA 376.2	Sulfide by Colorimetry	+/- 30%	< MDL	90-110%	na	na	3.18 * MDL
SOP 13 Appendix AB							

1 RPD is the relative percent difference between two or more measurements calculated as percent recovery:

$$RPD = \left[\frac{S_1 - S_2}{(S_1 + S_2)/2} \right] \times 100$$

where,

S_1 = the larger test result value,

S_2 = the smaller test result value.

2 Measurement systematic error is set to 90-100% in the absence of any specific guidance for laboratory rejection criteria

3 AMS+ is the measurement precision uncertainty based on a sample size of 7 in-situ environmental samples (not blank) and 99% confidence. It is the stdev of 7 measurements * 3.018. It was computed as the average AMS+ for 6 site visits to LaVerkin creek, Zion National Park June through November 2006.

4 Defined by National Environmental Methods Index (NEMI) as the lowest "measurable" concentration for an analyte by the specified method (http://web1.er.usgs.gov/nemi/field_def.jsp?method_field_name=DL_VALUE).

5 The ML is generally the MDL multiplied by 3.18.

6 The bacteria detection limit is 0 to 2420 using the most probable number (MPN) technique.

7.1.7.1. Premobilization check to minimize bias

Systematic error in field measured core parameters will be minimized by pre-mobilization instrument check and calibration using NIST traceable standards in the office as outlined by NPS WRD (SOP 5 Appendix A). Guidelines for acceptable ranges of individual calibration parameters are included as rejection criteria in the "pre-mob" page in the Field_forms.xls spreadsheet. If users follow the premobilization evaluation of core parameters MQO's are determined and reported automatically. If MQO's are violated the hydrologist must either determine the source of the problem and ensure it did not jeopardize the previous sampling run data or assume the results from the previous field run are not valid and discard them. The comparability source for core parameter DQO's are from USGS NFM and NPS WRD (Wilde 2005 and Pete Penoyer personal comm.. respectively). Currently the more stringent USGS criteria are programmed into Field_forms.xls. With the exception of DO these criteria are routinely met, thus the less stringent WRD criteria will likely be dropped all together.

7.1.7.2. Field calibration to minimize bias

Multiparameter probes are re-calibrated for DO, SC and pH at each sampling site using NIST traceable standards. Field calibration procedures for UT Park units follow instructions outlined in SOP 8 and the Troll 9000 users manual (SOP 5 Appendix B). The same type of field calibration is performed by hydrologists in CO park units (SOP 8 Appendix E.)

During the sampling run hydrologists compare calibration results to MQO's on-the-fly to check for obvious errors that would indicate instrument malfunction or improper calibration. This is done by taking a post calibration reading of calibration standards for pH and specific conductivity. For example, after calibration with a 1413 us/cm standard, the instrument measurement must be reasonably close to 1413 us/cm while the conductivity sensor is still immersed in the conductivity standard. Likewise after the final pH calibration point at pH 10.01, the instrument should provide a reading very close to 10.01.Typically the post calibration reading will be very close to the standard. If not, the user must resolve the problem before taking ambient measurements. All of this data is recorded in Field_forms.xls so it can be re-evaluated later in the office if necessary.

7.1.7.3. Long term record of bias

All automatically generated reports from pre-mobilization and field calibration become part of the permanent data record by appending them to the electronic data form Field_forms.xls. The pre and post calibration instrument readings for pH and conductivity are recorded on the field form and will be evaluated for trends over time that would indicate systematic error. However, the likelihood of observing such a trend is minimized by replacing sensors when response to known stimulus values exceed acceptance criteria outlined in the pre-mob spreadsheet of Field_forms.xls.

Table 6. Recommended MP instrument post-calibration check-error limits.

CORE PARAMETER	Sensor Measurement (acceptance/rejection criteria)	Optimum Goal/Target (Range) (USGS criteria)
Temperature	± 0.3 º C	± 0.2 º C
[1]Specific Conductance[2]	± 5 µS/cm or ± 3%	± 5 µS/cm or ±3%
pH[3]	± 0.3 S.U.	± 0.2 S.U.
Dissolved Oxygen[2]	± 0.4 mg/L, ± 5% saturation	± 0.3 mg/L, ± 3% saturation

[1]Whichever is greater

[2] Parameter values (SC and DO) may be affected by 2 to 3% with each degree change in temperature

[3] If after a 2–point calibration and performance of any necessary pH sensor maintenance, error criteria are still not met, a 3-point calibration should be performed before sensor is rejected and replaced.

7.2. Decision Rules

The following is based on guidance from the EPA's Measurement Quality Objectives white paper as it pertains to controlling management decision errors – section 6A. (accessed 10/30/2006 http://www.epa.gov/QUALITY/qs-docs/g4-final.pdf)

In practical terms data collected in this program will be used for making resource management decisions. Therefore, the decision rule is stated as a statistical hypothesis test that allows users to minimize the probability of making a false conclusion - (i.e., rejecting the null hypothesis when, in fact, it is true, or failing to reject the null hypothesis when, in fact, it is false). The null hypothesis is the base-line condition, while the alternative hypothesis is a condition you hope to be false. For example,

Ho: the water quality standard is not being exceeded

Ha: the water quality standard is being exceeded

note: in practice for 305(b) reporting and 303(d) listing state by state guidelines are followed to determine if a number of exceedances warrant placing a water body on an impaired list. Check with state water quality divisions for guidance.

A false rejection decision error, or a Type I error, occurs when you reject the null hypothesis when it is actually true. The probability of this error occurring is called alpha (α) and is called the hypothesis test's level of significance.

A false acceptance decision error, or a Type II error, occurs when you fail to reject the null hypothesis when it is actually false. The probability that this error will occur is called beta (β).

Frequently, a false rejection decision error is the more severe decision error, and therefore, criteria placed on an acceptable value of alpha (α) are typically more stringent than for beta (β). However, in the case of a conservation agency like the NPS the better choice is to err on the side of caution and set beta (β) more stringently than alpha (α).

The probability of rejecting the null hypothesis when it is actually false is the *statistical power* of the hypothesis test. Statistical power is a measure of how likely the collected data will allow you to make the correct conclusion that the alternative condition is true rather than the default baseline condition and is a key concept in determining DQOs for decision-making problems. Note that statistical power represents the probability of "true rejection" (i.e., the opposite of false acceptance) and, therefore, is equal to 1-β.

In analysis of water quality data as per guidance from WRD (Irwin 2006 and Irwin personal comm.) it is a good idea to set $\alpha = 0.1$ and $\beta = 0.05$. This is because as a resource conservation agency, the NPS should be more concerned with β (the conservationist's risk) than α (the polluter's risk). We will use the one sample, two-tailed test and set the alternative hypothesis to +30% of the historic mean. In this way we limit the probability of making a false rejection of the null to 10% and the probability of falsely accepting the null to 5%. Our statistical power is therefore $1-\beta = 0.95$

Hypothesis tests are only as reliable as the data used in their evaluation. Therefore, the best way to control the probability of making decision errors is to control the factors that contribute to total study error. These include controlling both measurement and sampling error (usually the larger of the two) and bias. Ways of minimizing these potential sources of error have been discussed in previous sections and other SOP's (2, 3, 4, 5, 8, 9, 10, 12 and 13). Cumulative bias is so important as to warrant is own SOP (18). Another way to minimize the probability of drawing false conclusions is to collect data that has minimal variability, or collect data in such a way as to minimize variability (discussed in protocol narrative section 2 and in site selection and timing SOP 4). Data variability and sample sizes necessary to achieve a high degree of power are discussed in Section 2 of the Protocol Narrative.

Finally data quality assessment should include a holistic evaluation of the data including assurances that

1. proper field methods were followed
2. equipment was in good condition and working properly
3. blanks and reps had acceptable values
4. holding times and sample temperatures were not exceeded
5. data completeness goals were met
6. data was quality controlled and uploaded with appropriate flags if necessary
7. cumulative bias has been evaluated and controlled or normalized

7.3. Handling non-detects

Results cannot be reported for values less than the Method Detection Limit (MDL). Most MDLs are much lower than water quality standards and provide the rationale for confidence in the data. However some parameters particularly nutrients often have extremely low values in pristine waters. It is these water bodies that NPS hopes to keep clean and monitoring these requires an effective method for dealing with parameter values near detection limits of analytical procedures (Irwin 2004). Data values below an MDL will not be presented numerically. Data falling between the MDL and minimum level of quantitation (ML) are considered "semi-quantitative" and can be presented as greater than zero. They are detected but not quantifiable and can be given a flag of be presented as less than or greater than the ML in order to compare it to water quality criteria.

Non-detects will be handled according to Helsel (2005) consistent with the modernized STORET recommendation. Modernized STORET suggests not reporting into a database any value higher than the MDL but lower than the ML. Instead, the detection condition field is set to "Present, below Quantification Limit". With that detection condition, STORET automatically enters "*Present <QL" in the result field. A major advantage of this approach is

that no "estimates" are treated as quantitative when in fact they are not quantitative. In later statistical analyses these data between the two limits are best interpreted using either an interval-censored method (parametric), or a rank-based method (nonparametric) where all in-between values are represented as the same tied rank. Values above the PQL are classified in EPA's modernized STORET database with the detection condition of "Detected and Quantified" This is ideal, and according to EPA STORET Staff, this is optimally the only choice which permits reporting a single number (Irwin 2004).

7.4. Violation of Data Quality Objectives

The data quality objectives are designed to allow data comparability through space and time keeping in mind limitations of the sampling design when drawing conclusions. It is likely at times that data quality objectives cannot be met. In these cases data must either be discarded or considered in isolation of other similar types of data to avoid drawing misleading or erroneous conclusions. This is especially true near the detection limits for laboratory analyses and when evaluating data from different laboratory techniques that attempt to quantify the same parameter.

If measurements do not meet or exceed these criteria they will be discarded and re-runs for laboratory samples will be requested if possible. Appropriate measures will be taken to determine the source of error so that future measurements will comply with these minimum requirements.

8. Training Requirements / Certification

Staff training and evaluation are covered in SOP's 2 and 3 for CO and UT Park units respectively.

9. Documentation and Records

9.1. Utah parks

9.1.1. Field data

1. One file of Field_forms.xls exists for each monthly sampling run. Each site in the Field_forms.xls spreadsheet has its own page that contains all field measurements and calibration information for that site (SOP 8).

2. Both hardcopy and digital versions of Field_forms.xls are taken to the field for recording data. All core parameter data are entered in pencil while at the site to serve as backup in case of memory failure in any of the electronic field devices. But most of the field data including calibration reports are recorded directly into the Field_forms.xls spreadsheet on the Rugged Reader (SOP 8), or are downloaded, edited and appended to Field_forms.xls in the office (SOP 8).

3. All pre-mobilization instrument checks and field calibrations are stored with field measurements of core parameters in electronic form in the Field_forms.xls spreadsheets. Included in these spreadsheets are flow measurements and electronic output from Aquacalc flow meters when they are used.

4. In the office or field, as data is entered or appended to the electronic version of Field_forms.xls quality control for completeness is automatically determined. A visual scan for "finished" in every row of the completeness field indicates a completed site visit. At this point hard copy output is printed for archival purposes.

5. Both the original paper forms used in the field and the completed forms printed after completing all data entry are stored indefinitely with the NCPN hydrologist

6. A series of three rotating backups are made for all field data. Short term backups are made on the PC hard drive as data entry progresses. Daily backups are made on CD when significant data entry occurs on any day, and an archival backup is made on CD after complete QC checks are made. Additionally, data are uploaded to the X-drive in Moab periodically to serve as an off-site backup.

9.1.2. Laboratory data

1. By request and automatically on an annual basis laboratory results are e-mailed in Excel format to the NCPN hydrologist from the UT DWQ data manager (Arne Hultquist, arnehultquist@utah.gov)

2. These data are imported to the NCPN-water data base, linked to site visit core parameter data and with the help of the NCPN data manager are uploaded to NCPN water data base and NPSTORET.

9.2. Colorado parks

9.2.1. Field data

A blank copy of the data form accompanies the hydrologist in the field. All core parameter and calibration data are completed in pencil while at the site. These data are entered into the USGS personal computer field forms (PCFF) in the office.

9.2.2. Laboratory data

In the past data from samples submitted to NWQL were quality control checked by the NWQL laboratory and uploaded to NWIS, the USGS national water quality data base. Presently NWQL data are made available to the BLCA/CURE hydrologist for review before sample holding times expire. He does quality control checks and can then request re-runs on samples if necessary. It is now his responsibility to upload lab and field data to national data bases. There is some backlog since 2005 that will be addressed in the Data Management SOP (14).

9.3. Data management (SOP 14)

Data evaluation for completeness is the responsibility of hydrologists collecting data in the field. Likewise, hardcopy archive and interim digital backup are the responsibility of the individual hydrologists in charge of data collection for different cooperating groups within the NCPN. The NCPN hydrologist with the help of park hydrologists and the NCPN data manager is responsible for corralling and uploading data to NCPN and national data bases.

9.4. Evaluation (SOP 15)

Data evaluation for internal consistency, outliers and quality control checks other than for completeness are the responsibilities of the NCPN hydrologist. After these quality control checks the NCPN hydrologist will upload data to the NCPN-water database. Having one person responsible for this step will ensure consistency in a final quality control check before data is uploaded to WRD STORET and eventually EPA STORET. Additionally, this will provide consistency in reporting values near detection and quantitation limits.

9.5. Reporting (SOP 15)

The NCPN hydrologist with the help of park hydrologists is responsible for annual reports, multi-year comprehensive analysis and reporting, notification of exceedance criteria to State and NPS cooperators and coordination with laboratories and NCPN data manager to ensure data integrity and archive.

10. Sampling Process Design

10.1. Program design

The NCPN monitoring program is designed to sample priority water bodies identified through historic analysis of water quality data that show information gaps and input from park managers and water quality specialists who identified water bodies of concern. Section 2.2 of the monitoring protocol narrative has more information about the process of monitoring program development and site selection.

10.2. Type, frequency and timing of sample collection

CWA compliance and requirements of supporting laboratories in UT largely determined the type and frequency of data collection. The choice to focus sampling on base flow conditions was driven by the need to minimize variability in order to minimize sample sizes required for meaningful statistical analysis.

10.3. Sample site selection

Sample site selection on monitored water bodies can be found in SOP 4

10.4. Site identification

Details regarding sampling site descriptions and long term documentation are found in SOP 4

10.5. Sampling constraints

Weather and unanticipated problems including unsafe travel and sampling conditions may preclude the optimum 12 site visits. Therefore, completeness goals have been set for a minimum of 10 site visits annually in order to obtain data representative of the water year. The exceptions are the Yampa and Green rivers which are too expensive to monitor more frequently.

Safety considerations and emergency contacts are listed in SOP 1.

11. Sampling Methods Requirements

11.1. Parameters

Sampling for core parameters is discussed in SOP 8

Sample collection for laboratory analyses is discussed in SOP 9 and 10 for CO and UT parks respectively.

11.2. Equipment care and maintenance

Preparation of equipment for field work and its care and handling are discussed in SOP 5. A list of equipment and characteristics is provided in table 11-2.

Table 7. Equipment characteristics for NCPN water quality monitoring.

Parameter	Equipment UT Parks	Equipment CO Parks	Container UT Parks	Container CO Parks	Preservative	Hold time
pH	In situ Troll 9000	Hydrolab mini sonde 4a	none	none	none	none
Conductivity	In situ Troll 9000	Hydrolab mini sonde 4a	none	none	none	none
Dissolved oxygen	In situ Troll 9000	Hydrolab mini sonde 4a	none	none	none	none
Temperature	In situ Troll 9000	Hydrolab mini sonde 4a	none	none	none	none
Flow	Baski cutthroat flume, pygmy or price meter, v-notch weir	Pygmy meter	none	none	none	none
Turbidity	Hach 2100P turbidimeter	Hach 2100P turbidimeter	20 ml glass vial	20 ml glass vial	none	4 hours
Nutrients	Analysis performed at lab	Analysis performed at lab	LDPE screw top provided by lab	LDPE screw top, Preservative provided by lab	H_2SO_4 (2ml) Store on ice	7 days
Chemistry	Analysis performed at lab	Analysis performed at lab	LDPE screw top provided by lab	LDPE screw top, Preservative provided by lab	None Store on ice	14 days
Metals	Analysis performed at lab	Analysis performed at lab	LDPE screw top provided by lab	LDPE screw top, Preservative provided by lab	HNO_3 (2ml) Store on ice	14 days

12. Sample Handling and Custody Requirements

Details of sample collection are covered in SOP 9 and 10 for CO and UT Parks respectively.

12.1. Utah Park sample handling and custody

1. For monitoring in UT Park units, samples are labeled in the field with the STORET number, location, date and time of collection, sample type, sampler's name, and method used to preserve sample.

2. Samples are transported from site of collection to a vehicle and immediately placed on ice.

3. Filtering and turbidity measurements are made in the field at the vehicle.

4. On arrival at the office, samples are placed in a refrigerator and cooled 24 hours before shipping on ice in a cooler to the laboratory for analysis.

5. The chain of custody is very short and consists of the collector, shipping company and laboratory. A tracking number for each batch shipped to laboratories is used to verify when samples arrive. This information is kept in the Field_forms.xls spreadsheet.

6. For samples shipped to the UT DWQ laboratory, the Division of Epidemiology and Laboratory Services (DELS) assists users by providing sample containers and instructions for proper sample handling. A table in SOP 13 Appendix A lists available testing methods, respective containers, preservative and proper handling conditions, total holding times, and sample acceptance criteria.

12.2. Colorado Park sample handling and custody

A very similar chain of handling and custody occurs for samples collected in CO Park units. The only significant differences are that hydrologists in CO Park units,

1. use a churn splitter to thoroughly mix samples before sub-sampling and

2. add preservative to sample bottles before sending samples to the National Water Quality Laboratory.

3. The sub-sampling procedure is outlined in USGS NFM 5.1.1.A Churn-splitter Procedure. The filtering technique is outlined in 5.2.1.A Capsule-filter Procedure.

13. Analytical Methods Requirements

The methods used to measure core parameters are listed in SOP 8. The laboratory methods used by the NWQL and UT DWQ are listed in SOP 12 and 13 respectively.

14. Quality Control Requirements

Quality control for precision and accuracy of both field and laboratory measurement has been addressed in section 7 of this document.

15. Instrument/Equipment Testing, Inspection, and Maintenance Requirements

1. Description of instrument testing, care and handling is discussed in SOP 5.

2. Quality control for precision and accuracy of field equipment has been addressed in section 7 of this document.

3. A complete set of replacement parts for the multiparameter probes is kept current and available because parts wear out at inconsistent intervals.

4. Consumables such as gloves, bottles, filters and batteries are ordered as needed at the end of each sampling run so they will be available by the next run.

5. Fresh calibration solutions that have not exceeded expiration dates are kept on hand and ordered at the end of each sampling run as needed.

6. Log books are kept for the multi parameter instruments and flow meters to record maintenance and other issues that may affect readings. This is critical for interpretation of long term trends where changes in sensors or other factors could affect instrument performance.

16. Instrument Calibration and Frequency

Field instruments are checked for integrity and calibrated before each sampling run in a pre-mobilization check (SOP 5). Field instruments are calibrated for pH, DO and conductivity at each sampling site.

17. Inspection/Acceptance Requirements for Supplies

All reagents and standards are National Institutes of Standards and Technology (NIST) traceable and measurement instrumentation meets NPS WRD and USGS specifications. It is the responsibility of each field crew leader to order new supplies of consumables and replacement parts as necessary at the end of each sampling run so that adequate supplies within their expiration date are on hand for the next monthly sampling run.

18. Data Acquisition Requirements

1. In addition to direct measurements and laboratory analysis of samples, the Water Quality monitoring program uses data from USGS gauging stations when proximal and in-stream flow measurement is not possible.

2. Site descriptions rely on GPS readings to precisely locate sampling site on a water body.

3. USGS 7.5 minute topographic maps are also used to describe site locations.

4. Historic data compiled in the NCPN-water data base was collected from a wide range of sources and is used to describe the range and variability of water

parameters. Source and quality control flags alert users of this data to its perceived quality, and users should be wary of data quality that does not have known or implied levels of quality.

19. Data Management

A general discussion of data management and data custody is addressed in section 9 of this document. More detail on data management can be found in SOP 14

20. Assessment and Response Actions

1. Evaluation of field, lab and data management activities of cooperating groups within the NCPN will be a routine and on-going responsibility of the NCPN hydrologist as data "round-up" progresses.

2. Evaluation of NCPN hydrologist activities in the field, and data management and reporting will be made by NPS WRD personnel and USGS cooperators, and the NCPN network coordinator. This will occur within 1 year of monitoring start-up and bi-annually afterward, or as needed until MQO's defined in section 7 of this document are met.

3. The NCPN hydrologist will be responsible for checking completeness and internal consistency using automated routines that are being developed on an ad hoc basis. These routines will check for exceedances of water quality criteria, chemical balances, and spurious values that have no credible explanation. It will be up to the NCPN hydrologist to follow-up and determine if errors can be corrected, or if data should be discarded.

4. All cooperators with the UT DWQ are required to attend an annual sample collection refresher provided by UT DWQ laboratory.

21. Reports

1. The NCPN hydrologist will prepare an annual report that will be distributed each February using the most recent quality controlled / finalized data. Reports will be forwarded to the NCPN technical committee, park resource management staff where sampling occurred, and the NPS WRD.

2. The report will list exceedance occurrences, the probable cause, and potential outcomes of water quality degradation.

3. The report will present statistical summary of range and variability of water quality parameters and interpretation of observations relative to issues of concern for each water body.

4. Additionally, the report will contain results of QC audits and internal assessments.

22. Data Review, Validation, and Verification Requirements

There are three levels of data review for all data collected by NCPN monitoring efforts.

1. Field review of core parameters is built into the Field_forms.xls spread sheet. Review of data as it is collected by hydrologists in the field is paramount to successfully collecting representative field data. The test for "representativeness" is based on the reported range in parameter values found on in the field folder and professional judgment. All field data must pass rigorous quality control requirements for bias and repeatability listed in SOP 5 Table 1. Corrective measures to ensure un-biased and precise measurement of core parameters can only be made while the operator is in the field.

2. The next level of data review occurs at the laboratory where results are screened for spurious values before sending data to NCPN hydrologists for review. Prompt review (< 7 days after receipt of data) of laboratory data by cooperating hydrologists is critical to ensure re-runs are possible before samples are discarded or mature beyond their holding times.

   Staff at UDWQ will conduct an internal review of laboratory results as they are received from the laboratory that includes the following:

   a. Screening of results against standards to catch exceedances and outliers (effective for parameters for which there are standards).

   b. Comparison of duplicate samples using a 10% threshold for further investigation (turbidity and suspended solids use 20% threshold).

   c. Screen all the blanks and audit standards, and require lab to respond to every inconsistent result.

   d. Compare dissolved phosphorus to total phosphorus (dissolved should be less than total).

3. NPS staff will also conduct an initial review of data to ensure quality and to make note of results that may influence the conduct of the monitoring program. This will be facilitated by submitting a request to UDWQ for a "Station Visit Report" with each upload of field data. The state will reply via e-mail when results are received from the laboratory. NPS field water monitoring staff will be responsible for reviewing this data in a timely manner (2 to 3 weeks), and responding to the UDWQ with requests for "re-runs" or clarification from the lab. Data that appears to be sound on initial review will be entered into a local STORET interface called SIM (analogous to the NP-STORET interface).

4. The final level of review occurs when the NCPN hydrologist performs automated statistical checks on bounds and internal consistency. Other checks that occur at this stage include ensuring that data,

a. contain all required fields,

b. have encoded valid values from constrained look-up lists where specified, and

c. are in the correct format (text in text fields and values in numeric fields, etc.).

At any point in the data collection and review process hydrologists can make decisions to discard or flag data that they know is not representative.

23. Validation and Verification Methods

1. Spot checks will be performed by cooperating NCPN hydrologists to determine consistency between field sheets and the digital database with an eye toward agreement and data gaps.

2. Additional checks on data quality will be made as data are analyzed in the analysis and reporting phase. Often subtle trends or occasional data outliers are not detected until examined in context with other data collected from a site in the form of graphs, and tables. At this stage it is the collective responsibility of all cooperating hydrologists to track down potential sources of error and make corrections where appropriate or flag data as necessary. However, no data will be removed from the database unless a compelling and rational reason exists to explain why it should be removed.

3. A Matlab program has been written to evaluate data collected with Field_forms.xls that plots monthly trends with compliance limits and computes descriptive statistics. This provides a tool to rapidly evaluate core parameter field conditions for outliers.

24. Reconciliation with Data Quality Objectives

To ensure data collection, compilation and review meet the project objectives an important step in the annual report preparation will include summarizing accuracy, precision, and completeness. Accuracy and precision will be calculated based on results from the premobilization instrument check and calibration and from individual field calibration parameters.

24.1. Effectiveness

Effectiveness, E will be calculated as
$E = (U / C) \times 100$
where
U = number of usable parameter values
C = number of parameter values collected

24.2. Completeness

Completeness C, will be calculated as

C = (U / P) x 100
where
U = number of usable parameter values
P = number of potential parameter values collected (typically 12 for monthly sampling)

These results will be used to make corrective actions that will improve the quality and efficiency of data collection where necessary.

25. Citations

Eaton, A.D., L.S. Clesceri, and A.E. Greenberg, eds., *Standard Methods for the Examination of Water and Wastewater,* 19th edition, Washington, D.C.: American Public Health Association, American Water Works Association, and Water Environment Federation, 1995.

EPA 2006, Guidance on Systematic Planning Using the Data Quality Objectives Process EPA QA/G-4, Office of Environmental Information Washington, DC 20460 accessed at:

Faires, L.M., 1993. Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Metals in Water by Inductively Coupled Plasma-Mass Spectrometry: U.S. Geological Survey Open-File Report 92-634, 28p.

Fishman, M.J., ed., 1993. Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments: U.S. Geological Survey Open-File report 93-125, 217p.

Fishman, M.J., and Friedman, L.C., 1989, Methods for determination of inorganic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, 545 p. Method ID: I-2587-89.

Garbarino, John R., 1999. Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Dissolved Arsenic, Boron, Lithium, Selenium, Strontium, Thallium, and Vanadium using Inductively Coupled Plasma-Mass Spectrometry: U.S. Geological Survey Open-File Report 99-093, 31p.

Irwin, R.J. 2004. Draft Part B of Aquatic Habitat Park Service guidance for Park Service Vital Signs Monitoring. Planning Process Steps: Issues To Consider And Then to Document In A Detailed Study Plan That Includes A Quality Assurance Project Plan (QAPP) And Monitoring "Protocols" (Standard Operating Procedures), Available on Internet at http://science.nature.nps.gov/im/monitor/protocols/wqPartB.doc

Irwin, R.J. 2006. Draft Part B lite (Just the Basics) QA/QC Review Checklist for Aquatic Vital Sign Monitoring Protocols and SOPs, National Park Service, Water Resources Division. Fort Collins, Colorado, distributed on Internet only at
http://www.nature.nps.gov/water/Vital_Signs_Guidance/Guidance_Documents/PartBLite.pdf

Wilde, F.D., January 2005, Preparations for water sampling (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A1, accessed 8/7/06, from http://pubs.water.usgs.gov/twri9A/.

26. Appendix A: State of Utah Department of Environmental Quality Division of Water Quality Quality Assurance Program Plan for Water Monitoring Programs

Copied verbatim without alteration for reference. Not all sections are relevant to the NCPN water quality monitoring program (eg. fish, periphyton and plankton)

OLENE S. WALKER

Governor

State of Utah

Department of
Environmental Quality

Dianne R. Nielson, Ph.D.

STATE OF UTAH DEPARTMENT OF ENVIRONMENTAL QUALITY DIVISION OF WATER QUALITY QUALITY ASSURANCE PROGRAM PLAN FOR WATER MONITORING PROGRAMS

October 2003

A-1: Quality Assurance Program Plan Identification

Document Title: The Quality Assurance Program Plan for the Utah Division of Water Quality Water Monitoring Programs

Organization Title:	Division of Water Quality
Address:	Department of Environmental Quality Division of Water Quality 288 North 1460 West Salt Lake City, Utah 84114-4870
Responsible Official:	Don Ostler Phone (801) 538-6146
Quality Assurance Officer:	Richard Denton Phone (801) 538-6055

Plan Coverage: This is a document describing the Utah Division of Water Quality, Quality Assurance Plan and Standard Operating Procedures. The plan covers all water sampling done by the Division of Water Quality.

Approval for Agency: Name: Don Ostler
Title: Director
Division of Water Quality

Signature: _____ Date: _____

A-2: TABLE OF CONTENTS

Section

Title Page and Approval Page A-1

Table of Contents A-2

Organization and Responsibility A-4

Introduction, Problem Definition/Background A-5

Program/Project/Task Description A-6

Quality Assurance Objectives for Measurement Data in Terms of Precision and Accuracy A-7

Sampling Process Design B-1

Sampling Methods Requirements B-2

Sample Handling, Custody and Form Requirements B-3

Analytical Methods Requirements B-4

Performance and System Quality Control Checks and Audits B-5

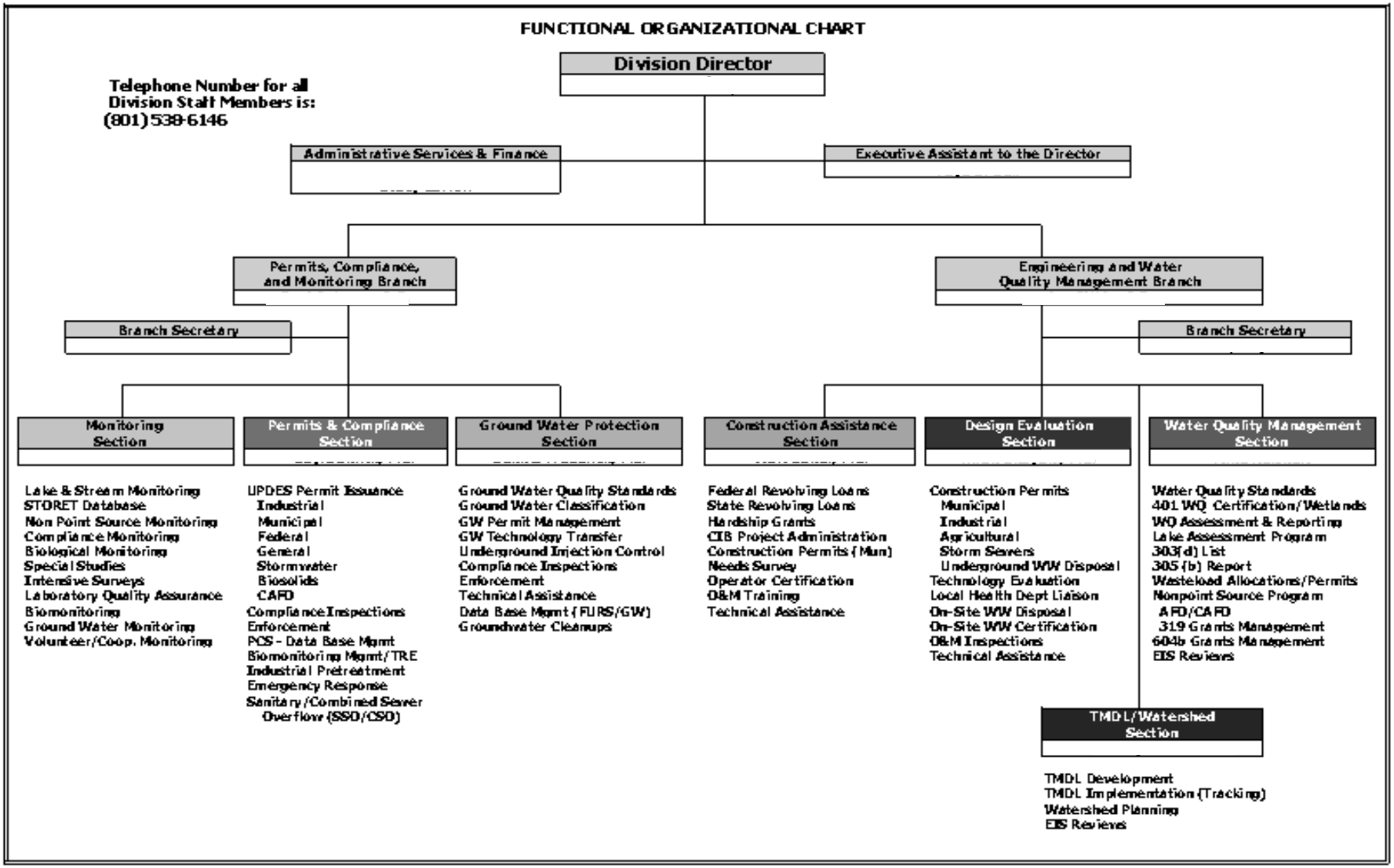
Instrument Calibration and Frequency B-7

Specific Routine Procedures Used to Assess Data Precision, and Response Actions C-1

Data Review, Validation, and Verification Requirements D-1, D-2, D-3

DIVISION OF WATER QUALITY

Department of Environmental Quality



A-5: Introduction, Problem Definition/Background

Environmental Protection Agency (EPA) policy requires participation in a centrally managed quality assurance program by all agencies whose monitoring and measurement efforts are supported or mandated through contracts, grants, regulations, or other formalized agreements with the EPA. To implement EPA policy, the Utah Division of Water Quality has prepared a Quality Assurance Program Plan (QAPP) covering all sampling that generates and processes environmental data for the Division of Water Quality.

The Division of Water Quality water assessment is confined to field analyses for a limited number of parameters; therefore, this document does not fully address the internal Quality Assurance program of laboratories doing the analysis. The State of Utah, Division of Laboratory Services Quality Assurance Program Plan is included for reference purposes in the Quality Assurance and Standard Operating Procedures Manual for the Utah Division of Water Quality as appendix 13. This document will be confined to the quality assurance of sampling, sample handling, field techniques, field analyses, and data treatment from the time samples are collected until the samples are submitted to the laboratory and after the results are reported until they are permanently stored.

The Division of Water Quality is responsible for various programs that include monitoring as an essential task. The rightness of the decisions made using that data is dependent on the quality of the data. The accurate assessment of stream quality for 305b reporting and 303d listing; and the effectiveness of water quality best management practices, pollution prevention measures, and pollution controls is determined by these analyses. Therefore, the success of the various programs UDWQ administers is also dependent on the quality of the data. The details of each program are outlined in the individual QAPP's for each program as well as the State of Utah Division of Water Quality Quality Assurance and Standard Operation Procedures Manual.

A-6: Program/Project/Task Description

Programs the State of Utah Division of Water Quality collects data for:

1: Section 305(b) of the Clean Water Act assessment program.
2: Section 319 of the Clean Water Act program.
3: The Total Maximum Daily Load Assessment program.
4: The Waste Load Allocation Program.
5: The Utah Pollution Discharge Elimination Systems oversight program.
6: The Clean Lake program.
7: The Groundwater Monitoring Program.
8: The Cooperative Agency Monitoring Program.

Several of these programs overlap in a variety of ways. For instance, all data collected for the various program can be used for the 305b assessment program. Each of these programs has similar data quality objectives. This fact is apparent in each of their individual QAPPs. Similar sections of each QAPP reference the same section of the Division of Water Quality Quality Assurance and Standard Operating Procedures Manual.

Several of these larger eight programs drive smaller projects. These smaller projects generally support the program and are spatially and temporally limited. The State of Utah participates in projects as project needs become a priority and funding becomes available. Site-specific analyses plans (SAP) are available on a project-by-project basis in the QAPP associated with that project. Although certain projects may require methodologies or techniques not commonly used, generally the methodologies and techniques are very similar between programs and projects.

The common goal of all these projects is to compare water quality data to Standards of Quality for Waters of the State (317-2) and to make accurate assessments of water quality. The type of data can be chemical, physical and/or biological in origin, but a certain amount of quality must be associated with each data element. The goal of this document is to detail the quality assurance practices that the State of Utah Department Environmental Quality applies to insure the quality and reproducibility of methodologies and analyses for most data elements.

A-7: Quality Assurance Objectives for Measurement Data In Terms of Precision and Accuracy

The measurement data should provide adequate precision and accuracy for each program objective. All data generated will be reported in units consistent with data generated by other organizations reporting similar analyses to allow comparability of data among organizations.

26.1.1.1. A-7.1 Chemical Measurement Data Quality Objectives

Specific chemical data quality objectives for accuracy precision and completeness for laboratory analyses are discussed in the Division of Laboratory Services Quality Assurance Program Plan (QAPP), appendix 13 of the Quality Assurance and Standard Operating Procedures Manual. Specific chemical data quality objectives for accuracy and precision of sampling are for measurements to fall within a 95% confidence interval around the true value. The confidence interval for each parameter except total suspended solids (TSS) and phosphorus determinations is based on prior knowledge of the measurement system and is generated from the EPA publication "Estimation of Generic Acceptance Limits for Quality Control Limits for Use in a Water Pollution Laboratory" May, 1991. If statistics are not available in the document, the EPA document entitled "Estimation of Generic Acceptance Limits for Quality Control Purposes in a Drinking Water Laboratory" will be used. For parameters not listed in either EPA document, the data quality objectives will be the same as those listed in the Division of Laboratory Services QAPP. The EPA documents are in appendix 14 and the Division of Laboratory Services QAPP is in appendix 13 of the Quality Assurance and Standard Operating Procedures Manual. The data quality objectives for TSS and phosphorus determinations are also based on prior knowledge of the measurement systems. TSS has a requirement of +/- 20 % for precision and accuracy. Phosphorus determinations for both total and dissolved total phosphorus have a two-tiered requirement. Determinations of phosphorus greater than .1 mg/l have a precision and accuracy requirement of +/- 20 %. Phosphorus determinations of less than .1mg/l have precision and accuracy requirement of +/- .02 mg/l.

26.1.1.2. A-7.2 Physical and Habitat Measurement Data Quality Objectives

Flow measurement can be determined by several means. At sites where applicable gauging stations exist (USGS or other entity), flow determinations will be acquired from the entity responsible for the station data. At sites where sampling personnel directly measure flow, flow determinations will have a precision and accuracy requirement of +/- 10%.

Specific data quality objectives for habitat and other physical measurement data are presented in the
methods documentation. The sampling methodologies and analyses techniques generally used are from *Standard Methods* or other EPA documents. The collection techniques, standard operating procedures and an overview of analytical methodologies are presented in Section 13 of the Quality Assurance and Standard Operating Procedures Manual.

26.1.1.3. A-7.3 Benthic Measurement Data Quality Objectives

Specific data quality objectives for benthic determinations are presented in the methods documentation. The National Aquatic Monitoring Center at Utah State University is currently performing the benthic sample analyses. The collection techniques, standard operating procedures and an overview of analytical methodologies are outlined in Section 8 of the Quality Assurance and Standard Operating Procedures Manual. The methodologies used are detailed in the National Aquatic Monitoring Center documentation, which is included as Appendix 19 of the Quality Assurance and Standard Operating Procedures Manual.

26.1.1.4. A-7.4 Periphyton and Phytoplankton Measurement Data Quality Objectives

Specific data quality objectives for periphyton and phytoplankton determinations are presented in the methods documentation. The sampling methodologies and analyses techniques generally used are from *Standard Methods* or other EPA documents. The collection techniques, standard operating procedures and an overview of analytical methodologies is presented in Section 9.0 of the Quality Assurance and Standard Operating Procedures Manual. The sample analyses are currently contracted to a private entity.

26.1.1.5. A-7.4 Fish Measurement Data Quality Objectives

Specific data quality objectives for fish determinations are presented in the methods documentation. Sampling methodologies and analyses techniques are from USEPA EMAP protocol. Species identification, length and species counts are determined by a "one pass" method over a predetermined reach length and the effort in time noted.

B-1: Sampling Process Design

The sampling design for every site is dictated by the project or projects. The projects, sites associated with the projects, and individual site analyses plans are listed in the State of Utah, Division of Water Quality Strategic Monitoring Guide. The guide is included in the Quality Assurance and Standard Operating Procedures Manual as section 4.3. This document is updated every fiscal year, and is implemented on the July 1 of every year. Projects that are scheduled for implementation on a rotational basis are also listed in that document, even if it is not scheduled for the current fiscal year. The site analyses plans from the most recent rotation are listed in the guide. Further information on rational and justification for site analyses plans in the Strategic Monitoring Guide can be found in individual quality assurance project plans and various sections of the Quality Assurance and Standard Operating Procedures Manual.

B-2: Sampling Methods and Procedures

26.1.1.6. B-2.1 Sampling Methods and Procedures for Laboratory Chemical Analyses

Water sample collection and preservation techniques for chemical analyses are presented in Section 4 of the Quality Assurance and Standard Operating Procedures Manual, and Section 7 the Division of Laboratory Services QAPP. Standard procedures and practices for the collection and preservation of chemical samples are detailed in those documents. The preservation techniques will conform to EPA Rules and Regulations listed in Federal Register VOL 49, No. 209.

26.1.1.7. B-2.2 Sampling Methods and Procedures for Field Chemical Analyses

Field measurement methods for chemical parameters are presented in Section 11 of the Quality Assurance and Standard Operating Procedures Manual. Standard procedures for the calibration and use of instruments used in field analyses are detailed in that document.

26.1.1.8. B-2.3 Sampling Methods and Procedures for Field Measurement of Physical Parameters

Field measurement methods for physical parameters are presented in Section 12 and Section 13 of the Quality Assurance and Standard Operating Procedures Manual. Standard procedures for the collection of physical measurements are detailed in that document.

B-2.4 Sampling Methods and Procedures for Benthic Analyses

Benthic sample collection techniques are presented in Section 8 of the Quality Assurance and Standard Operating Procedures Manual and the National Aquatic Monitoring Center Standard Operating Procedures and Quality Control documentation. The National Aquatic Monitoring Center documentation is presented as appendix 19 of the Quality Assurance and Standard Operating Procedures Manual. Standard procedures and practices for the collection and preservation of benthic samples are detailed in those documents.

B-2.5 Sampling Methods and Procedures for Phytoplankton and Periphyton Analyses

Phytoplankton and periphyton sample collection techniques are presented in Section 6 and Section 9 of the Quality Assurance and Standard Operating Procedures Manual. Standard procedures and practices for the collection and preservation of phytoplankton samples are detailed in that document.

B-2.5 Sampling Methods and Procedures for Fish Analyses

Fish sample collection methods are presented in Section 10 of the Quality Assurance and Standard Operating Procedures Manual.

B-3: Sample Custody and Form Identification

B-3.1 Sample Custody and Forms for Routine Analyses

For routine analyses (those not involved in possible litigation) the forms and codes used are presented in Section 14 of the Quality Assurance and Standard Operating Procedures Manual. Samplers will adhere to sampling and preservation techniques presented in the Quality Assurance and Standard Operating Procedures Manual. Samplers will prepare and complete all forms before samples will be delivered to the laboratory.

26.1.1.9. B-3.2 Sample Custody and Forms for Analyses Involved in Litigation

For samples that could possibly be involved in litigation procedures, chain of custody procedures will be followed in addition to procedures for routine sampling. Chain of custody procedures are presented in Section 15 of the Quality Assurance and Standard Operating Procedures Manual.

B-4: Analytical Methods Requirements

B-4.1 Analytical Methods for Laboratory Chemical Analyses

Analytical methods that are used for laboratory chemical analyses are listed in the Division of Laboratory Services Quality Assurance Program Plan (QAPP), appendix 13 of the Quality Assurance and Standard Operating Procedures Manual. The analytical methods in used generally are the same as listed in 40 CFR Part 136. Any deviation in analytical methodologies will be described in detail in the site-specific sampling and analysis plans.

B-4.2 Analytical Methods for Field Chemical Analyses

Analytical methods that are used for field chemical analyses are listed in Section 11 of the Quality Assurance and Standard Operating Procedures Manual.

B-4.3 Analytical Methods for Field Measurements of Physical Parameters

Analytical methods that are used for physical parameters are presented in Section 12 and Section 13 of the Quality Assurance and Standard Operating Procedures Manual.

B-4.4 Analytical Methods for Benthic Analyses

The National Aquatic Monitoring Center at Utah State University is currently performing the benthic sample analyses. Analytical methods that are used for benthic analyses are listed in Section 8 of the Quality Assurance and Standard Operating Procedures Manual and the National Aquatic Monitoring Center documentation, which is included as appendix 19 of the Quality Assurance and Standard Operating Procedures Manual.

B-4.5 Analytical Methods for Phytoplankton and Periphyton Analyses

Analytical methods that are used for phytoplankton and periphyton analyses are listed in Section 6 and Section 9 of the Quality Assurance and Standard Operating Procedures Manual.

B-4.6 Analytical Methods for Fish Analyses

Analytical Methods used for pesticide and heavy metal analysis in fish tissue follow *Standard Methods* and our outlined in the Division of Laboratory Services Quality Assurance Program Plan (QAPP), appendix 13 of the Quality Assurance and Standard Operating Procedures Manual.

B-5 Performance and System Quality Control Checks and Audits

26.1.1.10. B-5.1 Quality Control Checks and Audits for Laboratory Chemical Analyses

B-5.1.1 Performance Evaluating Audits.

The quality assurance officer will submit quality control samples and blind audit samples quarterly for routinely analyzed parameters to the Division of Laboratory Services. A "blank" sample will also be submitted quarterly.

B-5.1.2 Performance Evaluating Duplicate Sampling.

Duplicate samples will be collected at a frequency rate of 4 % frequency rate or greater. Most surveys will have a minimum of one duplicate. A volume of water will be collected in a container to allow filling of two sets of sample containers simultaneously. Both laboratory known duplicate and blind duplicate sampling will be performed. Dummy sites with storet numbers will be used to submit blind duplicated samples to the laboratory. Sites will be chosen to insure duplication of all parameters being analyzed for that survey.

B-5.1.3 Performance Evaluating Field Blanks

Field blanks will be submitted to the laboratory at a frequency rate 1% or greater. Most surveys will have a minimum of one field blank. Deionized water will be taken from the laboratory with the monitoring team and handled in the same manner as other routine samples. During the survey the blank water will be placed in the sample bottles required for submission to the laboratory. Filtering will take place at that time if necessary. All parameters being monitored during that survey will be analyzed for.

B-5.2 Quality Control Checks and Audits for Field Chemical Analyses

Comparing instrument results for water with known values achieves performance evaluation of field chemical analyses. Either standard reference material of known quality, or alternative analysis methodologies are used to determine the known values. Details of field instrument quality control checks are presented in Section 11 of the Quality Assurance and Standard Operating Procedures Manual.

B-5.3 Quality Control Checks and Audits for Field Measurements of Physical Parameters

Field personnel and the quality assurance officer perform quality control checks on field measurements of physical parameters. Field personnel check all forms and results for completeness
and accuracy before submittal to the quality assurance officer. The quality assurance officer also reviews all physical data before transfer to the database.

B-5.4 Quality Control Checks and Audits for Benthic Analyses

The National Aquatic Monitoring Center at Utah State University is currently performing the benthic sample analyses. The quality assurance officer at the laboratory performing the benthic analyses performs numerous quality control checks and audits on all results before they are reported. Procedures performed at the benthic laboratory are outlined in Section 8 of the Quality Assurance and Standard Operating Procedures Manual and detailed in the National Aquatic Monitoring Center's benthic laboratory documentation, which is included as appendix 19 of the Quality Assurance and Standard Operating Procedures Manual.

26.1.1.11. B-5.5 Quality Control Checks and Audits for Phytoplankton and Periphyton Analyses

Not applicable at this time.

26.1.1.12. B-5.6 Quality Control Checks and Audits for Fish Analyses

Not applicable at this time.

B-7 Instrument Calibration and Frequency

B-7.1 Calibration Frequency and Procedures for Laboratory Chemical Analyses

Analytical instrumentation calibration and frequency procedures used by the Division of Laboratory Services are detailed in the Division of Laboratory Services QAPP. Division of Laboratory Services QAPP is in Appendix 13 of the Quality Assurance and Standard Operating Procedures Manual.

B-7.2 Calibration Frequency and Procedures for Field Chemical Analyses.

Calibration standards for field measurements parameters will be prepared in accordance with the approved EPA methodology. These standards will be verified by comparison with standard reference materials provided by the quality assurance officer. Standards that do not provide standard reference materials measurement values within the 95% confidence interval for the parameter will not be used for calibration. Data from prepared standards will be documented on calibration forms. Standard protocol for instrument calibrations varies between instruments. At a minimum manufactures recommended calibration frequency and procedures will be used. All instruments will be calibrated before deployment in the field and will be calibrated every day they are used in the field. Calibration procedures of field instruments for chemical parameters are presented in Section 10 of the Quality Assurance and Standard Operating procedures Manual. The manufacturers' specifications are also included in the appendices of that document.

B-7-3 Calibration Frequency and Procedures for Field Measurements and Physical Parameters

At a minimum manufactures recommended calibration frequency for portable current measuring instruments will be used. Manufactures specifications and procedures will be used to calibrate portable current measuring instruments. At a minimum manufactures recommended calibration frequency for other instruments use for measuring physical parameters will be used. The manufacturers specifications are included in the appendices of the. Quality Assurance and Standard Operating Procedures Manual.

B-7.4 Calibration Frequency and Procedures for Benthic, Phytoplankton, Periphyton and Fish Analyses

Calibration procedures of analytical instrumentation used by laboratories performing benthic, phytoplankton, periphyton, and fish analyses are detailed in Quality Assurance Project Plans of each laboratory performing biological analyses.

C-1: Specific Routine Procedures Used to Asses Data Precision, Accuracy, Completeness and Corrective Action.

C-1.1 Chemical Data

Section 17 of the Quality Assurance and Standard Operating Procedures Manual also contains the following documentation and may contain greater detail.

C-1.1.1 Field Data.

Field Data Will Be Assessed Continuously. If a dissolved oxygen value greater than the 100% saturation level is measured, an immediate calibration of the instrument will be required and the site reanalyzed. If a pH value less than 6.5 or greater than 9.0 is measured an immediate calibration of the instrument will be required and the site reanalyzed. If a specific conductance value greater than 10 times or less than 1/10 the standard is measured, an immediate calibration of the instrument with a standard of proper magnitude will be required and the site reanalyzed. The quality assurance coordinator and quality assurance officer will assess field data for an entire run on a weekly basis.

C-1.1.2 Routine Laboratory Data.

After results are received from the laboratory, a program that compares the measured values to water quality standards analyzes all samples from sites with use classifications. The quality assurance officer and quality assurance coordinator review this report for accuracy and completeness before submittal to division personnel. The quality assurance officer reviews all other laboratory reports for accuracy and completeness.

C-1.1.3 Duplicate Sample Data.

After all duplicate sample data is received from the laboratory; the duplicate measurements will be compared to a 95% confidence interval generated around the original site value except total suspended solids (TSS) and phosphorus. The 95% confidence interval is generated from regression equations published in the EPA document "Estimation of Generic Quality Control Limits for Use in a Water Pollution Laboratory". If statistics are not available in the document, the document from the EPA entitled "Estimation of Generic Acceptance Limits for Quality Control Purposes in a Drinking Water Laboratory" will be used. If statistics are not available in either document, the Division of Laboratory Services Quality Assurance Project Plan quality assurance goals will be used. In any case the most current documentation available will be used. The data quality objectives for TSS and phosphorus determinations are also based on prior knowledge of the measurement systems. TSS has a requirement of +/- 20 % for precision and accuracy. Phosphorus determinations for both total and dissolved total phosphorus have a two-tiered requirement. Determinations of phosphorus greater than 0.1 mg/l have a precision and accuracy requirement of +/- 20 %. Phosphorus determinations of less than 0.1mg/l have

precision and accuracy requirement of +/- 0.02 mg/l. The data will be evaluated with every duplicate sample as it is received from the laboratory. The data will also be collected and evaluated yearly and reported to the quality assurance coordinator in writing before the end of the following year.

C-1.1.4 Performance Evaluation Audits.

After all quality control sample data are received from the laboratory, the sample measurements will be compared to a 95% confidence interval generated around the true value. The 95% confidence interval is generated form regression equations provided with the audit series. If statistics are not available with the audit series, the EPA publication "Estimation of Generic Quality Control Limits for Use in a Water Pollution Laboratory" will be used. If statistics are not available in the document, the document from the EPA entitled "Estimation of Generic Acceptance Limits for Quality Control Purposes in a Drinking Water Laboratory" will be used. If statistics are not available in either document, the Division of Laboratory Services Quality Assurance Project Plan quality assurance goals will be used. In all cases the most current documentation will be used. The samples will be submitted quarterly. They will be evaluated and reported to the quality assurance coordinator in writing before the end of the following quarter.

C-1.2 Biological Data.

Procedures used to assess data from biological sampling are contained in Section 8, Section 9, and Section 10 of the Quality Assurance and Standard Operating Procedures Manual. Standard assessment tools detailed in each biological analyses method will be used.

C-1.3 Physical Assessment Data.

Procedures used to assess physical data is contained in and Section 13 of the Quality Assurance and Standard Operating Procedures Manual. Standard assessment tools detailed in each physical measurement method will be used.

C-1.5 Corrective actions

Corrective actions will be initiated as a result of the following quality assurance activities:

1. Daily field data assessment found to be beyond control limits.
2. Unacceptable results on performance evaluation audits.
3. Unacceptable performance found in system audits.
4. Previously reported results found to be in error.

It is imperative that early and effective corrective action be taken when control data fall outside of acceptable limits. Since the samplers and laboratory are responsible for recording all quality assurance data on forms daily, they will be the first to determine that a method is out of control and will initiate the appropriate immediate corrective measures necessary to bring data within

control limits. Any corrective action by the samplers will be documented and the quality assurance office will maintain completed forms.

Corrective action will be initiated by the quality assurance officer as a result of unacceptable performance or system on audits and, and if appropriate, in cooperation with the Division of Laboratory Services quality assurance officer.

D-1: Data Reduction and Reporting D-1, Verification D-2, Validation D-3

D-1.1 Chemical Data Reduction and Verification.

The quality assurance officer verifies all water chemistry data received by the Division of Water Quality. Chemical data verification is done through a variety of process throughout the data reduction and storage process. Before monitoring is performed field sheets with site-specific field and laboratory analysis request forms are computer generated to insure completeness and accuracy. The referential integrity associated with the STORET database helps verifies sites and analysis requests. The quality control officer reviews field sheets before any fieldwork is initiated. Appropriate sample preservation is checked during the laboratory submittal sample process and recorded for each sample on the field sheet. Laboratory data reduction and verification processes are detailed in the Division of Laboratory Services QAPP. The Division of Laboratory Services QAPP is included as Appendix 13 of the Quality Assurance and Standard Operating Procedures Manual. After chemical data is received from the laboratory, several procedures are used to reduce, report and verify data. Chemical data analyzed at the Division of Laboratory Services is routinely assessed and verified using data reduction, verification, and validation procedures that are presented in Section 16 of the Quality Assurance and Standard Operating Procedures Manual. Data processing procedures, flow charts and examples of reports are detailed in that document.

D-1.2 Biological Data Reduction.

The quality assurance officer verifies all biological data received by the Division of Water Quality. The type of biological data and analyses dictate the review, verification and validation process that are used. The verification and validations assessments associated with the biological method are generally used. The methods for collection and analyses of biological data collected by the State of Utah are presented in Section 8, 9, and 10 of the Quality Assurance and Standard Operating Procedures Manual.

D-1.3 Physical Data Reduction and Verification.

The quality assurance officer verifies all physical data received by the Division of Water Quality. The type of biological data and analyses dictate the review, verification and validation process that are used. The verification and validations assessments associated with the physical measurement method are generally used. The methods for collection and analyses of physical data collected by the State of Utah are presented in Section 13 of the Quality Assurance and Standard Operating Procedures Manual.

27. Appendix B: Utah Environmental Testing Laboratory Certification Bulletin

Division of Epidemiology and Laboratory Services
Bureau of Laboratory Improvement
Environmental Laboratory Certification Program (ELCP) Volume 09, Number 02
June 2003

Matrix Types
The term matrix comes up in a couple different places when discussing laboratory certification. Field of Accreditation Matrix - When talking about matrix as it relates to certification [matrix, method/technology, analyte], these matrix definitions shall be:

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source.

Non-Potable Water: any aqueous sample
excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and TCLP or other extracts.

Solid and Chemical Materials: includes soils, sediments, sludges, products and by-products of an industrial process that results in a matrix not previously defined.
To complete the list from the NELAC standards, although not used in the Utah program:

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Air and Emissions: whole gas or vapor
samples including those contained in flexible or rigid wall containers and the extracted
concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.
(NELAC)

Quality System Matrix – When talking about a matrix used for purposes of batch and quality control requirements. A matrix is considered one of the following:

Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface
water, groundwater, effluents, and TCLP or
other extracts.

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

For the certification program the differentiation of matrix is done in parallel with the separate regulatory programs. That means certificates for Safe Drinking Water Act testing includes all analytes and methods for Drinking Water matrix,

Clean Water Act testing for Non-Potable Water matrix, and Resource Conservation and Recovery Act testing for Solid and Chemical Material matrix.

Internal Audits

NELAC 2001 5.5.3.1 Internal Audits

A slight clarification for the current NELAC standard is found in the 2003 standard at

5.4.13.1 *The laboratory shall periodically, In accordance with a predetermined schedule and procedure, and at least annually, conduct internal audits of its activities to verify that its operations continue to comply with the requirements of the quality system and this Standard. The internal audit program shall address all elements of the quality system, including the environmental testing activities. It is the responsibility of the quality manager to plan and organize audits as required by the schedule and requested by management. Such audits shall be carried out by* 2 *trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out.*

The purpose of the internal audit is to evaluate the effectiveness of the laboratory in complying with the laboratory's own quality system as well as the requirements for certification. The internal auditor should evaluate specifics such as the effectiveness of the corrective action procedures if finding and correcting problems, how close an SOP follows the declared method, the completeness of standard tracking logs, the contents of final reports and the accuracy of data packages, etc.

The audit must address **all** elements of the quality system and specifically mentions the testing itself.

What is required for the internal audit as documentation?
The laboratory:
1. needs a written procedure that covers what will be reviewed, by whom and how often;
2. needs a schedule outlining when each part of the complete quality system will be audited [keeping within the one-year minimum];
3. must have corrective action documentation;
3. must have the records of the reviews and reports of findings; and
4. must have reports to management.

How does the State assessor evaluate the internal audit?
The State assessors evaluate your quality system against the requirements of the State rule [R444-14] and industry standards.
They then evaluate your audit procedure against your quality system.
Continuing on, they will evaluate the actual documentation from internal audit against what your audit procedure describes.
The internal audit documentation is evaluated for completeness, timeliness and meeting certain technical requirements such as the independence of the auditor, organization of the audit and the minimum frequency of the audits.

NELAC, NELAP, EPA, ELCP
There are times when we get questions about the relationship of the State laboratory certification program, the National Environmental Laboratory Accreditation Conference [NELAC] standards, the National Environmental Laboratory Accreditation Program [NELAP] and the EPA.

EPA has federal statutory responsibility to “protect human health and to safeguard the natural environment — air, water, and land upon which life depends”. The states are charged to perform the hands on tasks within their boundaries. Part of the requirements of being a primacy state [the state takes the first responsibility for environmental protection] is to create a testing laboratory within the state to perform all environmental testing used for regulatory purposes. There is an option available where the state may create a laboratory certification program to allow for private sector laboratories to perform regulatory testing if they meet requirements acceptable to EPA.

The ELCP is that certification program and EPA reviews the program at least every three years.

The ELCP is required as part of the agreement with EPA to be a primacy state. The ELCP receives its authority to exist and act from state law. Laboratories are certified under state authority and are considered state-certified laboratories.

The administrative law we use to enforce the state law contains requirements that each laboratory must meet to become certified. These requirements now include

some standards developed by a national organization [NELAC] and are incorporated by reference into our administrative law.

NELAC is a voluntary association of State and Federal agencies with full opportunity for input from the private sector. NELAC's purpose is to establish and promote mutually acceptable performance standards for the operation of environmental laboratories. EPA's NELAP office provides support to NELAC and the evaluation of the accrediting authority programs.

NELAP oversees state and federal accrediting authorities to keep consistency between the programs. The ELCP is assessed according to NELAC standards to be considered a NELAP accrediting authority.

As an accrediting authority the ELCP evaluates laboratories to the Utah rule which meets the requirement of the NELAC standard. This evaluation allows for any laboratory assessed under the current rule to be considered both a Utah certified laboratory and a NELAP accredited laboratory.

A laboratory that becomes NELAP accredited by another accrediting authority is eligible for Utah certification through a process of recognition. Recognition assumes the laboratory has met the NELAC standards and as such is in substantial compliance with the Utah requirements found in rule.

Proficiency Testing

With the adoption of the NELAC 2001 standards the PT requirements change slightly. The Fields of PT move to matrix, technology, analyte. This change from program, analyte, and matrix.

What this means to you is the PT challenges will no longer be program specific. If the same method is used for two different matrices the method will need to be challenged by both matrices. If the same technology is used for two different methods and the same matrix only one will need to be challenged.

As an example, if a laboratory holds the following Fields of Accreditation:
1. Potable Water – ICPAES/EPA 200.7 – Lead
2. Non-Potable Water – ICPAES/EPA 200.7 – Lead
3. Non-Potable Water – ICPAES/EPA 6010 – Lead

The laboratory's Fields of Proficiency Testing would be:
1. Potable Water – ICPAES – Lead
2. Non-Potable Water – ICPAES – Lead

Therefore, in the Non-Potable Water matrix, the laboratory would be required to demonstrate proficiency twice yearly by **either** of its two accredited ICPAES methods. A satisfactory result would rule the laboratory satisfactory for both methods (EPA 200.7 and EPA 6010). Similarly, an unsatisfactory result would be considered unsatisfactory for both methods.

This change is effective July 1, 2003. Each laboratory should begin to

comply with the requirement for all existing fields of accreditation.

Laboratories will have until September 1, 2004 to become fully compliant with the new fields of accreditation as long as the laboratory remains compliant with all other NELAC Chapter 2 requirements.

Any PT sample analyzed after July 1, 2003 must be reported by technology/method, but laboratories may begin to analyze and report routinely scheduled PT samples by technology/method before this date.

A laboratory seeking to obtain accreditation for a new field of proficiency testing must analyze and report the PT samples by technologies/methods.

For either obtaining or maintaining accreditation, laboratories shall successfully complete two of the most recent three rounds of PT studies attempted as in the requirements of section 2.7.2.

The Environmental Testing Laboratory Certification Bulletin is published periodically by the Utah Department of Health, Division of Epidemiology and Laboratory Services, Bureau of Laboratory Improvement, to disseminate regulatory and general information to Utah certified laboratories.

Send comments to:
Bureau of Laboratory Improvement
46 North Medical Drive
Salt Lake City, UT 84113-1105
Voice (801) 584-8469 or Fax (801) 584-8501
Rod Betit, Executive Director Utah Department of Health
A. Richard Melton, Director Utah Department of Health
Charles D. Brokopp, Dr. P.H., Director DELS
David B Mendenhall, Director BLI

Certification Officers:
Tamara A. DeMorest, Acting Section Chief
Tiffini Adams
Kristin A. Brown
Lorna Ward –Office
Cassandra M. Barrett – Office
Web site -
www.health.state.ut.us/dels/labimp

28. Appendix C: Quality Control at United States Geological Survey National Water Quality Laboratory

http://nwql.usgs.gov/Public/pubs/QC_Fact/text.html

28.1. INTRODUCTION

The National Water Quality Laboratory (NWQL)--the primary analytical facility of the U.S. Geological Survey (USGS)--analyzes environmental samples from across the United States and around the world. Samples of water, sediment, aquatic biological materials, and air are analyzed at the laboratory using state-of-the-art instruments and methods. These analyses require a comprehensive quality-assessment process.

Quality assessment consists of two separate but related components: quality-control data and quality-assurance elements. Quality-control (QC) data are produced to measure bias and variability of the analytical method. Without QC data, the magnitude of errors, and thus the quality of the environmental data, is not known. Some examples of QC data sample types include standard reference materials, laboratory replicates, laboratory reagent blanks, spikes, and surrogates. Assessment of QC data is essential to evaluating environmental analytical data produced by a laboratory. Definitions of QC data sample types used by the NWQL are emphasized in this Fact Sheet.

Quality-assurance (QA) elements are also essential to producing high-quality analytical results. QA elements are defined as the procedures used to control the immeasurable components of the analytical method. QA elements include standard operating procedures, analysis-sequence protocols, instrument-maintenance logs, and personnel training records. Clearly, all of these elements could affect laboratory data quality. However, it is often difficult to ascertain the impact of QA elements on environmental analytical results. Therefore, the assessment of QA elements will not be discussed in this Fact Sheet.

Quality assessment is the process of reviewing the application of QA elements and the analysis of QC data. The objective of quality assessment is to provide evidence that the measurement processes are under control.

28.2. QUALITY-CONTROL PROCESS

The NWQL uses a three-tiered approach to QC divided into inorganic and organic analyses. For the first tier, "Method Performance," QC samples are analyzed by a chemist. At this level, chemists use the QC sample data to control the analytical process for each batch of environmental samples. If prescribed acceptance criteria are not met, the analysis is discontinued until corrected.

The second tier contains the "Data Review and Blind Sample Programs." Chemists use data from these programs to monitor method performance throughout the laboratory and over long periods.

The third and final tier consists of NWQL's participation in "Performance-Evaluation Studies." These studies are managed by local, State, or Federal agencies external to the NWQL. Data from these studies can be used to compare laboratories or to select a laboratory for analytical work. Summary reports from many of these studies are available on the NWQL Home Page Website at wwwnwql.cr.usgs.gov/USGS.

28.3. INORGANIC ANALYSIS

First Tier: Method Performance

Analysts trying to determine inorganic constituents in any environmental sample evaluate two areas of performance: instrument and method. Accurate instrument operation can be verified by analyzing known standards at different concentrations. Inorganic blank water, which has negligible concentrations of the constituents of interest, is also analyzed to evaluate and eliminate potential sources of bias.

The analyst uses QC samples to assess whether the method is working properly. Generally, data produced from these samples are the most useful for customers to evaluate the entire analytical process. In addition to inorganic blank water samples, standard reference materials are the primary tools for evaluating method bias and variability. Laboratory replicate data also might be evaluated to obtain method variability information. Definitions of these sample types follow.

Standard Reference Materials (SRMs). Generally, SRMs are supplied in one of two ways: as an ampoule in concentrated form that is added to the appropriate matrix, or as a prepared reference ready for analysis. The prepared reference is the predominant source when commercial materials are available. In either form, the SRM is thought to be sufficiently tested to be used to assess method performance. Several SRMs of differing concentrations and constituents are analyzed alongside environmental samples at the NWQL. The SRM data are evaluated to assess bias for each analysis. Additionally, as the data are compiled, long-term estimates of method variability can be obtained.

Laboratory Replicate. A laboratory replicate is one type of environmental sample replicate. The environmental sample is split at the laboratory to provide information about variability. If the sample is split into identical subsamples, then the replicate will provide information about variability inherent in the analytical process. However, true sample splits are often difficult to obtain. More frequently, the split samples will exhibit some differences because of sample heterogeneity. The sample variability inherent in more challenging matrices, such as sediment, will often mask the sample processing and analytical variability.

Second Tier: Data Review and Blind Sample Programs

Data Review Program. All inorganic analytical results initially are stored in the Laboratory Information Management System data base. A QC check program uses chemical logic checks to review all analytical results in the data base. All analytical results that fall outside the acceptance limits are sent to chemists in the Quality Assurance Unit for review. Examples of these logic checks include the following:

- Cation/Anion Balance
- Specific Conductance/Anion Ratio
- Specific Conductance/Cation Ratio
- Dissolved Solids/Specific Conductance Ratio
- Field Value/Laboratory Value Agreement (pH, alkalinity, specific conductance)
- Filtered Component Compared with Unfiltered Component

Blind Sample Programs. The inorganic blind sample programs are administered by two different organizations, one internal and the other external to the NWQL. The internal program quantifies bias caused by random laboratory contamination. It also supplements the external blind sample program by submitting double-blind reference samples for those analyses not evaluated externally (cyanide, whole water recoverable constituents, sediment, tissue). These reference samples are called "double blind" because both the sample origin and constituent concentrations are unknown to the analyst.

The external blind sample program is used to evaluate both the NWQL and the USGS laboratory in Ocala, Fla. Bias and variability information obtained from this program is made available to laboratory customers on the Web at wwwbtdqs.cr.usgs.gov.

Data analysis performed by either program typically involves preparing control charts to monitor long-term method performance. The number of standard deviations from the most probable value (otherwise known as the z-value) is calculated. Using this calculation, all data can be easily displayed in a single chart. The z-value is calculated as follows:

$$\frac{\text{laboratory measured concentration} - \text{most probable value}}{\text{standard deviation of most probable value}}$$

In figure 1, the z-values calculated for cadmium represent concentrations ranging from 0 to 50 micrograms per liter.

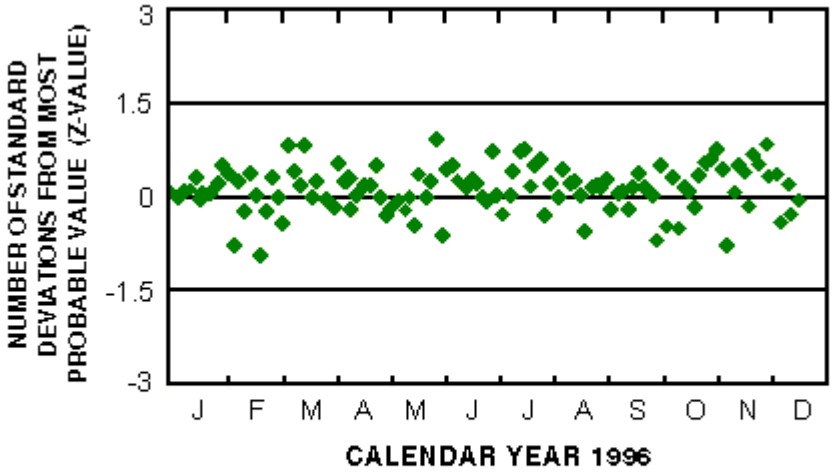


Figure 1. Control chart of cadmium determinations in standard reference material samples by graphite furnace atomic absorption spectrophotometry.

Third Tier: Performance-Evaluation Studies

The NWQL participates in the following national and international interlaboratory performance-evaluation studies:

- U.S. Environmental Protection Agency (USEPA) Water-Pollution (WP), Water-Supply (WS), and Safe Drinking Water Act (radiochemistry) studies;
- USGS Branch of Quality Systems Evaluation Program for Standard Reference Samples;
- National Water Research Institute of Canada; and
- National Oceanic and Atmospheric Administration (NOAA) Intercomparison Program for Marine Sediment and Tissue.

Field Supplies

The USGS warehouses stock sample containers, filters, and preservatives for collecting environmental samples. Inorganic blank water is also available for customer use. All supplies are rigorously tested to ensure compliance with quality standards. Contamination is tested using the lowest detection methodology for the constituents of interest. Certificates of analysis are provided with each manufacturer's lot. Other field QC supplies, such as inorganic spike materials, are available on request.

28.4. ORGANIC ANALYSIS

First Tier: Method Performance

Organic compounds may be degraded by heat, light, temperature, microorganisms, and other factors. Because the NWQL analyzes a wide range of samples and specializes in

trace and ultratrace compound concentrations, there is an increased need for QC information compared to inorganic analyses.

Samples received for organic determinations are grouped together for similar analyses. Laboratory reagent blanks and laboratory reagent spike samples are prepared and analyzed with each group of environmental samples to monitor method performance. Surrogate compounds are also added to all environmental and QC samples. These QC sample types are defined as follows.

Laboratory Reagent Blank. The laboratory reagent blank (LRB) is prepared using an appropriate matrix. The selected matrix should have negligible concentrations of compounds for that particular analysis. The LRB is processed in the same way as the accompanying environmental sample and so is used to monitor for random contamination introduced from laboratory processing. If a compound is detected and identified in the LRB, quantitation of that same compound in the environmental sample would be qualified. Not only does the analyst track LRB results with each sample set, but the laboratory can track a history of LRB results to determine possible trends.

Laboratory Reagent Spike. A laboratory reagent spike (LRS) is prepared and analyzed with each group of environmental samples. The LRS is a sample where known concentrations of the compounds of interest are added, usually into a synthetic (reagent) matrix. An evaluation of reagent spike data yields information about method performance. The LRS analytical results are compared to acceptance criteria for each method to assess potential bias of environmental sample results. In addition, LRS data are charted periodically to observe long-term method bias and variability. A graph of the percent recovery of the herbicide atrazine, an organic compound in the LRS, is shown in figure 2.

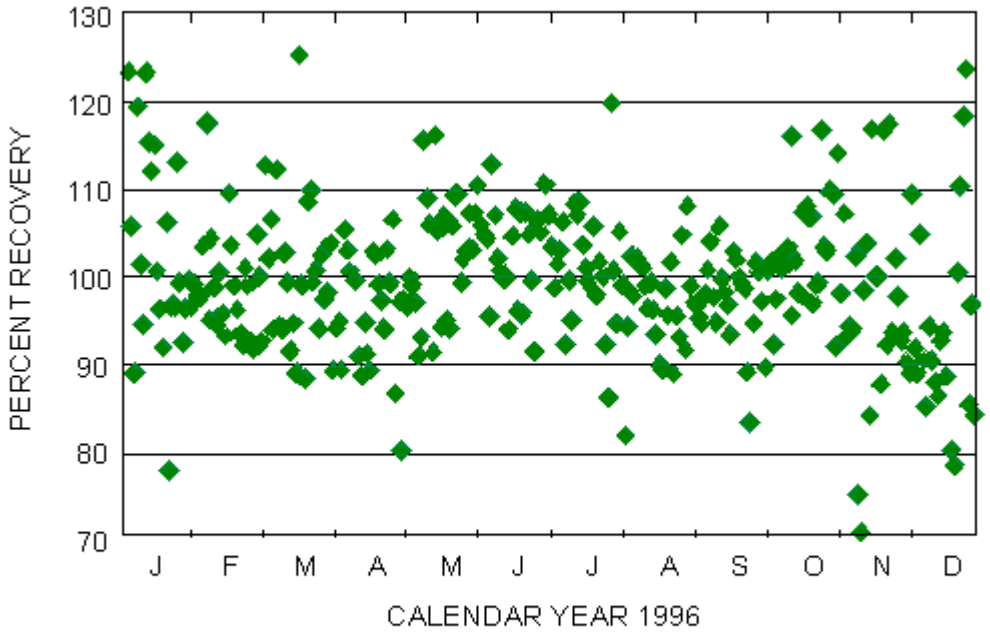


Figure 2. Percent recovery of the herbicide atrazine analyzed by solid-phase extraction, gas chromatography/mass spectrometry.

Surrogate. A surrogate is a compound that is expected to perform similarly to the compounds being analyzed in a laboratory method. The surrogate is not normally found in the environment and therefore can be used to monitor the recovery efficiency of analytical processes. The NWQL adds surrogate compounds to all environmental and QC samples to monitor compound recovery and potential environmental effects. For example, high dissolved organic carbon concentrations might interfere with analytical techniques. Surrogate analytical results are released electronically with environmental data for all organic analyses.

Second Tier: Blind Sample Program and Data Review

Blind Sample Program. The first major component within the second tier of quality control is the blind sample program where double-blind samples are submitted for analysis. Results of double-blind samples should accurately reflect the state of laboratory processes since the samples do not receive special treatment.

Data Review. Another major QC activity is long-term data-quality assessment. This type of data review involves evaluating control charts and analyzing statistics from the LRBs, LRSs, and surrogates.

Several statistical techniques are used for data analysis. Results are used to compute long-term control limits, which, in turn, are used to produce new acceptance criteria. Frequently, a control limit is set at three times the standard deviation of the expected

value. If only normal random errors are present, then 99.7 percent of the values will fall within this limit (Friedman and Erdmann, 1982, p. 91).

Third Tier: Performance-Evaluation Studies

Performance-evaluation studies for organic compounds are more difficult to administer than for inorganic constituents because some of the compounds are unstable. Special care is required to ensure that performance-evaluation studies evaluate method performance and are not biased by natural compound degradation. The special measures that must be taken (for example, chilling samples and protecting them from light), along with the high cost of organic sample analysis, can make the studies expensive. Nevertheless, the NWQL routinely participates in the following national performance-evaluation studies:

- USEPA Water-Pollution (WP) and Water-Supply (WS) studies,
- NOAA Intercomparison Program for Marine Sediment and Tissue, and
- National Water Research Institute of Canada (organic carbon).

Field Supplies

The USGS warehouses stock the necessary supplies for collecting environmental samples for organic analyses, including sample containers, preservatives, filters, and solid-phase extraction cartridges. In addition, organic blank water suitable for pesticide and volatile analyses is available. Blank water is rigorously tested and quality assured at the laboratory and is distributed with a certificate of analysis. Complete field spike kits, consisting of a volumetric measuring device, necessary solvents, gloves, and a waste storage container, can be purchased from the NWQL. Verified spike solutions for several compound classes are prepackaged and ready for use. Custom spike solutions tailored to meet field QC activities also can be prepared.

28.5. SELECTED REFERENCES

Fishman, M.J., ed., 1993, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory--Determination of inorganic and organic constituents in water and fluvial sediments: U.S. Geological Survey Open-File Report 93-125, 217 p.

Fishman, M.J., and Friedman, L.C., 1989, Methods for determination of inorganic substances in water and fluvial sediments: Techniques of Water-Resources Investigations of the U.S. Geological Survey, book 5, chap. A1, 545 p.

Friedman, L.C., and Erdmann, D.E., 1982, Quality assurance practices for the chemical and biological analyses of water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A6, 181 p.

D. Helsel. 2005. Nondetects and Data Analysis: Statistics for Censored Environmental Data. Wiley. 288 pp., http://www.wiley.com/WileyCDA/WileyTitle/productCd-0471671738.html).

Pritt, J.W., and Raese, J.W., 1995, Quality assurance/quality control manual--
National Water Quality Laboratory: U.S. Geological Survey Open-File Report 95-443, 35 p.

Irwin, R.J. (2004). Draft Part B of Aquatic Habitat Park Service guidance for Park Service Vital Signs Monitoring. Planning Process Steps: Issues To Consider And Then to Document In A Detailed Study Plan That Includes A Quality Assurance Project Plan (QAPP) And Monitoring "Protocols" (Standard Operating Procedures), Available on Internet
http://science.nature.nps.gov/im/monitor/protocols/wqPartB.doc

N. Taylor and C. E. Kuyatt. 1994. Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results NIST Publication TN 1297 (http://physics.nist.gov/Document/tn1297.pdf).

Wershaw, R.L., Fishman, M.J., Grabbe, R.R., and Lowe, L.E., 1987, Methods for
the determination of organic substances in water and fluvial sediments: Techniques
of Water-Resources Investigations of the U.S. Geological Survey, book 5, chap. A3, 80 p.

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T., eds., April 2004, Processing of water samples (version 2.1): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A5, accessed 1/30/06 at http://pubs.water.usgs.gov/twri9A5/ (Separate updates for 5.6.1.F, "Wastewater, pharmaceutical, and antibiotic compounds," 5.6.4.A, "Arsenic speciation," and 5.6.4.B, "Low-level Mercury" are provided on the Web page.)

UT-DWQ, 2006. Utah's 2006 Integrated Report Volume II - 303(d) List of Waters January 19, 2006 Department of Environmental Quality Division of Water Quality, Salt Lake City, UT. Report found on-line 1/30/06 at http://www.waterquality.utah.gov/documents/2006_303d_1-19-06draft.pdf

- Kimberly D. Pirkey and Stephen R. Glodt

April 1998

For more information, contact:

Chief, Branch of Analytical Services
USGS National Water Quality Laboratory
Box 25046, Mail Stop 407
Denver, Colorado 80225

Fact Sheet FS–02

This page is URL: http://wwwnwql.cr.usgs.gov/Public/pubs/QC_Fact
Created June 29, 1998

29. Appendix D: Example AMS+ and AMS calculations

Alternate Measurement Sensitivity Plus (AMS+)

Calculated as standard deviation of seven replicated measurements in-situ * 3.708 (two sided t-distrib 99% confidence)

AMS is the same calculation conducted on sink water measurements in premobilization procedure

Example AMS+ Calculation

lvk2-6-9-06	DO	Baro. Press.	Spc. Cond	Water	pH
Reading #	(mg/L)	(mm Hg)	(uS/cm@25 oC)	temp oC	unitless
1	7.89	632.74	811.04	21.48	8.53
2	7.93	632.74	817.26	21.46	8.53
3	7.97	632.71	807.88	21.45	8.53
4	7.99	632.71	806.77	21.44	8.53
5	7.99	632.69	803.32	21.44	8.53
6	7.97	632.66	811.47	21.44	8.52
7	7.96	632.69	808.05	21.46	8.53
AMS+	0.141	0.105	16.351	0.044	0.014

lvk-7-13-06	DO	Baro. Press.	Spc. Cond	Water	pH
Reading #	(mg/L)	(mm Hg)	(uS/cm@25 oC)	temp oC	unitless
1	7.79	638.45	807.53	21.44	8.49
2	7.78	638.48	806.25	21.43	8.48
3	7.84	638.45	809.27	21.44	8.48
4	7.83	638.45	795.60	21.44	8.48
5	7.81	638.45	803.65	21.45	8.48
6	7.78	638.45	798.51	21.44	8.48
7	7.86	638.45	801.29	21.45	8.48
AMS+	0.113	0.036	18.444	0.029	0.014

laverk3-8-21-06	DO	Baro. Press.	Spc. Cond	Water	pH
Reading #	(mg/L)	(mm Hg)	(uS/cm@25 oC)	temp oC	unitless

Example AMS calculation

	DO	Baro. Press.	Spc. Cond (uS/cm@25 oC)	Water temp oC	pH
Reading #	(mg/L)	(mm Hg)			unitless
1	4.75	573.02	525.43	19.46	7.61
2	4.75	573.05	525.59	19.46	7.61
3	4.75	573.05	525.92	19.46	7.61
4	4.75	573.05	525.75	19.46	7.61
5	4.74	573.05	526.03	19.46	7.61
6	4.74	573.02	525.79	19.47	7.61
7	4.74	573.05	525.79	19.48	7.61
AMS	0.018	0.046	0.740	0.026	0.000

	DO	Baro. Press.	Spc. Cond (uS/cm@25 oC)	Water temp oC	pH
Reading #	(mg/L)	(mm Hg)			unitless
1	8.10	573.00	582.25	23.04	7.50
2	8.09	573.00	582.03	23.04	7.50
3	8.09	573.00	581.96	23.04	7.50
4	8.08	573.00	581.95	23.03	7.50
5	8.06	573.00	582.10	23.03	7.50
6	8.06	573.00	582.08	23.02	7.50
7	8.06	573.00	582.01	23.02	7.50
AMS	0.061	0.000	0.381	0.028	0.000

	DO	Baro. Press.	Spc. Cond (uS/cm@25 oC)	Water temp oC	pH
Reading #	(mg/L)	(mm Hg)			unitless

1	7.45	637.44	682.33	25.72	8.41
2	7.45	637.41	682.44	25.72	8.41
3	7.46	637.44	682.74	25.72	8.41
4	7.46	637.44	682.78	25.72	8.41
5	7.46	637.41	682.74	25.72	8.41
6	7.46	637.39	682.67	25.72	8.41
7	7.46	637.41	682.85	25.72	8.41
AMS+	0.022	0.071	0.716	0.010	0.000

1	7.04	573.94	513.29	16.23	7.81
2	6.94	573.89	515.58	16.25	7.80
3	6.91	573.91	517.65	16.25	7.80
4	6.91	573.91	512.38	16.25	7.80
5	6.86	573.91	517.72	16.24	7.80
6	6.85	573.91	518.21	16.24	7.80
7	6.80	573.91	512.70	16.24	7.80
AMS	0.291	0.054	9.480	0.024	0.014

laverk-9-5-06	DO	Baro. Press.	Spc. Cond	Water	pH
Reading #	(mg/L)	(mm Hg)	(uS/cm@25 oC)	temp oC	unitless
1	9.13	640.26	704.21	14.06	8.32
2	9.13	640.28	704.30	14.06	8.33
3					
4	9.12	640.28	704.28	14.08	8.33
5	9.00	640.28	704.17	14.10	8.33
6	8.98	640.23	703.65	14.18	8.34
7	9.04	640.23	704.09	14.12	8.33
AMS+	0.253	0.093	0.887	0.179	0.023

	DO	**Baro. Press.**	**Spc. Cond (uS/cm@25 oC)**	**Water temp oC**	**pH**
Reading #	**(mg/L)**	**(mm Hg)**			**unitless**
1	7.04	573.94	513.29	16.23	7.81
2	6.94	573.89	515.58	16.25	7.80
3	6.91	573.91	517.65	16.25	7.80
4	6.91	573.91	512.38	16.25	7.80
5	6.86	573.91	517.72	16.24	7.80
6	6.85	573.91	518.21	16.24	7.80
7	6.80	573.91	512.70	16.24	7.80
AMS	0.291	0.054	9.480	0.024	0.014

lvrkncrk10-24-06	DO	Baro. Press.	Spc. Cond	Water	pH
Reading #	(mg/L)	(mm Hg)	(uS/cm@25 oC)	temp oC	unitless
1	10.11	635.13	684.67	8.84	8.36
2	10.10	635.10	684.51	8.84	8.36
3	10.08	635.13	684.60	8.84	8.36
4	10.09	635.13	684.60	8.84	8.36
5	10.06	635.13	684.69	8.84	8.36
6	10.06	635.10	684.51	8.84	8.36
7	10.04	635.10	684.92	8.84	8.36
AMS+	0.088	0.050	0.518	0.008	0.000

	DO	**Baro. Press.**	**Spc. Cond (uS/cm@25 oC)**	**Water temp oC**	**pH**
Reading #	**(mg/L)**	**(mm Hg)**			**unitless**
1	4.75	573.02	525.43	19.46	7.61
2	4.75	573.05	525.59	19.46	7.61
3	4.75	573.05	525.92	19.46	7.61
4	4.75	573.05	525.75	19.46	7.61
5	4.74	573.05	526.03	19.46	7.61
6	4.74	573.02	525.79	19.47	7.61
7	4.74	573.05	525.79	19.48	7.61
AMS	0.018	0.046	0.740	0.026	0.000

lvrkn11-7-06	DO	Baro. Press.	Spc. Cond	Water	pH
Reading #	(mg/L)	(mm Hg)	(uS/cm@25 oC)	temp oC	unitless
1	9.005	636.3208	731.68403	13.21667	8.47
2	8.997	636.3716	733.09059	13.21667	8.47
3	9.001	636.3462	732.5228	13.21667	8.47
4	8.999	636.3462	731.98772	13.20556	8.47
5	9.003	636.3208	733.8621	13.21111	8.47
6	9.009	636.3462	731.79713	13.21111	8.48
7	8.998	636.3462	732.91996	13.21111	8.48
AMS+	0.016	0.065	2.945	0.016	0.018

	DO	**Baro. Press.**	**Spc. Cond (uS/cm@25 oC)**	**Water temp oC**	**pH**
Reading #	**(mg/L)**	**(mm Hg)**			**unitless**
1	6.10	560.40	442.08	18.31	8.63
2	6.11	560.43	442.11	18.32	8.63
3	6.13	560.43	442.34	18.31	8.63
4	6.15	560.37	442.40	18.32	8.63
5	6.17	560.43	442.52	18.32	8.63
6	6.18	560.40	442.94	18.32	8.63
7	6.17	560.43	443.01	18.32	8.63
AMS	0.113	0.074	1.373	0.010	0.000

lvrk-12-6-06	**DO**	**Baro. Press.**	**Spc. Cond (uS/cm@25 oC)**	**Water temp oC**	**pH**
Reading #	**(mg/L)**	**(mm Hg)**			**unitless**
1	18.97	640.36	725.63	1.69	8.32
2	19.03	640.36	726.50	1.69	8.32
3	19.09	640.36	726.90	1.69	8.32
4	19.16	640.36	727.90	1.69	8.32
5	19.21	640.36	727.85	1.69	8.32
6	19.25	640.36	727.78	1.69	8.32
7	19.24	640.36	727.70	1.69	8.32
AMS+	0.402	0.000	3.227	0.008	0.000

	DO	**Baro. Press.**	**Spc. Cond (uS/cm@25 oC)**	**Water temp oC**	**pH**
Reading #	**(mg/L)**	**(mm Hg)**			**unitless**
1	6.71	568.60	454.90	19.46	8.56
2	6.71	568.60	454.86	19.46	8.56
3	6.69	568.63	455.04	19.45	8.56
4	6.71	568.60	454.94	19.46	8.57
5	6.71	568.63	455.03	19.46	8.56
6	6.71	568.63	454.76	19.46	8.56
7	6.72	568.63	454.85	19.46	8.56
AMS	0.029	0.050	0.368	0.014	0.014

	DO	Baro. Press.	Spc. Cond	Water	pH
7 month	(mg/L)	(mm Hg)	(uS/cm@25 oC)	temp oC	unitless
Avg AMS+	0.148	0.060	6.155	0.042	0.010

	DO	**Baro. Press.**	**Spc. Cond**	**Water temp**	**pH**
7 month	(mg/L)	(mm Hg)	(uS/cm@25 oC)	oC	unitless
Avg AMS	0.117	0.046	3.223	0.022	0.006

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #8

Field Methods for Core Parameters, Turbidity and Discharge, All Units

Version 1.1 (8/11/08)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
	4/11/2007	David Thoma			
1.0	8/11/2008	Kelly Lawrence	Added decontamination procedure Appendix J	Previously did not have a decontamination procedure	1.1

This SOP focuses primarily on techniques and equipment for discharge measurement and the use of multi-parameter probes for measuring 'core' physical and chemical characteristics of the water, namely pH, dissolved oxygen, conductivity and temperature.

It relies heavily on USGS National Field Manual Chapters A6.1, 6.2, 6.3, 6.4 and 6.7 (USGS variously dated), along with operating manuals for the equipment.

Colorado - The Hydrolab Minisonde 4a with a Surveyor 4 display will be used for determining core parameters. Procedures for CO park units follow the USGS protocol in the National Field Manual.

Utah - The Insitu TROLL 9000 with a Rugged Reader display will be used for determining core parameters. The Hach 2100 P Turbidimeter will be used to measure turbidity.

Table of Contents

1. Flow Measurement 4
2. Use of Established Stream Gages 5
3. Estimating Discharge from Distant Gages 6
4. Accessing Gage Data 6
5. Use of Current Meter 6
6. The current meter procedure - general 6
7. The current meter procedure –specific 7
8. Data storage and download for Aquacalc Pro (JBS Instruments) 8
8.1. To view data files stored on Aquacalc 8
8.2 Download to PC 8
9. Use of Weirs and Flumes 9
9.1. The portable V-notch weir plate procedure 9
9.2. The portable flume procedure 10
10. Volumetric Measurements 11
10.1 Pour-off procedure 11
10.2 The depression/sump procedure: 11
11. Methods of Last Resort 12
11.1 The float velocity procedure 12
11.2 The visual estimate procedure 12
12. Flow Severity and Hydrograph Limb 13
13. Data Fields – PCFF used in BLCA/CURE 14
14. Data Fields – Field_forms.xls (for use with Insitu Data Sondes) 14
15. Field Calibration of MultiParameter Probe 15
16. Using the In-Situ Rugged Reader for field data acquisition 15
16.1. Log core parameter data 15
16.2. Download data files and calibration results stored on Rugged Reader 17
16.2.1. Download data reports to the PC 17
16.2.2. Download calibration reports to PC 17
16.2.3. Troll calibration data file clean up 18
16.3. *Optional* - load data template onto Rugged Reader 18
16.4. *Optional* - Store calibration reports to Field_forms.xls in the field 19
17. Delete Rugged Reader files from PC 19
18. Open calibration reports on PC 19
19. To open data files on PC 20
20. Turbidity 20
21. Decontamination Procedures 20
22. Appendix A: Baski Cutthroat Flume User Manual 21
22.1. Baski Cutthroat Flume Rating Table (MS word) 25
23. Appendix B: Field_forms.xls premobilization worksheet meta data 27
24. Appendix C: Field_forms.xls meta data 31
25. Appendix D: State of Utah Lab Sheets Monthly 41
26. Appendix E: State of Utah Lab Sheets Quarterly 42
27. Appendix F: State of Utah Run Sheets Monthly 43
28. Appendix G: State of Utah Run Sheets Quarterly 44
29. Appendix H: EPA Method 180.1 Turbidity 45
30. Appendix I: Hach 2100P Turbidity User Manual 46
31. Appendix J: Decontamination Procedures 47

List of Tables

Table 1. Recommended approaches to discharge measurement. 4
Table 2. Accuracy and points to consider when selecting a measurement method. 5
Table 3. Flow Severity 13

Table 4. Hydrograph Limb 13
Table 5. Recommended instrument stabilization criteria for recording field measurements (from USGS National Field Manual) and WRD. Note the units to be used and recommended calibration frequencies. 16

1. Flow Measurement

Several alternative methods for measuring discharge are presented. The particular method used will vary by site and depend on the quantity of flow, channel characteristics, and accessibility. The following tables are provided to assist in selecting the most appropriate method. The various methods are listed for a recommended range of discharge conditions, and the relative accuracy of each method is described. Obtaining flow data from an established stream gage is the method of first choice, followed by use of a current meter. For sites with a smaller flow, like many springs, installation for weir or flume provides the greatest accuracy, though the impacts of repeated installations should be considered.

In addition to a measurement of flow, two descriptors of flow condition will be recorded, Flow Severity and Hydrograph Limb. These will be useful descriptors for later analysis of water quality data, even in cases where a quantitative measurement is not possible (Flow severity and hydrograph descriptions are provided in notebook page "field_codes" in the Field_forms.xls spreadsheet described in SOP 8 sections 10 and 11).

Table 1. Recommended approaches to discharge measurement.

	Discharge			
Discharge magnitude	**Gallons/minute (gpm)**	**Cubic Feet/ Second (cfs)**	**Metric mL, l or m^3/sec**	**Approach**
Unmeasurable	No discernable discharge to measure		No discernable discharge to measure	Depression
First	< 0.12		< 10 mL/s	Depression, Volumetric
Second	0.12 to 1.0		10 to 100 mL/s	Weir, Volumetric
Third	1.0 to 10.		0.10 to 1.0 L/s	Weir, Flume
Fourth	10 to 100	.02 to .22	1.0 to 10. L/s	Weir, Flume
Fifth	100 to 448.8	.22 to 1	10. to 100. L/s	Flume
Sixth	448.8 to 4,488	1 to 10	0.10 to 1.0 m^3/s	Current meter, Gaging station
Seventh	4,488 to 44, 880	10 to 100	1.0 to 10. m^3/s	Current meter, Gaging station
Eighth	> 44,880	>100	> 10. m^3/s	Current meter from bridge, Gaging station

Table 2. Accuracy and points to consider when selecting a measurement method.

Method	Relative Accuracy	Field Considerations
Established Gaging Station	Very High	First Choice if nearby due speed, accuracy and application during flood flows. Flow data may not be immediately available if the gage is not real-time.
Weir, Flume	High to Very High	Weir and Flume can be difficult to carry into backcountry sites. Impacts due to repeated installations at springs are a concern.
Current Meter	High	Requires wading, so unsafe during flood conditions. Not practical below
Volumetric	Moderate to High	Best applied where a natural pour-over exists.
Depression	Moderate	Best applied to very small discharges.
Float Velocity	Low to moderate	Applicable to a single channel with a minimum of vegetation or organic debris.
Visual Estimation	Low	Use only where other methods are ineffective or unsafe.

Note: of all of the instruments listed, the flume is the largest and most difficult to pack into backcountry sites. It should only be used in the back country if it is essential to obtain an accurate measurement or if the site has a discharge magnitude making a flume the optimal instrument.

If the discharge of the spring is low, the discharge measurement may take 10s of minutes and should be initiated early in the site visit. The name, serial number (if available), and accuracy of the instrument used to measure discharge should be recorded as well as any other important observations. Important observations may include the markers of any recent high discharges, such as high water marks, oriented vegetation or debris on or above the channel or floodplain. All such information can be entered into the Field_forms.xls spreadsheet in appropriate cells, or in comment cells.

2. Use of Established Stream Gages

The use of an established stream gage is the preferred method obtaining flow data. The time investment in the field is minimal and the accuracy is high. Gages provide the additional benefit of permitting an examination of the hydrograph before and after sample collection. Gages are typically constructed on rivers with base flows of about 1 cfs or greater. Some caution should be used for flows below 5 -10 cfs, where very minor changes in channel geometry can lead to inaccuracies in the measurement.

For large rivers, such as the Green and Colorado Rivers in our area, taking instantaneous flow measurements is beyond the capability of NCPN staff, so stream gages provide the only quantitative measure of flow.

In order to be useful, the stream gage should be located near the water quality sample site, without significant inflow or withdrawal of water occurring between the sample site and gage, or where these do occur they are predictable or measurable so the gaged flow

can be adjusted with some reliability. It may be necessary to take several instantaneous measurements to establish a relationship between flow at the gage and flow at the sample site.

3. Estimating Discharge from Distant Gages

In cases where making a reliable discharge measurement on site is not practical, such as large rivers, an estimate of the discharge can be made using gage data from several miles upstream if inputs on the intervening distance are minimal. Flow from the gage will take into consideration the travel time from the gage, and will be corrected for known significant inputs or depletions that occur in the intervening distance. The estimated discharge will be recorded along with a comment that it is based on the gage, and a descriptor of its reliability. Reliability can be high under relatively constant flow conditions, lower when complicated by flow fluctuations and tributary inputs.

4. Accessing Gage Data

Where real-time gage data exists it can be accessed at:

http://waterdata.usgs.gov/ut/nwis/current/?type=flow

http://waterdata.usgs.gov/wy/nwis/current/?type=flow

http://waterdata.usgs.gov/co/nwis/current/?type=flow&group_key=huc_cd

http://waterdata.usgs.gov/az/nwis/current/?type=flow

Where gage data is not available real-time, contact the USGS office responsible for the gage to obtain provisional data. Depending on the type of data logger at the gage and communication with it, it may take several weeks before data is downloaded and available.

The travel time between the gage and the water quality site should always be taken into consideration when selecting flow data from the gage. Measured travel times are desirable if they are available from the river segment. If not an estimate of 5 mph is reasonable for short distances.

5. Use of Current Meter

Typically, current meters are necessary in sixth to eighth magnitude discharge streams (SOP 4 - Table1). Text below is modified from USGS - Buchanan and Somers, 1984: p. 31-54.

Operator manuals for AquaCalc Pro, wading rod, pygmy, and price meters with rating charts are provided in SOP 5- Preparations, Equipment handling and calibration.

6. The current meter procedure - general

Choose a cross section in a straight reach that has the following characteristics.

1. Streambed is free of large rocks, weeds, and protruding obstructions that create turbulence
2. Streambed has a flat profile to eliminate vertical components of velocity.
3. All of the flow is directed downstream (i.e. there are no eddies) and lines of flow are as near parallel as possible.
4. Pull a tape taut perpendicular to the channel and secure.
5. Divide the cross section of the channel into 20 to 30 partial sections.
    a. A partial section is a rectangle whose depth is equal to the measured depth at the location and whose width is equal to the sum of half the distances of the adjacent verticals.
    b. The spacing between partial sections is adjusted so that no more than 5% of the flow is in any one partial section. This means that where water is deeper and swifter measurement points are spaced closer together, while in shallow and slow water they are spaced further apart.
6. Measure the area and mean velocity of each section separately.
    a. The person wading the channel should stand downstream of the velocity meter at an arms-length distance from the current meter so as not to disturb the flow path.
7. At each vertical, the following observations are recorded on the AquaCalc Pro
    a. the distance to a reference point on the bank along the tag line
    b. the 0.6 tenths of depth value
    c. The velocity should be measured at a depth which is 0.6 of the depth from the surface of water in the channel.
    d. the averaged 40 second velocity as indicated by the current meter.
8. The discharge of each partial section is calculated as the product of mean velocity times depth at the vertical times the sum of half the distances to adjacent verticals. The sum of the discharges of each partial section is the total discharge. All of the necessary data and calculations are performed by the AquaCalc Pro.

7. The current meter procedure –specific

1. Aquacalc User Manual can be found in SOP5 Appendix K
2. Turn on the AquaCalc Pro
3. Select 5, system preferences
4. Select 1, set time
    a. Enter time in specified format
4. Select 2, set date
    a. Enter date in specified format
5. Select menu to return to main menu
6. Select meter setup, 4
7. Set the meter name, ID and type
    a. Note: type is of most importance
    b. After setting, hit esc, or menu
8. Select sections, 2

9. Use soft keys to navigate to a screen that has space to enter a new section name
    a. Select a number corresponding to a blank row
    b. The number will flash, then press the soft key that specifies “new”
    c. Select GID, 1 to change the gage id name
    d. Use the soft keys and number pad to enter a Gage ID name
        i) Naming convention is NCPN 3 letter site abbreviation + month + year
        ii) Eg. NFVdec05 for North Fork Virgin December 2005
    e. Press enter when finished.
    f. Review the remainder of the information displayed and change as necessary, then press escape, then menu
    g. You should now be at the main menu and GID should be the name you just entered.
10. At the main menu press measure, 1
11. Enter tag line distance at edge of water
12. Press “New Vertical”
13. Press “distance” and enter the tag line distance
14. Press “stream depth” and enter the stream depth by reading increments on the wading rod
15. Press “measure” to take a 40 second reading.
16. Press “New Vertical” and repeat above steps until the last reading at edge of water
17. At that time after pressing “new vertical” press “distance” and enter the tag line distance then press “edge”.

8. Data storage and download for Aquacalc Pro (JBS Instruments)

8.1. To view data files stored on Aquacalc

1. Click 2 for sections on Aquacalc
2. Select the number for the section of interest. It will start blinking
3. Click open
4. Click measure
5. Click review totals soft key
6. Click section totals soft key
7. Record these data on the field sheet hard copy

8.2 Download to PC

1. Connect Aquacalc to serial port on PC
2. Turn on Aquacalc
3. Click 2 for sections
4. Click the appropriate number for the section you want to download
5. Click the open soft key on the Aquacalc
6. Open data link software (upgrades and manual available at http://www.jbsenergy.com/downloads/)

7. Click settings button, then OK
8. Click new data button
9. Provide a file name using convention “NCPNID-month-year” e.g. NFKdec05 for a file collected in December on the N.FK.Virgin River in 2005
10. Click open and yes to create this file. On PC to close dialog box that says press enter on Aquaclac. (don’t press enter button on Aquacalc)
11. Press the 3 button on the Aquacalc to start the data download to PC
12. You should see all of the data being downloaded.
13. Save the downloaded data in its raw csv format by clicking close on in the AquaCalc software on the PC. Save the data to the Field_forms.xls spread sheet by clicking the Excel icon in the Datalink software. Copy and paste the data into the appropriate location in the Field_forms.xls spreadsheet

9. Use of Weirs and Flumes

Typically, weirs are used to measure discharge in channels which have second to fourth magnitude values of discharge, while flumes are used to measure third to sixth order magnitude discharge (SOP4 - Table 1). Flumes and weirs may be heavy and difficult to pack into back- country sites, but they are the most accurate measurement method for some discharges. Try to minimize damage that may occur to the channel and banks when either device is set in place.

Operator manual for Baski Cutthroat flume and corresponding rating tables can be found in Appendix A. The rating table and equation for the 90 degree V-notch weir is found in the Field_forms.xls spread sheet on notebook page “rating_charts.”

Weirs and flumes measure discharge by being physically placed in the channel and forcing all of the flow to pass over or through the device. If the following conditions are met, the result is a highly accurate measure of discharge:

1. all the flow is indeed flowing through the device,
2. all of the flow is directed downstream (i.e. there are no eddies) and lines of flow are as near parallel as possible
3. the device is installed level and plumb,
4. there is a free fall on the downstream side of the device,
5. adequate time is allowed for the pond behind the device to fill and equilibrate,
6. water depth is measured accurately and in the correct location,
7. the channel is low gradient
8. bed materials are fine-grained to ease placement and minimize leakage around the device

9.1. The portable V-notch weir plate procedure

(modified from USGS - Buchanan and Somers, 1984: p. 57-59):

1. Push the weir into a channel of loose material – they do not work in gravel or bedrock channels without significant channel modification.

2. The weir has a “v” notch, or other regular geometric shape through which all discharge in the channel must be focused. The weir has a scale which is used with a rating chart to convert pool depth to discharge.
3. Once placed in the channel, level the weir using a bubble level.
4. Ensure the weir plate is plumb.
5. Allow flow through the weir to stabilize prior to measurement.
6. Record gage height 3 to 5 times over a 3 to 5 minute interval, as appropriate.
7. Calculate the mean from the three replicated measurements and record.
8. The volumetric discharge (m^3/s or l/s) is calculated using a standard equation specific to the weir plate, or a rating chart. This equation is implemented automatically in Field_forms.xls when users enter stage height.
9. The accuracy of the weir is dependent on the size of the notch in the weir and the resolution of the scale on the weir. Some seepage around the weir plate is common and should be estimated (e.g. measured discharge is 6 gpm with an estimated seepage of 20 % of the flow, so adjusted discharge is 7.2 gpm). Rags or plastic sheeting can be useful additions to seal around the weir plate.
10. Record stage and leakage on the Lab sheet data form and in the Field_forms.xls file
11. Record the method of discharge measurement
12. Record ‘M’ to indicate the discharge was a measured value.

9.2. The portable flume procedure

After selecting a measurement site, (modified from USGS - Buchanan and Somers, 1984: p. 59-61)

1. Orient the flume with the wing walls pointed upstream in the channel in such a fashion as to focus as much flow as possible through the regular profile of the opening of the flume.
2. Use a bubble level to make sure the flume is level.
    a. Level the floor of the upstream section both longitudinally and transversely.
3. Allow flow to stabilize prior to measurement.
4. Record gage height 3 to 5 times over a 3 to 5 minute interval.
5. Record a visual estimate of % total flow leaking around the flume
6. The standard rating curve for the flume automatically translates gage height to discharge in the Field_forms.xls spreadsheet.
7. The mean value for discharge (m^3/s or l/s) is calculated automatically and recorded. Accuracy of the instrument is dependent on the scale on the flume. On some occasions, it may not be possible to capture 100 % of the discharge in the flume. If less than 100 % of the discharge is captured by the flume, the percent of flow NOT captured by the flume should be estimated for each of the 3 to 5 measurements and recorded. A correction to the discharge measurement is made automatically in Field_forms.xls to account for the percent of discharge not captured by the flume.
8. Record discharge value on the hard copy data form and in the Field_forms.xls file

9. Record the method of discharge measurement
10. Record ‘M’ to indicate the discharge was a measured value.

10. Volumetric Measurements

Volumetric measurements are typically used in first and second magnitude discharge springs, where there is a pour off, or other feature that allows flow to be easily captured in a container (SOP4 - Table 1). The depression/sump method is typically used for unmeasurable to first magnitude springs with little to no surface expression of flow. It is used for relative comparison value of discharge.

(modified from USGS - Buchanan and Somers, 1984: p. 61-63):

10.1 Pour-off procedure

1. Identify a location in the channel where most of the discharge flows.
2. Construct a temporary earthen dam using soil / sediment and impermeable materials such as plastic bags.
3. Divert water through the temporary earthen dam with a pipe or constructed channel.
4. Allow flow to stabilize prior to measurement.
5. Catch discharge from pipe with a container of known volume.
6. Record the time to fill.
7. Repeat the time to fill measurement 3 to 5 times over a 3 to 5-minute interval
8. Calculate the mean value (ml/s).
9. Accuracy of the instrument is dependent on the accuracy of the volumetric container and time to fill measurement.
10. A suite of varying size of containers appropriate for first to second magnitude discharge springs should be taken to the field site.
11. When not used for volumetric measurements, the containers can be used to help pack field gear.
12. Record discharge value on the Lab sheet data form and in the Field_forms.xls file
13. Record the method of discharge measurement
14. Record ‘M’ to indicate the discharge was a measured value.

10.2 The depression/sump procedure*:*

1. Construct a depression in the seep area.
2. Calculate the volume of depression using volumetric calibration or multiplication of dimensions.
    a. The volumetric containers used for the volumetric measurement may be used to estimate the volume of the depression.
3. Evacuate the depression, and record the time required to fill.
4. Repeat this procedure 3 to 5 times
5. Compute the mean value.
6. Record discharge value on the Lab sheet data form and in the Field_forms.xls file
7. Record the method of discharge measurement

8. Record ‘M’ to indicate the discharge was a measured value.

11. Methods of Last Resort

11.1 The float velocity procedure

(modified from USGS - Buchanan and Somers, 1984: p. 63)

1. While the accuracy of this method is relatively poor, it has the advantage requiring very little field equipment (stopwatch, tape measure, notebook, and pencil), and can be applied in streams too shallow for effective use of a current meter. It is applicable to simple channels (i.e. not braided) with a minimum of vegetation or organic debris. This is a method of last choice compared to the more accurate velocity measurement techniques listed above.
2. Mark two cross sections with flagging along a reach of straight channel.
3. Measure the distance between the two sections with the measuring tape.
4. Measure the width and depth of each channel cross section and record.
5. Locate cross section to allow for a travel time of >20 sec float time (if possible).
6. Place a float, i.e., wooden disk(s) or short dry stick in the stream channel and allow it to reach stream velocity before the upstream cross section is crossed.
7. Note the position of the float relative to the channel sides.
8. Record the time it takes for the float to travel between the two cross sections.
9. Repeat this procedure 3 to 5 times, as the float is placed at different locations across the channel at the upstream cross-section.
10. Trials where the float hangs up on vegetation or other debris are discarded.
11. Compute the velocity of the float as the distance between the cross sections divided by the travel time.
12. Calculate the mean value of surface horizontal velocity (m/s) is calculated.
13. Multiply convert mean surface velocity to mean vertical velocity by multiplication with a coefficient of 0.85.
14. Calculate discharge (m^3/s) by multiplying the value of mean velocity by the average area of the section of the stream channel measured.
15. Record discharge value on the Lab sheet data form and in the Field_forms.xls file
16. Record the method of discharge measurement
17. Record ‘M’ to indicate the discharge was a measured value.

11.2 The visual estimate procedure

This method is only recommended as last resort when other methods are ineffective or unsafe. Site conditions, such as dense vegetation cover, steep or flat slope, diffuse discharge into a marshy area and dangerous access sometimes do not allow for a direct measurement of discharge by the techniques listed above. Although visual estimation is imprecise, it may be the only method possible for some springs.

1. Make a visual estimate using units appropriate to the magnitude of discharge.
2. Note high water level on prominent and permanent features

3. Record discharge value on the Lab sheet data form and in the Field_forms.xls file
4. Record the method of discharge measurement as ‘visual’
5. Be sure to record ‘E’ on all data forms to indicate the discharge is an estimated value.
6. Take photographs to record the surface area wetted or covered by water
    a. The photograph can be inserted into Field_forms.xls by clicking
        i. Insert, picture, from file

12. Flow Severity and Hydrograph Limb

Record a qualitative determination of flow severity and hydrograph limb based on the descriptions in the following two tables.

Table 3. Flow Severity

New STORET Code	WRD Description	Old STORET Code
Dry	No visible water in stream (typical of dry period for an ephemeral/intermittent stream).	1
No Flow	Discrete pools of water with no apparent connecting flow (at surface).	No old code
Low	Base flow for a stream or flow within roughly 10% to 20% of base flow condition.	2
Normal	When stream flow is considered normal (greatest time that stream is characterized by this in terms of flow quantity, level, or general range of flow during a falling or rising hydroperiod, but above base flow).	3
Above Normal	Bank full flow or approaching bank full (generally within upper 20% of bank full flow condition).	4
Flood	Flow extends outside normal bank full condition or spreads across floodplain.	5

Table 4. Hydrograph Limb

New STORET Code	STORET Description
BASE	Base. Old STORET Code 1.
RISING	Rising. Old STORET Code 2.
PEAK	Peak. Old STORET Code 3.
FALLING	Falling. Old STORET Code 4.

13. Data Fields – PCFF used in BLCA/CURE

Personal Computer Field Forms (PCFF) is software developed by USGS for field data entry, storage and analytical services requests. It is designed around USGS protocol and standard USGS laboratory procedures. There is no users guide available, but with personal tutoring can be learned quickly. It has similar but much greater functionality than Field_forms.xls.

Critical field data will be recorded on hard copy in the field. Make hard copy of PCFF pages for this purpose. This serves as a fail-safe backup in the event digital data are corrupted or lost between the field and office. Only the most critical data elements need be recorded on hard copy while in the field. These include,

1. Date and time of sample collection
2. Water color and hydrograph limb
3. Discharge and method of measurement
4. Core parameter data (i.e. 7 measurements each of pH, DO, SC, temp and pressure for conversion to %DO)
5. Weather conditions
6. Spin test results
7. Parameter value readings before and after calibration

14. Data Fields – Field_forms.xls (for use with Insitu Data Sondes)

For use with the Insitu Rugged Reader plam PC and Troll 9500 sonde, critical field data will be recorded on hard copy in the field. Make hard copy of only pages 1 and 2 in Field_forms.xls for this purpose. Print front and back so 1 page is all that is needed for each site. (Multiple pages are troublesome in windy conditions.)

1. Open Field_forms.xls
2. Highlight first two pages cells A1:L150
3. Click File then print
4. Click properties and click radio button “Flip on long edge”
5. Click “selection” radio button.

This serves as a fail-safe backup in the event digital data are corrupted or lost between the field and office. Only the most critical data elements need be recorded on hard copy while in the field. These include

1. Date and time of sample collection
2. Water color and hydrograph limb
3. Discharge and method of measurement
4. Core parameter data (i.e. measurements of pH, DO, SC, temp and pressure for conversion to %DO)
5. Weather conditions
6. Spin test results if using a flow meter

7. Parameter value readings before and after calibration

Most of these data will be over written when digital files from the Troll unit and AquaCalc are downloaded and pasted into Field_forms.xls. This eliminates transcription error and saves time once in the office. Download instructions for the digital data can be found elsewhere in this document.

Meta data for all fields in Field_forms.xls can be found in Appendix B. Field_forms.xls has cells color coded in blue that require data entry by the user. However, not all blue cells must be filled in. Therefore a data completion field has been created that tells users if all required data input has been completed. This field is column J.

15. Field Calibration of MultiParameter Probe

1. Cool standards to ambient water temperature by immersing in stream for 15 minutes.
2. Calibrate pH via 2 point method (7.0 and 10.01) as per SOP 5 Appendix B pg 67
3. Calibrate conductivity via 1 point method at 1413 us/cm SC as per SOP 5 Appendix B pg 73
4. Calibrate DO via 1 point method using air saturated with water as per SOP 5 Appendix B pg 81
5. Take pre and post calibration readings of standard solutions for pH and conductivity. Record these values on the field form. This last step is especially important because calibration parameters may be out of range as specified in the user manual on occasion yet results may be perfectly valid.

16. Using the In-Situ Rugged Reader for field data acquisition

16.1. Log core parameter data

1. Turn on Rugged Reader
2. Hit top bar with stylus
3. Start – pocket situ
    a. Double click com1 19200 to find Troll
    b. Under that double click Troll 9000 profiler
    c. Highlight parameters
4. Click profiler
    a. Troll data files have the following naming convention
    b. SN34051 2005-09-28 192249 site_date
    c. Where digits 1-7 are the probe serial number
        i. Digits 9-18 are the date
        ii. Digits 20-25 are hours, min, sec in military time
        iii. Digits >27 are the file name specified by user when file was opened
    d. Change name to ‘site_date’ using the keyboard icon
        i. Site is a short abbreviation for the site name

ii. Date is of format dd_mm_yy

5. Click OK
6. Allow the Troll sonde to equilibrate for several minutes (5 at a minimum) in the stream.
7. Monitor the status of equilibration by selecting graph on the bottom of the screen
8. A flat line means equilibrium achieved.
9. Do something else productive while waiting.
10. Continue to equilibrate until all plots of core parameters are asymptotic or completely horizontal, which indicates no change in parameter value is occurring with time. Toggle between parameters to verify.
11. Click "meter" then click "snapshot" 7 times to record 7 readings **without removing the Troll probe from the water**. The median is determined automatically in the Field_forms.xls spread sheet.
12. Click "close" to end data logging session.
13. Before leaving the site view the data file by navigating to
    a. \Home\Data Folder\Troll 9000 Profiler Data\SN_#of your unit\
    b. On occasion conductivity is not recorded digitally. If it does not get recorded in the file, start a new file and record again before leaving site.
    c. Click exit to exit pocket situ. This is required in order to download and view data files later.
14. Unfortunately there is no easy way to copy and paste data from within Winsitu on the Rugged Reader directly into Field_forms.xls also on the Rugged Reader. This is why it is important to record in writing the core parameter readings on paper. Later they can be copied electronically into Field_forms.xls in the office after opening Winsitu on the PC and exporting data to an Excel format file.

Table 5. Recommended instrument stabilization criteria for recording field measurements (from USGS National Field Manual) and WRD. Note the units to be used and recommended calibration frequencies.

Standard Direct Field Measurement	**Stabilization criteria for measurements (variability/repeatability should be within the value shown)**
[1]Temperature:	
Thermistor Thermometer	0.2°C
Liquid-in-glass Thermometer	0.5 °C
[2]Conductivity (Specific Cond.)	
When <100 uS/cm	5 percent
When > 100 uS/cm	3 percent
[3]pH:	
Meter displays to 0.01	0.1 unit
[3]Dissolved oxygen:	
Amperometric method	0.3 mg/L

** Resolution/Sensitivity is a data quality indicator related to detection limits but typically handled differently for field probes than for laboratory parameters. For more information, see Part B (http://science.nature.nps.gov/im/monitor/protocols/wqPartB.doc) (Irwin, 2004).

***In the case of field probes, accuracy is typically a "best case" maximum deviation from known correct values (typically based on comparisons with known NIST certified reference materials or standards). True accuracy is a combination of high precision and low bias (see PartB for more details).
[1] Recommended sensor calibration is quarterly
[2] Recommended sensor calibration is daily
[3] Recommended sensor calibration is at beginning and end of sampling at each station (twice a day minimum)

16.2. Download data files and calibration results stored on Rugged Reader

Note: All of the following commands are typed or entered on the PC.

Connect USB from Rugged Reader to PC

16.2.1. Download data reports to the PC

Connect Rugged Reader to PC via Active Sync and USB cable Tone indicates a positive connection

If any error messages pop up they can be eliminated by choosing which functions ActiveSync performs automatically.

a) **IMPORTANT!! DO NOT SYNCHRONIZE unless you know exactly what you are doing. It is very easy to irretrievably erase data using synchronize functions. Choose "do not synchronize" if unsure. IMPORTANT!!**
b) Click file explorer in Active Sync
c) Click up arrow with stylus on bottom of screen and navigate to
    i. \built in storage\ pocket situ\data
d) You can view file names here, but cannot view data in the files.
e) Copy and paste *.bin files on Rugged Reader to a data folder on PC.
f) Open Winsitu on the PC
g) Open a bin file using Winsitu by clicking "File" then "open bin file".
h) Navigate to *.bin file location on PC
i) Double click to open.
j) Click "File" then "export to Excel"
k) Copy and paste from the temporary Excel file into appropriate location in Field_forms.xls.
l) To navigate up the file structure tree click data at top of screen then click pocket situ in the drop down menu

16.2.2. Download calibration reports to PC

1. In Active Sync on the PC click file explorer
    a. Click up arrow with stylus on bottom of screen and navigate to
    b. \built in storage\ pocket situ\Calibration Reports\SN34051 (SN34051 is the Troll serial number)

    c. Click any of the reports to view calibration results.
    d. Troll Calibration files have the following naming convention
        i. SN34051 2005-09-29 083430 PH
        ii. Where digits 1-7 are the probe serial number
        iii. Digits 9-18 are the date

iv. Digits 20-25 are hours, min, sec in military time
v. Digits 27-29 are the type of calibration
1. possible values are PH, COND, DO, QICKCAL

2. In the ActiveSync window click the Explore icon
3. Double click my pocket pc, built in storage
4. Highlight file you want to copy, right click and click copy
5. On the PC navigate to file where you want to place the copied data and click paste
6. Open a file by double clicking it.
7. Copy and paste from the temporary html file into appropriate location in Field_forms.xls.

16.2.3. Troll calibration data file clean up

1. Troll calibration files are copied into Excel in a single column that will be difficult to handle in data management and analysis. For this reason we convert “text to columns”.
2. With the Field_form.xls spread sheet open
    a. Highlight all of the data in the column you want to convert from text to columns.
    b. Click data, text to columns
    c. Check the ‘space’ delimited box
    d. Preview results and make sure the format will turn out as expected
3. Also make sure when the text is expanded to columns it doesn’t over write any data in cells to the right

16.3. *Optional* - load data template onto Rugged Reader

This is useful primarily because there are lots of built in conversions and reference tables you may wish to access in the field. It is not a good way to enter data in the field unless you are using a laptop or tablet because the Rugged Reader is very slow at this.

You must first set up the connection between your Rugged Reader and the PC. Do this by following directions in the Rugged Reader manual. The following steps demonstrate how to copy a blank template in to non-volatile memory on the Rugged Reader. Opening and closing a file in non-volatile memory is slower, but data storage is more secure in case of power failure.

Create a shortcut to the desktop for the path

1. C:\Documents and Settings\your_name\My Documents\Pocket_PC My Documents
    a. Copy and paste into this short cut the version of Field_forms.xls you want loaded onto the Rugged Reader
2. Plug the USB cable into the Rugged Reader and a USB port on the computer.
3. If ActiveSync is installed an automatic synchronization will take place.
4. If not, install ActiveSync
5. ActiveSync will install Field_forms.xls into the active memory
6. On the Rugged Reader open File Explorer and navigate to active memory

7. This is done either by pressing \ tapping the yellow Rugged Reader icon at the bottom of the screen, or by clicking the "^" arrow on the bottom of the screen then the "\" symbol and then "My Documents".
8. Tap and hold the file name "Field_forms.xls" then lift and tap again to select "copy".
9. Navigate to Built-in-storage, tap a blank space on the screen then lift and tap paste to copy the Field_forms.xls file into built in storage.
10. Navigate back into active memory and delete the other copy of Field_forms.xls. This will prevent you from accidentally entering data into both forms which would be difficult to compile later.

16.4. *Optional* - Store calibration reports to Field_forms.xls in the field

Note this is not recommended because it's quite slow

With a calibration report open on the Rugged Reader

1. Highlight any part of the file with the stylus
2. Lift the stylus off the screen
3. Click "select all text"
4. Click "copy"
5. Click "X" in upper right hand corner of screen – multiple times if necessary to return to start page
6. Click "Start"
7. Click "File Explorer"
8. Click "^" on bottom of screen
9. Select "Built-in-storage"
10. Click Field_forms.xls
11. Click on the cell where you want to copy the calibration report
12. Click and hold stylus to screen until a dialog pops up then click paste. Click "X" in upper right hand corner of screen – multiple times if necessary to return to start page

17. Delete Rugged Reader files from PC

1. Use file Explorer to delete files from Rugged Reader first make sure to exit pocket situ
2. Click home in pocket situ on the Rugged Reader
3. Click exit.
4. From the start menu on Rugged Reader click file explorer
    a. Under Data and no flow
    b. Click and hold the file you want to delete.
        i. A drop down window will open with an option to delete the highlighted file.

18. Open calibration reports on PC

Navigate to file and double click.

19. To open data files on PC

1. Start winsitu 4
2. Click File
3. Click open bin file
4. Navigate to file of interest.
5. It's wise to save the bin file in Excel format using the save as function.

20. Turbidity

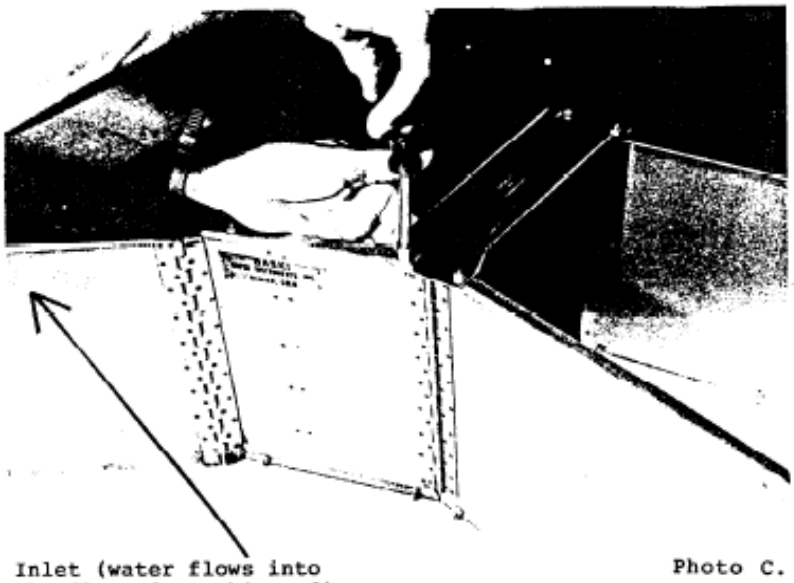
Turbidity is measured and reported in nephelometric turbitity units (NTU) using the Hach 2100P portable turbidity meter. A description of the method is provided in Appendix H and specific instruction for calibration and measurement are provided in Appendix I.

21. Decontamination Procedures

Decontaminating equipment that comes in contact with water is essential to minimized the potential for spreading aquatic invasive species. In 2007 Zebra Mussles were introduced into the Fremont River. Quagga mussles are likely to be introduced into reservoirs in the network as they are closing in geographically. Therefore it is exceedingly important that hydrologists and others doing water quality monitoring be vigilant to the potential that their activities could be the vector that spreads aquatic invasive species. A decontamination procedure (Appendix J) has been developed to prevent spread of invasives, but it will only work if followed diligently.

22. Appendix A: Baski Cutthroat Flume User Manual

Turn the flume right side up and install center brace. The center brace for the 1", 2" and 4" throat is a flat piece, but the center brace for the 8" throat has turned up edges...see Photo C and Photo D. With either type, rotate the center brace so the longest side (of the two uneven sides) is toward the **inlet** opening, thus, the holes will align. Insert bolt from the underside (these are not beveled holes), then place washer and hand tighten nut from the topside for each hole.



Inlet (water flows into the flume from this end)

Photo C.

Now, compare the handles. The handle with the greater angle fits on the **inlet** end of the flume and will match the angle of the flume side.

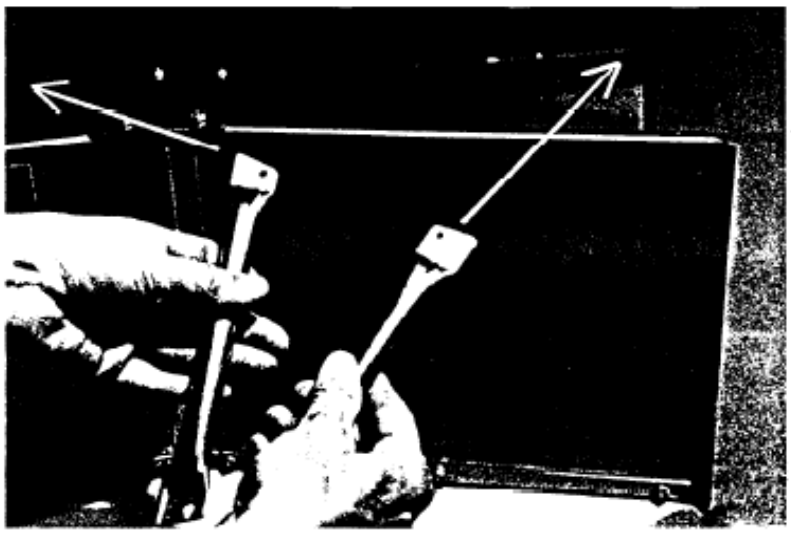


Photo D.

Place handle under the bent portion of side wall and secure by inserting bolt from the underside, then placing washer and screwing on nut from the topside for each hole. Follow same procedure for the second handle on other end of flume.

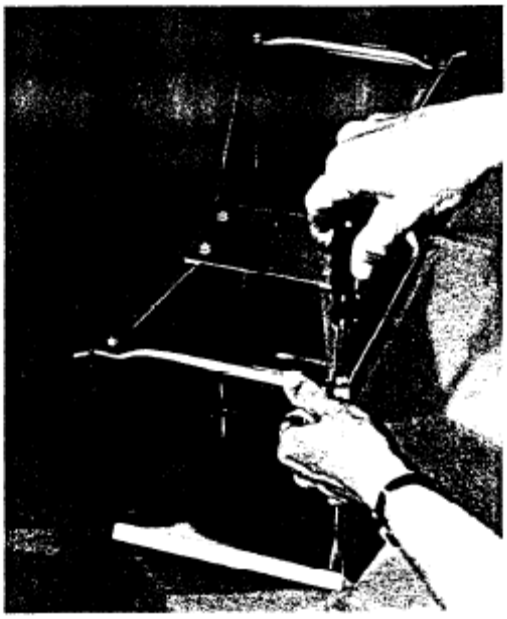


Photo E.

After all nuts are hand tightened on the three top pieces, use the nut driver to tighten somewhat more following the same procedure as for the bottom side. Finally, firmly and evenly, tighten all nuts on the top and bottom again (alternating as in Photo B). You may need to use the right angle screwdriver to hold some of the bolt heads while tightening.

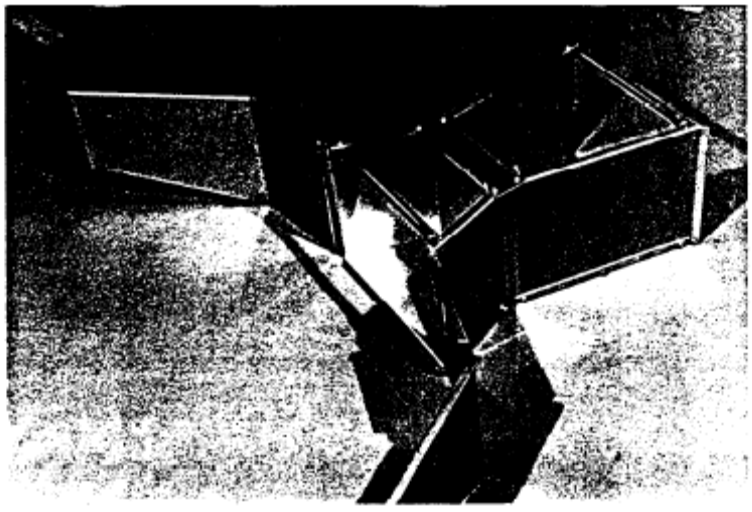


Photo F. Assembled 8" flume with optional 19" wing walls.

BASKI

Manufacturer of Inflatable Packers, Testing Tools and other products for investigating, controlling and producing the ***EARTH'S FLUIDS***
Phone: (303) 789-1200 • (800) 55 Baski • Fax (303) 789-0900

Collapsible Cutthroat Flume

GAGE (ft)	gpm 1"	gpm 2"	gpm 4"	gpm 8"	cfs 1"	cfs 2"	cfs 4"	cfs 8"
0.01	0.0225	0.0458	0.0932	0.190	0.00005	0.000102	0.000208	0.000422
0.02	0.090	0.183	0.373	0.760	0.0002	0.000408	0.000832	0.00169
0.03	0.203	0.412	0.839	1.71	0.00045	0.000918	0.00187	0.00380
0.04	0.360	0.733	1.49	3.04	0.0008	0.00163	0.00333	0.00675
0.05	0.563	1.15	2.33	4.75	0.00125	0.00255	0.00520	0.0106
0.06	0.810	1.65	3.36	6.84	0.0018	0.00367	0.00749	0.0152
0.07	1.10	2.24	4.57	9.31	0.00245	0.00500	0.0102	0.0207
0.08	1.44	2.93	5.96	12.2	0.0032	0.00653	0.0133	0.0270
0.09	1.82	3.71	7.55	15.4	0.00405	0.00826	0.0168	0.0342
0.10	2.25	4.58	9.32	19.0	0.005	0.0102	0.0208	0.0422
0.11	2.72	5.54	11.3	23.0	0.00605	0.0123	0.0252	0.0511
0.12	3.24	6.60	13.4	27.4	0.0072	0.0147	0.0300	0.0608
0.13	3.80	7.74	15.8	32.1	0.00845	0.0172	0.0352	0.0713
0.14	4.41	8.98	18.3	37.2	0.0098	0.0200	0.0408	0.0827
0.15	5.06	10.3	21.0	42.8	0.0113	0.0229	0.0468	0.0949
0.16	5.76	11.7	23.9	48.6	0.0128	0.0261	0.0532	0.108
0.17	6.50	13.2	26.9	54.9	0.0145	0.0295	0.0601	0.122
0.18	7.29	14.8	30.2	61.6	0.0162	0.0330	0.0674	0.137
0.19	8.12	16.5	33.6	68.6	0.0181	0.0368	0.0751	0.152
0.20	9.00	18.3	37.3	76.0	0.020	0.0408	0.0832	0.169
0.21	9.92	20.2	41.1	83.8	0.0221	0.0450	0.0917	0.186
0.22	10.9	22.2	45.1	92.0	0.0242	0.0494	0.101	0.204
0.23	11.9	24.2	49.3	101	0.0265	0.0540	0.110	0.223
0.24	13.0	26.4	53.7	109	0.0288	0.0588	0.120	0.243
0.25	14.1	28.6	58.3	119	0.0313	0.0638	0.130	0.264
0.26	15.2	31.0	63.0	128	0.0338	0.0690	0.141	0.285
0.27	16.4	33.4	67.9	139	0.0365	0.0744	0.152	0.308
0.28	17.6	35.9	73.1	149	0.0392	0.0800	0.163	0.331
0.29	18.9	38.5	78.4	160	0.0421	0.0858	0.175	0.355
0.30	20.3	41.2	83.9	171	0.0450	0.0918	0.187	0.380
0.31	21.6	44.0	89.6	183	0.0481	0.0980	0.200	0.406
0.32	23.0	46.9	95.4	195	0.0512	0.104	0.213	0.432
0.33	24.5	49.9	101	207	0.0545	0.111	0.227	0.460
0.34	26.0	52.9	108	220	0.0578	0.118	0.240	0.488
0.35	27.6	56.1	114	233	0.0613	0.125	0.255	0.517

Collapsible Cutthroat Flume

GAGE (ft)	gpm 1"	gpm 2"	gpm 4"	gpm 8"	cfs 1"	cfs 2"	cfs 4"	cfs 8"
0.36	29.2	59.4	121	246	0.0648	0.132	0.270	0.547
0.37	30.8	62.7	128	260	0.0685	0.140	0.285	0.578
0.38	32.5	66.1	135	274	0.0722	0.147	0.300	0.609
0.39	34.2	69.7	142	289	0.0761	0.155	0.316	0.642
0.40	36.0	73.3	149	304	0.0800	0.163	0.333	0.675
0.41	37.8	77.0	157	319	0.0841	0.171	0.350	0.709
0.42	39.7	80.8	164	335	0.0882	0.180	0.367	0.744
0.43	41.6	84.7	172	351	0.0925	0.189	0.385	0.780
0.44	43.6	88.7	180	368	0.0968	0.197	0.403	0.817
0.45	45.6	92.7	189	385	0.101	0.207	0.421	0.855
0.46	47.6	96.9	197	402	0.106	0.216	0.440	0.893
0.47	49.7	101	206	420	0.110	0.225	0.459	0.932
0.48	51.8	106	215	438	0.115	0.235	0.479	0.972
0.49	54.0	110	224	456	0.120	0.245	0.499	1.01
0.50	56.3	115	233	475	0.125	0.255	0.520	1.06
0.51	58.5	119	242	494	0.130	0.265	0.541	1.10
0.52	60.8	124	252	514	0.135	0.276	0.562	1.14
0.53	63.2	129	262	534	0.140	0.287	0.584	1.19
0.54	65.6	134	272	554	0.146	0.297	0.607	1.23
0.55	68.1	139	282	575	0.151	0.309	0.629	1.28
0.56	70.6	144	292	596	0.157	0.320	0.652	1.32
0.57	73.1	149	303	617	0.162	0.331	0.676	1.37
0.58	75.7	154	314	639	0.168	0.343	0.700	1.42
0.59	78.3	159	324	661	0.174	0.355	0.724	1.47
0.60	81.0	165	336	684	0.180	0.367	0.749	1.52
0.61	83.7	170	347	707	0.186	0.380	0.774	1.57
0.62	86.5	176	358	730	0.192	0.392	0.800	1.62
0.63	89.3	182	370	754	0.198	0.405	0.826	1.67
0.64	92.2	188	382	778	0.205	0.418	0.852	1.73
0.65	95.1	194	394	803	0.211	0.431	0.879	1.78
0.66	98.0	200	406	828	0.218	0.444	0.906	1.84
0.67	101	206	418	853	0.224	0.458	0.934	1.89
0.68	104	212	431	879	0.231	0.472	0.962	1.95
0.69	107	218	444	905	0.238	0.486	0.990	2.01
0.70	110	224	457	931	0.245	0.500	1.02	2.07
0.71	113	231	470	958	0.252	0.514	1.05	2.13
0.72	117	237	483	985	0.259	0.529	1.08	2.19
0.73	120	244	497	1013	0.266	0.544	1.11	2.25
0.74	123	251	510	1040	0.274	0.559	1.14	2.31

WORDS\INHOUSE\CTF.SAM Rev. 5/95

22.1. Baski Cutthroat Flume Rating Table (MS word)

BASKI Inc., Collapsible Cutthroat Flume, Rating Table, 1586 South Robb Way, Denver CO 80232

Gage (ft)	Discharge							
	gpm (gallons per minute) Throat Width				cfs (cubic feet per second) Throat Width			
	1”	2”	4”	8”	1”	2”	4”	8”
0.01	0.0225	0.0458	0.0932	0.190	0.00005	0.000102	0.000208	0.000422
0.02	0.090	0.183	0.373	0.760	0.0002	0.000408	0.000832	0.00169
0.03	0.203	0.412	0.839	1.71	0.00045	0.000918	0.00187	0.00380
0.04	0.360	0.733	1.49	3.04	0.0008	0.00163	0.00333	0.00675
0.05	0.563	1.15	2.33	4.75	0.00125	0.00255	0.00520	0.0106
0.06	0.810	1.65	3.36	6.84	0.0018	0.00367	0.00749	0.0152
0.07	1.10	2.24	4.57	9.31	0.00245	0.00500	0.0102	0.0207
0.08	1.44	2.93	5.96	12.2	0.0032	0.00653	0.0133	0.0270
0.09	1.82	3.71	7.55	15.4	0.00405	0.00826	0.0168	0.0342
0.10	2.25	4.58	9.32	19.0	0.005	0.0102	0.0208	0.0422
0.11	2.72	5.54	11.3	23.0	0.00605	0.0123	0.0252	0.0511
0.12	3.24	6.60	13.4	27.4	0.0072	0.0147	0.0300	0.0608
0.13	3.80	7.74	15.8	32.1	0.00845	0.0172	0.0352	0.0713
0.14	4.41	8.98	18.3	37.2	0.0098	0.0200	0.0408	0.0827
0.15	5.06	10.3	21.0	42.8	0.0113	0.0229	0.0468	0.0949
0.16	5.76	11.7	23.9	48.6	0.0128	0.0261	0.0532	0.108
0.17	6.50	13.2	26.9	54.9	0.0145	0.0295	0.0601	0.122
0.18	7.29	14.8	30.2	61.6	0.0162	0.0330	0.0674	0.137
0.19	8.12	16.5	33.6	68.6	0.0181	0.0368	0.0751	0.152
0.20	9.00	18.3	37.3	76.0	0.020	0.0408	0.0832	0.169
0.21	9.92	20.2	41.1	83.8	0.0221	0.0450	0.0917	0.186
0.22	10.9	22.2	45.1	92.0	0.0242	0.0494	0.101	0.204
0.23	11.9	24.2	49.3	101	0.0265	0.0540	0.110	0.223
0.24	13.0	26.4	53.7	109	0.0288	0.0588	0.120	0.243
0.25	14.1	28.6	58.3	119	0.0313	0.0638	0.130	0.264
0.26	15.2	31.0	63.0	128	0.0338	0.0690	0.141	0.285
0.27	16.4	33.4	67.9	139	0.0365	0.0744	0.152	0.308
0.28	17.6	35.9	73.1	149	0.0392	0.0800	0.163	0.331
0.29	18.9	38.5	78.4	160	0.0421	0.0858	0.175	0.355
0.30	20.3	41.2	83.9	171	0.0450	0.0918	0.187	0.380
0.31	21.6	44.0	89.6	183	0.0481	0.0980	0.200	0.406
0.32	23.0	46.9	95.4	195	0.0512	0.104	0.213	0.432
0.33	24.5	49.9	101	207	0.0545	0.111	0.227	0.460
0.34	26.0	52.9	108	220	0.0578	0.118	0.240	0.488
0.35	27.6	56.1	114	233	0.0613	0.125	0.255	0.517
0.36	29.2	59.4	121	246	0.0648	0.132	0.270	0.547
0.37	30.8	62.7	128	260	0.0685	0.140	0.285	0.578
0.38	32.5	66.1	135	274	0.0722	0.147	0.300	0.609
0.39	34.2	69.7	142	289	0.0761	0.155	0.316	0.642
0.40	36.0	73.3	149	304	0.0800	0.163	0.333	0.675
0.41	37.8	77.0	157	319	0.0841	0.171	0.350	0.709
0.42	39.7	80.8	164	335	0.0882	0.180	0.367	0.744
0.43	41.6	84.7	172	351	0.0925	0.189	0.385	0.780

Gage (ft)	Discharge							
	gpm (gallons per minute) Throat Width				cfs (cubic feet per second) Throat Width			
	1”	2”	4”	8”	1”	2”	4”	8”
0.44	43.6	88.7	180	368	0.0968	0.197	0.403	0.817
0.45	45.6	92.7	189	385	0.101	0.207	0.421	0.855
0.46	47.6	96.9	197	402	0.106	0.216	0.440	0.893
0.47	49.7	101	206	420	0.110	0.225	0.459	0.932
0.48	51.8	106	215	438	0.115	0.235	0.479	0.972
0.49	54.0	110	224	456	0.120	0.245	0.499	1.01
0.50	56.3	115	233	475	0.125	0.255	0.520	1.06
0.51	58.5	119	242	494	0.130	0.265	0.541	1.10
0.52	60.8	124	252	514	0.135	0.276	0.562	1.14
0.53	63.2	129	262	534	0.140	0.287	0.584	1.19
0.54	65.6	134	272	554	0.146	0.297	0.607	1.23
0.55	68.1	139	282	575	0.151	0.309	0.629	1.28
0.56	70.6	144	292	596	0.157	0.320	0.652	1.32
0.57	73.1	149	303	617	0.162	0.331	0.676	1.37
0.58	75.7	154	314	639	0.168	0.343	0.700	1.42
0.59	78.3	159	324	661	0.174	0.355	0.724	1.47
0.60	81.0	165	336	684	0.180	0.367	0.749	1.52
0.61	83.7	170	347	707	0.186	0.380	0.774	1.57
0.62	86.5	176	358	730	0.192	0.392	0.800	1.62
0.63	89.3	182	370	754	0.198	0.405	0.826	1.67
0.64	92.2	188	382	778	0.205	0.418	0.852	1.73
0.65	95.1	194	394	803	0.211	0.431	0.879	1.78
0.66	98.0	200	406	828	0.218	0.444	0.906	1.84
0.67	101	206	418	853	0.224	0.458	0.934	1.89
0.68	104	212	431	879	0.231	0.472	0.962	1.95
0.69	107	218	444	905	0.238	0.486	0.990	2.01
0.70	110	224	457	931	0.245	0.500	1.02	2.07
0.71	113	231	470	958	0.252	0.514	1.05	2.13
0.72	117	237	483	985	0.259	0.529	1.08	2.19
0.73	120	244	497	1013	0.266	0.544	1.11	2.25
0.74	123	251	510	1040	0.274	0.559	1.14	2.31

23. Appendix B: Field_forms.xls premobilization worksheet meta data

item 1	item 2	cell range	description
	Date	B3	date of premob test
	Start time	B4	start time of testing
	End time	D4	finish time of testing
	Operator	C5	name of person conducting test
	Inst. Mfr.	C6	maker of multiparameter probe
	Sensor serial number	C7	serial number of probe body
	Model	C8	model of probe body
	Display	C9	unit that allows viewing parameter data
	Display voltage (%)	C10	voltage of display unit
	Sonde (v)	C11	voltage of probe
	Sondepower (%)	C12	power remaining in probe
	Notes:	C13	any notes specific to this calibration and error check procedure
	Sensor	H4:H9	type of sensor attached to probe body
	Sensor ID	H4:H9	identification number of sensor attached to probe body

Error Check Solution: Sink water, and sodium sulfite solution

Pre-Cal. Measurements	MP temp ^{O}F	C18	multiparameter probe temp (degrees F)
Pre-Cal. Measurements	NIST Thermometer ^{O}C	C19	NITST thermometer (degrees C)
Pre-Cal. Measurements	SC (us/cm)	C22	multiparameter probe specific conductivity (uc/cm)
Pre-Cal. Measurements	pH	C23	multiparameter probe pH
Pre-Cal. Measurements	DO (mg/L)	C24	multiparameter probe DO (mg.L)
Pre-Cal. Measurements	BP (mmHg)	C25	multiparameter probe barometric pressure (mmHg)
Post-Cal. Measurements	MP temp ^{O}F	J18	multiparameter probe temp (degrees F)
Post-Cal. Measurements	NIST Thermometer ^{O}C	J19	NITST thermometer (degrees C)
Post-Cal. Measurements	SC (us/cm)	J22	multiparameter probe specific conductivity (uc/cm)
Post-Cal. Measurements	pH	J23	multiparameter probe pH
Post-Cal. Measurements	DO (mg/L)	J24	multiparameter probe DO (mg.L)
Post-Cal. Measurements	BP (mmHg)	J25	multiparameter probe barometric pressure (mmHg)
Temperature error check	%Error	I28	automatically calculated percent error
Temperature error check	Allowable?	J28	answers question is this level of erorr OK?
	pre-mob_pre file name	A31	name of data file where premobilization sink

			water data is stored
	pre_mob_post file name	G31	name of data file where premobilization post-calibration sink water data is stored

Specific Conductance Calibration (1413 µS/cm std.) & Error Check @ 100 and 10,000 µS/cm

	precal MP temp OC	C34:E34	multiparameter probe temperature reading in conductivity standard (degrees C)
	post cal MP temp OC	G34:I35	multiparameter probe temperature reading in conductivity standard (degrees C)
	Kcell	B40	Kcell constant resulting from calibration
	Allowable?	C40	answers is this cell constant in the expected range?
	%Error	H40:H42	automatically calculates percent error
	Allowable?	I40:I42	answers is this error allowable based on WRD guidance
	maintenance needed?	C45	answers is maintenance on the sensor needed?

pH Calibration @ pH 7 & pH 10 su and Error Check @ pH 4

Pre-Cal. Reading	reading	B52:B54	reading in the indicated pH buffer solution
Pre-Cal. Reading	lot #	C52:C55	lot number of the indicated pH buffer solution
Pre-Cal. Reading	Exp	D52:D56	expiration date of the indicated pH buffer solution
Pre-Cal. Reading	maker	E52:E57	manufacturer of the indicated pH buffer solution
Pre-Cal. Reading	Cat #	F52:F58	catalog number of the indicated pH buffer solution
Post-Cal. Reading	%Error	I52:I54	percent error
Post-Cal. Reading	Allowable?	J52:J54	answers is this error level allowable?
	voltage pH 4 std	C57	sensor voltage (mV)
	voltage pH 7 std	C58	sensor voltage (mV)
	voltage pH 10 std	C59	sensor voltage (mV)
	temp deg C	F57:F59	temperature of corresponding buffer solution
For 2 point calibration	Slope 1 mV/pH	E62	slope of calibration curve (mV/pH), 2pt calib
For 2 point calibration	Offset 1 mV	E63	offset of calibration curve (mV), 2 pt calib
If making a 3 point calibration	Slope 2 mV/pH	I62	slope of calibration curve (mV/pH), 3 pt calib
If making a 3 point calibration	Offset 2 mV	I63	offset of calibration curve (mV), 3 pt calib
	offset check	D66	check for offset within acceptable range
	slope check	D67	checl for slope within acceptable range
	maintenance needed?	D68	answers is maintenance needed?

Dissolved Oxygen (DO):

	Membrane thickness (mil)	C74	thickness of DO membrane (mil)
	Calib Method	C75	method used for calibration
	Edit baro. setting?	C76	was the barometric pressure value set by user?
	stimulus	C77	the maximum DO concentration for the measured temperature and pressure at time of calibration 9mg/L)
	Pre-Cal. Reading	C78	reading of DO prior to calibration with sensor in solution indicated in C75 (mg/L)
	Slope	C79	calibration slope (nA/mg/L)
	Offset	C80	calibration offset (nA)
	temp. during cal	C81	temperature during calibration (degrees C)
	baro. during cal	C82	barometric pressure during calibration (degrees C)
	Manuf. Date	E74	manufacture date of membrane cap
Error check1	Method	G75	method used for error check
Error check1	Edit baro. setting?	G76	was the barometric pressure value set by user?
Error check1	stimulus	G77	the maximum DO concentration for the measured temperature and pressure at time of calibration 9mg/L)
Error check1	Pre-Cal. Reading	G78	reading of DO prior to calibration with sensor in solution indicated in C75 (mg/L)
Error check2	Method	K75	method used for error check
Error check2	Edit baro. setting?	K76	was the barometric pressure value set by user?
Error check2	stimulus	K77	the maximum DO concentration for the measured temperature and pressure at time of calibration 9mg/L)
Error check2	Pre-Cal. Reading	K78	reading of DO prior to calibration with sensor in solution indicated in C75 (mg/L)
	Post-Cal. Reading1	C86	post calibration reading with sensor in C75 (mg/L)
	Post-Cal. Reading2	G86	post calibration reading with sensor in G75 (mg/L)
	Post-Cal. Reading3	K86	post calibration reading with sensor in K75 (mg/L)
	%Error	I89:I90	percent error between stimulus and reading in error check solutions
	Allowable?	J89:J91	is this error allowable based on WRD guidance
	Notes:	B99	notes realted to DO calibration
	maintenance needed?	C94	based on acceptable range of calibration curve slope and offset is maintenance needed?

<u>Turbidity Calibration</u>: with primary formazin standard (Minimum once every 3 months)

Gelex	Measured value (NTU)	C107:G107	value of currently measured gelex standard (NTU) Applies to EPA Method 180.1 and Hach 2100P field instrument

Gelex	% rel. diff.	C108:G108	difference between assigned value and current reading
	± 3% Y/N	C109:G109	is the relative difference greater than 3%?
	Recalibration necessary?	G110	is a formazin recalibration necessary?
	Standard Value	B112:E112	formazin standard value (NTU)
	Lot Number	B113:E113	lot number of formazin standard
	Exp. Date	B114:E114	expiration date of formazin standard
	Assigned value to Gelex	C116:E116	expected range of gelex standard (NTU)
	Secondary standards	C117:E117	assigned value based on most current calibration (NTU)
	Date of Latest calibration and assignment of Gelex values	G119	most recent calibration and assignment of values to gelex standards

24. Appendix C: Field_forms.xls meta data

The following fields exist in each individual sheet corresponding to unique field sites. The field site name is located in cell B5 of each sheet and also in abbreviated form on the notebook page tab at the bottom of each notebook page.

item 1	item2	data range	meta data
	Station #	B4	NCPN station identification number
	Station name	B5	abbreviated station name in text
	Lat	B6	latitude of station
	Long	B7	longitude of station
	altitude (ft)	B8	altitude of statoin
	7.5' quad	B9	7.5 minute quadrangle name on which station resides
	Storet #	B10	Environmental Protection Agency water quality data base station identification number
	Comments	B11	general comments related to sampling conditions
	Sample team	E5	sampling team members
	map datum	E8	map datum related to Lat and Long
	Date(m/d/yr)	I4	date of data and sample collect
	Start time	I5	time of data and sample collect
	End time	I6	end time of data and sample collect
	Mean time	I7	average of start and end times
	Sample collected at	I8	time when bottles filled with ambient water. This time agrees with the time labeled on the bottles
	Total chem	C17	was a water sample collected for total chemical analysis (y/n)?
	Filt. metals	D17	was a water sample collected for filtered metals analysis (y/n)?
	Nutrients	E17	was a water sample collected for nutrient analysis (y/n)?
	Turbidity	F17	was a water sample collected for turbidity analysis (y/n)?
	Carry	G17	was a water sample collected for carrying back to vehicle or lab (y/n)?
	Microbiology	C19	was a water sample collected for microbiological analysis (y/n)?
	Secchi disc	D19	was a secchi disc reading made?
	Chl. a	E19	was a sample collected for chlorophyl a?
	Algae	F19	was a sample for algae collected?
	Inverts	G19	were invertebrates collected?
	Date & time	C22	were the date and time recorded on the sample bottle?
	STORET #	D22	was the storet # recorded on the sample bottle
	Collector	E22	was the collectors name recorded on the sample bottle?
	Site name	F22	was the site name (text) recorded on the sample bottle?
	Sample period (m / q)	C23	were the water samples collected for standard monthly analysis run or quarterly metals run
	Filter packs	I23	manufacturer, model number and pore diameter of filter used to filter sample for trace elements analysis
	Lot#	I25	filter pack lot number
	suite Total Chemistry	C25	laboratory analysis suite for total chemistry analysis
	suite Filtered metals	C26	labotatory analysis suite for filtered metals
	suite Nutrients	C27	laboratory analysis suite for nutrients
	preservative chemistry	D25	preservative used in chemistry bottle shipped from lab
	preservative filtered metals	D26	preservative used in filtered metals bottle shipped from lab
	preservative	D27	preservative used in non-filtered nutrient bottle shipped from

item 1	item2	data range	meta data	
	non-filtered metals		lab	
	lot # Total Chemistry	E25	UT laboratory lot number for Total Chemistry bottle	
	lot # Filtered metals	E26	UT laboratory lot number for Filtered metals bottle	
	lot # Nutrients	E27	UT laboratory lot number for Nutrients bottle	
	CAS# Total Chemistry	F25	Chemical abstracts service # unique identifier for preservative in Total Chemistry bottle	Chemical Abstracts Service 2540 Olentangy River Road P.O. Box 3012, Columbus, Ohio 43210, Phone 800-848-6538, http://www.cas.org/EO/regsys.html
	CAS# Filtered metals	F26	Chemical abstracts service # unique identifier for preservative in Filtered metals bottle	Chemical Abstracts Service 2540 Olentangy River Road P.O. Box 3012, Columbus, Ohio 43210, Phone 800-848-6538, http://www.cas.org/EO/regsys.html
	CAS# Nutrients	F27	Chemical abstracts service # unique identifier for preservative in Nutrients bottle	Chemical Abstracts Service 2540 Olentangy River Road P.O. Box 3012, Columbus, Ohio 43210, Phone 800-848-6538, http://www.cas.org/EO/regsys.html
	Exp date Total Chemistry	G25	Expiration date for Total Chemistry bottle	
	Exp date Filtered metals	G26	Expiration date for Filtered Metals bottle	
	Exp date Nutrients	G27	Expiration date for Nutrients bottle	
	Sample type	B28	this is a state of UT field code referring to how sample was collected with some indication of its intended purpose	
	Source code	B29	this is a state of UT field code that indicates type of water body from which the sample was collected	
	Cost code	B30	this is a state of UT field code that indicates this sample is charged to National Park Service account	
	Agency	B31	this is a state of UT field code that indicates this sample is charged to National Park Service account	
	Date shipped (m/d/yr)	F29	date samples were shipped to state of UT lab from Bryce Canyon National Park	
	UPS tracking #	F30	United States Parcel Service tracking number	
	Date received (m/d/yr)	F31	date samples were received by state of UT laboratory	
measurable	dischar. (y/n)Color	A35	was there sufficient flow to be able to take a discharge measurement?	
Water	color	B35	what was the water color?	
Visual est.	of mixing	C35	was the water body well mixed?	
	disch.method	A38	method used to measure discharge	
	serial #	B38	instrument used to measure discharge	
	flow value	C38	discharge value	
units	(C,G,M,blank)	D38	units of discharge value	
meas type	(M,E,blank)	E38	was the discharge measured or estimated?	
avg veloc	(ft/s, blank)	F38	average velocity of water moving past the cross section where discharge was measured	

item 1	item2	data range	meta data
vtype	(M,E,blank)	G38	was the velocity of discharge measured or estimated?
	Air Temp(oC)	H40	air temperature in degrees celcius
	Gage ht.(ft)	B39	units in feet of stream gage height if present
	Q inst.(cfs)	B40	instantaneous discharge in cubic feet per second measured by any method
	AquaCalc file name	C41	file name of data collected with flow meter
	current meter discharge(cfs)	B43	measured flow in cubic feet per second if measured with a current meter
hydrograph	stage	E42	dynamic state of water level at time of sampling
flow	severity	F42	description of stage at time of sampling
	estimated discharge (cfs)	B4	discharge value determined by estimate
	estimate type	C45	description of how discharge was measured if not by quantitative method
Volumetric	*measurement*	A48:A52	number of measurement taken at site for determination of discharge volumetrically
	vol.(mL)	B48:B52	volume of water collected in mL
	time (s)	C48:C52	time to collect volume of water in seconds
	(mL/s)	D48:D52	automatically calculated flow rate mL/s
	(gpm)	E48:E52	automatically calculated flow rate in gallons per minute
	(cfs)	F48:F52	automatically calculated flow rate in cubic feet per second
estimated	% loss	G48:G52	estimate of flow not captured by measurement method, units in percentage eg 5% loss recorded as 5
adjusted flow	*(cfs)*	H48:H52	automatically calculated flow corrected for estimated percent loss not captured by method, units in cubic feet per second
	stable flow?	D53	automatically calculated determination of consistency of flow measurement within 5% of the mean of 5 measurements.
	average	H53	automatically calculated average flow value is the mean of 5 adjusted flow values, units cubic feet per second
Weir	*measurement*	A57:A61	number of measurement taken at site for determination of discharge with a weir
	stage (cm?)	B57:B61	water level height above weir notch in centimeters
Look up	*(gpm)*	C57:C61	table lookup value for flow associated with stage, units gallons per minute
	(cfs)	F57:F61	automatically calculated flow, units cubic feet per second
estimated	% loss	G57:G61	estimate of flow not captured by measurement method, units in percentage eg 5% loss recorded as 5
adjusted flow	*(cfs)*	H57:H61	automatically calculated flow corrected for estimated percent loss not captured by method, units in cubic feet per second
	stable flow?	D62	automatically calculated determination of consistency of flow measurement within 5% of the mean of 5 measurements.
	average	H62	automatically calculated average flow value is the mean of 5 adjusted flow values, units cubic feet per second
Flume	*measurement*	A66:A70	number of measurement taken at site for determination of discharge with a flume
	stage (cm?)	B66:B70	water level height above weir notch in centimeters
Look up	*(gpm)*	C66:C70	table lookup value for flow associated with stage, units gallons per minute
	(cfs)	F66:F70	automatically calculated flow corrected for estimated percent loss not captured by method, units in cubic feet per second
estimated	*% loss*	G66:G70	automatically calculated determination of consistency of flow measurement within 5% of the mean of 5 measurements.
adjusted	*(cfs)*	H66:H70	automatically calculated average flow value is the mean of 5

item 1	item2	data range	meta data
flow			adjusted flow values, units cubic feet per second
	stable flow?	D71	automatically calculated determination of consistency of flow measurement within 5% of the mean of 5 measurements.
	average	H71	automatically calculated average flow value is the mean of 5 adjusted flow values, units cubic feet per second
	Discharge comments	B74	any comments relative to flow determinatoin
	Reading #	B80	numeric order of core parameter collection
DO	(mg/L)	B78:84	dissolved oxygen, units milligrams/Liter
Baro. Press.	(mm Hg)	C78:C84	barometric pressure, units mg/L
Spc. Cond	uS/cm@25 oC	D78:D84	conductivity temperature corrected is specific conductance, units micro semens per centimeter
Water	temp oC	E78:E84	water temperature, units degrees celsius
pH	unitless	F78:F84	pH, unitless
	Troll 9000 file name	H79	Troll sonde file name where core parameter data are stored
	median	B85:F85	automatically calculated median of 5 values for DO, Pressure, Specific conductance, water temp and pH
	range	B86:F86	automatically calculated range of 5 values for DO, Pressure, Specific conductance, water temp and pH
	well mxd?	B87, D87:F87	automatically calculated estimate of water column mixing based on standard deviation of core parameters
	DO sat(%)	B88	automatically calculated percentage of dissolved oxygen saturation corrected for barometric pressure and salinity
	Turbid(ntu)	B89	turbidity, units ntu
	Secchi disc (m)	H88	depth beneath water surface where secchi disc is no longer visible, units meters
	E. Coli (col/100mL)	H89	count of Total fecal coliform bacteria colony forming units on petri plate after incubation, units colonies per 100 mL of sample water
	Tot. Coliform (col/100mL)	H90	count of E. coli colony forming units on petri plate after incubation, units colonies per 100 mL of sample water
	Weather	B91:E91	this is a state of UT field conditions code, up to 4 per site visit
	Field condns		this is a state of UT weather conditions code, up to 4 per site visit
Liquid filled	air temp oC	A97	temperature in shaded location in air measured with a liquid filled thermometer, units degrees C
Liquid filled	air temp oF	B97	temperature in shaded location in air measured with a liquid filled thermometer, units degrees F
Troll 9000	air temp oC	D97	temperature in shaded location in air measured with Troll 9000 sonde, units degrees C
Troll 9000	air temp oF	E97	temperature in shaded location in air measured with Troll 9000 sonde, units degrees F
Liquid filled	H2O temp oC	A100	temperature in water measured with a liquid filled thermometer, units degrees C
Liquid filled	H2O temp oF	B100	temperature in water measured with a liquid filled thermometer, units degrees F
Troll 9000	H2O temp oC	D100	temperature in water measured with Troll 9000 sonde, units degrees C
Troll 9000	H2O temp oF	E100	temperature in water measured with Troll 9000 sonde, units degrees F
Spin test	pygmy	B107:C107	length of time pygmy meter spins under stable conditions
Spin test	price AA	B108:C108	length of time price meter spins under stable conditions

item 1	item2	data range	meta data
Spin test	Date	D107:D108	date spin test was conducted
Turbidity	date	B113	date turbidity measured
Turbidity	time	C113	time turbidity measured
Signal avg	(on/off)	D113	instrument signal averaging turned on or off
Wavelength	low	F113	lower bandwidth of light used to measure turbidity, units nanometers
Wavelength	high	G113	upper bandwidth of light used to measure turbidity, units nanometers
	manufacturer	A117:A120	manufacturer of turbidity standards
	Cat #	B117:B120	Hach chemical company catalog numbers for turbidity standards
	NTU	C117:C120	value for turbidity standard, units ntu
	Variation	D117:D120	expected variance of turbidity standard, units ntu
	Lot #	E117:E120	lot number of turbidity standard
	expiration date	F117:F120	expiration date of turbidity standards
	Callibration accepted? (y/n)	D122	answers whether the turbidity calibration accepted?
	Secondary gelex standards (ntu)	C124:C126	after calibration, secondary standards are read as a double check on calibration, units ntu
	Cond of std used for calibration	D130	conductivity standard used for single point calibration, units (us/cm)
	Cond. reading prior to calibration	D131	conductivity value that instrument reads with standard in calibration cup prior to calibration
	manufacturer	D132	manufacturer of calibration solution
	lot #	D133	lot number of calibration standard
	CAS#	D134	chemical abstracts service identification number for calibration solution
	expiration date	D135	expiration data of calibration solution
	pH low	D138	pH of low end of 2 point calibration
	pH prior to calibration	D139	pH value that instrument reads with standard in calibration cup prior to calibration
	manufacturer	D140	manufacturer of pH solution
	lot #	D141	lot number of pH standard
	expiration date	D142	chemical abstracts service identification number for calibration solution
	pH high	H138	expiration data of calibration solution
	pH prior to calibration	H139	pH of high end of 2 point calibration
	manufacturer	H140	pH value that instrument reads with standard in calibration cup prior to calibration
	lot #	H141	manufacturer of pH solution
	expiration date	H142	expiration data of calibration solution
	DO pre calib	B146	DO reading in air saturated with water prior to calibration
	DO post calib	B147	DO reading in air saturated with water post calibration
Cond	Device	D154	Device serial number of Troll 9000 used to make insitu

item 1	item2	data range	meta data
			measurements
Cond	Sensor1	D155	sensor serial number for conductivity sensor
Cond	Sensor2	D156	conductivity range of sensor low or high
Cond	Sensor3	D157	date of sensor manufacture
Cond	Method	B158	method of calibration, usually "Traditional" meaning 1 point
Cond	Action	B159	answers whether the conductivity calibration was completed or cancelled
Cond	Status	B160	indicates the stability of readings as calibration was being conducted. If status is nominal then calibration point will be designated "user set"
Cond	Date	B161	date of calibration
Cond	Time	B162	time of calibration
Cond	Last	C163:D163	date and time of last calibration
Cond	Cal	A165:B166	number of calibration points used in calibration
Cond	Stimulus	C167	conductivity of calibration solution (uc/cm)
Cond	Response	C168	resistivity of measured solution, units = ohms
Cond	Final	D169	the conductivity reading after calibration, units (us/cm)
Cond	Temperature	C170	the temperature of the conductivity standard, units degrees C
Cond	Kcell	C171	cell constant for the calibration
PH	Device	K154	Device serial number of Troll 9000 used to make insitu measurements
PH	Sensor1	K155	sensor serial number for pH sensor
PH	Sensor2	K156	date of sensor manufacture
PH	Method	I157	method of calibration, usually "Traditional" meaning 2 points
PH	Action	I158	answers whether the pH calibration was completed or cancelled
PH	Status	I159	indicates the stability of readings as calibration was being conducted. If status is nominal then calibration point will be designated "user set"
PH	Date	I160	date of calibration
PH	Time	I161	time of calibration
PH	Last	K162:L162	date and time of last calibration
PH	Cal	K163	number of solution standards used in calibration
PH	Point1	I164	calibration point number
PH	Stimulus	J166	calibration solution standard pH value, units pH
PH	Response	K168	sensor reading in response to calibration solution standard number 1, units mV
PH	Final	K170	reading after calibrating point 1, units pH
PH	Temperature	J172	temperature of calibration solution, units degrees C
PH	Point2	I173	calibration point number
PH	Stimulus	J176	calibration solution standard pH value, units pH
PH	Response	J179	sensor reading in response to calibration solution standard number 1, units mV
PH	Final	K182	reading after calibrating point 1, units pH
PH	Temperature	J185	temperature of calibration solution, units degrees C
PH	Slope	J188	slope of calibration equation, units mV/pH
PH	Offset	J191	intercept of calibration equation, units mV
DO	Device	D194	serial number for Troll 9000
DO	Sensor	D195	Dissolved oxygen sensor serial number
DO	Sensor	D196	sensor manufacture date
DO	Method	B197	method used to calibrate dissolved oxygen, typically

item 1	item2	data range	meta data
			"Traditional"
DO	Action	B198	answers whether the dissolved oxygen calibration was completed or cancelled
DO	Status	B199:C199	indicates the stability of readings as calibration was being conducted. If status is nominal then calibration point will be designated "user set"
DO	Date	B200	date of calibration
DO	Time	B201	time of calibration
DO	Last	C202:CE202	date and time of last calibration
DO	Cal	C203	number of points used in calibration
DO	Membrane:	B204	thickness of dissolved oxygen sensor membrane
DO	Saturation1	C205:H205	sensor environment during calibration
DO	Saturation2	D206	did the user preset the dissolved oxygen concentration
DO	Temperature	C208	water temperature in chamber during calibration, units degrees C
DO	Barometric	D209	barometric pressure in chamber at time of calibration, units mBar
DO	Stimulus	C210	oxygen concentration of sensor in calibration conditions 1
DO	Response	C211	raw sensor reading, units nA
DO	Final	D212	oxygen concentration after calibration, units mg/L
DO	Slope	C213	slope of calibration equation, units nA/(mg/L)
DO	Offset	C214	intercept of calibration equation, units nA
Troll	Report generated:	B229:C229	date and time of report generation
Troll	Report from file:	B230	file path and name of file containing data collected at 5 equal increments across stream cross section
Troll	Win-Situ Version	B231	software and version used on hand held Rugged Reader computer to collect data and calibrate instrument
Troll	Serial number:	B233	serial number of Troll 9000
Troll	Firmware Version	B234	version of Troll 9000 operating system firmware
Troll	Unit name:	B235	name of instrument which was used to collect data
Troll	Test name:	C237	name of file where data is stored
Troll	Test defined on:	B239:C239	date and time when file was created
Troll	Test started on:	B240:C240	date and time when data collect began
Troll	Test stopped on:	B241:C241	date and time when data collect finished
Troll	Data stored if delta value exceeds:	B247	data stored if temperature > this value, units degrees F
Troll	Number of data samples:	B248	number of data points collected for each of the sensors mounted on the Troll sonde
Troll	TOTAL DATA SAMPLES	B251	total number of data samples collected foreach sensor mounted on the Troll 9000
Troll	Date	A282:undefined	date of data collect
Troll	Time	B282:undefined	time of data collect
Troll	ET (sec)	C282: undefined	time since "Test Started", units seconds
Troll	Temperature	D282: undefined	temperature of water, units F

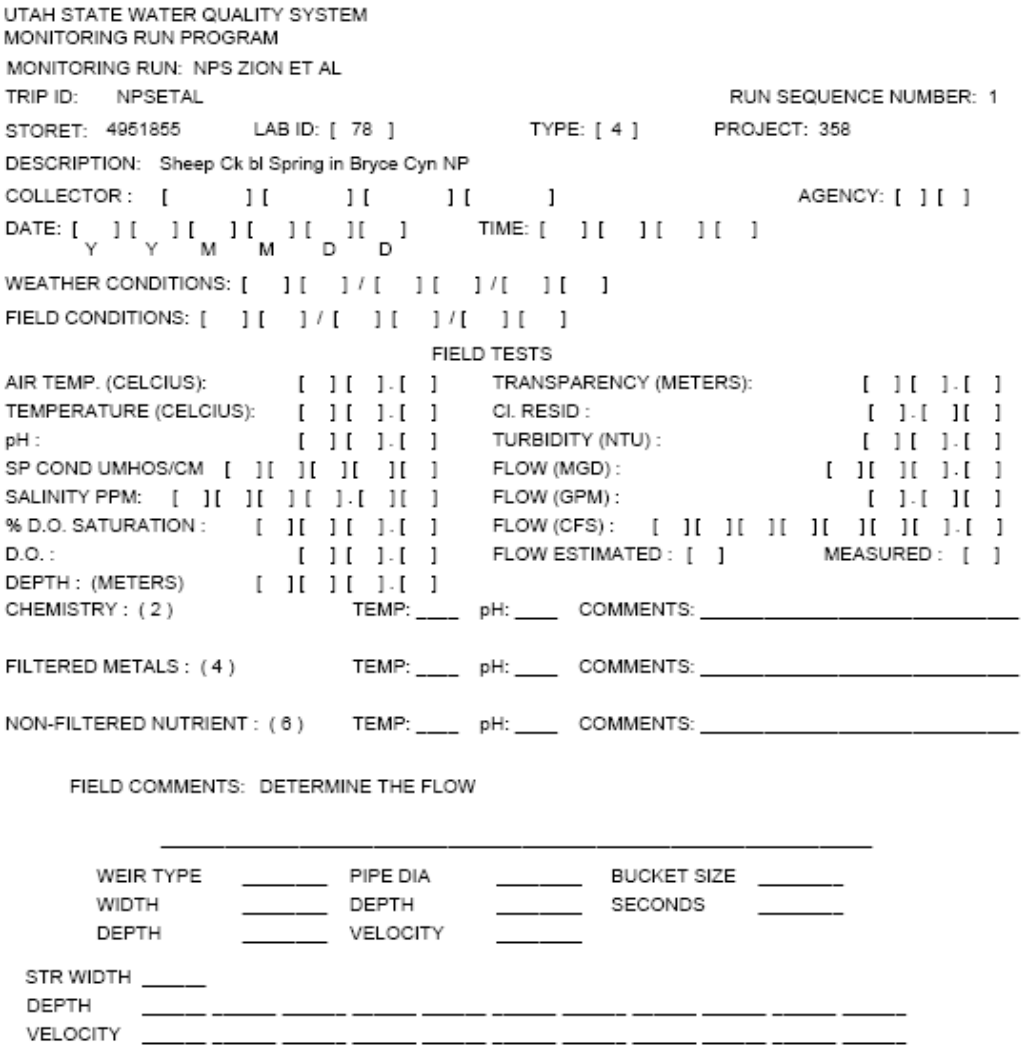
item 1	item2	data range	meta data
Troll	Barometric	EC282: undefined	barometric pressure, units mmHg
Troll	Battery	F282: undefined	battery voltage in Troll 9000, units Volts
Troll	Clark DO	G282: undefined	Dissolved Oxygen, units micrograms/Liter
Troll	pH	H282: undefined	pH
Troll	Conductivity	I282: undefined	Actual conductivity - not temperature corrected, units us/cm
AquaCalc	S/N:	B304	AquaCalc Pro serial number
AquaCalc	Firmware Version:	B305	firm ware version loaded onto AquaCalc pro
AquaCalc	File Version:	B306	file version of AquaCalc pro
AquaCalc	Gage ID:	B308	15 digit alpha numeric site identifier
AquaCalc	User ID:	B309	user name
AquaCalc	Meter name:	B310	name of flow meter used with AquaCalc pro
AquaCalc	Meter id:	B311	id number of meter
AquaCalc	Meter type:	B312	type of meter, either pygmy or price
AquaCalc	Meter Standard:	B313	measurement type SAE=english, SI=metric
AquaCalc	Meter Revs/Pulses:	B314	number of meter revolutions that equal one pulse
AquaCalc	Meter Const.S1:	B315	meter constant
AquaCalc	Meter Const.O1:	B316	meter constant
AquaCalc	Meter Const.C1:	B317	meter constant
AquaCalc	Meter Const.S2:	B318	meter constant
AquaCalc	Meter Const.O2:	B319	meter constant
AquaCalc	Meter Const.C1:	B320	meter constant
AquaCalc	Meter Const.S3:	B321	meter constant
AquaCalc	Meter Const.O3:	B322	meter constant
AquaCalc	Beg Time:	B323	beginning time and date of measurement
AquaCalc	End Time:	B324	end time and date of measurement
AquaCalc	Meas Time:	B325	time for duration of entire measurement
AquaCalc	Section Diff:	B326	percent difference from estimated flwo
AquaCalc	Beg Gage height:	B327	stream gage reading at beginning of measurement, units feet
AquaCalc	End Gage height:	B328	stream gage reading at end of measurement, units feet
AquaCalc	Beg Staff height:	B329	stream staff reading at beginning of measurement, units feet
AquaCalc	End Staff height:	B330	stream staff reading at end of measurement, units feet
AquaCalc	Estimated Q:	B331	user input estimate of flow, units cubic feet per second
AquaCalc	Adjusted Q:	B332	user input estimate of adjustment to estimated flow, units cubic

item 1	item2	data range	meta data
			feet per second
AquaCalc	Measure time:	B333	duration of measurement at each stream vertical, units seconds
AquaCalc	Measure standard:	B334	measurement type SAE=english, SI=metric
AquaCalc	Measure equipment:	B335	typ of rod to which meter is attached
AquaCalc	Sounding Weight:	B336	standard weight and hanger bar configuration used if measurement mad from a bridge
AquaCalc	Measure ice:	B337	idnetifies whether ice draft measurement mode is used
AquaCalc	Flood Measurement:	B338	identifies whether .2 flood measure mode is used
AquaCalc	Flood Coef:	B339	a factor used in computation of flood flow when only a 0.2 depth measurement is possible
AquaCalc	Max Vertical Q:	B340	maximum allowable proportion of flow allowed through each vertical before error warning issued
AquaCalc	Percent Slope:	B341	slope of stream surface, required to calculate chezy and manning coefficients
AquaCalc	Measure Start at:	B342	edge of stream relative to flow direction from which measurements start, REW is right edge of water, LEW is left
AquaCalc	Vertical Count:	B343	number of verticals in the measurement cross section
AquaCalc	Section Velocity:	B344	40 second average velocity of water moving through the vertical, units feet per second
AquaCalc	Section Width:	B345	width of cross section measured, feet
AquaCalc	Section Area:	B346	cross sectional area of measured cross section, units feet squared
AquaCalc	Section Q:	B347	calculated flow through cross section, units cubic feet per second
AquaCalc	Section Diff:	B348	percent difference from estimated flwo
AquaCalc	Section Pct Err:	B349	% difference from estimated flow
AquaCalc	Section Quality:	B350	user assessment as to the quality of the discharge measurement, units excellent, good, fair, or poor
AquaCalc	Section WetPerim:	B351	wetted perimeter of cross section, units feet
AquaCalc	Section Hyd Rad:	B352	hydraulic radius of cross section, units feet
AquaCalc	Section Manning:	B353	manning coefficient
AquaCalc	Section Chezy:	B354	chezy coefficient
AquaCalc	VERT	A357	measured vertical in cross section
AquaCalc	DIST	B357	distance along tag line, not necessairly distance from edge, units feet
AquaCalc	TDPTH	C357	user entered depth of water, units feet
AquaCalc	IDRFT	D357	ice draft is the thickness of surface ice, units feet
AquaCalc	EDPTH	E357	estimated depth? Couldn't find documentation in the manual
AquaCalc	OBS	F357	percentage of total depth where flow measurement was taken, units 2 tenths, 6 tenths, 8 tenths, E for edge
AquaCalc	TIME	G357	duration of measurement at each stream vertical, units seconds
AquaCalc	REVS	H357	number of meter revolutions in "TIME" length of time

item 1	item2	data range	meta data
AquaCalc	HA	I357	horizontal angle used to correct flow? No mention in manual
AquaCalc	HC:VF	J357	Couldn't find documentation in the manual
AquaCalc	METH	K357	user defined coefficient to correct a measurement at a vertical
AquaCalc	CLOCK	L357	AquaCalc clock time
AquaCalc	MVEL	M357	mean velocity at vertical, units feet per second
AquaCalc	OVEL	N357	observed velocity at the measurement depth, units ft/s
AquaCalc	VVEL	O357	calculated mean velocity for the vertical, units ft/s
AquaCalc	SSAREA	P357	subsection area for the vertical, units square feet
AquaCalc	SSQ	Q357	subsection flow
AquaCalc	SSPCT	R357	subsection percentage of total flow
AquaCalc	FLAGS	S357	warnings issued during measurement if errors arise

25. Appendix D: State of Utah Lab Sheets Monthly

Double-click picture to open PDF file for viewing and printing.



UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 1

STORET: 4951855 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: Sheep Ck bl Spring in Bryce Cyn NP

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

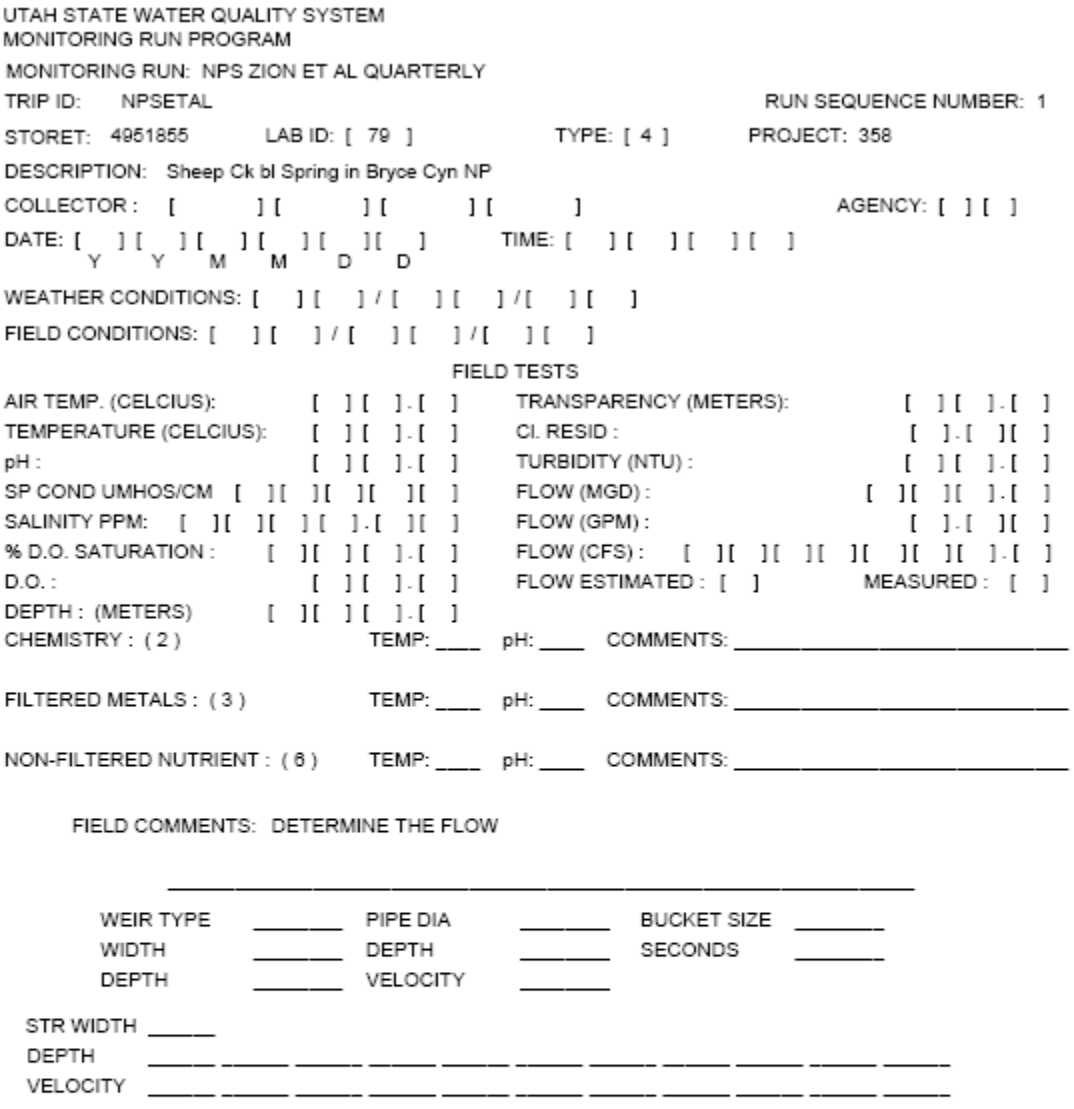
STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

26. Appendix E: State of Utah Lab Sheets Quarterly

Double-click picture to open PDF file for viewing and printing.



UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 1

STORET: 4951855 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: Sheep Ck bl Spring in Bryce Cyn NP

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ][ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ][ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ][ ][ ][ ]	FLOW (MGD) :	[ ][ ][ ] . [ ]
SALINITY PPM:	[ ][ ][ ] [ ] . [ ][ ]	FLOW (GPM) :	[ ] . [ ][ ]
% D.O. SATURATION :	[ ][ ] [ ] . [ ]	FLOW (CFS) :	[ ][ ][ ][ ][ ][ ][ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ][ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

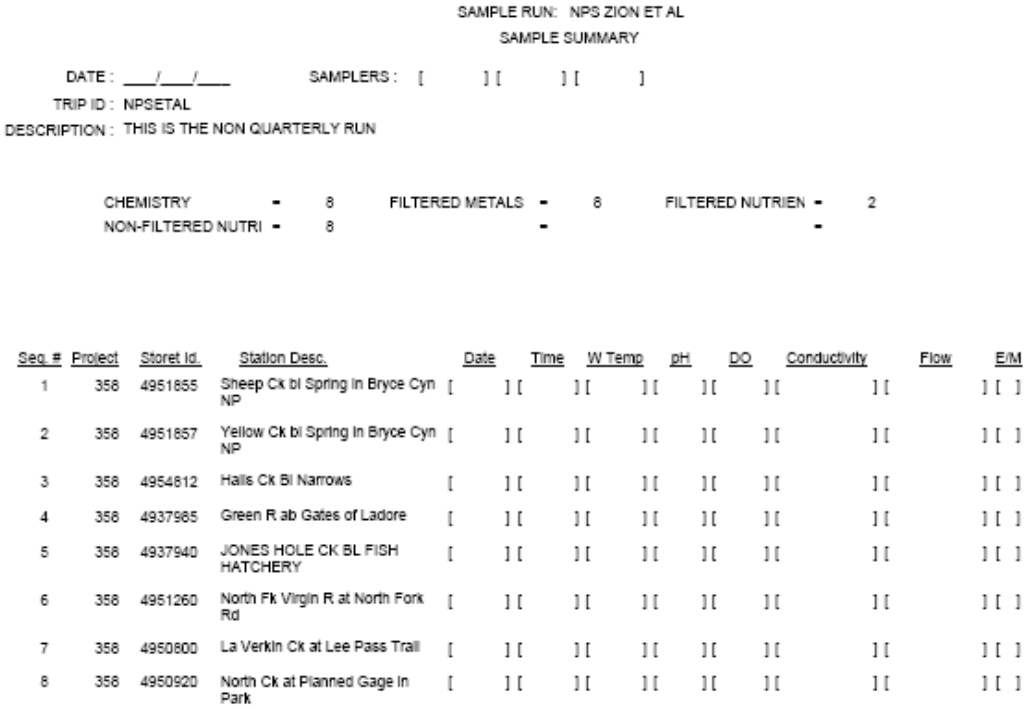
STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

27. Appendix F: State of Utah Run Sheets Monthly

Double-click picture to open PDF file for viewing and printing.



SAMPLE RUN: NPS ZION ET AL

SAMPLE SUMMARY

DATE : ___/___/___ SAMPLERS : [ ] [ ] [ ]

TRIP ID : NPSETAL

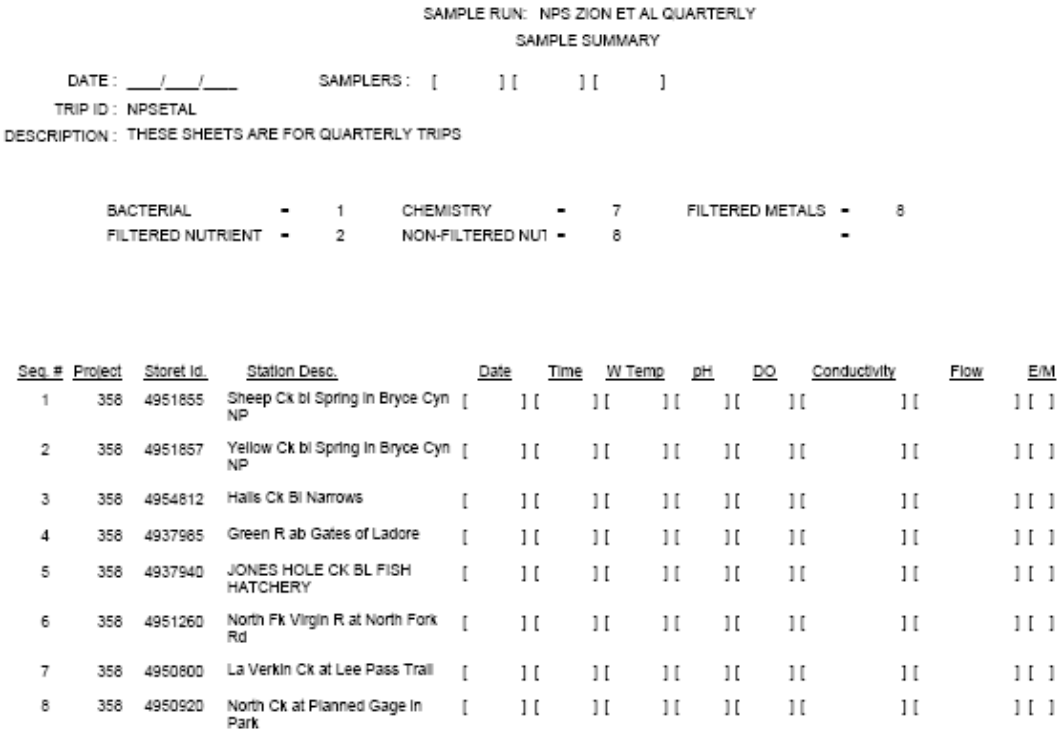
DESCRIPTION : THIS IS THE NON QUARTERLY RUN

CHEMISTRY	- 8	FILTERED METALS	- 8	FILTERED NUTRIEN	- 2
NON-FILTERED NUTRI	- 8		-		-

Seq. #	Project	Storet Id.	Station Desc.	Date	Time	W Temp	pH	DO	Conductivity	Flow	E/M
1	358	4951855	Sheep Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
2	358	4951857	Yellow Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
3	358	4954812	Halls Ck Bl Narrows	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
4	358	4937965	Green R ab Gates of Ladore	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
5	358	4937940	JONES HOLE CK BL FISH HATCHERY	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
6	358	4951260	North Fk Virgin R at North Fork Rd	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
7	358	4950800	La Verkin Ck at Lee Pass Trail	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
8	358	4950920	North Ck at Planned Gage in Park	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]

28. Appendix G: State of Utah Run Sheets Quarterly

Double-click picture to open PDF file for viewing and printing.



SAMPLE RUN: NPS ZION ET AL QUARTERLY

SAMPLE SUMMARY

DATE : ___/___/___ SAMPLERS : [ ] [ ] [ ]

TRIP ID : NPSETAL

DESCRIPTION : THESE SHEETS ARE FOR QUARTERLY TRIPS

BACTERIAL	- 1	CHEMISTRY	- 7	FILTERED METALS	- 8		
FILTERED NUTRIENT	- 2	NON-FILTERED NUT	- 6		-		

Seq. #	Project	Storet Id.	Station Desc.	Date	Time	W Temp	pH	DO	Conductivity	Flow	E/M
1	358	4951655	Sheep Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]
2	358	4951657	Yellow Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]
3	358	4954812	Halls Ck Bl Narrows	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]
4	358	4937985	Green R ab Gates of Ladore	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]
5	358	4937940	JONES HOLE CK BL FISH HATCHERY	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]
6	358	4951260	North Fk Virgin R at North Fork Rd	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]
7	358	4950800	La Verkin Ck at Lee Pass Trail	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]
8	358	4950920	North Ck at Planned Gage in Park	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ] [ ]

29. Appendix H: EPA Method 180.1 Turbidity

Double-click picture to open PDF file for viewing and printing.

METHOD #: 180.1	Approved for NPDES (Editorial Revision 1974, 1978)
TITLE:	Turbidity (Nephelometric)
ANALYTE:	Turbidity
INSTRUMENTATION:	Turbidimeter
STORET No.	00076

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters in the range of turbidity from 0 to 40 nephelometric turbidity units (NTU). Higher values may be obtained with dilution of the sample.

NOTE 1: NTU's are considered comparable to the previously reported Formazin Turbidity Units (FTU) and Jackson Turbidity Units (JTU).

2.0 Summary of Method

2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTU's, are made in a nephelometer designed according to specifications outlined in Apparatus. A standard suspension of Formazin, prepared under closely defined conditions, is used to calibrate the instrument.

2.1.1 Formazin polymer is used as the turbidity reference suspension for water because it is more reproducible than other types of standards previously used for turbidity standards.

2.1.2 A commercially available polymer standard is also approved for use for the National Interim Primary Drinking Water Regulations. This standard is identified as AMCO-AEPA-1 available from Amco Standard International, Inc.

3.0 Sample Handling and Preservation

3.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.

4.0 Interferences

4.1 The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles will affect the results in a positive manner.

4.2 The presence of true color, that is the color of water which is due to

30. Appendix I: Hach 2100P Turbidity User Manual

Double-click picture to open PDF file for viewing and printing.



CAT. NO. 46500-88

PORTABLE TURBIDIMETER

Model 2100P

Instrument and Procedure Manual

© Hach Company, 1991-2004.
All rights reserved. Printed in the U.S.A.

te/dk 3/04 8ed

31. Appendix J: Decontamination Procedures

Appendix __: Decontamination Considerations During and After Field Activities

National Park Service

Northern Colorado Plateau Network Inventory and Monitoring Program

Water Quality

07/02/2008

Excerpts from the U.S. Fish and Wildlife Service's national "Help Stop Aquatic Hitchhikers" program, and the USU Buglab's "New Zealand Mudsnail" website, as well as recommendations from Melissa Trammel, NPS fisheries biologist, are presented below as a reference when conducting water quality monitoring. A brief background of known local threats is provided along with an overview of useful decontamination procedures. The intent of adding this section is to help reduce or prevent the spread of aquatic diseases and invasive species while conducting routine field procedures.

Specific threats by invasive plants, animals, or diseases are of course dependant on location, however section 2 is meant as a blanket decontamination procedure. Additional considerations may be necessary as new threats are realized.

1. Contamination Types

1.1 Mollusks: New Zealand Mud Snail

1.1.2 Background

Identification- New Zealand Mudsnails are small (3 to 6 mm or 1/8 inch) snails that have brown or black cone-shaped shells with five whorls (USFWS, 2008).

Where dense populations have developed they often look like BBs scattered on the river's bottom. Taxonomically, New Zealand mud snails are in the snail family Hydrobiidae. Hydrobiids can be distinguished from other aquatic snail families by having dextral (opening to the right with the spire pointing away from you) shells with an operculum (a hard calcareous flat that can seal the opening of the shell). New Zealand mud snails are small (~ 1-2 mm) and generally dark colored. A key characteristic for identifying them is that they carry immature snails in their shells. You will need a microscope to see this.

Habitat- New Zealand mud snails appear to prefer flowing water habitats with stable flows. Springs, spring creeks, and river sections downstream from dams are all places that they thrive in. They are most typically found on larger cobble substrates or on pieces of wood. Routine aquatic invertebrate monitoring using a kick net or Surber net may not detect them as they stick well to rocks and they do not tend to float downstream very far. Pick up and evaluate individual softball sized rocks and larger sticks.

- If you think you have found some, please store them in small container with preservative (either isopropyl or ethyl alcohol) and mail them to Mark Vinson, BLM Western Bioassessment Center, Department of Aquatic, Watershed, and Earth Resources Utah State University, Logan, Utah 84322-5210 (Buglab, 2007).

1.1.3 Environmental Impact

Densities & Competition with Native Species – This snail has the ability to reproduce quickly and mass in high densities. When snails become as dense as one-half million per meter square, this has been a cause for concern in western streams. Because the West is known for its great trout fishing, there is concern that the mudsnails will impact the food chain of native trout and alter the physical characteristics of the streams themselves. Research is needed to determine the impacts of large populations of mudsnails on the native fauna, such as aquatic insects and native snails, and on any changes in the physical environment.

Ability to Survive in Variable Conditions - The rapid spread of the mudsnail throughout the Yellowstone watershed may have been assisted by human transport. Mudsnails are able to withstand dessication, a variety of temperature regimes, and are small enough that many types of water users (anglers, swimmers, picnicers, pets) could inadvertently be the mechanism for interbasin transfer of this nuisance species. Because the mudsnails reproduce asexually, it only takes one individual to become introduced into new water to make an impact. Also, the mudsnails can readily attach themselves to boots and waders (USFWS, 2008).

1.2 Whirling Disease

1.2.1 Background

Identification- While the transmission stage of Whirling Disease is a microscopic spore so the only way to identify the disease is from infected fish. Young fish may be exhibiting the “Whirling” behavior that gave the disease its name, in which the fish swims in circles. The tail of an infected fish often turns black. Because the parasite attacks the cartilage tissue of a fish's head and spine if the fish survives to be 1.5 months the spinal development of the fish is often altered giving the fish a crocked or spiral shape and/or deformed head (UTDWR, 2008).

Lifecycle- The physical appearance of Whirling Disease is not as important as understanding what it is and how to prevent its spread. In layman's terms, whirling disease is the descriptive side effect produced by a complex relationship involving a non-native parasite and a common aquatic worm that exclusively impacts coldwater sportfish, specifically trout and salmon. The parasite becomes engulfed by a tubifex worm, which acts as an intermediate host for the parasite. Eventually, this relationship produces a new free-floating life phase of the parasite that attaches itself to trout and salmon. After coming into contact with the host fish, the parasite penetrates the head and spinal cartilage of fingerling trout where it multiplies very rapidly, putting pressure on the organ of equilibrium. This causes the fish to swim erratically (whirl) and have difficulty feeding and avoiding predators. It is this whirling effect that has significantly impacted our coldwater fishery resources.

1.2.2 Environmental Impact

Native to Eurasia, whirling disease is having devastating impacts on coldwater fisheries in North America. All species of trout and salmon may be susceptible to whirling disease. Other members of the trout and salmon family, such as mountain whitefish are also at risk. Rainbow trout and cutthroat trout appear to be more susceptible than other trout species. Brown trout become infected with the parasite, but they appear to have immunity to the infection and have not been as greatly impacted as rainbow trout. Scientific studies have demonstrated that grayling and bull trout are very resistant to infection. However, regardless of species, when each infected fish dies, many thousands to millions of the parasite spores are released to the water. These parasitic spores are virtually indestructible -- they can withstand freezing and desiccation, and can survive in a stream for 20 to 30 years. The parasite is sure to continue to spread to drainages now clean, since it is so easily and unknowingly transported by animals, birds and humans (USFWS, 2008).

1.3 Didymosphenia, a.k.a Didymo or Rock Snot

1.3.1 Background

It is found in streams and rivers in much of North America. Didymo increasingly poses a threat to aquatic ecosystems because it forms extensive mats on stream beds. Didymo attaches to the streambed by a stalk. These stalks have a rough texture similar to wet wool and mimic strands of toilet paper, as opposed to other algal species which feel "slimy". *Didymosphenia geminata* is expanding its geographic

range. Historically, this diatom occurred mostly in northern latitudes in low nutrient waters; now it occurs in more nutrient-rich water and at lower latitudes. Central Colorado as well as Northern Utah have both reported spread of this species ("Didymosphenia," 2008).

2. Decontamination Procedures

2.1 General/Recreational Procedures

General Prevention Procedures for Stopping Aquatic Hitchhikers: *A must read for all recreational users*

Follow a general set of procedures every time you come in contact with any body of water. By doing so, you can protect your waters from harmful aquatic hitchhikers. Because you never know where a nuisance species has been introduced, but has yet to be discovered.

There are hundreds of different harmful species ranging from plants, fish, amphibians, crustaceans, mollusks, diseases or pathogens. Some organisms are so small, you may not even realize they are hitching a ride with you. So, it is important to follow this general procedure every time you leave any body of water.

Remove all visible mud, plants, fish/animals.
Before leaving any body of water, it is important to examine all your equipment, boats, trailers, clothing, boots, buckets etc and:

- Remove any visible plants, fish or animals.
- Remove mud and dirt since it too may contain a hitchhiker.*
- Remove even plant fragments as they may contain a hitchhiker.*
- Do not transport any potential hitchhiker, even back to your home. Remove and leave them at the site you visited.

*The larvae (immature form) of an animal can be so tiny that you cannot see it. However, it can live in mud, dirt, sand, and on plant fragments.

Eliminate water from all equipment before transporting anywhere.
Much of the recreational equipment used in water contains many spots where water can collect and potentially harbor these aquatic hitchhikers. Thus, make sure that you:

- Eliminate all water from every conceivable item before you leave the area you are visiting.
- Remove water from motors, jet drives, live wells, boat hulls, scuba tanks and regulators, boots, waders, bait buckets, seaplane floats, swimming floats.
- Once water is eliminated, follow the cleaning instructions listed below.

Clean and dry anything that came in contact with the water.
(boats, trailers, equipment, dogs, boots, clothing, etc.).
Basic procedures

- Use hot (< 40° C or 104° F) or salt water to clean your equipment.
- The following recipes are recommended for cleaning hard-to-treat equipment that cannot be exposed to hot water:
  - Dipping equipment into 100% vinegar for 20 minutes will kill harmful aquatic hitchhiker species.
  - A 1 % table salt solution for 24 hours can replace the vinegar dip. This table provides correct mixtures for the 1 % salt solution in

include: water:

Gallons of Water	Cups of Salt
5	2/3
10	1 ¼
25	3
50	6 1/4
100	12 2/3

- If hot water is not available, **spray** equipment such as boats, motors, trailers, anchors, decoys, floats, nets, **with high-pressure water.**
- **DRY Equipment.** If possible, allow for 5 days of drying time before entering new waters.

(USFWS, 2008)

2.2 Formal USFS Decontamination Procedures

Alternatively complying with some form of the following tailored version of the US Forest Service "2008 Fire Ops Guidance for Aquatic Invasive Species" may be used (Trammell, 2008).

Prevention, where possible

- Avoid dumping water directly from one stream or lake into another.
- Avoid obtaining water from multiple sources during a single operational period unless drafting/dipping equipment is sanitized between sources.
- Minimize driving equipment through water bodies.
- Obtain maps of invasive presence in or around your sample area, be aware and respond accordingly at each site. You can never be certain that invasives are NOT present, but at least you will know ahead of time where they ARE present.

Sanitation

The following outlines the use of Quat128 and Sparquat256, preferred alternatives to bleach which may harm stainless steal water quality equipment as well as personal gear. Refer to the US Forest Service "**2008** Fire Ops Guidance for Aquatic Invasive Species" (p. 4-6,), for complete tables of aquatic invasives of concern and other methods of control.

- Set up a portable disinfection tank using a 5% cleaning solution of quaternary ammonium compound – common cleaning agents used in homes, swimming pools, and hospitals that are safe for gear and equipment when used at the recommended concentration. Two brands are readily available from GSA or local suppliers: *Quat128*® (by Waxie) or *Sparquat 256*® (by Spartan). Costs and effectiveness are comparable; use either.

  Recipe for 5% cleaning solution using either *Quat128*® ***or*** *Sparquat 256*®

Volume of tap water	**Volume of *Quat128*®**	**Volume of *Sparquat 256*®**
100 mL water	4.63 mL	3.00 mL
1 gallon water	6.35 liquid oz.	4.12 liquid oz.
1 gallon water	12.7 tbsp	8.2 tbsp
1 gallon water	0.79 cups	0.51 cups
100 gallons water	4.96 gallons	3.22 gallons
1000 gallons water	49.6 gallons	32.2 gallons

- Where feasible dip gear or equipment into the cleaning solution. Alternatively, put the 5% cleaning solution in backpack spray pumps to clean portable equipment. The solution must be in contact with the surface being sanitized for at least 10 minutes and then rinsed with water.
- Under the direction of the Resource Advisor, test cleaning solution at least daily according to the directions below. The cleaning solution can be used repeatedly for up to a week unless heavily muddied or diluted. If the concentration is too weak, dispose of the used solution properly and make a new solution.

Safety

- Use protective, unlined rubber gloves and splash goggles or face shield when handling the cleaning solution and take extra precautions when handling undiluted chemicals. Have eye wash and clean water available on-site to treat accidental exposure.
- Consult the product label and Material Safety Data Sheet for additional information.

Testing Solution

- To determine if the solution is below the 5% strength use "Quat Chek 1000" Test Papers (purchase these from the supplier of the cleaning compound). The used cleaning solution needs to be diluted to about 600 ppm of ammonium compounds before it can be tested with these papers.
  - Take **one** cup of used *Sparquat 256*® cleaning solution, pour into a bucket. Add **5** cups of water. Mix. OR
  - Take **one** cup of used *Quat128*® cleaning solution, pour into a bucket. Add **4** cups of water. Mix.
- Test the diluted solution with "Quat Chek" Test Paper. Match up the color of the paper with the ppm's on the color chart. For optimal disinfection, the diluted solution should

have a concentration between 600 and 800 ppm. If it is too dilute, dispose of properly and make a new cleaning solution.

Disposal

- Do not dump cleaning solution into any stream or lake, or on areas where it can migrate into any stormdrain, waterbody, or sensitive habitat. It may be possible to dispose of used cleaning solution over open land. Consult Resource Advisor before using this method.
- Depending on volume, used cleaning solution may be disposed of by trucking off-site for disposal in a wastewater treatment facility.
- Used cleaning solution may or may not be suitable for disposal in on-site septic systems. Consult the local agency's Utilities Supervisor or Facilities Manager prior to disposal.

Storage

Sparquat 256® and *Quat128*® can be stored up to two years in an unopened container without losing its effectiveness. Both should be stored in a cool, dry place, out of direct sunlight. Temperatures can range from 32° to 110° F.

Purchase

Both products are available from GSA (https://www.gsaadvantage.gov) and are commonly available through local janitorial and swimming pool chemical suppliers.

- *Quat 128*® by Waxie's Enterprises Inc.; GSA (NSN No. 170304) =$36/case (4 gal); Additional info can be found at http://www.waxie.com

- *Sparquat 256*® by Spartan Chemical Company; GSA (NSN No. 1025-04) = $54/case (4 gal); Additional info can be found at http://www.spartanchemical.com
- Remember to buy "Quat Chek 1000" test papers when you purchase the chemicals ("Operational Guidelines", 2008).

Citations

BLM / USU National Aquatic Monitoring Center, 2007. New Zealand Mud Snails. Western Aquatic Invertebrate Nuisance Species. June 2007. BLM / USU National Aquatic Monitoring Center Department of Aquatic, Watershed, & Earth Resources (AWER), Utah State University. 2 July 2008 <http://www1.usu.edu/buglab/projects/nzms.htm>.

EPA, 2008. Didymosphenia Geminata: a Nuisance Freshwater Alga. Region 8 - Mountains & Plains. 3 July 2008. U.S. Environmental Protection Agency. 2 July 2008 <http://www.epa.gov/region8/////////water/didymosphenia/>.

U.S. Fish and Wildlife Service,2008. Harmful Aquatic Hitchhikers. Protect Your Waters. U.S. Fish and Wildlife Service and the U.S. Coast Guard. 2 July 2008 <http://www.protectyourwaters.net/hitchhikers/>.

National Park Service 2008, Operational Guidelines for Aquatic Invasive Species Prevention and Equipment Cleaning. Wildland Fires. Salt Lake City, UT,, 2008. 1-2.

Trammell, Melissa. Telephone interview. 2 July 2008.

Utah Division of Wildlife Resources, 2008. Whirling Disease in Utah. Fisheries Experimentation Station. 31 Mar. 2008. 6 Aug. 2008 <http://http://wildlife.utah.gov/fes/whirling_disease.html>.

Reference Material

Decontamination Procedure Power Point Presentation located in:
X:\Active_Monitoring_Projects\Water_Quality\presentations\Decontamination_Aug08

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #9

Field Sample Collection and Processing for Water Chemistry, Nutrients and TSS, Colorado Units

Version 1.0 (4/11/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

The purpose of this SOP is to outline procedures for the collection and handling of water samples to ensure accurate field data collection and that representative samples of surface water bodies arrive uncontaminated at the laboratory in a timely manner.

Table of Contents

1. Introduction....3
2. Citations....3
3. Appendix A: National Field Manual for the Collection of Water-Quality Data....4
3.1. Preparations for Water Sampling (1/2005)....4
3.2. Selection of Equipment for Water Sampling (Version 2.0, 3/2003)....5
3.3. Cleaning of Equipment for Water Sampling (Version 2.0, 4/2004)....6
3.4. Collection of Water Samples (9/99)....7
3.5. Processing of Water Samples, Version 2.1 (4/04)....8
3.6. Field Measurements....9
3.6.1. General Information and Guidelines (Version 1.2, 8/2005)....10
3.6.2. Temperature (Version 2, 3/2006)....11
3.6.3. Dissolved Oxygen (6/2006)....12
3.6.4. Specific Electrical Conductance (8/2005)....13
3.6.5. pH (Version 1.3, 1/2006)....14
3.6.6. Turbidity (9/2005)....15
3.6.7. Fecal Indicator Bacteria....16
3.6.8. Safety in Field Activities (10/97)....17
4. Appendix B: Errata sheets for all chapters (downloaded 11/17/06)....18
4.1. Preparations for Water Sampling....18
4.2. Selection of Equipment for Water Sampling....19
4.3. Cleaning of Equipment for Water Sampling....20
4.4. Collection of Water Samples....23
4.5. Processing of Water Samples....25
4.6. Field Measurements....30
4.7. Biological Indicators....34
4.8. Bottom-Material Samples....34
4.9. Safety in Field Activities....35

1. Introduction

Sample collection for CO park units follows USGS protocol (USGS, variously dated). Methods used by the USGS are published as chapters in the National Field Manual by their most recent update as methods evolve over time. Pertinent chapters are reproduced in this SOP without alteration as Appendices. Many changes to the NFM are apparent and ongoing so this should be viewed as a dynamic document and regularly reviewed for new updates. Relevant National Field Manual Chapters and errata sheets were downloaded 11/17/06. To view individual chapters in Adobe pdf format double click on title page in each appendix.

The online National Field Manual document (NFM) will be checked for revisions and Network protocols (or appropriate sections) will be updated annually.

http://water.usgs.gov/owq/FieldManual/

Due to partnering with various organizations in (e.g. other states) in network, other water quality/aquatic monitoring protocols may be followed and are provided elsewhere

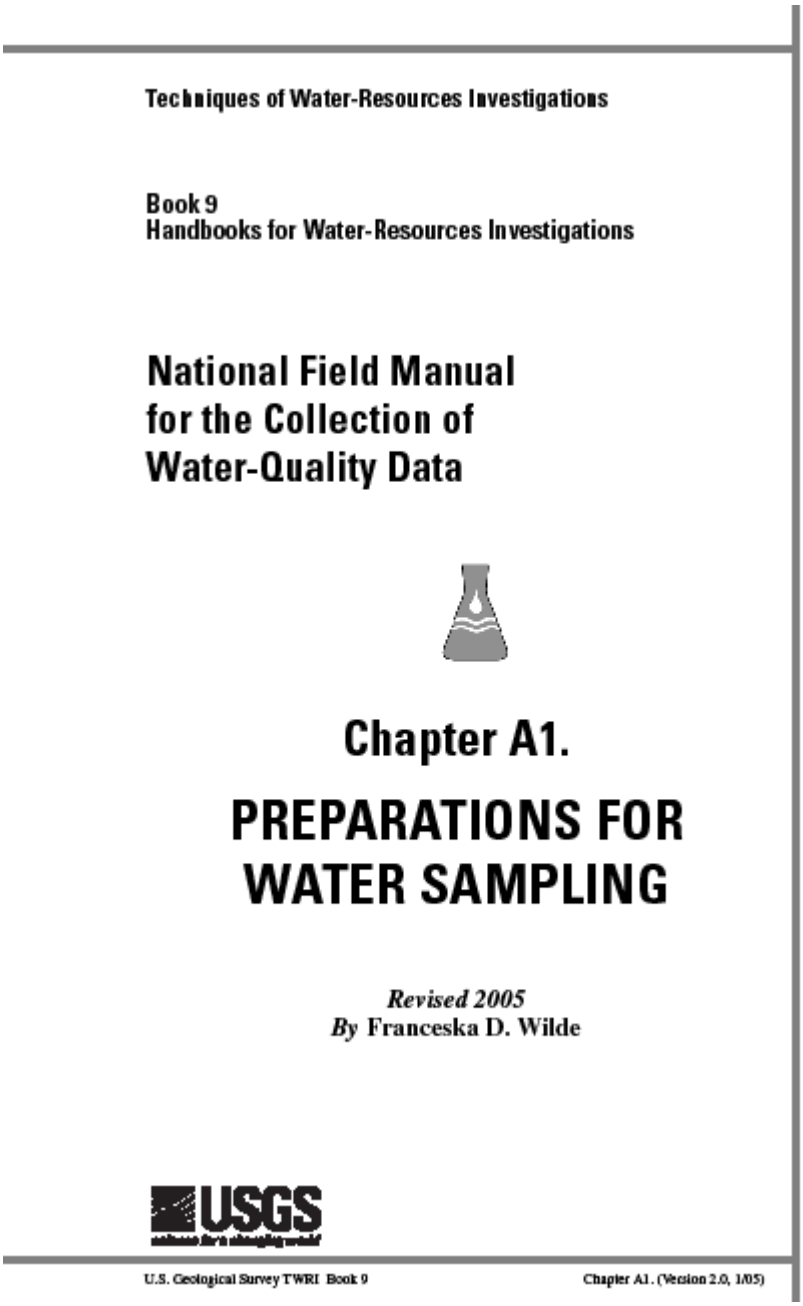
2. Citations

U.S. Geological Survey, variously dated, National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chaps. A1-A9, available online at http://pubs.water.usgs.gov/twri9A.

3. Appendix A: National Field Manual for the Collection of Water-Quality Data

3.1. Preparations for Water Sampling (1/2005)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual for the Collection of Water-Quality Data

Chapter A1.

PREPARATIONS FOR WATER SAMPLING

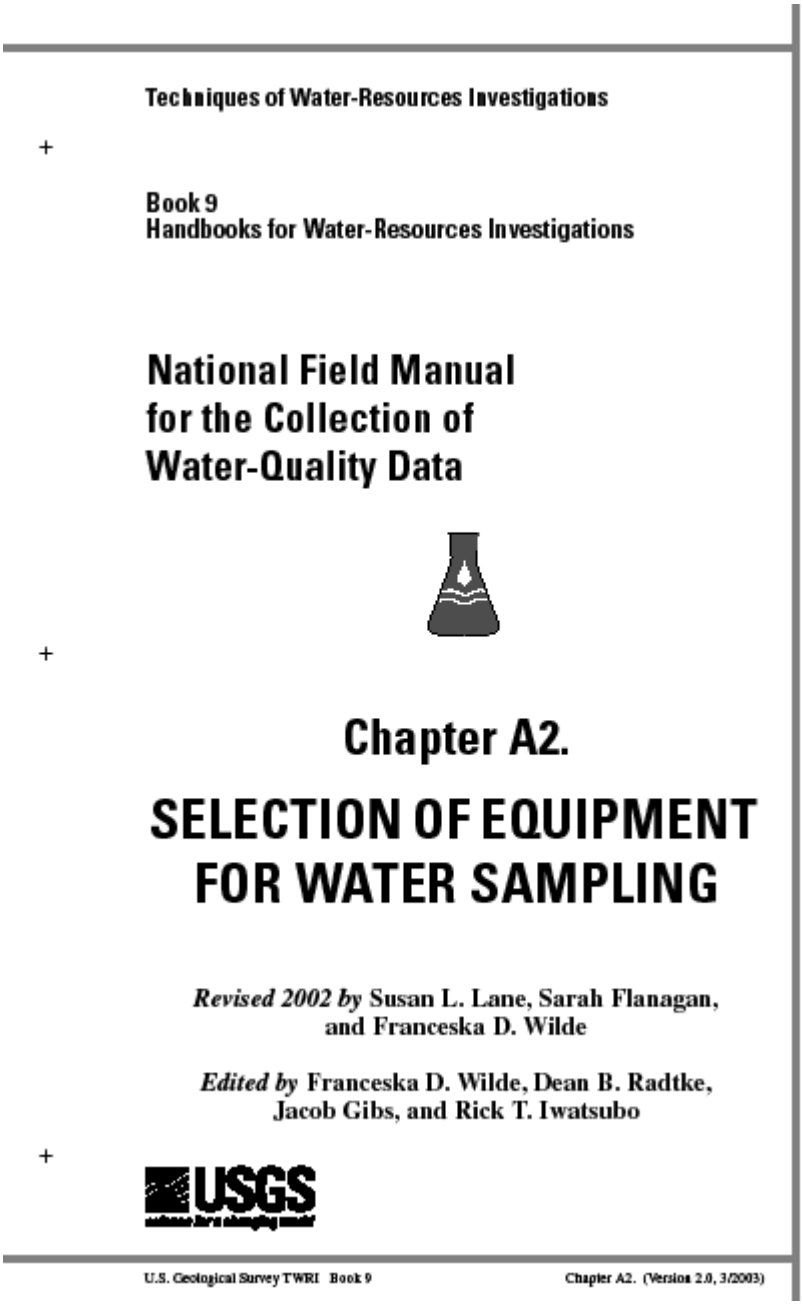
Revised 2005
By Franceska D. Wilde

USGS

U.S. Geological Survey TWRI Book 9 Chapter A1. (Version 2.0, 1/05)

3.2. Selection of Equipment for Water Sampling (Version 2.0, 3/2003)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual for the Collection of Water-Quality Data

Chapter A2.

SELECTION OF EQUIPMENT FOR WATER SAMPLING

Revised 2002 by Susan L. Lane, Sarah Flanagan, and Franceska D. Wilde

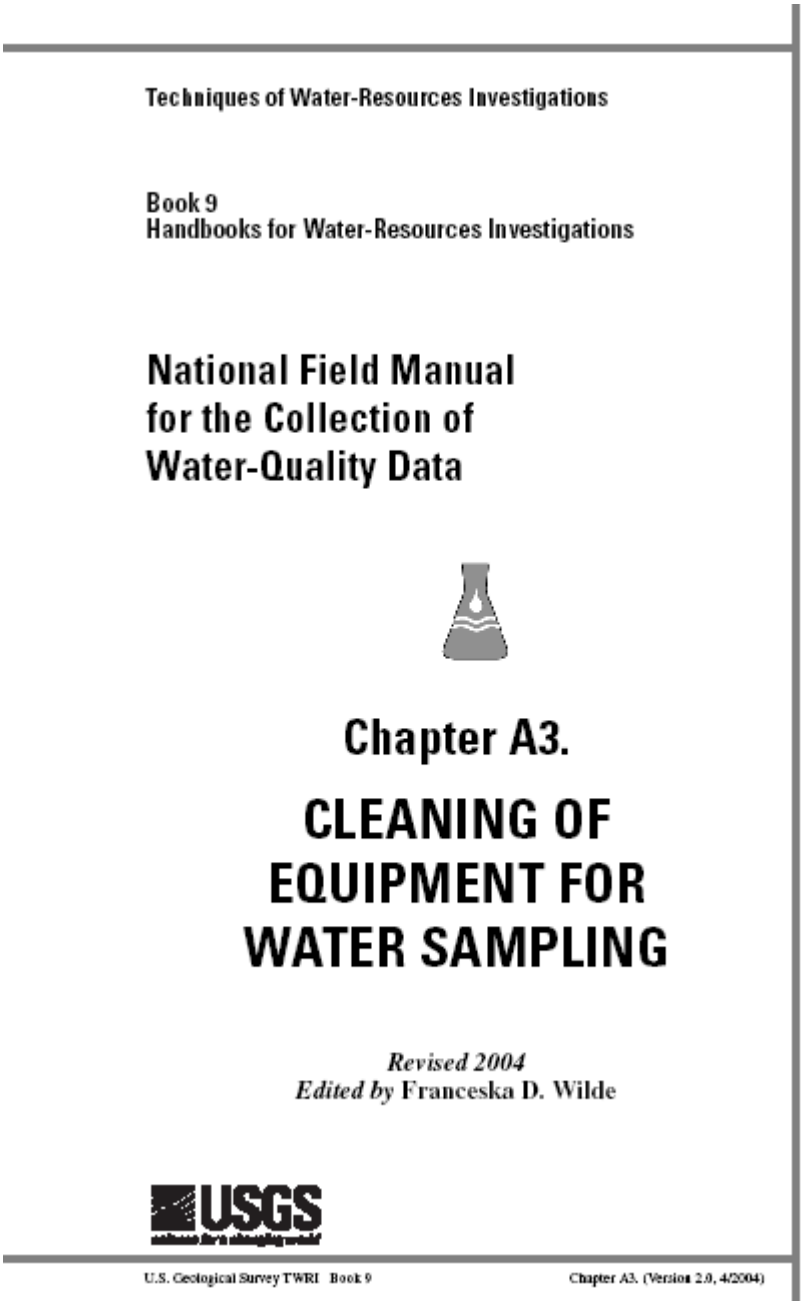
Edited by Franceska D. Wilde, Dean B. Radtke, Jacob Gibs, and Rick T. Iwatsubo

USGS

U.S. Geological Survey TWRI Book 9 Chapter A2. (Version 2.0, 3/2003)

3.3. Cleaning of Equipment for Water Sampling (Version 2.0, 4/2004)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual for the Collection of Water-Quality Data

Chapter A3.

CLEANING OF EQUIPMENT FOR WATER SAMPLING

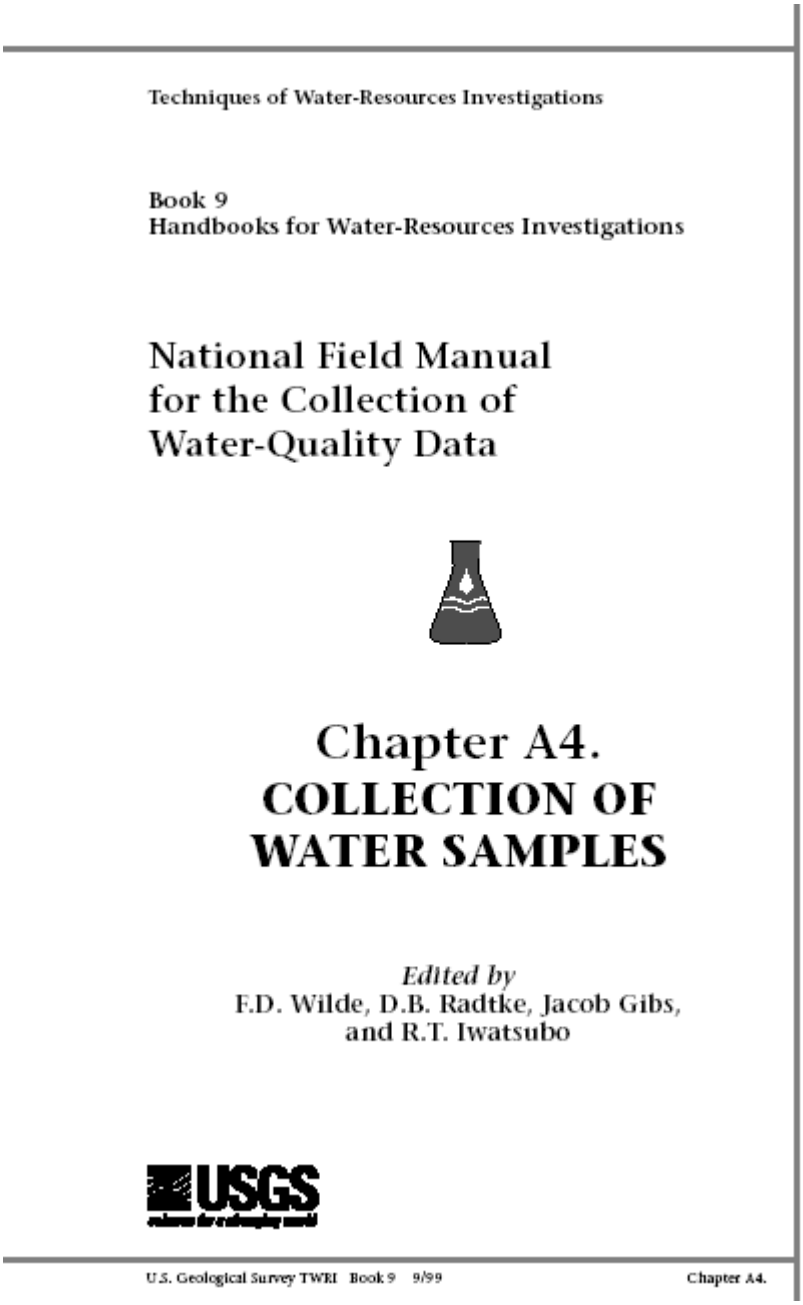
Revised 2004
Edited by Franceska D. Wilde

USGS

U.S. Geological Survey TWRI Book 9 Chapter A3. (Version 2.0, 4/2004)

3.4. Collection of Water Samples (9/99)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual for the Collection of Water-Quality Data

Chapter A4.
COLLECTION OF WATER SAMPLES

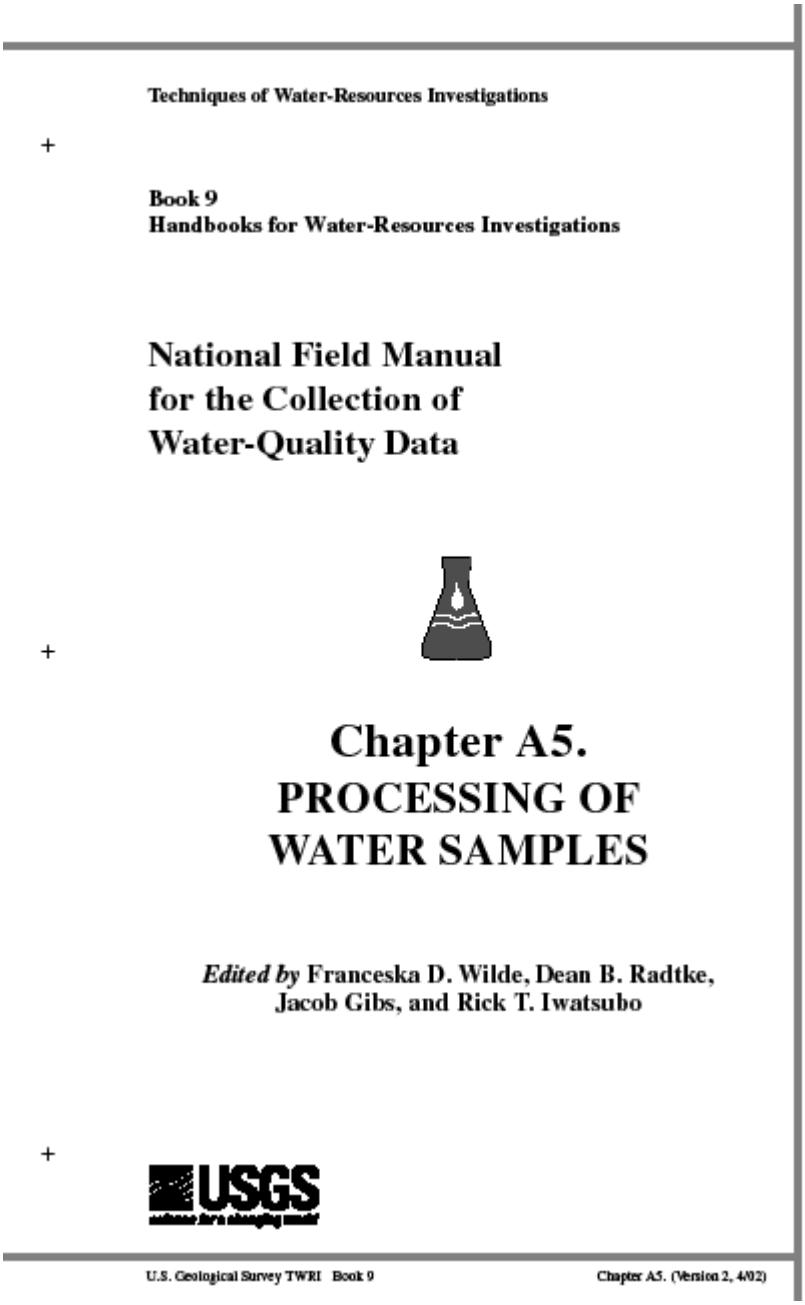
Edited by
F.D. Wilde, D.B. Radtke, Jacob Gibs, and R.T. Iwatsubo

USGS

U.S. Geological Survey TWRI Book 9 9/99 Chapter A4.

3.5. Processing of Water Samples, Version 2.1 (4/04)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual
for the Collection of
Water-Quality Data

Chapter A5.
PROCESSING OF
WATER SAMPLES

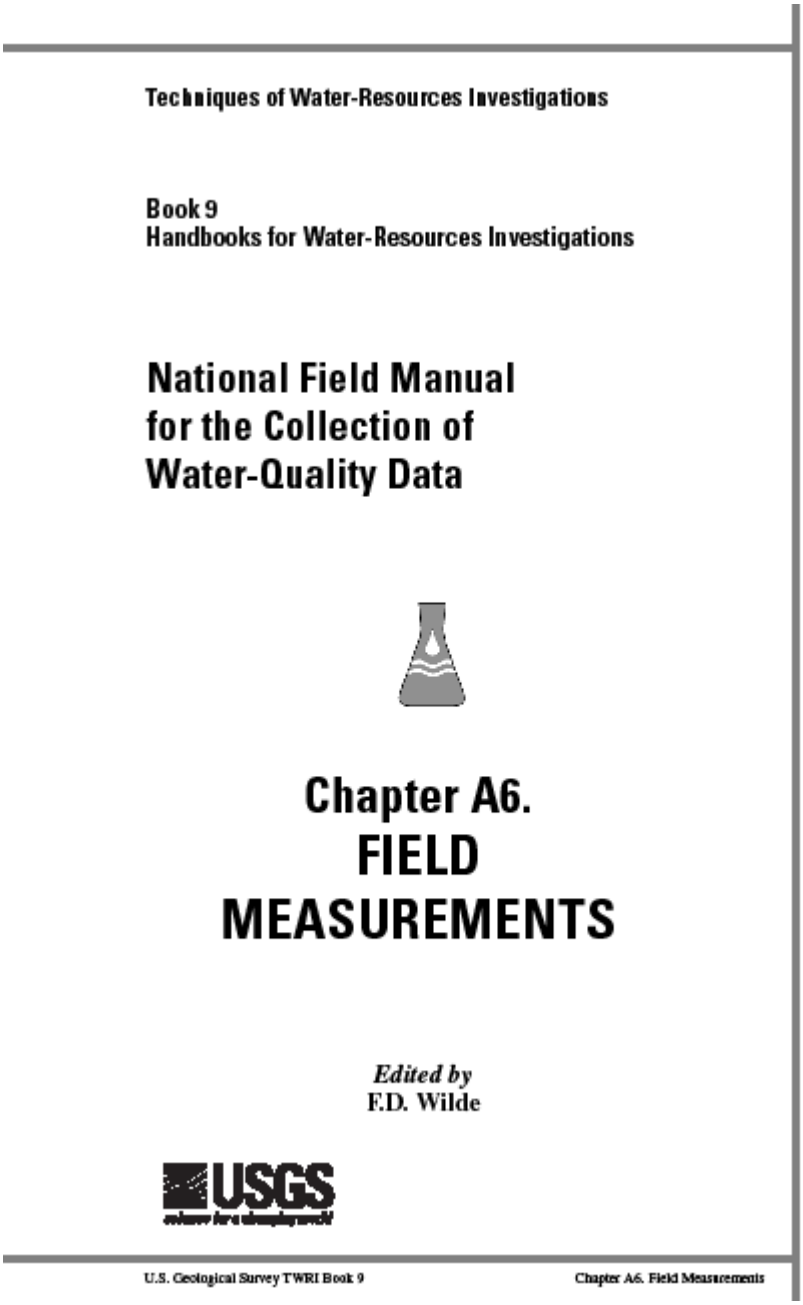
Edited by Franceska D. Wilde, Dean B. Radtke,
Jacob Gibs, and Rick T. Iwatsubo

USGS

U.S. Geological Survey TWRI Book 9 Chapter A5. (Version 2, 4/02)

3.6. Field Measurements

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual for the Collection of Water-Quality Data

Chapter A6.
FIELD MEASUREMENTS

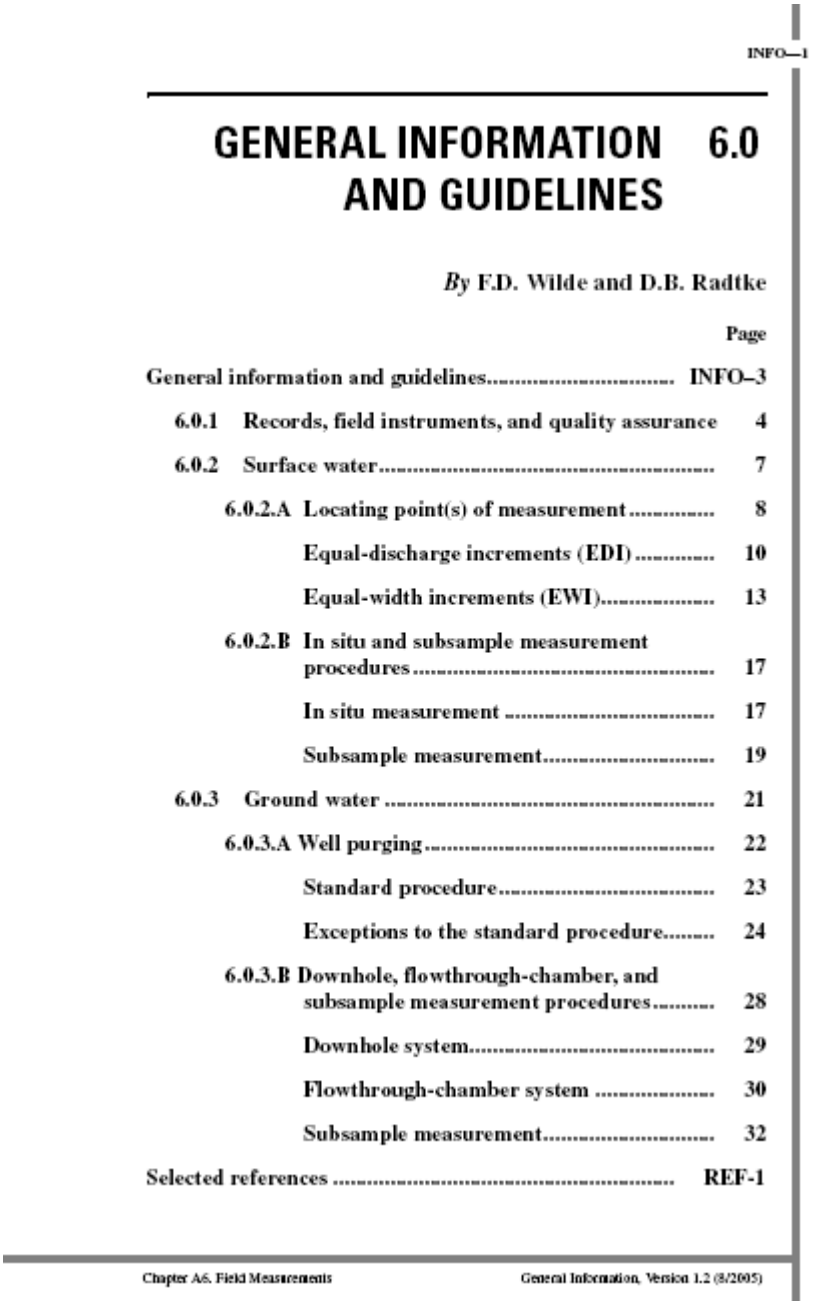
Edited by
F.D. Wilde

USGS

U.S. Geological Survey TWRI Book 9 Chapter A6. Field Measurements

3.6.1. General Information and Guidelines (Version 1.2, 8/2005)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



GENERAL INFORMATION AND GUIDELINES 6.0

By F.D. Wilde and D.B. Radtke

Page

General information and guidelines..... INFO–3

6.0.1 Records, field instruments, and quality assurance 4

6.0.2 Surface water..... 7

6.0.2.A Locating point(s) of measurement..... 8

Equal-discharge increments (EDI)..... 10

Equal-width increments (EWI)..... 13

6.0.2.B In situ and subsample measurement procedures..... 17

In situ measurement..... 17

Subsample measurement..... 19

6.0.3 Ground water..... 21

6.0.3.A Well purging..... 22

Standard procedure..... 23

Exceptions to the standard procedure..... 24

6.0.3.B Downhole, flowthrough-chamber, and subsample measurement procedures..... 28

Downhole system..... 29

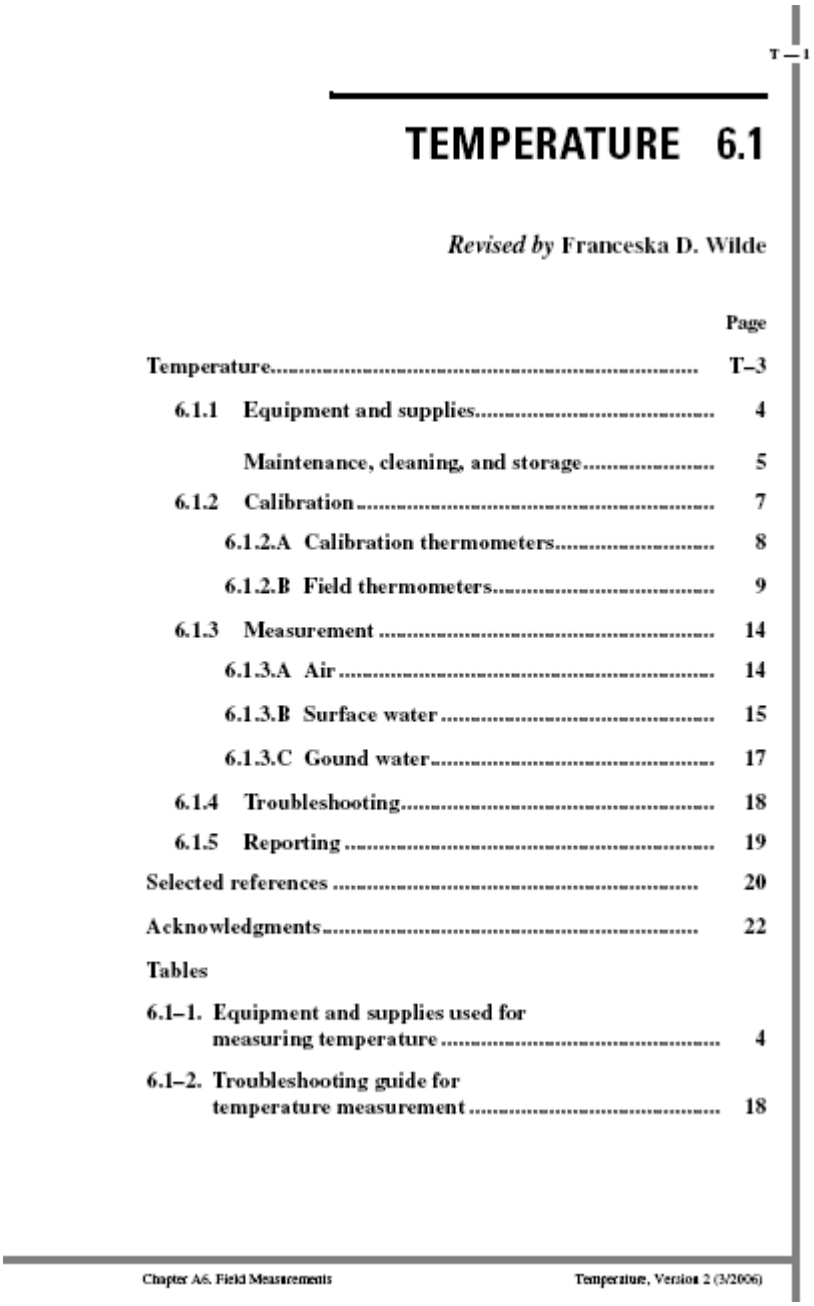
Flowthrough-chamber system..... 30

Subsample measurement..... 32

Selected references..... REF-1

3.6.2. Temperature (Version 2, 3/2006)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



TEMPERATURE 6.1

Revised by Franceska D. Wilde

Page

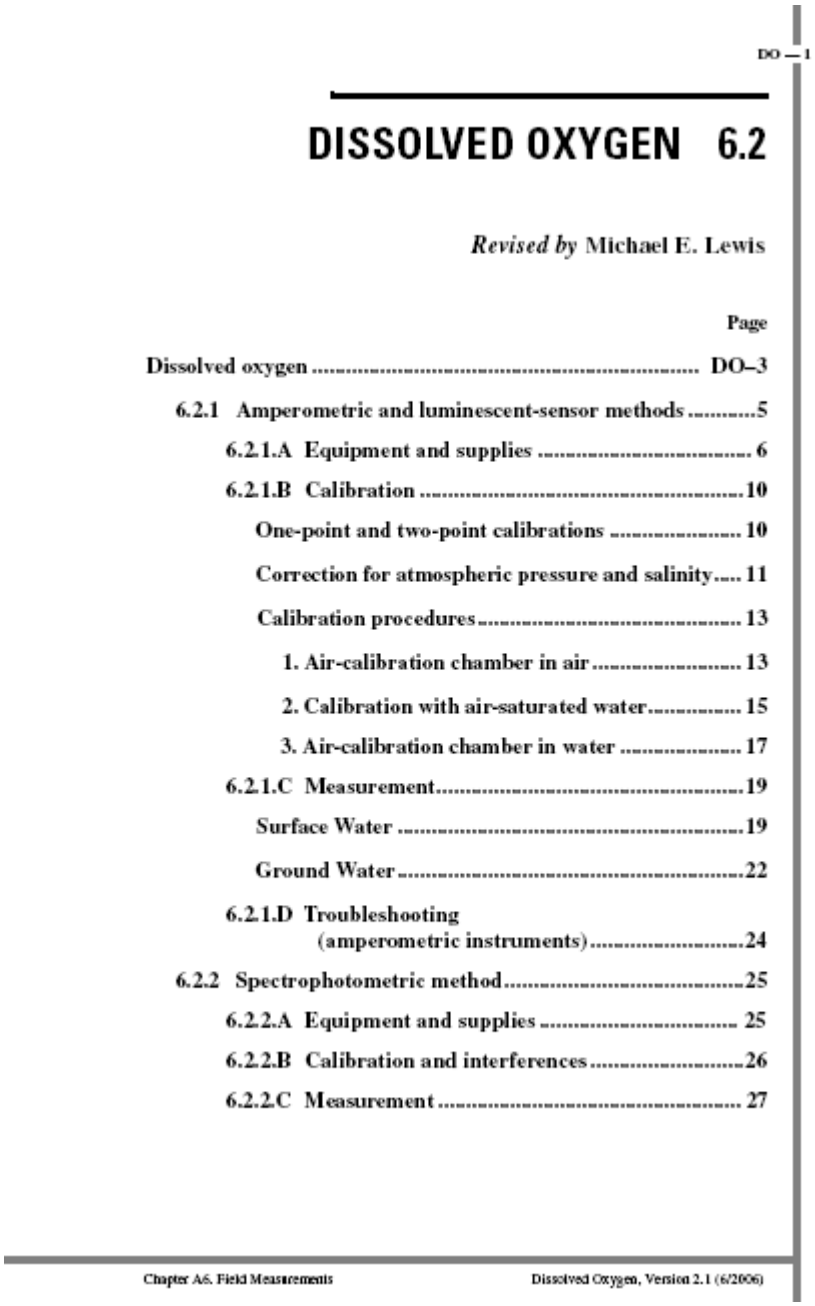
Temperature T–3
6.1.1 Equipment and supplies 4
Maintenance, cleaning, and storage 5
6.1.2 Calibration 7
6.1.2.A Calibration thermometers 8
6.1.2.B Field thermometers 9
6.1.3 Measurement 14
6.1.3.A Air 14
6.1.3.B Surface water 15
6.1.3.C Gound water 17
6.1.4 Troubleshooting 18
6.1.5 Reporting 19
Selected references 20
Acknowledgments 22

Tables

6.1–1. Equipment and supplies used for measuring temperature 4
6.1–2. Troubleshooting guide for temperature measurement 18

3.6.3. Dissolved Oxygen (6/2006)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



DISSOLVED OXYGEN 6.2

Revised by Michael E. Lewis

Page

Dissolved oxygen DO–3

6.2.1 Amperometric and luminescent-sensor methods 5

6.2.1.A Equipment and supplies 6

6.2.1.B Calibration 10

One-point and two-point calibrations 10

Correction for atmospheric pressure and salinity 11

Calibration procedures 13

1. Air-calibration chamber in air 13

2. Calibration with air-saturated water 15

3. Air-calibration chamber in water 17

6.2.1.C Measurement 19

Surface Water 19

Ground Water 22

6.2.1.D Troubleshooting (amperometric instruments) 24

6.2.2 Spectrophotometric method 25

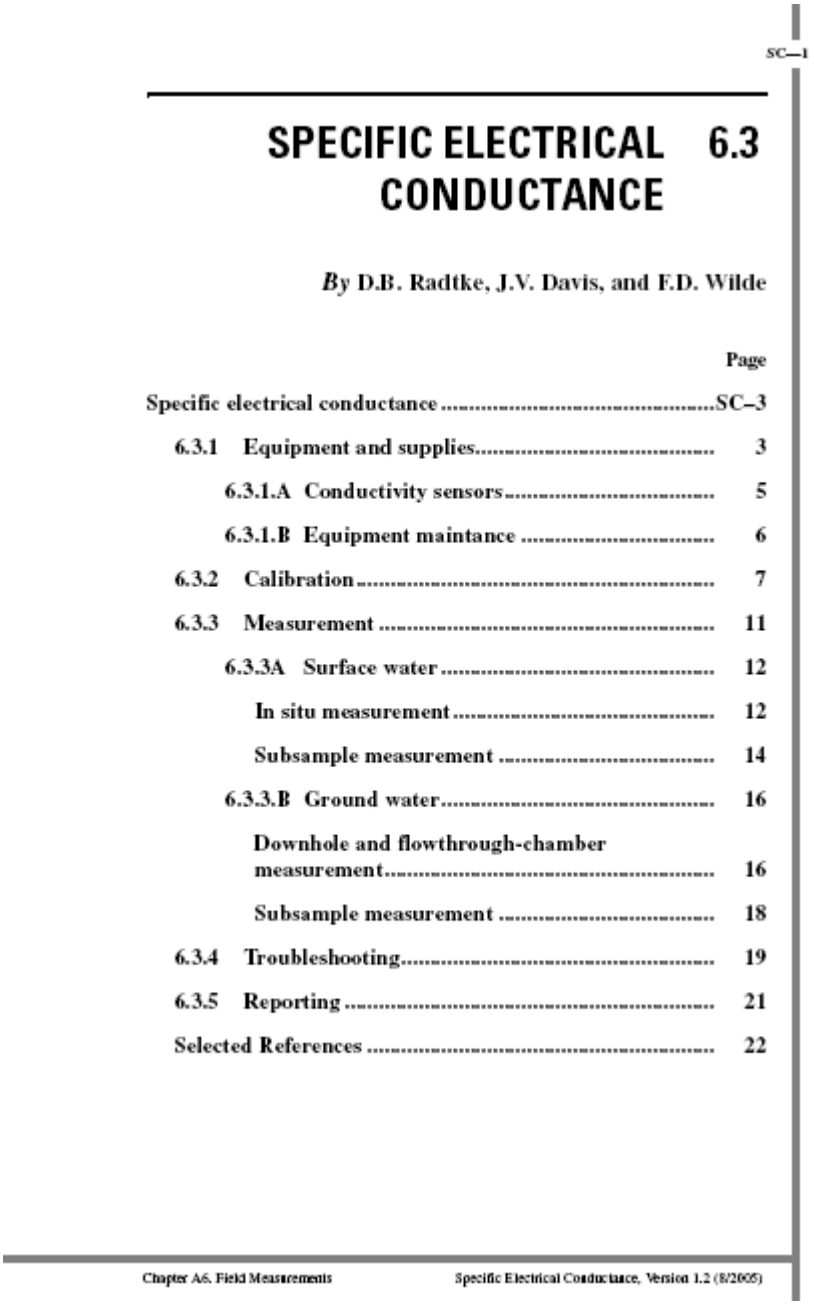
6.2.2.A Equipment and supplies 25

6.2.2.B Calibration and interferences 26

6.2.2.C Measurement 27

3.6.4. Specific Electrical Conductance (8/2005)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



SPECIFIC ELECTRICAL CONDUCTANCE 6.3

By D.B. Radtke, J.V. Davis, and F.D. Wilde

Page

Specific electrical conductance SC–3

6.3.1 Equipment and supplies 3

6.3.1.A Conductivity sensors 5

6.3.1.B Equipment maintance 6

6.3.2 Calibration 7

6.3.3 Measurement 11

6.3.3A Surface water 12

In situ measurement 12

Subsample measurement 14

6.3.3.B Ground water 16

Downhole and flowthrough-chamber measurement 16

Subsample measurement 18

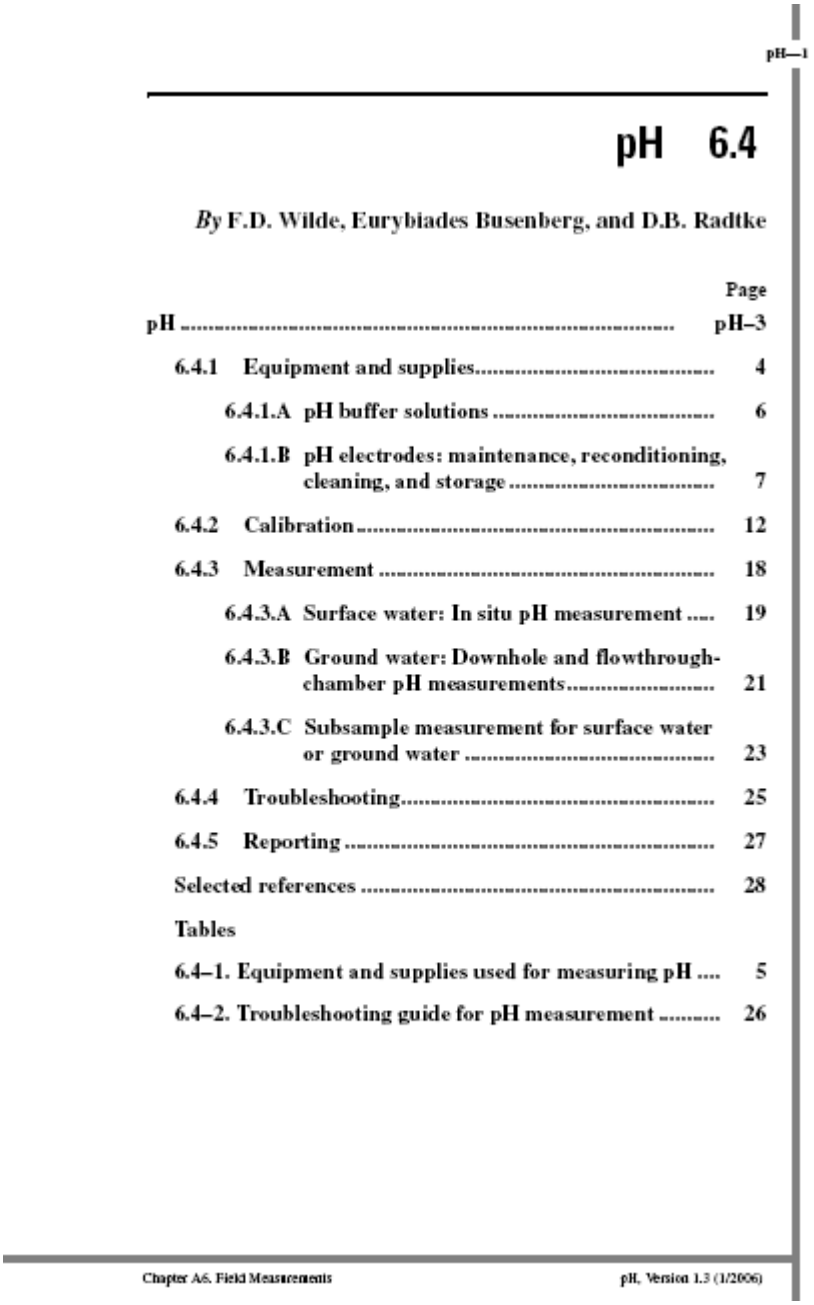
6.3.4 Troubleshooting 19

6.3.5 Reporting 21

Selected References 22

3.6.5. pH (Version 1.3, 1/2006)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



pH 6.4

By F.D. Wilde, Eurybiades Busenberg, and D.B. Radtke

Page

pH pH–3

6.4.1 Equipment and supplies 4

6.4.1.A pH buffer solutions 6

6.4.1.B pH electrodes: maintenance, reconditioning, cleaning, and storage 7

6.4.2 Calibration 12

6.4.3 Measurement 18

6.4.3.A Surface water: In situ pH measurement 19

6.4.3.B Ground water: Downhole and flowthrough-chamber pH measurements 21

6.4.3.C Subsample measurement for surface water or ground water 23

6.4.4 Troubleshooting 25

6.4.5 Reporting 27

Selected references 28

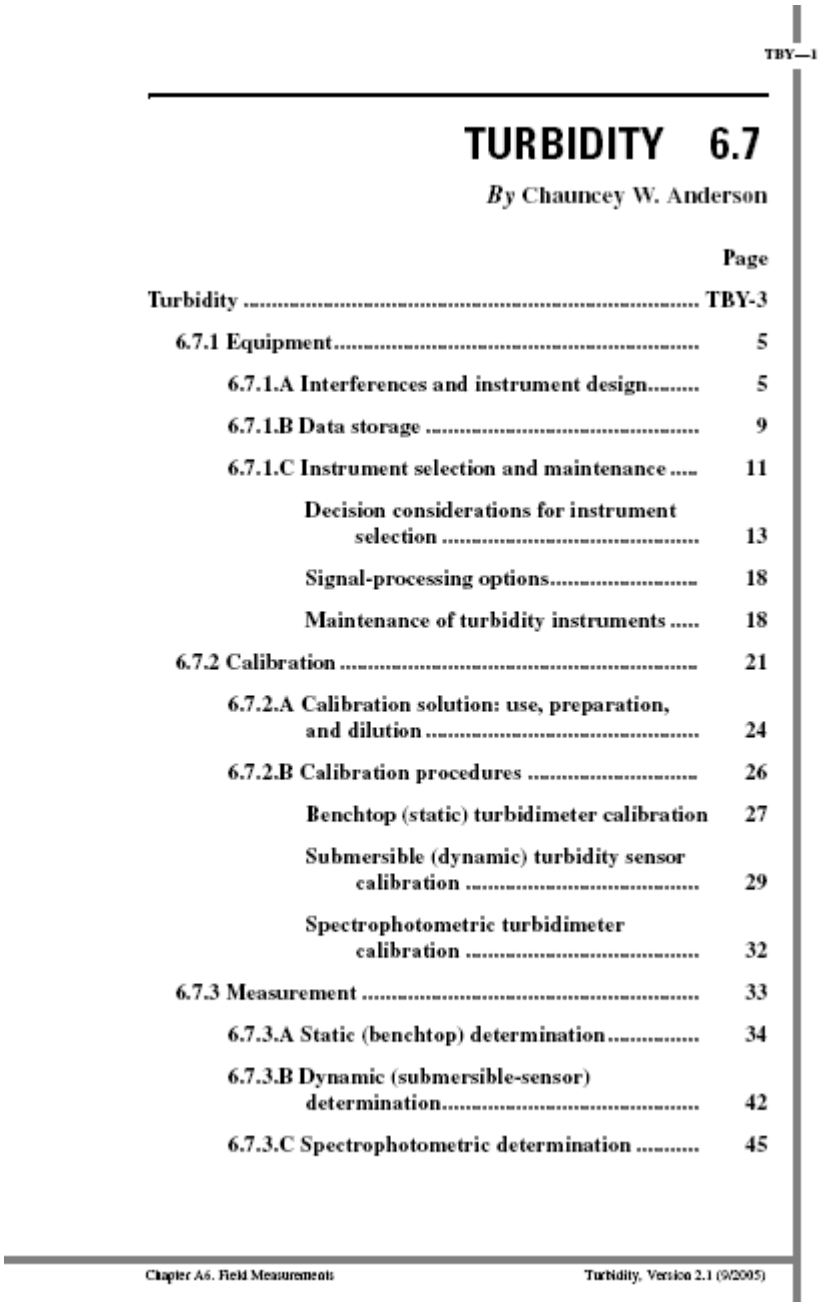
Tables

6.4–1. Equipment and supplies used for measuring pH 5

6.4–2. Troubleshooting guide for pH measurement 26

3.6.6. Turbidity (9/2005)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



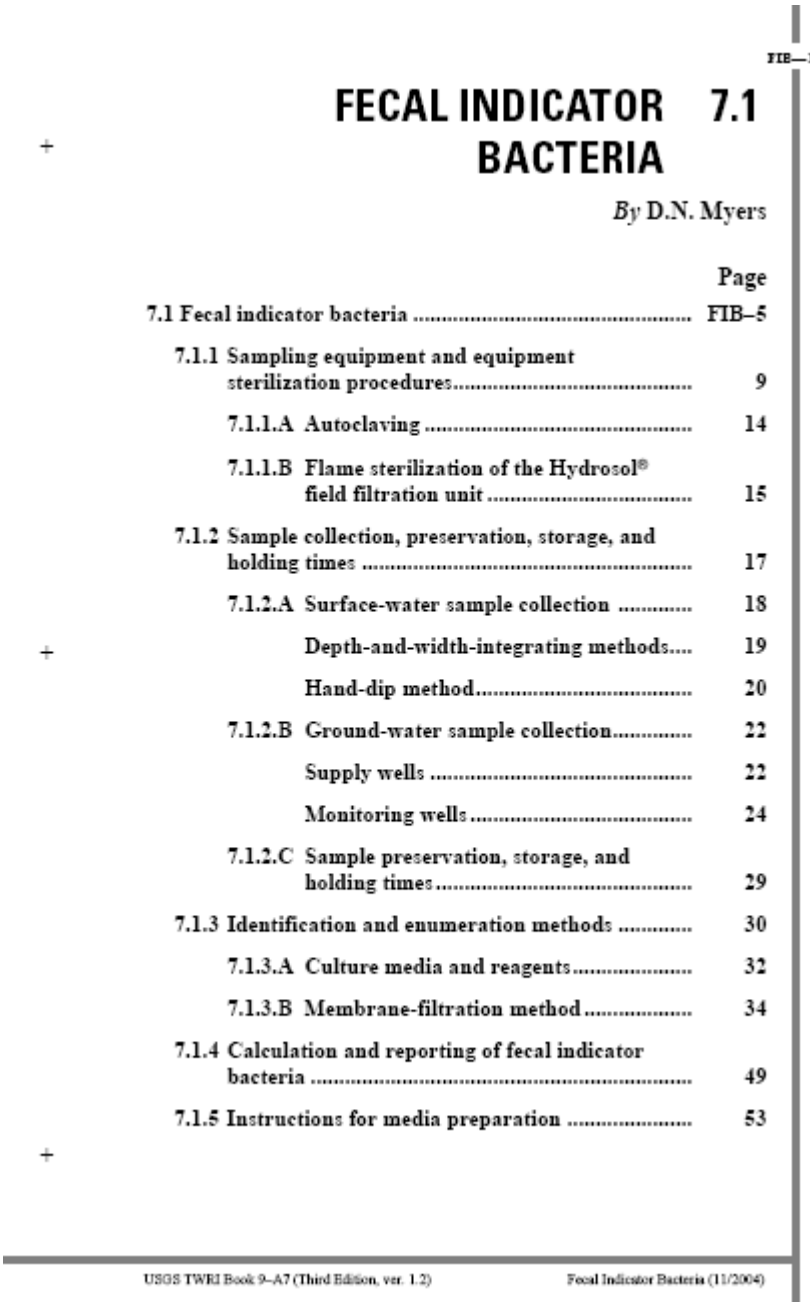
TURBIDITY 6.7

By Chauncey W. Anderson

	Page
Turbidity	TBY-3
6.7.1 Equipment	5
6.7.1.A Interferences and instrument design	5
6.7.1.B Data storage	9
6.7.1.C Instrument selection and maintenance	11
Decision considerations for instrument selection	13
Signal-processing options	18
Maintenance of turbidity instruments	18
6.7.2 Calibration	21
6.7.2.A Calibration solution: use, preparation, and dilution	24
6.7.2.B Calibration procedures	26
Benchtop (static) turbidimeter calibration	27
Submersible (dynamic) turbidity sensor calibration	29
Spectrophotometric turbidimeter calibration	32
6.7.3 Measurement	33
6.7.3.A Static (benchtop) determination	34
6.7.3.B Dynamic (submersible-sensor) determination	42
6.7.3.C Spectrophotometric determination	45

3.6.7. Fecal Indicator Bacteria

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



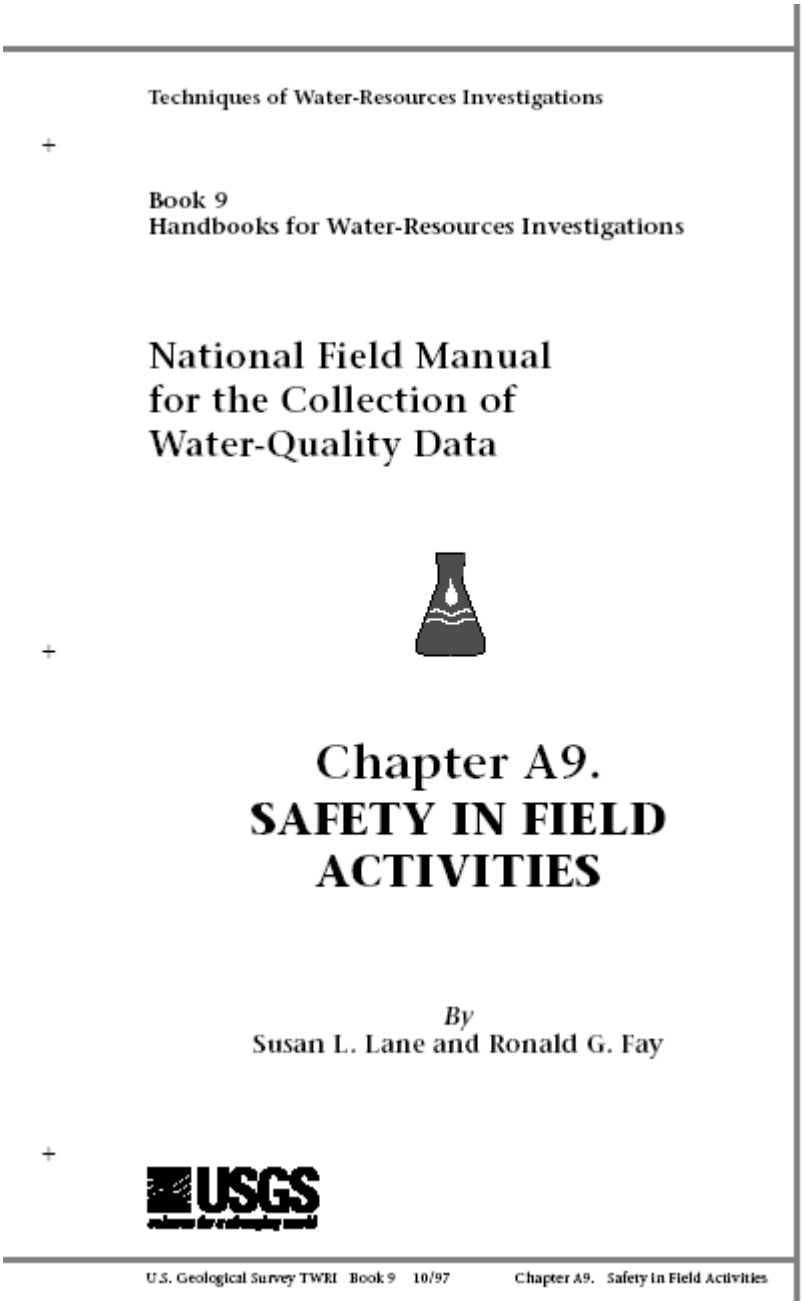
FECAL INDICATOR BACTERIA 7.1

By D.N. Myers

	Page
7.1 Fecal indicator bacteria	FIB–5
7.1.1 Sampling equipment and equipment sterilization procedures	9
7.1.1.A Autoclaving	14
7.1.1.B Flame sterilization of the Hydrosol® field filtration unit	15
7.1.2 Sample collection, preservation, storage, and holding times	17
7.1.2.A Surface-water sample collection	18
Depth-and-width-integrating methods	19
Hand-dip method	20
7.1.2.B Ground-water sample collection	22
Supply wells	22
Monitoring wells	24
7.1.2.C Sample preservation, storage, and holding times	29
7.1.3 Identification and enumeration methods	30
7.1.3.A Culture media and reagents	32
7.1.3.B Membrane-filtration method	34
7.1.4 Calculation and reporting of fecal indicator bacteria	49
7.1.5 Instructions for media preparation	53

3.6.8. Safety in Field Activities (10/97)

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



Techniques of Water-Resources Investigations

Book 9
Handbooks for Water-Resources Investigations

National Field Manual
for the Collection of
Water-Quality Data

Chapter A9.
SAFETY IN FIELD ACTIVITIES

By
Susan L. Lane and Ronald G. Fay

USGS

U.S. Geological Survey TWRI Book 9 10/97 Chapter A9. Safety in Field Activities

4. Appendix B: Errata sheets for all chapters (downloaded 11/17/06)

Comments and Errata
(Revision history for the *National Field Manual*)

A Comments and Errata **archive file** lists changes incorporated into the *National Field Manual* from 1999 through July 2005. The home page for each chapter provides links to archived versions or editions.

4.1. Preparations for Water Sampling

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
7/2006	URL for cited Water Resources Policy Memo No. 99.34 was http://water.usgs.gov/admin/memo/WRD/wrdpolicy99.34.html.	Updated URL is http://water.usgs.gov/admin/memo/policy/wrdpolicy99.34.html.
5/2006	Purpose, title, and contents for Chapter 1 are in need of updating.	Work in process for FY 2006-2007 includes change of chapter title to Quality Assurance and Quality Control for Water Sampling. The quality control section that currently resides in NFM 4 will move to Chapter 1, and the information currently in Chapter 1 that is relevant to surface-water and ground-water sample collection is being merged into Chapter 4.
8/2005	For changes incorporated from 1999 thorugh July 2005, go to the **archive file**.	Currently no changes are noted for version 2.0 dated 1/2005.

4.2. Selection of Equipment for Water Sampling

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
8/2006 - section 2.4, p. 101	Sunscreen and insect repellent are missing from table 2-17.	Add to table 2-17: Sunscreen (wash hands thoroughly after application) Insect repellent - instead of a DEET-containing repellent, recommend either (a) oil of lemon eucalyptus, or (b) picaridin (a chemical). Reference: http://www.cdc.gov/od/oc/Media/pressrel/r050428.htm
8/2006 section 2.1.1.A, p. 24-30	US DH-2 and US D-96 A-1 samplers are missing and should be added.	Add US DH-2 and US D-96 A-1 samplers to table 2.2; add US DH-2 to fig. 2-1. Incorporate descriptions into text. A commercial version of the DH-2 bag sampler can be found at www.carnettechnology.com as "US DH-2A." The D-96 A-1 is an aluminum version of the US D-96.
6/2006		"Universal" blank water has been discontinued for use as inorganic blank water.
8/2005	For changes incorporated from 1999 thorugh July 2005, go to the **archive file**.	Currently no changes are noted for version 2.0 dated 3/2003.

4.3. Cleaning of Equipment for Water Sampling

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
8/2006 Section 3.3.9 p. 54	Instructions for cleaning the Teflon bag are missing. Instructions are provided courtesy of A. Amsberg and H. Johnson (USGS).	**Add to section 3.3.9, at the end of the first paragraph:** "The Teflon bag used in bag samplers must be cleaned and field rinsed before use. The special cleaning procedures recommended are described under step 2 on p. 54. The field-rinse procedure is described in NFM 4.1." **Change step 2, p. 54, to read:** Soak components (except for the Teflon bag) in a detergent solution for 30 minutes. Wearing appropriate disposable, powderless gloves, scrub components with a soft brush or sponge and rinse thoroughly (section 3.2.1 or 3.2.2). Change gloves. To clean the Teflon bag: 1. Fill the bottom of the bag with warm 0.1% Liquinox solution (approx. 100-200 mL). 2. Pull the bag (gently) into a tube-like shape; loosely fold the top (opening) over and slosh the solution back and forth from the bottom to the top of the bag. 3. With the bag still pulled into a tube-like shape, hold it horizontally and rotate it around, making sure the Liquinox flows into all of the folds. 4. Rinse with hot tap water, in the same fashion as the soapy rinses; back and forth from top to bottom, then rotating while holding horizontally. 5. Repeat this procedure with 5% HCl. 6. Repeat the rinse technique three times, using fresh DIW. 7. Holding the bag open with one hand, squirt the inside of the bag with methanol (only necessary if samples will be analyzed for organic compounds). Ensure full contact of inside bag surfaces by repeating the sloshing and rolling techniques before decanting waste methanol into approriate containers.
6/2006		"Universal" blank water has been discontinued for use as inorganic blank water.
11/21/2005 Ver. 2.0, 4/2004, pg. 12.		Add a new first arrow above the existing first arrow, with the following text: Clean all surfaces (for example, bench and counter tops) on which you will be working. If a disinfectant is used, thoroughly rinse and dry the surface afterwards and be sure to air out the area to remove any fumes from the dininfection agent. Do not expose a sample to the area if fumes remain that could contaminate the sample with an analyte of interest.
11/21/2005 Ver. 2.0, 4/2004, pg. 13.		Insert after the first sentence or add as a footnote to the first sentence the following: Refer to NFM 7.1 for sterilization and disinfection requirements when sampling for microbial organisms.

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
11/21/2005 Ver. 2.0, 4/2004, pg. 14, Table 3-1.	Add a row to the table after the "Detergent" row.	LEFT COLUMN: Disinfectant RIGHT COLUMN: Hypochlorite wipes (commercially available), dilute bleach solution, or methyl or ethyl alcohol solution. ADD FOOTNOTE TO TABLE: **Hypochlorite wipes:** At 0.525-percent (1:10) dilution, these wipes are more concentrated than that of the bleach solution and could be more corrosive to equipment if not followed by the standard DIW rinse.
11/21/2005 Ver 2.0, 4/2004, pg. 53. #1 under "**Disinfecting an electric or steel tape**:"	1. Select a disinfectant: either a chlorine bleach solution (described below) or a methyl or ethyl alcohol solution. If using bleach, prepare a dilute 50 mg/L (0.005 percent) solution of common household chlorine bleach (1 mL of bleach to 900 mL water).	1. Select a disinfectant: either a chlorine bleach solution (described below), commercially available hypochlorite (disinfectant) wipes, or a methyl or ethyl alcohol solution. o If using the hypochlorite wipes, be sure to check the expiration date printed on the packet; do not use wipes with expired dates. Note: as these wipes are more concentrated in hypochlorite (0.525 percent) than the bleach solution (0.005 percent), care should be taken to avoid equipment damage. o To prepare a 0.005-percent (50 mg/L) bleach solution[9]: (a) Wearing laboratory gloves, add 1 mL of common household chlorine bleach to 900 mL water, then add water to bring it to a volume of 1,000 mL (USEPA, 1982, p. 253, and 1996, p. VIII-41). The bleach solution should be prepared fresh for each day of use (NFM 7.3). (b) Label a polyethylene sample bottle as "Well-Tape Disinfectant" and record the date of preparation, using an indelible marker. (c) Fill the bottle with the dilute disinfectant solution. Cap the bottle tightly, and double-bag it in ziplock plastic bags for transport. 2. At the well site, put on disposable gloves before disinfecting the equipment. Wipe down the section of the tape that was submerged in the well water, using the disinfectant of choice. o For the bleach or alcohol solution, wet a clean cloth. o If disinfecting an electric tape, check with the manufacturer before exposing the tape and related equipment to a solvent. 3. Rinse the tape thoroughly with DIW or tap water: prolonged exposure of the tape to chlorine bleach can damage the tape. Take care to keep the tape clean; prevent it from dragging on the ground. 4. Rewind the tape onto the reel and place it into a clean plastic bag for storage and transport. o Using a clean, dry cloth, dry the steel tape completely before rewinding, to prevent it from rusting.

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
		o Recommendation: If the tape will be stored for a month or more, put a drying agent such as a silica-gel packet into the plastic bag before sealing the bag. Footnote #9 (see second bullet from no. 1): Prepare a 0.02-percent (200 mg/L) solution if pH is less than 6 or greater than 8.
8/2005	For changes incorporated from 1999 thorugh July 2005, go to the **archive file**.	Currently no changes are noted for version 2.0 dated 4/2004.

4.4. Collection of Water Samples

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
6/2006		"Universal" blank water has been discontinued for use as inorganic blank water.
5/2006	Surface-water and ground-water sampling protocols need updating.	Revisions are being completed in FY 2006 and will be published in FY 2007 or before. Updates include changes to surface-water sampler tables (in the appendix), in addition to other information. Important changes are being made to the ground-water guidance for purging and sampling, including guidance when working at low-yield wells. The quality-control section (NFM 4.3) is being moved from Chap. 4 to Chap. 1.
11/08/04 -- p. CF-5, App. A4-B, and App.A4-C table 1	Update chapter to reflect the closure of the Ocala Water Quality and Research Laboratory	Delete reference to the Ocala laboratory. Supplies are available from the USGS "One-Stop Shopping."
5/2/03 -- NFM 4.2.1, Step 3, page 78	Additional guidance is needed to properly rinse out the sample line during the purge and before samples are collected.	Insert as first sentence for Step 3b: "Discharge initial well water through the waste line that goes to the flowthrough chamber." Insert as first sentence for Step 3c: "When the water runs clear, switch the flow to the sample line (line that goes to the collection chamber) and allow at least two tubing volumes to pass through so as to rinse and condition the sample line with well water. Then. . . " (continue into next sentence, changing the B in Begin to lower case).
5/2/03 -- NFM 4.2.2, Step 3d, page 86	Additional guidance is needed to properly rinse out the sample line during the purge and before samples are collected.	Add a fourth bullet: "While purging the first two well volumes, and before starting field measurements, once the well water is running clear, switch the flow from the "waste" line to the sample line, to ensure that the sample line is thoroughly rinsed of methanol and other equipment-cleaning agents."
11/29/02--REF-2		Add reference for Izbicki, John, Christensen, Allen, and Hanson, Randy, 1999, U.S. Geological Survey combined well-bore flow and depth-dependent water sampler, U.S. Geological Survey Fact Sheet 196-99, 2 p., at http://ca.water.usgs.gov/archive/reports/fs19699.pdf Add reference for Miller, James, ed., 2002, Ground water atlas of the United States, http://capp.water.usgs.gov/gwa/index.html
4/4/01 -- p. 69 of NFM A4	Third bullet, second to last sentence: "... (2) field	"...(2) field-measurement values should be determined before collecting samples, if possible."

	measurements must be monitored before collecting samples."	

4.5. Processing of Water Samples

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
10/2006		The silver-filter method for processing DOC samples no longer is used by USGS water-quality field personnel. This method will be removed in the forthcoming revision of NFM 5.
6/2006		"Universal" blank water has been discontinued for use as inorganic blank water.
5/16/05	Chapter needs to be updated and compiled to include new sections.	Update is in process. Expected completion date of 10/30/05 has been extended into FY 2007.
1/2005 -- 5.6.4.A	Update references to McCleskey and others (in press)	McCleskey and others was published in Applied Science in 2004.
9/30/04 -- 5.4.2, Chemical Treatment, p. 93, step 5.c.	Correct the preservation order and update text to reflect current chemical treatments	Correct preservation order and preservatives as follows: 1. Nutrients (4.5 N H_2SO_4) 2. Organic carbon (4.5 N H_2SO_4) 3. Trace elements (HNO_3) 4. Major ions (HNO_3) 5. Mercury (6 N HCL) 6. Others that require acidifying (HCL first) 7. Samples requiring other preservatives (zinc acetate, sodium hydroxide, etc.)
2/23/04 -- 5.2.2, "Filtration"		Starting 2/23/2004, a low-capacity (19.6 cm^2) disposable filter-- the "Aquaprep"^{TM}--will be available for USGS use through the One-Stop Shopping program ('Q460FLD--Filter, Aquaprep^{TM}, 0.45 micrometer'). This filtration device only is acceptable for filtration of small-volume water samples at sites at which the amount of suspended material is sufficiently low so that the filter will not clog during the sample-filtering process. These special conditions for use must be adhered to strictly. **To prepare the 19.6 cm^2-Aquaprep^{TM} for field use, follow the same procedures as for the 600-cm^2 disposable Aquaprep^{TM} capsule filter, but use 100 mL** (instead of 1,000 mL) **of quality-controlled DIW to rinse the interior of the unit.**
1/5/04 -- 5.6.1.E, Oil and Grease	**COMMENT** -- The procedure for adding acid to adjust sample pH to 2 or less should provide guidance	**ACTION** -- The next version of section 5.6.1.E will be revised by adding the following technical note under Step 3: TECHNICAL NOTE: To establish the volume of H_2SO_4 required, collect a separate aliquot of sample and adjust the pH of this aliquot by adding the acid until pH

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
	as to how to determine the volume of acid needed for a given sample.	indicator paper verifies that the pH has been lowered to or less than 2. This aliquot is for testing purposes only and is not to be used for oil and grease analysis. Add the volume of acid determined to the sample to be analyzed. Do not dip pH paper, a pH electrode, a stirring rod, or other materials into a sample that will be used for Hexane Extractable Materials or Silica Gel Treated - Hexane Extractable Materials determination.
1/2/04 -- 5.0.1, 5.0.3, 5.6.2	COMMENT: IBW should be used instead of DIW for rinsing bottles used for mercury and other trace-element samples if the DIW is not of known quality and could contaminate the sample.	NFM 5.0.1 -- RESPONSE: (page 16, 2nd arrow, 2nd dash), add "inorganic-grade blank water (IBW) or" before the words "deionized water (DIW)" and after "(DIW)" add "of appropriate quality." Same section, page 18: second dash, add "IBW or" before each occurrence of "DIW." NFM 5.0.3 -- RESPONSE: (page 21), first sentence, before the word "DIW" add "IBW or"; Table 5-2, add "IBW, inorganic-grade blank water" to headnote. Changes to both bullets -- add "IBW or" before "DIW" in all places and delete footnote 1. NFM 5.6.2 -- RESPONSE: (page 107), after first sentence add: "Use IBW instead of DIW for the pre-field bottle rinse/fill if the composition of the DIW could contain target analytes at detectable concentrations for the analysis to be performed on the environmental samples. (DIW could have measureable concentrations of trace elements, for example, if the ion-exchange column is nearing its exchange capacity or is defective; DIW should be analyzed periodically to determine that the system is operating as intended). Alternatively, if the pre-field bottle rinse/fill procedure is not being used, the sample bottle should be rinsed in the field twice with IBW, followed by a field rinse with sample (wholewater for wholewater samples, filtrate for filtered samples) before filling the bottle (NFM 5.0.3). The DIW/IBW rinse of bottles for samples being collected for wholewater analysis can be substituted by field rinsing with sample a minimum of three times before filling the bottle."
5/21/03 -- 5.6.3.K, Chlorofluorocarbons, p. 122-125.	A new method for collecting CFC age dating samples has been developed that eliminates the need for flame sealing of the samples for most sampling applications. Add the description of the new method	A new method for collecting CFC age dating samples has been developed that eliminates the need for flame sealing of the samples for most sampling applications. The new method is similar to the method for collecting dissolved gas samples. Bottles with special caps are filled and capped under water. Check for information at the webpage water.usgs.gov/lab/cfc/ under "New Sampling Method for CFCs." The new method simplifies sample collection greatly. If long-term archival of samples is desired, the original flame sealing technique can still be applied.

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
	to all appropriate references in Chapter 5, including section 5.6.3.K. (Chapter 5 is in the process of being revised and new instructions will be incorporated.)	
11/29/02 -- REF-1		Add reference to Izbicki, John, Christensen, Allen, and Hanson, Randy, 1999, U.S. Geological Survey combined well-bore flow and depth-dependent water sampler, U.S. Geological Survey Fact Sheet 196-99, 2 p., at ca.water.usgs.gov/archive/ reports/fs19699.pdf
3/17/01 -- CFC sampling, p. 123-128 of NFM A5	**COMMENT:** CFC sampling -- SF_6 sampling methods and information can be found at http://water.usgs.gov/lab/cfc/sampling/sf6.html. This information should be used in conjunction with the instructions for CFC sampling on p. 123-128 of NFM A5, for samples to be analyzed by the USGS CFC Laboratory.	Section is being updated.
3/17/01 -- Dissolved gases sampling (in ground water).	**Comment:** Sampling procedures for collection of methane and other dissolved gases in ground water can be found at http://water.usgs.gov/lab/dissolved-gas/ and in "Baedecker, M.J., and Cozzarelli, I.M., 1992, The	Suggestion: Collection and processing methods for commonly sampled gases should be added to NFM Chapter A5

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
	determination and fate of unstable constituents of contaminated groundwater, *in* Lesage, Suzanne, and Jackson, R.E., eds., 1992, Groundwater contamination and analysis at hazardous waste sites: New York, Marcel Dekkar, Inc., p. 425-461." The method to be selected depends on study objectives, site conditions, and the analyzing laboratory.	
11/8/00 -- Appendix A5-A, p. APP-A-2, column 1, Append to table a listing for "Ultraviolet-absorbing substances"	Comment: An entry for ultraviolet absorbing substances should be added to the table in the future.	In progress. Bottle type and other instructions are in development. Contact Ron Brenton/NWQL (rbrenton@usgs.gov, 303-236-3210) for update.
10/27/00 -- 5.3 Solid-Phase Extraction by C-18 Column - pages 77-82	A COMMENT has been made to update the C-18 SPE method, method 2011, and change the reference to Sandstrom and others (2000). Add to Fig. 5-2 a line for "Extraction Date". Add a line to Fig. 5-3 about the addition of NaCl to the sample in the sample field form for 2051.	NO CHANGE AT THIS TIME. ANALYSIS: Users are alerted to this information and that these changes will be made in a future version but, at this time, have been assigned a noncritical status.
10/19/00 -- Appendix A5-A,	In fourth row, last column, change	Replace with "Raw, untreated sample. Chill/maintain at 4 degrees C."

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
page APP-A-1	"Filtered sample (0.7-um GF/F), untreated. Chill/maintain at 4 degrees C."	

4.6. Field Measurements

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
August 22, 2006 -- Section 6.6, Alkalinity and Acid Neutralizing Capacity	2nd Edition, 9/2001	Version 3.0, 7/2006, contains minor updates. The term "edition" was changed to "version" to be consist with the tracking convention for other sections.
August 14, 2006 -- NFM 6.5.1.B, page 6 and NFM 6.5.2, page 8	Add information about disposal of ZoBell's solution.	Page 6, add new paragraph after step 3: ***To dispose of ZoBell's solution after use***: o **Do not dispose of through the sewer system. Use a qualified hazardous waste hauler and Treatment, Storage, or Disposal Facility (TSDF)** (see http://www.epa.gov/radiation/mixed-waste/mw_pg11a.htm) o Note that the cyanide compounds in this waste stream are at a concentration of one percent and can be classified as a RCRA D003 (reactive) or non-hazardous waste stream. It is up to the generator of the waste to make this determination. o Whether you dispose of this as RCRA haz or non haz, the U.S. Department of Transportation (DOT) shipping name is cyanide solutions, n.o.s. 6.1 (Toxics). Also on p. 6, change the Black Box to: "CAUTION -- ZoBell's solution is toxic - handle with care and dispose of ZoBell's-solution waste according to governmental regulations." Page 8, third arrow, add a third dash: "- ZoBell's is considered a hazardous waste; after use, contain the waste properly for subsequent appropriate and legal disposal. If in doubt, consult your safety officer."
June 20, 2006 -- NFM 6.5, Section 6.5.5	Report the calculated Eh in mV to two significant figures. Potentials are reported to the nearest 10 mV, along with the temperature at which the measurement was made, the electrode system employed, and the pH at the time of measurement.	Report Eh in millivolts (mV), calculated relative to the standard hydrogen electrode. Do not report raw data. Replicate measurements should stabilize within plus or minus 10 mV. Report the median of at least three replicate measurements to the nearest mV. If continual drifting without stabilization occurs after extending the measurement period, record this difficulty on the field form along with the length of time during which Eh values were monitored.
June 16, 2006 -- Section 6.2	Update description of the equipment for the spectrophotometric method.	Version 2.1, 6/2006; table 6.2-4. the VacuVial catalog number was changed to K-7553 and information was added about the

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
		photometer.
June 7, 2006 -- Section 6.2	Version 2.0, May 2006	Version 2.1, June 2006. Updates were made to the example information listed for CHEMetrics, Inc.
June 6, 2006 -- Section 6.0, p. 27	In the footnote for fig. 6.0-3, the URL was http://water.usgs.gov/owq/turbidity/turbidity.pcodes_mcodes.update.xls	The updated URL is: http://water.usgs.gov/owq/turbidity_codes.xls
May 23, 2006 Section 6.2, Dissolved Oxygen	The original version, dated April 1998, is due for an update to reflect new technology and method changes.	Version 2.0, 5/2006: A new method has been added for DO measurement by luminescent sensor technology. Updates have been made for the amperometric and spectrophotometric methods. At DO concentrations of less that 2 mg/L (instead of 1 mg/L), the spectrophotometric method is recommended. The iodometric (Winkler) method has been moved to the main body of the report.
May 23, 2006	A section on the use of multiparameter instruments is needed.	A new section is being prepared on the use of multiparameter instruments to collect field measurements. Publication is scheduled for FY 2007.
May 3, 2006 -- Reference update for Chapter 6	In "Selected References" for NFM 6 sections, the following reference is being replaced: Wagner, R.J., Mattraw, H.C., Ritz, G.F., and Smith, B.A., 2000, Guidelines and standard procedures for continuous water-quality monitors--Site selection, field operation, calibration, record computation, and reporting: U.S. Geological Survey Water-Resources Investigations Report 00-4252, 53 p.	The updated report is: Wagner, R.J., Boulger, R.W., Jr., Oblinger, C.J., and Smith, B.A., 2006, Guidelines and standard procedures for continuous water-quality monitors--Station operation, record computation, and data reporting: U.S. Geological Survey Techniques and Methods 1-D3, 51 p. + 8 attachments, online only at http://pubs.water.usgs.gov/tm1d3
April 5, 2006 -- 6.1, Temperature	Version 1.2, April 2004	Version 2, March 2006. The Calibration section received major updates. Other sections received minor modifications.
March 1, 2006 -- pH, Section 6.4	Version 1.2, 7/2003	Version 1.3, 1/2006. The new version includes minor updates and reformatting of subsections.
9/30/05 -- Section 6.7	Revise the Excel spreadsheet of turbidity parameter and method codes.	Changes are ongoing and can be accessed from water.usgs.gov/owq/FieldManual/Chapter6/6.7_contents.html.

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
9/30/05 -- Change from 5/16/05 entry (below)	Update 6.0, 6.6, 6.7, as needed.	Updated versions of sections 6.0 and 6.3 have been posted.
5/16/05 -- Sections 6.1, 6.2, 6.3, 6.4, 6.5	Updates are being made to listed sections, and references will be included with each section rather than being grouped in the back of the chapter.	6.1 Temperature (updates needed) 6.2 Dissolved Oxygen (rewrite in completion) (8/2005) 6.3 Specific Electrical Conductance (updates needed) 6.4 pH (updates needed) 6.5 Redox (updates needed)
2/2/05 -- Section 6.7 - "Tubidity" (version 2.0, 8/2004)	Selected references (see "Change To" column) to NTU should be updated to include NTRU.	p. 36, section 1b, 3rd line: Insert "or NTRU" after "in NTU" p. 38, section 1, first bullet, 2nd line down: Insert "or NTRU" after "and NTU" p. 41, section 6e, first bullet: Insert "or NTRU" after "use NTU".
1/31/05 -- Section 6.7 - "Turbidity." On Web page water.usgs.gov/owq/FieldManual/Chapter6/6.7_contents.html, go to the link for "Turbidity parameter codes and methods codes" (Excel spreadsheet)	For the Orion AQ4500 turbidity meter (Thermo Electron Corporation), the correct reporting unit and NWIS pcode for white-light source are NTRU (63676). Delete NTU 63675 codes. Also, add the AU 63678 and FAU 63683 codes.	The changes for the Orion AQ4500 listed below will be entered with the next update of the Excel spreadsheet as follows: Instrument: Orion AQ4500 Method NWIS Pcode Rep. Unit Description/ Source 63676 NTRU EPA 180 63680 FNU ISO-NEPH 63683 FAU ISO-ABSB and IR%T 63681 FNRU IR RATIO 63678 AU WHITE %T
8/11/04 -- Section 6.7, "Turbidity"	Add a section or explanation on how to use calibration constants to troubleshoot and quality assure turbidity calibrations.	This suggestion is likely to be incorporated into the next update of section 6.7. An example of use of calibration constants for quality assurance of the turbidity determination follows: The submersible probes manufactured by YSI (models 6026 and 6136) have a desired operating range for their constants (see the YSI 6-series manual); these can be quite valuable in determining the validity of a calibration. For example, a discrepancy was found in some calibrations when going from an old bottle of Formazin to a new one (from the same manufacturer). Technical support at YSI suggested checking the constants, and the resulting data showed significant improvement in the constant when a new

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
		bottle of standard was used. Also, data showing deterioration of the constant after instrument calibration indicates that there may be a problem with the instrument sensor or with the particular bottle of standard used. Constants also can be used to determine how carefully field personnel mix standards. For the YSI units, the constants can be easily retrieved using a display unit or they can be downloaded as a printable historic record/file using their Ecowatch software (usually provided free with the purchase of a sonde unit). Assuming that one keeps good notes and careful track of their standards, monitoring the constants could potentially save time and money when trying to clear up confusion or subtle problems that might arise with successive calibrations.
8/28/03-- Section 6.4.1	Second paragraph begins: "Measurement of pH can be either electrometric or colorimetric."	Delete this sentence.

4.7. Biological Indicators

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
May 2006	Need to add information on chylorphyll sampling.	A new section on chlorophyll sampling is being prepared. Publication is scheduled for FY 2007.
9/30/2005	Updates are needed to relevant sections of Chapter 7 to reflect information in Office of Water Quality Technical Memorandum 2005.02 (available from water.usgs.gov/ admin/memo/).	Updates are scheduled for completion by October 2006.
8/26/2005 Section 7.1.5, first sentence.	1. mENDO medium for total coliform analysis. a. Empty the vial containing 4.8 g of dehydrated mENDO medium into a 250-mL flask and add 100 mL of a 2 percent ethanol solution.	1. mENDO medium for total coliform analysis. a. Empty the vial containing 5.1 g of dehydrated mENDO medium into a 250-mL flask. Add 100 mL of an ethanol solution. To prepare the ethanol solution, combine 2 mL of 95% undenatured ethanol with 98 mL of deionized/distilled water and mix well.
8/2005	For changes incorporated from 1999 thorugh July 2005, go to the **archive file**.	Currently no changes are noted for the third edition of chapter 7.

4.8. Bottom-Material Samples

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
8/2005	For changes incorporated from 1999 thorugh July 2005, go to the **archive file**.	Currently no changes are noted for version 1.1 dated 6/2005.

4.9. Safety in Field Activities

Comment: For a comprehensive safety policy on streamgaging, see USGS Water Resources Division (WRD) Memorandum 99.32, SAFETY--WRD Policy for Safety Associated with Discharge Measurements, Sampling, and Related Streamgaging Activities. The memo is available at: http://water.usgs.gov/admin/memo/. Click on "WRD Policy and Safety", scroll down the page and click on "wrdpolicy99.32.html". This memo not only provides policy, but it also is meant to improve safety awareness. Emphasizing safety in the workplace does reduce the chance of serious injury and benefits everyone.

DATE POSTED/ SECTION/PAGE	CHANGE FROM	CHANGE TO
8/2006 -- p. 45 Section 9.9.1	Danger from mosquitoes is missing.	Add information on mosquitoes to section 9.9.1, including the following recommendation on use of insect repellent to avoid contaminating the sample with DEET: Instead of a DEET-containing repellent, use (a) oil of lemon eucalyptus, or (b) picaridin (a chemical). Reference: http://www.cdc.gov/od/oc/Media/pressrel/r050428.htm
10/10/00 -- p. 11 Section 9.1.1, first paragraph	Reference for *A Guide to Safe Field Operations* is U.S. Geological Survey, 1995	Change reference to read: *A Guide to Safe Field Operations* (Yobbi and others, 1996)

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #10

Field Sample Collection and Processing for Water Chemistry, Nutrients and TSS, Utah Units

Version 1.0 (4/11/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

The purpose of this SOP is to outline procedures for the collection and handling of water samples to ensure accurate field data collection and that representative samples of surface water bodies arrive uncontaminated at the state laboratory in a timely manner.

Table of Contents

1. Introduction **3**
2. Preparations for sample collection **3**
2.1. Equipment needed for sample collection 3
2.1.1. Site visit *3*
2.1.2. At vehicle *3*
3. Label bottles **3**
4. Sample Collection **4**
5. Filtering **4**
6. Bacteria **5**
7. Holding Samples **5**
8. Data Forms **5**
8.1. Paper 5
8.2. Digital 6
8.2.1. Lab sheets *6*
8.2.2. Run sheets *6*
8.3. State of Utah codes 6
8.4. Run sheet digital summary 7
9. Mailing **8**
10. Contacts **8**
11. Appendix A: Methods of Sampling Based on Discharge **9**
11.1. Equal-Discharge Increments (EDI) 9
11.2. Equal-width increments (EWI) 11
12. Appendix B: NCPN Custom Data Forms **15**
12.1. Monthly Lab Sheets 15
12.2. Quarterly Lab Sheets 23
12.3. Monthly Run Sheets 31
12.4. Quarterly Run Sheets 32
13. Appendix C: Utah Excel Spread sheet instructions **33**
13.1. Example hard copy of UT Excel spreadsheet 35

List of Tables

Table 1. Number and type of bottles for sampling. 4
Table 2. Example of field notes for a discharge-weighted conductivity measurement 11
Table 3. Example of field notes for an area-weighted conductivity measurement 13

1. Introduction

Sample collection for UT park units is fairly straightforward, but must follow a standard procedure to ensure samples are not contaminated, stored properly and to insure internal consistency between labels so that field and laboratory data can be matched later when data are catalogued and analyzed.

2. Preparations for sample collection

2.1. Equipment needed for sample collection

2.1.1. Site visit

1. Bottles have an expiration date printed on the label. Discard expired bottles and request new ones from Andy Christiansen at the UTDWQ lab 801-584-8204.
    a. Chemistry bottle
    b. Filtered metals bottle
    c. Non-filtered nutrient bottle
    d. Filtered nutrient bottle (DINO only)
    e. Carry bottle
2. Lab sheet
3. Turbidity cell and protective baggie
4. Sharpie
5. Rugged Reader
6. Forms

2.1.2. At vehicle

1. 0.45µM filter pack
2. Rubber tubing
3. Peristaltic pump
4. DI water
5. Cooler with ice in the vehicle for cooling samples
6. Turbidity meter with reference cells

3. Label bottles

Record the following on each bottle **label** with a sharpie:

1. STORET number
2. Date
3. Time
4. Sample point name
5. Sampler Initials

Record the following on each bottle somewhere **outside of the label** on the bottle incase the label peels or wears off. Do not write on the lids, or on shoulder of bottle - this space is for lab use

1. STORET number

2. Date
3. Sample point name

Table 1. Number and type of bottles for sampling.

Bottle	Number per Site	Volume	Acid Preservative
Chemistry (major ions)	1	1000 ml	None
Filtered Metals	1	250 ml	HNO_3
Non-Filtered Nutrients	1	500 ml	H_2SO_4
Filtered Nutrients	1 (DINO only)	250 ml	H_2SO_4

4. Sample Collection

After labeling:

1. In well mixed streams, simply immerse bottle just below surface (if possible) to fill.
    a. In non-well mixed or large rivers see Appendix A – Methods of sampling based on discharge.
2. Do not overfill as acid preservative may be lost
3. To maintain acid preservative, do not rinse in stream.
    b. In low flow situations or shallow waters you may have to improvise by filling sample containers from a carry bottle rinsed 4 times.
4. There is no requirement for composite samples, because most streams will be thoroughly mixed.
5. Use gloves
6. No Smoking
7. Do not leave vehicle running
8. Do NOT fill the "Filtered Metals" bottle directly from the stream.

5. Filtering

Filtering from stream into sample bottles that require filtering is ideal, but not always practical. A "carry" bottle can be used if this is not possible. You can collect water in a larger container such as an extra chemistry bottle requested from lab, but do not filter from the chemistry bottle to be sent to the lab for analysis. Use a new carry bottle every day.

1. Rinse the carry bottle 4 times with sample water. Let it settle a bit before filtering metals.
2. Use 0.45µ filter pack to filter nutrients (DINO only) and metals as soon as possible after collection.
    a. Prepare tubing
        i. Rinse both ends of tubing exterior well with DI water before inserting in peristaltic pump or carry bottle.
        ii. Insert inflow end into carry bottle

iii. Purge tubing with sample water for 30 seconds without filter.
iv. Turn off pump

b. Prepare filter pack

i. Attach filter pack to outflow end of tubing with attention to flow direction labeled on the filter pack.
ii. Invert filter pack to 45 degree angle and turn on peristaltic pump
iii. Thump the filter pack vigorously to help dislodge any air bubbles.

3. The pump frequency will increase when air blockages have been removed
4. Continue rinsing filter pack with sample water for 30 sec. New or rinsed tubing will be preferable where the unfiltered sample has been hiked out.
5. Can use either individual filters in an in-line filter holder, or a filter cassette. The individual filters have much less surface area and will clog if the water is even a little turbid. A prefilter can be used if needed. Cassette filters are also prone to leakage around filter edges.
6. After prepping the filter pack filter directly into the filtered metals and or filtered nutrient (DINO only) sample bottle.

6. Bacteria

See SOP 11

UDEQ is still in the process of switching from fecal coliform to E-coli. As yet they have standards, but no official protocol. NCPN and SEUG purchased the Colilert system and has implemented this procedure in UT parks.

7. Holding Samples

1. Hiking out without ice is OK for all but bacteria, but chill them soon after
2. Bacteria samples must be iced at sample collection and kept below 10°C until incubation.
3. Get samples on ice or in refrigerator as soon as practical. Hold at 4°C.
4. Chill samples in refrigerator overnight to ensure that they are at 4°C before shipping.
5. Holding time is as short as 48 hours for turbidity and pH. Since these are measured accurately in the field, the practical holding limit is 7 days for TSS and TDS. Note: Samples are analyzed in the order they were received. Due to large sample numbers submitted to the lab samples may sit for several days before they are analyzed. For this reason cooperators should not hold samples more than 4 to 5 days to ensure holding times are not exceeded.
6. Get samples from multiple sites mailed expeditiously so lab has time to run them before holding time expires.

8. Data Forms

8.1. Paper

Blank forms can be found in SOP 8 Appendix D thru G. Data forms include pre-made Lab and Run sheets that differ slightly by location and sampling frequency. Namely, the

chemistry code is 4 for monthly sampling and 3 for quarterly sampling. Quarterly sheets are used in January, April, July and October

8.2. Digital

Both forms that accompany samples to the lab are automatically filled in using Field_forms.xls. Lab forms can be found at the bottom of each site page. Run forms have their own page titled "UT_master". Both can be printed by swiping the area of interest and printing "selected area".

The two data forms are described in the following sections.

8.2.1. Lab sheets

Lab sheets (ironically) are taken to each field sampling site and filled out while on location, recording core parameters and other ancillary information that provide context to the chemical analysis data. Hardcopy lab sheets must always accompany samples sent to the lab. These are generated automatically in the Field_forms.xls spreadsheet at the very bottom of each site page and can be printed after all pertinent information has been entered into Field_forms.xls.

8.2.2. Run sheets

Run sheets summarize Lab sheet data and should be submitted electronically (see Run sheet digital summary below). Including hardcopy Run sheets is optional if digital version is e-mailed. Run sheets are completed for each shipment of samples to the lab. This may include samples from one or more locations. Field parameters for each site sampled are reported. Enter sampler's full name even though the space is too small.

Make copies of forms to take in the field for recording data. Photo copy and archive field forms before sending in with samples.

8.3. State of Utah codes

Topic	Code	Instructions
Time	Military time	
Type	4 (grab sample)	
Lab ID	78	
Agency code	04 (NPS)	
Weather Condition	01 Clear 02 Overcast 03 Partly Cloudy 04 Wind 05 Fog 06 Dust 07 Rain 08 Hail 09 Light Snow 10 Heavy Snow	
Field Conditions	01 Normal 02 Evidence of Recent High Water 03 Flood	

Topic	Code	Instructions
	04 Shore Ice 05 Anchor Ice	
Velocity		1) Record as measured (**M**) if based on gage, current meter measurement, flume or volumetric. 2) Record as estimated (**E**) if visually estimated or floating stick method. 3) Bottom of page is for flow calculations if needed 4) The state of UT suggests having the current meter calibrated once per year.
Discharge		There are three approaches to measuring flow: 1. USGS calculates discharge at each of 20 or so sub-sections measured then sums to determine total flow. NCPN suggests using this approach, with Aquacalc performing the calculations. 2. Utah method – Average of Depths x Average of velocity = Q. This method is quicker with a claimed difference of only 2%: 3. If using a USGS Gage for discharge there are two options: a. Just note "USGS" on the run sheet and lab will enter daily mean flow from USGS (This is typically several months later when USGS data is available.) b. If real time gage data is available NPS determines flow based on gage and travel time. This will be more accurate if discharge is varying. Note: it will be desirable to check the gage before heading out to sample in order to ensure it is working. If not then a discharge measurement will be necessary.
Dissolved oxygen		Record as mg/l
Chemistry	(2) = ions (3) = minerals + metals (filtered) (4) = minerals only (filtered) (6) = nutrients, NO_2+NO_3, Total P (nonfiltered) (9) = Dissolved P (filtered) Note: The "Temp, pH and Comment Fields" here are filled in by the lab on arrival	

8.4. Run sheet digital summary

Arne Hultquist, data manager for UTDWQ, strongly encourages electronic data collection in the field and transmission of field data via e-mal in the form of an Excel spreadsheet. The digital run sheet summaries are automatically generated in Field_forms.xls on worksheet page NPSetal021406. This file should be checked for

formatting. The format for time and date column A should be mmddyy hhmmss. This formatting code is found under the formatting tab 'custom' in Field_forms.xls. This page only should be saved as a *.csv formatted text file and named NPSetalmmddyy. Attach this file to an e-mail that goes to ahultquist@utah.gov.

Note: There is no electronic version of the Lab sheet that can be e-mailed. However, it is generated automatically in the Field_forms.xls spreadsheet and should be printed as hard copy to accompany bottles to the lab.

9. Mailing

1. Pre cool samples to 4 oC. Do not freeze samples.
2. Ship with ice. Do not hold or ship with "blue ice," it will not chill effectively.
    a. Water can be frozen in milk jugs or similar containers.
    b. In a 48 quart cooler, pack samples between layers of ice obtained from the maintenance yard and bagged in medium sized plastic garbage bags.
3. Enclose Lab and Run sheets in a large Ziploc baggie and place on top of ice inside the cooler.
4. Write the return address with a Sharpie on the inside of the cooler lid.
5. Wrap the cooler circumference top to bottom with packing tape.
6. From BRCA use UPS ground. It will arrive in less than 24 hours to Salt Lake City and costs less than $11.00.
7. Send samples on M, T or W only (no F or weekend arrival to lab!) to:
    UTDEQ Water Quality Laboratory
    46 North Medical Drive
    Salt Lake City, UT 84113
        i. Lab will return the cooler with a new set of sample bottles.
    a. Request a tracking number and receipt from the person who ships the cooler.
    b. On the Field_forms.xls file record
        i. Date samples shipped (mm/dd/yr)
        ii. UPS tracking #
        iii. Date samples received (mm/dd/yy)
        iv. Date data received from UT lab (mm/dd/yy)

10. Contacts

Director: Jeff Ostermiller 801- 538-6370; jostermiller@utah.gov
Data: Arne Hultquist; ahultquist@utah.gov
Laboratory: Sanwat Chandhuri
Assistant: Jack Oman; 801-584-8400 ask for "sample receiving" or "mailroom" if you need additional sample bottles

11. Appendix A: Methods of Sampling Based on Discharge

From (Wilde and Radtke 2005)

11.1. Equal-Discharge Increments (EDI)

The stream cross section is divided into increments of equal discharge. Field measurements can be made in situ at the centroid of each increment or by collecting an isokinetic depth-integrated sample at the centroid of each increment and determining the value either of each sample or of a composite of the samples. These methods result in data that are discharge-weighted.

Knowledge of streamflow distribution in the cross section is required to select verticals at which measurements will be made or subsamples collected. Streamflow distribution can be based on long-term discharge record for the site or on a discharge measurement made just prior to sample collection.

If the stream is well-mixed with relatively uniform discharge, the EDI can consist of a single vertical at the centroid of flow.

RULE OF THUMB: Divide the stream into a minimum of four increments. More increments could be needed for a stream site that is poorly mixed.

To divide the cross section into increments of equal discharge:

1. Visually inspect the stream from bank to bank, observing velocity, width, depth distribution, and the apparent distribution of sediment and aquatic biota in the cross section. Note location of stagnant water, eddies, backwater, reverse flows, areas of faster than normal flow, or obstructions.

2. If the channel and the control governing the stage are stable, historic streamflow data can be used to determine the measurement locations. If the channel is unstable or if no historic data are available, make a discharge measurement and preliminary field measurements across the selected section of channel.

    a. From the available discharge data, either

        - construct a graph using cumulative discharge or cumulative percent of total discharge plotted against the cross-sectional width, or
        - determine EDI sections directly from the discharge measurement note sheet.

    b. If profile values of pH, conductivity, temperature, and DO differ by less than 5 percent and show that the stream is well mixed both across the section and from top to bottom, a single measurement point at the centroid of flow can be used to represent field-measurement values of the cross section.

3. From the graph or measurement notes, determine the number and locations of EDIs and the centroids of those increments.

EXAMPLE:

If 5 increments will be used, select points of measurement by dividing the total stream discharge by 5 to determine increment discharge: in this case, each EDI equals 20 percent of discharge. The first vertical is located at the centroid of the initial EDI, the point where cumulative discharge equals 10 percent of the total discharge. The remaining 4 centroids are found by adding increment discharge to the discharge at the initial EDI centroid. The EDI centroids will correspond to points along the stream cross section where 10, 30, 50, 70, and 90 percent of the total discharge occur.

When making field measurements:

1. Select either the in situ or subsample method.

   - In-situ method—Go to the centroid of the first equal-discharge increment. Using submersible sensors, measure at mid-depth (or multiple depths) in the vertical. Repeat at each vertical. The value recorded at each vertical represents the median of values observed within approximately 60 seconds after sensor(s) have equilibrated with stream water.
   - Subsample method—Collect an isokinetic depth-integrated sample at the centroid of each equal-discharge increment, emptying the increment sample into a compositing device. Measure field parameters either in the sample collected at each increment or in a subsample taken from the composite of all the increment samples.

2. The final field-measurement value is the mean of the in situ or individual increment-sample value for all the EDI verticals in the section (the composite subsample yields a single value). (Note: for computation of mean pH, use the method described in SOP 8).

3. Enter data on field forms.

EXAMPLE:

Table 1 is an example of how mean conductivity measured in situ is calculated using the equal-discharge-increment method

- In the example, the correct value for the discharge-weighted mean conductivity is 163 µS/cm, calculated from 815 divided by 5 (the sum of the recorded median values divided by the number of median measurements).
- Note that at the midpoint of the center centroid of flow (increment 3) the median conductivity would have been reported as 155 µS/cm; if conductivity had been measured near the left edge of the water (increment 1), the conductivity would have been reported as 185 µS/cm.

Table 2. Example of field notes for a discharge-weighted conductivity measurement

[ft/sec, feet per second; ft, feet; ft^2 square feet; ft^3/sec, cubic feet per second; µS/cm, microsiemens per centimeter at 25 degrees Celsius; LEW, left edge of water; —, not available; REW, right edge of water]

Equal discharge increment	Percent of flow in increment	Mean velocity (ft/sec)	Width of increment (ft)	Depth of increment (ft)	Area of increment (ft^2)	Increment discharge (ft^3/sec)	Median conductivity (µS/cm)
LEW	0	—	—	—	—	—	—
1	20	2.0	22	5.7	125	250	185
2	20	2.2	11	10.4	114	250	170
3	20	2.3	9	12.0	109	250	155
4	20	3.9	5	12.8	64	250	155
5	20	3.4	10	7.4	74	250	150
REW	0	—	—	—	—	—	—

Calculation of conductivity: mean of median conductivity measurements

$(815 / 5) = 163 \mu Scm$

11.2. Equal-width increments (EWI)

The stream cross section is divided into increments of equal width. Knowledge of the streamflow distribution in the cross section is not required.

In situ field measurements are made at the midpoints of each increment. Area-weighted concentrations can be computed from these measurements (Table 2).

Subsample field measurements are made in discrete samples that usually are withdrawn from a composite sample collected using an isokinetic sample and isokinetic depth-integrating method. The volume of the isokinetic sample must be proportional to the amount of discharge in each increment and measurements in subsamples taken from the compositing device result in discharge-weighted values.

To divide the cross section into increments of equal width:

1. Visually inspect the stream from bank to bank, observing velocity, width, depth distribution, and the apparent distribution of sediment and aquatic biota in the cross section. Note location of stagnant water, eddies, backwater, areas of faster than normal flow, and piers or other obstructions.

2. Determine stream width using a tagline or from station markings on bridge railings or cableways.

3. Divide the section into equal-width increments based on flow and stream-channel characteristics along the cross section, field-measurement variability from the cross-section profile, and data objectives for the study. This interval width will govern the number of verticals used, and applies also to streams in which flow is divided (for instance, in a braided channel).

RULE OF THUMB:

- In streams 5-ft wide or greater, a minimum of 10 equal-width increments is generally recommended.
- In streams less than 5-ft wide, use as many increments as practical, but equally spaced a minimum of 3 inches apart.

4. Locate the midpoint of the first vertical at a distance of one-half of the selected increment width from edge of water. Locate other measurement verticals at the centers of the remaining increments.

EXAMPLE:

In a stream 60-ft wide that has been divided into 15 increments of 4 ft each, the first measurement vertical would be 2 ft from water's edge and subsequent verticals would be at 6, 10, 14 ft, and so forth, from the starting point at water's edge.

When making field measurements:

1. Select either the in situ or subsample method.

- In situ method—Measure at the midpoint of each equal-width increment. Using submersible sensors, measure at mid-depth in the vertical.
- Subsample method—Collect an isokinetic depth-integrated sample at the midpoint of each equal-width increment, emptying each sample into a compositing device. Use of the correct sampling equipment is critical to execute this method successfully: standard samplers cannot meet isokinetic requirements when stream velocity is less than 1.5 ft/sec.

2. Record a value for each field measurement at each vertical. The value recorded represents the stabilized values observed within approximately 60 seconds after the sensor(s) have equilibrated with the stream or subsample water.

EXAMPLE:

Table 2 provides an example of an area-weighted median measurement for conductivity measured in situ.

In the example, the area-weighted median conductivity equals 130 µS/cm.

To calculate an area-weighted median, multiply the area of each increment by its corresponding field measurement, sum the products of all the increments, and divide by total cross-sectional area.

Note that if the conductivity reported were selected at mid-depth of the vertical of centroid of flow (section 10), it would have been reported as 125 µS/cm; if the conductivity reported were near the left edge of water, it would have been reported as 150 µS/cm.

The final field-measurement value normally is calculated as the mean of the values recorded at all EWI increments, resulting in an area-weighted mean. (Note: for pH, calculate the mean using the method described in SOP 8).

Alternatively for EWI, if the area-weighted median best represents integrated stream chemistry, you can report the median instead of the mean—but, be sure to document this on the field form and in the final data report .

Table 3. Example of field notes for an area-weighted conductivity measurement

[ft, feet; LEW, left edge of water; ft^2, square feet; µS/cm, microsiemens per centimeter at 25 degrees Celsius;—, not available; REW, right edge of water]

Section number	Cumulative percent of flow in section	Vertical location from LEW (ft)	Width of section (ft)	Depth of vertical (ft)	Area of section(ft^2)	Median conduc-tivity (µS/cm)	Product of median conduc-tivity and area
LEW	0	0	—	—	—	—	—
1	2	2	4	1.0	4.0	150	600
2	4	6	4	2.0	8.0	145	1,160
3	6	10	4	2.6	10.4	145	1,508
4	10	14	4	3.2	12.8	140	1,792
5	16	18	4	3.5	14.0	135	1,890
6	22	22	4	4.0	16.0	130	2,080
7	28	26	4	4.5	18.0	130	2,340
8	34	30	4	5.4	21.6	125	2,700
9	42	34	4	6.0	24.0	125	3,000
10	50	38	4	5.7	22.8	125	2,850
11	62	42	4	5.1	20.4	125	2,550
12	76	46	4	4.6	18.4	125	2,300
13	88	50	4	3.5	14.0	125	1,750
14	96	54	4	1.4	5.6	135	756
15	99	58	4	1.0	4.0	140	560
REW	100	60	—	—	—	—	—

Calculation of conductivity: sum of values in last column divided by the total cross-sectional area

(27836 / 214) = 130 µ*S/cm*

When making field measurements:

1. Select either the in situ or subsample method and follow the instructions in 6.0.2.B.
    - In-situ method—Go to the centroid of the first equal-discharge increment. Using submersible sensors, measure at mid-depth (or multiple depths) in the vertical. Repeat at each vertical. The value recorded at each vertical represents the median of values observed within approximately 60 seconds after sensor(s) have equilibrated with stream water.
    - Subsample method—Collect an isokinetic depth-integrated sample at the centroid of each equal-discharge increment, emptying the increment sample into a compositing device. Measure field parameters either in the sample collected at each increment or in a subsample taken from the composite of all the increment samples.
2. The final field-measurement value is the mean of the in situ or individual increment-sample value for all the EDI verticals in the section (the composite subsample yields a single value). (Note: for computation of mean pH, use the method described in section 6.4.3.A).
3. Enter data on field forms

NOTE: The field measurement profile is a useful guideline, but the sample size determined at the time of cross section sampling may not provide a good measure of central tendency in extreme (low) flow conditions.

TECHNICAL NOTE: The preferred sampling method and number of verticals to be sampled within the stream cross section that are needed to obtain a sample that is representative depends on stream homogeneity as indicated by the field measurement profile and stream-discharge or other data, as well as by study objectives. Note that the field-measurement profile is a useful guideline, but might not be representative of chemical homogeneity because it will not be possible to make a preliminary profile for chemical constituents that require laboratory analysis. Also, it might be desirable to move to a sampling site upstream or downstream to adjust for seasonal variation or extreme flow conditions.

Guidelines used for selecting sampling sites on ephemeral and intermittent streams are the same as those for perennial streams. Ephemeral and intermittent stream sites need additional planning and examination to account for unstable channel form and conditions related to rapidly changing stage and discharge that can occur as a result of flash flooding.

12. Appendix B: NCPN Custom Data Forms

12.1. Monthly Lab Sheets

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 1

STORET: 4951855 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: Sheep Ck bl Spring in Bryce Cyn NP

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____
DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____
VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 2

STORET: 4951857 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: Yellow Ck bl Spring in Bryce Cyn NP

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 3

STORET: 4954812 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: Halls Ck Bl Narrows

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED :	[ ] MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 4

STORET: 4937985 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: Green R ab Gates of Ladore

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED NUTRIENT : (9) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (2) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 5

STORET: 4937940 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: JONES HOLE CK BL FISH HATCHERY

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED NUTRIENT : (9) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (2) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 6

STORET: 4951260 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: North Fk Virgin R at North Fork Rd

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____
VELOCITY	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 7

STORET: 4950800 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: La Verkin Ck at Lee Pass Trail

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 8

STORET: 4950920 LAB ID: [78] TYPE: [4] PROJECT: 358

DESCRIPTION: North Ck at Planned Gage in Park

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (4) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____
DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____
VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

12.2. Quarterly Lab Sheets

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 1

STORET: 4951855 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: Sheep Ck bl Spring in Bryce Cyn NP

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 2

STORET: 4951857 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: Yellow Ck bl Spring in Bryce Cyn NP

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH	_____									
DEPTH	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____
VELOCITY	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 3

STORET: 4954812 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: Halls Ck Bl Narrows

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH	_____									
DEPTH	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____
VELOCITY	_____	_____	_____	_____	_____	_____	_____	_____	_____	_____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 4

STORET: 4937985 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: Green R ab Gates of Ladore

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] /[ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] /[ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

BACTERIAL : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED NUTRIENT : (9) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (2) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____
DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____
VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 5

STORET: 4937940 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: JONES HOLE CK BL FISH HATCHERY

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] /[ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] /[ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED NUTRIENT : (9) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (2) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 6

STORET: 4951260 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: North Fk Virgin R at North Fork Rd

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 7

STORET: 4950800 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: La Verkin Ck at Lee Pass Trail

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] / [ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

UTAH STATE WATER QUALITY SYSTEM
MONITORING RUN PROGRAM

MONITORING RUN: NPS ZION ET AL QUARTERLY

TRIP ID: NPSETAL RUN SEQUENCE NUMBER: 8

STORET: 4950920 LAB ID: [79] TYPE: [4] PROJECT: 358

DESCRIPTION: North Ck at Planned Gage in Park

COLLECTOR : [ ] [ ] [ ] [ ] AGENCY: [ ] [ ]

DATE: [ ] [ ] [ ] [ ] [ ] [ ] TIME: [ ] [ ] [ ] [ ]
Y Y M M D D

WEATHER CONDITIONS: [ ] [ ] / [ ] [ ] /[ ] [ ]

FIELD CONDITIONS: [ ] [ ] / [ ] [ ] /[ ] [ ]

FIELD TESTS

AIR TEMP. (CELCIUS):	[ ] [ ] . [ ]	TRANSPARENCY (METERS):	[ ] [ ] . [ ]
TEMPERATURE (CELCIUS):	[ ] [ ] . [ ]	Cl. RESID :	[ ] . [ ] [ ]
pH :	[ ] [ ] . [ ]	TURBIDITY (NTU) :	[ ] [ ] . [ ]
SP COND UMHOS/CM	[ ] [ ] [ ] [ ] [ ]	FLOW (MGD) :	[ ] [ ] [ ] . [ ]
SALINITY PPM:	[ ] [ ] [ ] [ ] . [ ] [ ]	FLOW (GPM) :	[ ] . [ ] [ ]
% D.O. SATURATION :	[ ] [ ] [ ] . [ ]	FLOW (CFS) :	[ ] [ ] [ ] [ ] [ ] [ ] [ ] . [ ]
D.O. :	[ ] [ ] . [ ]	FLOW ESTIMATED : [ ]	MEASURED : [ ]
DEPTH : (METERS)	[ ] [ ] [ ] . [ ]		

CHEMISTRY : (2) TEMP: ____ pH: ____ COMMENTS: _____

FILTERED METALS : (3) TEMP: ____ pH: ____ COMMENTS: _____

NON-FILTERED NUTRIENT : (6) TEMP: ____ pH: ____ COMMENTS: _____

FIELD COMMENTS: DETERMINE THE FLOW

_____

WEIR TYPE	_____	PIPE DIA	_____	BUCKET SIZE	_____
WIDTH	_____	DEPTH	_____	SECONDS	_____
DEPTH	_____	VELOCITY	_____		

STR WIDTH _____

DEPTH _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

VELOCITY _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

12.3. Monthly Run Sheets

SAMPLE RUN: NPS ZION ET AL

SAMPLE SUMMARY

DATE : ___/___/___ SAMPLERS : [ ] [ ] [ ]

TRIP ID : NPSETAL

DESCRIPTION : THIS IS THE NON QUARTERLY RUN

CHEMISTRY	=	8	FILTERED METALS	=	8	FILTERED NUTRIEN	= 2
NON-FILTERED NUTRI	=	8		=			=

Seq. #	Project	Storet Id.	Station Desc.	Date	Time	W Temp	pH	DO	Conductivity	Flow	E/M
1	358	4951855	Sheep Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
2	358	4951857	Yellow Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
3	358	4954812	Halls Ck Bl Narrows	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
4	358	4937985	Green R ab Gates of Ladore	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
5	358	4937940	JONES HOLE CK BL FISH HATCHERY	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
6	358	4951260	North Fk Virgin R at North Fork Rd	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
7	358	4950800	La Verkin Ck at Lee Pass Trail	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
8	358	4950920	North Ck at Planned Gage in Park	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]

12.4. Quarterly Run Sheets

SAMPLE RUN: NPS ZION ET AL QUARTERLY

SAMPLE SUMMARY

DATE : ___/___/___ SAMPLERS : [ ] [ ] [ ]

TRIP ID : NPSETAL

DESCRIPTION : THESE SHEETS ARE FOR QUARTERLY TRIPS

BACTERIAL	=	1	CHEMISTRY	=	7	FILTERED METALS	= 8
FILTERED NUTRIENT	=	2	NON-FILTERED NUT	=	8		=

Seq. #	Project	Storet Id.	Station Desc.	Date	Time	W Temp	pH	DO	Conductivity	Flow	E/M
1	358	4951855	Sheep Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
2	358	4951857	Yellow Ck bl Spring in Bryce Cyn NP	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
3	358	4954812	Halls Ck Bl Narrows	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
4	358	4937985	Green R ab Gates of Ladore	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
5	358	4937940	JONES HOLE CK BL FISH HATCHERY	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
6	358	4951260	North Fk Virgin R at North Fork Rd	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
7	358	4950800	La Verkin Ck at Lee Pass Trail	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]
8	358	4950920	North Ck at Planned Gage in Park	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]	[ ]

13. Appendix C: Utah Excel Spread sheet instructions

Instructions for Data Entry into Water Quality Spreadsheet

1. Make a working copy of the water quality spreadsheet on the floppy disk. Choose any easy-to -remember name. Don't make changes to the original file.
2. Enter the date and time the sample was taken in column A (labeled "Annotation") in the following format:
   MMDDYY HHMMSS
   Make sure there is one space between the year and the hours. The date and time is automatically copied to the appropriate column. The time portion of this entry is also automatically copied to the appropriate column.
3. Enter the seven digit storet number in column B (labeled "station id").
4. Enter the three digit cost code in column C (labeled "program"). Omit any letters which appear in the cost code.
5. Enter the one or two digit sample type, omitting any leading zeros, in column D (labeled "activity").
6. Enter the one or two digit agency code in column E (labeled "org code").
7. Enter up to four weather condition codes in columns F through I (labeled "weath"). Leading zeros may be omitted. Leave unused weather condition code cells blank. Do not repeat the same weather condition code.
8. Enter up to four field condition codes in columns J through M (labeled "field"). Leading zeros may be omitted. Leave unused field condition code cells blank. Do not repeat the same field condition code.
9. Enter any comment, e.g. "dead cow in stream", in column N (labeled "comment").
10. Enter the numeric value of the flow in column P (labeled "flow"). Leave this blank if the flow was not measured or estimated.
11. Enter a single upper case letter to indicate type of units in column Q (labeled "units"). C indicates cubic feet per second, G indicates gallons per minute, and M indicates million gallons per day. Do not use any other units. Leave this blank if the flow was not measured or estimated.
12. Enter either an upper case M to indicate a measured flow value or an upper case E to indicate an estimated flow value in column R (labeled "type"). Leave this blank if the flow was not measured or estimated.
13. Enter the average velocity of the flow in feet per second in column S (labeled "velocity"). Leave this blank if the velocity was not measured or estimated.
14. Enter either an upper case M to indicate a measured velocity value or an upper case E to indicate an estimated velocity value in column T (labeled "type"). Leave this blank if the velocity was not measured or estimated.
15. Enter the numeric value of the residual chlorine in column U (labeled "res cl"). Leave this blank if the residual chlorine was not measured.
16. Enter the numeric value of the water temperature (degrees Celsius) in column X (labeled "temp"). Leave this blank if the water temperature was not measured.
17. Enter the numeric value of the pH in column Y (labeled "pH"). Leave this blank if the pH was not measured.

18. Enter the numeric value of the specific conductivity (microSiemens per centimeter) in column Z (labeled “sp cond”). Leave this blank if the specific conductivity was not measured.
19. Enter the Salinity values in column AA. Leave this blank if the salinity was not measured.
19. Enter the numeric value of the %DO saturation in column AB (labeled “%DO Saturation”). Leave this blank if the %DO Saturation was not measured
20. Enter the numeric value of the dissolved oxygen (milligrams per liter) in column AC (labeled “DO”). Leave this blank if the dissolved oxygen was not measured.
22. LAKE SAMPLES ONLY. Enter the depth the sample was TAKEN in column AD. Leave blank if this is a stream sample
23. LAKE SAMPLES ONLY. Enter the numeric value of the secchi disk reading (meters) in column AE (labeled “transparency”). Leave this blank if a secchi disk reading was not taken.
24. Save the File with the appropriate Trip ID.

13.1. Example hard copy of UT Excel spreadsheet

A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
Annotation	**station_id**	**Project**	**activity**	**org_code**	**weath1**	**weath2**	**weath3**	**weath4**	**field1**	**field2**	**field3**	**field4**	**comments**	**Annotation**
This is an example line														
120604 132500 :	4954430	350	4	13	3	4			1					

R	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF
utype	**velocity**	**vtype**	**res_cl**	**aDate**	**atime**	**temp**	**pH**	**sp_cond**	**Salinity**	**percentDO**	**DisOxy**	**depth**	**sechhi**	**Turbidity**
M						9.0	8.0	1400	1.2	85.8	7.8			

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #11

Fecal Indicator Bacteria, All Units

Version 1.00 (10/30/06)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

This Water Quality Monitoring Standard Operating Procedure (SOP) describes sample collection, handling and processing for the enumeration of Fecal Indicator Bacteria in natural waters, specifically the historic method of fecal coliform bacteria in Utah units and now the IDEXX method for *E. coli.* The reason for covering two techniques for Utah units is because the state is currently changing from fecal coliform testing to *E. Coli*.

It also covers the membrane filter technique for *E. coli* used in Colorado units.

Specific step-by-step procedures for Utah units are adopted from Standard Methods for the Examination of Water and Wastewater (APHA, 1998) Section 9000, and for Colorado units from EPA Method 1604: Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium). Copies of both references are reproduced in Appendix A and B respectively.

The remainder of this SOP (pages 4 – 11) supplements the APHA, EPA and IDEXX methods with background and approaches to sample collection, transport and processing that are specific to NCPN monitoring sites.

Table of Contents

List of Tables **3**
List of Acronyms and Abbreviations Used **4**
1. Introduction **5**
2. Procedures Used in Park Units **5**
3. Supplements to Published Filtration Methods **9**
3.1. Field preparations 9
3.1.1. Things to bring into the field *9*
3.1.2. To do prior to sampling *9*
3.1.3. Record on field/lab data sheet *9*
3.1.4. Label sample bottle/whirl pack with: *9*
3.2. Sample collection 9
3.3. Sample preservation and storage 10
3.4. Sample filtration and incubation 10
3.5. Quantity filtered 10
4. Supplements to IDEXX Quantitray/2000 Method **12**
4.1. Field preparations 12
4.1.1. Things to bring into the field *12*
4.1.2. To do prior to sampling *12*
4.1.3. Record on Field_forms.xls data sheet *12*
4.1.4. Label sample bottle with: *12*
4.2. Sample collection: Quantitray/2000 method 12
4.3. The Quantitray/2000 methodology 12
4.4. Computing reported values: Quantitray/2000 method 13
4.4.1. Total coliforms *13*
4.4.2. E. coli. *13*
5. Computing Reported Values – Filtration method **13**
5.1. Example computations 15
6. Criteria and Actions **16**
6.1. Numeric criteria 16
7. Safety and Disposal of Bacteria Cultures **16**
8. References **17**
9. Appendix A. Standard Methods for the Examination of Water and Wastewater, Applicable Portions of Section 9000. ..... **18**
10. Appendix B. EPA Method 1604: Total Coliforms and Escherichia coli in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium) **52**

List of Tables

Table 1. Pertinent sections of Standard Methods for the Examination of Water and Wastewater 8
Table 2. Location and method of sample collection for bacterial analysis. 10
Table 3. Guide for the volume to filter for Fecal Coliform or E coli membrane filter analysis 11
Table 4. Methods of computation of reported values for Fecal Indicator Bacteria. 15
Table 5. Bacteria criteria by designated beneficial use for UT and CO water bodies. 16

List of Acronyms and Abbreviations Used

AZ	Arizona
APHA	American Public Health Association
CDPHE	Colorado Department of Public Health and Environment
cfs	cubic feet per second
cfu	colony forming unit
CO	Colorado
EPA	United States Environmental Protection Agency
FC	Fecal Coliform Bacteria
FIB	Fecal Indicator Bacteria
mg/L	milligrams per liter
NPS	National Park Service
ONRW	Outstanding Natural Resource Waters
Q	Discharge (in cubic feet per second)
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
SOP	Standard Operating Procedure
STORET	USEPA Storage and Retrieval System database
TNTC	To Numerous to Count
µg/L	micrograms per liter
UT	Utah
UDEQ	Utah Division of Environmental Quality
UDWQ	Utah Division of Water Quality
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
USGS-NFM	USGS National Field Manual for Collection of Water-Quality Data
WRD	National Park Service Water Resources Division

1. Introduction

The purpose of monitoring fecal indicator bacteria is to provide an indication of the risk to recreational users of contracting enteric diseases after coming into contact with park waters through immersion or full-body contact recreation like swimming, but was based on the probability of an incidental ingestion of the water during those activities. Sampling frequency is at a screening level (monthly) at NCPN water quality monitoring sites where visitors use the water as a source of drinking water or for full– or partial-body contact recreation. This sample frequency is adequate as a general indicator of the relative magnitude of contamination, but is not adequate for enforcement level monitoring, such as for swimming beaches where a minimum of 5 samples/month are required. For this reason, the results will be used to inform the NPS and visitors about general conditions, but not to take actions, such as a closure, in response to the results of individual samples.

Actual legal enforcement of the recreational bacterial standard would rest with the state or with EPA if the state failed to take action. The NPS does not have authority to do so. The NPS has authorities for visitor protection to issue advisories and to restrict access if necessary.

The procedures differ slightly between units in Colorado and in Utah because the current standards are for *E. coli* in the former and for Fecal Coliform in Utah. The state of Utah proposes to change to *E. coli* in the near future, hence the addition of procedures to enumerate *E. coli* using the IDEXX method that does not require filtering.

In summary, the filtration methods used consist of collection of a water sample in a sterile container, filtering that sample through a membrane filter with a pore size of 0.49µm that traps bacteria or groups of bacteria on the surface. The filter is placed on a nutrient media and incubated for 24 hours, at which time the bacteria have grown into colonies that are large enough to be counted. Positive colonies have the ability to grow on the selected media at the incubation temperature to produce a distinctive appearance. They are counted and the results reported as colony-forming units (cfu) per 100 ml of sample.

2. Procedures Used in Park Units

The fecal coliform procedures used in **Utah** units are presented in Standard Methods for the Examination of Water and Wastewater (APHA, 1998) with applicable sections reproduced in Appendix A. A guide to the specific sections used is presented in Table SOP11-1. As a secondary reference, the USGS National Field Manual, Book 9 Section 7.1 (USGS-NFM, various dates), can be used (see Appendix B). While it is entirely consistent with Standard Methods, some useful points of clarification have been added. Although Utah is moving away from filtration methods to the IDEXX *E. Coli.* method of Most Probable Number (MPN) the older techniques are retained here to aid interpretation of historic data.

The *E. coli* method called Quantitray-2000 used in **Utah** units is presented in a video on the IDEXX web site. **http://www.idexx.com/water/quantitray/quantitray2000300_en.jsp**

The field methodology is entirely consistent with the video explanation but a few useful points are mentioned in the text below.

The procedures used in **Colorado** units are found in EPA Method 1604: Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium). A copy is presented in Appendix C, and a guide to the specific sections used is presented in

Table 1.

Table 1. Pertinent sections of standard methods for the examination of water and wastewater (APHA 1998).

	Primary Reference for Procedures		Secondary Reference
	Utah Units	**Colorado Units**	**All Units**
	Standard Methods for the Examination of Water and Wastewater (APHA, 1998) Reproduced in Appendix A	EPA Method 1604: Total Coliforms and *Escherichia coli* in Water by Membrane Filtration (MI Medium) Reproduced in Appendix B	USGS National Field Manual, Book 9 Section 7.1 (USGS-NFM, various dates)
Activity	**Section(s) applicable to NCPN Activities**		
Equipment specifications	9030 Laboratory Apparatus 9030 B. Equipment Specifications	6.0 Equipment and Supplies 9.0 Calibration and Standardization	7.1.1 Equipment and Equipment Sterilization Procedures
Sample Collection	9060 Samples 1. Containers 2. Dechlorination 3. Sampling Procedures	8.0 Sample Collection, Preservation and Storage	7.1.2 Sample Collection, Preservation and Storage 7.1.2.A Surface-Water Sample Collection
Sample Preservation and Storage	9060 B. Preservation and Storage 1. Holding time and Temperature	8.2.1 Storage Temperature and Handling Conditions 8.2.2 Holding time Limitations	7.1.2.C Sample Preservation and Storage
Preparation of Buffered Dilution Water	9050 C. Media Specifications 1. Dilution Water	7.0 Reagents and Standards	7.1.3.A Preparation of Media and Reagents
Preparation of M-FC Media or MI Agar	9050 Preparation of Culture Media 9222 D. 1. Materials and Culture Medium for M-FC medium	7.5 MI Agar	7.1.3.A Preparation of Media and Reagents 7.1.5 Instructions for Media Preparation
Membrane Filter Procedure	9222 Membrane Filter Technique for Members of the Coliform Group, and 9222 D. Fecal Coliform Membrane Filter Procedure 2. Procedure	11.0 Procedure	7.1.3.C Membrane Filtration Procedure
Incubation	9222 D. Fecal Coliform Membrane Filter Procedure d. Incubation	Step 11.10	7.1.3.C Membrane Filtration Procedure, Step 14
Enumeration	9222 D. Fecal Coliform Membrane Filter Procedure e. counting f. verification	Step 11.11, 11.12 and 11.13	7.1.3.C Membrane Filtration Procedure, Steps 15 through 18
Calculation of Bacteria Density	9222 D. 3. Calculation of Fecal Coliform Density	12.0 Data Analysis and Calculations	7.1.4 Calculation and Reporting of fecal Indicator Bacteria

3. Supplements to Published Filtration Methods

The remainder of this section presents information that supplements the referenced procedures with actions specific to NCPN parks and applies to **filtration** methods used in CO and UT park units.

3.1. Field preparations

3.1.1. Things to bring into the field

Field data sheets (sufficient for planned sampling + extras)
Turbidimeter
Sample bottles or Whirl Packs (sufficient for planned sampling + extras)
Pens
Sharpies
Cooler and ice
Thermometer
Watch
Sodium Thiosulfate (if any sites have residual chlorine)

3.1.2. To do prior to sampling

Obtain Ice
Inventory and stock supplies.

3.1.3. Record on field/lab data sheet

Name of sample collector
Time of sample collection
Time filtered
Quantity collected
Quantity filtered
Counts for each plate
Reported value
Observations

3.1.4. Label sample bottle/whirl pack with:

Site Name
Name of sample collector
Date of collection
Time of collection

3.2. Sample collection

Preferences for the location of sample collection are presented in Table 2, along with a summary of the sample collection process.

Table 2. Location and method of sample collection for bacterial analysis.

Site Type	Locations(s) of Sample Collection	Method of Sample Collection
Large Rivers	1. Mid Channel 2. Near bank if mid channel is not accessible	Sampling procedures are described in detail in Sections 9060A and 9060B of the 20th edition of Standard Methods for the Examination of Water and Wastewater, or in the USEPA Microbiology Methods Manual, Section II, A.
Small Streams	1. Mid Channel 2. Near bank if flooding precludes mid channel sample	Water samples are collected in sterile polypropylene sample containers (bottle or whirlpack®) with leak-proof closures.
Springs and Seeps	In Order of Preference 1. At discrete point of discharge 2. Flowing water downstream of discharge point 3. Standing water downstream of discharge point	Samples are collected by immersing uncapped sample bottle to a depth of 0.3 m in a location upstream of the person collecting the sample and away from any sediment plume created by the sampler. Contact with the sterile inner surface of the sample bottle or cap is avoided. Examine the sample. If it contains large sticks, leaves or other detritus, discard the sample and collect another using the same bottle. (Objects will interfere with counting the bacteria colonies.)
Lake	Epilimnion, at a depth of 1 meter	Residual chlorine in drinking water (or chlorinated effluent) samples should be neutralized with sodium thiosulfate (1 ml of a 10% solution per liter of water) at the time of collection.1 The sample bottle is capped and placed on ice as soon as practical after sample collection.

[1] Neutralization of chlorine residual is necessary only in waters where chlorine residual is present due to discharge of treated wastewater. This does not occur at any current NCPN sites.

3.3. Sample preservation and storage

Samples will be placed on ice and filtered within 6 hours as provided for in the detailed methodologies. For sites accessed by hiking, sample collection will be the last activity conducted, and the sample will be wrapped in insulating material to shield it from sunlight and heat and transported as quickly as possible to a vehicle where it can be placed on ice.

3.4. Sample filtration and incubation

Sample filtration and incubation will take place at park laboratories and be conducted by park water quality monitoring staff.

3.5. Quantity filtered

With both the MF-C method and EPA Method 1064 , the volume of sample filtered must be adjusted to achieve a countable filter. Filters with more than 60 colonies for M-FC media or 80 colonies for MI agar are difficult to count and inaccuracies are introduced when colonies overlap. So where large numbers of bacteria are expected, the volume filtered is reduced to achieve a countable plate. When a large range of bacteria numbers is possible two or more sample volumes can be filtered in order to increase the chance of a countable plate. Suggested volumes for filtration are presented in Table 3.

Table 3. Guide for the volume to filter for Fecal Coliform or E coli membrane filter analysis. This is only an initial guide and the amount of samples filtered should be adjusted based on experience with results from the specific sampling site.

		Water Condition	Clear		Moderately Turbid		Very Turbid	
		Contamination Sources[1]	Few	Many	Few	Many	Few	Many
Water Source Being Sampled	Rivers	Suggested Volumes to Filter	100ml 50ml	50ml 10ml 5ml	50ml 10ml 5ml	50ml 10ml 5ml	10ml 5ml 1ml	5ml 1ml .5ml
		Range of Coverage for FC2	20 - 120	40–1,200	40–1,200	40–1,200	200–6,000	400–12,000
		Range Of Coverage E. coli3	20 - 160	40–1,600	40–1,600	40–1,600	200–8,000	400-16,000
		Volume of Sample to be Collected	200ml	100ml	100ml	100ml	100ml	100ml
	Isolated Springs or Small Streams with pools	Suggested Volumes to Filter	100ml	100ml 50ml	100ml 50ml 10ml	50ml 10ml 5ml	10ml 5ml 1ml	5ml 1ml .5ml
		Range of Coverage for FC2	20 - 60	20 - 120	20 - 600	40–1,200	200–6,000	400–12,000
		Range Of Coverage E. coli3	20 - 80	20 - 160	20 – 800	40–1,600	200–8,000	400-16,000
		Volume of Sample to be Collected	100ml	200ml	200ml	100ml	100ml	100ml
	Lakes	Suggested Volumes to Filter	100ml 50ml	100ml 50ml	100ml 50ml	100ml 50ml 10ml	-	-
		Range of Coverage for FC2	20 - 120	20 - 120	20 - 120	20 - 600		
		Range Of Coverage E. coli3	20 - 160	20 - 160	20 - 160	20 – 800		
		Volume of Sample to be Collected	200ml	200ml	200ml	200ml		

[1] Contamination sources include municipal sewage discharge, livestock use, human recreational use and wildlife.

[2] The range of coverage is the reportable range of values if the fecal coliform plate is assumed to have the ideal colony count of 20 – 60 colonies per plate.

[3] For E. coli MF-MI technique the ideal plate count is 20-80 colonies and countable up to 200 colonies.

4. Supplements to IDEXX Quantitray/2000 Method

This section presents information that supplements the referenced procedures with actions specific to NCPN parks and applies to **IDEXX** methods used in **Utah** park units.

4.1. Field preparations

4.1.1. Things to bring into the field

Field data sheets
Sample bottles containing Sodium Thiosulfate – to mitigate chlorine if present
Water bottle parka, 1 L Nalgene with ice. This will last up to 8 hours even in summer if kept in shade
Sealer with power supply / inverter
Sample bottles
Quantitrays
Colilert reagent
Portable incubator and power supplies.

4.1.2. To do prior to sampling

Obtain Ice
Inventory and stock supplies.

4.1.3. Record on Field_forms.xls data sheet

Date and time of sample collection
Date and time of start incubation
Duration sample held prior to incubation
Was sample kept < 10 deg C from time of collection
Date and time plate counts read
Number of wells large and small (+) wells under ambient and UV light
Most Probable Number (MPN) from look up table

4.1.4. Label sample bottle with:

Site Name
Date of collection
Time of collection

4.2. Sample collection: Quantitray/2000 method

Preferences for the location of sample collection are presented in Table 11-2.

4.3. The Quantitray/2000 methodology

presented in an internet video at:
http://www.idexx.com/water/quantitray/quantitray2000300_en.jsp

The basic steps are as follows.

- Record date and location on sample bottle
- Record date, time on field form

- Collect 100 mL in sample bottle with a gloved hand
- Place sample bottle in 1 L Nalgene bottle packed in ice and insulated with water bottle parka for hike to car
- At the car fire up the Quantitray sealer and incubator and allow each to warm up
- Remove sample from ice and add Colilert reagent and shake gently until dissolved
- Label a Quantitray 2000 plate with date location and start time of incubation
- Pour sample into Quantitray and run through sealer
- Place sealed quantitray in incubator for 24 hours before reading

4.4. Computing reported values: Quantitray/2000 method

4.4.1. Total coliforms

Count large and small wells that have (+) reaction under ambient light. Use these values to look up MPN for Total Coliforms. (+) wells are more yellow than the comparator. Note: the look up function is built into Field_forms.xls

4.4.2. E. coli.

Count large and small wells that have (+) reaction under ultra violet light. Use these values to look up MPN for *E. Coli*. (+) wells are more yellow than the comparator. Note: the look up function is built into Field_forms.xls

5. Computing Reported Values – Filtration method

(see APHA, 1998 section 9222 D.3 or NFM section 7.1.4)

Computation of bacterial density (FC or E. coli) is made using the count of colonies on each plate. It is based on the assumption that 1 bacteria or group of bacteria, together called a "colony forming unit" (cfu), will grow into a countable colony. The computation is based on the number of typical colonies counted on ideal plates, that is, those with 20 to 60 positive colonies. Plates with more colonies tend to exhibit overlap or coalescing of colonies resulting in counting errors, and those with fewer represent a poor sample of occurrence. Plates with counts outside the ideal range can be used if no ideal plates exist, so long as the total number of colonies on the plate (including both typical FC or E coli colonies and extraneous colonies) does not exceed 200. When the total number of colonies exceeds 200, or there are fewer colonies but they cannot be counted due to overlap or coalescing, that plate is counted as "to numerous to count", or TNTC. Methods of computation are presented in

Table 4.

Table 4. Methods of computation of reported values for Fecal Indicator Bacteria.

Number of Plates in Ideal Range (20-60 colonies for both FC and 20-80 colonies for E. coli)	Plates outside the Ideal Range are:	Compute Reported Value by: (all values will be adjusted to 100ml of sample)
1	Irrelevant	Use the 1 ideal Plate
2	Irrelevant	Total Colonies on the 2 ideal plates
3	Irrelevant	Total Colonies on the 3 ideal plates
0	All TNTC (>200)	Report as >60 (FC) or >80 (E. coli) (the upper end of the ideal range) adjusted for smallest amount filtered. (See example 3 below)
0	1 or more plates to high but not TNTC	Compute from the countable plate(s)
0	Some plates >60 and some <20, but not TNTC	Compute from all the countable plates
0	1 or more plates too low, but greater than zero	Compute from the countable plate(s)
0	All plates zero	Report as < 1 cfu/100ml adjusted for the largest amount filtered.

5.1. Example computations

Compute the count using the following equation:

$$cfu\ /100\,ml = \frac{total\ colonies\ counted\ \ x100}{total\ \ ml\ sample\ \ filtered}$$

Example 1: 100 ml are filtered resulting in a count of 45 colonies, and 50 ml are filtered resulting in a count of 28 colonies, 10ml filtered resulting in 7 colonies. Use only the ideal 100ml and 50ml plates to compute.

$$\frac{\left[(45+28)\times 100\right]}{(100+50)ml} = 49\,cfu/100ml$$

Example 2: 100 ml and 50 ml filtered are both TNTC and the 10 ml plate has 89 colonies. No plates are ideal, so use the only countable plate.

$$\frac{\left[(89)\times 100\right]}{10ml} = 890\,cfu/100ml$$

Example 3: Three plates are filtered for 1, 5 and 10 ml and all are TNTC. Compute using the 1ml plate and report on >60 colonies.

$$\frac{\left[(>60)\times 100\right]}{1ml} => 6{,}000\,cfu/100ml$$

Example 4: Three plates are filtered for 5, 10 and 50ml and all are zero. Compute using the plate with the largest volume filtered.

$$\frac{[(<1)\times 100]}{50ml} =< 2cfu/100ml$$

The colony count data are recorded on the hard copy field form and are entered into the digital data base with other field data.

6. Criteria and Actions

6.1. Numeric criteria

Acute and chronic exceedance criteria serve as a guide for informing park resource management staff of the severity of contamination (Table 5).

Table 5. Bacteria criteria by designated beneficial use for UT and CO water bodies.

			----------Beneficial Use----------		
State	**Parameter**	**Domestic source**	**Recreation and Aesthetics**	**Recreation and Aesthetics**	**Agriculture**
		1C	2A	2B	4
			----------(No.)/100 ML----------		
UT	E. coli: 0-Day Geometric mean	206	126	206	
UT	E.coli: Maximum	940	576	940	
			----------Beneficial Use----------		
		CLASS 1a Existing Primary Contact	CLASS 1b Potential Primary Contact	CLASS Secondary Contact	Domestic Water Supply
			----------(No.)/100 ML----------		
CO	Fecal Coliforms: 0-Day Geometric mean	200	325	2000	2000
CO	E. coli	126	205	630	630

7. Safety and Disposal of Bacteria Cultures

It is important to remember that by incubating and culturing the samples to enhance bacterial growth so that you may enumerate them you may also encourage the growth of colonies (or media) that may contain enteric pathogens, such as Salmonella, Shigella, and

Legionella. As such, the waste Petri plates and culture media (such as would be used with IDEXX) should be treated as a biohazard and disinfected before it is discarded. The most effective way to do this is with an autoclave. A cheaper and effective yet not fool proof method is a 30 minute boil in a pressure cooker.

8. References

American Public Health Association, American Water Works Association, and Water Environment Federation (1998): **Standard Methods for the Examination of Water and Wastewater** (20th ed.), pp. 9-1 to 9-65, American Public Health Association, Washington, D.C.

Myers, D.N., and Wilde, F.D., eds., November 2003, Biological Indicators (3d ed.): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A7, accessed __date__ at http://pubs.water.usgs.gov/twri9A7/ (Chapter sections are cited by author and date.)

USEPA, 2002. Method 1604: Total Coliforms and Escherichia coli in Water by Membrane Filtration (MI Medium). US Environmental Protection Agency publication 821-R-02-024, September 2002. 14p.

U.S. Geological Survey, variously dated, *National Field Manual for the Collection of Water-Quality Data*: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A1-A9, available online at http://pubs.water.usgs.gov/twri9A. **National field manual for the collection of water-quality data (USGS Techniques of Water-Resources Investigations Book 9, Chapter A1-A9)**

9. Appendix A. Standard Methods for the Examination of Water and Wastewater, Applicable Portions of Section 9000.

Extracted and reporoduced without alteration from: American Public Health Association, American Water Works Association, and Water Environment Federation (1998): **Standard Methods for the Examination of Water and Wastewater** (20th ed.), pp. 9-1 to 9-65, parts, American Public Health Association, Washington, D.C.

Presented here to ensure availability.

9010 INTRODUCTION*

The following sections describe procedures for making microbiological examinations of water samples to determine sanitary quality. The methods are intended to indicate the degree of contamination with wastes. They are the best techniques currently available; however, their limitations must be understood thoroughly.

Tests for detection and enumeration of indicator organisms, rather than of pathogens, are used. The coliform group of bacteria, as herein defined, is the principal indicator of suitability of a water for domestic, industrial, or other uses. The cultural reactions and characteristics of this group of bacteria have been studied extensively.

Experience has established the significance of coliform group density as a criterion of the degree of pollution and thus of sanitary quality. The significance of the tests and the interpretation of results are well authenticated and have been used as a basis for standards of bacteriological quality of water supplies.

The membrane filter technique, which involves a direct plating for detection and estimation of coliform densities, is as effective as the multiple-tube fermentation test for detecting bacteria of the coliform group. Modification of procedural details, particularly of the culture medium, has made the results comparable with those given by the multiple-tube fermentation procedure. Although there are limitations in the application of the membrane filter technique, it is equivalent when used with strict adherence to these limitations and to the specified technical details. Thus, two standard methods are presented for the detection and enumeration of bacteria of the coliform group.

It is customary to report results of the coliform test by the multiple-tube fermentation procedure as a Most Probable Number (MPN) index. This is an index of the number of coliform bacteria that, more probably than any other number, would give the results shown by the laboratory examination; it is not an actual enumeration. By contrast, direct plating methods such as the membrane filter procedure permit a direct count of coliform colonies. In both procedures coliform density is reported conventionally as the MPN or membrane filter count per 100 mL. Use of either procedure permits appraising the sanitary quality of water and the effectiveness of treatment processes. Because it is not necessary to provide a quantitative assessment of coliform bacteria for all samples, a qualitative, presence-absence test is included.

Fecal streptococci and enterococci also are indicators of fecal pollution and methods for their detection and enumeration are given. A multiple-tube dilution and a membrane filter procedure are included.

Methods for the differentiation of the coliform group are included. Such differentiation generally is considered of limited value in assessing drinking water quality because the presence of any coliform bacteria renders the water potentially unsatisfactory and unsafe. Speciation may provide information on colonization of a distribution system and further confirm the validity of coliform results.

Coliform group bacteria present in the gut and feces of warm-blooded animals generally include organisms capable of producing gas from lactose in a suitable culture medium at 44.5 ± 0.2°C. Inasmuch as coliform organisms from other sources often cannot produce gas under these conditions, this criterion is used to define the fecal component of the coliform group. Both the multiple-tube dilution technique and the membrane filter procedure have been modified to incorporate incubation in confirmatory tests at 44.5°C to provide estimates of the density of fecal organisms, as defined. Procedures for fecal coliforms and *Escherichia coli* include a 24-h multiple-tube test using A-1 medium, a 7-h rapid method, and chromogenic substrate coliform tests. This differentiation yields valuable information concerning the possible source of pollution in water, and especially its remoteness, because the *nonfecal* members of the coliform group may be expected to survive longer than the *fecal* members in the unfavorable environment provided by the water.

The heterotrophic plate count may be determined by pour plate, spread plate, or membrane filter method. It provides an approximate enumeration of total numbers of viable bacteria that may yield useful information about water quality and may provide supporting data on the significance of coliform test results. The heterotrophic plate count is useful in judging the efficiency of various treatment processes and may have significant application as an in-plant control test. It also is valuable for checking quality of finished water in a distribution system as an indicator of microbial regrowth and sediment buildup in slow-flow sections and dead ends.

Experience in the shipment of un-iced samples by mail indicates that noticeable changes may occur in type or numbers of bacteria during such shipment for even limited periods of time. Therefore, refrigeration during transportation is recommended to minimize changes, particularly when ambient air temperature exceeds 13°C.

Procedures for the isolation of certain pathogenic bacteria and protozoa are presented. These procedures are tedious and complicated and are not recommended for routine use. Likewise, tentative procedures for enteric viruses are included but their routine use is not advocated.

Examination of routine bacteriological samples cannot be regarded as providing complete information concerning water quality. Always consider bacteriological results in the light of information available concerning the sanitary conditions surrounding the sample source. For a water supply, precise evaluation of quality can be made only when the results of laboratory examinations are interpreted in the light of sanitary survey data. Consider inadequate the results of the examination of a single sample from a given source. When possible, base evaluation of water quality on the examination of a series of samples collected over a known and protracted period of time.

Pollution problems of tidal estuaries and other bodies of saline water have focused attention on necessary modification of existing bacteriological techniques so that they may be used effectively. In the following sections, applications of specific techniques to saline water are not discussed because the methods used for fresh waters generally can be used satisfactorily with saline waters.

Methods for examination of the waters of swimming pools and other bathing places are included. The standard procedures for the plate count, fecal coliforms, and fecal streptococci are identical with those used for other waters. Procedures for *Staphylococcus* and *Pseudomonas aeruginosa,* organisms commonly associated with the upper respiratory tract or the skin, are included.

* Approved by Standard Methods Committee, 1993.

Procedures for aquatic fungi and actinomycetes are included.

Sections on rapid methods for coliform testing and on the recovery of stressed organisms are included. Because of increased interest and concern with analytical quality control, this section continues to be expanded.

The bacteriological methods in Part 9000, developed primarily to permit prompt and rapid examination of water samples, have been considered frequently to apply only to routine examinations. However, these same methods are basic to, and equally valuable in, research investigations in sanitary bacteriology and water treatment. Similarly, all techniques should be the subject of investigations to establish their specificity, improve their procedural details, and expand their application to the measurement of the sanitary quality of water supplies or polluted waters.

9020 QUALITY ASSURANCE/QUALITY CONTROL*

9020 A. Introduction

1. General Considerations

The growing emphasis on microorganisms in water quality standards and enforcement activities and their continuing role in research, process control, and compliance monitoring require the establishment and effective operation of a quality assurance (QA) program to substantiate the validity of analytical data.

A laboratory quality assurance program is the integration of intralaboratory and interlaboratory quality control (QC), standardization, and management practices into a formal, documented program with clearly defined responsibilities and duties to ensure that the data are of the type, quality, and quantity required.

The program must be practical and require only a reasonable amount of time or it will be bypassed. Generally, about 15% of overall laboratory time should be spent on different aspects of a quality assurance program. However, more time may be needed for more important analytical data, e.g., data for enforcement actions. When properly administered, a balanced, conscientiously applied QA program will optimize data quality without adversely affecting laboratory productivity.

Because microbiological analyses measure constantly changing living organisms, they are inherently variable. Some quality control tools used by chemists, such as reference standards, instrument calibration, and quality control charts, may not be available to the microbiologist.

Because QA programs vary among laboratories as a result of differences in organizational mission, responsibilities, and objectives; laboratory size, capabilities, and facilities; and staff skills and training, this provides only general guidance. Each laboratory should determine the appropriate QA level for its purpose.

2. Guidelines for a Quality Assurance Program

Develop a QA program to meet the laboratory's specific needs and the planned use of the data. Emphasis on the use of data is particularly important where significant and costly decisions depend on analytical results. An effective QA program will confirm the quality of results and increase confidence in the data.

a. Management responsibilities: Management must recognize the need for quality assurance, commit monetary and personnel resources, assume a leadership role, and involve staff in development and operation of the QA program. Management should meet with the laboratory supervisor and staff to develop and maintain a comprehensive program and establish specific responsibility for management, supervisors, and analysts.

b. Quality assurance officer: In large laboratories, a QA officer has the authority and responsibility for application of the QA program. Ideally, this person should have a staff position reporting directly to upper management, not a line position. The QA officer should have a technical education, be acquainted with all aspects of laboratory work, and be familiar with statistical techniques for data evaluation. The QA officer is responsible for initiating the program, convincing staff of its value, and providing necessary information and training to the staff. Once the QA program is functioning, the coordinator conducts frequent (weekly to monthly) reviews with the laboratory supervisor and staff to determine the current status and accomplishments of the program and to identify and resolve problems. The QA officer also reports periodically to management to secure backing in actions necessary to correct problems that threaten data quality.

c. Staff: Laboratory and field staffs participate with management in planning the QA program, preparing standard operating procedures, and most importantly, implementing the QC program in their daily tasks of collecting samples, conducting analyses, performing quality control checks, and calculating and reporting results. Because the staffs are the first to see potential problems, they should identify them and work with the supervisor to correct and avoid them. It is critical to the success of the QA program that staff understand and actively support it.

3. Quality Assurance Program Objectives

The objectives of a QA program include providing data of known quality, ensuring a high quality of laboratory performance, maintaining continuing assessment of laboratory operations, identifying weaknesses in laboratory operations, detecting training needs, and improving documentation and recordkeeping.

4. Elements of a Quality Assurance Program

Each laboratory should develop and implement a written QA plan describing the QA program and QC activities of the labo-

* Approved by Standard Methods Committee, 1997.

ratory. The plan should address the following basic common aspects:

a. Statement of objectives, describing the specific goals of the laboratory.

b. Sampling procedures, including selection of representative sites and specified holding time and temperature conditions. If data may be subjected to litigation, use chain-of-custody procedures.

c. Personnel policies, describing specific qualification and training requirements for supervisors and analysts.

d. Equipment and instrument requirements, providing calibration procedures and frequency and maintenance requirements.

e. Specifications for supplies, to ensure that reagents and supplies are of high quality and are tested for acceptability.

f. Analytical methods, i.e., standardized methods established by a standards-setting organization and validated. Ideally, these laboratory methods have documented precision, bias, sensitivity, selectivity, and specificity.

g. Analytical quality control measures, including such analytical checks as duplicate analyses, positive and negative controls, sterility checks, and verification tests.

h. Standard operating procedures (SOPs), i.e., written statement and documentation of all routine laboratory operations.

i. Documentation requirements, concerning data acquisition, recordkeeping, traceability, and accountability.

j. Assessment requirements:

1) Internal audits of the laboratory operations, performed by the QA officer and supervisor.

2) On-site evaluations by outside experts to ensure that the laboratory and its personnel are following an acceptable QA program.

3) Performance evaluation studies, in which the QA officer works with the supervisor to incorporate unknown challenge samples into routine analytical runs and laboratories are encouraged to participate in state and national proficiency testing and accreditation programs. The collaborative studies confirm the abilities of a laboratory to generate acceptable data comparable to those of other laboratories and identify potential problems.

k. Corrective actions: When problems are identified by the staff, supervisor, and/or QA coordinator, use standard stepwise procedures to determine the causes and correct them. Nonconformances identified by external laboratory evaluation are corrected, recorded, and signed off by the laboratory manager and QA officer.

Detailed descriptions of quality assurance programs are available.[1–4]

The QC guidelines discussed in 9020B and 9020C are recommended as useful source material, but all elements need to be addressed in developing a QA program.

5. References

1. GASKIN, J.E. 1992. Quality Assurance in Water Quality Monitoring. Inland Water Directorate, Conservation & Protection, Ottawa, Ont., Canada.
2. RATLIFF, T.A., JR. 1990. The Laboratory Quality Assurance System. A Manual of Quality Procedures with Related Forms. Van Nostrand Reinhold, New York, N.Y.
3. GARFIELD, F.M. 1984. Quality Assurance Principles of Analytical Laboratories, Assoc. Official Analytical Chemists, Arlington, Va.
4. DUX, J.P. 1983. Quality assurance in the analytical laboratory. *Amer. Lab.* 26:54.

9020 B. Intralaboratory Quality Control Guidelines

All laboratories have some intralaboratory QC practices that have evolved from common sense and the principles of controlled experimentation. A QC program applies practices necessary to minimize systematic and random errors resulting from personnel, instrumentation, equipment, reagents, supplies, sampling and analytical methods, data handling, and data reporting. It is especially important that laboratories performing only a limited amount of microbiological testing exercise strict QC. A listing of key QC practices is given in Table 9020:I. Other sources of QC practices are available.[1–3] These practices and guidelines will assist laboratories in establishing and improving QC programs. Laboratories should address all of the QC guidelines discussed herein, but the depth and details may differ for each laboratory.

1. Personnel

Microbiological testing should be performed by a professional microbiologist or technician trained in environmental microbiology whenever possible. If not, a professional microbiologist should be available for guidance. Train and evaluate the analyst in basic laboratory procedures. The supervisor periodically should review procedures of sample collecting and handling, media and glassware preparation, sterilization, routine analytical testing, counting, data handling, and QC techniques to identify and eliminate problems. Management should assist laboratory personnel in obtaining additional training and course work to advance their skills and career.

2. Facilities

a. Ventilation: Plan well-ventilated laboratories that can be maintained free of dust, drafts, and extreme temperature changes. Whenever possible, laboratories should have air conditioning to reduce contamination, permit more stable operation of incubators, and decrease moisture problems with media and instrumentation.

b. Space utilization: Design and operate the laboratory to minimize through traffic and visitors, with a separate area for preparing and sterilizing media, glassware, and equipment. Use a vented laminar-flow hood for dispensing and preparing sterile media, transferring microbial cultures, or working with pathogenic materials. In smaller laboratories it may be necessary, although undesirable, to carry out these activities in the same room.

c. Laboratory bench areas: Provide at least 2 m of linear bench space per analyst and additional areas for preparation and support activities. For stand-up work, typical bench dimensions are 90 to 97 cm high and 70 to 76 cm deep. For sit-down activities such

TABLE 9020:I. KEY QUALITY CONTROL PRACTICES

Item	Action	Frequency	Further Information in Section 9020B, ¶
Reagent water	Monitor quality	See Table 9020:II	
Bench surface	Monitor for contamination	Weekly	2*e*
Air in workplace	Monitor bacterial density	Monthly	2*e*
Thermometers	Check accuracy	Semiannually	3*a*
Balances and weights	Check accuracy	Monthly	3*b*
Balances	Service and recalibrate	Annually	3*b*
pH meter	Standardize	Each use	3*c*
	Check against another meter	Monthly	3*c*
Media-dispensing apparatus	Check volume accuracy	Each use	3*f*
Hot-air oven	Check performance	Monthly	3*g*
Autoclave	Check performance	Each use	3*h*
Refrigerator	Check temperature	Daily	3*i*
Freezer	Check temperature	Daily	3*j*
	Defrost	Semiannually	3*j*
Membrane filtration equipment	Check for leaks and surface scratches	Each use	3*k*
UV lamps	Test with UV meter	Quarterly	3*l*
Biohazard hood	Monitor air and UV lamps	Monthly	3*m*
	Inspect for airflow	Quarterly	3*m*
Incubator	Check temperature	Twice daily	3*n* and *o*
Microscope	Clean optics and stage	Each use	3*p*
Glassware	Inspect for cleanliness, chips, and etching	Each use	4*a*
	Check pH	Each batch	4*a*1)
	Conduct inhibitory residue test	Annually	4*a*2)
Dilution water bottles	Check pH and volume	Each use	4*c*
Media	Check pH and appearance	Each use	4*i*1)
Autoclave	Check performance	Weekly	4*i*2)
Plate counts	Perform duplicate analyses	Weekly	8*a*4)
	Repeat counts	Monthly	8*a*2)

as microscopy and plate counting, benches are 75 to 80 cm high. Specify bench tops of stainless steel, epoxy plastic, or other smooth, impervious surface that is inert and corrosion-resistant, has a minimum number of seams, and has adequate sealing of any crevices. Install even, glare-free lighting with about 1000 lux (100 ft-candles) intensity at the working surface.

d. Walls and floors: Assure that walls are covered with a smooth finish that is easily cleaned and disinfected. Specify floors of smooth concrete, vinyl, asphalt tile, or other impervious, sealed washable surfaces.

e. Work-area monitoring: Maintain high standards of cleanliness in work areas. Monitor air, at least monthly, with air density plates. The number of colonies on the air density plate test should not exceed 160/m^2/15 min exposure (15 colonies/plate/15 min).

Plate or the swab method[1] can be used weekly or more frequently to monitor bench surface contamination. Although uniform limits for bacterial density have not been set, each laboratory can use these tests to establish a base line and take action on a significant increase.

f. Laboratory cleanliness: Regularly clean laboratory rooms and wash benches, shelves, floors, and windows. Wet-mop floors and treat with a disinfectant solution; do not sweep or dry-mop. Wipe bench tops and treat with a disinfectant before and after use. Do not permit laboratory to become cluttered.

3. Laboratory Equipment and Instrumentation

Verify that each item of equipment meets the user's needs for precision and minimization of bias. Perform equipment maintenance on a regular basis as recommended by the manufacturer or obtain preventive maintenance contracts on autoclave, balances, microscopes, and other equipment. Directly record all quality control checks in a permanent log book.

Use the following quality control procedures:

a. Thermometer/temperature-recording instruments: Check accuracy of thermometers or temperature-recording instruments semiannually against a certified National Institute of Standards and Technology (NIST) thermometer or one traceable to NIST and conforming to NIST specifications. For general purposes use thermometers graduated in increments of 0.5°C or less. Maintain in water or glycerol for air incubators and refrigerators and glycerol for freezers and seal in a flask. For a 44.5°C water bath, use a submersible thermometer graduated to 0.2°C or less. Record temperature check data in a quality control log. Mark the necessary NIST calibration corrections on each thermometer and incubator, refrigerator, or freezer. When possible, equip incubators and water baths with temperature-recording instruments that provide a continuous record of operating temperature.

b. Balances: Follow manufacturer's instructions in operation and routine maintenance of analytical and top-loading balances. Balances should be serviced and recalibrated by a manufacturer technician annually or more often as conditions change or problems occur. In weighing 2 g or less, use an analytical balance with a sensitivity less than 1 mg at a 10-g load. For larger quantities use a pan balance with sensitivity of 0.1 g at a 150-g load.

Wipe balance before use with a soft brush. Clean balance pans after use and wipe spills up immediately with a laboratory tissue. Inspect weights with each use and replace if corroded. Use only a plastic-tip forceps to handle weights. Check balance and working weights monthly against a set of reference weights (ANSI/

ASTM Class 1 or NIST Class S) for accuracy, precision, and linearity.[4] Record results.

c. pH meter: Use a meter graduated in 0.1 pH units or less, that includes temperature compensation. Preferably use digital meters and commercial buffer solutions. With each use, standardize meter with two buffers that bracket the pH of interest and record. Date buffer solutions when opened and check monthly against another pH meter. Discard solution after each use and replace buffer supply before expiration date. For full details of pH meter use and maintenance, see Section 4500-H^+.

d. Water purification system: Commercial systems are available that include some combination of prefiltration, activated carbon, mixed-bed resins, and reverse-osmosis with final filtration to produce a reagent-grade water. The life of such systems can be extended greatly if the source water is pretreated by distillation or by reverse osmosis to remove dissolved solids. Such systems tend to produce the same quality water until resins or activated carbon are near exhaustion and quality abruptly becomes unacceptable. Some deionization components are available now that automatically regenerate the ion exchange resins. Do not store reagent water unless a commercial UV irradiation device is installed and is confirmed to maintain sterility.

Monitor reagent water continuously or daily with a calibrated conductivity meter and analyze at least annually for trace metals. Replace cartridges at intervals recommended by the manufacturer based on the estimated usage and source water quality. Do not wait for column failure. If bacteria-free water is desired, include aseptic final filtration with a 0.22-μm-pore membrane filter and collect in a sterile container. Monitor treated water for contamination and replace the filter as necessary.

e. Water still: Stills produce water of a good grade that characteristically deteriorates slowly over time as corrosion, leaching, and fouling occur. These conditions can be controlled with proper maintenance and cleaning. Stills efficiently remove dissolved substances but not dissolved gases or volatile organic chemicals. Freshly distilled water may contain chlorine and ammonia (NH_3). On storage, additional NH_3 and CO_2 are absorbed from the air. Use softened water as the source water to reduce frequency of cleaning the still. Drain and clean still and reservoir according to manufacturer's instructions and usage.

f. Media dispensing apparatus: Check accuracy of volumes dispensed with a graduated cylinder at start of each volume change and periodically throughout extended runs. If the unit is used more than once per day, pump a large volume of hot reagent water through the unit to rinse between runs. Correct leaks, loose connections, or malfunctions immediately. At the end of the work day, break apparatus down into parts, wash, rinse with reagent water, and dry. Lubricate parts according to manufacturer's instructions or at least once per month.

g. Hot-air oven: Test performance monthly with commercially available *Bacillus subtilis* spore strips or spore suspensions. Monitor temperature with a thermometer accurate in the 160 to 180°C range and record results. Use heat-indicating tape to identify supplies and materials that have been exposed to sterilization temperatures.

h. Autoclave: Record items sterilized, temperature, pressure, and time for each run. Optimally use a recording thermometer. Check and record operating temperature weekly with a minimum/maximum thermometer. Test performance with *Bacillus stearothermophilus* spore strips, suspensions, or capsules monthly. Use heat-indicating tape to identify supplies and materials that have been sterilized.

i. Refrigerator: Maintain temperature at 1 to 4°C. Check and record temperature daily and clean monthly. Identify and date materials stored. Defrost as required and discard outdated materials quarterly.

j. Freezer: Maintain temperature at −20°C to −30°C. Check and record temperature daily. A recording thermometer and alarm system are highly desirable. Identify and date materials stored. Defrost and clean semiannually; discard outdated materials.

k. Membrane filtration equipment: Before use, assemble filtration units and check for leaks. Discard units if inside surfaces are scratched. Wash and rinse filtration assemblies thoroughly after use, wrap in nontoxic paper or foil, and sterilize.

l. Ultraviolet lamps: Disconnect lamps monthly and clean bulbs with a soft cloth moistened with ethanol. Test lamps quarterly with an appropriate (short- or long-wave) UV light meter* and replace bulbs if output is less than 70% of the original. For short-wave lamps used in disinfecting work areas, expose plate count agar spread plates containing 200 to 300 organisms of interest, for 2 min. Incubate plates at 35°C for 48 h and count colonies. Replace bulb if count is not reduced 99%.

CAUTION: *Although short-wave (254-nm) UV light is known to be more dangerous than long-wave UV (365-nm), both types of UV light can damage eyes and skin and potentially are carcinogenic.[5] Protect eyes and skin from exposure to UV light. (See Section 1090B.)*

m. Biohazard hood: Once per month expose plate count agar plates to air flow for 1 h. Incubate plates at 35°C for 48 h and examine for contamination. A properly operating biohazard hood should produce no growth on the plates. Disconnect UV lamps and clean monthly by wiping with a soft cloth moistened with ethanol. Check lamps' efficiency as specified above. Inspect cabinet for leaks and rate of air flow quarterly. Use a pressure monitoring device to measure efficiency of hood performance. Have laminar-flow safety cabinets containing HEPA filters serviced by the manufacturer. Maintain hoods as directed by the manufacturer.

n. Water bath incubator: Verify that incubators maintain test temperature, such as 35 ± 0.5°C or 44.5 ± 0.2°C. Keep an appropriate thermometer (¶ 3*a*, above) immersed in the water bath; monitor and record temperature twice daily (morning and afternoon). For optimum operation, equip water bath with a gable cover. Use only stainless steel, plastic-coated, or other corrosion-proof racks. Clean bath as needed.

o. Incubator (air, water jacketed, or aluminum block): Verify that incubators maintain appropriate test temperatures. Also, verify that cold samples are incubated at the test temperature for the required time. Check and record temperature twice daily (morning and afternoon) on the shelves in use. If a glass thermometer is used, submerge bulb and stem in water or glycerine to the stem mark. For best results use a recording thermometer and alarm system. Place incubator in an area where room temperature is maintained between 16 and 27°C (60 to 80°F).

p. Microscopes: Use lens paper to clean optics and stage after each use. Cover microscope when not in use.

Permit only trained technicians to use fluorescence microscope and light source. Monitor fluorescence lamp with a light meter

* Fisher Scientific, short wave meter (Cat. No. 11-924-54) and long wave meter (Cat. No. 11-984-53), Pittsburgh, PA 15219-4785, or equivalent.

and replace when a significant loss in fluorescence is observed. Log lamp operation time, efficiency, and alignment. Periodically check lamp alignment, particularly when the bulb has been changed; realign if necessary. Use known positive 4 + fluorescence slides as controls.

4. Laboratory Supplies

a. Glassware: Before each use, examine glassware and discard items with chipped edges or etched inner surfaces. Particularly examine screw-capped dilution bottles and flasks for chipped edges that could leak and contaminate the analyst and the area. Inspect glassware after washing for excessive water beading and rewash if necessary. Make the following tests for clean glassware as necessary:

1) pH check—Because some cleaning solutions are difficult to remove completely, spot check batches of clean glassware for pH reaction, especially if soaked in alkali or acid. To test clean glassware for an alkaline or acid residue add a few drops of 0.04% bromthymol blue (BTB) or other pH indicator and observe the color reaction. BTB should be blue-green (in the neutral range).

To prepare 0.04% bromthymol blue indicator solution, add 16 mL 0.01*N* NaOH to 0.1 g BTB and dilute to 250 mL with reagent water.

2) Test for inhibitory residues on glassware and plasticware—Certain wetting agents or detergents used in washing glassware may contain bacteriostatic or inhibiting substances that require 6 to 12 rinsings to remove all traces and insure freedom from residual bacteriostatic action. Perform this test annually and before using a new supply of detergent. If prewashed, presterilized plasticware is used, test it for inhibitory residues. Although the following procedure describes testing of petri dishes for inhibitory residue, it is applicable to other glass or plasticware.

a) Procedure—Wash and rinse six petri dishes according to usual laboratory practice and designate as Group A.

Wash six petri dishes as above, rinse 12 times with successive portions of reagent water, and designate as Group B.

Rinse six petri dishes with detergent wash water (in use concentration), and air-dry without further rinsing, and designate as Group C.

Sterilize dishes in Groups A, B, and C by the usual procedure.

For presterilized plasticware, set up six plastic petri dishes and designate them as Group D.

Prepare and sterilize 200 mL plate count agar and hold in a 44 to 46°C water bath.

Prepare a culture of *E. aerogenes* known to contain 50 to 150 colony-forming units/mL. Preliminary testing may be necessary to achieve this count range. Inoculate three dishes from each test group with 0.1 mL and the other three dishes from each group with 1 mL culture.

Analyze the four sets of six plates each, following heterotrophic plate count method (Section 9215B), and incubate at 35°C for 48 h. Count plates with 30 to 300 colonies and record results as CFU/mL.

b) Interpretation of results—Difference in averaged counts on plates in Groups A through D should be less than 15% if there are no toxic or inhibitory effects.

Differences in averaged counts of less than 15% between Groups A and B and greater than 15% between Groups A and C indicate that the cleaning detergent has inhibitory properties that are eliminated during routine washing. Differences between B and D greater than 15% indicate an inhibitory residue.

b. Utensils and containers for media preparation: Use utensils and containers of borosilicate glass, stainless steel, aluminum, or other corrosion-resistant material (see Section 9030). Do not use copper utensils.

c. Dilution water bottles: Use scribed bottles made of nonreactive borosilicate glass or plastic with screwcaps containing inert liners. Clean before use. Disposable plastic bottles prefilled with dilution water are available commercially and are acceptable. Before use of each lot, check pH and volume and examine sterile bottles of dilution water for a precipitate; discard if present. Reclean bottles with acid if necessary, and remake the dilution water. If precipitate repeats, procure a different source of bottles.

d. Reagent-grade water quality: The quality of water obtainable from a water purification system differs with the system used and its maintenance. See 3*d* and *e* above. Recommended limits for reagent water quality are given in Table 9020:II. If these limits are not met, investigate and correct or change water source. Although pH measurement of reagent water is characterized by drift, extreme readings are indicative of chemical contamination.

e. Use test for evaluation of reagent water, media, and membranes: When a new lot of culture medium, membrane filters, or a new source of reagent-grade water is to be used make comparison tests, at least quarterly, of the current lot in use (reference lot) against the new lot (test lot).

1) Procedure—Use a single batch of control water (redistilled or distilled water polished by deionization), glassware, membrane filters, or other needed materials to control all variables except the one factor under study. Make parallel pour or spread plate or

TABLE 9020:II. QUALITY OF REAGENT WATER USED IN MICROBIOLOGY TESTING

Test	Monitoring Frequency	Maximum Acceptable Limit
Chemical tests:		
Conductivity	Continuously or with each use	>0.5 megohms resistance or <2 μmhos/cm at 25°C
pH	With each use	5.5–7.5
Total organic carbon	Monthly	<1.0 mg/L
Heavy metals, single (Cd, Cr, Cu, Ni, Pb, and Zn)	Annually*	<0.05 mg/L
Heavy metals, total	Annually*	<0.10 mg/L
Ammonia/organic nitrogen	Monthly	<0.10 mg/L
Total chlorine residual	Monthly or with each use	<0.01 mg/L
Bacteriological tests:		
Heterotrophic plate count (See Section 9215)	Monthly	< 1000 CFU/mL
Use test (see 4*e*)	Quarterly and for a new source	Student's $t \leq 2.78$

* Or more frequently if there is a problem.

membrane filter plate tests on reference lot and test lot, according to procedures in Sections 9215 and 9222. As a minimum, make single analyses on five different water samples positive for the target organism. Replicate analyses and additional samples can be tested to increase the sensitivity of detecting differences between reference and test lots.

When conducting the use test on reagent water, perform the quantitative bacterial tests in parallel using a known high-quality water as a control water. Prepare dilution/rinse water and media with new source of reagent and control water. Test water for all uses (dilution, rinse, media preparation, etc.).

2) Counting and calculations—After incubation, compare bacterial colonies from the two lots for size and appearance. If colonies on the test lot plates are atypical or noticeably smaller than colonies on the reference lot plates, record the evidence of inhibition or other problem, regardless of count differences. Count plates and calculate the individual count per 1 mL or per 100 mL. Transform the count to logarithms and enter the log-transformed results for the two lots in parallel columns. Calculate the difference, d, between the two transformed results for each sample, including the + or − sign, the mean, $\overline{d}$ and the standard deviation s_d of these differences (see Section 1010B).

Calculate Student's t statistic, using the number of samples as n:

$$t = \frac{\overline{d}}{s_d/\sqrt{n}}$$

These calculations may be made with various statistical software packages available for personal computers.

3) Interpretation—Use the critical t value, from a Student's t table for comparison against the calculated value. At the 0.05 significance level this value is 2.78 for five samples (four degrees of freedom). If the calculated t value does not exceed 2.78, the lots do not produce significantly different results and the test lot is acceptable. If the calculated t value exceeds 2.78, the lots produce significantly different results and the test lot is unacceptable.

If the colonies are atypical or noticeably smaller on the test lot or the Student's t exceeds 2.78, review test conditions, repeat the test, and/or reject the test lot and obtain another one.

f. Reagents: Because reagents are an integral part of microbiological analyses, their quality must be assured. Use only chemicals of ACS or equivalent grade because impurities can inhibit bacterial growth, provide nutrients, or fail to produce the desired reaction. Date chemicals and reagents when received and when first opened for use. Make reagents to volume in volumetric flasks and transfer for storage to good-quality inert plastic or borosilicate glass bottles with borosilicate, polyethylene, or other plastic stoppers or caps. Label prepared reagents with name and concentration, date prepared, and initials of preparer. Include positive and negative control cultures with each series of cultural or biochemical tests.

g. Dyes and stains: In microbiological analyses, organic chemicals are used as selective agents (e.g., brilliant green), as indicators (e.g., phenol red), and as microbiological stains (e.g., Gram stain). Dyes from commercial suppliers vary from lot to lot in percent dye, dye complex, insolubles, and inert materials. Because dyes for microbiology must be of proper strength and stability to produce correct reactions, use only dyes certified by the Biological Stain Commission. Check bacteriological stains before use with at least one positive and one negative control culture and record results.

h. Membrane filters and pads: The quality and performance of membrane filters vary with the manufacturer, type, brand, and lot. These variations result from differences in manufacturing methods, materials, quality control, storage conditions, and application.

1) Membrane filters and pads for water analyses should meet the following specifications:

a) Filter diam 47 mm, mean pore diam 0.45 μm. Alternate filter and pore sizes may be used if the manufacturer provides data verifying performance equal to or better than that of 47- mm-diam, 0.45-μm-pore size filter. At least 70% of filter area must be pores.

b) When filters are floated on reagent water, the water diffuses uniformly through the filters in 15 s with no dry spots on the filters.

c) Flow rates are at least 55 mL/min/cm^2 at 25°C and a differential pressure of 93 kPa.

d) Filters are nontoxic, free of bacterial-growth-inhibiting or stimulating substances, and free of materials that directly or indirectly interfere with bacterial indicator systems in the medium; ink grid is nontoxic. The arithmetic mean of five counts on filters must be at least 90% of the arithmetic mean of the counts on five agar spread plates using the same sample volumes and agar media.

e) Filters retain the organisms from a 100-mL suspension of *Serratia marcescens* containing 1×10^3 cells.

f) Water-extractables in filter do not exceed 2.5% after the membrane is boiled in 100 mL reagent water for 20 min, dried, cooled, and brought to constant weight.

g) Absorbent pad has diam 47 mm, thickness 0.8 mm, and is capable of absorbing 2.0 ± 0.2 mL Endo broth.

h) Pads release less than 1 mg total acidity calculated as $CaCO_3$ when titrated to the phenolphthalein end point with 0.02N NaOH.

i) If filter and absorbent pad are not sterile, they should not be degraded by sterilization at 121°C for 10 min. Confirm sterility by absence of growth when a membrane filter is placed on a pad saturated with tryptone glucose extract broth or tryptone glucose extract agar and incubated at 35 ± 0.5°C for 24 h.

j) Some lots of membrane filters yield low recoveries, poor differentiation, or malformation of colonies due to toxicity, chemical composition, or structural defects.[6] Perform the use test (¶ 4*e*) on new lots of filters.

2) Standardized tests:

Standardized tests are available for evaluating retention, recovery, extractables, and flow rate characteristics of membrane filters.[7]

Some manufacturers provide information beyond that required by specifications and certify that their membranes are satisfactory for water analysis. They report retention, pore size, flow rate, sterility, pH, percent recovery, and limits for specific inorganic and organic chemical extractables. Although the standard membrane filter evaluation tests were developed for the manufacturers, a laboratory can conduct its own tests.

To maintain quality control inspect each lot of membranes before use and during testing to insure they are round and pliable, with undistorted gridlines after autoclaving. After incubation, colonies should be well-developed with well-defined color and shape as defined by the test procedure. The gridline ink should not channel growth along the ink line nor restrict colony development. Colonies should be distributed evenly across the membrane surface.

i. Culture media: Because cultural methods depend on properly prepared media, use the best available materials and techniques in media preparation, storage, and application. For control of quality, use commercially prepared media whenever available but note that such media may vary in quality among manufacturers and even from lot to lot from the same manufacturer.

Order media in quantities to last no longer than 1 year. Use media on a first-in, first-out basis. When practical, order media in quarter pound (114 g) multiples rather than one pound (454 g) bottles, to keep the supply sealed as long as possible. Record kind, amount, and appearance of media received, lot number, expiration date, and dates received and opened. Check inventory quarterly for reordering.

Store dehydrated media at an even temperature in a cool dry place, away from direct sunlight. Discard media that cake, discolor, or show other signs of deterioration. If expiration date is given by manufacturer, discard unused media after that date. A conservative time limit for unopened bottles is 2 years at room temperature. Compare recovery of newly purchased lots of media against proven lots, using recent pure-culture isolates and natural samples.

Use opened bottles of media within 6 months. Dehydrated media are hygroscopic. Protect opened bottles from moisture. Close bottles as tightly as possible, immediately after use. If caking or discoloration of media occurs, discard media. Store opened bottles in a dessicator.

1) Preparation of media—Prepare media in containers that are at least twice the volume of the medium being prepared. Stir media, particularly agars, while heating. Avoid scorching or boil-over by using a boiling water bath for small batches of media and by continually attending to larger volumes heated on a hot plate or gas burner. Preferably use hot plate-magnetic stirrer combinations. Label and date prepared media. Prepare media in reagent water. Measure water volumes and media with graduates or pipets conforming to NIST and APHA standards, respectively. Do not use blow-out pipets. After preparation and storage, remelt agar media in boiling water or flowing steam.

Check and record pH of a portion of each medium after sterilization and cooling. Check pH of solid medium with a surface probe. Record results. Make minor adjustments in pH (<0.5 pH units) with 1*N* NaOH or HCl solution to the pH specified in formulation. If the pH difference is larger than 0.5 units, discard the batch and check preparation instructions and pH of reagent water to resolve the problem. Incorrect pH values may be due to reagent water quality, medium deterioration, or improper preparation. Review instructions for preparation and check water pH. If water pH is unsatisfactory, prepare a new batch of medium using water from a new source (see 9020B.3*d* and *e*). If water is satisfactory, remake medium and check; if pH is again incorrect, prepare medium from another bottle.

Record pH problems in the media record book and inform the manufacturer if the medium is indicated as the source of error. Examine prepared media for unusual color, darkening, or precipitation and record observations. Consider variations of sterilization time and temperature as possible causes for problems. If any of the above occur, discard the medium.

2) Sterilization—Sterilize media at 121 to 124°C for the minimum time specified. A double-walled autoclave permits maintenance of full pressure and temperature in the jacket between loads and reduces chance for heat damage. Follow manufacturer's directions for sterilization of specific media. The required exposure time varies with form and type of material, type of medium, presence of carbohydrates, and volume. Table 9020:III gives guidelines for typical items. Do not expose media containing carbohydrates to the elevated temperatures for more than 45 min. Exposure time is defined as the period from initial exposure to removal from the autoclave.

TABLE 9020:III. TIME AND TEMPERATURE FOR AUTOCLAVE STERILIZATION

Material	Time at 121°C*
Membrane filters and pads	10 min
Carbohydrate-containing media (lauryl tryptose, BGB broth, etc.)	12–15 min
Contaminated materials and discarded cultures	30 min
Membrane filter assemblies (wrapped), sample collection bottles (empty)	15 min
Buffered dilution water, 99 mL in screw-cap bottle	15 min
Rinse water, volume > 100 mL	Adjust for volume

* Except for media, times are guidelines; check for sterility.

Some currently available autoclave models are automatic and include features such as vertical sliding, self-sealing and opening doors, programmable sterilization cycles, and continuous multipoint monitoring of chamber temperature and pressure. These units also may incorporate solution cooling and vapor removal features. When sterilizer design includes heat exchangers and solution cooling features as part of a factory-programmed liquid cycle, strict adherence to the 45-min total elapsed time in the autoclave is not necessary provided that printout records verify normal cycle operation and chamber cooling during exhaust and vapor removal.

Remove sterilized media from autoclave as soon as chamber pressure reaches zero, or, if a fully automatic model is used, as soon as the door opens. Do not reautoclave media.

Check effectiveness of sterilization weekly by placing *Bacillus stearothermophilus* spore suspensions or strips (commercially available) inside glassware. Sterilize at 121°C for 15 min. Place in trypticase soy broth tubes and incubate at 55°C for 48 h. If growth of the autoclaved spores occurs after incubation at 55°C, sterilization was inadequate. A small, relatively inexpensive 55°C incubator is available commercially.†

Sterilize heat-sensitive solutions or media by filtration through a 0.22-µm-pore-diam filter in a sterile filtration and receiving apparatus. Filter and dispense medium in a safety cabinet or biohazard hood if available. Sterilize glassware (pipets, petri dishes, sample bottles) in an autoclave or an oven at 170°C for 2 h. Sterilize equipment, supplies, and other solid or dry materials that are heat-sensitive, by exposing to ethylene oxide in a gas sterilizer. Use commercially available spore strips or suspensions to check dry heat and ethylene oxide sterilization.

3) Use of agars and broths—Temper melted agars in a water bath at 44 to 46°C until used but do not hold longer than 3 h. To monitor agar temperature, expose a bottle of water or medium to the same heating and cooling conditions as the agar. Insert a thermometer in the monitoring bottle to determine when the temperature is 45 to 46°C and suitable for use in pour plates. If possible, prepare media on the day of use. After pouring agar plates for streaking, dry agar surfaces by keeping dish slightly open for at

† 3M Health Care, St. Paul, MN 55144, or equivalent.

least 15 min in a bacteriological hood to avoid contamination. Discard unused liquid agar; do not let harden or remelt for later use.

Handle tubes of sterile fermentation media carefully to avoid entrapping air in inner tubes, thereby producing false positive reactions. Examine freshly prepared tubes to determine that gas bubbles are absent.

4) Storage of media—Prepare media in amounts that will be used within holding time limits given in Table 9020:IV. Protect media containing dyes from light; if color changes occur, discard the media. Refrigerate poured agar plates not used on the day of preparation. Seal agar plates with loose-fitting lids in plastic bags if held more than 2 d. Prepare broth media that will be stored for more than 2 weeks in screw-cap tubes, other tightly sealed tubes, or in loose-capped tubes placed in a sealed plastic bag or other tightly sealed container to prevent evaporation.

Mark liquid level in several tubes and monitor for loss of liquid. If loss is 10% or more, discard the batch. If media are refrigerated, incubate overnight at test temperature before use and reject the batch if false positive responses occur. Prepared sterile broths and agars available from commercial sources may offer advantages when analyses are done intermittently, when staff is not available for preparation work, or when cost can be balanced against other factors of laboratory operation. Check performance of these media as described in ¶ 5 below.

5) Quality control of prepared media—Maintain in a bound book a complete record of each prepared batch of medium with name of preparer and date, name and lot number of medium, amount of medium weighed, volume of medium prepared, sterilization time and temperature, pH measurements and adjustments, and preparations of labile components. Compare quantitative recoveries of new lots with previously acceptable ones. Include sterility and positive and negative control culture checks on all media as described below.

5. Standard Operating Procedures (SOPs)

SOPs are the operational backbone of an analytical laboratory. SOPs describe in detail all laboratory operations such as preparation of reagents, reagent water, standards, culture media, proper use of balances, sterilization practices, and dishwashing procedures, as well as methods of sampling, analysis, and quality control. The SOPs are unique to the laboratory. They describe the tasks as performed on a day-to-day basis, tailored to the laboratory's own equipment, instrumentation, and sample types. The SOPs guide routine operations by each analyst, help to assure uniform operations, and provide a solid training tool.

TABLE 9020:IV. HOLDING TIMES FOR PREPARED MEDIA

Medium	Holding Time
Membrane filter (MF) broth in screw-cap flasks at 4°C	96 h
MF agar in plates with tight-fitting covers at 4°C	2 weeks
Agar or broth in loose-cap tubes at 4°C	2 weeks
Agar or broth in tightly closed screw-cap tubes or other sealed containers	3 months
Poured agar plates with loose-fitting covers in sealed plastic bags at 4°C	2 weeks
Large volume of agar in tightly closed screw-cap flask or bottle at 4°C	3 months

6. Sampling

a. Planning: Microbiologists should participate in the planning of monitoring programs that will include microbial analyses. They can provide valuable expertise on the selection of sampling sites, number of samples and analyses needed, workload, and equipment and supply needs. For natural waters, knowledge of the probable microbial densities, and the impact of season, weather, tide and wind patterns, known sources of pollution, and other variables, are needed to formulate the most effective sampling plan.

b. Methods: Sampling plans must be specific for each sampling site. Prior sampling guidance can be only general in nature, addressing the factors that must be considered for each site. Sampling SOPs describe sampling equipment, techniques, frequency, holding times and conditions, safety rules, etc., that will be used under different conditions for different sites. From the information in these SOPs sampling plans will be drawn up.

7. Analytical Methods

a. Method selection: Because minor variations in technique can cause significant changes in results, microbiological methods must be standardized so that uniform data result from multiple laboratories. Select analytical methods appropriate for the sample type from *Standard Methods* or other source of standardized methods and ensure that methods have been validated in a multilaboratory study with the sample types of interest.

b. Data objectives: Review available methods and determine which produce data to meet the program's needs for precision, bias, specificity, selectivity, and detection limit. Ensure that the methods have been demonstrated to perform within the above specifications for the samples of interest.

c. Internal QC: The written analytical methods should contain required QC checks of positive and negative control cultures, sterile blank, replicate analyses (precision), and a known quantitative culture, if available.

d. Method SOPs: As part of the series of SOPs, provide each analyst with a copy of the analytical methods written in step-wise fashion exactly as they are to be performed and specific to the sample type, equipment, and instrumentation used in the laboratory.

8. Analytical Quality Control Procedures

a. General quality control procedures:

1) New methods—Conduct parallel tests with the standard procedure and a new method to determine applicability and comparability. Perform at least 100 parallel tests across seasons of the year before replacement with the new method for routine use.

2) Comparison of plate counts—For routine performance evaluation, repeat counts on one or more positive samples at least monthly and compare the counts with those of other analysts testing the same samples. Replicate counts for the same analyst should agree within 5% and those between analysts should agree within 10%. See 9020B.10*b* for a statistical calculation of data precision.

3) Control cultures—For each lot of medium check analytical procedures by testing with known positive and negative control cultures for the organism(s) under test. See Table 9020:V for examples of test cultures.

TABLE 9020:V. CONTROL CULTURES FOR MICROBIOLOGICAL TESTS

Group	Control Culture	
	Positive	Negative
Total coliforms	*Escherichia coli* *Enterobacter aerogenes*	*Staphylococcus aureus* *Pseudomonas* sp.
Fecal coliforms	*E. coli*	*E. aerogenes* *Streptococcus faecalis*
Escherichia coli	*E. coli*	*E. aerogenes*
Fecal streptococci	*Enterococcus faecalis*	*Staphylococcus aureus* *E. coli*
Enterococci	*S. faecalis*	*S. mitis/salivarius*

4) Duplicate analyses—Perform duplicate analyses on 10% of samples and on at least one sample per test run. A test run is defined as an uninterrupted series of analyses. If the laboratory conducts less than 10 tests/week, make duplicate analyses on at least one sample each week.

5) Sterility checks—For membrane filter tests, check sterility of media, membrane filters, buffered dilution and rinse water, pipets, flasks and dishes, and equipment as a minimum at the end of each series of samples, using sterile reagent water as the sample. If contaminated, check for the source. For multiple-tube and presence-absence procedures, check sterility of media, dilution water, and glassware. To test sterility of media, incubate a representative portion of each batch at an appropriate temperature for 24 to 48 h and observe for growth. Check each batch of buffered dilution water for sterility by adding 20 mL water to 100 mL of a nonselective broth. Alternatively, aseptically pass 100 mL or more dilution water through a membrane filter and place filter on growth medium suitable for heterotrophic bacteria. Incubate at 35 ± 0.5°C for 24 h and observe for growth. If any contamination is indicated, determine the cause and reject analytical data from samples tested with these materials. Request immediate resampling and reanalyze.

b. Precision of quantitative methods: Calculate precision of duplicate analyses for each different type of sample examined, for example, drinking water, ambient water, wastewater, etc., according to the following procedure:

1) Perform duplicate analyses on first 15 positive samples of each type, with each set of duplicates analyzed by a single analyst. If there is more than one analyst, include all analysts regularly running the tests, with each analyst performing approximately an equal number of tests. Record duplicate analyses as D_1 and D_2.

2) Calculate the logarithm of each result. If either of a set of duplicate results is <1, add 1 to both values before calculating the logarithms.

3) Calculate the range (R) for each pair of transformed duplicates as the mean ($\overline{R}$) of these ranges.

See sample calculation in Table 9020:VI.

4) Thereafter, analyze 10% of routine samples in duplicate. Transform the duplicates as in ¶ 2) and calculate their range. If the range is greater than 3.27 $\overline{R}$, there is greater than 99% probability that the laboratory variability is excessive. Determine if increased imprecision is acceptable; if not, discard all analytical results since the last precision check (see Table 9020:VII). Identify and resolve the analytical problem before making further analyses.

5) Update the criterion used in ¶ 4) by periodically repeating the procedures of ¶s 1) through 3) using the most recent sets of 15 duplicate results.

TABLE 9020:VI. CALCULATION OF PRECISION CRITERION

Sample No.	Duplicate Analyses		Logarithms of Counts		Range of Logarithms (R_{log}) ($L_1 - L_2$)
	D_1	D_2	L_1	L_2	
1	89	71	1.9494	1.8513	0.0981
2	38	34	1.5798	1.5315	0.0483
3	58	67	1.7634	1.8261	0.0627
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
14	7	6	0.8451	0.7782	0.0669
15	110	121	2.0414	2.0828	0.0414

Calculations:

1) Σ of R_{log} = 0.0981 + 0.0483 + 0.0627 + ... + 0.0669 + 0.0414
= 0.718 89

2) $\overline{R} = \frac{\Sigma R_{log}}{n} = \frac{0.718\,89}{15} = 0.0479$

3) Precision criterion = 3.27 $\overline{R}$ = 3.27 (0.0479) = 0.1566

9. Verification

For the most part, the confirmation/verification procedures for drinking water differ from those for other waters because of specific regulatory requirements.

a. Multiple-tube fermentation (MTF) methods:

1) Total coliform procedure (9221B)

a) Drinking water—Carry samples through confirmed phase only. Verification is not required. For QC purposes, if normally there are no positive results, analyze at least one positive source water quarterly to confirm that the media produce appropriate responses. For samples with a history of heavy growth without gas in presumptive-phase tubes, carry the tubes through the confirmed phase to check for false negative responses for coliform bacteria. Verify any positives for fecal coliforms or *E. coli.*

b) Other water types—Verify by performing the completed MTF Test on 10% of samples positive through the confirmed phase.

2) Enzyme substrate coliform test (total coliform/*E. coli*) (9223B)

a) Drinking water—Verify at least 5% of total coliform positive results from enzyme substrate coliform tests by inoculating growth from a known positive sample and testing for lactose fermentation or for β-D-galactopyranosidase by the *o*-nitrophenyl-β-D-galactopyranoside (ONPG) test and indophenol by the cyto-

TABLE 9020:VII. DAILY CHECKS ON PRECISION OF DUPLICATE COUNTS*

Date of Analysis	Duplicate Analyses		Logarithms of Counts		Range of Logarithms	Acceptance of Range†
	D_1	D_2	L_1	L_2		
8/29	71	65	1.8513	1.8129	0.0383	A
8/30	110	121	2.0414	2.0828	0.0414	A
8/31	73	50	1.8633	1.6990	0.1643	U

* Precision criterion = (3.27$\overline{R}$) = 0.1566.
† A = acceptable; U = unacceptable.

chrome oxidase (CO) test. See 9225D for these tests. Coliforms are ONPG-positive and cytochrome-oxidase-negative. Verify *E. coli* using the EC MUG test (see 9221F).

b) Other water types—Verify at least 10% of total coliform positive samples as in ¶ 2*a* above.

3) Fecal streptococci procedure—Verify as in 9230C.5. Growth of catalase-negative, gram-positive cocci on bile esculin agar at 35°C and in brain-heart infusion broth at 45°C verifies the organisms as fecal streptococci. Growth at 45°C and in 6.5% NaCl broth indicates the streptococci are members of the enterococcus group.

4) Include known positive and negative pure cultures as a QC check.

b. Membrane filter methods:

1) Total coliform procedures

a) Drinking water—Pick all, up to 5 typical and 5 atypical (nonsheen) colonies from positive samples on M-Endo medium and verify as in 9222B.5*g*. Also verify any positives for fecal coliforms or *E. coli*. If there are no positive samples, test at least one known positive source water quarterly.

b) Other water types—Verify positives monthly by picking at least 10 sheen colonies from a positive water sample as in 9222B.5*g*. Adjust counts based on percent verification.

c) To determine false negatives, pick representative atypical colonies of different morphological types and verify as in 9222B.5*g*.

2) Fecal coliform procedure

a) Verify positives monthly by picking at least 10 blue colonies from one positive sample. Verify in lauryl tryptose broth and EC broth as in 9221B.3 and 9221E. Adjust counts based on percent verification.

b) To determine false negatives, pick representative atypical colonies of different morphological types and verify as in 9221B.3 and 9221E.

3) *Escherichia coli* procedure

a) Drinking water—Verify at least 5% of MUG-positive and MUG-negative results. Pick from well-isolated sheen colonies that fluoresce on nutrient agar with MUG (NA MUG), taking care not to pick up medium, which can cause a false positive response. Also verify nonsheen colonies that fluoresce. Verify by performing the citrate test and the indole test as described in 9225D, but incubate indole test at 44.5°C. *E. coli* are indole-positive and yield no growth on citrate.

b) Other water types—Verify one positive sample monthly as in ¶ a) above. Adjust counts based on percentage of verification.

4) Fecal streptococci procedure—Pick to verify monthly at least 10 isolated esculin-positive red colonies from m-Enterococcus agar to brain heart infusion (BHI) media. Verify as described in 9230C. Adjust counts based on percentage of verification.

5) *Enterococci* procedures—Pick to verify monthly at least 10 well-isolated pink to red colonies with black or reddish-brown precipitate from EIA agar. Transfer to BHI media as described in 9230C. Adjust counts based on percentage of verification.

6) Include known positive and negative pure cultures as a quality control check.

10. Documentation and Recordkeeping

a. QA plan: The QA program documents management's commitment to a QA policy and sets forth the requirements needed to support program objectives. The program describes overall policies, organization, objectives, and functional responsibilities for achieving the quality goals. In addition, the program should develop a project plan that specifies the QC requirements for each project. The plan specifies the QC activities required to achieve the data representativeness, completeness, comparability, and compatibility. Also, the QA plan should include a program implementation plan that ensures maximum coordination and integration of QC activities within the overall program (sampling, analyses, and data handling).

b. Sampling records: A written SOP for sample handling records sample collection, transfer, storage, analyses, and disposal. The record is most easily kept on a series of printed forms that prompt the user to provide all the necessary information. It is especially critical that this record be exact and complete if there is any chance that litigation may occur. Such record systems are called chain of custody. Because laboratories do not always know whether analytical results will be used in future litigation, some maintain chain-of-custody on all samples. Details on chain of custody are available in Section 1060B and elsewhere.[1]

c. Recordkeeping: An acceptable recordkeeping system provides needed information on sample collection and preservation, analytical methods, raw data, calculations through reported results, and a record of persons responsible for sampling and analyses. Choose a format agreeable to both the laboratory and the customer (the data user). Ensure that all data sheets are signed and dated by the analyst and the supervisor. The preferable record form is a bound and page-numbered notebook, with entries in ink and a single line drawn through any change with the correction entered next to it.

Keep records of microbiological analyses for at least 5 years. Actual laboratory reports may be kept, or data may be transferred to tabular summaries, provided that the following information is included: date, place, and time of sampling, name of sample collector; identification of sample; date of receipt of sample and analysis; person(s) responsible for performing analysis; analytical method used; the raw data and the calculated results of analysis. Verify that each result was entered correctly from the bench sheet and initialed by the analyst. If an information storage and retrieval system is used, double check data on the printouts.

11. Data Handling

a. Distribution of bacterial populations: In most chemical analyses the distribution of analytical results follows the Gaussian curve, which has symmetrical distribution of values about the mean (see Section 1010B). Microbial distributions are not necessarily symmetrical. Bacterial counts often are characterized as having a skewed distribution because of many low values and a few high ones. These characteristics lead to an arithmetic mean that is considerably larger than the median. The frequency curve of this distribution has a long right tail, such as that shown in Figure 9020:1, and is said to display positive skewness.

Application of the most rigorous statistical techniques requires the assumption of symmetrical distributions such as the normal curve. Therefore it usually is necessary to convert skewed data so that a symmetrical distribution resembling the normal distribution results. An approximately normal distribution can be obtained from positively skewed data by converting numbers to their logarithms, as shown in Table 9020:VIII. Comparison of the fre-

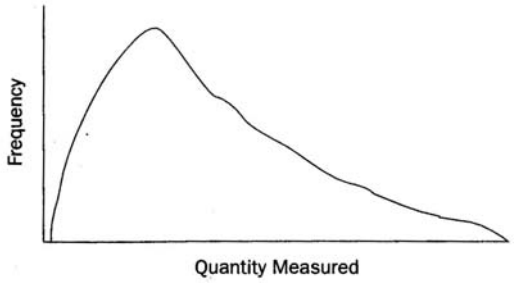


Figure 9020:1. Frequency curve (positively skewed distribution).

quency tables for the original data (Table 9020:IX) and their logarithms (Table 9020:X) shows that the logarithms approximate a symmetrical distribution.

b. Central tendency measures of skewed distribution: If the logarithms of numbers from a positively skewed distribution are approximately normally distributed, the original data have a log-normal distribution. The best estimate of central tendency of log-normal data is the geometric mean, defined as:

$$\bar{x}_g = n\sqrt{(x_1)(x_2)\cdots(x_n)}$$

and

$$\log \bar{x}_g = \frac{\sum (\log x_i)}{n}$$

that is, the geometric mean is equal to the antilog of the arithmetic mean of the logarithms. For example, the following means

TABLE 9020:VIII. COLIFORM COUNTS AND THEIR LOGARITHMS

MPN Coliform Count *No./100 mL*	log MPN
11	1.041
27	1.431
36	1.556
48	1.681
80	1.903
85	1.929
120	2.079
130	2.114
136	2.134
161	2.207
317	2.501
601	2.779
760	2.881
1020	3.009
3100	3.491
$\bar{x}$ = 442	$\bar{x}_g$ = antilog 2.1825 = 152

TABLE 9020:IX. COMPARISON OF FREQUENCY OF MPN DATA

Class Interval	Frequency (MPN)
0 to 400	11
400 to 800	2
800 to 1200	1
1200 to 1600	0
1600 to 2000	0
2000 to 2400	0
2400 to 2800	0
2800 to 3200	0

calculated from the data in Table 9020:VIII are drastically different.

$$\log \bar{x}_g = \frac{\sum (\log x_i)}{n} = \frac{32.737}{15} = 2.1825$$

geometric mean

$$\bar{x}_g = \text{antilog } (2.1825) = 152$$

and arithmetic mean

$$\bar{x} = \frac{\sum x_i}{n} = \frac{6632}{15} = 442$$

Therefore, although regulations or tradition may require or cause microbiological data to be reported as the arithmetic mean or median, the preferred statistic for summarizing microbiological monitoring data is the geometric mean. An exception may be in the evaluation of data for risk assessment. The arithmetic mean may be a better measure for this purpose because it may generate a higher central tendency value and possibly provide a greater safety factor.[8]

c. "Less than" (<) values: There has always been uncertainty as to the proper way to include "less than" values in calculation and evaluation of microbiological data because such values cannot be treated statistically without modification. Proposed modifications involve changing such numbers to zero, choosing values halfway between zero and the "less than" value, or assigning the "less than" value itself, i.e., changing <1 values to 1, ½, or 0.

There are valid reasons for not including < values, whether modified or not. If the database is fairly large with just a few < values, the influence of these uncertain values will be minimal

TABLE 9020:X. COMPARISON OF FREQUENCY OF LOG MPN DATA

Class Interval	Frequency (log MPN)
1.000 to 1.300	1
1.300 to 1.600	2
1.600 to 1.900	1
1.900 to 2.200	5
2.200 to 2.500	1
2.500 to 2.800	2
2.800 to 3.100	2
3.100 to 3.400	0
3.400 to 3.700	1

and of no benefit. If the database is small or has a relatively large number of < values, inclusion of modified < values would exert an undue influence on the final results and could result in an artificial negative or positive bias. Including < values is particularly inappropriate if the < values are <100, <1000, or higher because the unknown true values could be anywhere from 0 to 99, 0 to 999, etc. When < values are first noted, adjust or expand test volumes. The only exception to this caution would be regulatory testing with defined compliance limits, such as the <1/100 mL values reported for drinking water systems where the 100-mL volume is required.

12. References

1. Bordner, R.H., J.A. Winter & P.V. Scarpino, eds. 1978. Microbiological Methods for Monitoring the Environment, Water and Wastes. EPA-600/8-78-017, Environmental Monitoring & Support Lab., U.S. Environmental Protection Agency, Cincinnati, Ohio.
2. American Society for Testing and Materials. 1995. Standard guide for good laboratory practices in laboratories engaged in sampling and analysis of water. D-3856-95, Annual Book of ASTM Standards, Vol. 11.01, American Soc. Testing & Materials, Philadelphia, Pa.
3. American Society for Testing and Materials. 1996. Standard practice for writing quality control specifications for standard test methods for water analysis. D-5847-96, Annual Book of ASTM Standards, Vol. 11.01, American Soc. Testing & Materials, West Conshohocken, Pa.
4. American Society for Testing and Materials. 1993. Annual Book of ASTM Standards, Vol. 14.02, General Methods and Instrumentation. E-319-86 (reapproved 1993), Standard Practice for Evaluation of Single-Pan Mechanical Balances, and E-898-88 (reapproved 1993), Standard Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances. American Soc. Testing & Materials, Philadelphia, Pa.
5. Schmitz, S., C. Garbe, B. Tebbe & C. Orfanos. 1994. Long wave ultraviolet radiation (UVA) and skin cancer. *Hautarzt* 45:517.
6. Brenner, K. & C.C. Rankin. 1990. New screening test to determine the acceptability of 0.45 µm membrane filters for analysis of water. *Appl. Environ. Bacteriol.* 56:54.
7. American Society for Testing and Materials. 1977. Annual Book of ASTM Standards. Part 31, Water. American Soc. Testing & Materials, Philadelphia, Pa.
8. Haas, C.N. 1996. How to average microbial densities to characterize risk. *Water Res.* 30:1036.

9020 C. Interlaboratory Quality Control

1. Background

Interlaboratory QC programs are a means of establishing an agreed-upon, common performance criteria system that will assure an acceptable level of data quality and comparability among laboratories with similar interests and/or needs.

These systems may be volunteer, such as that for the cities in the Ohio River Valley Water Sanitation Commission (ORSANCO), or regulatory, such as the Federal Drinking Water Laboratory Certification Program (see below). Often, the term "accreditation" is used interchangeably with certification. Usually, interlaboratory quality control programs have three elements: uniform criteria for laboratory operations, external review of the program, and external proficiency testing.

2. Uniform Criteria

Interlaboratory quality control programs begin as a volunteer or mandatory means of establishing uniform laboratory standards for a specific purpose. The participants may be from one organization or a group of organizations having common interests or falling under common regulations. Often one group or person may agree to draft the criteria. If under regulation, the regulating authority may set the criteria for compliance-monitoring analyses.

Uniform sampling and analytical methods and quality control criteria for personnel, facilities, equipment, instrumentation, supplies, and data handling and reporting are proposed, discussed, reviewed, modified if necessary, and approved by the group for common use. Criteria identified as necessary for acceptable data quality should be mandatory. A formal document is prepared and provided to all participants.

The QA/QC responsibilities of management, supervisors, and technical staff are described in 9020A. In large laboratories, a QA officer is assigned as a staff position but may be the supervisor or other senior person in smaller laboratories.

After incorporation into laboratory operations and confirmation that the QA program has been adapted and is in routine use, the laboratory supervisor and the QA officer conduct an internal program review of all operations and records for acceptability, to identify possible problems and assist in their resolution. If this is done properly, there should be little concern that subsequent external reviews will find major problems.

3. External Program Review

Once a laboratory has a QA program in place, management informs the organization and a qualified external QA person or team arranges an on-site visit to evaluate the QA program for acceptability and to work with the laboratory to solve any problems. An acceptable rating confirms that the laboratory's QA program is operating properly and that the laboratory has the capability of generating valid defensible data. Such on-site evaluations are repeated and may be announced or unannounced.

4. External Proficiency Testing

Whenever practical, the external organization conducts formal performance evaluation studies among all participant laboratories. Challenge samples are prepared and sent as unknowns on a set schedule for analyses and reporting of results. The reported data are coded for confidentiality and evaluated according to an agreed-upon scheme. The results are summarized for all laboratories and individual laboratory reports are sent to participants. Results of such studies indicate the quality of routine analyses of each laboratory as compared to group performance. Also, results of the group as a whole characterize the performance that can be expected for the analytical methods tested.

5. Example Program

In the Federal Drinking Water Laboratory Certification Program, public water supply laboratories must be certified according to minimal criteria and procedures and quality assurance described in the EPA manual on certification:[1] criteria are established for laboratory operations and methodology; on-site inspections are required by the certifying state agency or its surrogate to verify minimal standards; annually, laboratories are required to perform acceptably on unknown samples in formal studies, as samples are available; the responsible authority follows up on problems identified in the on-site inspection or performance evaluation and requires corrections within a set period of time. Individual state programs may exceed the federal criteria.

On-site inspections of laboratories in the present certification program show that primary causes for discrepancies in drinking water laboratories have been inadequate equipment, improperly prepared media, incorrect analytical procedures, and insufficiently trained personnel.

6. References

1. U.S. ENVIRONMENTAL PROTECTION AGENCY. 1997. Manual for the Certification of Laboratories Analyzing Drinking Water, 4th ed. EPA-814B-92-002, U.S. Environmental Protection Agency, Cincinnati, Ohio.

9030 LABORATORY APPARATUS*

9030 A. Introduction

This section contains specifications for microbiological laboratory equipment. For testing and maintenance procedures related to quality control, see Section 9020.

* Approved by Standard Methods Committee, 1993.

9030 B. Equipment Specifications

1. Incubators

Incubators must maintain a uniform and constant temperature at all times in all areas, that is, they must not vary more than ±0.5°C in the areas used. Obtain such accuracy by using a water-jacketed or anhydric-type incubator with thermostatically controlled low-temperature electric heating units properly insulated and located in or adjacent to the walls or floor of the chamber and preferably equipped with mechanical means of circulating air.

Incubators equipped with high-temperature heating units are unsatisfactory, because such sources of heat, when improperly placed, frequently cause localized overheating and excessive drying of media, with consequent inhibition of bacterial growth. Incubators so heated may be operated satisfactorily by replacing high-temperature units with suitable wiring arranged to operate at a lower temperature and by installing mechanical air-circulation devices. It is desirable, where ordinary room temperatures vary excessively, to keep laboratory incubators in special rooms maintained at a few degrees below the recommended incubator temperature.

Alternatively, use special incubating rooms well insulated and equipped with properly distributed heating units, forced air circulation, and air exchange ports, provided that they conform to desired temperature limits. When such rooms are used, record the daily temperature range in areas where plates or tubes are incubated. Provide incubators with open metal wire or perforated sheet shelves so spaced as to assure temperature uniformity throughout the chamber. Leave a 2.5-cm space between walls and stacks of dishes or baskets of tubes.

Maintain an accurate thermometer, traceable to the National Institute of Standards and Technology (NIST), with the bulb immersed in liquid (glycerine, water, or mineral oil) on each shelf in use within the incubator and record daily temperature readings (preferably morning and afternoon). It is desirable, in addition, to maintain a maximum and minimum registering thermometer within the incubator on the middle shelf to record the gross temperature range over a 24-h period. At intervals, determine temperature variations within the incubator when filled to maximum capacity. Install a recording thermometer whenever possible, to maintain a continuous and permanent record of temperature.

Ordinarily, a water bath with a gabled cover to reduce water and heat loss, or a solid heat sink incubator, is required to maintain a temperature of 44.5 ± 0.2°C. If satisfactory temperature control is not achieved, provide water recirculation. Keep water

depth in the incubator sufficient to immerse tubes to upper level of media.

2. Hot-Air Sterilizing Ovens

Use hot-air sterilizing ovens of sufficient size to prevent internal crowding; constructed to give uniform and adequate sterilizing temperatures of 170 ± 10°C; and equipped with suitable thermometers. Optionally use a temperature-recording instrument.

3. Autoclaves

Use autoclaves of sufficient size to prevent internal crowding; constructed to provide uniform temperatures within the chambers (up to and including the sterilizing temperature of 121°C); equipped with an accurate thermometer the bulb of which is located properly on the exhaust line so as to register minimum temperature within the sterilizing chambers (temperature-recording instrument is optional); equipped with pressure gauge and properly adjusted safety valves connected directly with saturated-steam supply lines equipped with appropriate filters to remove particulates and oil droplets or directly to a suitable special steam generator (do not use steam from a boiler treated with amines for corrosion control); and capable of reaching the desired temperature within 30 min. Confirm, by chemical or toxicity tests, that the steam supply has not been treated with amines or other corrosion-control chemicals that will impart toxicity.

Use of a vertical autoclave or pressure cooker is not recommended because of difficulty in adjusting and maintaining sterilization temperature and the potential hazard. If a pressure cooker is used in emergency or special circumstances, equip it with an efficient pressure gauge and a thermometer the bulb of which is 2.5 cm above the water level.

4. Gas Sterilizers

Use a sterilizer equipped with automatic controls capable of carrying out a complete sterilization cycle. As a sterilizing gas use ethylene oxide (CAUTION: *Ethylene oxide is toxic—avoid inhalation, ingestion, and contact with the skin. Also, ethylene oxide forms an explosive mixture with air at 3-80% proportion.*) diluted to 10 to 12% with an inert gas. Provide an automatic control cycle to evacuate sterilizing chamber to at least 0.06 kPa, to hold the vacuum for 30 min, to adjust humidity and temperature, to charge with the ethylene oxide mixture to a pressure dependent on mixture used, to hold such pressure for at least 4 h, to vent gas, to evacuate to 0.06 kPa, and finally, to bring to atmospheric pressure with sterile air. The humidity, temperature, pressure, and time of sterilizing cycle depend on the gas mixture used.

Store overnight sample bottles with loosened caps that were sterilized by gas, to allow last traces of gas mixture to dissipate. Incubate overnight media sterilized by gas, to insure dissipation of gas.

In general, mixtures of ethylene oxide with chlorinated hydrocarbons such as freon are harmful to plastics, although at temperatures below 55°C, gas pressure not over 35 kPa, and time of sterilization less than 6 h, the effect is minimal. If carbon dioxide is used as a diluent of ethylene oxide, increase exposure time and pressure, depending on temperature and humidity that can be used.

Determine proper cycle and gas mixture for objects to be sterilized and confirm by sterility tests.

5. Optical Counting Equipment

a. Pour and spread plates: Use Quebec-type colony counter, dark-field model preferred, or one providing equivalent magnification (1.5 diameters) and satisfactory visibility.

b. Membrane filters: Use a binocular microscope with magnification of 10 to 15×. Provide daylight fluorescent light source at angle of 60 to 80° above the colonies; use low-angle lighting for nonpigmented colonies.

c. Mechanical tally.

6. pH Equipment

Use electrometric pH meters, accurate to at least 0.1 pH units, for determining pH values of media.

7. Balances

Use balances providing a sensitivity of at least 0.1 g at a load of 150 g, with appropriate weights. Use an analytical balance having a sensitivity of 1 mg under a load of 10 g for weighing small quantities (less than 2 g) of materials. Single-pan rapid-weigh balances are most convenient.

8. Media Preparation Utensils

Use borosilicate glass or other suitable noncorrosive equipment such as stainless steel. Use glassware that is clean and free of residues, dried agar, or other foreign materials that may contaminate media.

9. Pipets and Graduated Cylinders

Use pipets of any convenient size, provided that they deliver the required volume accurately and quickly. The error of calibration for a given manufacturer's lot must not exceed 2.5%. Use pipets having graduations distinctly marked and with unbroken tips. Bacteriological transfer pipets or pipets conforming to APHA standards may be used. *Do not pipet by mouth; use a pipet aid.*

Use graduated cylinders meeting ASTM Standards (D-86 and D-216) and with accuracy limits established by NIST where appropriate.

10. Pipet Containers

Use boxes of aluminum or stainless steel, end measurement 5 to 7.5 cm, cylindrical or rectangular, and length about 40 cm. When these are not available, paper wrappings for individual pipets may be substituted. To avoid excessive charring during sterilization, use best-quality sulfate pulp (kraft) paper. *Do not use copper or copper alloy cans or boxes as pipet containers.*

11. Refrigerator

Use a refrigerator maintaining a temperature of 1 to 4.4°C to store samples, media, reagents, etc. Do not store volatile solvents, food, or beverages in a refrigerator with media. Frost-free refrig-

erators may cause excessive media dehydration on storage longer than 1 week.

12. Temperature-Monitoring Devices

Use glass or metal thermometers graduated to 0.5°C to monitor most incubators and refrigerators. Use thermometers graduated to 0.1°C for incubators operated above 40°C. Use continuous recording devices that are equally sensitive. Verify accuracy by comparison with a NIST-certified thermometer, or equivalent.

13. Dilution Bottles or Tubes

Use bottles or tubes of resistant glass, preferably borosilicate glass, closed with glass stoppers or screw caps equipped with liners that do not produce toxic or bacteriostatic compounds on sterilization. Do not use cotton plugs as closures. Mark graduation levels indelibly on side of dilution bottle or tube. Plastic bottles of nontoxic material and acceptable size may be substituted for glass provided that they can be sterilized properly.

14. Petri Dishes

For the plate count, use glass or plastic petri dishes about 100 × 15 mm. Use dishes the bottoms of which are free from bubbles and scratches and flat so that the medium will be of uniform thickness throughout the plate. For the membrane filter technique use loose-lid glass or plastic dishes, 60 × 15 mm, or tight-lid dishes, 50 × 12 mm. Sterilize petri dishes and store in metal cans (aluminum or stainless steel, but not copper), or wrap in paper—preferably best-quality sulfate pulp (kraft)—before sterilizing. Presterilized petri dishes are commercially available.

15. Membrane Filtration Equipment

Use filter funnel and membrane holder made of seamless stainless steel, glass, or autoclavable plastic that does not leak and is not subject to corrosion. Field laboratory kits are acceptable but standard laboratory filtration equipment and procedures are required.

16. Fermentation Tubes and Vials

Use fermentation tubes of any type, if their design permits conforming to medium and volume requirements for concentration of nutritive ingredients as described subsequently. Where tubes are used for a test of gas production, enclose a shell vial, inverted. Use tube and vial of such size that the vial will be filled completely with medium, at least partly submerged in the tube, and large enough to make gas bubbles easily visible.

17. Inoculating Equipment

Use wire loops made of 22- or 24-gauge nickel alloy* or platinum-iridium for flame sterilization. Use loops at least 3 mm in diameter. Sterilize by dry heat or steam. Single-service hardwood or plastic applicators also may be used. Make these 0.2 to 0.3 cm in diameter and at least 2.5 cm longer than the fermentation tube; sterilize by dry heat and store in glass or other nontoxic containers.

* Chromel, nichrome, or equivalent.

18. Sample Bottles

For bacteriological samples, use sterilizable bottles of glass or plastic of any suitable size and shape. Use bottles capable of holding a sufficient volume of sample for all required tests and an adequate air space, permitting proper washing, and maintaining samples uncontaminated until examinations are completed. Ground-glass-stoppered bottles, preferably wide-mouthed and of resistant glass, are recommended. Plastic bottles of suitable size, wide-mouthed, and made of nontoxic materials such as polypropylene that can be sterilized repeatedly are satisfactory as sample containers. Presterilized plastic bags, with or without dechlorinating agent, are available commercially and may be used. Plastic containers eliminate the possibility of breakage during shipment and reduce shipping weight.

Metal or plastic screw-cap closures with liners may be used on sample bottles provided that no toxic compounds are produced on sterilization.

Before sterilization, cover tops and necks of sample bottles having glass closures with aluminium foil or heavy kraft paper.

19. Bibliography

COLLINS, W.D. & H.B. RIFFENBURG. 1923. Contamination of water samples with material dissolved from glass containers. *Ind. Eng. Chem.* 15:48.

CLARK, W.M. 1928. The Determination of Hydrogen Ion Concentration, 3rd ed. Williams & Wilkins, Baltimore, Md.

ARCHAMBAULT, J., J. CUROT & M.H. MCCRADY. 1937. The need of uniformity of conditions for counting plates (with suggestions for a standard colony counter). *Amer. J. Pub. Health* 27:809.

BARKWORTH, H. & J.O. IRWIN. 1941. The effect of the shape of the container and size of gas tube in the presumptive coliform test. *J. Hyg.* 41:180.

RICHARDS, O.W. & P.C. HEIJN. 1945. An improved dark-field Quebec colony counter. *J. Milk Technol.* 8:253.

COHEN, B. 1957. The measurement of pH, titratable acidity, and oxidation-reduction potentials. *In* Manual of Microbiological Methods. Society of American Bacteriologists. McGraw-Hill Book Co., New York, N.Y.

MORTON, H.E. 1957. Stainless-steel closures for replacement of cotton plugs in culture tubes. *Science.* 126:1248.

MCGUIRE, O.E. 1964. Wood applicators for the confirmatory test in the bacteriological analysis of water. *Pub. Health Rep.* 79:812.

BORDNER, R.H., J.A. WINTER & P.V. SCARPINO, eds. 1978. Microbiological Methods for Monitoring the Environment, Water and Wastes. EPA-600/8-78-017, Environmental Monitoring & Support Lab., U.S. Environmental Protection Agency, Cincinnati, Ohio.

AMERICAN PUBLIC HEALTH ASSOCIATION. 1993. Standard Methods for the Examination of Dairy Products, 16th ed. American Public Health Assoc., Washington, D.C.

9040 WASHING AND STERILIZATION*

Cleanse all glassware thoroughly with a suitable detergent and hot water, rinse with hot water to remove all traces of residual washing compound, and finally rinse with laboratory-pure water. If mechanical glassware washers are used, equip them with influent plumbing of stainless steel or other nontoxic material. Do not use copper piping to distribute water. Use stainless steel or other nontoxic material for the rinse water system.

Sterilize glassware, except when in metal containers, for not less than 60 min at a temperature of 170°C, unless it is known from recording thermometers that oven temperatures are uniform, under which exceptional condition use 160°C. Heat glassware in metal containers to 170°C for not less than 2 h.

Sterilize sample bottles not made of plastic as above or in an autoclave at 121°C for 15 min.

For plastic bottles loosen caps before autoclaving to prevent distortion.

* Approved by Standard Methods Committee, 1993.

9050 PREPARATION OF CULTURE MEDIA*

9050 A. General Procedures

1. Storage of Culture Media

Store dehydrated media (powders) in tightly closed bottles in the dark at less than 30°C in an atmosphere of low humidity. Do not use them if they discolor or become caked and lose the character of a free-flowing powder. Purchase dehydrated media in small quantities that will be used within 6 months after opening. Additionally, use stocks of dehydrated media containing selective agents such as sodium azide, bile salts or derivatives, antibiotics, sulfur-containing amino acids, etc., of relatively current lot number (within a year of purchase) so as to maintain optimum selectivity. See also Section 9020.

Prepare culture media in batches that will be used in less than 1 week. However, if the media are contained in screw-capped tubes they may be stored for up to 3 months. See Table 9020:IV for specific details. Store media out of direct sun and avoid contamination and excessive evaporation.

Liquid media in fermentation tubes, if stored at refrigeration or even moderately low temperatures, may dissolve sufficient air to produce, upon incubation at 35°C, a bubble of air in the tube. Incubate fermentation tubes that have been stored at a low temperature overnight before use and discard tubes containing air.

Fermentation tubes may be stored at approximately 25°C; but because evaporation may proceed rapidly under these conditions—resulting in marked changes in concentration of the ingredients—do not store at this temperature for more than 1 week. Discard tubes with an evaporation loss exceeding 1 mL.

2. Adjustment of Reaction

State reaction of culture media in terms of hydrogen ion concentration, expressed as pH.

The decrease in pH during sterilization will vary slightly with the individual sterilizer in use, and the initial reaction required to obtain the correct final reaction will have to be determined. The decrease in pH usually will be 0.1 to 0.2 but occasionally may be as great as 0.3 in double-strength media. When buffering salts such as phosphates are present in the media, the decrease in pH value will be negligible.

Make tests to control adjustment to required pH with a pH meter. Measure pH of prepared medium as directed in Section 4500-H^+. Titrate a known volume of medium with a solution of NaOH to the desired pH. Calculate amount of NaOH solution that must be added to the bulk medium to reach this reaction. After adding and mixing thoroughly, check reaction and adjust if necessary. The required final pH is given in the directions for preparing each medium. If a specific pH is not prescribed, adjustment is unnecessary.

The pH of reconstituted dehydrated media seldom will require adjustment if made according to directions. Such factors as errors in weighing dehydrated medium or overheating reconstituted medium may produce an unacceptable final pH. Measure pH, especially of rehydrated selective media, regularly to insure quality control and media specifications.

3. Sterilization

After rehydrating a medium, dispense promptly to culture vessels and sterilize within 2 h. Do not store nonsterile media.

Sterilize all media, except sugar broths or broths with other specifications, in an autoclave at 121°C for 15 min after the temperature has reached 121°C. When the pressure reaches zero, remove medium from autoclave and cool quickly to avoid decomposition of sugars by prolonged exposure to heat. To permit uniform heating and rapid cooling, pack materials loosely and in small containers. Sterilize sugar broths at 121°C for 12 to 15 min. The maximum elapsed time for exposure of sugar broths to any heat (from time of closing loaded autoclave to unloading) is 45 min. Preferably use a double-walled autoclave to permit preheating before loading to reduce total needed heating time to within the 45-min limit. Presterilized media may be available commercially.

* Approved by Standard Methods Committee, 1993.

4. Bibliography

BUNKER, G.C. & H. SCHUBER. 1922. The reaction of culture media. *J. Amer. Water Works Assoc.* 9:63.

RICHARDSON, G.H., ed. 1985. Standard Methods for the Examination of Dairy Products, 15th ed. American Public Health Assoc., Washington, D.C.

BALOWS, A., W.J. HAUSLER, JR., K.L. HERRMANN, H.D. ISENBERG & H.J. SHADOMY, eds. 1991. Manual of Clinical Microbiology, 5th ed. American Soc. Microbiology, Washington, D.C.

9050 B. Water

1. Specifications

To prepare culture media and reagents, use only distilled or demineralized reagent-grade water that has been tested and found free from traces of dissolved metals and bactericidal or inhibitory compounds. Toxicity in distilled water may be derived from fluoridated water high in silica. Other sources of toxicity are silver, lead, and various unidentified organic complexes. Where condensate return is used as feed for a still, toxic amines or other boiler compounds may be present in distilled water. Residual chlorine or chloramines also may be found in distilled water prepared from chlorinated water supplies. If chlorine compounds are found in distilled water, neutralize them by adding an equivalent amount of sodium thiosulfate or sodium sulfite.

Distilled water also should be free of contaminating nutrients. Such contamination may be derived from flashover of organics during distillation, continued use of exhausted carbon filter beds, deionizing columns in need of recharging, solder flux residues in new piping, dust and chemical fumes, and storage of water in unclean bottles. Store distilled water out of direct sunlight to prevent growth of algae and turn supplies over as rapidly as possible. Aged distilled water may contain toxic volatile organic compounds absorbed from the atmosphere if stored for prolonged periods in unsealed containers. Good housekeeping practices usually will eliminate nutrient contamination.

See Section 9020.

2. Bibliography

STRAKA, R.P. & J.L. STOKES. 1957. Rapid destruction of bacteria in commonly used diluents and its elimination. *Appl. Microbiol.* 5:21.

GELDREICH, E.E. & H.F. CLARK. 1965. Distilled water suitability for microbiological applications. *J. Milk Food Technol.* 28:351.

MACLEOD, R.A., S.C. KUO & R. GELINAS. 1967. Metabolic injury to bacteria. II. Metabolic injury induced by distilled water or Cu^{++} in the plating diluent. *J. Bacteriol.* 93:961.

9050 C. Media Specifications

The need for uniformity dictates the use of dehydrated media. Never prepare media from basic ingredients when suitable dehydrated media are available. Follow manufacturer's directions for rehydration and sterilization. Commercially prepared media in liquid form (sterile ampule or other) also may be used if known to give equivalent results. See Section 9020 for quality-control specifications.

The terms used for protein source in most media, for example, peptone, tryptone, tryptose, were coined by the developers of the media and may reflect commercial products rather than clearly defined entities. It is not intended to preclude the use of alternative materials provided that they produce equivalent results.

NOTE—The term "percent solution" as used in these directions is to be understood to mean "grams of solute per 100 mL solution."

1. Dilution Water

a. Buffered water: To prepare stock phosphate buffer solution, dissolve 34.0 g potassium dihydrogen phosphate (KH_2PO_4), in 500 mL reagent-grade water, adjust to pH 7.2 ± 0.5 with 1*N* sodium hydroxide (NaOH), and dilute to 1 L with reagent-grade water.

Add 1.25 mL stock phosphate buffer solution and 5.0 mL magnesium chloride solution (81.1 g $MgCl_2 \cdot 6H_2O$/L reagent-grade water) to 1 L reagent-grade water. Dispense in amounts that will provide 99 ± 2.0 mL or 9 ± 0.2 mL after autoclaving for 15 min.

b. Peptone water: Prepare a 10% solution of peptone in distilled water. Dilute a measured volume to provide a final 0.1% solution. Final pH should be 6.8.

Dispense in amounts to provide 99 ± 2.0 mL or 9 ± 0.2 mL after autoclaving for 15 min.

Do not suspend bacteria in any dilution water for more than 30 min at room temperature because death or multiplication may occur.

2. Culture Media

Specifications for individual media are included in subsequent sections. Details are provided where use of a medium first is described.

9060 SAMPLES*

9060 A. Collection

1. Containers

Collect samples for microbiological examination in nonreactive borosilicate glass or plastic bottles that have been cleansed and rinsed carefully, given a final rinse with deionized or distilled water, and sterilized as directed in Sections 9030 and 9040. For some applications samples may be collected in presterilized plastic bags.

2. Dechlorination

Add a reducing agent to containers intended for the collection of water having residual chlorine or other halogen unless they contain broth for direct planting of sample. Sodium thiosulfate ($Na_2S_2O_3$) is a satisfactory dechlorinating agent that neutralizes any residual halogen and prevents continuation of bactericidal action during sample transit. The examination then will indicate more accurately the true microbial content of the water at the time of sampling.

For sampling chlorinated wastewater effluents add sufficient $Na_2S_2O_3$ to a clean sterile sample bottle to give a concentration of 100 mg/L in the sample. In a 120-mL bottle 0.1 mL of a 10% solution of $Na_2S_2O_3$ will neutralize a sample containing about 15 mg/L residual chlorine. For drinking water samples, the concentration of dechlorinating agent may be reduced: 0.1 mL of a 3% solution of $Na_2S_2O_3$ in a 120-mL bottle will neutralize up to 5 mg/L residual chlorine.

Cap bottle and sterilize by either dry or moist heat, as directed (Section 9040). Presterilized plastic bags or bottles containing $Na_2S_2O_3$ are available commercially.

Collect water samples high in metals, including copper or zinc (>1.0 mg/L), and wastewater samples high in heavy metals in sample bottles containing a chelating agent that will reduce metal toxicity. This is particularly significant when such samples are in transit for 4 h or more. Use 372 mg/L of the disodium salt of ethylenediaminetetraacetic acid (EDTA). Adjust EDTA solution to pH 6.5 before use. Add EDTA separately to sample bottle before bottle sterilization (0.3 mL 15% solution in a 120-mL bottle) or combine it with the $Na_2S_2O_3$ solution before addition.

3. Sampling Procedures

When the sample is collected, leave ample air space in the bottle (at least 2.5 cm) to facilitate mixing by shaking, before examination. Collect samples that are representative of the water being tested, flush or disinfect sample ports, and use aseptic techniques to avoid sample contamination.

Keep sampling bottle closed until it is to be filled. Remove stopper and cap as a unit; do not contaminate inner surface of stopper or cap and neck of bottle. Fill container without rinsing, replace stopper or cap immediately, and if used, secure hood around neck of bottle.

a. Potable water: If the water sample is to be taken from a distribution-system tap without attachments, select a tap that is supplying water from a service pipe directly connected with the main, and is not, for example, served from a cistern or storage tank. Open tap fully and let water run to waste for 2 or 3 min, or for a time sufficient to permit clearing the service line. Reduce water flow to permit filling bottle without splashing. If tap cleanliness is questionable, choose another tap. If a questionable tap is required for special sampling purposes, disinfect the faucet (inside and outside) by applying a solution of sodium hypochlorite (100 mg NaOCl/L) to faucet before sampling; let water run for additional 2 to 3 min after treatment. Do not sample from leaking taps that allow water to flow over the outside of the tap. In sampling from a mixing faucet remove faucet attachments such as screen or splash guard, run hot water for 2 min, then cold water for 2 to 3 min, and collect sample as indicated above.

If the sample is to be taken from a well fitted with a hand pump, pump water to waste for about 5 to 10 min or until water temperature has stabilized before collecting sample. If an outdoor sampling location must be used, avoid collecting samples from frost-proof hydrants. If there is no pumping machinery, collect a sample directly from the well by means of a sterilized bottle fitted with a weight at the base; take care to avoid contaminating samples by any surface scum. Other sterile sampling devices, such as a trip bailer, also may be used.

In drinking water evaluation, collect samples of finished water from distribution sites selected to assure systematic coverage during each month. Carefully choose distribution system sample locations to include dead-end sections to demonstrate bacteriological quality throughout the network and to ensure that localized contamination does not occur through cross-connections, breaks in the distribution lines, or reduction in positive pressure. Sample locations may be public sites (police and fire stations, government office buildings, schools, bus and train stations, airports, community parks), commercial establishments (restaurants, gas stations, office buildings, industrial plants), private residences (single residences, apartment buildings, and townhouse complexes), and special sampling stations built into the distribution network. Preferably avoid outdoor taps, fire hydrants, water treatment units, and backflow prevention devices. Establish sampling program in consultation with state and local health authorities.

b. Raw water supply: In collecting samples directly from a river, stream, lake, reservoir, spring, or shallow well, obtain samples representative of the water that is the source of supply to consumers. It is undesirable to take samples too near the bank or too far from the point of drawoff, or at a depth above or below the point of drawoff.

c. Surface waters: Stream studies may be short-term, high-intensity efforts. Select bacteriological sampling locations to include a baseline location upstream from the study area, industrial and municipal waste outfalls into the main stream study area, tributaries except those with a flow less than 10% of the main stream, intake points for municipal or industrial water facilities, down-

* Approved by Standard Methods Committee, 1997.

stream samples based on stream flow time, and downstream recreational areas. Dispersion of wastewaters into the receiving stream may necessitate preliminary cross-section studies to determine completeness of mixing. Where a tributary stream is involved, select the sampling point near the confluence with the main stream. Samples may be collected from a boat or from bridges near critical study points. Choose sampling frequency to be reflective of changing stream or water body conditions. For example, to evaluate waste discharges, sample every 4 to 6 h and advance the time over a 7- to 10-d period.

To monitor stream and lake water quality establish sampling locations at critical sites. Sampling frequency may be seasonal for recreational waters, daily for water supply intakes, hourly where waste treatment control is erratic and effluents are discharged into shellfish harvesting areas, or even continuous.

d. Bathing beaches: Sampling locations for recreational areas should reflect water quality within the entire recreational zone. Include sites from upstream peripheral areas and locations adjacent to drains or natural contours that would discharge stormwater collections or septic wastes. Collect samples in the swimming area from a uniform depth of approximately 1 m. Consider sediment sampling of the water-beach (soil) interface because of exposure of young children at the water's edge.

To obtain baseline data on marine and estuarine bathing water quality include sampling at low, high, and ebb tides.

Relate sampling frequency directly to the peak bathing period, which generally occurs in the afternoon. Preferably, collect daily samples during the recognized bathing season; as a minimum include Friday, Saturday, Sunday, and holidays. When limiting sampling to days of peak recreational use, preferably collect a sample in the morning and the afternoon. Correlate bacteriological data with turbidity levels or rainfall over the watershed to make rapid assessment of water quality changes.

e. Sediments and biosolids: The bacteriology of bottom sediments is important in water supply reservoirs, in lakes, rivers, and coastal waters used for recreational purposes, and in shellfish-growing waters. Sediments may provide a stable index of the general quality of the overlying water, particularly where there is great variability in its bacteriological quality.

Sampling frequency in reservoirs and lakes may be related more to seasonal changes in water temperatures and stormwater runoff. Bottom sediment changes in river and estuarine waters may be more erratic, being influenced by stormwater runoff, increased flow velocities, and sudden changes in the quality of effluent discharges.

Microbiological examination of biosolids from water and wastewater treatment processes is desirable to determine the impact of their disposal into receiving waters, ocean dumping, land application, or burial in landfill operations.

Collect and handle biosolids with less than 7% total solids using the procedures discussed for other water samples. Biosolids with more than 7% solids and exhibiting a "plastic" consistency or "semisolid" state typical of thickened sludges require a finite shear stress to cause them to flow. This resistance to flow results in heterogeneous distribution of biosolids in tanks and lagoons. Use cross-section sampling of accumulated biosolids to determine distribution of organisms within these impoundments. Establish a length-width grid across the top of the impoundment, and sample at intercepts. A thief sampler that samples only the solids layer may be useful. Alternatively use weighted bottle samplers that can be opened up at a desired depth to collect samples at specific locations.

Processed biosolids having no free liquids are best sampled when they are being transferred. Collect grab samples across the entire width of the conveyor and combine into a composite sample. If solids are stored in piles, classification occurs. Exteriors of uncovered piles are subject to various environmental stresses such as precipitation, wind, fugitive dusts, and fecal contamination from scavengers. Consequently, surface samples may not reflect the microbiological quality of the pile. Therefore, use cross-section sampling of these piles to determine the degree of heterogeneity within the pile. Establish a length-width grid across the top of the pile, and sample intercepts. Sample augers and corers may prove to be ineffective for sampling piles of variable composition. In such cases use hand shovels to remove overburden.

f. Nonpotable samples (manual sampling): Take samples from a river, stream, lake, or reservoir by holding the bottle near its base in the hand and plunging it, neck downward, below the surface. Turn bottle until neck points slightly upward and mouth is directed toward the current. If there is no current, as in the case of a reservoir, create a current artificially by pushing bottle forward horizontally in a direction away from the hand. When sampling from a boat, obtain samples from upstream side of boat. If it is not possible to collect samples from these situations in this way, attach a weight to base of bottle and lower it into the water. In any case, take care to avoid contact with bank or stream bed; otherwise, water fouling may occur.

g. Sampling apparatus: Special apparatus that permits mechanical removal of bottle stopper below water surface is required to collect samples from depths of a lake or reservoir. Various types of deep sampling devices are available. The most common is the ZoBell J-Z sampler,[1] which uses a sterile 350-mL bottle and a rubber stopper through which a piece of glass tubing has been passed. This tubing is connected to another piece of glass tubing by a rubber connecting hose. The unit is mounted on a metal frame containing a cable and a messenger. When the messenger is released, it strikes the glass tubing at a point that has been slightly weakened by a file mark. The glass tube is broken by the messenger and the tension set up by the rubber connecting hose is released and the tubing swings to the side. Water is sucked into the bottle as a consequence of the partial vacuum created by sealing the unit at time of autoclaving. Commercial adaptations of this sampler and others are available.

Bottom sediment sampling also requires special apparatus. The sampler described by Van Donsel and Geldreich[2] has been found effective for a variety of bottom materials for remote (deep water) or hand (shallow water) sampling. Construct this sampler preferably of stainless steel and fit with a sterile plastic bag. A nylon cord closes the bag after the sampler penetrates the sediment. A slide bar keeps the bag closed during descent and is opened, thereby opening the bag, during sediment sampling.

For sampling wastewaters or effluents the techniques described above generally are adequate; in addition see Section 1060.

4. Size of Sample

The volume of sample should be sufficient to carry out all tests required, preferably not less than 100 mL.

5. Identifying Data

Accompany samples by complete and accurate identifying and descriptive data. Do not accept for examination inadequately identified samples.

6. References

1. ZOBELL, C.E. 1941. Apparatus for collecting water samples from different depths for bacteriological analysis. *J. Mar. Res.* 4:173.
2. VAN DONSEL, D.J. & E.E. GELDREICH. 1971. Relationships of Salmonellae to fecal coliforms in bottom sediments. *Water Res.* 5:1079.

7. Bibliography

PUBLIC HEALTH LABORATORY SERVICE WATER SUB-COMMITTEE. 1953. The effect of sodium thiosulphate on the coliform and *Bacterium coli* counts of non-chlorinated water samples. *J. Hyg.* 51:572.

SHIPE, E.L. & A. FIELDS. 1956. Chelation as a method for maintaining the coliform index in water samples. *Pub. Health Rep.* 71:974.

HOATHER, R.C. 1961. The bacteriological examination of water. *J. Inst. Water Eng.* 61:426.

COLES, H.G. 1964. Ethylenediamine tetra-acetic acid and sodium thiosulphate as protective agents for coliform organisms in water samples stored for one day at atmospheric temperature. *Proc. Soc. Water Treat. Exam.* 13:350.

DAHLING, D.R. & B.A. WRIGHT. 1984. Processing and transport of environmental virus samples. *Appl. Environ. Microbiol.* 47:1272.

U.S. ENVIRONMENTAL PROTECTION AGENCY. 1992. Environmental Regulations and Technology Control of Pathogens and Vector Attraction in Sewage Sludge. EPA-625/R-92-013. Washington, D.C.

9060 B. Preservation and Storage

1. Holding Time and Temperature

a. General: Start microbiological analysis of water samples as soon as possible after collection to avoid unpredictable changes in the microbial population. For most accurate results, ice samples during transport to the laboratory, if they cannot be processed within 1 h after collection. If the results may be used in legal action, employ special means (rapid transport, express mail, courier service, etc.) to deliver the samples to the laboratory within the specified time limits and maintain chain of custody. Follow the guidelines and requirements given below for specific water types.

b. Drinking water for compliance purposes: Preferably hold samples at <10°C during transit to the laboratory. Analyze samples on day of receipt whenever possible and refrigerate overnight if arrival is too late for processing on same day. Do not exceed 30 h holding time from collection to analysis for coliform bacteria. Do not exceed 8 h holding time for heterotrophic plate counts.

c. Nonpotable water for compliance purposes: Hold source water, stream pollution, recreational water, and wastewater samples below 10°C during a maximum transport time of 6 h. Refrigerate these samples upon receipt in the laboratory and process within 2 h. When transport conditions necessitate delays in delivery of samples longer than 6 h, consider using either field laboratory facilities located at the site of collection or delayed incubation procedures.

d. Other water types for noncompliance purposes: Hold samples below 10°C during transport and until time of analysis. Do not exceed 24 h holding time.

2. Bibliography

CALDWELL, E.L. & L.W. PARR. 1933. Present status of handling water samples—Comparison of bacteriological analyses under varying temperatures and holding conditions, with special reference to the direct method. *Amer. J. Pub. Health* 23:467.

COX, K.E. & F.B. CLAIBORNE. 1949. Effect of age and storage temperature on bacteriological water samples. *J. Amer. Water Works Assoc.* 41: 948.

PUBLIC HEALTH LABORATORY SERVICE WATER SUB-COMMITTEE. 1952. The effect of storage on the coliform and *Bacterium coli* counts of water samples. Overnight storage at room and refrigerator temperatures. *J. Hyg.* 50:107.

PUBLIC HEALTH LABORATORY SERVICE WATER SUB-COMMITTEE. 1953. The effect of storage on the coliform and *Bacterium coli* counts of water samples. Storage for six hours at room and refrigerator temperatures. *J. Hyg.* 51:559.

MCCARTHY, J.A. 1957. Storage of water sample for bacteriological examinations. *Amer. J. Pub. Health* 47:971.

LONSANE, B.K., N.M. PARHAD & N.U. RAO. 1967. Effect of storage temperature and time on the coliform in water samples. *Water Res.* 1: 309.

LUCKING, H.E. 1967. Death rate of coliform bacteria in stored Montana water samples. *J. Environ. Health* 29:576.

MCDANIELS, A.E. & R.H. BORDNER. 1983. Effect of holding time and temperature on coliform numbers in drinking water. *J. Amer. Water Works Assoc.* 75:458.

MCDANIELS, A.E. et al. 1985. Holding effects on coliform enumeration in drinking water samples. *Appl. Environ. Microbiol.* 50:755.

U.S. ENVIRONMENTAL PROTECTION AGENCY. 1985. Test Methods for *Escherichia coli* and Enterococci in Water by the Membrane Filter Procedure. EPA-600/4-85/076.

HURST, C.J. 1991. Disinfection of drinking water, swimming-pool-water and treated sewage effluent. *In* S.S. Block. Disinfection, Sterilization and Preservation, 4th ed. Lea & Febiger, Philadelphia, Pa.

9213 C. Whirlpools

1. General Discussion

a. Characteristics: A whirlpool is a shallow pool with a maximum water depth of 1.2 m; it has a closed-cycle water system, a heated water supply, and usually a hydrojet recirculation system. It may be constructed of plastic, fiberglass, redwood, or epoxy-lined surfaces. Whirlpools are designed for recreational as well as therapeutic use and may accommodate one or more bathers. These pools usually are not cleaned, drained, and refilled after each use. They are located in homes, apartments, hotels, athletic facilities, rehabilitation centers, and hospitals.

b. Monitoring requirements: Whirlpool-associated infections are common because of the inherent design and characteristics of whirlpools, which include high temperature, reduced disinfection efficacy, and increased organic material. All these factors contribute to favorable conditions for growth of microorganisms, especially *Pseudomonas aeruginosa*. Studies have shown that whirlpools can serve as a reservoir of *Legionella pneumophila*. Therefore, frequent testing for residual disinfectant levels and pH, along with scheduled maintenance, is necessary for safe whirlpool water quality.[1–5]

c. Microbiological indicators: The primary indicator of disinfection efficacy is *P. aeruginosa*, with total coliforms, heterotrophic plate count, and staphylococci as supporting indicators of water quality. The standard index of water quality, i.e., total coliforms, may be insufficient to judge the microbiological quality of whirlpool water. *Pseudomonas aeruginosa* is frequently isolated from whirlpool water that is coliform-negative.[6] In the event of a whirlpool-associated outbreak, collect samples as close as possible to the time of the outbreak. Analyze for the suspected pathogen and *P. aeruginosa*. Methods for *P. aeruginosa* are described in Sections 9213E and 9213F.

d. Sample preservation: Examine samples as soon as possible after collection. See Section 9060B.

2. References

1. CENTERS FOR DISEASE CONTROL. 1981. Suggested Health and Safety Guidelines for Public Spas and Hot Tubs. DHHS-CDC #99-960. United States Government Printing Off., Washington, D.C.
2. SOLOMON, S.L. 1985. Host factors in whirlpool-associated *Pseudomonas aeruginosa* skin disease. *Infect. Control* 6:402.
3. HIGHSMITH, A.K., P.N. LEE, R.F. KHABBAZ & V.P. MUNN. 1985. Characteristics of *Pseudomonas aeruginosa* isolated from whirlpools and bathers. *Infect. Control* 6:407.
4. GROOTHUIS, D.G., A.H. HAVELAAR & H.R. VEENENDAAL. 1985. A note on legionellas in whirlpools. *J. Appl. Bacteriol.* 58:479.
5. HIGHSMITH, A.K. & M.S. FAVERO. 1985. Microbiological aspects of public whirlpools. *Clin. Microbiol. Newsletter* 7:9.
6. HALL, N. 1984. Whirlpools and *Pseudomonas aeruginosa*. UHL Lab Hotline 21:9.

3. Bibliography

GELDREICH, E.E., A.K. HIGHSMITH & W.J. MARTONE. 1985. Public whirlpools—the epidemiology and microbiology of disease. *Infect. Control* 6:392.

9213 D. Natural Bathing Beaches

1. General Discussion

a. Characteristics: A natural bathing beach is any area of a stream, lake, ocean, impoundment, or hot spring that is used for recreation. A wide variety of pathogenic microorganisms can be transmitted to humans through use of natural fresh and marine recreational waters contaminated by wastewater.[1,2] These include enteric pathogens such as *Salmonella, Shigella,* enteroviruses, protozoa, multicellular parasites, and "opportunists" such as *P. aeruginosa, Klebsiella* sp., *Vibrio* sp., and *Aeromonas hydrophila,* which can multiply in recreational waters with sufficient nutrients. Other organisms of concern are those associated with the skin, mouth, or nose of bathers, such as *Staphylococcus aureus* and other organisms, e.g., nontuberculous mycobacteria and leptospira, and *Naegleria* sp..[3–9]

b. Monitoring requirements: Historically, fecal coliforms have been recommended as the indicator of choice for evaluating the microbiological quality of recreational waters. Many states have adopted use of this indicator in their water quality standards. Recent studies have demonstrated that *E. coli* and enterococci showed a stronger correlation with swimming-associated gastroenteritis than do fecal coliforms, and that both indicators were equally acceptable for monitoring fresh-water quality. For marine water, enterococci showed the strongest relationship of density to gastroenteritis. The recommended densities of these indicator organisms were calculated to approximate the degree of protection previously accepted for fecal coliforms. EPA-recommended water quality criteria are based on these findings.[10] While the primary indicators of water quality are *E. coli* and enterococci, the enumeration of *P. aeruginosa, Aeromonas hydrophila,* and *Klebsiella*

sp. in recreational waters may be useful in cases of discharge of pulp and paper wastes and effluents from textile finishing plants into receiving waters.

2. Samples

a. Containers: Collect samples as directed in Section 9060A. The size of the container varies with the number and variety of tests to be performed. Adding $Na_2S_2O_3$ to the bottle is unnecessary.

b. Sampling procedure: Collect samples 0.3 m below the water surface in the areas of greatest bather load. Take samples over the range of environmental and climatic conditions, especially during times when maximal pollution can be expected, i.e., periods of tidal, current, and wind influences, stormwater runoff, wastewater treatment bypasses. See Section 9213B.2*b* for methods of sample collection and Section 9222 for suggested sample volumes.

c. Sample storage: See Section 9060B.

3. Tests for *Escherichia coli*

a. Media:

1) mTEC agar:*

Proteose peptone	5.0	g
Yeast extract	3.0	g
Lactose	10.0	g
Sodium chloride, NaCl	7.5	g
Dipotassium phosphate, K_2HPO_4	3.3	g
Monopotassium phosphate KH_2PO_4	1.0	g
Sodium lauryl sulfate	0.2	g
Sodium desoxycholate	0.1	g
Bromcresol purple	0.08	g
Bromphenol red	0.08	g
Agar	15.0	g
Reagent-grade water	1	L

Sterilize by autoclaving; pH should be 7.3 ± 0.2. Pour 4 to 5 mL liquefied agar into culture dishes (50 × 10 mm). Store in refrigerator.

2) Urea substrate:*

Urea	2.0	g
Phenol red	10	mg
Reagent-grade water	100	mL

Adjust pH to between 3 and 4. Store at 2 to 8°C. Use within 1 week.

b. Procedure: Filter sample through a membrane filter (see Section 9222), place membrane on mTEC agar, incubate at 35 ± 0.5°C for 2 h to rejuvenate injured or stressed bacteria, and then incubate at 44.5 ± 0.2°C for 22 h. Transfer filter to a filter pad saturated with urea substrate. After 15 min, count yellow or yellow-brown colonies, using a fluorescent lamp and a magnifying lens. *E. coli* produces yellow or yellow-brown colonies. Verify a portion of these differentiated colonies with a commercial multitest system [see Section 9222B.5*f*2)*b*)].

* Difco or equivalent.

4. Tests for Enterococci

Perform tests for enterococci by the multiple-tube technique (Section 9230B) or membrane filter technique (Section 9230C).

5. Tests for *Pseudomonas aeruginosa*

Perform tests for *P. aeruginosa* as directed in Sections 9213E and F. Use the multiple-tube test with samples but note that the procedures may not be applicable to marine samples.

6. Tests for *Salmonella/Shigella*

See Section 9260.

7. References

1. CABELLI, V.J. 1980. Health Effects Criteria for Marine Recreational Waters. EPA-600/1-80-031, U.S. Environmental Protection Agency, Research Triangle Park, N.C.
2. DUFOUR, A.P. 1984. Health Effects Criteria for Fresh Recreational Waters. EPA-600/1-84-004, U.S. Environmental Protection Agency, Research Triangle Park, N.C.
3. KESWICK, B.H., C.P. GERBA & S.M. GOYAL. 1981. Occurrence of enteroviruses in community swimming pools. *Amer. J. Pub. Health* 71: 1026.
4. DUTKA, B.J. & K.K. KWAN. 1978. Health indicator bacteria in water surface microlayers. *Can. J. Microbiol.* 24:187.
5. CABELLI, V.J., H. KENNEDY & M.A. LEVIN. 1976. *Pseudomonas aeruginosa* and fresh recreational waters. *J. Water Pollut. Control Fed.* 48: 367.
6. SHERRY, J.P., S.R. KUCHMA & B.J. DUTKA. 1979. The occurrence of *Candida albicans* in Lake Ontario bathing beaches. *Can. J. Microbiol.* 25:1036.
7. STEVENS, A.R., R.L. TYNDALL, C.C. COUTANT & E. WILLAERT. 1977. Isolation of the etiological agent of primary amoebic meningoencephalitis from artificially heated waters. *Appl. Environ. Microbiol.* 34:701.
8. WELLINGS, F.M., P.T. AMUSO, S.L. CHANG & A.L. LEWIS. 1977. Isolation and identification of pathogenic *Naegleria* from Florida lakes. *Appl. Environ. Microbiol.* 34:661.
9. N'DIAYE, A., P. GEORGES, A. N'GO & B. FESTY. 1985. Soil amoebas as biological markers to estimate the quality of swimming pool waters. *Appl. Environ. Microbiol.* 49:1072.
10. U.S. ENVIRONMENTAL PROTECTION AGENCY. 1986. Ambient Water Quality Criteria for Bacteria—1986. EPA-440/5-84-002, U.S. Environmental Protection Agency, Washington, D.C.

8. Bibliography

OLIVIERI, V.P., C.W. DRUSE & K. KAWATA. 1977. Microorganisms in Urban Stormwater. EPA-600/2-77-087, U.S. Environmental Protection Agency, Cincinnati, Ohio.

*RICE, E.W., T.C. COVERT, D.K. WILD, D. BERMAN, S.A. JOHNSON & C.H. JOHNSON. 1993. Comparative resistance of *Escherichia coli* and *Enterococci* to chlorination. *J. Environ. Health.* A28:89.

9222 MEMBRANE FILTER TECHNIQUE FOR MEMBERS OF THE COLIFORM GROUP*

9222 A. Introduction

The membrane filter (MF) technique is highly reproducible, can be used to test relatively large sample volumes, and usually yields numerical results more rapidly than the multiple-tube fermentation procedure. The MF technique is extremely useful in monitoring drinking water and a variety of natural waters. However, the MF technique has limitations, particularly when testing waters with high turbidity or large numbers of noncoliform (background) bacteria. When the MF technique has not been used previously, it is desirable to conduct parallel tests with the method the laboratory is using currently to demonstrate applicability and comparability.

1. Definition

As related to the MF technique, the coliform group is defined as those facultative anaerobic, gram-negative, non-spore-forming, rod-shaped bacteria that develop red colonies with a metallic (golden) sheen within 24 h at 35°C on an Endo-type medium containing lactose. Some members of the total coliform group may produce dark red, mucoid, or nucleated colonies without a metallic sheen. When verified these are classified as atypical coliform colonies. When purified cultures of coliform bacteria are tested, they produce negative cytochrome oxidase and positive β-galactosidase test reactions.† Generally, pink (non-mucoid), blue, white, or colorless colonies lacking sheen are considered noncoliforms by this technique.

2. Applications

Turbidity caused by the presence of algae, particulates, or other interfering material may not permit testing of a sample volume sufficient to yield significant results. Low coliform estimates may be caused by the presence of high numbers of noncoliforms or of toxic substances. The MF technique is applicable to the examination of saline waters, but not wastewaters that have received only primary treatment followed by chlorination because of turbidity in high volume samples or wastewaters containing toxic metals or toxic organic compounds such as phenols. For the detection of stressed total coliforms in treated drinking water and chlorinated secondary or tertiary wastewater effluents, use a method designed for stressed organism recovery (see Section 9212B.1). A modified MF technique for fecal coliforms (Section 9212) in chlorinated wastewater may be used if parallel testing over a 3-month period with the multiple-tube fermentation technique shows comparability for each site-specific type of sample.

* Approved by Standard Methods Committee, 1997.

† ONPG is a substrate for the β-galactosidase test.

The standard volume to be filtered for drinking water samples is 100 mL. This may be distributed among multiple membranes if necessary. However, for special monitoring purposes, such as troubleshooting water quality problems of identification of coliform breakthrough in low concentrations from treatment barriers, it may be desirable to test 1-L samples. If particulates prevent filtering a 1-L sample through a single filter, divide sample into four portions of 250 mL for analysis. Total the coliform counts on each membrane to report the number of coliforms per liter. Smaller sample volumes will be necessary for source or recreational waters and wastewater effluents that have much higher coliform densities.

Statistical comparisons of results obtained by the multiple-tube method and the MF technique show that the MF is more precise (compare Tables 9221:II and III with Table 9222:II). Data from each test yield approximately the same water quality information, although numerical results are not identical (see Section 9010B for drinking water).

3. Bibliography

CLARK, H.F., E.E. GELDREICH, H.L. JETER & P.W. KABLER. 1951. The membrane filter in sanitary bacteriology. *Pub. Health Rep.* 66:951.

KABLER, P.W. 1954. Water examinations by membrane filter and MPN procedures. *Amer. J. Pub. Health* 44:379.

THOMAS, H.A. & R.L. WOODWARD. 1956. Use of molecular filter membranes for water potability control. *J. Amer. Water Works Assoc.* 48: 1391.

MCCARTHY, J.A., J.E. DELANEY & R.J. GRASSO. 1961. Measuring coliforms in water. *Water Sewage Works* 108:238.

LIN, S. 1973. Evaluation of coliform test for chlorinated secondary effluents. *J. Water Pollut. Control Fed.* 45:498.

MANDEL, J. & L.F. NANNI. 1978. Measurement evaluation. *In* S.L. Inhorn, ed. Quality Assurance Practices for Health Laboratories, p. 209. American Public Health Assoc., Washington, D.C.

9222 B. Standard Total Coliform Membrane Filter Procedure

1. Laboratory Apparatus

For MF analyses use glassware and other apparatus composed of material free from agents that may affect bacterial growth.

a. Sample bottles: See Section 9030B.18.

b. Dilution bottles: See Section 9030B.13.

c. Pipets and graduated cylinders: See Section 9030B.9. Before sterilization, loosely cover opening of graduated cylinders with metal foil or a suitable heavy wrapping-paper substitute. Immediately after sterilization secure cover to prevent contamination.

d. Containers for culture medium: Use clean borosilicate glass flasks. Any size or shape of flask may be used, but erlenmeyer flasks with metal caps, metal foil covers, or screw caps provide for adequate mixing of the medium contained and are convenient for storage.

e. Culture dishes: Use sterile borosilicate glass or disposable, presterilized plastic petri dishes, 60 × 15 mm, 50 × 9 mm, or other appropriate size. Wrap convenient numbers of clean, glass culture dishes in metal foil if sterilized by dry heat, or suitable heavy wrapping paper when autoclaved. Incubate loose-lidded glass and disposable plastic culture dishes in tightly closed containers with wet paper or cloth to prevent moisture evaporation with resultant drying of medium and to maintain a humid environment for optimum colony development.

Presterilized disposable plastic dishes with tight-fitting lids that meet the specifications above are available commercially and are used widely. Reseal opened packages of disposable dish supplies for storage.

f. Filtration units: The filter-holding assembly (constructed of glass, autoclavable plastic, porcelain, or stainless steel) consists of a seamless funnel fastened to a base by a locking device or by magnetic force. The design should permit the membrane filter to be held securely on the porous plate of the receptacle without mechanical damage and allow all fluid to pass through the membrane during filtration. Discard plastic funnels with deep scratches on inner surface or glass funnels with chipped surfaces.

Wrap the assembly (as a whole or separate parts) in heavy wrapping paper or aluminum foil, sterilize by autoclaving, and store until use. Alternatively expose all surfaces of the previously cleaned assembly to ultraviolet radiation (2 min exposure) for the initial sanitization before use in the test procedure, or before reusing units between successive filtration series. Field units may be sanitized by dipping or spraying with alcohol and then igniting or immersing in boiling water for 2 min. After submerging unit in boiling water, cool it to room temperature before reuse. Do not ignite plastic parts. Sterile, disposable field units may be used.

For filtration, mount receptacle of filter-holding assembly on a 1-L filtering flask with a side tube or other suitable device (manifold to hold three to six filter assemblies) such that a pressure differential (34 to 51 kPa) can be exerted on the filter membrane. Connect flask to a vacuum line, an electric vacuum pump, a filter pump operating on water pressure, a hand aspirator, or other means of securing a pressure differential (138 to 207 kPa). Connect a flask of approximately the same capacity between filtering flask and vacuum source to trap carry-over water.

g. Membrane filter: Use membrane filters (for additional specifications, see Section 9020) with a rated pore diameter such that there is complete retention of coliform bacteria. Use only those filter membranes that have been found, through adequate quality control testing and *certification by the manufacturer*, to exhibit: full retention of the organisms to be cultivated, stability in use, freedom from chemical extractables that may inhibit bacterial growth and development, a satisfactory speed of filtration (within 5 min), no significant influence on medium pH (beyond ± 0.2 units), and no increase in number of confluent colonies or spreaders compared to control membrane filters. Use membranes gridmarked in such a manner that bacterial growth is neither inhibited nor stimulated along the grid lines when the membranes with entrapped bacteria are incubated on a suitable medium. Preferably use fresh stocks of membrane filters and if necessary store them in an environment without extremes of temperature and humidity. Obtain no more than a year's supply at any one time.

Preferably use presterilized membrane filters for which the manufacturer has certified that the sterilization technique has neither induced toxicity nor altered the chemical or physical properties of the membrane. If membranes are sterilized in the laboratory, autoclave for 10 min at 121°C. At the end of the sterilization period, let the steam escape rapidly to minimize accumulation of water of condensation on filters.

h. Absorbent pads consist of disks of filter paper or other material certified for each lot by the manufacturer to be of high quality and free of sulfites or other substances of a concentration that could inhibit bacterial growth. Use pads approximately 48 mm in diameter and of sufficient thickness to absorb 1.8 to 2.2 mL of medium. Presterilized absorbent pads or pads subsequently sterilized in the laboratory should release less than 1 mg total acidity (calculated as $CaCO_3$) when titrated to the phenolphthalein end point, pH 8.3, using 0.02*N* NaOH and produce pH levels of 7 ± 0.2. Sterilize pads simultaneously with membrane filters available in resealable kraft envelopes, or separately in other suitable containers. Dry pads so they are free of visible moisture before use. See sterilization procedure described for membrane filters above and Section 9020 for additional specifications on absorbent pads.

i. Forceps: Smooth flat forceps, without corrugations on the inner sides of the tips. Sterilize before use by dipping in 95% ethyl or absolute methyl alcohol and flaming.

j. Incubators: Use incubators to provide a temperature of 35 ± 0.5°C and to maintain a humid environment (60% relative humidity).

k. Microscope and light source: To determine colony counts on membrane filters, use a magnification of 10 to 15 diameters and a cool white fluorescent light source adjusted to give maximum sheen discernment. Optimally use a binocular wide-field dissecting microscope. Do not use a microscope illuminator with optical system for light concentration from an incandescent light source for discerning coliform colonies on Endo-type media.

2. Materials and Culture Media

The need for uniformity dictates the use of commercial dehydrated media. Never prepare media from basic ingredients when suitable dehydrated media are available. Follow manufacturer's directions for rehydration. Store opened supplies of dehydrated media in a desiccator. Commercially prepared media in liquid form (sterile ampule or other) may be used if known to give equivalent results. See Section 9020 for media quality control specifications.

Test each new medium lot against a previously acceptable lot for satisfactory performance as described in Section 9020B. With each new lot of Endo-type medium, verify a minimum 10% of coliform colonies, obtained from natural samples or samples with known additions, to establish the comparative recovery of the medium lot.

Before use, test each batch of laboratory-prepared MF medium for performance with positive and negative culture controls. Check for coliform contamination at the beginning and end of each filtration series by filtering 20 to 30 mL of dilution or rinse water through the filter. If controls indicate contamination, reject all data from affected samples and request resample.

*a. LES Endo agar:**

Yeast extract	1.2	g
Casitone or trypticase	3.7	g
Thiopeptone or thiotone	3.7	g
Tryptose	7.5	g
Lactose	9.4	g
Dipotassium hydrogen phosphate, K_2HPO_4	3.3	g
Potassium dihydrogen phosphate, KH_2PO_4	1.0	g
Sodium chloride, NaCl	3.7	g
Sodium desoxycholate	0.1	g
Sodium lauryl sulfate	0.05	g
Sodium sulfite, Na_2SO_3	1.6	g
Basic fuchsin	0.8	g
Agar	15.0	g
Reagent-grade water	1	L

Rehydrate product in 1 L water containing 20 mL 95% ethanol. Do not use denatured ethanol, which reduces background growth and coliform colony size. Bring to a near boil to dissolve agar, then promptly remove from heat and cool to 45 to 50°C. Do not sterilize by autoclaving. Final pH 7.2 ± 0.2. Dispense in 5- to 7-mL quantities into lower section of 60-mm glass or plastic petri dishes. If dishes of any other size are used, adjust quantity to give an equivalent depth of 4 to 5 m. Do not expose poured plates to direct sunlight; refrigerate in the dark, preferably in sealed plastic bags or other containers to reduce moisture loss. Discard unused medium after 2 weeks or sooner if there is evidence of moisture loss, medium contamination, or medium deterioration (darkening of the medium).

b. M-Endo medium:†

Tryptose or polypeptone	10.0	g
Thiopeptone or thiotone	5.0	g
Casitone or trypticase	5.0	g
Yeast extract	1.5	g
Lactose	12.5	g
Sodium chloride, NaCl	5.0	g
Dipotassium hydrogen phosphate, K_2HPO_4	4.375	g
Potassium dihydrogen phosphate, KH_2PO_4	1.375	g
Sodium lauryl sulfate	0.05	g
Sodium desoxycholate	0.10	g
Sodium sulfite, Na_2SO_3	2.10	g
Basic fuchsin	1.05	g
Agar (optional)	15.0	g
Reagent-grade water	1	L

1) Agar preparation—Rehydrate product in 1 L water containing 20 mL 95% ethanol. Heat to near boiling to dissolve agar, then promptly remove from heat and cool to between 45 and 50°C. Dispense 5- to 7-mL quantities into 60-mm sterile glass or plastic petri dishes. If dishes of any other size are used, adjust quantity to give an equivalent depth. Do not sterilize by autoclaving. Final pH should be 7.2 ± 0.2. A precipitate is normal in Endo-type media.

Refrigerate finished medium in the dark and discard unused agar after 2 weeks.

2) Broth preparation—Prepare as above, omitting agar. Dispense liquid medium (at least 2.0 mL per plate) onto absorbent pads (see absorbent pad specifications, Section 9222B.1) and carefully remove excess medium by decanting the plate. The broth may have a precipitate but this does not interfere with medium performance if pads are certified free of sulfite or other toxic

* Dehydrated Difco M-Endo Agar LES (No. 0736), dehydrated BBL M-Endo Agar LES (No. 11203), or equivalent.

† Dehydrated Difco M-Endo Broth MF (No. 0749), dehydrated BBL *m*-Coliform Broth (No. 11119), or equivalent may be used if absorbent pads are used.

agents at a concentration that could inhibit bacterial growth. Refrigerated broth may be stored for up to 4 d.

c. Buffered dilution rinse water: See Section 9050C.1.

3. Samples

Collect samples as directed in Sections 9060A and B.

4. Coliform Definition

Bacteria that produce a red colony with a metallic (golden) sheen within 24 h incubation at 35°C on an Endo-type medium are considered members of the coliform group. The sheen may cover the entire colony or may appear only in a central area or on the periphery. The coliform group thus defined is based on the production of aldehydes from fermentation of lactose. While this biochemical characteristic is part of the metabolic pathway of gas production in the multiple-tube test, some variations in degree of metallic sheen development may be observed among coliform strains. However, this slight difference in indicator definition is not considered critical to change its public health significance, particularly if suitable studies have been conducted to establish the relationship between results obtained by the MF and those obtained by the standard multiple-tube fermentation procedure.

5. Procedures

a. Selection of sample size: Size of sample will be governed by expected bacterial density. In drinking water analyses, sample size will be limited only by the degree of turbidity or by the noncoliform growth on the medium (Table 9222:I). For regulation purposes, 100 mL is the official sample size.

An ideal sample volume will yield 20 to 80 coliform colonies and not more than 200 colonies of all types on a membrane-filter surface. Analyze drinking waters by filtering 100 to 1000 mL, or by filtering replicate smaller sample volumes such as duplicate 50-mL or four replicates of 25-mL portions. Analyze other waters by filtering three different volumes (diluted or undiluted), depending on the expected bacterial density. See Section 9215B.2 for preparation of dilutions. When less than 10 mL of sample (diluted or undiluted) is to be filtered, add approximately 10 mL sterile dilution water to the funnel before filtration or pipet the sample volume into a sterile dilution bottle, then filter the entire dilution. This increase in water volume aids in uniform dispersion of the bacterial suspension over the entire effective filtering surface.

b. Sterile filtration units: Use sterile filtration units at the beginning of each filtration series as a minimum precaution to avoid accidental contamination. A filtration series is considered to be interrupted when an interval of 30 min or longer elapses between sample filtrations. After such interruption, treat any further sample filtration as a new filtration series and sterilize all membrane filter holders in use. See Section 9222B.1*f* for sterilization procedures and Section 9020B.2*m* and *n* for UV cleaning and safety guidelines.

c. Filtration of sample: Using sterile forceps, place a sterile membrane filter (grid side up) over porous plate of receptacle. Carefully place matched funnel unit over receptacle and lock it in place. Filter sample under partial vacuum. With filter still in place, rinse the interior surface of the funnel by filtering three 20- to 30-mL portions of sterile dilution water. Alternatively, rinse funnel by a flow of sterile dilution water from a squeeze bottle. This is satisfactory only if the squeeze bottle and its contents do not become contaminated during use. Rinsing between samples prevents carryover contamination. Upon completion of final rinse and the filtration process disengage vacuum, unlock and remove funnel, immediately remove membrane filter with sterile forceps, and place it on selected medium with a rolling motion to avoid entrapment of air. If the agar-based medium is used, place prepared filter directly on agar, invert dish, and incubate for 22 to 24 h at 35 ± 0.5°C.

If liquid medium is used, place a pad in the culture dish and saturate with at least 2.0 mL M-Endo medium and carefully remove excess medium by decanting the plate. Place prepared filter directly on pad, invert dish, and incubate for 22 to 24 h at 35 ± 0.5°C.

Differentiation of some colonies from either agar or liquid medium substrates may be lost if cultures are incubated beyond 24 h.

Insert a sterile rinse water sample (100 mL) after filtration of a series of 10 samples to check for possible cross-contamination or contaminated rinse water. Incubate the rinse water control membrane culture under the same conditions as the sample.

For nonpotable water samples, preferably decontaminate filter unit after each sample (as described above) because of the high number of coliform bacteria present in these samples. Alternatively, use an additional buffer rinse of the filter unit after the filter is removed to prevent carryover between samples.

TABLE 9222:I. SUGGESTED SAMPLE VOLUMES FOR MEMBRANE FILTER TOTAL COLIFORM TEST

	Volume (X) To Be Filtered *mL*							
Water Source	100	50	10	1	0.1	0.01	0.001	0.0001
Drinking water	X							
Swimming pools	X							
Wells, springs	X	X	X					
Lakes, reservoirs	X	X	X					
Water supply intake			X	X	X			
Bathing beaches			X	X	X			
River water				X	X	X	X	
Chlorinated sewage				X	X	X		
Raw sewage					X	X	X	X

d. Alternative enrichment technique: Place a sterile absorbent pad in the lid of a sterile culture dish and pipet at least 2.0 mL lauryl tryptose broth, prepared as directed in 9221B.1.*a*1), to saturate pad. Carefully remove any excess liquid from absorbent pad by decanting plate. Aseptically place filter through which the sample has been passed on pad. Incubate filter, without inverting dish, for 1.5 to 2 h at 35 ± 0.5°C in an atmosphere of at least 60% relative humidity.

If the agar-based Endo-type medium is used, remove enrichment culture from incubator, lift filter from enrichment pad, and roll it onto the agar surface, which has been allowed to equilibrate to room temperature. Incorrect filter placement is at once obvious, because patches of unstained membrane indicate entrapment of air. Where such patches occur, carefully reseat filter on agar surface. If the liquid medium is used, prepare final culture by removing enrichment culture from incubator and separating the dish halves. Place a fresh sterile pad in bottom half of dish and saturate with at least 2.0 mL of M-Endo medium and carefully remove excess liquid from absorbent pad by decanting plate. Transfer filter, with same precautions as above, to new pad. Discard used enrichment pad.

With either the agar or the liquid medium, invert dish and incubate for 20 to 22 h at 35 ± 0.5°C. Proceed to ¶ *e* below.

e. Counting: To determine colony counts on membrane filters, use a low-power (10 to 15 magnifications) binocular wide-field dissecting microscope or other optical device, with a cool white fluorescent light source directed to provide optimal viewing of sheen. The typical coliform colony has a pink to dark-red color with a metallic surface sheen. Count both typical and atypical coliform colonies. The sheen area may vary in size from a small pinhead to complete coverage of the colony surface. Atypical coliform colonies can be dark red, mucoid, or nucleated without sheen. Generally pink, blue, white, or colorless colonies lacking sheen are considered noncoliforms. The total count of colonies (coliform and noncoliform) on Endo-type medium has no consistent relationship to the total number of bacteria present in the original sample. A high count of noncoliform colonies may interfere with the maximum development of coliforms. Refrigerating cultures (after 22 h incubation) with high densities of noncoliform colonies for 0.5 to 1 h before counting may deter spread of confluence while aiding sheen discernment.

Samples of disinfected water or wastewater effluent may include stressed organisms that grow relatively slowly and produce maximum sheen in 22 to 24 h. Organisms from undisinfected sources may produce sheen at 16 to 18 h, and the sheen subsequently may fade after 24 to 30 h.

f. Coliform verification: Occasionally, typical sheen colonies may be produced by noncoliform organisms and atypical colonies (dark red or nucleated colonies without sheen), may be coliforms. Preferably verify all typical and atypical colony types. For drinking water, verify all suspect colonies by swabbing the entire membrane or pick at least five typical colonies and five atypical colonies from a given membrane filter culture. For waters other than drinking water, at a minimum, verify at least 10 sheen colonies (and representative atypical colonies of different morphological types) from a positive water sample monthly. See Section 9020B.8. Based on need and sample type, laboratories may incorporate more stringent quality control measures (e.g., verify at least one colony from each typical or atypical colony type from a given membrane filter culture, verify 10% of the positive samples). Adjust counts on the basis of verification results. Verification tests are listed below.

1) Lactose fermentation—Transfer growth from each colony or swab the entire membrane with a sterile cotton swab (for presence-absence results in drinking water samples) and place in lauryl tryptose broth; incubate the lauryl tryptose broth at 35 ± 0.5°C for 48 h. Gas formed in lauryl tryptose broth and confirmed in brilliant green lactose broth (Section 9221B.2 for medium preparation) within 48 h verifies the colony as a coliform. Simultaneous inoculation of both media for gas production is acceptable. Inclusion of EC broth inoculation for 44.5 ± 0.2°C incubation will provide information on the presence of fecal coliforms. Use of EC-MUG with incubation at 44.5 ± 0.2°C for 24 h will provide information on presence of *E. coli*. See Section 9222G for MF partition procedures.

2) Alternative coliform verifications—Apply this alternative coliform verification procedure to isolated colonies on the membrane filter culture. If a mixed culture is suspected or if colony separation is less than 2 mm, streak the growth to M-Endo medium or MacConkey agar to assure culture purity or submit the mixed growth to the fermentation tube method.

a) Rapid test—A rapid verification of colonies utilizes test reactions for cytochrome oxidase (CO) and β-galactosidase. Coliform reactions are CO negative and β-galactosidase positive within 4 h incubation of tube culture or micro (spot) test procedure.

b) Commercial multi-test systems—Verify the colony by streaking it for purification, selecting a well-isolated colony, and inoculating into a multi-test identification system for Enterobacteriaceae that includes lactose fermentation and/or β-galactosidase and CO test reactions.

6. Calculation of Coliform Density

Compute the count, using membrane filters with 20 to 80 coliform colonies and not more than 200 colonies of all types per membrane, by the following equation:

$$\text{(Total) coliforms/100 mL} = \frac{\text{coliform colonies counted} \times 100}{\text{mL sample filtered}}$$

If no coliform colonies are observed, report the coliform colonies counted as "<1 coliform/100 mL."

For verified coliform counts, adjust the initial count based upon the positive verification percentage and report as "verified coliform count/100 mL."

$$\text{Percentage verified coliforms} = \frac{\text{number of verified colonies}}{\text{total number of coliform colonies subjected to verification}} \times 100$$

a. Water of drinking water quality: While the EPA Total Coliform Rule for public water supply samples requires only a record of coliform presence or absence in 100-mL samples, it may be advisable to determine coliform densities in repeat sampling situations. This is of particular importance when a coliform biofilm problem is suspected in the distribution system. Quantitative information may provide an indication of the magnitude of a contaminating event.

With water of good quality, the occurrence of coliforms generally will be minimal. Therefore, count all coliform colonies (dis-

regarding the lower limit of 20 cited above) and use the formula given above to obtain coliform density.

If confluent growth occurs, covering either the entire filtration area of the membrane or a portion thereof, and colonies are not discrete, report results as "confluent growth with (or without) coliforms." If the total number of bacterial colonies, coliforms plus noncoliforms, exceeds 200 per membrane, or if the colonies are not distinct enough for accurate counting, report results as "too numerous to count" (TNTC) or "confluent," respectively. For drinking water, the presence of coliforms in such cultures showing no sheen may be confirmed by either transferring a few colonies or placing the entire membrane filter culture into a sterile tube of brilliant green lactose bile broth. As an alternative, brush the entire filter surface with a sterile loop, applicator stick, or cotton swab and inoculate this growth to the tube of brilliant green lactose bile broth. If gas is produced from the brilliant green bile broth tube within 48 h at 35 ± 0.5°C, coliforms are present. For compliance with the EPA Total Coliform Rule, report confluent growth or TNTC with at least one detectable coliform colony (which is verified) as a total coliform positive sample. Report confluent growth or TNTC without detectable coliforms as invalid. For invalid samples, request a new sample from the same location within 24 h and select more appropriate volumes to be filtered per membrane, observing the requirement that the standard drinking water portion is 100 mL, or choose another coliform method that is less subject to heterotrophic bacterial interferences. Thus, to reduce interference from overcrowding, instead of filtering 100 mL per membrane, filter 50-mL portions through two separate membranes, 25-mL portions through each of four membranes, etc. Total the coliform counts observed on all membranes and report as number per 100 mL.

b. Water of other than drinking water quality: As with potable water samples, if no filter has a coliform count falling in the ideal range, total the coliform counts on all filters and report as number per 100 mL. For example, if duplicate 50-mL portions were examined and the two membranes had five and three coliform colonies, respectively, report the count as eight coliform colonies per 100 mL, i.e.,

$$\frac{[(5 + 3) \times 100]}{(50 + 50)} = 8 \text{ coliforms/100 mL}$$

Similarly, if 50-, 25-, and 10-mL portions were examined and the counts were 15, 6, and <1 coliform colonies, respectively, report the count as 25/100 mL, i.e.,

$$\frac{[(15 + 6 + 0) \times 100]}{(50 + 25 + 10)} = 25 \text{ coliforms/100 mL}$$

On the other hand, if 10-, 1.0-, and 0.1-mL portions were examined with counts of 40, 9, and <1 coliform colonies, respectively, select the 10-mL portion only for calculating the coliform density because this filter had a coliform count falling in the ideal range. The result is 400/100 mL, i.e.,

$$\frac{(40 \times 100)}{10} = 400 \text{ coliforms/100 mL}$$

In this last example, if the membrane with 40 coliform colonies also had a total bacterial colony count greater than 200, report the coliform count as ≥400/100 mL.

TABLE 9222:II. CONFIDENCE LIMITS FOR MEMBRANE FILTER COLIFORM RESULTS USING 100-mL SAMPLE

Number of Coliform Colonies Counted	95% Confidence Limits	
	Lower	Upper
0	0.0	3.7
1	0.1	5.6
2	0.2	7.2
3	0.6	8.8
4	1.0	10.2
5	1.6	11.7
6	2.2	13.1
7	2.8	14.4
8	3.4	15.8
9	4.0	17.1
10	4.7	18.4
11	5.4	19.7
12	6.2	21.0
13	6.9	22.3
14	7.7	23.5
15	8.4	24.8
16	9.2	26.0
17	9.9	27.2
18	10.7	28.4
19	11.5	29.6
20	12.2	30.8

Report confluent growth or membranes with colonies too numerous to count as described in *a* above. Request a new sample and select more appropriate volumes for filtration or utilize the multiple-tube fermentation technique.

c. Statistical reliability of membrane filter results: Although the precision of the MF technique is greater than that of the MPN procedure, membrane counts may underestimate the number of viable coliform bacteria. Table 9222:II illustrates some 95% confidence limits. These values are based on the assumption that bacteria are distributed randomly and follow a Poisson distribution. For results with counts, *c*, greater than 20 organisms, calculate the approximate 95% confidence limits using the following normal distribution equations:

$$\text{Upper limit} = c + 2\sqrt{c} \quad \text{Lower limit} = c - 2\sqrt{c}$$

7. Bibliography

FIFIELD, C.W. & C.P. SCHAUFUS. 1958. Improved membrane filter medium for the detection of coliform organisms. *J. Amer. Water Works Assoc.* 50:193.

MCCARTHY, J.A. & J.E. DELANEY. 1958. Membrane filter media studies. *Water Sewage Works* 105:292.

RHINES, C.E. & W.P. CHEEVERS. 1965. Decontamination of membrane filter holders by ultraviolet light. *J. Amer. Water Works Assoc.* 57: 500.

GELDREICH, E.E., H.L. JETER & J.A. WINTER. 1967. Technical considerations in applying the membrane filter procedure. *Health Lab. Sci.* 4:113.

WATLING, H.R. & R.J. WATLING. 1975. Note on the trace metal content of membrane filters. *Water SA* 1:28.

LIN, S.D. 1976. Evaluation of Millipore HA and HC membrane filters for the enumeration of indicator bacteria. *Appl. Environ. Microbiol.* 32:300.

STANDRIDGE, J.H. 1976. Comparison of surface pore morphology of two brands of membrane filters. *Appl. Environ. Microbiol.* 31:316.

GELDREICH, E.E. 1976. Performance variability of membrane filter procedure. *Pub. Health Lab.* 34:100.

GRABOW, W.O.K. & M. DU PREEZ. 1979. Comparison of m-Endo LES, MacConkey and Teepol media for membrane filtration counting of total coliform bacteria in water. *Appl. Environ. Microbiol.* 38:351.

DUTKA, B.D., ed. 1981. Membrane Filtration Applications, Techniques and Problems. Marcel Dekker, Inc., New York, N.Y.

EVANS, T.M., R.J. SEIDLER & M.W. LECHEVALLIER. 1981. Impact of verification media and resuscitation on accuracy of the membrane filter total coliform enumeration technique. *Appl. Environ. Microbiol.* 41: 1144.

FRANZBLAU, S.G., B.J. HINNEBUSCH, T.M. KELLEY & N.A. SINCLAIR. 1984. Effect of noncoliforms on coliform detection in potable groundwater: improved recovery with an anaerobic membrane filter technique. *Appl. Environ. Microbiol.* 48:142.

MCFETERS, G.A., J.S. KIPPIN & M.W. LECHEVALLIER. 1986. Injured coliforms in drinking water. *Appl. Environ. Microbiol.* 51:1.

9222 D. Fecal Coliform Membrane Filter Procedure

Fecal coliform bacterial densities may be determined either by the multiple-tube procedure or by the MF technique. See Section 9225 for differentiation of *Escherichia coli*, the predominant fecal coliform. If the MF procedure is used for chlorinated effluents, demonstrate that it gives comparable information to that obtainable by the multiple-tube test before accepting it as an alternative. The fecal coliform MF procedure uses an enriched lactose medium and incubation temperature of 44.5 ± 0.2°C for selectivity. Because incubation temperature is critical, submerge waterproofed (plastic bag enclosures) MF cultures in a water bath for incubation at the elevated temperature or use an appropriate solid heat sink incubator or other incubator that is documented to hold the 44.5°C temperature within 0.2°C throughout the chamber, over a 24-h period. Areas of application for the fecal coliform method in general are stated in the introduction to the multiple-tube fecal coliform procedures, Section 9221E.

1. Materials and Culture Medium

a. M-FC medium: The need for uniformity dictates the use of dehydrated media. Never prepare media from basic ingredients when suitable dehydrated media are available. Follow manufacturer's directions for rehydration. Commercially prepared media in liquid form (sterile ampule or other) also may be used if known to give equivalent results. See Section 9020 for quality control specifications.

M-FC medium:

Tryptose or biosate	10.0 g
Proteose peptone No. 3 or polypeptone	5.0 g
Yeast extract	3.0 g
Sodium chloride, NaCl	5.0 g
Lactose	12.5 g
Bile salts No. 3 or bile salts mixture	1.5 g
Aniline blue	0.1 g
Agar (optional)	15.0 g
Reagent-grade water	1 L

Rehydrate product in 1 L water containing 10 mL 1% rosolic acid in 0.2*N* NaOH.* Heat to near boiling, promptly remove from heat, and cool to below 50°C. Do not sterilize by autoclaving. If agar is used, dispense 5- to 7-mL quantities to 50- × 12-mm petri plates and let solidify. Final pH should be 7.4 ± 0.2. Refrigerate finished medium, preferably in sealed plastic bags or other containers to reduce moisture loss, and discard unused broth after 96 h or unused agar after 2 weeks.

Test each medium lot against a previously acceptable lot for satisfactory performance as described in Section 9020B, by making dilutions of a culture of *E. coli* (Section 9020) and filtering appropriate volumes to give 20 to 60 colonies per filter. With each new lot of medium verify 10 or more colonies obtained from several natural samples, to establish the absence of false positives. For most samples M-FC medium may be used without the 1% rosolic acid addition, provided there is no interference with background growth. Such interference may be expected in stormwater samples collected during the first runoff (initial flushing) after a long dry period.

Before use, test each batch of laboratory-prepared MF medium for performance with positive and negative culture controls. Check for coliform contamination at the beginning and end of each filtration series by filtering 20 to 30 mL of dilution or rinse

* Rosolic acid reagent will decompose if sterilized by autoclaving. Refrigerate stock solution in the dark and discard after 2 weeks or sooner if its color changes from dark red to muddy brown.

water through filter. If controls indicate contamination, reject all data from affected samples and request resample.

b. Culture dishes: Tight-fitting plastic dishes are preferred because the membrane filter cultures are submerged in a water bath during incubation. Place fecal coliform cultures in plastic bags or seal individual dishes with waterproof (freezer) tape to prevent leakage during submersion. Specifications for plastic culture dishes are given in Section 9222B.1*e*.

c. Incubator: The specificity of the fecal coliform test is related directly to the incubation temperature. Static air incubation may be a problem in some types of incubators because of potential heat layering within the chamber, slower heat transfer from air to the medium, and the slow recovery of temperature each time the incubator is opened during daily operations. To meet the need for greater temperature control use a water bath, a heat-sink incubator, or a properly designed and constructed incubator shown to give equivalent results. A temperature tolerance of 44.5 ± 0.2°C can be obtained with most types of water baths that also are equipped with a gable top for the reduction of water and heat losses.

2. Procedure

a. Selection of sample size: Select volume of water sample to be examined in accordance with the information in Table 9222:III. Use sample volumes that will yield counts between 20 and 60 fecal coliform colonies per membrane.

When the bacterial density of the sample is unknown, filter several volumes or dilutions to achieve a countable density. Estimate volume and/or dilution expected to yield a countable membrane and select two additional quantities representing one-tenth and ten times this volume, respectively.

b. Filtration of sample: Follow the same procedure and precautions as prescribed under Section 9222B.5*b* above.

c. Preparation of culture dish: Place a sterile absorbent pad in each culture dish and pipet at least 2.0 mL M-FC medium, prepared as directed above, to saturate pad. Carefully remove any excess liquid from culture dish by decanting the plate. Aseptically, place prepared filter on medium-impregnated pad as described in Section 9222B above.

As a substrate substitution for the nutrient-saturated absorbent pad, add 1.5% agar to M-FC broth as described in Section 9222B above.

d. Incubation: Place prepared dishes in waterproof plastic bags or seal, invert, and submerge petri dishes in water bath, and incubate for 24 ± 2 h at 44.5 ± 0.2°C. Anchor dishes below water surface to maintain critical temperature requirements. Place all prepared cultures in the water bath within 30 min after filtration. Alternatively, use an appropriate, accurate solid heat sink or equivalent incubator.

e. Counting: Colonies produced by fecal coliform bacteria on M-FC medium are various shades of blue. Nonfecal coliform colonies are gray to cream-colored. Normally, few nonfecal coliform colonies will be observed on M-FC medium because of selective action of the elevated temperature and addition of rosolic acid salt reagent. Count colonies with a low-power (10 to 15 magnifications) binocular wide-field dissecting microscope or other optical device.

f. Verification: Verify typical blue colonies and any atypical grey to green colonies as described in Section 9020 for fecal coliform analysis. Simultaneous inoculation at both temperatures is acceptable.

3. Calculation of Fecal Coliform Density

a. General: Compute the density from the sample quantities that produced MF counts within the desired range of 20 to 60 fecal coliform colonies. This colony density range is more restrictive than the 20 to 80 total coliform range because of larger colony size on M-FC medium. Calculate fecal coliform density as directed in Section 9222B.6 above. Record densities as fecal coliforms per 100 mL.

b. Sediment and biosolid samples: For total solid (dry weight basis) see Section 2540G.

Calculate fecal coliforms per gram dry weight for biosolid analysis as follows:

$$\text{Fecal coliforms per gram dry weight} = \frac{\text{colonies counted}}{\text{(dilution chosen)} \times \text{(\% dry solids)}}$$

where dilution and % dry solids are expressed in decimal form.

Example 1: There were 22 colonies observed on the 1:10 000 dilution plate of a biosolid with 4% dry solids.

$$\frac{22}{(0.0001)(0.04)} = 5.5 \times 10^6 \text{ fecal coliform/g dry weight}$$

If no filter has a coliform count falling in the ideal range (20

TABLE 9222:III. SUGGESTED SAMPLES VOLUMES FOR MEMBRANE FILTER FECAL COLIFORM TEST

	Volume (*X*) To Be Filtered, *mL*							
Water Source	100	50	10	1	0.1	0.01	0.001	0.0001
Lakes, reservoirs	X	X						
Wells, springs	X	X						
Water supply intake		X	X	X				
Natural bathing waters		X	X	X				
Sewage treatment plant			X	X	X			
Farm ponds, rivers				X	X	X		
Stormwater runoff				X	X	X		
Raw municipal sewage					X	X	X	
Feedlot runoff					X	X	X	
Sewage sludge						X	X	X

to 60), total the coliform counts on all countable filters and report as fecal coliforms per gram dry weight.

Example 2: There were 18 colonies observed on the 1:10 000 dilution plate and 2 colonies observed on the 1:100 000 dilution plate of a biosolid sample with 4% dry solids.

$$\frac{(18 + 2)}{(0.0001 + 0.00001)(0.04)} = 4.5 \times 10^6$$

To compute a geometric mean of samples, convert coliform densities of each sample to $\log_{10}$ values. Determine the geometric mean for the given number of samples (usually seven) by averaging the $\log_{10}$ values of the coliform densities and taking the antilog of that value.

4. Bibliography

GELDREICH, E.E., H.F. CLARK, C.B. HUFF & L.C. BEST. 1965. Fecal-coliform-organism medium for the membrane filter technique. *J. Amer. Water Works Assoc.* 57:208.

ROSE, R.E., E.E. GELDREICH & W. LITSKY. 1975. Improved membrane filter method for fecal coliform analysis. *Appl. Microbiol.* 29:532.

LIN, S.D. 1976. Membrane filter method for recovery of fecal coliforms in chlorinated sewage effluents. *Appl. Environ. Microbiol.* 32:547.

PRESSWOOD, W.G. & D.K. STRONG. 1978. Modification of M-FC medium by eliminating rosolic acid. *Appl. Environ. Microbiol.* 36:90.

GREEN, B.L., W. LITSKY & K.J. SLADEK. 1980. Evaluation of membrane filter methods for enumeration of faecal coliforms from marine waters. *Mar. Environ. Res.* 67:267.

SARTORY, D.P. 1980. Membrane filtration faecal coliform determinations with unmodified and modified M-FC medium. *Water SA* 6:113.

GRABOW, W.O.K., C.A. HILNER & P. COUBROUGH. 1981. Evaluation of standard and modified M-FC, MacConkey, and Teepol media for membrane filter counting of fecal coliform in water. *Appl. Environ. Microbiol.* 42:192.

RYCHERT, R.C. & G.R. STEPHENSON. 1981. Atypical *Escherichia coli* in streams. *Appl. Environ. Microbiol.* 41:1276.

PAGEL, J.E., A.A. QURESHI, D.M. YOUNG & L.T. VLASSOFF. 1982. Comparison of four membrane filter methods for fecal coliform enumeration. *Appl. Environ. Microbiol.* 43:787.

U.S. ENVIRONMENTAL PROTECTION AGENCY. 1992. Environmental Regulations and Technology. Control of Pathogens and Vector Attraction in Sewage Sludge. EPA-626/R-92-013, Washington, D.C.

U.S. ENVIRONMENTAL PROTECTION AGENCY. 1993. Standards for the Use or Disposal of Sewage Sludge: Final Rule. 40 CFR Part 257; *Federal Register* 58:9248, Feb. 19, 1993.

10. Appendix B. EPA Method 1604: Total Coliforms and Escherichia coli in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)

Extracted and reproduced without alteration. Presented here to ensure availability.

EPA

United States Environmental Protection Agency	Office of Water Washington DC 20460	EPA 821-R-02-024 September 2002

Method 1604: Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)

U.S. Environmental Protection Agency
Office of Water (4303T)
1200 Pennsylvania Avenue, NW
Washington, DC 20460

EPA-821-R-02-024

Disclaimer

The Engineering and Analysis Division, of the Office of Science and Technology, has reviewed and approved this report for publication. The Office of Science and Technology directed, managed, and reviewed the work of DynCorp in preparing this report. Neither the United States Government nor any of its employees, contractors, or their employees make any warranty, expressed or implied, or assumes any legal liability or responsibility for any third party's use of or the results of such use of any information, apparatus, product, or process discussed in this report, or represents that its use by such party would not infringe on privately owned rights.
The content of this method version is identical to the February 2000 version of *Membrane Filter Method for the Simultaneous Detection of Total Coliforms and Escherichia coli in Drinking Water* (EPA-600-R- 00-013) with one exception, the addition of MI broth. Since MI broth was approved on November 6, 2001, as a minor modification of the MI agar method, it has also been included in this document.
Mention of trade names or commercial products does not constitute endorsement or recommendation for use.
Questions concerning this method or its application should be addressed to:

Robin K. Oshiro
Engineering and Analysis Division (4303T)
U.S. EPA Office of Water, Office of Science and Technology
1200 Pennsylvania Avenue, NW
Washington, DC 20460
oshiro.robin@epa.gov
202-566-1075
202-566-1053 (facsimile)

Table of Contents

1.0 Scope and Application 1
2.0 Summary of Method 1
3.0 Definitions 1
4.0 Interferences and Contamination 2
5.0 Safety 2
6.0 Equipment and Supplies 3
7.0 Reagents and Standards 4
8.0 Sample Collection, Preservation, and Storage 6
9.0 Calibration and Standardization 6
10.0 Quality Control (QC) 6
11.0 Procedure 7
12.0 Data Analysis and Calculations 8
13.0 Method Performance 8
14.0 Pollution Prevention 9
15.0 Waste Management 9
16.0 References 10
17.0 Tables and Figures 12

Method 1604: Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)

September 2002

1.0 Scope and Application

1.1 This test method describes a sensitive and differential membrane filter (MF) medium, using MI agar or MI broth, for the simultaneous detection and enumeration of both total coliforms (TC) and *Escherichia coli* (*E. coli*) in water samples in 24 hours or less on the basis of their specific enzyme activities. Two enzyme substrates, the fluorogen 4-Methylumbelliferyl-$-Dgalactopyranoside (MUGal) and a chromogen Indoxyl-$-D-glucuronide (IBDG), are included in the medium to detect the enzymes $-galactosidase and $-glucuronidase, respectively, produced by TC and *E. coli*, respectively.

1.2 Total coliforms include species that may inhabit the intestines of warm-blooded animals or occur naturally in soil, vegetation, and water. They are usually found in fecally-polluted water and are often associated with disease outbreaks. Although they are not usually pathogenic themselves, their presence in drinking water indicates the possible presence of pathogens. *E. coli*, one species of the coliform group, is always found in feces and is, therefore, a more direct indicator of fecal contamination and the possible presence of enteric pathogens. In addition, some strains of *E. coli* are pathogenic (Reference 16.12).

1.3 This method, which has been validated for use with drinking water in single-lab and multi-lab studies (References 16.8 - 16.10), will be used primarily by certified drinking water laboratories for microbial analysis of potable water. Other uses include recreational, surface or marine water, bottled water, groundwater, well water, treatment plant effluents, water from drinking water distribution lines, drinking water source water, and possibly foods, pharmaceuticals, clinical specimens (human or veterinary), other environmental samples (*e.g.*, aerosols, soil, runoff, or sludge) and/or isolation and separation of transformants though the use of *E. coli lac* Z or *gus* A/*uid* reporter genes (Reference 16.11).

1.4 Since a wide range of sample volumes or dilutions can be analyzed by the MF technique, a wide range of *E. coli* and TC levels in water can be detected and enumerated.

2.0 Summary of Method

2.1 An appropriate volume of a water sample (100 mL for drinking water) is filtered through a 47- mm, 0.45-µm pore size cellulose ester membrane filter that retains the bacteria present in the sample. The filter is placed on a 5-mL plate of MI agar or on an absorbent pad saturated with 2-3 mL of MI broth, and the plate is incubated at 35°C for up to 24 hours. The bacterial colonies that grow on the plate are inspected for the presence of blue color from the breakdown of IBDG by the *E. coli* enzyme $-glucuronidase and fluorescence under longwave ultraviolet light (366 nm) from the breakdown of MUGal by the TC enzyme $-galactosidase (Reference 16.8).

3.0 Definitions

3.1 Total coliforms (TC) - In this method, TC are those bacteria that produce fluorescent colonies upon exposure to longwave ultraviolet light (366 nm) after primary culturing on MI agar or broth (See Figure 1.). The fluorescent colonies can be completely blue-white (TC other than *E. coli*) or blue-green (*E. coli*) in color or fluorescent halos may be observed around the edges of the blue- green *E. coli* colonies. In addition, non-fluorescent blue colonies, which rarely occur, are added to the total count because the fluorescence is masked by the blue color from the breakdown of IBDG (Reference 16.8).

3.2 *Escherichia coli* - In this method, the *E. coli* are those bacteria that produce blue colonies under ambient light after primary culturing on MI agar or broth (See Figures 1 and 2.). These colonies can be fluorescent or non-fluorescent under longwave ultraviolet light (366 nm) (Reference 16.8).

4.0 Interferences and Contamination

4.1 Water samples containing colloidal or suspended particulate material can clog the membrane filter, thereby preventing filtration, or cause spreading of bacterial colonies which could interfere with identification of target colonies. However, the blue *E. coli* colonies can often be counted on plates with heavy particulates or high concentrations of total bacteria (See Figures 2 and 3.) (Reference 16.8).

4.2 The presence of some lateral diffusion of blue color away from the target *E. coli* colonies can affect enumeration and colony picking on plates with high concentrations of *E. coli*. This problem should not affect filters with low counts, such as those obtained with drinking water or properly diluted samples (Reference 16.8).

4.3 Tiny, flat or peaked pinpoint blue colonies (**#** 0.5-mm in diameter on filters containing **#** 200 colonies) may be due to species other than *E. coli*. These colonies occur occasionally in low numbers and should be excluded from the count of the *E. coli* colonies, which are usually much larger in size (1-3-mm in diameter). The small colonies have never been observed in the absence of typical *E. coli*, but, if such should occur, the sample should not be considered *E. coli*-positive unless at least one colony has been verified by another method [*e.g.*, EC medium with 4-Methylumbelliferyl-$-D-glucuronide (MUG) or API 20E strips] (Reference 16.8).

4.4 Bright green, fluorescent, non-blue colonies, observed along with the typical blue/white or bluegreen fluorescent TC colonies, may be species other than coliforms. These colonies, which generally occur in low numbers (**#** 5%) and can usually be distinguished from the TC, should be eliminated from the TC count. An increase in the number of bright green colonies may indicate an unusual sample population or a breakdown of the cefsulodin in the medium (Reference 16.8).

5.0 Safety

5.1 The analyst/technician must know and observe the normal safety procedures required in a microbiology laboratory while preparing, using, and disposing of cultures, reagents, and materials, and while operating sterilization equipment.

5.2 Mouth-pipetting is prohibited.

5.3 Avoid prolonged exposure to longwave or germicidal ultraviolet light.

5.4 Autoclave all contaminated plates and materials at the end of the analysis.

6.0 Equipment and Supplies

6.1 Incubator set at 35°C ± 0.5°C, with approximately 90% humidity if loose-lidded petri dishes are used.

6.2 Stereoscopic microscope, with magnification of 10-15x, wide-field type.

6.3 A microscope lamp producing diffuse light from cool, white fluorescent lamps adjusted to give maximum color.

6.4 Hand tally.

6.5 Pipet container of stainless steel, aluminum, or Pyrex glass, for pipets.

6.6 Graduated cylinders (100-mL for drinking water), covered with aluminum foil or kraft paper and sterilized.

6.7 Membrane filtration units (filter base and funnel), glass, plastic or stainless steel. These are wrapped with aluminum foil or kraft paper and sterilized.

6.8 Germicidal ultraviolet (254 nm) light box for sanitizing the filter funnels is desirable, but optional.

6.9 A line vacuum, electric vacuum pump, or aspirator is used as a vacuum source. In an emergency, a hand pump or a syringe can be used. Such vacuum-producing devices should be equipped with a check valve to prevent the return flow of air.

6.10 Vacuum filter flask, usually 1 liter, with appropriate tubing. Filter manifolds to hold a number of filter bases are desirable, but optional.

6.11 Safety trap flask, placed between the filter flask and the vacuum source.

6.12 Forceps, straight (preferred) or curved, with smooth tips to permit easy handling of filters without damage.

6.13 Alcohol, 95% ethanol, in small wide-mouthed vials, for sterilizing forceps.

6.14 Bunsen or Fisher-type burner or electric incinerator unit.

6.15 Sterile T.D. (To Deliver) bacteriological or Mohr pipets, glass or plastic (1-mL and 10-mL volumes).

6.16 Membrane Filters (MF), white, grid-marked, cellulose ester, 47-mm diameter, 0.45 µm ± 0.02- µm pore size, presterile or sterilized for 10 minutes at 121°C (15-lb pressure).

6.17 Longwave ultraviolet lamp (366 nm), handheld 4-watt (preferred) or 6-watt, or microscope attachment.

6.18 *Dilution water*: Sterile phosphate-buffered dilution water, prepared in large volumes (*e.g.*, 1 liter) for wetting membranes before addition of the sample and for rinsing the funnel after sample filtration or in 99-mL dilution blanks [Section 9050C in *Standard Methods* (Reference 16.2)].

6.19 Indelible ink marker for labeling plates.

6.20 Thermometer, checked against a National Institute of Science and Technology (NIST)-certified thermometer, or one traceable to an NIST thermometer.

6.21 Petri dishes, sterile, plastic, 9 x 50 mm, with tight-fitting lids, or 15 x 60 mm, glass or plastic, with loose-fitting lids; 15 x 100 mm dishes may also be used.

6.22 Bottles, milk dilution, borosilicate glass, screw-cap with neoprene liners, marked at 99 mL for 1:100 dilutions (if needed). Dilution bottles marked at 90 mL, or tubes marked at 9 mL may be used for 1:10 dilutions.

6.23 Flasks, borosilicate glass, screw-cap, 250- to 2000-mL volume, for agar preparation.

6.24 Waterbath maintained at 50°C for tempering agar.

6.25 Syringe filter, sterile, disposable, 25-mm diameter, 0.22-µm pore size, to filter cefsulodin for MI agar.

6.26 Syringe, sterile, plastic, disposable, 20-cc capacity. Autoclaved glass syringes are also acceptable.

6.27 Test tubes, sterile, screw-cap, 20 x 150-mm, borosilicate glass or plastic, with lids.

6.28 Sterilization filter units, presterile, disposable, 500- or 1000-mL capacity, 0.2-µm pore size, to filter stock buffer solutions.

6.29 Sterile 47-mm diameter absorbent pads (used with MI broth).

Note: *Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

7.0 Reagents and Standards

7.1 *Purity of Reagents*: Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (Reference 16.1). The agar used in preparation of culture media must be of microbiological grade.

7.2 Whenever possible, use commercial culture media as a means of quality control.

7.3 *Purity of Water*: Reagent-grade distilled water conforming to Specification D1193, Type II water or better, *ASTM Annual Book of Standards* (Reference 16.3).

7.4 Buffered Dilution Water (Reference 16.2)

7.4.1 *Stock Phosphate Buffer Solution* (Reference 16.2):

Potassium Dihydrogen Phosphate (KH_2PO_4)	34.0 g
Reagent-Grade Distilled Water	500 mL

7.4.2 *Preparation of Stock Buffer Solution*: Adjust the pH of the solution to 7.2 with 1 N NaOH, and bring volume to 1000 mL with reagent-grade distilled water. Sterilize by filtration or autoclave for 15 minutes at 121°C (15-lb pressure).

7.4.3 *$MgCl_2$ Solution* (Reference 16.2): Dissolve 38 g anhydrous $MgCl_2$ (or 81.1 g $MgCl_2C6H_2O$) in one liter of reagent-grade distilled water. Sterilize by filtration or autoclave for 15 minutes at 121°C (15-lb pressure).

7.4.4 *Storage of Stock Buffer and $MgCl_2$ Solutions*: After sterilization of the stock solutions, store in the refrigerator until used. Handle aseptically. If evidence of mold or other

contamination appears in either stock, the solution should be discarded, and a fresh solution should be prepared.

7.4.5 *Working Solution* (Final pH 7.0 ± 0.2): Add 1.25 mL phosphate buffer stock (Section 7.4.2) and 5 mL $MgCl_2$ stock (Section 7.4.3) for each liter of reagent-grade distilled water prepared. Mix well, and dispense in appropriate amounts for dilutions in screw-cap dilution bottles or culture tubes, and/or into larger containers for use as rinse water. Autoclave at 121°C (15-lb pressure) for 15 minutes. Longer sterilization times may be needed depending on the container and load size and the amount of time needed for the liquid to reach 121°C.

7.5 MI Agar (Reference 16.8)

7.5.1 *Composition:*

Proteose Peptone #3	5.0 g
Yeast Extract	3.0 g
$-D-Lactose	1.0 g
4-Methylumbelliferyl-$-D-Galactopyranoside (MUGal) (Final concentration 100µg/mL)	0.1 g
Indoxyl-$-D-Glucuronide (IBDG) (Final concentration 320 µg/mL)	0.32 g
NaCl	7.5 g
K_2HPO_4	3.3 g
KH_2PO_4	1.0 g
Sodium Lauryl Sulfate	0.2 g
Sodium Desoxycholate	0.1 g
Agar	15.0 g
Reagent-Grade Distilled Water	1000 mL

7.5.2 *Cefsulodin Solution* (1 mg / 1 mL): Add 0.02 g of cefsulodin to 20 mL reagent-grade distilled water, sterilize using a 0.22-µm syringe filter, and store in a sterile tube at 4°C until needed. Prepare fresh solution each time. Do not save the unused portion.

7.5.3 *Preparation*: Autoclave the medium for 15 minutes at 121°C (15-lb pressure), and add 5 mL of the freshly-prepared solution of Cefsulodin (5 µg/mL final concentration) per liter of tempered agar medium. Pipet the medium into 9 x 50-mm Petri dishes (5 mL/plate). Store plates at 4°C for up to 2 weeks. The final pH should be 6.95 ± 0.2.

7.6 MI Broth: The composition of MI broth is the same as MI agar, but without the agar. The final pH of MI broth should be 7.05 ± 0.2. The broth is prepared and sterilized by the same methods described for MI agar in Sections 7.5.1, 7.5.2, and 7.5.3, except that absorbent pads are placed in 9 x 50 mm Petri dishes and saturated with 2-3 mL of MI broth containing 5 :g/mL final concentration of Cefsulodin. Alternately, the broth can be filter-sterilized. Excess broth is poured off before using the plates. Plates should be stored in the refrigerator and discarded after 96 hours (Reference 16.15).

7.7 Tryptic Soy Agar/Trypticase Soy Agar (Difco 0369-17-6, BD 4311043, Oxoid CM 0129B, or equivalent) (TSA)

7.7.1 *Composition*:

Tryptone	15.0 g
Soytone	5.0 g
NaCl	5.0 g
Agar	15.0 g

7.7.2 *Preparation*: Add the dry ingredients listed above to 1000 mL of reagent-grade distilled water, and heat to boiling to dissolve the agar completely. Autoclave at 121°C (15-lb pressure) for 15 minutes. Dispense the agar into 9 x 50-mm petri dishes (5 mL/plate). Incubate the plates for 24 - 48 hours at 35°C to check for contamination. Discard any plates with growth. If > 5% of the plates show contamination, discard all plates, and make new medium. Store at 4°C until needed. The final pH should be 7.3 ± 0.2.

8.0 Sample Collection, Preservation, and Storage

8.1 Water samples are collected in sterile polypropylene sample containers with leakproof lids.

8.2 Sampling procedures are described in detail in Sections 9060A and 9060B of the 18th edition of *Standard Methods for the Examination of Water and Wastewater* (Reference 16.2) or in the *USEPA Microbiology Methods Manual*, Section II, A (Reference 16.6). Residual chlorine in drinking water (or chlorinated effluent) samples should be neutralized with sodium thiosulfate (1 mL of a 10% solution per liter of water) at the time of collection. Adherence to sample preservation procedures and holding time limits are critical to the production of valid data. Samples not collected according to these rules should not be analyzed.

8.2.1 *Storage Temperature and Handling Conditions:* Ice or refrigerate water samples at a temperature of 1-4°C during transit to the laboratory. Use insulated containers to assure proper maintenance of storage temperature. Take care that sample bottles are not totally immersed in water from melted ice during transit or storage.

8.2.2 *Holding Time Limitations:* Analyze samples as soon as possible after collection. Drinking water samples should be analyzed within 30 h of collection (Reference 16.13). Do not hold source water samples longer than 6 h between collection and initiation of analyses, and the analyses should be complete within 8 h of sample collection.

9.0 Calibration and Standardization

9.1 Check temperatures in incubators twice daily to ensure operation within stated limits (Reference 16.14).

9.2 Check thermometers at least annually against an NIST-certified thermometer or one traceable to NIST. Check mercury columns for breaks.

10.0 Quality Control (QC)

10.1 Pretest each batch of MI agar or broth for performance (*i.e.*, correct enzyme reactions) with known cultures (*E. coli*, TC, and a non-coliform).

10.2 Test new lots of membrane filters against an acceptable reference lot using the method of Brenner and Rankin (Reference 16.7).

10.3 Perform specific filtration control tests each time samples are analyzed, and record the results.

10.3.1 *Filter Control*: Place one or more membrane filters on TSA plates, and incubate the plates for 24 hours at 35°C. Absence of growth indicates sterility of the filter(s).

10.3.2 *Phosphate-Buffered Dilution Water Controls*: Filter a 50-mL volume of sterile dilution water before beginning the sample filtrations and a 50-mL volume of dilution water after completing the filtrations. Place the filters on TSA plates, and incubate the plates for 24 hours at 35°C. Absence of growth indicates sterility of the dilution water.

10.3.3 *Agar or Broth Controls*: Place one or more TSA plates and one or more MI agar plates or MI broth pad plates in the incubator for 24 hours at 35°C. Broth pad plates should be incubated *grid-side up*, not inverted like the agar plates. Absence of growth indicates sterility of the plates.

10.4 See recommendations on quality control for microbiological analyses in the "*Manual for the Certification of Laboratories Analyzing Drinking Water*: *Criteria and Procedures; Quality Assurance*" (Reference 16.15) and the *USEPA Microbiology Methods Manual*, part IV, C (Reference 16.6).

11.0 Procedure

11.1 Prepare MI agar or MI broth and TSA as described in Sections 7.5, 7.6, and 7.7. If plates are made ahead of time and stored in the refrigerator, remove them and allow them to warm to room temperature. The crystals that form on MI agar after refrigeration will disappear as the plates warm up (Reference 16.8).

11.2 Label the bottom of the MI agar or MI broth plates with the sample number/identification and the volume of sample to be analyzed. Label QC TSA plates and the MI agar or MI broth sterility control plate(s).

11.3 Using a flamed forceps, place a membrane filter, grid-side up, on the porous plate of the filter base. If you have difficulties in removing the separation papers from the filters due to static electricity, place a filter with the paper on top of the funnel base and turn on the vacuum. The separation paper will curl up, allowing easier removal.

11.4 Attach the funnel to the base of the filter unit, taking care not to damage or dislodge the filter. The membrane filter is now located between the funnel and the base.

11.5 Put approximately 30 mL of sterile dilution water in the bottom of the funnel.

11.6 Shake the sample container vigorously 25 times.

11.7 Measure an appropriate volume (100 mL for drinking water) or dilution of the sample with a sterile pipette or graduated cylinder, and pour it into the funnel. Turn on the vacuum, and leave it on while rinsing the funnel twice with about 30 mL sterile dilution water.

11.8 Remove the funnel from the base of the filter unit. A germicidal ultraviolet (254 nm) light box can be used to hold and sanitize the funnel between filtrations. At least 2 minutes of exposure time is required for funnel decontamination. Protect eyes from UV irradiation with glasses, goggles, or an enclosed UV chamber.

11.9 Holding the membrane filter at its edge with a flamed forceps, gently lift and place the filter gridside up on the MI agar plate or MI broth pad plate. Slide the filter onto the agar or pad, using a rolling action to avoid trapping air bubbles between the membrane filter and the underlying agar or absorbent pad. Run the tip of the forceps around the outside edge of the filter to be sure the filter makes contact with the agar or pad. Reseat the membrane if non-wetted areas occur due to air bubbles

11.10 Invert the agar petri dish, and incubate the plate at 35°C for 24 hours. Pad plates used with MI broth should be incubated *grid-side up* at 35°C for 24 hours. If loose-lidded plates are used for MI agar or broth, the plates should be placed in a humid chamber.

11.11 Count all blue colonies on each MI plate under normal/ambient light, and record the results (See Figures 1 and 2.). This is the *E. coli* count. Positive results that occur in less than 24 hours are valid, but the results cannot be recorded as negative until the 24-hour incubation period is complete (Reference 16.14).

11.12 Expose each MI plate to longwave ultraviolet light (366 nm), and count all fluorescent colonies [blue/green fluorescent *E. coli*, blue/white fluorescent TC other than *E. coli*, and blue/green with fluorescent edges (also *E. coli*)] (See Figure 1.). Record the data.

11.13 Add any blue, non-fluorescent colonies (if any) found on the same plate to the TC count (Reference 16.8).

12.0 Data Analysis and Calculations

12.1 Use the following general rules to calculate the *E. coli* or TC per 100 mL of sample:

12.1.1 Select and count filters with **#** 200 total colonies per plate.

12.1.2 Select and count filter with **#** 100 target colonies (ideally, 20-80).

12.1.3 If the total number of colonies or TC on a filter is too-numerous-to-count or confluent, record the results as "TC_+ (TNTC)" and count the number of *E. coli*. If both target organisms are **$** 200, record the results as "TC_+ EC_+ (TNTC)".

12.1.4 Calculate the final values using the formula:

$$\textit{E. coli}/100\ \text{mL} = \frac{\text{Number of blue colonies}}{\text{Volume of sample filtered (mL)}} \times 100$$

$$\text{TC}/100\ \text{mL} = \frac{\text{Number of fluorescent colonies + Number of blue, non-fluorescent colonies (if any)}}{\text{Volume of sample filtered (mL)}} \times 100$$

12.2 See the *USEPA Microbiology Manual*, Part II, Section C, 3.5, for general counting rules (Reference 16.6).

12.3 Report results as *E. coli* or TC per 100 mL of drinking water.

13.0 Method Performance

13.1 The detection limits of this method are one *E. coli* and/or one total coliform per sample volume or dilution tested (Reference 16.8).

13.2 The false-positive and false-negative rates for *E. coli* are both reported to be 4.3% (Reference 16.8).

13.3 The single lab recovery of *E. coli* is reported (Reference 16.8) to be 97.9% of the Heterotrophic Plate Count (pour plate) (Reference 16.2) and 115% of the R2A spread plate (Reference 16.2). For *Klebsiella pneumoniae* and *Enterobacter aerogenes*, two total coliforms, the recoveries are 87.5% and 85.7% of the HPC (Reference 16.8), respectively, and 89.3% and 85.8% of the R2A spread plate, respectively.

13.4 The specificities for *E. coli* and total coliforms are reported to be 95.7% and 93.1% (Reference 16.8), respectively.

13.5 The single lab coefficients of variation for *E. coli* and total coliforms are reported to be 25.1% and 17.6% (Reference 16.8), respectively, for a variety of water types.

13.6 In a collaborative study (References 16.4, 16.5, and 16.9), 19 laboratories concurrently analyzed six wastewater-spiked Cincinnati tap water samples, containing 3 different concentrations of *E. coli* (**#** 10, 11-30, and > 30 per 100 mL).

13.6.1 The single laboratory precision (coefficient of variation), a measure of the repeatability, ranged from 3.3% to 27.3% for *E. coli* and from 2.5% to 5.1% for TC for the six samples tested, while the overall precision (coefficient of variation), a measure of reproducibility, ranged from 8.6% to 40.5% and from 6.9% to 27.7%, respectively. These values are based on $\log_{10}$-transformed data (Reference 16.5).

13.6.2 Table 1 contains the statistical summary of the collaborative study (Reference 16.9) results.

14.0 Pollution Prevention

14.1 Pollution prevention is any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. It is the environmental management tool preferred over waste disposal or recycling. When feasible, laboratory staff should use a pollution prevention technique, such as preparation of the smallest practical volumes of reagents, standards, and media or downsizing of the test units in a method.

14.2 The laboratory staff should also review the procurement and use of equipment and supplies for other ways to reduce waste and prevent pollution. Recycling should be considered whenever practical.

15.0 Waste Management

15.1 The Environmental Protection Agency requires that laboratory waste management practices be consistent with all applicable rules and regulations. The Agency urges

laboratories to protect the air, water, and land by minimizing and controlling releases from hoods and bench operations, complying with the letter and spirit of sewer discharge permits and regulations and by complying with solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. All infectious wastes should be autoclaved before disposal.

16.0 References

16.1 American Chemical Society. 1981. Reagent Chemicals. *In* American Chemical Society Specifications, 6th edition. American Chemical Society, Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K. and the United States Pharmacopeia.

16.2 American Public Health Association. 1992. Standard Methods for the Examination of Water and Wastewater, 18th edition. American Public Health Association, Washington, D.C.

16.3 American Society for Testing and Materials. 1993. Standard Specification for Reagent Water, Designation D1193-91, p. 45-47. *In* 1993 Annual Book of ASTM Standards: Water and Environmental Technology, Volume 11.01. American Society for Testing and Materials, Philadelphia, PA.

16.4 American Society for Testing and Materials. 1994. Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water, Designation D 2777-86, p. 31-44. *In* 1994 Annual Book of ASTM Standards, Section 11: Water and Environmental Technology, Volume 11.01. American Society for Testing and Materials, Philadelphia, PA.

16.5 Association of Official Analytical Chemists. 1989. Guidelines for Collaborative Study Procedure to Validate Characteristics of a Method of Analysis. Journal of the Association of Official Analytical Chemists 72 (4): 694-704.

16.6 Bordner, R., J. Winter, and P. Scarpino (ed). 1978. Microbiological Methods for Monitoring the Environment: Water and Wastes. EPA-600/8-78-017, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH.

16.7 Brenner, K.P., and C.C. Rankin. 1990. New Screening Test to Determine the Acceptability of 0.45-µm Membrane Filters for Analysis of Water. Applied and Environmental Microbiology 56: 54-64.

16.8 Brenner, K.P., and C.C. Rankin, Y.R. Roybal, G.N. Stelma, Jr., P.V. Scarpino, and A.P. Dufour. 1993. New Medium for the Simultaneous Detection of Total Coliforms and *Escherichia coli* in Water. Applied and Environmental Microbiology 59: 3534-3544.

16.9 Brenner, K.P., C.C. Rankin, and M. Sivaganesan. 1996. Interlaboratory Evaluation of MI Agar and the U.S. Environmental Protection Agency-Approved Membrane Filter Method for the Recovery of Total Coliforms and *Escherichia coli* from Drinking Water. Journal of Microbiological Methods 27: 111-119.

16.10 Brenner, K.P., C.C. Rankin, M. Sivaganesan, and P.V. Scarpino. 1996. Comparison of the Recoveries of *Escherichia coli* and Total Coliforms from Drinking Water by the MI Agar Method and the U.S. Environmental Protection Agency-Approved Membrane Filter Method. Applied and Environmental Microbiology 62 (1): 203-208.

16.11 Buntel, C.J. 1995. *E. coli* $-Glucuronidase (GUS) as a Marker for Recombinant Vaccinia Viruses. BioTechniques 19 (3); 352-353.

16.12 Federal Register. 1985. National Primary Drinking Water Regulations; Synthetic Organic Chemicals, Inorganic Chemicals and Microorganisms; Proposed Rule. *Federal Register* 50: 46936-47022.

16.13 Federal Register. 1994. National Primary and Secondary Drinking Water Regulations: Analytical Methods for Regulated Drinking Water Contaminants; Final Rule. *Federal Register* 59: 62456-62471.

16.14 Federal Register. 1999. National Primary and Secondary Drinking Water Regulations: Analytical Methods for Chemical and Microbiological Contaminants and Revisions to Laboratory Certification Requirements; Final Rule. *Federal Register* 64: 67450-67467.

16.15 U.S. Environmental Protection Agency. 1992. Manual for the Certification of Laboratories Analyzing Drinking Water: Criteria and Procedures, Quality Assurance, Third Edition. EPA-814B-92-002, Office of Ground Water and Drinking Water, Technical Support Division, U.S. Environmental Protection Agency, Cincinnati, OH.

17.0 Tables and Figures

Table 1. Statistical Summary of the Collaborative Study Results[1]

Target Organism	Sample Number	*E. coli* Count Category (Range)[2]	Initial n[3]	Final n[4]	S_r[5]	RSD_r[6] (%)	$\overline{X}$[7]	S_R[8]	RSD_R[9] (%)	$\frac{RSD_R}{RSD_r}$ Ratio
Escherichia coli	1	Low (≤ 10)	63	63	0.17	27.3	0.64	0.26	40.5	1.49
	2		63	63	0.21	25.0	0.84	0.33	39.0	1.56
	3	Medium (11-30)	63	63	0.10	7.9	1.27	0.15	12.1	1.52
	4		63	60	0.07	5.6	1.32	0.12	9.2	1.65
	5	High (> 30)	63	60	0.06	3.3	1.87	0.16	8.6	2.62
	6		63	63	0.09	4.3	1.99	0.25	12.6	2.91
Total Coliforms	1	Low (≤ 10)	63	63	0.10	4.3	2.35	0.62	26.4	6.11
	2		63	63	0.09	3.8	2.31	0.64	27.7	7.25
	3	Medium (11-30)	63	63	0.11	5.1	2.17	0.47	21.8	4.28
	4		63	57	0.10	3.3	3.07	0.21	6.9	2.08
	5	High (> 30)	63	63	0.15	4.8	3.10	0.43	14.0	2.96
	6		63	63	0.08	2.5	3.14	0.46	14.7	5.97

[1] The values are based on $\log_{10}$ transformed data (Reference 16.5).

[2] The samples were grouped by their *E. coli* count on MI agar into the following categories:
Low (≤ 10 *E. coli* / 100 mL, samples 1 and 2),
Medium (11-30 *E. coli* / 100 mL, samples 3 and 4), and
High (> 30 *E. coli* / 100 mL, samples 5 and 6).
[3] These values are based on triplicate analyses by each laboratory. The reference laboratory analyzed three sets of samples: the initial and final samples prepared and a sample shipped along with the other 18 lab samples.
[4] These values were obtained after removing outliers by the AOAC procedure (Reference 16.5).
[5] Sr, Single Operator Standard Deviation, a measure of repeatability.
[6] RSDr, Single Operator Relative Standard Deviation (Coefficient of Variance), a measure of repeatability.
[7] $\overline{X}$, The mean of the replicate analyses for all laboratories.
[8] SR, Overall Standard Deviation, a measure of reproducibility.
[9] RSDR, Overall Relative Standard Deviation (Coefficient of Variation), a measure of reproducibility.

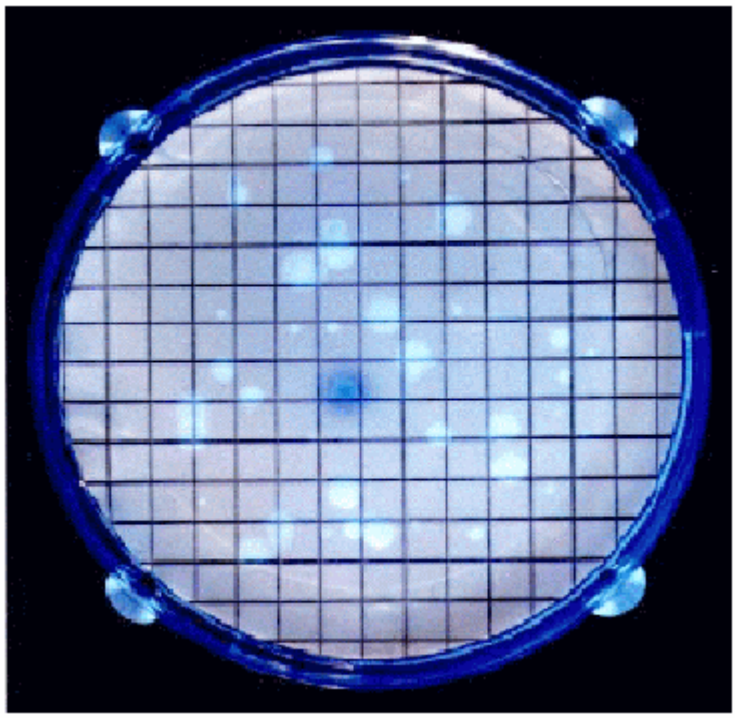


Figure 1. This photograph shows *Escherichia coli* (blue/green fluorescence) and total coliforms other than *E. coli* (blue/white fluorescence) on MI agar under longwave UV light (366 nm). The sample used was a wastewater-spiked Cincinnati, Ohio tap water.

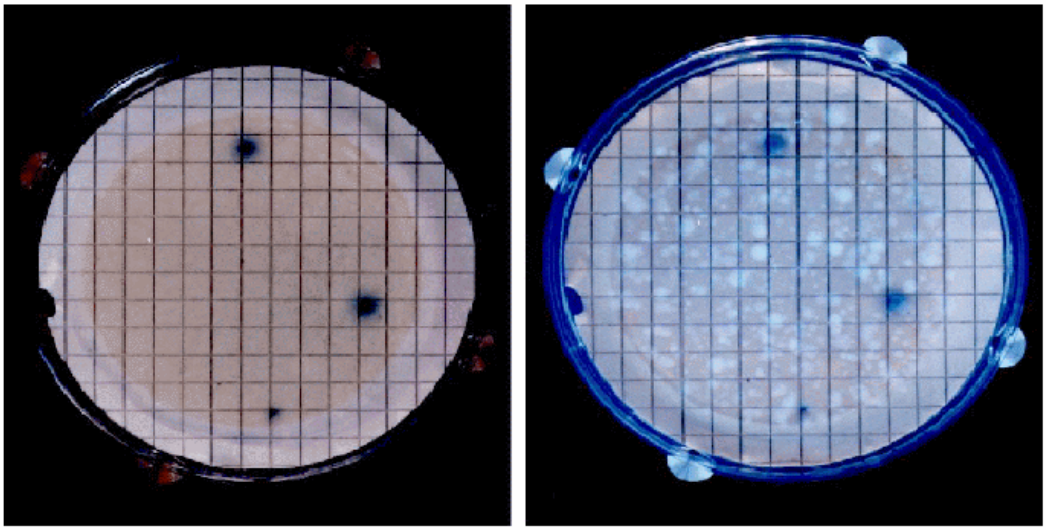


Figure 2. These photographs show *Escherichia coli* and total coliforms from cistern water on MI agar. The confluent plate was photographed under different lighting: ambient light on the left, and longwave UV light (366 nm) on the right. Under ambient light, *E. coli* are blue, and total coliforms other than *E. coli* and non-coliforms are their natural color. Under longwave UV light, all total coliforms, including *E. coli*, are fluorescent, and noncoliforms are non-fluorescent (*i.e.*, they are not visible).

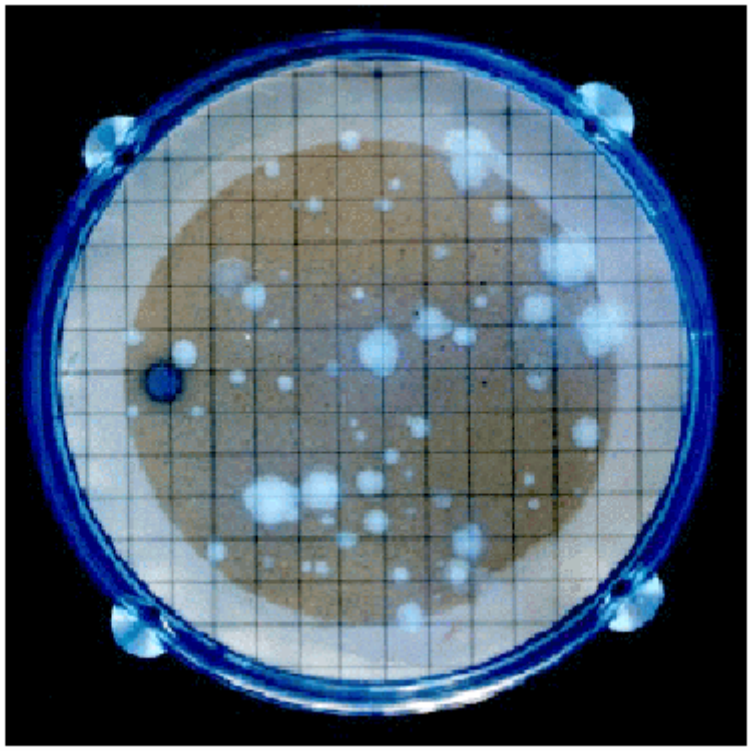


Figure 3. This photograph shows that *Escherichia coli* (blue/green fluorescence) and total coliforms other than *E. coli* (blue/white fluorescence) can easily be detected on MI agar plates from samples with high turbidity levels. The sample used was surface waterspiked Cincinnati, Ohio tap water.

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #12

Laboratory Analysis Methods for Water Quality, Colorado Units

Version 1.0 (4/11/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

This Water Quality Monitoring Standard Operating Procedure (SOP) describes laboratory analysis methods for water samples collected in NCPN Units in the state of Colorado. All samples collected in these parks are sent to the USGS-National Water Quality Laboratory (USGS-NWQL) in Lakewood, Colorado for chemical analysis. Samples are collected and processed as prescribed in SOP 9 and shipped to the laboratory.

Table of Contents

1. Introduction 3
2. References 6
3. Appendix A. Method of analysis for dissolved Cadmium, Copper, Lead, Manganese, Silver, and Zinc using USGS method I-2477-92. 7
4. Appendix B. Method of analysis for dissolved Calcium and Magnesium using USGS Method I-1472-87 44
5. Appendix C. Method of analysis for Ammonia Nitrogen using USGS Method I-6522-90. 58
6. Appendix D. Method of analysis for Low Ionic Strength Nitrate Nitrogen using USGS Method I-2542-89 68
7. Appendix E. Method of analysis for Low Ionic Strength Nitrite plus Nitrate Nitrogen using USGS Method I-2546-91. 76
8. Appendix F. Method of analysis for dissolved Total Phosphorus using USGS Method I-4607-90. .. 86
9. Appendix G. Method of analysis for Laboratory pH using USGS Method I-1586-85 or I-2587-89. 95
10. Appendix H. Method of analysis for dissolved Selenium using USGS Method I-2477-92. 99
11. Appendix I. Glossary of terms from USGS National Water Quality Laboratory 138

List of Tables

Table 1. Laboratory Analyses conducted on samples from DINO, CURE and BLCATable 12-1. 4
Table 2. Explanation of containers, sample sizes, and treatments. 5

1. Introduction

Methods of analysis used by the USGS-NWQL are published by the U.S. Geological Survey and are available in various reports. These are reproduced in this SOP without alteration as Appendices A through H. In some cases, portions of larger documents have been excerpted.

The USGS is responsible for laboratory operations and the quality of the results produced.

Since the USGS has developed numerous methods for laboratory analysis, the specific methods used for the analytes identified for NCPN-Colorado units are presented in Table 12-1. This is provided to avoid confusion between the many methods used at various times and for various reasons for the same analyte. Also included on Table 12-1 are:

1. Laboratory reporting limits. The National Water Quality Laboratory collects quality-control data from selected analytical methods on a continuing basis to determine long-term method detection levels (LT-MDL's) and establish laboratory reporting levels (LRL's). These values are re-evaluated annually based on the most current quality-control data and may change.
2. The USGS Method Identifier,
3. The specific report and page numbers where the step-by-step procedures can be found,
4. The Container used and volume sampled, and
5. The appropriate Appendix in this SOP where the method is reproduced.

Hydrologists in Colorado parks collect 1 field blank and 1 field replicate per 21 samples. (1 blank, 1 rep per field event). USGS recommends 1 blank and 1 rep per 10 sites, but only requires 1 blank and 1 rep per 20 sites. The analysis of blanks is for the same suite of parameters as that being analyzed for the native site water (Malick personal comm.). Historically, the replicates have always been nearly identical, and in only one case has a blank had detection. In that instance it was obvious what caused the problem and thus was easily corrected. Such a high level of success in quality control is testimony to protocol and methods execution implemented in Colorado park units.

Table 1. Laboratory Analyses conducted on samples from DINO, CURE and BLCATable 12-1.

Analyte	USGS-NWQL Lab Code 1	USGS-NWIS Parameter Code 2	USGS Method Version Code 3	Reporting Limit 4	Unit	Reporting Limit Type 4	Method Description [5]	USGS Method Identifier	Reference	Page	Container (Table 12-2)	SOP Appendix for step-by-step
cadmium	**1788**	**01025**	**G**	0.04	µg/L	LRL	ICP-MS	I-2477-92	OFR 92-634	i-28	**FA**	**A**
calcium	**659**	**00915**	**D**	0.02	mg/L	LRL	ICP – AES	I-1472-87	OFR 93-125	101-112	**FA**	**B**
copper	**1791**	**01040**	**G**	0.4	µg/L	LRL	ICP-MS	I-2477-92	OFR 92-634	i-28	**FA**	**A**
lead	**1792**	**01049**	**G**	0.08	µg/L	LRL	ICP-MS	I-2477-92	OFR 92-634	i-28	**FA**	**A**
magnesium	**663**	**00925**	**C**	0.008	mg/L	LRL	ICP – AES	I-1472-87	OFR 93-125	101-112	**FA**	**B**
manganese	**1793**	**01056**	**G**	0.2	µg/L	LRL	ICP-MS	I-2477-92	OFR 92-634	i-28	**FA**	**A**
pH, laboratory	**68**	**00403**	**A**	0.1	pH	MRL	Electrometric, Glass-Electrode	I-1586-85	TWRI B5-A1/89	363-364	**RU**	**G**
selenium	**2506**	**01145**	**C**	0.4	µg/L	LRL	ICP-MS	I-2477-92	OFR 99-093	i-31	**FA**	**H**
silver	**1796**	**01075**	**G**	0.20	µg/L	LRL	ICP-MS	I-2477-92	OFR 92-634	i-28	**FA**	**A**
zinc	**1798**	**01090**	**G**	0.6	µg/L	LRL	ICP-MS	I-2477-92	OFR 92-634	i-28	**FA**	**A**
nitrogen, ammonia	**1980**	**00608**	**H**	0.010	mg/L	LRL	Colorimetry, Salicylate-hypochlorate, automated-segmented flow	I-6522-90	OFR 93-125	125-132	**FCC**	**C**
nitrogen, nitrite (Low Ionic strength)	**1977**	**00613**	**H**	0.002	mg/L	LRL	Colorimetry, diazotization, automated-segmented flow	I-2542-89	OFR 93-125	137-142	**FCC**	**D**
nitrogen, nitrite +	**1979**	**00631**	**G**	0.016	mg/L	LRL	Colorimetry, cadmium reduction-	I-2546-	OFR 93-125	149-156	**FCC**	**E**

Analyte	USGS-NWQL Lab Code [1]	USGS-NWIS Parameter Code [2]	USGS Method Version Code [3]	Reporting Limit [4]	Unit	Reporting Limit Type [4]	Method Description [5]	USGS Method Identifier	Reference	Page	Container (Table 12-2)	SOP Appendix for step-by-step
nitrate (Low Ionic strength)							diazotization, automated-segmented flow	91				
phosphorus, total	**2333**	**00665**	**G**	.004	mg/L	LRL	Colorimetry, phosphomolybdate, automated-segmented flow	I-4607-90	OFR 93-125	175-181	**WCA**	**F**

[1] USGS-NWQL Lab Code – Specific Laboratory analysis as requested from the USGS National Water Quality Laboratory Schedule of Analyses.
[2] USGS-NWIS Parameter Code – Parameter identifier under which results will be reported to the USGS National Water Information System (water quality database).
[3] USGS Method Code – This letter designation is assigned sequentially with each revision of the method. Identical Method code letters assigned to two or more analytes do not necessarily mean that the same method is used. For example a method "D" only indicates that this is the forth version of methods used for that analyte.
[4] Reporting Limit and Reporting Limit Type – The minimum concentration reported by the Laboratory. These can change a little year-to year based on laboratory performance checks. Reporting limit types include:
LRL = Lab Reporting Limit (2 x the Long-Term Minimum Detection Limit (LTMDL))
MRL = Minimum Reporting Limit
[5] Method Description - abbreviations used:
ICP-MS = Mass Spectrometry
ICP – AES = Inductively Coupled Plasma - Atomic Emission Spectrometry

Table 2. Explanation of containers, sample sizes, and treatments

Container Code	Description	Volume	Container Type	Treatment and Preservation
FCC	Filtered	125 mL	Brown polyethylene bottle	Filter through 0.45-um filter, use filtered sample to rinse containers, chill and maintain at 4 deg C, ship immediately.
WCA	Whole Water Chilled Acidified	125 mL	Plain (translucent) polyethylene bottle	Use unfiltered sample to rinse bottles. Acidify with 1 mL of 4.5N (4.5 normal) sulfuric acid (H2S04), chill and maintain at 4 deg C, ship immediately.
FA	Filtered and Acidified	250 mL	Polyethylene bottle, acid-rinsed	Filter through 0.45-um filter, use filtered sample to rinse containers and acidify sample with nitric acid (HNO3) to pH < 2.
RU	Unfiltered	250 or 500 mL	Polyethylene bottle	Use unfiltered sample to rinse bottles.

2. References

Faires, L.M., 1993. Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Metals in Water by Inductively Coupled Plasma-Mass Spectrometry: U.S. Geological Survey Open-File Report 92-634, 28p.

Fishman, M.J., ed., 1993. Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments: U.S. Geological Survey Open-File report 93-125, 217p.

Fishman, M.J., and Friedman, L.C., 1989, Methods for determination of inorganic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, 545 p. Method ID: I-2587-89.

Garbarino, John R., 1999. Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Dissolved Arsenic, Boron, Lithium, Selenium, Strontium, Thallium, and Vanadium using Inductively Coupled Plasma-Mass Spectrometry: U.S. Geological Survey Open-File Report 99-093, 31p.

3. Appendix A. Method of analysis for dissolved Cadmium, Copper, Lead, Manganese, Silver, and Zinc using USGS method I-2477-92.

Extracted and reproduced without alteration from USGS Open File Report 92-634, Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Metals in Water by Inductively Coupled Plasma-Mass Spectrometry (Faires, 1993). Pages i–28.

METHODS OF ANALYSIS BY THE U.S. GEOLOGICAL SURVEY NATIONAL WATER QUALITY LABORATORY— DETERMINATION OF METALS IN WATER BY INDUCTIVELY COUPLED PLASMA—MASS SPECTROMETRY

By Lynda M. Faires

U.S. GEOLOGICAL SURVEY

Open–File Report 92–634



Denver, Colorado
1993

U.S. DEPARTMENT OF THE INTERIOR

MANUEL LUJAN, JR., Secretary

U. S. GEOLOGICAL SURVEY

Dallas L. Peck, Director

For additional information
write to:

U.S. Geological Survey
Chief, National Water Quality Laboratory
Box 25046, Mail Stop 407
Federal Center
Denver, CO 80225

Copies of this report can be
purchased from:

U.S. Geological Survey
Open-File Reports--ESIC
Box 25425, Mail Stop 517
Federal Center
Denver, CO 80225

CONTENTS

Page

Abstract 1
Introduction 1
Analytical method 3
1. Scope and application 3
2. Summary of method 4
3. Interferences 6
4. Instrumentation and equipment 8
5. Materials, reagents, and standards 10
6. Sample collection and preparation 12
7. Instrument performance 12
8. Procedure 15
9. Calculations 16
10. Reporting of results 16
Discussion of results 16
11. Detection limits and reporting limits 16
12. Accuracy and precision 18
13. Method comparison 21
Conclusions 24
References cited 27

FIGURE

Figure 1. Block diagram showing inductively coupled plasma - mass spectrometer 5

TABLES

Table 1. Metals, masses, detection and reporting limits 4
2. Typical instrument operating parameters 9
3. Preparation of Stock I solutions 13
4. Instrument detection limits 17
5. Method detection limits 18
6. Analyses of U.S. Geological Survey standard reference water sample T-101 19
7. Analyses of National Institute of Standards and Technology SRM 1643b 20
8. Short-term precision at 1- and 2-micrograms-per-liter concentration level 21
9. Short-term precision at 25- and 50-micrograms-per-liter concentration level 22
10. Within-day analyses of U.S. Geological Survey standard reference water sample T-113 23

TABLES--Continued

Page

11. Long-term precision at 25- and 50-micrograms-per-liter concentration level 24
12. Analyses of surface-water samples 25
13. Analyses of ground-water samples 26
14. Analyses of U.S. Geological Survey standard reference water sample T-113 27

CONVERSION FACTORS, ABBREVIATED WATER-QUALITY UNITS, AND ADDITIONAL ABBREVIATIONS

Multiply	By	To obtain
liter per minute (L/min)	0.265	gallon per minute
micrometer (µm)	3.3×10^{-6}	feet
milliliter (mL)	2.64×10^{-4}	gallon
milliliter per minute (mL/min)	3.38×10^{-2}	ounce per minute

Degree Celsius (°C) may be converted to degree Fahrenheit (°F) by using the following equation:

$$°F = 9/5(°C)+32.$$

Abbreviated water-quality units used in this report:

mg/L	milligram per liter
µg/L	microgram per liter
µS/cm	microsiemens per centimeter at 25 degrees Celsius

Other abbreviations used in this report:

amu	atomic mass unit
ASTM	American Society for Testing and Materials
GFAAS	graphite furnace atomic absorption spectrometry
ICP	inductively coupled plasma
ICP-AES	inductively coupled plasma - atomic emission spectrometry
ICP-MS	inductively coupled plasma - mass spectrometry
kW	kilowatt
lb/in^2	pound per square inch
MHz	megahertz
MPV	most probable value
MPVSDV	most probable value standard deviation
m/z	mass-to-charge ratio
SDV	standard deviation
USEPA	U.S. Environmental Protection Agency

The use of firm, trade, and brand names in this report is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.

METHODS OF ANALYSIS BY THE U.S. GEOLOGICAL SURVEY NATIONAL WATER QUALITY LABORATORY — DETERMINATION OF METALS IN WATER BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY

By LYNDA M. FAIRES

ABSTRACT

A method for the determination of dissolved concentration of 15 selected metals in water samples by inductively coupled plasma - mass spectrometry is described. Analysis requires only a few milliliters of sample solution and a few minutes of instrument time. Samples are filtered and acidified at time of collection. Specific conductance of samples is limited to less than 2,500 microsiemens per centimeter at 25 degrees Celsius or equivalent after dilution. The method reporting limits for routine analysis are 1 microgram per liter. Average short-term precision is 1.7 percent relative standard deviation for metal concentrations ranging from 25 to 50 micrograms per liter. The method may be used either as an alternative or as a supplement to existing methods for trace metal analysis in water samples.

INTRODUCTION

Dissolved concentrations of metals in water samples are commonly determined by either graphite furnace atomic absorption spectrometry (GFAAS) (Van Loon, 1985) or inductively coupled plasma - atomic emission spectrometry (ICP-AES) (Montaser and Golightly, 1987; Boumans, 1987; Thompson and Walsh, 1983). GFAAS is used to detect many metals at the microgram-per-liter or submicrogram-per-liter level, but the technique is slow, requiring a separate analysis for each element being determined. ICP-AES is a rapid multielement technique, but its detection limits are higher than GFAAS, sometimes by an order of magnitude or more. A new instrumental technique, inductively coupled plasma - mass spectrometry (ICP-MS) (Montaser and Golightly, 1987; Date and Gray, 1989), combines the low detection limits of GFAAS with the multielement speed of analysis of ICP-AES. ICP-MS is ideally suited to the determination of trace metals in water samples with low total-dissolved-solids content. Instrument detection limits are submicrogram per liter for most metals; method reporting limits

for routine analysis are set at 1 µg/L to ensure a high level of confidence in results from this method and to minimize problems with field or laboratory contamination at the submicrogram-per-liter level.

The ICP-MS technique was co-developed in laboratories in the United Kingdom and the United States in the late 1970s, and the first commercial instrument was available in 1983. Scientists soon began to publish a range of applications (Date and Gray, 1989), such as the certification of standard reference materials and the analysis of geologic and hydrologic samples. At present (1992) there are multiple manufacturers of the instrumentation and several hundred instruments being operated worldwide. The technology is accepted as a rugged and reliable tool for routine analysis and is operational in many government, academic, and commercial analytical laboratories. The U.S. Environmental Protection Agency (USEPA) is in the process of approving two independently developed methods for the analysis of water samples by ICP-MS: USEPA 6020 (Laing and others, 1992) and USEPA 200.8 (Long and Martin, 1992, p. 95-137). The American Society for Testing and Materials (ASTM) also is in the process of approving a standardized ICP-MS method for the analysis of water samples (Edward Zayhowski, U.S. Geological Survey, oral commun., 1992).

This report describes a method for determining dissolved concentrations in the range from 1.0 to 1,000 µg/L for 15 metals in filtered, acidified water samples with a specific conductance less than 2,500 µS/cm. This method is similar to both USEPA 200.8 (Long and Martin, 1992, p. 95-137) and the proposed ASTM method. The method can be used either as an alternative or as a supplement to existing methods at the U.S. Geological Survey's National Water Quality Laboratory (Fishman and Friedman, 1989):

I-1472-87 (metals, atomic emission spectrometric, ICP),
I-2138-87 (cadmium, atomic absorption spectrometric, graphite furnace),
I-2243-87 (cobalt, atomic absorption spectrometric, graphite furnace),
I-2274-87 (copper, atomic absorption spectrometric, graphite furnace),
I-2403-87 (lead, atomic absorption spectrometric, graphite furnace),
I-2503-87 (nickel, atomic absorption spectrometric, graphite furnace),
I-2724-87 (silver, atomic absorption spectrometric, graphite furnace),
I-1055-85 (antimony, atomic absorption spectrometric, hydride).

The ICP-MS method was implemented at the National Water Quality Laboratory in October 1992.

This report provides a detailed description of the method, including sampling and preservation, analysis, quality control, calculation, and reporting of results. Precision and accuracy data based on the analysis of standard reference materials, calculated instrument and method detection limits, method reporting limits, and comparison data based on the analysis of unspiked and spiked surface-water and ground-water samples by both ICP-

AES and ICP-MS are presented for 15 metals. The data in this report apply only to dissolved concentrations of 15 selected metals in filtered, acidified water samples. Subsequent reports may provide supporting data for the analysis of digested whole-water samples, digested tissue samples, or the determination of additional metals by the same analytical protocol.

ANALYTICAL METHOD

Parameters and Codes: Metals, dissolved, I-2477-92

[Lab, laboratory; WATSTORE, Water Data Storage and Retrieval System; CAS, Chemical Abstract Services]

Parameter	Lab code	WATSTORE code	Method code	CAS registry number
Aluminum (µg/L as Al)	1784	01106	G	7429-90-5
Antimony (µg/L as Sb)	1785	01095	G	7440-36-0
Barium (µg/L as Ba)	1786	01005	G	7440-39-3
Beryllium (µg/L as Be)	1787	01010	G	7440-41-7
Cadmium (µg/L as Cd)	1788	01025	G	7440-43-9
Chromium (µg/L as Cr)	1789	01030	G	7440-47-3
Cobalt (µg/L as Co)	1890	01035	G	7440-48-4
Copper (µg/L as Cu)	1791	01040	G	7440-50-8
Lead (µg/L as Pb)	1792	01049	G	7439-92-1
Manganese (µg/L as Mn)	1793	01056	G	7439-96-5
Molybdenum (µg/L as Mo)	1794	01060	G	7439-98-7
Nickel (µg/L as Ni)	1795	01065	G	7440-02-0
Silver (µg/L as Ag)	1796	01075	G	7440-22-4
Uranium (µg/L as U)	1797	22703	G	7440-61-1
Zinc (µg/L as Zn)	1798	01090	G	7440-66-6

1. Scope and application

This method is suitable for the single-element or multielement determination of dissolved concentrations of 15 selected trace metals (see table 1) in water. The method is applicable to surface-water, ground-water, drinking-water, and precipitation samples that have a measured specific conductance of less than 2,500 µS/cm at 25°C. The method is applicable to metals in the concentration range from 1.0 to 1,000 µg/L. The use of new or upgraded instrumentation with an "extended dynamic range" option can extend the upper concentration limit to 200 mg/L. Also samples with a specific conductance greater than the specified limit may be analyzed after appropriate dilution to conform to the specified limit; however, method reporting limits for the original sample will be increased according to the dilution factor.

Table 1.--*Metals, masses, detection and reporting limits*

[amu, atomic mass unit; IDL, instrument detection limit; MDL, method detection limit; MRL, method reporting limit]

Metal	Recommended masses (amu)	Limits measured in micrograms per liter		
		IDL	MDL	MRL
Ag silver	107, 109	0.03	0.07	1
Al aluminum	27	.12	1.18	1
Ba barium	137, 138	.10	.12	1
Be beryllium	9	.13	.07	1
Cd cadmium	111, 114	.17	.16	1
Co cobalt	59	.03	.07	1
Cr chromium	52	.11	.16	1
Cu copper	63, 65	.09	.10	1
Mn manganese	55	.06	.18	1
Mo molybdenum	95, 98	.06	.22	1
Ni nickel	60	.15	.19	1
Pb lead	206, 207, 208	.05	.18	1
Sb antimony	121, 123	.02	.26	1
U uranium	238	.01	.16	1
Zn zinc	64, 66	.12	.22	1

2. Summary of method

2.1 A generalized block diagram of an ICP-MS instrument is shown in figure 1

2.2 Sample solution is pumped by a peristaltic pump into a pneumatic nebulizer (NEB in fig. 1) which generates a liquid aerosol. This aerosol is transported by argon gas flow into a water-cooled spray chamber where the large droplets are removed by gravity and condensation. The small droplets are further swept into a radiofrequency inductively coupled plasma (ICP) where evaporation, molecular dissociation, atomization, and ionization occur. The ions are physically extracted from the center of the plasma by a differentially pumped vacuum system, through a water-cooled sampler cone (SM in fig. 1) and skimmer cone (SK in fig. 1) assembly, which is in physical contact with the horizontally mounted ICP.

2.3 The extracted ions are focused by an electrostatic ion lens (IL in fig. 1) assembly and accelerated into a unit-resolution quadrupole mass spectrometer (QUAD in fig. 1). For a given combination of radiofrequency and direct current voltages applied to the quadrupoles, only ions of a specific

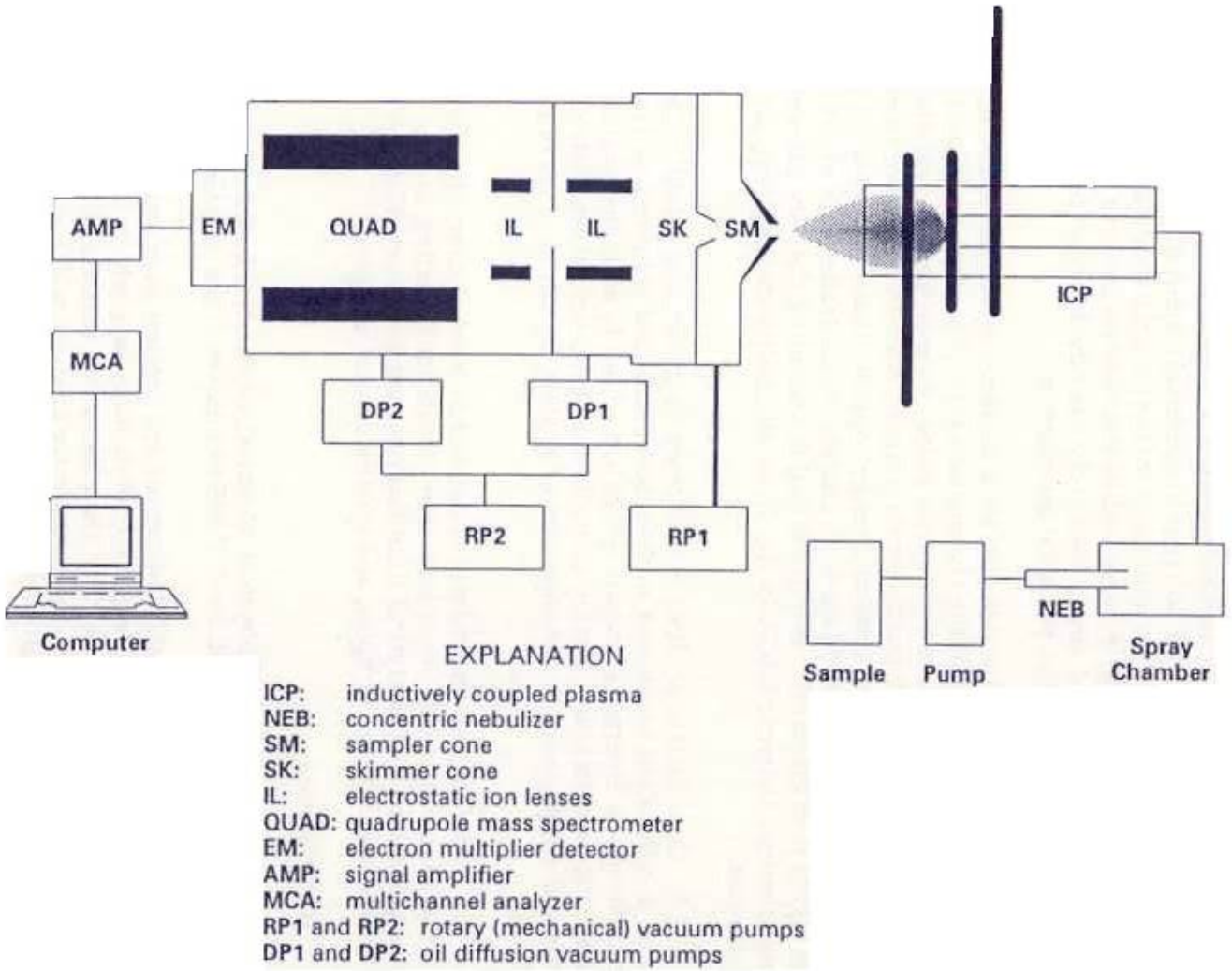


Figure 1.--Block diagram of inductively coupled plasma - mass spectrometer.

mass-to-charge ratio (m/z) pass through the quadrupoles and reach the detector. Rapidly scanning the voltages on the quadrupoles has the effect of rapidly scanning the mass spectrum at the detector. Selected mass values, where high ion signals are expected, can be pre-programmed to be skipped in the mass scan to avoid overload damage to the detector. The mass spectrometer alternatively can be operated in a peak-jumping mode, which rapidly changes the applied voltages to effectively jump to pre-selected masses instead of operating in the continuous mass-scanning mode already described. Mass-scanning is suggested in this method because the recorded spectrum can be recalled and qualitatively examined for the presence or absence of any analytes or potential interferents.

2.4 The ion signal is detected by a continuous dynode electron multiplier (EM in fig. 1) of the channeltron type. The physical impact of an ion on the detector surface produces a pulse of electrons. The resulting signal is processed in a digital, pulse-counting mode. New or upgraded instrumentation with an "extended dynamic range" option allows operation of the detector in an analog mode for high analyte concentrations. In either case, the ion signal is electronically amplified, and the resulting data are processed by a multichannel analyzer (MCA in fig. 1), or alternative data system, and computer.

2.5 Quantitative analysis is calibrated by generating a calibration curve using a calibration blank and multielement standard solutions of known concentrations. Internal standard spikes are added to all blanks, calibration standards, samples, and quality-control samples to correct for instrumental drift and suppression or enhancement of analyte signals because of matrix effects.

2.6 An "analysis procedure" is defined as a set of computer files, manual and automated operations, and calculations relating to a corresponding set of analytical solutions consisting of a calibration blank, calibration standards, samples, and quality-control samples.

3. Interferences

3.1 Several types of physical, chemical, or spectral interferences are recognized and documented for ICP-MS techniques (Date and Gray, 1989).

3.2 Isobaric elemental interferences are spectral overlaps caused when two or more isotopes of different elements form ions which have the same nominal mass-to-charge ratio. The analytes in this method all have at least one isotope that can be used for quantitative analysis which is free of potential isobaric elemental interference.

3.3 Isobaric polyatomic interferences are spectral overlaps caused when polyatomic ions formed by combinations of plasma gas, atmospheric gases, sample solvent, sample acid, or sample matrix constituents have the same nominal mass-to-charge ratio as the analyte isotope ion. These potential interferences have been systematically documented and tabulated (Tan and Horlick, 1986).

3.3.1 In the development of this method, isobaric spectral interferences occurred for copper in some samples containing sodium and for zinc in some samples containing sulfate. Copper analysis can be interfered by the formation of $NaAr^+$ at m/z 63, and zinc analysis can be interfered by the formation of SO_2^+ at m/z 64. These interferences were observed to be erratic and unpredictable with the parameters and instrumentation used in the development of this method, requiring the precautions described in Section 3.3.2.

3.3.2 In order to recognize isobaric spectral interferences, independently monitor two isotopes of each analyte, whenever possible, during quantitative analysis. If the calculated analytical results for each isotope of a given analyte agree within the acceptable method precision limits, then spectral interferences are not indicated. However, if the calculated analytical results for each isotope of a given analyte differ beyond the acceptable method precision limits, then spectral interferences are indicated for that analyte. In this case, the results for that analyte are not reported, and it needs to be reanalyzed or determined by an alternative approved method.

3.4 Physical interferences are associated with the physical processes of sample transport to the plasma, evaporation, dissociation, and ionization in the plasma, extraction of the ions from the plasma, and transmission of the ions through the mass spectrometer. Physical interferences cause the instrument response to be different, either suppressed or enhanced, for samples than for standard solutions, causing erroneous analytical results. High levels of dissolved salts can particularly cause physical interferences by affecting the sample transport or clogging the small orifice to the mass spectrometer.

3.4.1 In order to minimize physical interferences, the specific conductance of samples is limited to 2,500 µS/cm or equivalent dilution prior to analysis. The use of internal standards compensates for minor physical interferences and also provides a means of detecting major physical interferences. The integrated counts (or calculated concentrations) for each sample replicate need to be printed and monitored during the analysis procedure. Changes in the integrated counts for the internal standards in excess of ±30 percent (or changes in the calculated concentrations in excess

of acceptable method precision) alert the operator to the likely occurrence of physical interferences. Suspect samples need to be diluted and reanalyzed or analyzed by an alternative approved method.

3.5 Memory interferences result when constituents of a prior sample contribute to the measured signal of the current sample. These interferences are caused by sample deposition and subsequent release from the sample tubing, nebulizer, spray chamber, plasma torch, or sampler or skimmer cones.

3.5.1 Memory interferences are minimized by limiting the specific conductance of individual samples and by sufficient flushing of the system between samples with rinse blank. Memory interferences are suspected if the sequential replicate integrated counts (or calculated concentrations) for a given isotope in a sample decrease consecutively while the other isotopes for the sample vary only within acceptable method precision limits. In that case the concentration of that analyte in the previous sample needs to be checked to determine if it was high. If so, the system needs to be thoroughly flushed and the affected sample reanalyzed.

4. Instrumentation and equipment

The instrumentation and equipment needed for this method are listed as follows. Specific brands and models used during the development of this method along with typical operating parameters, where applicable, used to obtain the data in this report, are listed in table 2. Equivalent instrumentation and equipment, demonstrating equivalent or superior performance, may be substituted.

4.1 *Inductively coupled plasma-mass spectrometry system.*

Capable of scanning the mass range from 1 to 280 amu.

4.1.2 Minimum resolution capability of 1 amu peak width at 5 percent of maximum peak height.

4.1.3 Capable of skipping preselected amu values during scan or with "extended dynamic range" option.

4.1.4 Digital pulse-counting detection system or "extended dynamic range" option.

4.1.5 Radio-frequency generator--27.1 or 40 MHz, minimum power 1.5 kW.

Mass flow controller on nebulizer gas supply.

Table 2.--*Typical instrument operating parameters*

Parameter	Unit, value, or instrument manufacturer
Inductively coupled plasma - mass spectrometer	Fisons/VG Instruments, UK, model PlasmaQuad PQ-1
Radiofrequency generator	Henry, 1.25 kilowatts
Inductively coupled plasma torch	Fassel type, quartz
Inductively coupled plasma spray chamber	Scott type, water cooled at 5°C by Neslab RBC-3
Inductively coupled plasma nebulizer	Meinhard type, concentric
Coolant flow rate	13 liters per minute argon
Auxiliary flow rate	0.6 liter per minute argon
Nebulizer flow rate	0.7 liter per minute argon
Solution uptake rate	0.7 milliliter per minute
Peristalic pump	Ismatec
Vacuum water chiller temperature	15°C
Detector mode	Channeltron type, pulse-counting
Replicate integrations	3
Mass range	5-240 atomic mass units
Multichannel analyzer	2,048 channels
Dwell time	320 microseconds
Scans per integration	100
Time per integration	1 minute
Sample uptake time	2 minutes
Rinse time	1 minute
Data acquisition time	3 minutes
Total sample cycle time	6 minutes
Data processing system	Compaq Deskpro 386/20
Printer	Epson FX-86e

4.1.7 ICP glassware--Nebulizer, water-cooled spray chamber, quartz plasma torch.

4.2 *Instrument control and data processing system.*

4.2.1 Capable of acquiring and storing data during analytical procedure, performing calculations, storing, displaying, and printing acquired spectra, printing real-time integrations of sample counts or real-time calculated concentrations, and printing calculated results.

4.2.2 Data transfer system for archiving data.

4.2.3 On-line printer.

4.3 *Peristaltic pump*: Capable of pumping aqueous solutions at adjustable rates ranging from 0.2 to 2.0 mL/min; minimum of four channels.

4.4 *Argon gas regulator*: Capable of delivery of high-purity (99.99 percent) grade argon at variable rates from 0 to 25 L/min with constant back pressure of 80 lb/in^2.

4.5 *Autosampler*: Capable of holding 10-mL sample tubes, with continuously flushing sample probe rinse.

4.6 *Labware*: For trace metal analysis, particular attention is necessary to avoid contamination or loss of analytes, both in the field and in the laboratory. Sample bottles, sample tubes, pipette tips, volumetric ware, and storage ware need to be Teflon, polytetrafluoroethylene, fluorinated ethylene propylene, high density polyethylene, or similar materials. These items need to be acid-cleaned using ASTM Type I water and ultrapure grade nitric acid prior to use.

4.7 *Operating parameters*: Refer to the manufacturers' operating manuals for installation, performance testing, tuning procedures, optimization, and routine maintenance of all instrumentation and equipment used by this method; operating parameters will be specific to a given model and make of instrumentation. The parameters found to be optimum for the equipment used in the development of this method are listed in table 2.

5. Materials, reagents, and standards

5.1 For trace metal analysis, high-purity or ultrapure-grade reagents need to be used. All acids for sample preservation or solution preparation need to be ultrapure grade; nitric acid (HNO_3) is the acid of choice, whenever possible, to minimize isobaric polyatomic spectral interferences and to matrix-match standards and samples.

5.2 *Argon gas supply* (99.99-percent purity).

5.3 *Water*, ASTM Type I reagent water (American Society for Testing and Materials, 1992) or equivalent deionized water.

5.4 *Nitric acid* (HNO_3), concentrated, specific gravity 1.41.

5.5 *Nitric acid*, 2 percent (volume/volume): Dilute 20 mL of concentrated nitric acid to 1,000 mL with ASTM Type I reagent water. Use for rinse blank and calibration blank.

5.6 *Calibration standard and internal standard Stock I solutions*: Commercially prepared and certified single-element or multielement Stock I solutions may be used, or they may be prepared from high-purity chemicals and metals according to approved U.S. Geological Survey methods (Fishman and Friedman, 1989) such as those listed in table 3, approved USEPA methods (Long and Martin, 1992), or approved ASTM methods (American Society for Testing and Materials, 1992). A convenient concentration for Stock I solutions is 1,000 mg/L in 5-percent (volume/volume) nitric acid. These Stock I solutions are stable at room temperature for extended periods (up to 1 year). Stock I solutions need to be analyzed for possible contamination prior to further use.

NOTE: Chemicals combined in any multielement solutions must be chemically compatible and stable.

5.7 *Calibration standard and internal standard Stock II solutions*: Commercially prepared and certified single-element or multielement Stock II solutions may be used, or they may be prepared by dilution from Stock I solutions. A convenient concentration for Stock II solutions is 10 mg/L in 5-percent (volume/volume) nitric acid. Stock II solutions are stable at room temperature for extended periods (up to 1 year).

5.7.1 *Calibration standards*: Single-element or multielement calibration standards of the desired concentrations need to be prepared fresh at the start of each analytical procedure by diluting Stock II solutions to the appropriate concentrations using 2-percent (volume/volume) nitric acid. Single-point calibration can be used, but multipoint calibration is suggested, with concentrations of the standards distributed over the anticipated range of analyte concentrations.

5.7.2 *Internal standards*: Internal standards are added in equal concentrations to all blanks, standards, and samples in an analysis procedure. This may be done by manually spiking each solution with an aliquot of the internal standard Stock II solution or by using a second channel of the peristaltic pump for continuous on-line addition. A convenient resultant concentration for the internal standard spike in the sample solution is 100 µg/L of each internal standard used. Indium may be used as a single internal standard, but the use of three or more internal standards, distributed across the mass range, is suggested. Commonly used internal standards include scandium, yttrium, indium, terbium, and bismuth. Any element may be used as an internal standard if it is similar in mass and chemical behavior to the analyte element and if it is not already present in the sample.

5.8 *Tuning solution*: A multielement solution, distributed across the mass range, in 2-percent (volume/volume) nitric acid is used for daily instrument tuning, mass calibration, resolution, and sensitivity checks. Commonly used elements are beryllium, magnesium, cobalt, indium, and lead. A convenient concentration of the tuning solution is 100 µg/L of each element. Prepare this solution by dilution from Stock I or Stock II solutions. Tuning solution is stable at room temperature for extended periods (up to 1 year).

6. Sample collection and preparation

Filter surface- or ground-water samples collected for the determination of dissolved concentrations of metals through a 0.45-µm filter and acidify to pH less than 2.0 with ultrapure-grade nitric acid as soon after collection as possible. Samples are then stable for moderate periods (up to 6 months) without refrigeration, although extreme temperatures need to be avoided. To avoid contamination, collect and store samples in previously acid-rinsed Teflon, polytetrafluoroethylene, fluorinated ethylene propylene, high density polyethylene, or similar containers, following appropriate field protocols for clean sampling at the microgram-per-liter level.

7. Instrument performance

7.1 *Analytical productivity*: According to the typical operating parameters used in the development of this method (table 2), each sample requires 6 minutes for a complete sample analysis cycle (uptake, three replicate integrations, rinse). A typical analysis procedure of 38 solutions could include 1 blank, 3 calibration standards, 4 quality-control samples, and 30 samples. This setup would require a total analysis time of 3 hours, 48 minutes. On the basis of an 8-hour working day for the analyst, approximately 50 to 60 samples per day can be analyzed using the typical parameters listed in table 2. Productivity can be increased by (a) reducing the scanning time for each integration, (b) reducing the number of replicate integrations for each sample, or (c) increasing the number of samples within each analysis procedure (each calibration). Some loss of accuracy and precision, compared to the data presented in this report, will result from such changes in the operating parameters.

7.2 *Instrument performance evaluation*: Evaluate and optimize instrument performance each day for sensitivity, resolution, and mass calibration using the tuning solution (Section 5.8). Instructions for instrument performance evaluation are given in Procedure (Section 8.2). After tuning, instrument sensitivity needs to be at least 100,000 counts/sec for 100 µg/L solution of indium; instrument resolution needs to be at least 0.75 amu at 5-percent peak height; and mass calibration needs to be within 0.1 amu to proceed with analysis. If the software allows, determine the instrument stability during the course of an analysis procedure by monitoring the integration counts of the internal standards, which need to remain within ±30 percent of their starting values to proceed with analysis.

Table 3.--*Preparation of Stock I solutions*

[°C, degree Celsius; mg/L, milligram per liter; g, gram; mL, milliliter; ASTM, American Society for Testing and Materials]

Obtain ultrahigh-purity grade chemicals or metals from reputable commercial sources. Dry all salts for 1 hour at 105°C and cool in a desiccator, unless otherwise specified. All references to water shall be understood to mean ASTM Type I reagent water (American Society for Testing and Materials, 1992). Prepare Stock I solutions with concentrations of 1,000 mg/L from the following directions.

Aluminum: Dissolve 1.000 g aluminum metal in 100 mL concentrated HCl and 20 mL concentrated HNO_3. Heat to increase rate of dissolution. Continue heating until volume is reduced to 40 mL. Cool and add 40 mL water. Heat until volume is reduced to 20 mL. Cool and dilute to 1,000 mL with water.

Antimony: Dissolve 1.000 g antimony powder in 20 mL (1+1) nitric acid and 5 mL concentrated HCl. Heat to increase rate of dissolution. Cool, add 200 mL water and 1.5 g tartaric acid. Warm the solution to dissolve the white precipitate. Cool and dilute to 1,000 mL with water.

Barium: Dissolve 1.512 g $BaCl_2$, dried at 180°C for 1 hour, in a 1,000-mL volumetric flask containing 100 mL water. In a well-ventilated hood, slowly add 100 mL concentrated HNO_3 with stirring. After the $BaCl_2$ has dissolved, dilute to 1,000 mL with water.

Beryllium: Wash 1.000 g beryllium flakes into a 1,000-mL volumetric flask with a minimum amount of water. Use a well-ventilated hood. Heat to increase rate of dissolution. Add 90 mL concentrated HNO_3 and 10 mL HCl. Cool and dilute to 1,000 mL with water.

Cadmium: Wash 1.000 g cadmium spatters into a 1,000-mL volumetric flask with a minimum amount of water. Add 50 mL concentrated HCl and stir. Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

Chromium: Wash 1.000 g chromium metal into a 1,000-mL volumetric flask with a minimum amount of water. Add 50 mL concentrated HCl and stir. Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

Cobalt: Wash 1.000 g cobalt powder into a 1,000-mL volumetric flask with a minimum amount of water. Add 100 mL concentrated HNO_3. Place in an ultrasonic bath to increase rate of dissolution. Dilute to 1,000 mL with water.

Table 3.--*Preparation of Stock I solutions*--Continued

Copper: Wash 1.000 g copper powder into a 1,000-mL volumetric flask with a minimum amount of water. Slowly add 100 mL concentrated HNO_3 with stirring. If necessary, heat to increase rate of dissolution. Dilute to 1,000 mL with water.

Lead: Wash 1.000 g lead powder into a 1,000-mL volumetric flask with a minimum amount of water. Place flask in an ultrasonic bath in a well-ventilated hood and add 15 mL concentrated HCl. Periodically add 1-mL increments of concentrated HNO_3 for a total volume of 85 mL. Dilute to 1,000 mL with water.

Manganese: Wash 1.000 g manganese flakes into a 1,000-mL volumetric flask with a minimum amount of water. In a well-ventilated hood, slowly add 100 mL concentrated HNO_3. Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

Molybdenum: Wash 1.000 g molybdenum powder into a 1,000-mL volumetric flask with a minimum amount of water. In a well-ventilated hood, while stirring, slowly add 10 mL concentrated HNO_3. After fumes dissipate, add an additional 90 mL of concentrated HNO_3 and 20 mL water. Heat to increase rate of dissolution. If solution turns cloudy and a precipitate appears, increase heat and stir for an additional 15 to 30 minutes. Cool and dilute to 1,000 mL with water.

Nickel: Wash 1.000 g nickel powder into a 1,000-mL volumetric flask containing 100 mL water. In a well-ventilated hood, while stirring, slowly add 100 mL concentrated HNO_3. Heat and stir for 30 minutes or until dissolution is complete. Cool and dilute to 1,000 mL with water.

Silver: Wash 1.000 g silver powder into a 1,000-mL volumetric flask containing 100 mL water. In a well-ventilated hood, while stirring, slowly add 100 mL concentrated HNO_3. Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water. Store in an opaque bottle.

Uranium: Dissolve 2.110 g $UO_2(NO_3)_2 \cdot 6H_2O$ (do not dry) in 200 mL water and dilute to 1,000 mL with water.

Zinc: Wash 1.000 g zinc powder into a 1,000-mL volumetric flask containing 200 mL water. While stirring, slowly add 100 mL concentrated HNO_3. Dilute to 1,000 mL with water.

8. Procedure

8.1 Begin operating the instrument and data system. Allow at least 30 minutes for instrument warm-up and stabilization prior to starting analysis.

8.2 Check the instrument performance to meet the specifications in Section 7.2. Using the tuning solution, tune the ion lenses for maximum sensitivity of the indium 115 peak. Obtain a survey scan of the tuning solution and check the resolution and mass calibration at low-, middle-, and high-mass values; adjust if necessary.

8.3 Set up the appropriate software routines to perform the analysis procedure.

8.4 Prepare blanks, standards, and samples for analysis by pipetting aliquots into tubes for the autosampler rack. If the internal standard is added manually by spiking, then the sample aliquots need to be pipetted to an exact volume. A convenient volume for analysis is 10 mL, but smaller volumes can be analyzed by the use of appropriate tubes and racks. Internal standards are added, either manually or on-line, in equal concentration to all blanks, standards, and samples; the exact concentration need not be known as long as it is consistent in all solutions within a given analysis procedure.

8.5 Calibrate for the analysis procedure by using a calibration blank and one or more single-element or multielement standard solutions at one or more concentration levels for each metal. The first sample in the analytical procedure needs to be a quality-control sample to verify the calibration.

8.6 Analyze the samples. Allow sufficient uptake time for each sample prior to data acquisition for sample transport and signal stabilization. Obtain replicate integrations for each sample. Allow sufficient time to flush the system with rinse blank between samples.

8.7 Verify the on-going operation of the analytical system by inserting quality-control samples after the calibration standards, after every 10 samples or fewer, and at the end of the analysis procedure. The measured concentrations for the quality-control samples need to be within acceptable limits of the expected concentrations to verify the analysis of each subset of samples between two quality-control samples. Reanalyze samples not bracketed by acceptable quality-control samples.

8.8 Print the integrated counts (or calculated concentrations) for each sample replicate during the course of the analysis procedure. The analyst needs to review these values for any abnormalities that indicate interferences (see Sections 3.3.2, 3.4.1, 3.5.1).

9. Calculations

9.1 Obtain replicate integrations for each blank, standard, and sample.

9.2 Specify standards and sample to be blank-subtracted in the setup of the analytical procedure.

9.3 Request full statistics so that results are calculated independently for each isotope specified in the analysis procedure.

9.4 Calibration and calculations are performed by the instrument data system. Integrated counts for analyte isotopes are compared to the appropriate internal standards during the calculation of the results.

9.5 If dilutions were made, apply the appropriate multiplication factors to the sample results and to the method reporting limits for that sample.

9.6 Review the results to determine if the independently calculated values for two isotopes of the same analyte in the same sample are within the acceptable method precision. If so, then report the abundance-weighted average of the two isotope results. If not, the result is not acceptable, and the sample will need to be reanalyzed or analyzed by an alternative approved method for that metal.

10. Reporting of results

Report dissolved concentrations of metals as follows: less than 10 µg/L, two significant figures; 10 to 100 µg/L, three significant figures; 100 to 1,000 µg/L, four significant figures. Report results less than 1.0 µg/L as "less than method reporting limit."

DISCUSSION OF RESULTS

11. Detection limits and reporting limits

11.1 Instrument detection limits are listed in table 4. These values were obtained by analyzing seven aliquots of calibration blank solution (2-percent ultrapure HNO_3 in ASTM Type I water) consecutively in a single analysis procedure, following the procedure described in Section 8. The results were not blank-subtracted, in order to have nonzero numbers for the calculations. The standard deviation of each metal for the seven replicate analyses was multiplied by the appropriate t-value for 99-percent confidence level to obtain the corresponding instrument detection limit. The average instrument detection limit for the 15 analytes was 0.08 µg/L.

11.2 Method detection limits are listed in table 5. These values were obtained by analyzing seven aliquots of a laboratory-prepared multielement solution with concentrations of the metals near the expected detection limit

Table 4.--*Instrument detection limits*

[meas. conc., measured concentration; IDL, instrument detection limit. All measurements in micrograms per liter]

Metal	Average meas. conc.	Standard deviation	IDL
Ag	0.03		0.03
Al	.58		.12
Ba	.07		.10
Be	.22		.13
Cd	.14		.17
Co	.07		.03
Cr	1.49		.11
Cu	.20		.09
Mn	.23		.06
Mo	.08		.06
Ni	.62		.15
Pb	.09		.05
Sb	.05		.02
U	.01		.01
Zn	.39		.12

Samples were not blank-subtracted.
Number of replicates = 7.
Degrees of freedom = 6.
t-value (99-percent confidence) = 3.143.
Average IDL = 0.08 µg/L.

of the method. The seven aliquots were analyzed consecutively in a single analysis procedure, following the procedure described in Section 8. The results were blank-subtracted. The standard deviation of each analyte for the seven replicate analyses was multiplied by the appropriate t-value for 99-percent confidence level to obtain the corresponding method detection limit. The average method detection limit for the 15 analytes was 0.22 µg/L.

11.3 Method reporting limits for routine analysis following the procedure described in Section 8 are suggested to be 1 µg/L for each metal. This method reporting limit is approximately five times greater than the average determined method detection limit. The purpose of using method reporting limits rather than method detection limits is to minimize the problems associated with field and laboratory contamination at the submicrogram-per-liter level and to ensure a high degree of confidence in

Table 5.--*Method detection limits*

[meas. conc., measured concentration; MDL, method detection limit. All measurements in micrograms per liter]

Metal	Nominal concentration	Average meas. conc.	Standard deviation	MDL
Ag	2	2.06	0.02	0.07
Al	2	2.21	.38	1.18
Ba	2	2.10	.04	.12
Be	1	.91	.02	.07
Cd	1	1.00	.05	.16
Co	1	.96	.02	.07
Cr	2	1.87	.05	.16
Cu	1	.94	.03	.10
Mn	1	.93	.06	.18
Mo	1	1.05	.07	.22
Ni	1	.91	.06	.19
Pb	1	.97	.06	.18
Sb	1	1.01	.08	.26
U	1	1.10	.05	.16
Zn	2	1.91	.07	.22

Number of replicates = 7.
Degrees of freedom = 6.
t-value (99-percent confidence) = 3.143.
Average MDL = 0.22 µg/L.

analytical results from the routine mode of operation. Reporting limits less than the suggested 1 µg/L method reporting limit could be used in the case of custom analyses where particular care is taken in both the field and laboratory work associated with the samples and the analyses.

12. Accuracy and precision

12.1 The accuracy of the method was verified by the analyses of standard reference water samples with certified or most probable values for the dissolved concentrations of the constituent analytes. The results for U.S. Geological Survey standard reference water sample T-101 (trace constituents) are shown in table 6, and the results for National Institute of Standards and Technology SRM 1643b (trace elements in water) are listed in table 7.

Table 6.--*Analyses of U.S. Geological Survey standard reference water sample T-101*

[MPV, most probable value; MPVSDV, most probable value standard deviation; meas. conc., measured concentration; SDV, measured standard deviation; NC, not certified. All measurements in micrograms per liter]

Metal	MPV	MPVSDV	Average meas. conc.	SDV
Ag	4.94	1.11	4.89	0.23
Al	87.65	13.62	81.46	8.51
Ba	65.00	15.93	60.38	3.44
Be	14.45	2.24	14.56	1.04
Cd	9.93	1.49	9.42	.26
Co	11.94	3.50	9.55	.73
Cr	18.00	3.22	15.27	.34
Cu	49.96	6.62	48.45	4.69
Mn	50.41	4.09	50.02	1.32
Mo	49.50	3.29	48.82	3.16
Ni	31.81	5.78	31.60	2.32
Pb	17.90	6.08	18.58	.54
Sb	10.28	1.42	10.93	.52
U	NC	NC	13.57	.59
Zn	66.25	7.58	76.45	6.74

Twenty-one analyses over 9 days.

These results were obtained from multiple analyses over several days, following the procedure described in Section 8. Agreement was found between the most probable values and the average measured concentrations. In most cases, the average measured concentration was within two times the most probable standard deviation of the most probable value. Also in most cases, the measured standard deviation was less than, or similar to, the most probable standard deviation.

12.2 Short-term (single-operator, within-day) precision was determined by analyzing seven aliquots each of two laboratory-prepared multielement solutions consecutively in a single analysis procedure following the procedure described in Section 8. The results are listed in tables 8 and 9. Relative standard deviation at the 1- and 2-µg/L concentration level ranged from 1 to 8 percent, with the exception of Al which was about 17 percent

Table 7.--*Analyses of National Institute of Standards and Technology SRM 1643b*

[MPV, most probable value; MPVSDV, most probable value standard deviation; meas. conc., measured concentration; SDV, measured standard deviation; NC, not certified; <MRL, less than method reporting limit. All measurements in micrograms per liter]

Metal	MPV	MPVSDV	Average meas. conc.	SDV
Ag	9.80	0.80		1.23
Al	NC	NC		2.11
Ba	44.00	2.00		2.80
Be	19.00	2.00		1.66
Cd	20.00	1.00		.53
Co	26.00	1.00		1.93
Cr	18.60	.40		.48
Cu	21.90	.40		2.27
Mn	28.00	2.00		1.28
Mo	85.00	3.00		7.90
Ni	49.00	3.00		4.11
Pb	23.70	.70		.89
Sb	NC	NC		<MRL
U	NC	NC		<MRL
Zn	66.00	2.00		5.36

Twenty-one analyses over 9 days.

(table 8). The average relative standard deviation at this concentration level for the 15 metals was 5.2 percent. Relative standard deviations at the 25- and 50-µg/L concentration level ranged from 1 to 4 percent (table 9). The average relative standard deviation at this concentration level for the 15 metals was 1.7 percent. Short-term precision and accuracy results for U.S. Geological Survey standard reference water sample T-113 are given in table 10.

12.3 Long-term (single-operator, multiday) precision was determined by analyzing several aliquots of a laboratory-prepared multielement solution in several analysis procedures on multidays. The results are listed in table 11. The relative standard deviations at the 25- and 50-µg/L concentration level ranged from 2 to 9 percent. The average relative standard deviation at this concentration level for the 15 metals was 4.6 percent. Long-term precision data also are listed in tables 6 and 7 for the analyses of standard reference water samples.

Table 8.--*Short-term precision at 1- and 2-micrograms-per-liter concentration level*

[meas. conc., measured concentration; µg/L, microgram per liter]

Metal	Nominal concentration (µg/L)	Average meas. conc. (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
Ag	2	2.06	0.02	1.15
Al	2	2.21	.38	16.99
Ba	2	2.10	.04	1.78
Be	1	.91	.02	2.46
Cd	1	1.00	.05	5.06
Co	1	.96	.02	2.26
Cr	2	1.87	.05	2.78
Cu	1	.94	.03	3.47
Mn	1	.93	.06	6.30
Mo	1	1.05	.07	6.70
Ni	1	.91	.06	6.51
Pb	1	.97	.06	5.75
Sb	1	1.01	.08	8.12
U	1	1.10	.05	4.72
Zn	2	1.91	.07	3.70

Number of replicates = 7.
Average precision = 5.2 percent relative standard deviation.

13. Method comparison

A surface-water sample was collected from the South Platte River, near Henderson, Colorado. A ground-water sample was collected from a U.S. Geological Survey well in Jefferson County, Colorado. Each water sample was filtered and acidified at time of collection and subsequently split into four subsamples, which were analyzed by ICP-MS and ICP-AES to compare analytical performance of the two methods. One subsample was unspiked; one subsample was spiked at 20 µg/L for each metal; one subsample was spiked at 100 µg/L for each metal; and one subsample was spiked at 500 µg/L for each metal. Each subsample was analyzed in replicates of seven aliquots consecutively within an analysis procedure. Results for the surface-water study are presented in table 12, and results for the ground-water study are presented in table 13. U.S. Geological Survey standard reference water sample T-113 (trace constituents) was used in all the analyses as a quality-control sample. The results for both ICP-MS and ICP-AES analyses of T-113 are presented in table 14.

Table 9.--*Short-term precision at 25- and 50-micrograms-per-liter concentration level*

[meas. conc., measured concentration; µg/L, microgram per liter]

Metal	Nominal concentration (µg/L)	Average meas. conc. (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
Ag	50	50.76	0.48	0.94
Al	50	46.04	.83	1.79
Ba	50	49.40	.81	1.64
Be	25	23.65	.25	1.05
Cd	25	24.88	.42	1.69
Co	25	24.74	.31	1.26
Cr	50	47.75	.40	.84
Cu	25	23.78	.24	1.02
Mn	25	24.15	.15	.63
Mo	25	26.06	.81	3.11
Ni	25	23.96	.38	1.58
Pb	25	26.06	1.10	4.21
Sb	25	25.08	.41	1.63
U	25	28.32	.71	2.51
Zn	50	49.25	.64	1.30

Number of replicates = 7.
Average precision = 1.7 percent relative standard deviation.

Analyses of unspiked and spiked surface-water and ground-water samples and U.S. Geological Survey standard reference water sample T-113 by ICP-MS and ICP-AES were in agreement. The concentrations of several metals were less than the method reporting limit for ICP-AES in the unspiked samples, but all analytes were within the detection range of both techniques in the spiked samples.

Some disagreement in the results for Al by the two techniques was observed. On the basis of the measured recoveries in the spiked samples, the Al results by ICP-AES probably are more accurate than by ICP-MS. Al results by ICP-MS have a greater degree of uncertainty than the other metals included in the method, as indicated by the data in tables 5, 8, 11, and 12. During development of this method, it was observed that the quality of results for Al was variable. This problem might be instrument dependent rather than technique or method dependent. Also, it was observed that Al and Zn contamination at the 1- to 2-µg/L level can occur easily and randomly

Table 10.-*Within-day analyses of U.S. Geological Survey standard reference water sample T-113*

[MPV, most probable value; MPVSDV, most probable value standard deviation; meas. conc., measured concentration; SDV, measured standard deviation; NR, not reported; NC, not certified; <MRL, less than method reporting limits. All measurements in micrograms per liter]

Metal	MPV	MPVSDV	Average meas. conc.	SDV
Ag	5.00	0.30	5.43	0.22
Al	317.00	8.00	331.00	6.56
Ba	70.00	2.00	73.07	2.16
Be	10.00	.90	10.99	.72
Cd	4.23	.18	4.40	.29
Co	10.20	.40	10.19	.27
Cr	2.50	.70	1.05	.25
Cu	47.00	1.00	NR	NR
Mn	65.00	1.00	63.06	1.08
Mo	34.00	2.00	37.00	1.37
Ni	2.10	.30	2.76	.37
Pb	1.30	1.30	.43	.08
Sb	18.90	1.60	20.40	.61
U	NC	NC	<MRL	<MRL
Zn	55.50	1.30	50.09	1.70

Eighteen analyses within-day.

in noncleanroom laboratory conditions. Quality-control sample results need to be carefully monitored during use of this method to ensure reliable results for all metals, especially Al.

Measured recoveries in the spiked samples were good for all metals except Ag, which was observed to be low by both techniques. This result is assumed to be due to the presence of chloride in the natural-water samples, causing the Ag to be lost from solution by precipitation. The recoveries by ICP-MS for Ag were lower than by ICP-AES probably because the samples were analyzed by ICP-MS later than by ICP-AES, and more Ag had been lost by precipitation during the interval. Unspiked natural-water samples are unlikely to contain Ag at concentrations greater than a few micrograms per liter, and loss by precipitation is not anticipated to be a problem.

Table 11.--*Long-term precision at 25- and 50-micrograms-per-liter concentration level*

[meas. conc., measured concentration; µg/L, microgram per liter]

Metal	Nominal concentration (µg/L)	Average meas. conc. (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
Ag	50	52.95	1.59	3.01
Al	50	51.85	2.55	4.92
Ba	50	49.53	2.03	4.09
Be	25	27.73	1.81	6.51
Cd	25	27.13	1.16	4.28
Co	25	24.91	.61	2.44
Cr	50	50.83	1.60	3.15
Cu	25	25.78	.76	2.94
Mn	25	25.53	.74	2.91
Mo	25	25.00	.96	3.85
Ni	25	25.76	.87	3.38
Pb	25	23.56	2.20	9.34
Sb	25	26.31	.87	3.31
U	25	23.34	2.19	9.40
Zn	50	54.94	3.05	5.56

Number of replicates = 7.
Average precision = 4.6 percent relative standard deviation.

CONCLUSIONS

From the data presented in this report, inductively coupled plasma - mass spectrometry is shown to be a useful and reliable analytical method for the determination of trace metals in water. This report presents a method for the routine analysis of 15 metals in natural-water samples with a specific conductance less than 2,500 µS/cm or equivalent after dilution. Method reporting limits are set at 1 µg/L to ensure reliability of analytical results and avoid problems of sample contamination at the submicrogram-per-liter level. Average short-term, single-operator, within-day precision is 5.2 percent at the 1- and 2-µg/L concentration level and 1.7 percent at the 25- and 50-µg/L concentration level. Average long-term, single-operator, multiday precision is 4.6 percent at the 25- and 50-µg/L concentration level.

Table 12.--*Analyses of surface-water samples*

[ICP-AES, inductively coupled plasma - atomic emission spectrometry; ICP-MS, inductively coupled plasma - mass spectrometry; µg/L, microgram per liter; <MRL, less than method reporting limit; NA, not analyzed]

Metal	ICP-AES (µg/L)				ICP-MS (µg/L)			
	Unspiked	Low	Medium	High	Unspiked	Low	Medium	High
				Average measured concentrations				
Ag	<MRL	17.7	85.0	421.9	<MRL	16.5	77.9	344.8
Al	21.9	41.2	118.6	524.2	7.1	22.1	124.1	495.7
Ba	40.4	60.2	136.4	530.4	43.0	63.3	147.0	565.0
Be	<MRL	20.3	99.8	507.3	<MRL	19.1	111.4	516.8
Cd	<MRL	20.6	95.8	491.9	<MRL	19.0	102.1	502.3
Co	<MRL	20.3	100.9	504.5	<MRL	19.3	109.7	429.6
Cr	<MRL	23.7	99.5	499.2	1.6	19.3	109.7	197.5
Cu	<MRL	23.3	102.9	498.4	3.5	20.5	105.6	460.6
Mn	290.5	315.0	389.8	770.7	298.0	312.7	442.6	786.1
Mo	27.1	47.4	120.5	528.7	24.3	44.4	136.9	572.3
Ni	<MRL	27.7	106.4	499.2	10.4	27.4	92.1	480.0
Pb	<MRL	20.0	104.1	497.6	<MRL	21.3	103.9	502.3
U	NA	NA	NA	NA	10.3	34.6	116.2	601.0
Zn	28.5	51.1	126.9	523.2	30.4	48.7	147.4	486.0
				Standard deviations of average measured concentrations				
Ag	<MRL	0.3	1.1	3.5	<MRL	0.4	1.1	4.4
Al	1.8	2.5	3.1	6.7	.7	.8	2.1	5.2
Ba	.1	.1	.3	1.8	.4	1.0	1.8	4.4
Be	<MRL	.1	.3	2.1	<MRL	.3	2.5	5.3
Cd	<MRL	.4	1.0	2.8	<MRL	.5	.6	3.9
Co	<MRL	.5	.9	2.4	<MRL	.3	1.1	3.4
Cr	<MRL	.5	1.3	3.0	.1	.3	1.3	3.8
Cu	<MRL	.3	.3	2.2	.1	.6	1.4	6.2
Mn	.6	.5	2.2	4.3	3.3	4.5	5.4	6.6
Mo	2.2	1.6	1.6	2.2	.5	1.2	.7	5.6
Ni	5.5	1.7	1.4	3.3	.2	.8	1.1	4.4
Pb	<MRL	1.5	4.6	3.9	<MRL	.3	.6	5.1
U	NA	NA	NA	NA	.2	.7	2.7	5.5
Zn	1.1	1.4	1.3	5.4	.5	.5	2.3	6.5

Recovery of spike additions

	ICP-AES (percent)			ICP-MS (percent)		
	Low	Medium	High	Low	Medium	High
Ag	88.4	85.0	84.4	82.6	77.9	69.0
Al	96.4	96.7	100.5	75.3	117.1	97.7
Ba	99.0	95.9	98.0	101.4	104.0	104.4
Be	101.2	99.7	101.4	95.6	111.4	103.4
Cd	102.8	95.8	98.4	93.8	101.8	100.4
Co	101.6	100.9	100.9	91.8	108.8	85.7
Cr	114.8	98.8	99.7	88.8	108.1	99.2
Cu	116.4	102.9	99.7	84.8	102.1	91.4
Mn	122.6	99.3	96.0	73.9	144.6	97.6
Mo	101.4	93.4	100.3	100.4	112.5	109.6
Ni	121.2	103.0	99.2	84.8	81.7	93.9
Pb	100.2	104.1	99.5	103.0	103.2	100.3
U	NA	NA	NA	121.6	105.9	118.1
Zn	113.1	98.4	98.9	91.4	117.0	91.1

Table 13.--*Analyses of ground-water samples*

[ICP-AES, inductively coupled plasma - atomic emission spectrometry; ICP-MS, inductively coupled plasma - mass spectrometry; µg/L, microgram per liter; <MRL, less than method reporting limit; NA, not analyzed]

Metal	ICP-AES (µg/L)				ICP-MS (µg/L)			
	Unspiked	Low	Medium	High	Unspiked	Low	Medium	High
Average measured concentrations								
Ag	<MRL	18.1	66.5	219.7	<MRL	18.4	31.9	141.4
Al	2.7	19.6	95.5	492.6	<MRL	12.8	106.9	486.6
Ba	118.2	133.6	211.5	585.4	133.1	156.3	230.7	619.2
Be	<MRL	19.7	99.7	498.8	<MRL	20.7	114.5	520.0
Cd	<MRL	21.3	98.9	492.0	<MRL	20.1	100.6	495.4
Co	<MRL	20.1	99.0	491.4	<MRL	17.8	106.0	491.1
Cr	<MRL	22.2	100.4	491.3	<MRL	17.1	112.2	512.5
Cu	<MRL	18.1	96.6	477.4	1.3	19.6	100.3	458.1
Mn	263.8	279.9	345.2	737.2	218.0	225.4	407.5	785.5
Mo	<MRL	22.9	102.8	506.2	1.0	17.8	109.2	526.1
Ni	<MRL	19.8	99.6	484.6	12.1	32.7	109.5	481.9
Pb	<MRL	20.0	97.2	492.2	<MRL	19.0	103.1	510.8
U	NA	NA	NA	NA	<MRL	18.4	107.7	565.9
Zn	<MRL	23.6	97.3	494.4	4.2	24.3	112.7	493.4
Standard deviations of average measured concentrations								
Ag	<MRL	0.5	0.8	2.3	<MRL	0.2	3.2	12.8
Al	1.6	2.7	2.1	6.2	<MRL	.3	1.0	6.4
Ba	1.6	1.1	.5	3.4	2.8	1.3	2.6	4.9
Be	<MRL	.2	.5	4.8	<MRL	.3	1.1	11.8
Cd	<MRL	.8	2.0	8.8	<MRL	.3	1.2	3.8
Co	<MRL	.3	1.1	5.8	<MRL	.2	.9	2.3
Cr	<MRL	1.2	1.9	6.9	<MRL	.1	1.4	2.0
Cu	<MRL	.3	.6	2.4	.5	.3	1.6	4.7
Mn	12.7	7.0	4.4	9.7	5.4	5.7	6.1	5.4
Mo	<MRL	2.0	1.5	6.9	.1	.2	1.7	3.8
Ni	<MRL	1.1	2.7	7.8	2.5	1.2	1.7	2.6
Pb	<MRL	1.4	4.3	5.8	<MRL	.4	1.3	3.3
U	NA	NA	NA	NA	<MRL	.7	2.0	11.0
Zn	<MRL	1.9	2.5	9.5	.6	.8	2.9	7.0

Recovery of spike additions

	ICP-AES (percent)			ICP-MS (percent)		
	Low	Medium	High	Low	Medium	High
Ag	90.3	66.5	43.9	91.8	31.9	28.3
Al	84.7	92.8	98.0	64.0	106.9	97.3
Ba	77.0	93.3	93.4	116.0	97.6	97.2
Be	98.3	99.7	99.8	103.4	114.5	104.0
Cd	106.6	98.9	98.4	100.2	100.6	99.1
Co	100.7	99.0	98.3	85.6	105.4	98.1
Cr	110.8	100.4	98.3	80.7	111.2	102.3
Cu	90.4	96.6	95.5	91.2	99.0	91.4
Mn	80.5	81.4	94.7	37.0	189.4	113.5
Mo	114.3	102.8	101.2	83.6	108.2	105.0
Ni	98.9	99.6	96.9	102.8	97.4	94.0
Pb	100.0	97.2	98.4	95.0	103.1	102.2
U	NA	NA	NA	91.0	107.5	113.1
Zn	117.8	97.3	98.7	100.7	108.5	97.8

Table 14.--*Analyses of U.S. Geological Survey standard reference water sample T-113*

[MPV, most probable value; ICP-AES, inductively coupled plasma - atomic emission spectrometry; ICP-MS, inductively coupled plasma - mass spectrometry; <MRL, less than method reporting limit; NC, not certified; NA, not analyzed. All measurements in micrograms per liter]

Metal	MPV	Average measured concentration	
		ICP-AES	ICP-MS
Ag	5.0	5.9	5.1
Al	317.0	319.3	315.5
Ba	70.0	68.2	71.3
Be	10.0	10.5	11.5
Cd	4.2	4.4	4.5
Co	10.2	11.5	10.6
Cr	2.5	<MRL	1.6
Cu	47.0	46.4	46.4
Mn	65.0	65.0	63.0
Mo	34.0	36.4	35.0
Ni	2.1	<MRL	3.1
Pb	1.3	<MRL	<MRL
Sb	18.9	NA	19.6
U	NC	NA	<MRL
Zn	55.5	56.0	60.7

ICP-AES: 12 analyses on 4 days.
ICP-MS: 11 analyses on 4 days.

REFERENCES CITED

American Society for Testing and Materials, 1992, Water (sec. 11), *in* Annual book of ASTM standards: Philadelphia, Pa., v. 11.01, p. 45-47.

Boumans, P.W.J.M., ed., 1987, Inductively coupled plasma emission spectroscopy, pts. I and II: New York, John Wiley & Sons, 1070 p.

Date, A.R., and Gray, A.L., eds., 1989, Applications of inductively coupled plasma mass spectrometry: Glasgow, UK, Blackie & Son Limited, 273 p.

Fishman, M.J., and Friedman, L.C., eds., 1989, Methods for determination of inorganic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, 545 p.

Laing, G.A., Stapanian, M.A., Aleckson, K.A., Dobb, D.E., Rowan, J.T., and Garner, F.C., 1992, Final report of the multi-laboratory evaluation of method 6020 CLP-M inductively coupled plasma-mass spectrometry, version 8.1, *in* Statement of work for low concentration water inorganics analytical services superfund document No. ILC02.0: Las Vegas, Nev., Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, variable pagination.

Long, S.E., and Martin, T.D., 1992, Method 200.8--Determination of trace elements in waters and wastes by inductively coupled plasma - mass spectrometry, version 4.4, *in* Methods for the determination of metals in environmental samples: Cincinnati, Ohio, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, p. 95-137.

Montaser, Akbar, and Golightly, D.W., eds., 1987, Inductively coupled plasmas in analytical atomic spectrometry: New York, VCH, 660 p.

Tan, S.H., and Horlick, Gary, 1986, Background spectral features in inductively coupled plasma/mass spectrometry: Applied Spectroscopy, v. 40, no. 4., p. 445-460.

Thompson, Michael, and Walsh, J.N., 1983, A handbook of inductively coupled plasma spectrometry: Glasgow, UK, Blackie & Son Limited, 273 p.

Van Loon, J.C., 1985, Selected methods of trace metal analysis: New York, John Wiley & Sons, 357 p.

*U.S. GOVERNMENT PRINTING OFFICE: 1993-774-207/60024

4. Appendix B. Method of analysis for dissolved Calcium and Magnesium using USGS Method I-1472-87.

Extracted and reproduced without alteration from USGS Open File Report 93-125 Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments (Fishman, 1993). Pages 101 through 112.

METHODS OF ANALYSIS BY THE U.S. GEOLOGICAL SURVEY NATIONAL WATER QUALITY LABORATORY--DETERMINATION OF INORGANIC AND ORGANIC CONSTITUENTS IN WATER AND FLUVIAL SEDIMENTS

Excerpt: Method I-1472-87
Pages 101-112

By Marvin J. Fishman, Editor

U.S. GEOLOGICAL SURVEY

Open-File Report 93-125

Denver, Colorado
1993

Metals, atomic emission spectrometry, inductively coupled plasma (ICP)

Parameters and Codes:
Metals, dissolved, I-1472-87 (see below)

Parameter	Code	Parameter		Code
Barium (µg/L as Ba)	01005	Magnesium	(mg/L as Mg)	00925
Beryllium (µg/L as Be)	01010	Manganese	(µg/L as Mn)	01056
Cadmium (µg/L as Cd)	01025	Molybdenum	(µg/L as Mo)	01060
Calcium (mg/L as Ca)	00915	Nickel	(µg/L as Ni)	01065
Chromium (µg/L as Cr)	01030	Silica	(mg/L as SiO_2)	00955
Cobalt (µg/L as Co)	01035	Silver	(µg/L as Ag)	01075
Copper (µg/L as Cu)	01040	Sodium	(mg/L as Na)	00930
Iron (µg/L as Fe)	01046	Strontium	(µg/L as Sr)	01080
Lead (µg/L as Pb)	01049	Vanadium	(µg/L as V)	01085
Lithium (µg/L as Li)	01130	Zinc	(µg/L as Zn)	01090

1. Application

1.1 This method is used only for the determination of dissolved metals in water samples that have a measured specific conductance of less than 2,000 µS/cm at 25°C. The concentration limits are listed in table 8. Samples containing analyte concentrations greater than the maximum concentration limit can be analyzed for calcium, magnesium, silica, and sodium if the sample is diluted and if, after dilution, the specific conductance is less than 2,000 µS/cm. Trace metals also can be determined after appropriate dilution in samples that have a measured specific conductance greater than 2,000 µS/cm; however, detection levels and sensitivity will change proportionally. This modified method was implemented in the National Water Quality Laboratory in August 1987.

Table 8.--*Concentration limits of metals for inductively coupled plasma*

[µg/L, microgram per liter; mg/L, milligram per liter]

Metals	Minimum limit (µg/L, except where noted)	Maximum limit (µg/L, except where noted)	Wavelength
Barium	1	10,000	455.5
Beryllium	.5	10,000	313.0
Cadmium	1	10,000	214.4
Calcium (1)	.02 mg/L	25 mg/L	396.8
Calcium (2)	25 mg/L	400 mg/L	315.8
Chromium	5	10,000	267.7
Cobalt	3	10,000	238.8
Copper	10	10,000	324.7
Iron	3	10,000	259.9
Lead	10	10,000	220.3
Lithium	4	10,000	670.7
Magnesium (1)	.01 mg/L	5 mg/L	279.5
Magnesium (2)	5 mg/L	100 mg/L	382.9
Manganese	1	10,000	257.6
Molybdenum	10	10,000	203.8
Nickel	10	10,000	[1]231.6
Silica (SiO_2)	.01 mg/L	100 mg/L	288.1
Silver	1	10,000	328.1
Sodium (1)	.2 mg/L	50 mg/L	589.0
Sodium (2)	50 mg/L	400 mg/L	330.2
Strontium	.5	10,000	421.5
Vanadium	6	10,000	292.4
Zinc	3	10,000	[1]206.0

[1]Second order.

1.2 Analyze filtered and acidified samples. Water-suspended sediment or bottom material samples cannot be analyzed.

1.3 The inductively coupled plasma (ICP) methods approved for U.S. Geological Survey use are linked to a specific instrument and associated software. This does not imply endorsement of one product over another.

2. Summary of method

All metals are determined simultaneously on a single sample by a direct-reading emission spectrometric method using an inductively coupled argon plasma as an excitation source. Samples are pumped into a crossflow pneumatic nebulizer, and introduced into the plasma through a spray chamber and torch assembly. Each analysis is determined on the basis of the average of three replicate integrations, each of which is background corrected by a spectrum-shifting technique except for lithium (670.7 nm) and sodium (589.0 nm). A series of five mixed-element standards and a blank are used for calibration.

3. Interferences

3.1 Several interelement interference effects have been evaluated. Interelement correction factors are programmed into the Thermo Jarrell-Ash system software, and corrections are automatically applied internally to the data before they are printed.

3.2 Samples containing high dissolved solids show a variety of unidentified interference effects. Therefore, analyses need to be limited to samples with a specific conductance of 2,000 µS/cm or less.

4. Apparatus

Emission spectrometry system consisting of the following

4.1.1 *Autosampler.*

4.1.2 *Computer,* IBM PS/2 or equivalent.

Peristaltic pump.

4 .4 *Quartz torch assembly.*

4.1.5 *Spectrometer,* Jarrell-Ash Plasma Spectrometer, 0.75-m focal curve with spectrum shifter background correction, crossflow pneumatic nebulizer, and radio frequency generator. (See table 8 for element wavelengths.)

4.2 Refer to the Jarrell-Ash instruction manual (ICAP61E) for operating techniques. Operating conditions (see NOTE 1):

NOTE 1. Operating conditions are approximate and will vary from instrument to instrument.

Incident radio frequency power	1.25 kW
Reflected radio frequency power	<10 W
Vertical observation position	16 mm above load coil
Horizontal observation position	Center
Argon head pressure	40 lb/in^2
Sample argon pressure for crossflow nebulizer	17 lb/in^2
Sample argon flow rate for crossflow nebulizer	0.9 L/min
Coolant argon flow rate	18 L/min
Sample pumping rate for crossflow nebulizer	10 percent above aspiration
Refractor plate position	Optimized for Hg profile
Spectrum shifter	0.03 nm on high side of wavelength

5. Reagents

5.1 Use Ultrex grade acids or equivalent to prepare standards.

5.1.1 *Aqua regia*: Cautiously mix three parts concentrated HCl (sp gr 19) and one part concentrated HNO_3 (sp gr 1.41) just before use.

5.1.2 *Hydrochloric acid, 6M*: Add 500 mL concentrated HCl (sp gr 1.19) to 400 mL water, and dilute to 1 L.

5.2 *Water:* All references to water shall be understood to mean ASTM Type I reagent water (American Society for Testing and Materials, 1991).

5.3 Prepare standard stock solutions from Spex HiPure grade chemicals or equivalent. Dry all salts for 1 h at 105°C unless otherwise specified. Do not dry hydrated salts. Clean all metals thoroughly with the appropriate acid, and dry prior to weighing.

5.3.1 *Barium standard solution I,* 1.00 mL = 1,000 µg Ba: Dissolve 1.512 g $BaCl_2$ dried at 180°C for 1 h and cooled in a desiccator, in a 1,000-mL volumetric flask containing 100 mL water. In a well-ventilated hood, slowly add 100 mL concentrated HNO_3 (sp gr 1.41) while stirring. After the $BaCl_2$ has dissolved, dilute to 1,000 mL with water.

5.3.2 *Beryllium standard solution I,* 1.00 mL = 1,000 µg Be: Transfer 1.000 g Be flakes into a 1,000-mL volumetric flask with minimum water. Use a well-ventilated hood (Caution, see Note 2). Add 90 mL concentrated HNO_3 (sp gr 1.41) and 10 mL concentrated HCl (sp gr 1.19). Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

NOTE 2 Beryllium is extremely toxic. May be fatal if swallowed or inhaled.

5.3.3 *Cadmium standard solution I,* 1.00 mL = 1,000 µg Cd: Transfer 1.000 g Cd splatters into a 1,000-mL volumetric flask containing 100 mL water. Add 100 mL concentrated HNO_3 (sp gr 1.41). Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

5.3.4 *Calcium (1) standard solution I,* 1.00 mL = 1,000 µg Ca: Weigh 2.4973 g $CaCO_3$ dried at 180°C for 1 h and quickly transfer into a 1,000-mL volumetric flask containing 100 mL water. Dissolve cautiously with 10 mL concentrated HNO_3 (sp gr 1.41). Cool and dilute the solution to 1,000 mL with water.

5.3.5 *Calcium (2) standard solution I,* 1.00 mL = 5,000 µg Ca: Dry 12.4865 g $CaCO_3$ at 180°C for 1 h and cool in a desiccator. Weigh quickly, transfer into a 1,000-mL volumetric flask containing 100 mL water, and dissolve cautiously with 10 mL concentrated HNO_3 (sp gr 1.41). Dilute to 1,000 mL with water. After cooling, adjust volume to 1,000 mL.

5.3.6 *Chromium standard solution I,* 1.00 mL = 1,000 µg Cr: Transfer 1.000 g Cr metal into a 1,000-mL volumetric flask with minimum water. Add 50 mL concentrated HCl (sp gr 1.19) and stir. Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

5.3.7 *Cobalt standard solution I,* 1.00 mL = 1,000 µg Co: Transfer 1.000 g Co powder into a 1,000-mL volumetric flask with minimum water. Add 100 mL concentrated HNO_3 (sp gr 1.41). Place in an ultrasonic bath to increase rate of dissolution. Dilute to 1,000 mL with water.

5.3.8 *Copper standard solution I,* 1.00 mL = 1,000 µg Cu: Transfer 1.000 g Cu powder into a 1,000-mL volumetric flask with minimum water. Slowly add 100 mL concentrated HNO_3 (sp gr 1.41) while stirring. If necessary, heat to increase rate of dissolution. Dilute to 1,000 mL with water.

5.3.9 *Iron standard solution I,* 1.00 mL = 1,000 µg Fe: Transfer 1.000 g Fe powder into a 1,000-mL volumetric flask with minimum water. Place flask in hood and, while stirring, add 15 mL concentrated HCl (sp gr 1.19). Heat to increase rate of dissolution. After dissolution, slowly add 85 mL concentrated HNO_3 (sp gr 1.41). Cool and dilute to 1,000 mL with water.

5.3.10 *Lead standard solution I,* 1.00 mL = 1,000 µg Pb: Transfer 1.000 g Pb powder into a 1,000-mL Teflon volumetric flask with minimum water. Place flask into an ultrasonic bath in a well-ventilated hood and add 15 mL concentrated HCl (sp gr 1.19). Slowly add 85 mL concentrated HNO_3 (sp gr 1.41) while stirring. Dilute to 1,000 mL with water.

5.3.11 *Lithium standard solution I,* 1.00 mL = 1,000 µg Li: Dry $LiCO_3$ in oven at 180°C for 2 h, cool, and transfer 5.3235 g into a 1,000-mL volumetric flask containing 100 mL water. Slowly add 10 mL concentrated HNO_3 (sp gr 1.41). Dilute to 1,000 mL with water.

5.3.12 *Magnesium standard solution I,* 1.00 mL = 1,000 µg Mg: Transfer 1.000 g Mg granules into a 1,000-mL volumetric flask containing 100 mL water. In a well-ventilated hood, slowly add 100 mL concentrated HNO_3 (sp gr 1.41). Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

5.3.13 *Manganese standard solution I,* 1.00 mL = 1,000 µg Mn: Transfer 1.000 g Mn flakes into a 1,000-mL volumetric flask with minimum water. In a well-ventilated hood, slowly add 100 mL concentrated HNO_3 (sp gr 1.41). Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water.

5.3.14 *Molybdenum standard solution I,* 1.00 mL = 1,000 µg Mo: Transfer 1.000 g Mo powder into a 1,000-mL volumetric flask with minimum water. In a well-ventilated hood while stirring, slowly add 10 mL concentrated HNO_3 (sp gr 1.41). After fumes dissipate, add 90 mL concentrated HNO_3 (sp gr 1.41) and 20 mL water. Heat to increase rate of dissolution. If solution turns cloudy and a precipitate appears, turn heat up on hot plate and stir for an additional 15 to 30 minutes until precipitate disappears. Cool and dilute to 1,000 mL with water.

5.3.15 *Nickel standard solution I,* 1.00 mL = 1,000 µg Ni: Transfer 1.000 g Ni powder into a 1,000-mL volumetric flask containing 100 mL water. In a well-ventilated hood while stirring, slowly add 100 mL concentrated HNO_3 (sp gr 1.41). Heat and stir for 30 minutes or until dissolution is complete. Cool and dilute to 1,000 mL with water.

5.3.16 *Silica standard solution I,* 1.00 mL = 1,000 µg SiO_2: Transfer 1.7654 g $Na_2SiO_3 \cdot 5H_2O$ into a 500-mL polypropylene or Teflon volumetric flask and dissolve with small amount of water. Add 50 mL concentrated HNO_3 (sp gr 1.41) and dilute to 500 mL with water.

5.3.17 *Silver standard solution I,* 1.00 mL = 1,000 µg Ag: Transfer 1.000 g Ag powder into a 1,000-mL volumetric flask containing 100 mL water. In a well-ventilated hood while stirring, slowly add 100 mL concentrated HNO_3 (sp gr 1.41). Heat to increase rate of dissolution. Cool and dilute to 1,000 mL with water. Store in an opaque bottle.

5.3.18 *Sodium standard solution I,* 1.00 mL = 5,000 µg Na: Dissolve 12.7107 g NaCl in a 1,000-mL volumetric flask containing 100 mL water. Slowly add 100 mL concentrated HNO_3 (sp gr 1.41) while stirring. Cool and dilute to 1,000 mL with water.

5.3.19 *Strontium standard solution I,* 1.00 mL = 1,000 µg Sr: Dissolve 2.4153 g $Sr(NO_3)_2$ dried for 1 h at 180°C in a 1,000-mL volumetric flask containing 100 mL water. While stirring, add 100 mL concentrated HNO_3 (sp gr 1.41). Dilute to 1,000 mL with water.

5.3.20 *Vanadium standard solution I,* 1.00 mL = 1,000 µg V: Transfer 2.2963 g NH_4VO_3 into a 1,000-mL volumetric flask containing 20 mL concentrated HNO_3 (sp gr 1.41). Heat to increase rate of dissolution and continue to heat while stirring for 1 h. Cool and dilute to 1,000 mL with water.

5.3.21 *Zinc standard solution I,* 1.00 mL = 1,000 µg Zn: Transfer 1.0000 g Zn powder into a 1,000-mL volumetric flask containing 200 mL water. While stirring, slowly add 100 mL concentrated HNO_3 (sp gr 1.41). Dilute to 1,000 mL with water.

5.4 *Standard solution II,* 1.00 mL = 100 µg metal [except calcium (2) and sodium, 1.00 mL = 500 µg]: Dilute 100 mL of each individual metal standard solution I to 1,000 mL with water.

Mixed working standard solutions.

5.5.1 Prepare five mixed working standard solutions as follows: Pipet 25.0 mL of each appropriate standard solution II into a 250-mL volumetric flask. Dilute to 250 mL with water. Transfer to acid-rinsed PTFE bottle for storage. Fresh mixed standards need to be prepared weekly. Final concentration will be 1.00 mL = 10.0 µg for all metals with the exception of sodium and calcium (2), which will be 1.00 mL = 50.0 µg. Composition for mixed standard solutions will be as follows:

5.5.2 *Mixed standard solution I*--Iron, cadmium, chromium, lead, and zinc.

5.5.3 *Mixed standard solution II*--Barium, beryllium, copper, nickel, strontium, vanadium, and cobalt.

5.5.4 *Mixed standard solution III*--Molybdenum, lithium, silica, silver, and calcium (2).

5.5.5 *Mixed standard solution IV*--Calcium (1), manganese, and sodium.

5.5.6 *Standard solution V*--Magnesium.

5.5.7 *Rinse solution* is prepared by diluting 20 mL concentrated HNO_3 (sp gr 1.41) with 2,000 mL water.

5.6 *Check standard solution*: Pipet 25 mL of each standard solution II into a 500-mL volumetric flask. Dilute to 500 mL with water. Transfer to PTFE bottle for storage. Fresh check standard solution is to be prepared as needed. Final concentration will be 1.00 mL = 5.00 µg for all metals, with the exception of Na, which will be 1.00 mL = 28.83 µg (because of Na in the Si standard solution).

6. Procedure

6.1 Set up instrument with proper operating conditions (paragraph 4.2) and ignite plasma. Warm the instrument for 30 minutes prior to standardization.

6.2 Initiate the *operation* sequence in the software.

6.3 Aspirate a profiling solution or position the mercury pen lamp in front of the entrance slit. Initiate the *profile* computer command and profile the instrument by averaging the micrometer settings obtained at identical intensity positions on each side of the profiling spectral line. Position the micrometer to the average setting.

6.4 Standardize the data system by running a blank and the series of five mixed standard solutions. Pump rinse solution for 30 seconds between standards. Allow 30 seconds for equilibration each time a new solution is introduced.

6.5 Analyze check standard solution described in paragraph 5.6. Concentration values are not to deviate from the actual values by more than 2 percent. If values do deviate more than 2 percent, recalibrate.

6.6 Check standardization by running certified reference samples in natural matrix materials. The determined concentrations need to be within one standard deviation unit of the concentration given for each constituent in the reference material.

6.7 Analyze samples, allowing 30 seconds for equilibration. Pump rinse solution for 30 seconds between samples. Check calibration after analyzing 10 samples by rerunning a reference sample and the check standard solution. The results for the reference sample and check standard solution need to be within one standard deviation unit of the concentration given for each metal in the reference material and less than ± 2 percent for each element. If not, the data system has to be restandardized as described starting at paragraph 6.3.

6.8 Reprofile instrument (paragraph 6.3) as necessary. If profile position changes by more than 4 µm units, the instrument will need to be restandardized (starting with paragraph 6.2).

7. Calculations

7.1 All calculations are performed internally by the computer data system. The compound SiO_2 will be labeled Si. Headings are used to identify the results.

7.2 If dilutions were performed, multiply the results by the appropriate dilution factor using computer software.

8. Report

8.1 All results are printed out directly in the appropriate reporting units.

8.2 Report the dissolved constituent concentrations as follows:

8.2.1 Calcium (00915), magnesium (00925), silica (00955), and sodium (00930): less than 10 mg/L, one decimal; 10 mg/L and greater, two significant figures.

8.2.2 Beryllium (01010), cadmium (01025), manganese (01056), silver (01075), and strontium (01080): less than 10 µg/L, nearest microgram per liter; 10 µg/L and greater, two significant figures.

8.2.3 Barium (01005), chromium (01030), cobalt (01035), iron (01046), lithium (01130), vanadium (01085), and zinc (01090): less than 10 µg/L, nearest microgram per liter to the lower limit of detection as specified in table 8; 10 µg/L and greater, two significant figures.

8.2.4 Copper (01040), lead (01049), molybdenum (01060), and nickel (01065): less than 100 µg/L, nearest 10 µg/L; 100 µg/L and greater, two significant figures.

9. Precision

Within its designated range, single-operator precision of the method for each metal can be expressed as listed in table 9. A minimum of 10 replicates was analyzed to obtain each regression equation shown.

Table 9.--*Single-operator precision data for inductively coupled plasma*

[µg/L, microgram per liter; mg/L, milligram per liter]

Metal	Slope[1]	Intercept
Barium (µg/L)	0.0061	0.83
Beryllium (µg/L)	.0061	.06
Cadmium (µg/L)	.0203	.30
Calcium (mg/L)	.0044	.30
Cobalt (µg/L)	.0650	.40
Copper (µg/L)	.0039	1.32
Iron (µg/L)	.0071	.059
Lead (µg/L)	.1210	5.0
Lithium (µg/L)	.0240	.076
Magnesium (mg/L)	.0060	.018
Manganese (µg/L)	.0042	.30
Molybdenum (µg/L)	.1220	.18
Silica (mg/L)	.0040	.019
Sodium (mg/L)	.0077	.26
Strontium (µg/L)	.0089	.076
Vanadium (µg/L)	.1000	1.80
Zinc (µg/L)	.0059	1.24

[1]The standard deviation S_o is calculated by $S_o=mx+b$, where m is slope of line, x is concentration of constituent in units specified, and b is intercept.

9.3 Single-operator precision for dissolved chromium, nickel, and silver

for five samples, expressed as the standard deviation and percentage relative standard deviation, is as follows:

Chromium

Number of replicates	Mean (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
11	1.1	1.2	109
12	10.9	.76	7
18	16.6	1.0	6
11	19.7	1.4	7
12	24.6	.98	4

Nickel

Number of replicates	Mean (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
12	10.6	7.0	66
12	12.7	4.1	32
9	19.8	5.3	27
12	25.2	3.3	13
18	46.8	4.2	9

Silver

Number of replicates	Mean (µg/L)	Standard deviation (µg/L)	Relative standard deviation (percent)
9	0.37	0.38	102
12	2.48	.52	21
12	3.87	.66	17
12	4.95	1.5	30
18	10.4	1.8	17

Reference

American Society for Testing and Materials, 1991, Annual book of ASTM standards, Section 11, Water: Philadelphia, v. 11.01, p. 45-47.

5. Appendix C. Method of analysis for Ammonia Nitrogen using USGS Method I-6522-90.

Extracted and reproduced without alteration from USGS Open File Report 93-125 Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments (Fishman, 1993). Pages 125 through 132.

METHODS OF ANALYSIS BY THE U.S. GEOLOGICAL SURVEY NATIONAL WATER QUALITY LABORATORY--DETERMINATION OF INORGANIC AND ORGANIC CONSTITUENTS IN WATER AND FLUVIAL SEDIMENTS

Excerpt: Method I-6522-90 & I2522-90
Pages 125-132

By Marvin J. Fishman, Editor

U.S. GEOLOGICAL SURVEY

Open-File Report 93-125

Denver, Colorado
1993

Nitrogen, ammonia, colorimetry, salicylate-hypochlorite, automated-segmented flow

Parameters and Codes:
Nitrogen, ammonia, dissolved, I-2522-90 (mg/L as N): 00608
Nitrogen, ammonia, total-in-bottom-material, I-6522-90 (mg/L as N): 00611

1. Application

1.1 This method is used to analyze samples of surface, domestic, and industrial water, and brines containing from 0.01 to 1.5 mg/L of ammonia-nitrogen. Concentrations greater than 1.50 mg/L must be diluted. This modified method was implemented in the National Water Quality Laboratory in March 1988.

1.2 This method also is used to determine concentrations of ammonia-nitrogen in samples of bottom material containing at least 0.2 mg/kg NH_3-N. Prepared sample solutions containing more than 1.5 mg/L NH_3-N need to be diluted.

1.3 Sodium ion is a good replacement for ammonium ion in the slow-exchange positions of soil minerals (Jackson, 1958). Bottom material is treated with an acidified sodium chloride solution, and the resulting mixture is allowed to settle and then decanted to obtain a clear supernatant solution for analysis.

2. Summary of method

Ammonia reacts with salicylate and hypochlorite ions in the presence of ferricyanide ions to form the salicylic acid analog of indophenol blue (Reardon and others, 1966; Patton and Crouch, 1977; Harfmann and Crouch, 1989). The resulting color is directly proportional to the concentration of ammonia present.

3. Interferences

3.1 Sulfide interferes. Bromide and nitrite can interfere. Calcium and magnesium in highly alkaline waters (pH greater than 13.6) can exceed the ability of the tartrate to complex both ions.

3.2 The samples are easily contaminated by ammonia in the laboratory atmosphere; therefore, sample handling and analysis need to be performed where there is no possibility of ammonia contamination.

4. Apparatus

4.1 *Shaker,* wrist action.

4.2 *Alpkem rapid flow analyzer (RFA),* consisting of sampler, peristaltic pump, analytical cartridge, heating bath, colorimeter, data station, and printer.

4.3. With this equipment, the following operating conditions are satisfactory for the range from 0.01 to 1.50 mg/L of ammonia-nitrogen:

Flow cell .....15 mm
Wavelength.....660 nm
Sample time .....24 seconds
Wash time.....16 seconds
Sampling rate.....90 per hour
Heating bath (2 mL).....37°C
Pecking .....ON
Damp (RC).....1 second

5. Reagents

5.1 *Ammonia standard solution I,* 1.00 mL = 0.50 mg NH_3-N: Dissolve 1.9095 g NH_4Cl, dried overnight over sulfuric acid, in ammonia-free water and dilute to 1,000 mL. Refrigerate.

5.2 *Ammonia standard solution II,* 1.00 mL = 0.0015 mg NH_3-N: Dilute 3.0 mL ammonia standard solution I to 1,000 mL with ammonia-free water. Prepare fresh weekly and refrigerate.

5.2.1 *Ammonia working solutions,* bottom materials: Prepare an ammonia-free blank and 250 mL each of a series of ammonia working solutions by appropriate quantitative dilution of ammonia standard solution II or working solutions with acidified sodium chloride solution (paragraph 5.7), as follows:

Working solution No.	Solution added (mL)	Solution used	Ammonia concentration (mg/L)
1	250	Standard solution II	1.50
2	125	Standard solution II	.75
3	50	Standard solution II	.30
4	25	Standard solution II	.15
5	25	Working solution No. 2	.075
6	25	Working solution No. 4	.015

5.2.2 *Ammonia working solutions,* water: Prepare an ammonia-free blank and 250 mL each of a series of ammonia working solutions by dilution of ammonia standard solution II or working solutions with ammonia-free water as listed in the following table. If the samples to be analyzed are preserved, the ammonia working solutions need to contain an equivalent concentration of the same preservative.

Working solution No.	Solution added (mL)	Solution used	Ammonia concentration (mg/L)
1	250	Standard solution II	1.50
2	125	Standard solution II	.75
3	50	Standard solution II	.30
4	25	Standard solution II	.15
5	25	Working solution No. 2	.075
6	25	Working solution No. 4	.015

Prepare weekly and refrigerate.

5.3 *Buffer stock solution,* 71 g/L: Dissolve 134 g $Na_2HPO_4 \cdot 7H_2O$ in 800 mL of ammonia-free water. Add 100 mL 5*M* NaOH, dilute to 1 L with ammonia-free water, and mix thoroughly.

5.4 *Buffer working solution*: Add, while stirring, 250 mL stock potassium sodium tartrate solution to 200 mL buffer stock solution. Slowly, while stirring, add 120 mL 5*M* NaOH. Dilute to 1 L with ammonia-free water, add 1 mL of Brij-35 solution, and mix thoroughly.

5.5 *Hydrochloric acid,* concentrated (sp gr 1.19).

5.6 *Potassium sodium tartrate solution*, 149 g/L: Dissolve 200 g $NaKC_4H_4O_6 \cdot 4H_2O$ in about 600 mL ammonia-free water. Dilute to 1 L.

5.7 *Sodium chloride solution*, 100 g/L: Dissolve 100 g NaCl in 800 mL ammonia-free water, mix thoroughly, adjust the pH to 2.5 using concentrated HCl (sp gr 1.19), and dilute to 1 L.

5.8 *Sodium hydroxide solution*, 5*M*: CAUTION: Add, while cooling and stirring, 200 g NaOH to about 800 mL ammonia-free water. Cool and dilute to 1 L.

5.9 *Sodium hypochlorite solution*: Dilute 50 mL sodium hypochlorite solution (a commercial bleach solution containing 5.25-percent available chlorine is satisfactory) to 500 mL with ammonia-free water. Prepare fresh daily.

5.10 *Sodium salicylate–sodium nitroferricyanide solution*: Dissolve 150 g sodium salicylate and 0.30 g sodium nitroferricyanide [$Na_2Fe(CN)_5NO \cdot 2H_2O$] in about 600 mL ammonia-free water. Filter through Whatman 41 filter paper or equivalent, and dilute to 1 L. Add 1.0 mL Brij-35 solution and store in a light-resistant container.

5.11 *Sulfuric acid,* concentrated (sp gr .84).

5.12 *Sulfuric acid*, 2.5*M*: Cautiously add 138 mL concentrated H_2SO_4 (sp gr 1.84) to about 700 mL ammonia-free water. Cool and dilute to 1 L with ammonia-free water.

6. Procedure

6.1 Proceed to paragraph 6.2 for water. For bottom materials, begin with paragraph 6.1.1.

6.1.1 Weigh, to the nearest milligram, about 5 g of sample prepared as directed in method P-0520, and transfer to a 250-mL Erlenmeyer flask.

6.1.2 Add 50 mL of the acidic sodium chloride solution (paragraph 5.7). shake on the wrist-action shaker for 30 minutes, and allow to settle.

6.1.3 Transfer the supernatant solution to a 200-mL volumetric flask, taking care not to disturb the residue in the bottom of the Erlenmeyer flask.

6.1.4 Wash the sediment in the Erlenmeyer flask with 20 mL acidic sodium chloride solution (paragraph 5.7), let settle, and transfer the clear wash solution to the volumetric flask. Adjust to volume with acidic sodium chloride solution (paragraph 5.7). Proceed to paragraph 6.2.

6.2 Set up manifold (fig. 4). If the laboratory air is contaminated with ammonia, the air needs to be passed through a scrubber containing 2.5*M* H_2SO_4 before it enters the air manifold tube.

6.3 Allow the colorimeter, recorder, and heating bath to warm for at least 10 minutes or until the temperature of the heating bath reaches 37°C.

6.4 After all reagents are on line (NOTE 1), adjust the sample output of the photometer to 5 V. Then switch the photometer to "absorbance" mode and use the reference detector "fine gain" control to adjust the baseline absorbance to about 0.2 V. See operation manuals for complete details (Alpkem Corp., 1986). The solution remaining in the wash reservoir from previous determinations might be contaminated; therefore, this reservoir needs to be emptied and rinsed, and then refilled with fresh solution before proceeding.

NOTE 1. Place each reagent line except salicylate into its respective container; allow at least 5 minutes for the introduction of these reagents, and then place the salicylate line into its reagent container. If a precipitate forms after the addition of the salicylate, the pH of the solution stream is too low; check for contaminated reagents and remake them, and start again using the aforementioned procedure.

6.5 Place the most concentrated working solution in two cups before analysis. As the peaks appear on the recorder, adjust the STD CAL control until the peak obtains 95 percent of full scale.

6.6 When the system is clear of all working solutions, determine a dwell time using the most concentrated working solution.

6.7 Place a complete set of working solutions and a blank in the first positions of the sample tray beginning with the most concentrated working solution (NOTES 2 and 3). Place individual working solutions of differing concentrations in approximately every tenth position on the tray following the accepted protocol. Fill the remainder of each tray with unknown samples.

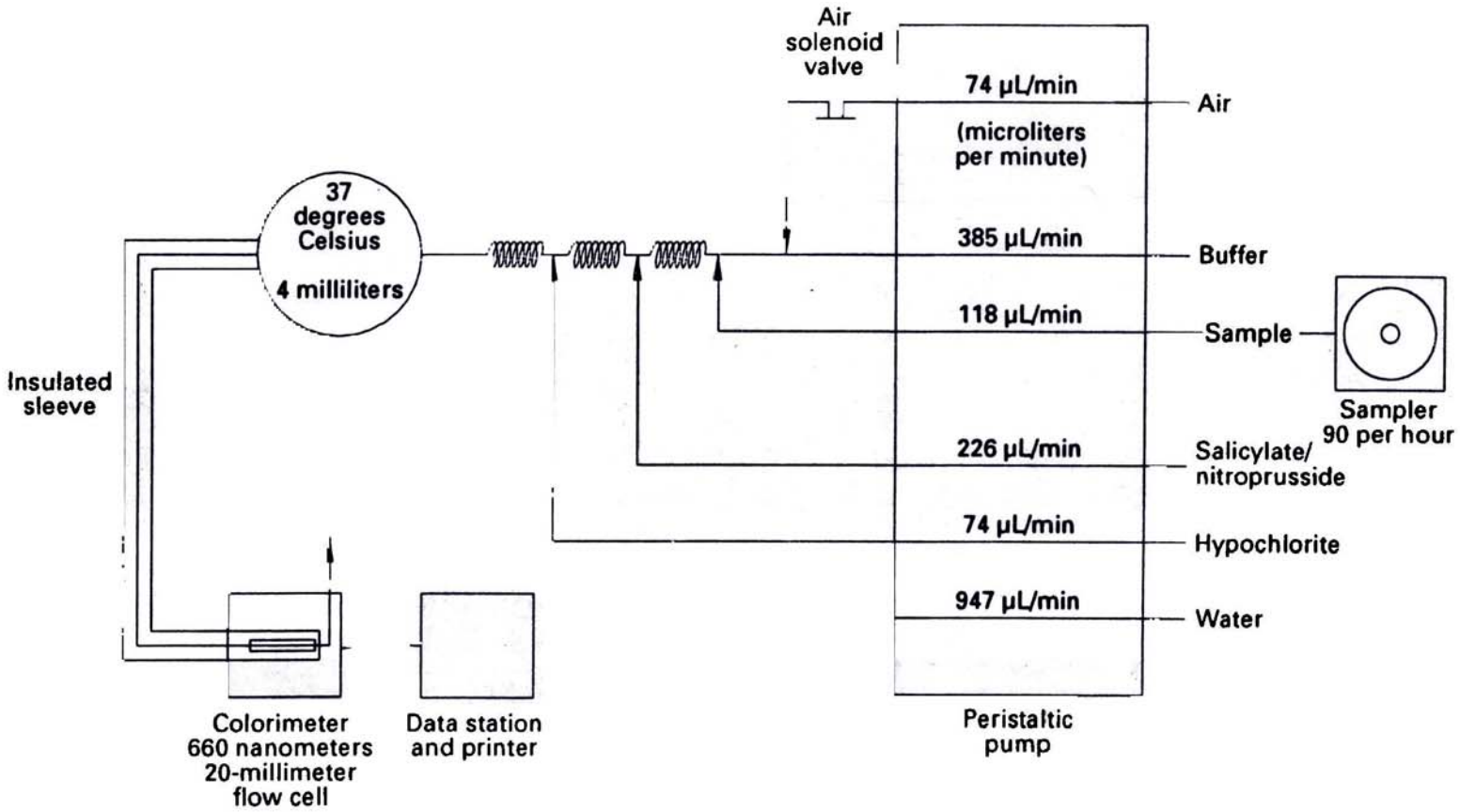


Figure 4.—Nitrogen, ammonia, salicylate-hypochlorite manifold.

NOTE 2. For analysis of bottom materials, use blank and working solutions as prepared in paragraph 5.2.1. For analysis of water, use blank and working solutions as prepared in paragraph 5.2.2.

NOTE 3. To avoid possible contamination of the sample cups, they need to remain sealed in their packages until just prior to use. Rinse each sample cup with sample prior to filling.

6.8 Begin analysis.

7. Calculations

7.1 Prepare an analytical curve by plotting the voltage of each working solution peak in relation to its respective ammonia-nitrogen concentration, or by using the RFA Softpak data reduction package. See operation manuals for complete details (Alpkem Corp., 1986).

7.2 Compute the concentration of dissolved ammonia-nitrogen in each sample by comparing its voltage to the analytical curve or by using the software. Any baseline drift needs to be accounted for when computing the voltage of a sample or standard peak; the RFA software automatically corrects for baseline drift.

7.3 Compute the concentration of ammonia-nitrogen in each sample of bottom material, as follows:

$$NH_3\text{-N (mg/kg)} = \frac{C_N \times 200 \text{ (NOTE 4)}}{\text{wt of sample, in g}}$$

where $C_N = NH_3$-N concentration in sample, in milligrams per liter.

NOTE 4. The factor 200 is used in converting to milligrams per kilogram.

8. Report

8.1 Report concentrations of ammonia-nitrogen, dissolved (00608), as follows: less than 1.0 mg/L, two decimals; 1.0 mg/L and greater, two significant figures.

8.2 Report ammonia-nitrogen, total-in-bottom-material (00611), as follows: less than 10 mg/kg, one decimal; 10 mg/kg and greater, two significant figures.

9. Precision

Single operator precision for ammonia-nitrogen, as determined for natural-water samples, expressed as standard deviation and percentage relative standard deviation, is as follows:

Number of determinations	Mean (mg/L)	Standard deviation (mg/L)	Relative standard deviation (percent)
180	1.23	0.011	0.89
193	.17	.001	.59
147	.28	.004	1.4
252	.71	.005	.70
240	.54	.006	1.1
209	.06	.001	1.7
240	.12	.002	1.7

References

Alpkem Corp., 1986, Rapid flow analyzer operator's manual, ALPKEM, methodology section.

Harfmann, R.G., and Crouch, S.R., 1989, Kinetic study of Berthelot reaction steps in the absence and presence of coupling reagents: Talanta, v. 36, p. 261-269.

Jackson, M.L., 1958, Soil chemical analysis: Englewood Cliffs, N.J., Prentice-Hall, p. 193.

Patton, C.J., and Crouch, S.R., 1977, Spectrophotometric and kinetics investigation of the Berthelot reaction for the determination of ammonia: Analytical Chemistry, v. 49, p. 464-469.

Reardon, J., Foreman, J.A., and Searcy, R.L., 1966, New reactants for the colorimetric determination of ammonia: Clinical Chimica Acta, v. 14, p. 403-405.

6. Appendix D. Method of analysis for Low Ionic Strength Nitrate Nitrogen using USGS Method I-2542-89.

Extracted and reproduced without alteration from USGS Open File Report 93-125 Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments (Fishman, 1993). Pages 137 through 142.

METHODS OF ANALYSIS BY THE U.S. GEOLOGICAL SURVEY NATIONAL WATER QUALITY LABORATORY--DETERMINATION OF INORGANIC AND ORGANIC CONSTITUENTS IN WATER AND FLUVIAL SEDIMENTS

Marvin J. Fishman, Editor

Excerpt: Method I-2542-89
Pages 137-142

GEOLOGICAL SURVEY

Open-File Report 93-125

Denver, Colorado
1993

Nitrogen, nitrite, low ionic-strength water, colorimetry, diazotization, automated-segmented flow

Parameter and Code:
Nitrogen, nitrite, dissolved, I-2542-89 (mg/L as N): 00613

1. Application

This method is used to analyze samples of precipitation or natural water containing from 0.001 to 0.20 mg/L of nitrite-nitrogen. Concentrations greater than 0.20 mg/L must be diluted. This method was implemented in the National Water Quality Laboratory in March 1986 and modified in May 1989.

2. Summary of method

Nitrite ion reacts with sulfanilamide under acidic conditions to form a diazo compound which then couples with N-1-naphthylethylenediamine dihydrochloride to form a red compound, the absorbance of which is measured colorimetrically (Shinn, 1941; Bendschneider and Robinson, 1952; Fox, 1979, 1985; Pai and others, 1990).

3. Interferences

Concentrations of potentially interfering substances generally are negligible. For specific details of inorganic and organic compounds that interfere with the reaction, see Norwitz and Keliher (1985, 1986) as well as more general information from the American Society for Testing and Materials (1991).

4. Apparatus

4.1 *Alpkem rapid flow analyzer (RFA)*, consisting of sampler, peristaltic pump, analytical cartridge, colorimeter, data station, and printer.

4.2 With this equipment, the following operating conditions are satisfactory for the range from 0.001 to 0.20 mg/L N:

Flow cell .....10 mm
Wavelength.....540 nm
Sampling rate.....64 per hour
Sample time.....24 seconds
Wash time.....32 seconds
Pecking .....OFF
Damp (RC).....1 second

5. Reagents

5.1 *Color reagent*: Add 200 mL of concentrated phosphoric acid (sp gr 1.69) and 20 g sulfanilamide to about 1,500 mL demineralized water. Dissolve completely (warm if necessary). Add 1.0 g N-1-naphthylethylenediamine dihydrochloride and dissolve completely. Dilute to 2 L with demineralized water. Add 1 mL Brij-35 solution. Store in an amber bottle and refrigerate. The reagent is stable for about 1 month.

5.2 *Nitrite-nitrogen standard solution I*, 1.00 mL = 0.100 mg NO_2-N: Dissolve 0.6076 g KNO_2, dried overnight over sulfuric acid, in demineralized water and dilute to 1,000 mL. This solution and the following nitrite standard solutions (paragraphs 5.3 and 5.4) are not stable indefinitely; their concentrations need to be checked frequently.

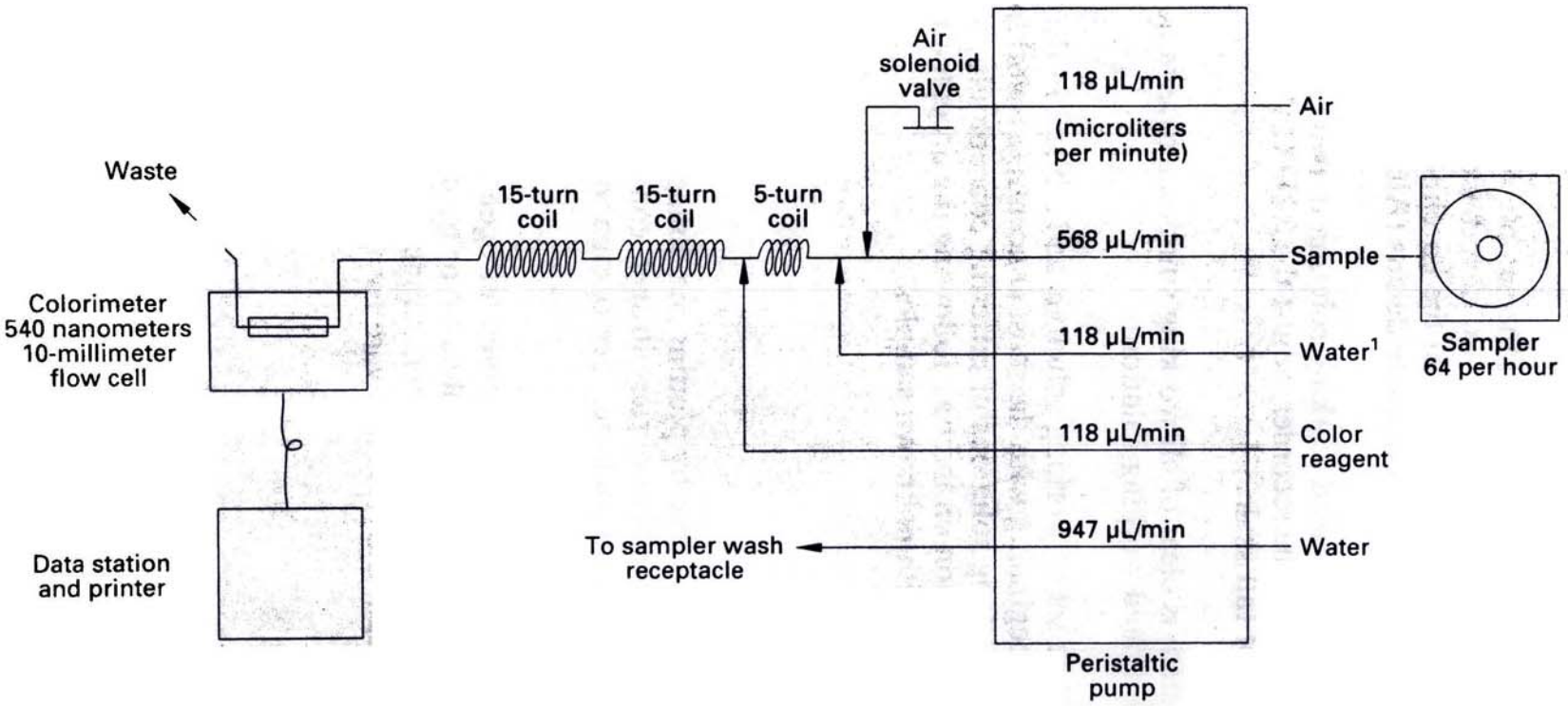
5.3 *Nitrite-nitrogen standard solution II*, 1.00 mL = 0.001 mg NO_2-N: Dilute 10.0 mL nitrite-nitrogen standard solution I to 1,000 mL with demineralized water.

5.4 *Nitrite-nitrogen working solutions*: Prepare a blank and 200 mL of a series of working solutions by appropriate dilution of nitrite-nitrogen standard solution II or working solution No. 3, as listed in the following table. If the samples to be analyzed are preserved, the nitrite-nitrogen working solutions need to contain an equivalent concentration of the same preservative.

Working solution No.	Solution added (mL)	Solution used	Nitrite-nitrogen concentration (mg/L)
1	40	Standard solution II	0.200
2	20	Standard solution II	.100
3	10	Standard solution II	.050
4	2	Standard solution II	.010
5	20	Working solution No. 3	.005
6	8	Working solution No. 3	.002
7	4	Working solution No. 3	.001

6. Procedure

6.1 Set up manifold (fig. 5).



[1] Contains 0.5-milliliter Brij solution per liter.

Figure 5.—Nitrogen, nitrite, low ionic-strength water, diazotization manifold.

Allow colorimeter recorder to warm for at least 10 minutes.

6.3 After all reagents are on line, adjust the sample output of the photometer to 5 V. Then switch the photometer to "absorbance" mode and use the reference detector "fine gain" control to adjust the baseline absorbance to about 0.2 V. See operation manuals for complete details (Alpkem Corp., 1986).

6.4 Place the most concentrated working solution in two cups before analysis. As the peaks appear on the recorder, adjust the STD CAL control until the peak obtains 95 percent of full scale.

6.5 When the system is clear of all working solutions, determine a dwell time using the most concentrated working solution.

6.6 Place a complete set of working solutions and a blank in the first positions of the sample tray beginning with the most concentrated working solution. Place individual working solutions of differing concentrations in approximately every eighth position on the tray following the accepted protocol. Fill the remainder of each tray with unknown samples.

Begin analysis.

7 Calculations

7.1 Prepare an analytical curve by plotting the voltage of each working solution peak in relation to its respective nitrite-nitrogen concentration, or by using the RFA Softpac data reduction package. See operation manuals for complete details (Alpkem Corp., 1986).

7.2 Compute the concentration of dissolved nitrogen in each sample either by comparing its voltage to the analytical curve or by using the software. Any baseline drift needs to be accounted for when computing the voltage of a sample or working solution peak; the RFA software automatically corrects for baseline drift.

8. Report

Report concentrations of nitrite-nitrogen, dissolved (00613), as follows: less than 0.10 mg/L, three decimals; 0.10 mg/L and greater, two significant figures.

9. Precision

Single operator precision for nitrite-nitrogen, as determined for natural-water samples, expressed as standard deviation and percentage relative standard deviation, is as follows:

Number of determinations	Mean (mg/L)	Standard deviation (mg/L)	Relative standard deviation (percent)
22	0.011	0.001	9.1
22	.063	.002	3.2
20	.144	.003	2.1

References

Alpkem Corp., 1986, Rapid flow analyzer operator's manual: ALPKEM, methodology section.

American Society for Testing and Materials, 1991, Annual book of ASTM standards, Section 11, Water: Philadelphia, v. 11.01, p. 508-515.

Bendschneider, Kenneth, and Robinson, R.J., 1952, A new spectrophotometric method for the determination of nitrite in sea water: Journal of Marine Research, v. 11, p. 87-96.

Fox, J.B., 1979, Kinetics and mechanism of the Greiss reaction: Analytical Chemistry, v. 51, p. 1493-1502.

. 1985, The determination of nitrite, a critical review: CRC Critical Reviews in Analytical Chemistry, v. 15, p. 283-313.

Norwitz, George, and Keliher, P.N., 1985, Study of interferences in the spectrophotometric determination of nitrite using composite diazotization - coupling reagents: Analyst, v. 110, p. 689-694.

. 1986, Study of organic interferences in the spectrophotometric determination of nitrite using composite diazotization - coupling reagents: Analyst, v. 111, p. 1033-1037.

Pai, S.C., Yang, C.C., and Riley, J.P., 1990, Formation kinetics of the pink azo dye in the determination of nitrite in natural waters: Analytica Chimica Acta, v. 232, p. 345-349.

Shinn, M.B., 1941, A colorimetric method for the determination of nitrite (ann. ed.): Industrial Engineering Chemistry, v. 13, p. 33-35.

7. Appendix E. Method of analysis for Low Ionic Strength Nitrite plus Nitrate Nitrogen using USGS Method I-2546-91.

Extracted and reproduced without alteration from USGS Open File Report 93-125 Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments (Fishman, 1993). Pages 149 through 156.

METHODS OF ANALYSIS BY THE U.S. GEOLOGICAL SURVEY NATIONAL WATER QUALITY LABORATORY--DETERMINATION OF INORGANIC AND ORGANIC CONSTITUENTS IN WATER AND FLUVIAL SEDIMENTS

Excerpt: Method I-2546-91
Pages 149-156

Marvin J. Fishman, Editor

GEOLOGICAL SURVEY

Open-File Report 93-125

Denver, Colorado
1993

Nitrogen, nitrite plus nitrate, low ionic-strength water, colorimetry, cadmium reduction-diazotization, automated-segmented flow

Parameter and Code:
Nitrogen, nitrite plus nitrate, dissolved, I-2546-91
(mg/L as N): 00631

1. Application

This method is used to analyze samples of precipitation or natural water, containing from 0.005 to 1.0 mg/L of nitrite- plus nitrate-nitrogen. Concentrations greater than 1.0 mg/L must be diluted. This method was implemented in the National Water Quality Laboratory in March 1986 and modified in May 1989.

2. Summary of method

Nitrate is reduced to nitrite by cadmium metal. The sample stream then is treated with sulfanilamide under acidic conditions to yield a diazo compound, which couples with N-1-naphthylethylenediamine dihydrochloride to form an azo dye, the absorbance of which is measured colorimetrically. The result is the sum of the nitrite originally present plus that formed by the reduction of the nitrate (Morris and Riley, 1963; Brewer and Riley, 1965; Wood and others, 1967; Strickland and Parsons, 1972; Nydahl, 1976; Sherwood and Johnson, 1981; Patton, 1982; U.S. Environmental Protection Agency, 1983).

3. Interferences

3.1 Concentrations of potentially interfering substances generally are negligible in unpolluted surface and ground water. For specific details of inorganic and organic compounds that interfere, see Norwitz and Keliher (1985, 1986) as well as more general information from the American Society for Testing and Materials (1991).

3.2 Sulfides, often present in anoxic water, rapidly deactivate cadmium reactors by forming an insoluble layer of cadmium sulfide on the active metal surface (Strickland and Parsons, 1972).

3.3 The buffer capacity of the imidazole solution and the approximate 10:1 volume ratio of buffer to sample eliminate the possibility of erroneous results for moderately acidic ($pH \geq 1$) samples.

4. Apparatus

4.1 *Alpkem rapid flow analyzer (RFA),* consisting of sampler, peristaltic pump, analytical cartridge (including copper-cadmium reduction column), colorimeter, data station, and printer.

4.2 With this equipment, the following operating conditions are satisfactory for the range from 0.005 to 1.0 mg/L ($NO_2 + NO_3$) as N:

Flow cell .....10 mm
Wavelength.....540 nm
Sample time.....24 seconds
Sampling rate.....64 per hour
Wash time.....32 seconds
Pecking .....OFF
Damp (RC).....1 second

5. Reagents

5.1 *Brij-35 solution,* 30-percent aqueous solution.

5.2 *Cadmium powder,* 100 mesh: Weigh 10 g cadmium powder into a 50-mL Griffin beaker and wash with 1*M* HCl. Stir vigorously to break clumps of cadmium formed by the addition of HCl. Rinse with copper sulfate solution (20 g/L). Wash thoroughly with imidazole solution to remove colloidal copper which is visible as a blue color in the wash solution. A minimum of five washings usually is required to eliminate perceptible blue color. Store in imidazole solution.

5.3 *Cadmium reduction column:* The reduction column is 4 cm long, 1/8-in. OD standard Teflon tubing (about 3.0-mm OD x 1.5-mm ID). Plugs to retain the cadmium in the column are cut from a sheet of hydrophilic, porous plastic with an average pore size of 40 µm. The cadmium column is plugged at the bottom with a plastic frit and attached to a syringe filled with imidazole solution. A custom-made funnel is attached to the top of the cadmium column, and the column and part of the funnel are filled with imidazole solution. The cadmium is added slowly to prevent air bubbles and pockets. Decant the solution in the funnel to facilitate the insertion of the top frit without introducing air to the column. The reduction efficiency of the column needs to be checked regularly by comparing the peak heights of nitrite and nitrate standards. Equal concentration standards should give equal heights. Replace the column if the efficiency falls to less than 90 percent.

5.4 *Color reagent*: Add 200 mL concentrated phosphoric acid (sp gr 1.69) and 20 g sulfanilamide to about 1,500 mL demineralized water. Dissolve completely (warm if necessary). Add 1.0 g N-1-naphthylethylenediamine dihydrochloride and dissolve completely. Dilute to 2 L with demineralized water. Add 1 mL Brij-35 solution. Store in an amber bottle and refrigerate. This reagent is stable for about 1 month.

5.5 *Copper sulfate solution*, 20 g/L: Dissolve 20 g $CuSO_4$ in demineralized water and dilute to 1 L.

5.6 *Hydrochloric acid, 1M*: Add 83 mL concentrated HCl (sp gr 1.19) to demineralized water and dilute to 1 L.

5.7 *Imidazole solution:* Dissolve 6.8 g of imidazole in demineralized water. Stir and dilute to about 950 mL. Adjust pH to 7.5 $\pm$ 0.1 with concentrated HCl (about 4 mL). Add 0.5 mL copper sulfate solution and dilute to 1 L. Add 1 mL Brij-35 solution.

5.8 *Nitrate-nitrogen standard solution I*, 1.00 mL = 0.50 mg NO_3-N: Dissolve 3.609 g KNO_3, dried overnight over concentrated H_2SO_4, in demineralized water and dilute to 1 L.

5.9 *Nitrate-nitrogen standard solution II*, 1.00 mL = 0.005 mg NO_3-N: Dilute 10.0 mL nitrate-nitrogen standard solution I to 1,000 mL with demineralized water.

5.10 *Nitrate-nitrogen working solutions:* Prepare a blank and 200 mL of a series of working solutions by appropriate dilution of nitrate-nitrogen standard solution II or working solution No. 3 as listed in the following table. If the samples to be analyzed are preserved, the nitrate-nitrogen working solutions need to contain an equivalent concentration of the same preservative.

Working solution No.	Solution added (mL)	Solution used	Nitrate-nitrogen concentration (mg/L)
1	40	Standard solution II	1.000
2	20	Standard solution II	.500
3	10	Standard solution II	.250
4	2	Standard solution II	.050
5	20	Working solution No. 3	.025
6	8	Working solution No. 3	.010
7	4	Working solution No. 3	.005

6. Procedure

Set up manifold (fig. 7).

Allow colorimeter recorder to warm for at least 10 minutes.

Allow the color reagent to come to room temperature.

6.4 Begin pumping reagents but do not connect the reduction column to the analytical cartridge until all air has been removed from the reagent and sample tubes (NOTE 1).

NOTE 1. It is important to avoid introduction of air bubbles into the reduction column because they adversely affect sample contact with the cadmium powder and decrease the reduction efficiency. Column needs to be replaced if air bubbles are introduced.

6.5 After all reagents are on line, adjust the sample output of the photometer to 5 V. Then switch the photometer to "absorbance" mode and use the reference detector "fine gain" control to adjust the baseline absorbance to about 0.2 V. See operation manuals for complete details (Alpkem Corp., 1986).

6.6 Place the most concentrated working solution in two cups before analysis. As the peaks appear on the recorder, adjust the STD CAL control until the peak obtains 95 percent of full scale.

6.7 When the system is clear of all working solutions, determine a dwell time using the most concentrated working solution.

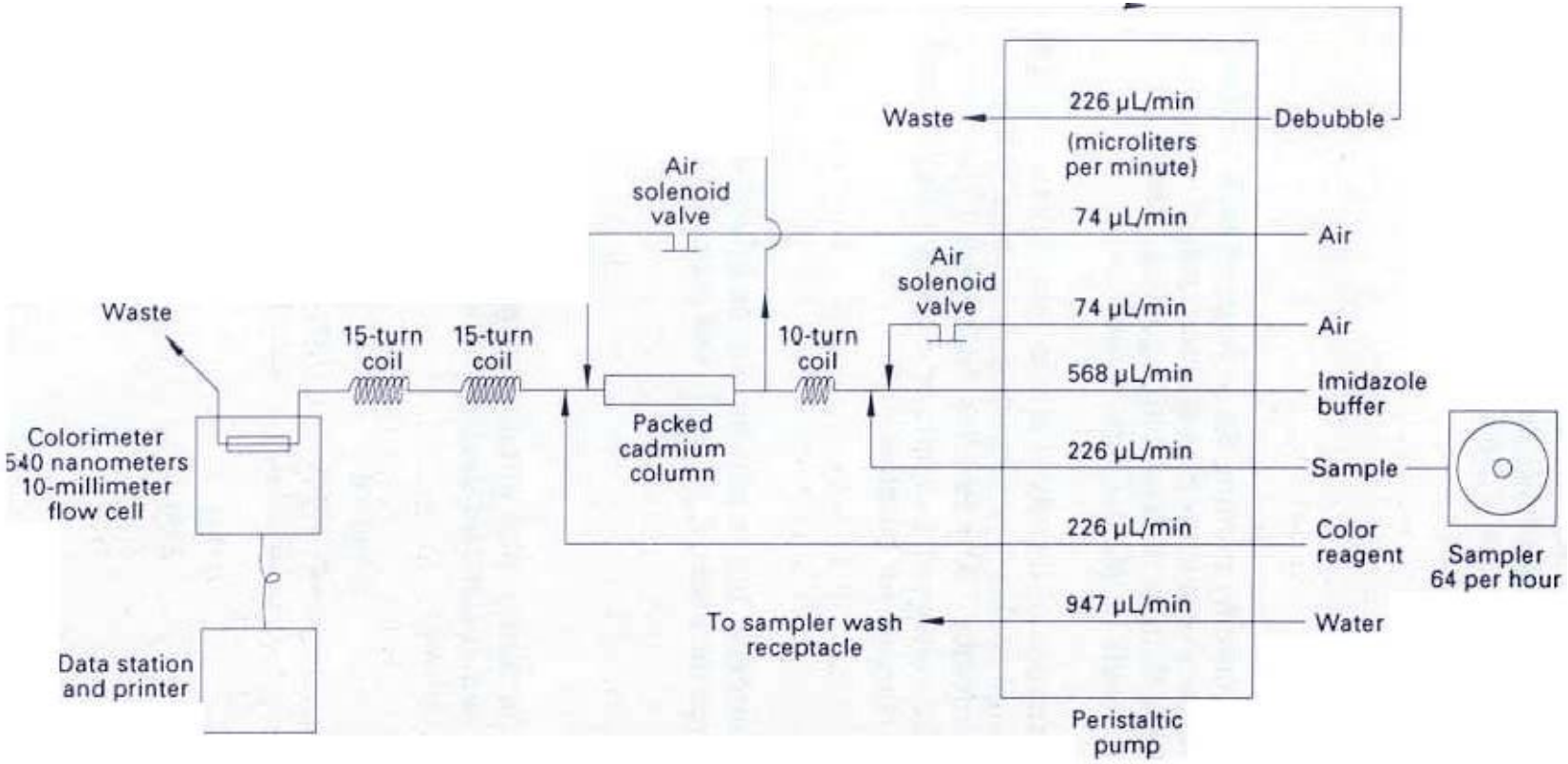


Figure 7.—Nitrogen, nitrite plus nitrate, low ionic-strength water, cadmium reduction-diazotization manifold.

6.8 Place a complete set of working solutions and a blank in the first positions of the sample tray beginning with the most concentrated working solution. Place individual working solutions of differing concentrations in about every eighth position on the tray following the accepted protocol. Fill the remainder of each tray with unknown samples.

6.9 Begin analysis.

7. Calculations

7.1 Prepare an analytical curve by plotting the voltage of each working solution peak in relation to its respective nitrite- plus nitrate-nitrogen concentration, or by using the RFA Softpac data reduction package. See operation manuals for complete details (Alpkem Corp., 1986).

7.2 Compute the concentration of dissolved nitrite- plus nitrate-nitrogen in milligrams per liter in each sample either by comparing its voltage to the analytical curve or by using the software. Any baseline drift needs to be accounted for when computing the voltage of a sample or working solution peak; the RFA software automatically corrects for baseline drift.

8. Report

Report concentrations of nitrogen, nitrite plus nitrate, dissolved (00631), as follows: 0.005 to 0.10 mg/L, three decimals; 0.10 mg/L and greater, two significant figures.

9. Precision

Single operator precision for nitrite- plus nitrate-nitrogen, as determined for natural-water samples, expressed as standard deviation and percentage relative standard deviation, is as follows:

Number of determinations	Mean (mg/L)	Standard deviation (mg/L)	Relative standard deviation (percent)
11	0.009	0.001	
21	.105	.006	5.7
22	.465	.015	3.2
22	.717	.011	1.5

References

Alpkem Corp., 1986, Rapid flow analyzer operator's manual: ALPKEM, methodology section.

American Society for Testing and Materials, 1991, Annual book of ASTM standards, Section 11, Water: Philadelphia, American Society for Testing and Materials, v. 11.01, p. 508-515.

Brewer, P.G., and Riley, J.P., 1965, The automatic determination of nitrate in sea water: Deep Sea Research, v. 12, p. 765-772.

Morris, A.W., and Riley, J.P., 1963, The determination of nitrate in sea water: Analytica Chimica Acta, v. 29, p. 272-279.

Norwitz, George, and Keliher, P.N., 1985, Study of interferences in the spectrophotometric determination of nitrite using composite diazotization-coupling reagents: Analyst, v. 110, p. 689-694.

. 1986, Study of organic interferences in the spectrophotometric determination of nitrite using composite diazotization-coupling reagents: Analyst, v. 111, p. 1033-1037.

Nydahl, Folke, 1976, On the optimum conditions for the reduction of nitrate to nitrite by cadmium: Talanta, v. 23, p. 349-357.

Patton, C.J., 1982, Novel cadmium reactors for determination of nitrate in water and seawater by segmented, continuous flow colorimetry, chap. 4 *of* Design, characterization, and applications of a miniature continuous flow analysis system: East Lansing, Michigan, Michigan State University, Ph.D. dissertation, p. 60-121.

Sherwood, G.A., and Johnson, D.C., 1981, Electrocatalysis of the reduction of nitrate ion at cadmium electrodes by electrodeposited copper: Analytica Chimica Acta, v. 129, p. 87-100.

Strickland, J.D.H., and Parsons, T.R., 1972, A manual of sea water analysis: Canada Fisheries Research Board Bulletin 167, p. 310.

U.S. Environmental Protection Agency, 1983, Methods for the chemical analysis of water and wastes: Washington, D.C., U.S. Government Printing Office, p. 353.2-1.

Wood, E.D., Armstrong, F.A.J., and Richards, F.A., 1967, Determination of nitrate in sea water by cadmium-copper reduction to nitrite: Journal of Marine Biology Association, U.K., v. 47, p. 23-31.

8. Appendix F. Method of analysis for dissolved Total Phosphorus using USGS Method I-4607-90.

Extracted and reproduced without alteration from USGS Open File Report 93-125 Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments (Fishman, 1993). Pages 175 through 181.

METHODS OF ANALYSIS BY THE U.S. GEOLOGICAL SURVEY NATIONAL WATER QUALITY LABORATORY--DETERMINATION OF INORGANIC AND ORGANIC CONSTITUENTS IN WATER AND FLUVIAL SEDIMENTS

Marvin J. Fishman, Editor

Excerpt: Method I-4607-90 & I-2607-90
Pages 175-181

GEOLOGICAL SURVEY

Open-File Report 93-125

Denver, Colorado
1993

Phosphorus, low ionic-strength water, colorimetry, phosphomolybdate, automated-segmented flow

Parameters and Codes:
Phosphorus, dissolved, I-2607-90 (mg/L as P): 00666
Phosphorus, total, I-4607-90 (mg/L as P): 00665

1. Application

This method is used to analyze samples of precipitation or natural water with a specific conductance of less than 100 µS/cm containing from 0.001 to 0.20 mg/L of phosphorus. Concentrations greater than 0.20 mg/L must be diluted. This method was implemented in the National Water Quality Laboratory in March 1990.

2. Summary of method

2.1 All forms of phosphorus, including organic phosphorus compounds, are converted to orthophosphate by an acid-persulfate digestion.

2.2 Orthophosphate ion reacts with ammonium molybdate in acidic solution to form phosphomolybdic acid, which upon reaction with ascorbic acid produces an intensely blue complex. Antimony potassium tartrate is added to increase the rate of reduction (Murphy and Riley, 1962; Gales and others, 1966; Pai and others, 1990).

3. Interferences

3.1 Barium, lead, and silver interfere by forming a phosphorus precipitate but the effect is negligible in natural water. The interference from silica, which forms a pale blue complex, is small and may be considered negligible. Nitrite interferes but can be oxidized to nitrate with hydrogen peroxide before analysis. Residual chloride needs to be removed by boiling the sample.

3.2 Arsenic as arsenate (AsO_4^{-3}) produces a similar color as phosphate (Murphy and Riley, 1962) and might cause a positive interference. Arsenic concentrations as much as 100 µg/L do not interfere.

4. Apparatus

4.1 *Autoclave.*

4.2 *Alpkem rapid flow analyzer (RFA),* consisting of sampler, analytical cartridge, peristaltic pump, heating bath, colorimeter, data station, and printer.

4.3 With this equipment, the following operating conditions are satisfactory for the range from 0.001 to 0.200 mg/L P:

Flow cell ..... 30 mm
Wavelength..... 880 nm
Sample time..... 38 seconds
Sampling rate..... 60 per hour
Wash time..... 22 seconds
Heating bath (2 mL)..... 37°C
Pecking ..... OFF
Damp (RC)..... 4 seconds

4.4 *Glass tubes with plastic caps,* disposable, 16 x 150 mm.

5. Reagents

5.1 *Ammonium molybdate solution,* 35.6 g/L: Dissolve 40 g ammonium molybdate $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ in 800 mL demineralized water and dilute to 1 L.

5.2 *Ascorbic acid solution,* 18 g/L: Dissolve 18 g ascorbic acid ($C_6H_8O_6$) in 800 mL demineralized water and dilute to 1 L. Keep in dark bottle and refrigerate. The solution is stable for 1 week.

5.3 *Antimony potassium tartrate solution*, 3 g/L: Dissolve 3.0 g antimony potassium tartrate $K(SbO)C_4H_4O_6{\cdot}1/2H_2O$ in 800 mL demineralized water and dilute to 1 L.

5.4 *Combined working reagent:* Combine reagents in following order (this reagent is stable for about 8 h):

Sulfuric acid, 2.45*M* ..... 100 mL
Ammonium molybdate solution..... 30 mL
Ascorbic acid solution..... 60 mL
Antimony potassium tartrate solution... 10 mL

5.5 *Sodium lauryl sulfate (SLS) solution*, 15 percent w/w: Dissolve 30 g SLS in 170 mL demineralized water. CAUTION: Solid sodium lauryl sulfate is a nasal irritant; work in a well-ventilated hood. Place flask in an ultrasonic bath to aid in dissolving SLS.

5.6 *Phosphorus standard solution I*, 1.00 mL = 0.100 mg P: Dissolve 0.4394 g KH_2PO_4, dried overnight over concentrated sulfuric acid (sp gr 1.84), in demineralized water and dilute to 1,000 mL.

5.7 *Phosphorus standard solution II*, 1.00 mL = 0.001 mg P: Dilute 10 mL phosphorus standard solution I to 1,000 mL with demineralized water.

5.8 *Phosphorus working solutions*: Prepare a blank and 1,000 mL each of a series of working solutions by appropriate dilution of phosphorus standard solutions I and II, as listed in the following table. If the samples to be analyzed are preserved, the phosphorus working solutions need to contain an equivalent concentration of the same preservative.

Working solution No.	Solution added (mL)	Solution used	Phosphorus concentration (mg/L)
1	2	Standard solution I	0.200
2	1	Standard solution I	.100
3	50	Standard solution II	.050
4	10	Standard solution II	.010
5	5	Standard solution II	.005
6	1	Standard solution II	.001

5.9 *Sulfuric acid solution*, 2.45*M:* Cautiously, add slowly, with constant stirring, 136 mL concentrated sulfuric acid (sp gr 1.84) to 800 mL demineralized water. Cool, and dilute to 1,000 mL with demineralized water.

5.10 *Phosphorus catalyst*: Add slowly 25 mL concentrated sulfuric acid and 4.0 g potassium persulfate ($K_2S_2O_8$) to 1,800 mL demineralized water and dilute to 2 L.

5.11 *Water diluent:* Add 5 mL sodium lauryl sulfate solution to 250 mL demineralized water.

6. Procedure

6.1 Pipet a volume of well-mixed sample containing less than 0.002 mg total phosphorus (10.0 mL maximum) into a disposable glass tube, and adjust volume to 10 mL.

6.2 Pipet 10 mL of blank and each of the working solutions into disposable glass tubes.

6.3 Add 4.0 mL phosphorus catalyst to each sample, working solution, and blank.

6.4 Place plastic caps on top of tubes. Autoclave for 30 minutes at 15 lb/in^2 pressure.

6.5 Set up manifold (fig. 9).

6.6 Allow colorimeter, recorder, and heating bath to warm up for at least 10 minutes or until the heating bath temperature reads 37°C.

6.7 After all reagents are on line, adjust the sample output of the photometer to 5 V. Then switch the photometer to "absorbance" mode and use the reference detector "fine gain" control to adjust the baseline absorbance to about 0.2 V. See operation manuals for complete details (Alpkem Corp., 1986).

6.8 Place the most concentrated working solution in two cups before analysis. As the peaks appear on the recorder, adjust the STD CAL control until the peak obtains 95 percent of full scale.

6.9 When the system has cleared of all working solutions, determine a dwell time using the most concentrated working solution.

6.10 Place a complete set of working solutions and a blank in the first positions of the first sample tray, beginning with the most concentrated working solution. Place individual working solutions of differing concentrations in approximately every eighth position on the tray following the accepted protocol. Fill the remainder of each tray with unknown samples.

6.1 Begin analysis.

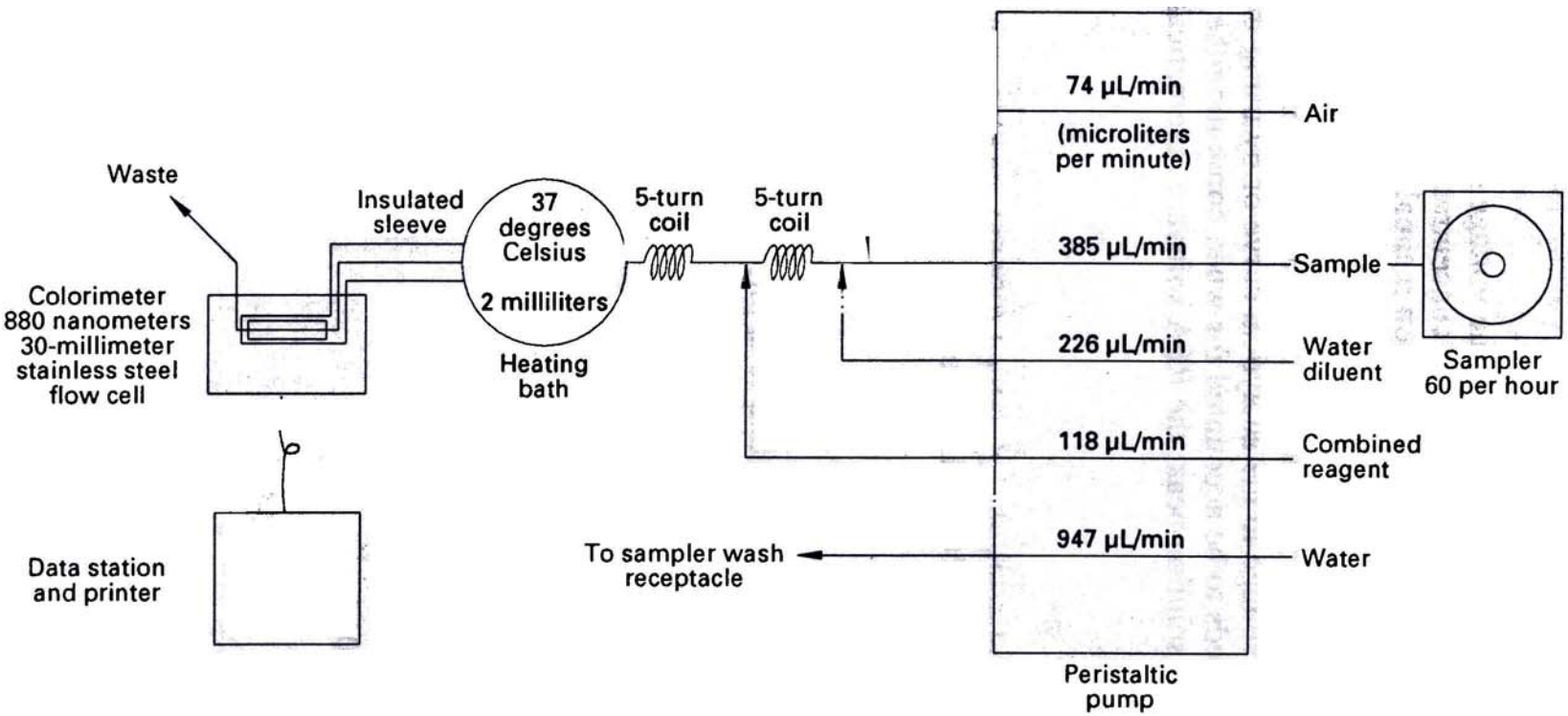


Figure 9.—Phosphorus, low ionic-strength water, phosphomolybdate manifold.

7. Calculations

7.1 Prepare an analytical curve by plotting the voltage of each working solution peak in relation to its respective phosphorus concentration, or by using the RFA Softpac data reduction package. See operation manual for complete details (Alpkem Corp., 1986).

7.2 Compute the concentration of dissolved or total phosphorus in each sample either by comparing its voltage to the analytical curve or by using the software. Any baseline drift needs to be accounted for when computing the voltage of a sample or working solution peak; the RFA software automatically corrects for baseline drift.

8. Report

Report concentrations of phosphorus, dissolved (00666), or phosphorus, total (00665), as follows: less than 0.10 mg/L, three decimals; 0.10 mg/L and greater, two significant figures.

9. Precision

Single operator precision for dissolved and total phosphorus, as determined for natural-water samples, expressed as standard deviation and percentage relative standard deviation, is as follows:

Mean (mg/L)	Standard deviation (mg/L)	Relative standard deviation (percent)
0.177	0.005	2.69
.176	.004	2.17
.092	.006	6.89
.038	.002	6.64
.004	.001	31.43

References

Alpkem Corp., 1986, Rapid flow analyzer operator's manual: ALPKEM, methodology section.

Gales, M.E., Jr., Julian, E.C., and Kroner, R.C., 1966, Method for quantitative determination of total phosphorus in water: American Water Works Association Journal, v. 58, p. 1363 -1368.

Murphy, J., and Riley, J.P., 1962, A modified single-solution method for the determination of phosphorus in natural water: Analytica Chimica Acta, v. 27, p. 31-36.

Pai, Su-Cheng, Yang, Chung-Cheng, and Riley, J.P., 1990, Effects of acidity and molybdate concentration on the kinetics of the formation of the phosphoantimonylmolybdenum blue complex: Analytica Chimica Acta, v. 229, p. 115-120.

9. Appendix G. Method of analysis for Laboratory pH using USGS Method I-1586-85 or I-2587-89.

Extracted and reproduced without alteration from USGS Techniques of Water-Resources Investigations, book 5, chap. A1, Methods for determination of inorganic substances in water and fluvial sediments (Fishman and Friedman, 1989). Pages 363 and 364.

Click here to return to USGS publications

USGS
science for a changing world

Techniques of Water-Resources Investigations
of the United States Geological Survey

Chapter A1

METHODS FOR DETERMINATION OF INORGANIC SUBSTANCES IN WATER AND FLUVIAL SEDIMENTS

By Marvin J. Fishman and Linda C. Friedman, Editors

First Edition 1970
Second Edition 1979
Third Edition 1989

Book 5
LABORATORY ANALYSIS

pH, electrometric, glass-electrode

Parameters and Codes:

pH lab, I-1586-85 (units): 00403
pH lab, automated, I-2587-85 (units): 00403

1. Application

This method may be used to determine the pH of any natural or treated water and any industrial or other wastewater.

2. Summary of method

2.1 See the introduction to electrometry in this chapter for the principles of pH-meter operation. See also Barnes (1964), Bates (1964), and Willard and others (1965).

2.2 This procedure may be automated with commercially available instrumentation.

3. Interferences

3.1 The determination is not affected by the presence of color or turbidity, or of organic or colloidal material. Oxidizing and reducing substances do not impair the accuracy of method.

3.2 The pH measurement is temperature dependent, and a significant error results if the temperatures of the buffers and samples differ appreciably. However, a variation of less than 5 °C has no significant effect except in the most exacting work.

3.3 For samples having abnormally high sodium levels, corrections may be necessary. This correction varies with the type of electrodes used; hence, see the manufacturer's instructions for the necessary computations.

4. Apparatus

4.1 *pH meter*, with glass and reference electrodes or combination pH electrode.

4.2 Several types of pH meters are available, including digital and expanded-scale models. Unless a different type is needed for special purposes, an ordinary laboratory, line-operated, pH meter—capable of a reproducibility of 0.05 of a pH unit—is adequate.

4.3 A new glass electrode or one that has dried completely may require several hours of soaking in water or buffer solution before it produces stable, reliable readings. The tip of the glass electrode must be kept immersed in water when not in use. Although the glass tip is reasonably durable, it can be damaged, and should never be cleaned or wiped with an abrasive or dirty tissue or cloth.

5. Reagents

Standard buffer solutions, pH 4.00, 7.00, and 9.00: These buffers should cover the range of pH of the samples to be measured. If samples of pH less than 4.00 or greater than 9.00 are to be analyzed, additional buffers will be required. Ready-made buffer reagents are satisfactory.

6. Procedure

6.1 After an appropriate warmup period, standardize the instrument with the buffer solutions, bracketing the pH values of the samples. Samples and buffers must be at the same temperature.

6.2 With a minimum of aeration or agitation, measure the pH of samples in accordance with the manufacturer's instructions.

7. Calculations

The pH is read directly from the meter.

8. Report

Report pH values (00403) to the nearest 0.1 pH unit.

9. Precision

9.1 Precision for pH for five of the 36 samples expressed in terms of standard deviation is as follows:

Number of laboratories	Mean (pH units)	Relative standard deviation (pH units)
27	6.21	0.26
59	7.14	.32
33	7.52	.15
48	8.00	.21
60	8.54	.15

9.2 Using automated instrumentation, analysis of two test samples by a single laboratory for 25 replicates of each resulted in mean values of 7.58 and 8.07 pH units and standard deviations of 0.05 and 0.03 pH units, respectively.

References

Barnes, Ivan, 1964, Field measurement of alkalinity and pH: U.S. Geological Survey Water- Supply Paper 1535-H, 17 p.

Bates, R. G., 1964, Determination of pH—theory and practice: New York, John Wiley and Sons, 435 p.

Willard, H. H., Merritt, L. L., Jr., and Dean, J. A., 1974, Instrumental methods of analysis (5th ed.): New York, D. Van Nostrand, 860 p.

10. Appendix H. Method of analysis for dissolved Selenium using USGS Method I-2477-92.

Extracted and reproduced without alteration from USGS Open-File Report 99-093, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory – Determination of Dissolved Arsenic, Boron, Lithium, Selenium, Strontium, Thallium, and Vanadium using Inductively Coupled Plasma-Mass Spectrometry (Garbarino, 1999).



Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Dissolved Arsenic, Boron, Lithium, Selenium, Strontium, Thallium, and Vanadium Using Inductively Coupled Plasma–Mass Spectrometry

Open-File Report 99–093

U.S. Department of the Interior
U.S. Geological Survey

Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Dissolved Arsenic, Boron, Lithium, Selenium, Strontium, Thallium, and Vanadium Using Inductively Coupled Plasma–Mass Spectrometry

By John R. Garbarino

U.S. Geological Survey
Open-File Report 99–093

Denver, Colorado
1999

U.S. DEPARTMENT OF THE INTERIOR
BRUCE BABBITT, Secretary

U.S. GEOLOGICAL SURVEY
Charles G. Groat, Director

The use of brand, firm, and trade names in this report is for identification purposes only and does not constitute endorsement by the U.S. Government.

For additional information write to:

U.S. Geological Survey
Chief, National Water Quality Laboratory
Box 25046, Mail Stop 407
Federal Center
Denver, CO 80225-0046

Copies of this report can be purchased from:

U.S. Geological Survey
Branch of Information Services
Box 25286
Federal Center
Denver, CO 80225-0286

CONTENTS

Abstract 1
Introduction 1
Analytical method 2
1. Application 2
2. Summary of method 3
3. Interferences 3
4. Apparatus, instrumentation, and operating conditions 15
5. Reagents and calibration standards 17
6. Sample preparation 18
7. Analytical procedure 18
8. Calculations 18
9. Reporting results 18
Discussion of results 19
Results for standard reference material 19
Spike recoveries in natural-water samples 20
Comparison of inductively coupled plasma–mass spectrometry to former methods of analysis 22
Conclusions 30
References cited 30

FIGURES

1–7. Graphs showing relations among selected cations and anions and new elements determined by inductively coupled plasma–mass spectrometry:

1. Arsenic 5
2. Boron 6
3. Lithium 7
4. Selenium 8
5. Strontium 9
6. Thallium 10
7. Vanadium 11

8. Accuracy of inductively coupled plasma–mass spectrometric arsenic determinations in the presence of bromide using different correction equations 14
9. Relation of apparent chromium-52 signal from $^{40}Ar^{12}C^{+}$ as a function of increasing concentrations of carbon 16
10. The accuracy of inductively coupled plasma–mass spectrometric chromium-52 determinations in the presence of carbon 16

FIGURES—Continued

11–16. Graphs showing statistical results for filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and former methods of analysis:
- 11. Arsenic results for 59 samples 24
- 12. Boron results for 62 samples 25
- 13. Lithium results for 63 samples 26
- 14. Selenium results for 64 samples 27
- 15. Strontium results for 63 samples 28
- 16. Vanadium results for 17 samples 29

TABLES

1. Inorganic constituents and codes 3
2. Former methods and ICP–MS method detection limits and calibration limits for new elements determined in filtered, acidified natural water 4
3. Concomitant concentrations that result in more than a 20-percent ionization suppression on new elements determined by ICP–MS 13
4. Statistical analysis of long-term ICP–MS results for U.S. Geological Survey Standard Reference Water Sample T145 20
5. Short-term analytical variability as a function of elemental concentration for ICP–MS 21
6. Average percent spike recoveries in reagent-water, surface-water, and ground-water matrices by inductively coupled plasma–mass spectrometry 21
7. Chemical characteristics of natural-water samples used to evaluate ICP–MS 22
8. Statistical analysis summary of inductively coupled plasma–mass spectrometry and former methods of analysis 23

CONVERSION FACTORS, ABBREVIATED WATER-QUALITY UNITS, OTHER ABBREVIATIONS, AND DEFINITIONS

Multiply	By	To obtain
gram (g)	3.53×10^{-2}	ounce
liter (L)	2.64×10^{-1}	gallon
microgram (µg)	3.53×10^{-8}	ounce
micrometer (µm)	3.94×10^{-5}	inch
milligram (mg)	3.53×10^{-5}	ounce
milliliter (mL)	2.64×10^{-4}	gallon

Degree Celsius (°C) may be converted to degree Fahrenheit (°F) by using the following equation:

$$°F = 9/5\ (°C) + 32.$$

Abbreviated water-quality units used in this report are as follows:

mg/L	milligram per liter
µg/L	microgram per liter
µS/cm	microsiemens per centimeter at 25°C

Other abbreviations also used in this report:

amu	atomic mass unit
ASTM	American Society for Testing and Materials
CC	catalyzed colorimetry
DCP–AES	direct current plasma–atomic emission spectrometry
F–AAS	flame–atomic absorption spectrophotometry
FEP	fluorinated ethylene propylene (Teflon)
GF–AAS	graphite furnace–atomic absorption spectrophotometry
HG–AAS	hydride generation–atomic absorption spectrophotometry
ICP–MS	inductively coupled plasma–mass spectrometry
ICP–AES	inductively coupled plasma–atomic emission spectrometry, also known as inductively coupled plasma–optical emission spectrometry (ICP–OES)
MDL(s)	method detection limit(s)
MRL(s)	minimum reporting level(s)
MPV(s)	most probable value(s)
NIST	National Institute of Standards and Technology
NWQL	National Water Quality Laboratory
SRWS(s)	U.S. Geological Survey Standard Reference Water Sample(s)
SOP	standard operating procedure
USGS	U.S. Geological Survey
>	greater than
<	less than
≤	less than or equal to
±	plus or minus

Definitions:

MDL — The method detection limit (MDL) is defined as the minimum concentration of an element that can be measured and reported with 99-percent confidence that the concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the element of interest (U.S. Environmental Protection Agency, 1997).

MPV — The most probable value (MPV) is equal to the median value for numerous interlaboratory analyses from multiple analytical methods.

Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Dissolved Arsenic, Boron, Lithium, Selenium, Strontium, Thallium, and Vanadium Using Inductively Coupled Plasma–Mass Spectrometry

By John R. Garbarino

ABSTRACT

The inductively coupled plasma–mass spectrometric (ICP–MS) methods have been expanded to include the determination of dissolved arsenic, boron, lithium, selenium, strontium, thallium, and vanadium in filtered, acidified natural water. Method detection limits for these elements are now 10 to 200 times lower than by former U.S. Geological Survey (USGS) methods, thus providing lower variability at ambient concentrations. The bias and variability of the method was determined by using results from spike recoveries, standard reference materials, and validation samples. Spike recoveries at 5 to 10 times the method detection limit and 75 micrograms per liter in reagent-water, surface-water, and ground-water matrices averaged 93 percent for seven replicates, although selected elemental recoveries in a ground-water matrix with an extremely high iron sulfate concentration were negatively biased by 30 percent. Results for standard reference materials were within 1 standard deviation of the most probable value. Statistical analysis of the results from about 60 filtered, acidified natural-water samples indicated that there was no significant difference between ICP–MS and former USGS official methods of analysis.

INTRODUCTION

The U.S. Geological Survey (USGS) National Water Quality Laboratory (NWQL) offers several methods for the determination of dissolved arsenic, boron, lithium, selenium, strontium, thallium, and vanadium in filtered, acidified water samples. Former USGS methods use single-element quantification, such as flame– (F–AAS), graphite furnace– (GF–AAS), and hydride generation–atomic absorption spectrophotometry (HG–AAS), direct current plasma–atomic emission spectrometry (DCP–AES), catalyzed colorimetry (CC), or the simultaneous multielement technique of inductively coupled plasma–atomic emission spectrometry (ICP–AES). This report provides data that validates the addition of the aforementioned elements to the existing inductively coupled plasma–mass spectrometric (ICP–MS) method.

Elements that are being added to the existing multielement ICP–MS method and their corresponding former USGS methods of analysis are listed in the following table: Dissolved arsenic, boron, lithium, strontium, thallium, and vanadium are validated for use in ICP–MS method I-2477-92 (see Faires, 1993; Garbarino and Taylor, 1994).

Element	CC	F–AAS	GF–AAS	HG–AAS	DCP–AES	ICP–AES
Arsenic			✓	✓		
Boron					✓	✓
Lithium		✓				✓
Selenium			✓	✓		
Strontium		✓				✓
Thallium			✓			
Vanadium	✓					✓

ICP–MS is compared to one former method from the list in the preceding table. In all comparisons, the most current (as of January 1998) former method is used. Dissolved arsenic and selenium ICP–MS results are compared to HG–AAS. The HG–AAS methods require that the sample be digested to oxidize organocompounds. The former HG–AAS method for dissolved arsenic is based on an online sulfuric acid/potassium persulfate digestion. The former HG–AAS method for dissolved selenium is based on an offline hydrochloric acid/potassium persulfate digestion. No such digestions are necessary to determine dissolved elements by the ICP–MS method because the high-temperature plasma dissociates organometallic species. The ICP–MS method for dissolved boron, lithium, strontium, and vanadium is compared to ICP–AES. The ICP–MS thallium method was compared to GF–AAS.

The objectives of this report are as follows:

- To determine whether ICP–MS results are biased with respect to former USGS methods of analysis for determination of the new elements in filtered natural water. Multiple strategies are used in the statistical analysis of the experimental data to provide a practical estimate of the expected accuracy.

- To compare the variability of the ICP–MS method to former USGS methods of analysis.

- To estimate potential effects of using results from the ICP–MS method on long-term water-quality studies.

The revised methods were developed by the USGS for use at the NWQL. These methods supplement other official USGS inorganic methods (Fishman, 1993; Fishman and Friedman, 1989). The new elements will be available in the ICP–MS schedules.

ANALYTICAL METHOD

1. Application

The determination of dissolved arsenic, boron, lithium, selenium, strontium, thallium, and vanadium in filtered, acidified natural water has been added to the ICP–MS method (I-2477-92). Table 1 lists the new lab code, parameter code, method code, and reporting unit for every element. A report by Garbarino and Taylor (1994) describes the determination of these elements in filtered, acidified natural water; therefore, only supplementary information and validation data are provided here. Filtered, acidified natural water is processed as described in Horowitz and others (1994).

Table 1. Inorganic constituents and parameter codes

Metals, Dissolved, Method I-2477-92

Element	Lab code	Parameter and method codes
Arsenic, µg/L	2503	01000D
Boron, µg/L	2504	01020G
Lithium, µg/L	2505	01130C
Selenium, µg/L	2506	01145C
Strontium, µg/L	2507	01080I
Thallium, µg/L	2508	01057I
Vanadium, µg/L	2509	01085E

The short-term method detection limits (MDLs) and analytical concentration ranges are listed in table 2. Even though the upper calibration standard is listed as 200 µg/L or less, the elemental linear dynamic range is extended to at least 1 mg/L by calibrating both the pulse and analog measurement modes. Short-term MDLs were calculated by using the U.S. Environmental Protection Agency's (1997) definition and represent pooled averages on the basis of four MDLs determined on different days over several weeks. The MDLs have not been established for most former methods, therefore, minimum-reporting levels (MRLs) are listed that are probably within a factor of 5 of the MDL.

2. Summary of Method

The ICP–MS methods have been described previously in Faires (1993) and Garbarino and Taylor (1994). The following sections only provide additional information specific to the elements that are being added to the method.

3. Interferences

Several types of physical and spectral interference are recognized and documented for ICP–MS techniques (see Horlick and Shao, 1992; Garbarino and Taylor, 1994). Physical interferences are associated primarily with sample introduction and are minimized by using the internal standardization technique. Isotopes measured in this procedure have been selected specifically to minimize spectral interferences from isobaric, doubly charged, and molecular ions. Multiple isotopes can be measured for selected elements that have potential isobaric or molecular ion interference. The analyst must be aware of these interferences because they might be present with certain types of sample matrices.

3.1 *Physical interferences.* The use of the ratio of elemental ion intensity to the internal standard element ion intensity for calibration reduces the effects of sample transport, instrumental drift, and matrix-induced fluctuations in plasma characteristics. Accurate results depend on having a constant internal standard ion intensity throughout the analysis of all standards and samples. Stable internal standard ion intensity requires that any interference associated with the internal standard element can be corrected and that the internal standard element is not naturally present in the samples being analyzed. Groups of elements with similar response characteristics or mass-to-charge ratios are often referenced to an internal standard element with similar characteristics.

Three internal standard elements are routinely used to cover the mass range from 6 to 240—for example, $^{72}Ge^{+}$, $^{115}In^{+}$, and $^{209}Bi^{+}$ (Garbarino and Taylor, 1994). Alternative isotopes may be substituted after ensuring that there are no spectral interferences associated with the new selections.

Memory effects related to sample transport are negligible for most elements that are normally present in natural water. Carryover from samples that have arsenic,

Table 2. Former methods and ICP–MS method detection limits and calibration limits for new elements determined in filtered, acidified natural water

[All concentrations are in micrograms per liter; MRL, minimum reporting level; MDL, method detection limit; CC, catalyzed colorimetry; DCP–AES, direct current plasma–atomic emission spectrometry; F–AAS, flame–atomic absorption spectrophotometry; GF–AAS, stabilized temperature graphite furnace–atomic absorption spectrophotometry; HG–AAS, hydride generation–atomic absorption spectrophotometry; ICP–AES, inductively coupled plasma–atomic emission spectrometry; ICP–MS, inductively coupled plasma–mass spectrometry]

Element	Former methods			ICP–MS	
	Technique	MRL	Upper concentration limit (without dilution)	Short-term MDL	Upper calibration standard[1]
Arsenic	GF–AAS	0.9	50	0.07	200
	HG–AAS	1	20		
Boron	ICP–AES	16	10,000	[2] 0.5	100
Lithium	F–AAS	10	1,000	0.03	200
	ICP–AES	4	100,000		
Selenium	GF–AAS	1	50	0.1	200
	HG–AAS	1	20		
Strontium	F–AAS	10	5,000	0.008	200
	ICP–AES	1	10,000		
Thallium	GF–AAS	1	9	0.005	200
Vanadium	CC	1	40	0.08	200
	ICP–AES	10	10,000		

[1]Upper concentration limit extends to greater than 1 milligram per liter without dilution by calibrating the analog stage of the detector.

[2]Method detection limit for boron is limited by reagent blank concentration.

boron, lithium, selenium, and strontium concentrations less than or equal to 200 µg/L is negligible when using the sample introduction described in Garbarino and Struzeski (1998). However, thallium and vanadium did not recover to reagent-blank intensity levels within the rinse period; thallium and vanadium intensities were 10 and 2 times greater than reagent-blank levels, respectively. Consequently, the analyst must review all analytical results to ensure that errors from carryover are minimized.

Sample matrix composition could also affect the bias and variability of ICP–MS determinations. The use of internal standardization compensates for most matrix effects, however, some matrix interferences remain problematic. Matrix composition can suppress the ionization efficiency of the plasma and result in negatively biased elemental concentrations. Aluminum sulfate, calcium sulfate, iron sulfate, manganese sulfate, sodium sulfate, and sodium chloride were evaluated for their matrix interference potential. A solution containing about 25 µg/L of arsenic, boron, lithium, selenium, strontium, thallium, and vanadium was prepared in increasing concentrations of each compound.

The effects of increasing concomitant concentration are shown in figures 1 through 7. For example, the graphs in figure 1 show the effects of increasing concentrations of cations (aluminum, calcium, iron, manganese, and sodium) and anions (sulfate

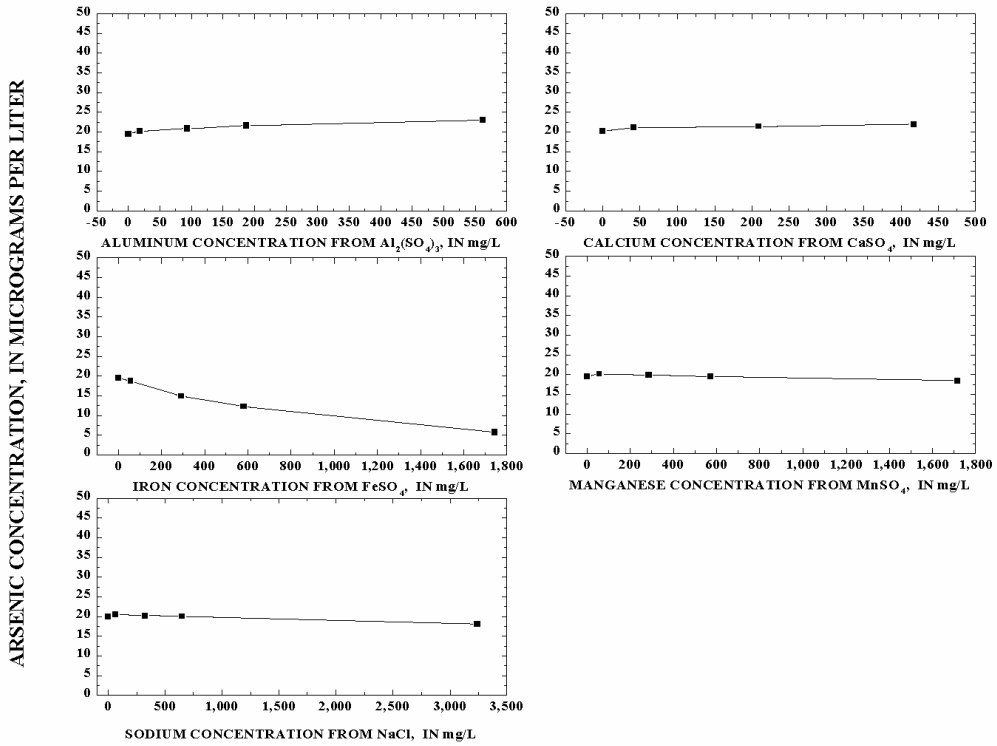


Figure 1. Relations among selected cations and anions and **arsenic** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

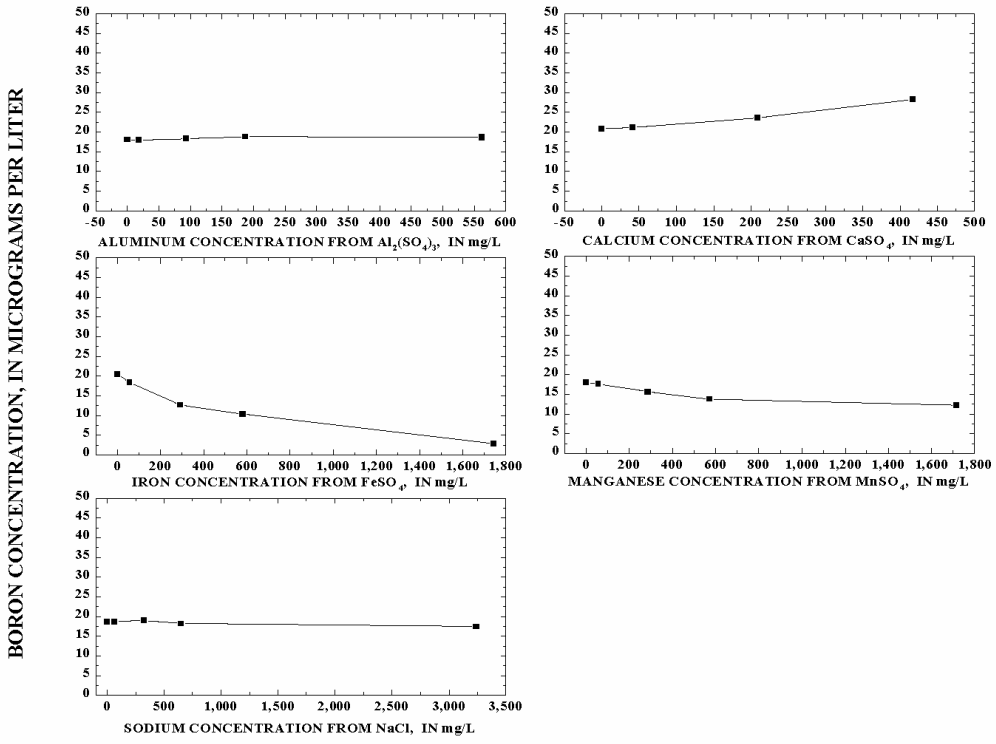


Figure 2. Relations among selected cations and anions and **boron** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

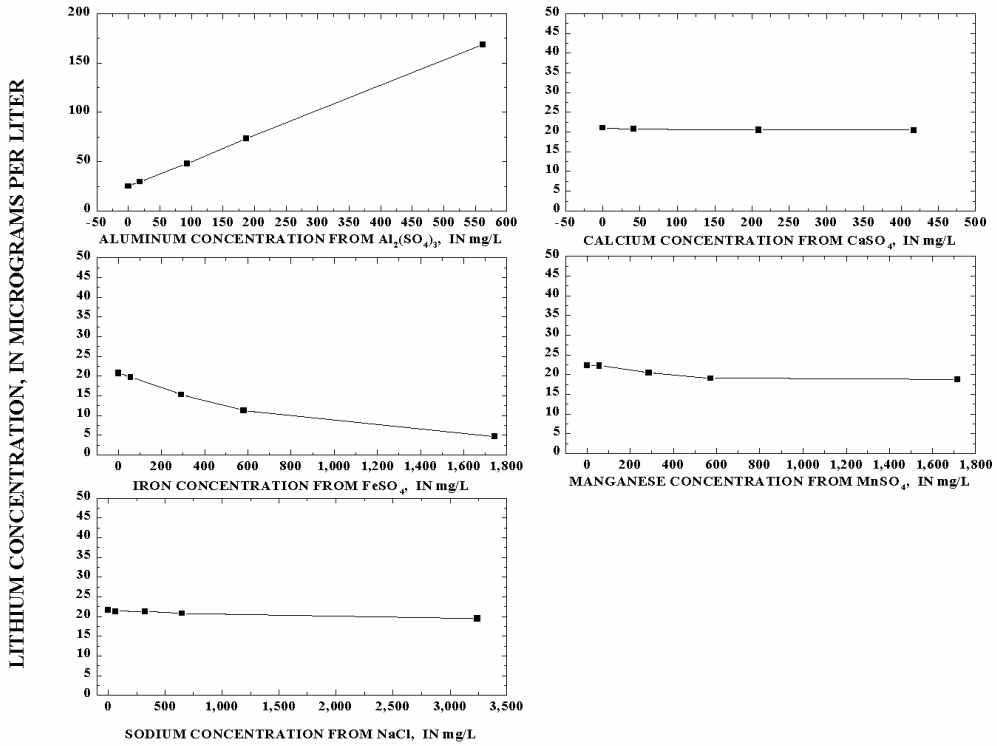


Figure 3. Relations among selected cations and anions and **lithium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

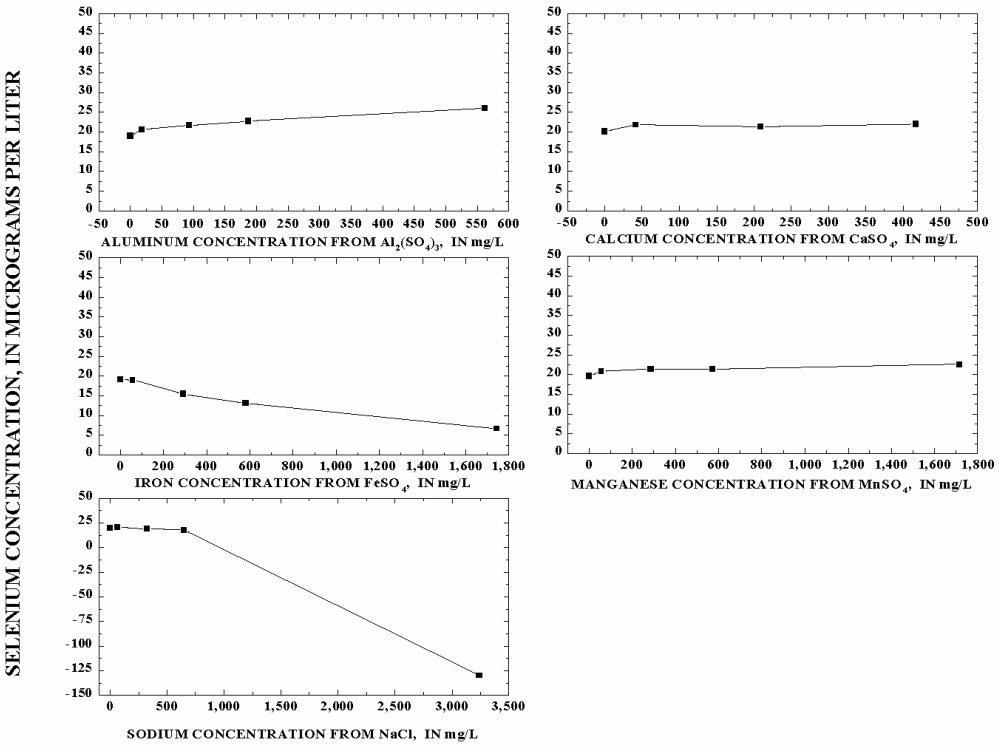


Figure 4. Relations among selected cations and anions and **selenium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

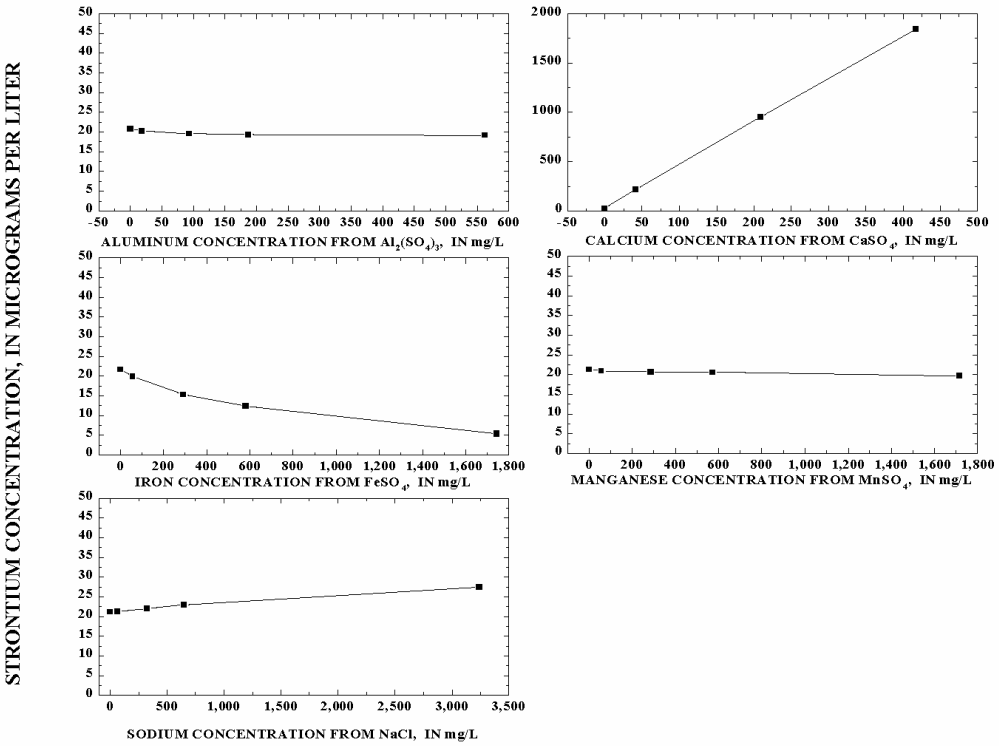


Figure 5. Relations among selected cations and anions and **strontium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

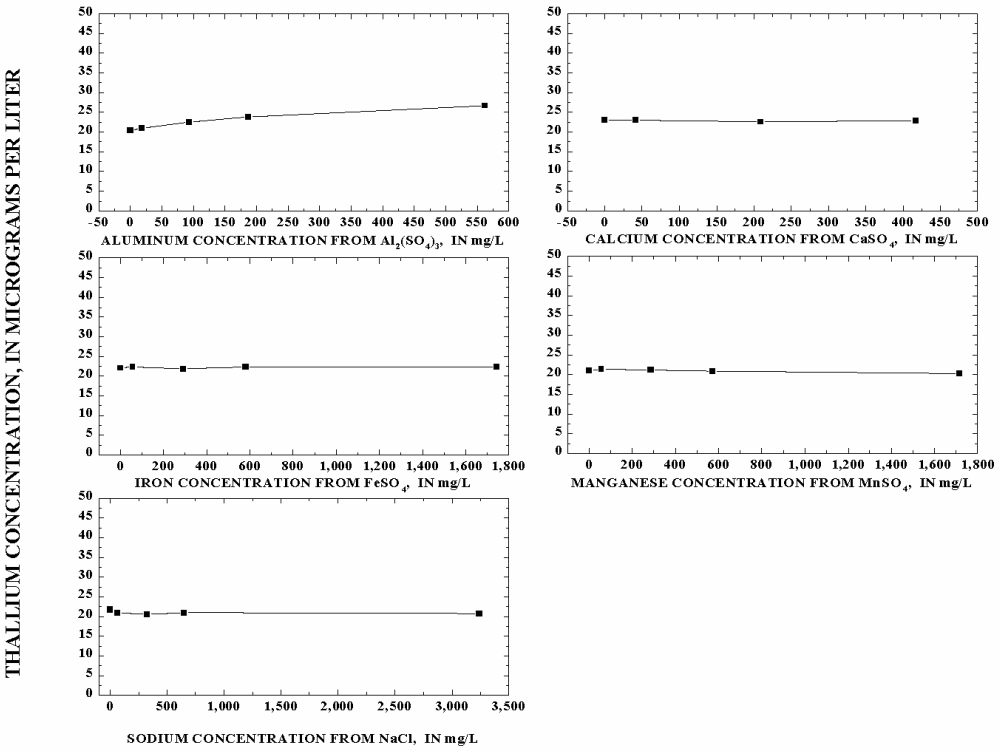


Figure 6. Relations among selected cations and anions and **thallium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

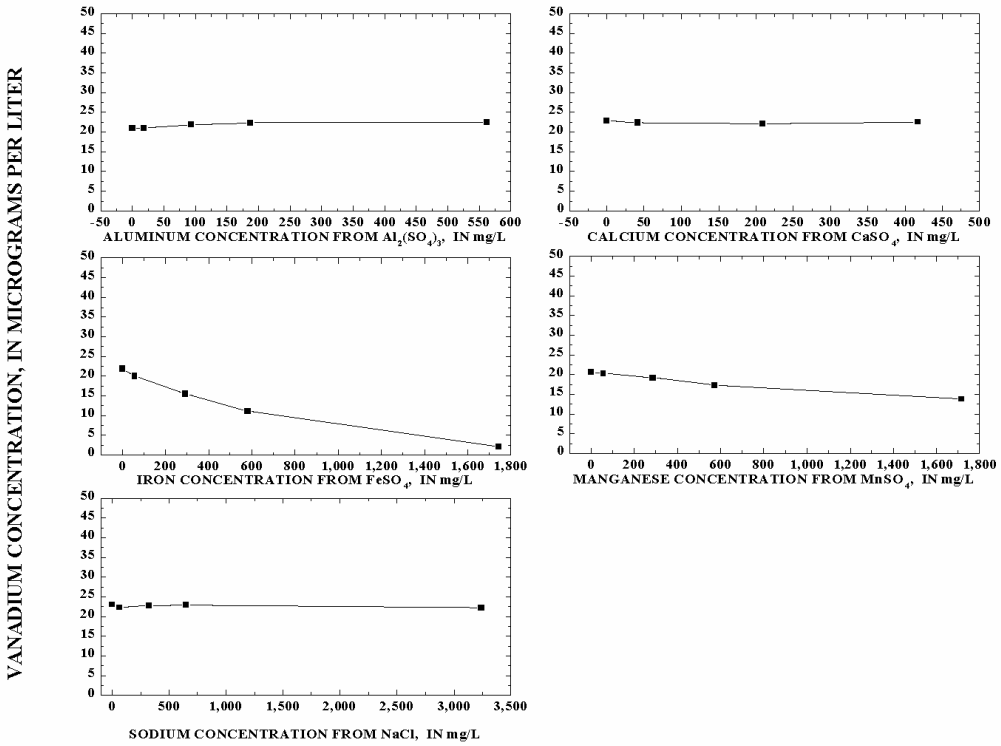


Figure 7. Relations among selected cations and anions and **vanadium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

and chloride) on the determination of arsenic. A relation with a slope equal to zero indicates no interference. A slope greater than zero suggests a positive interference or contamination; a slope of less than zero indicates signal suppression. Some elemental concentrations, for example, boron in calcium sulfate (fig. 2), lithium in aluminum sulfate (fig. 3), and strontium in calcium sulfate (fig. 5), increased with concomitant concentration. Such increases are from contamination (confirmed by using ICP–AES). Signal suppressions greater than 20 percent are indicated for some elements, depending on the nature of the concomitant. The results indicate that the suppression is not from sulfate or chloride but rather from the accompanying metal cation. Sulfur and chloride have low degrees of ionization, 14 and 0.9 percent, respectively, and therefore, they should not significantly induce signal suppression (Douglas, 1992).

In contrast, all the metal cations tested have degrees of ionization exceeding 90 percent. High iron concentration significantly suppressed all elements except for thallium. The response function was similar for each suppressed element, reaching a maximum of 75-percent suppression at about 1,700 mg/L iron. Lighter elements (less than 80 amu) were affected to a higher degree than heavier elements; for example, thallium was not affected. Manganese suppressed boron and vanadium in a similar manner, although to a lesser degree than iron. Selenium is severely suppressed in the presence of 3,200 mg/L sodium; the suppression is enhanced by selenium's 33-percent degree of ionization. In general, light elements are affected more severely than heavy elements, and heavier concomitant cations cause more severe suppression (Horlick and Shao, 1992). Concomitant concentrations that produce more than a 20-percent suppression are summarized in table 2. For most concomitant elements, 99 percent of the samples submitted for analysis at NWQL have concentrations that are much less than the concentrations listed in table 3. Nevertheless, the analyst must be aware of possible suppression whenever any element is present in a sample at unusually high concentrations; the sample can be diluted to eliminate the suppression.

3.2 *Spectral interferences.* Whenever possible, the isotope used for quantitation either has no spectral interferences or has a small number of potential spectral interferences. Spectral interferences can originate from isobaric ions, molecular ions, or doubly charged ions. The analyst must be aware of these potential spectral interferences when reviewing analytical results. Known spectral interferences for elements being added to the ICP–MS method are listed in the following sections.

3.3 *Isobaric interferences.* The only isobaric interference that affects the new elements is krypton on $^{82}Se^{+}$ and $^{78}Se^{+}$. Krypton can be a minor contaminant found in argon gas that is used to support the plasma, however, its concentration will remain constant for the standards and samples, and, therefore, usually does not require correction.

3.4 *Molecular-ion interferences.* There are several known molecular-ion interferences associated with arsenic, selenium, and vanadium. Chloride-associated molecular-ion interferences have been documented on arsenic ($^{75}As^{+}$) and vanadium ($^{51}V^{+}$) (see Horlick and Shao, 1992; Garbarino and Taylor, 1994). The molecular ions $^{40}Ar^{35}Cl^{+}$ and $^{35}Cl^{16}O$ interfere with $^{75}As^{+}$ and $^{51}V^{+}$, respectively. The $^{81}BrH^{+}$ ion interferes with the determination of selenium at 82 amu.

Table 3. Concomitant concentrations that result in more than a 20-percent ionization suppression on new elements determined by ICP–MS

[ICP–MS, inductively coupled plasma–mass spectrometry; mg/L, milligrams per liter; NWQL 99th percentile, the elemental concentration that is greater than 99 percent of the samples submitted to the National Water Quality Laboratory (NWQL); >, the greater than symbol signifies that the concentration causing 20-percent ionization suppression is greater than the stated concentration; nd, not determined because concomitant solution was contaminated with element]

Element	Aluminum, mg/L	Calcium, mg/L	Iron, mg/L	Manganese, mg/L	Sodium, mg/L
NWQL 99th percentile	*55*	*570*	*19*	*11*	*1,800*
Arsenic	>500	>400	300	>1,700	>3,200
Boron	>500	nd	300	300	>3,200
Lithium	nd	>400	300	300	>3,200
Selenium	nd	>400	300	>1,700	600
Strontium	>500	nd	300	>1,700	nd
Thallium	nd	>400	>1,700	>1,700	>3,200
Vanadium	>500	>400	300	300	>3,200

Because arsenic is monoisotopic, and vanadium has only one isotope with suitable natural abundance, no other interference-free isotopes are available for measurement. Equation 2 can be used to correct chloride interference on $^{75}As^{+}$; it is derived as follows:

$$^{75}As^{+}_{corr} = {}^{75}As^{+} - [{}^{40}Ar^{35}Cl^{+}/{}^{40}Ar^{37}Cl^{+}] \times \{{}^{40}Ar^{37}Cl^{+} - [{}^{77}Se/{}^{82}Se] \times {}^{82}Se^{+}\}. \quad (1)$$

The portion of the signal at 75 amu from the $^{40}Ar^{35}Cl^{+}$ interference is calculated by converting the signal at 77 amu from $^{40}Ar^{37}Cl^{+}$ (by measuring $^{77}Se^{+}$) after subtracting the contribution from $^{77}Se^{+}$ (by measuring $^{82}Se^{+}$) to its proportion at 75 amu. All the terms identified by *corr* are the corrected isotope intensities, and the terms in square brackets are the natural abundance isotope ratios; all other terms represent isotope intensities. After substituting constant terms that are based on natural abundance, equation 1 simplifies to

$$^{75}As^{+}_{corr} = {}^{75}As^{+} - 3.127 \times \{{}^{77}Se^{+} - [0.874] \times {}^{82}Se^{+}\}. \quad (2)$$

However, if bromide is also present in a sample, using equation 2 will give positively biased results because of the $^{81}BrH^{+}$ interference at $^{82}Se^{+}$. Therefore, whenever bromide is present, the correction must use $^{78}Se^{+}$ as shown in equation 3.

$$^{75}As^{+}_{corr} = {}^{75}As^{+} - 3.127 \times \{{}^{77}Se^{+} - [0.322] \times {}^{78}Se^{+}\}. \quad (3)$$

The positive bias in arsenic results when using equation 2 in the presence of bromide is shown in figure 8. The arsenic concentration will be 40 percent greater than is actually present with only 200 µg/L bromide in a sample. When using equation 3, such bias is not indicated. Nevertheless, in the absense of bromide, equation 2 is accurate as shown in figure 1 for up to 5,000 mg/L chloride.

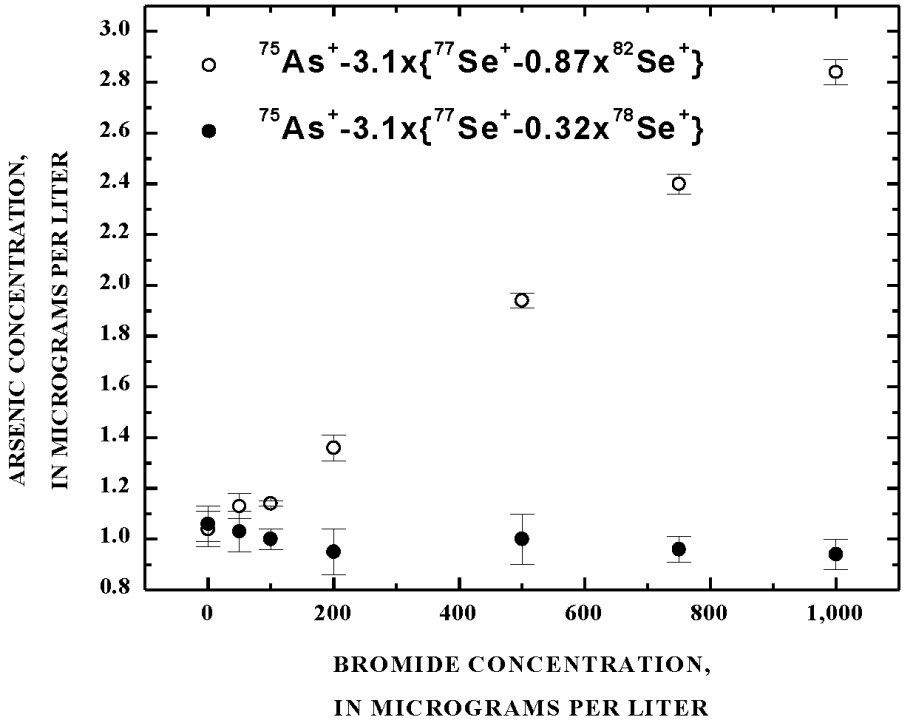


Figure 8. Accuracy of inductively coupled plasma–mass spectrometric arsenic determinations in the presence of bromide using different correction equations. The error bars correspond to one standard deviation based on three instrumental measurements.

Equation 5 must be used to correct chloride interference on $^{51}V^{+}$; it is derived as follows:

$$^{51}V^{+}_{corr} = {}^{51}V^{+} - [{}^{35}Cl^{16}O/{}^{37}Cl^{16}O] \times \{{}^{37}Cl^{16}O - [{}^{53}Cr/{}^{52}Cr] \times {}^{52}Cr^{+}\}. \quad (4)$$

The portion of the signal at 51 amu from the $^{35}Cl^{16}O^{+}$ interference is calculated by converting the signal at 53 amu from $^{37}Cl^{16}O^{+}$ (by measuring $^{53}Cr^{+}$) after subtracting the contribution from $^{53}Cr^{+}$ (by measuring $^{52}Cr^{+}$) to its proportion at 51 amu. After substituting constant terms that are based on natural abundance, equation 4 simplifies to

$$^{51}V^{+}_{corr} = {}^{51}V^{+} - 3.127 \times \{{}^{53}Cr^{+} - [0.113] \times {}^{52}Cr^{+}\}. \quad (5)$$

The accuracy of using equation 5 is shown in figure 7 for chloride concentrations less than 5,000 mg/L.

Masses at 52 and 53 amu are effected by interference from $^{40}Ar^{12}C^{+}$ and $^{40}Ar^{13}C^{+}$, respectively, that arise from any carbon present in the sample. The interference correction is based on the linear relation of the response at 52 amu from $^{40}Ar^{12}C^{+}$ to the concentration of carbon as shown in figure 9 (a similar linear relation is used for $^{53}Cr^{+}$). A water-soluble carbon compound such as sodium carbonate can be used to determine the relation (see Krushevska and others, 1998). The corrections are based on equations 6 and 7.

$$^{52}Cr^{+}_{corr} = {}^{52}Cr^{+} - ({}^{52}Cr^{+} / {}^{13}C^{+}) \times {}^{13}C^{+}. \quad (6)$$

$$^{53}Cr^{+}_{corr} = {}^{53}Cr^{+} - ({}^{53}Cr^{+} / {}^{13}C^{+}) \times {}^{13}C^{+}. \quad (7)$$

The terms ($^{52}Cr^{+}$ / $^{13}C^{+}$) and ($^{53}Cr^{+}$ / $^{13}C^{+}$) are variables that equal about 0.2 and 0.002, respectively. These variables depend on the instrument operating conditions and must be determined before every calibration. When using equation 6, figure 10 shows that the carbon interference can be eliminated. Such a correction is accurate at carbon concentrations to 1,000 mg/L. The $^{40}Ar^{13}C^{+}$ interference at $^{53}Cr^{+}$ is less severe than the corresponding interference at $^{52}Cr^{+}$ because of the lower natural abundance of ^{13}C. Nevertheless, the accuracy of using equation 7 is comparable to that shown in figure 10 for $^{52}Cr^{+}$.

3.5 *Doubly charged ion interferences.* A doubly charged ion is created in the plasma for any element that has a second ionization potential less than the ionization potential of argon. Strontium is the only new element that is potentially affected by doubly charged ion interferences. However, lutetium and ytterbium rarely are found in environmental samples at significant concentrations, and their interfering isotopes ($^{176}Lu^{2+}$ and $^{176}Yb^{2+}$) have low natural abundance.

4. Apparatus, Instrumentation, and Operating Conditions

4.1 *Labware.* Use clean Type A glass volumetric flasks to prepare all solutions. Store solutions in fluorinated ethylene propylene (FEP Teflon) bottles to maintain stable elemental concentrations. Regularly verify the accuracy of all pipets and volumetric flasks for preparing standard solutions by using either an analytical balance or an automatic calibrating spectrophotometer.

4.2 *Instrumentation.* Instrumentation previously described in methods by Faires (1993) and Garbarino and Taylor (1994) has been replaced by a Perkin-Elmer Elan 6000 ICP–MS. The Elan 6000 operates under the same general principles as earlier instrumentation but has higher sensitivity and lower MDLs. The standard Perkin-Elmer cross-flow nebulizer and spray chamber is used to introduce samples. The cross-flow nebulizer resists clogging (≤ 0.5 percent total dissolved solids) and is chemically inert.

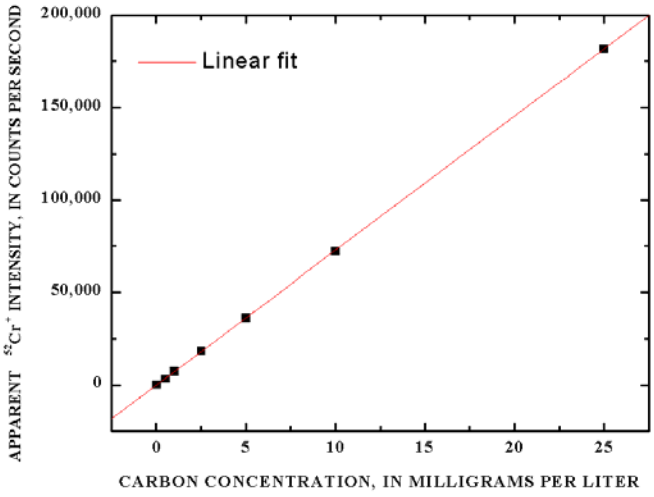


Figure 9. Relation of apparent chromium-52 signal from $^{40}Ar^{12}C^+$ as a function of increasing concentrations of carbon.

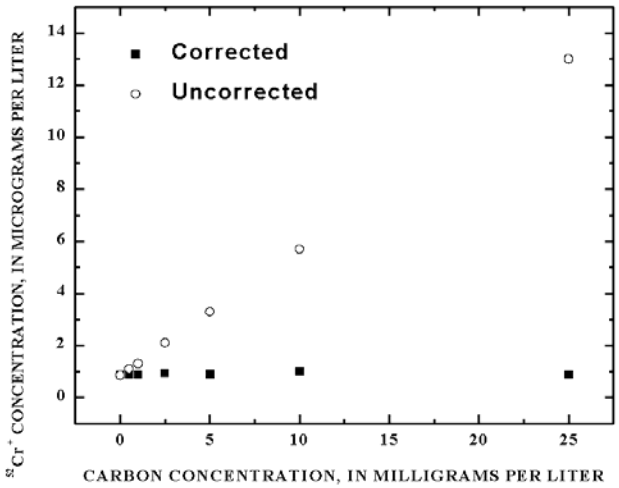


Figure 10. The accuracy of inductively coupled plasma–mass spectrometric chromium-52 determinations in the presence of carbon.

Other nebulizer designs can be used but must be resistant to clogging and capable of providing MDLs that are within a factor of two of those listed in table 1. Details of instrument operation are fully documented in the NWQL Standard Operating Procedure IM0011.1 (T.M. Struzeski, U.S. Geological Survey, written commun., 1998) and in the Perkin-Elmer (1997a and 1997b) software and hardware manuals.

Primary isotopes used to determine dissolved concentrations for the new elements are as follows:

Arsenic	75 amu	Boron	11 amu
Lithium	7 amu	Selenium	78 & 82 amu
Strontium	88 amu	Thallium	205 amu
Vanadium	51 amu		

5. Reagents and Calibration Standards

Earlier reports (Faires, 1993; Garbarino and Taylor, 1994) describe the preparation of calibration standards, internal standard solution, performance check solution, and tuning solution. New elements are calibrated by including them in the multielement standards described in method I-2477-92 at the same concentrations. Multielement standards are prepared in a matrix that is matched to the acid concentration of the samples being analyzed. Arsenic, selenium, and vanadium must not be included in a multielement standard that contains bromide or chloride. The interference solution has been added to verify the accuracy of interference corrections on arsenic and vanadium.

ASTM Type I reagent water (American Society for Testing and Materials, 1995, p. 122–124), spectroscopic grade commercial standards, and ultrapure acids must be used to prepare all solutions. All percentages represent volume-to-volume ratios. All concentrated acids and commercial standards must be verified to contain concentrations of concomitant elements that are less than the MDLs after the prescribed dilution. Every solution must be stored in a designated FEP Teflon bottle.

5.1 *Nitric acid* (HNO_3): Concentrated, specific gravity 1.41.

5.2 *Calibration blank*: Reagent water acidified to 0.4 percent HNO_3.

5.3 *Commercial single-element standard solutions*, 1.00 mL = 10 mg preserved in HNO_3 for each of the following: As, B (in water), Li, Se, Sr, Tl, and V.

5.4 *Commercial standard solutions*, chloride: 1.00 mL = 100 mg in water and bromide: 1.00 mL = 1.0 mg in water.

5.5 *Carbon standard solution,* 1.00 mL = 10 mg of carbon. Weigh 88.3 g dry Na_2CO_3 and transfer quantitatively into a 1-L volumetric flask; dissolve salt using 1,000 mL of reagent water.

5.6 *Multielement stock solution I*, 1.00 mL = 0.010 mg of As, B, Li, Se, Sr, Tl, and V: Dilute 1.0 mL of each commercial single-element standard to 1,000 mL in a volumetric flask with 1 percent HNO_3.

5.7 5.7 *Multielement calibration standard I,* 1.00 mL = 0.025 µg of As, B, Li, Se, Sr, Tl, and V: Include new elements in an existing calibration standard by diluting 0.250 mL of multielement stock solution I in a 100-mL volumetric flask with the calibration blank.

5.8 *Multielement calibration standard II,* 1.00 mL = 0.100 µg of As, B, Li, Se, Sr, Tl, and V: Include new elements in an existing calibration standard by diluting 1.0 mL of multielement stock solution I in a 100-mL volumetric flask with the calibration blank.

5.9 *Multielement calibration standard III,* 1.00 mL = 0.200 µg of As, B, Li, Se, Sr, Tl, and V: Include new elements in an existing calibration standard by diluting 2.0 mL of multielement stock solution I in a 100-mL volumetric flask with the calibration blank.

5.10 *Interference check standard,* 1.00 mL = 0.500 mg Cl, 0.0005 mg Br, 0.025 mg C, and 0.005 µg As and V: Dilute 5.0 mL of the commercial chloride standard solution, 0.50 mL of commercial bromide standard solution, 2.5 mL of carbon standard solution, and 0.50 mL of multielement stock solution I in a 1,000-mL volumetric flask with the calibration blank.

6. Sample Preparation

Filtered, acidified natural-water samples analyzed by ICP–MS for dissolved arsenic, boron, lithium, selenium, strontium, thallium, and zinc and other elements do not require additional processing.

7. Analytical Procedure

Refer to Perkin-Elmer (1997a, 1997b) and NWQL Standard Operating Procedure IM0011.1 (T.M. Struzeski, U.S. Geological Survey, written commun., 1998) for details of the analytical procedure. In addition, the accuracy of interference-correction equations must be determined by analyzing the interference check standard (see section 5.10) with every batch of samples. The Elan software automatically verifies that the results meet acceptance criteria.

8. Calculations

No additional calculations are required in this method.

9. Reporting Results

The number of significant figures reported varies with element and is a function of concentration. Whenever the concentration is less than the MDL for an element, the result is reported as less than the MDL (< MDL). All other elemental results should be reported using the criteria listed below. These criteria are based on the uncertainty suggested in the following Discussion of Results section. Alternatively, the variability in the mean concentration could be used to establish the appropriate number of significant figures to report for each individual sample matrix. The use of such a procedure would provide the most accurate estimate of the uncertainty associated with each sample.

For arsenic, lithium, and vanadium—

- If the concentration is greater than the MDL, but less than 10 µg/L, report result to two decimal places.

- If the concentration is greater than 10 µg/L, but less than 100 µg/L, report result to one decimal place.

- If the concentration is greater than 100 µg/L, report result to three significant figures.

For strontium and thallium—

- If the concentration is greater than the MDL, but less than 0.5 µg/L, report result to three decimal places.

- If the concentration is greater than 0.5 µg/L, but less than 10 µg/L, report result to two decimal places.

- If the concentration is greater than 10 µg/L, but less than 100 µg/L, report result to one decimal place.

- If the concentration is greater than 100 µg/L, report result to three significant figures.

For boron—

- If the concentration is greater than or equal to the MDL, but less than 100 µg/L, report result to one decimal place.

- If the concentration is greater than 100 µg/L, report result to three significant figures.

DISCUSSION OF RESULTS

The bias of the ICP–MS method for the determination of dissolved concentrations is established by comparing results to former USGS methods of analysis. ICP–MS method variability was determined from replicate analyses over a range of elemental concentrations prepared in a calibration-blank matrix. Results from the analysis of standard reference material, spiked samples, and up to 64 filtered natural-water samples are used to verify that dissolved arsenic, boron, lithium, selenium, strontium, thallium, and vanadium can be determined accurately by using ICP–MS. All former methods are USGS-approved methods (Fishman and Friedman, 1989; Fishman, 1993).

Results for Standard Reference Material

U.S. Geological Survey SRWS T145 was analyzed repetitively for 3 weeks to determine the long-term bias and variability of the ICP–MS method (see table 4). Results for all elements are within one standard deviation of the most probable value (MPV). The average long-term variability is 4±2 percent for elemental concentrations ranging from 10 to 200 µg/L. The paired Student *t*-Test was used to test the null hypothesis that the ICP–MS method yields mean elemental concentrations that are not significantly different from the MPVs. The p-values were calculated for each element to provide a level of confidence in accepting the null hypothesis. The larger the p-value (the level of significance) the greater the confidence in accepting the null hypothesis. When the p-value exceeds 0.05, the null hypothesis is acceptable at the 95-percent confidence level. The Student *t*-Test indicated that only the boron and lithium experimental results are not significantly different from the MPV. Nevertheless, the differences between the MPV and the experimental mean for arsenic, selenium, strontium, thallium, and vanadium are analytically insignificant.

The short-term variability of ICP–MS over an extended concentration range is shown in table 5. The variability is based on three replicate determinations (an acquisition time of about 1 minute) at each elemental concentration in the calibration blank matrix. The short-term variability was less than or equal to 5 percent at 0.5 µg /L for all elements except for boron which was 5 percent at 1 µg/L.

Table 4. Statistical analysis of long-term ICP–MS results for U.S. Geological Survey Standard Reference Water Sample T145

[ICP–MS, inductively coupled plasma–mass spectrometry; elemental results are in micrograms per liter; MPV, the most probable value; ±, the plus or minus symbol precedes the F-pseudosigma in the MPV column and the standard deviation in the experimental mean column; n, number of replicates used to calculate the experimental mean; p-value, level of significance; <, less than]

Element	MPV	Experimental mean, n=12	*t*-Test statistic	p-value
Arsenic	10 ± 1	10.3 ± 0.2	9.00	<0.0001
Boron	46 ± 6	45 ± 3	-0.23	0.8253
Lithium	27 ± 2	28 ± 1	1.54	0.1528
Selenium	10 ± 1	11.2 ± 0.2	18.63	<0.0001
Strontium	203 ± 9	208 ± 4	3.93	0.0024
Thallium	15 ± 3	14 ± 1	-3.12	0.0098
Vanadium	12 ± 2	10.6 ± 0.5	-7.86	<0.0001

Spike Recoveries in Natural-Water Samples

Spike recovery percentages listed in table 6 were determined for the new elements in matrices that are representative of reagent water, surface water, and ground water. Seven replicate recoveries at 5 to 10 times the MDL (the low-level spike) and 75 µg/L (the high-level spike) were determined in each matrix over a period of about 1 week. Average recoveries in the reagent-water matrix ranged from 93 to 105 percent for all elements except boron, whose low-level spike recovery was 77 percent. Recovery variability for the low-level spike ranged from 4 to 11 percent, depending on the element and matrix.

Recoveries of the high-level spike in the surface-water matrix were similar to those of the reagent-water matrix. Ambient concentrations of lithium, strontium, thallium, and vanadium in the surface water, however, hindered the recovery of the low-level spike. Recovery of low-level arsenic, boron, and selenium ranged from 88 to 126 percent. The variability in the recovery of 1 µg/L boron in the presence of 40 µg/L boron was 52 percent; the variability for arsenic and selenium was less than 6 percent at about the same spike concentration.

The ground-water matrix used for spike recoveries was selected to examine the effects of interferent species on elemental determinations. The ground water had high iron (340 mg/L) and sulfate (2,300 mg/L) concentrations, which exceed the level found in most samples analyzed by NWQL. Ambient concentrations of boron, lithium, and strontium precluded the recovery of the low-level spike, however, recoveries for arsenic, selenium, thallium, and vanadium averaged 70 percent. High-level spike recoveries averaged 70±4 percent, excluding thallium, which had 97 percent. The low percent recoveries for the lighter elements confirm the effects from ionization suppression (see section 3.1). Only thallium recovery, a heavier element, was not affected.

Table 5. Short-term analytical variability as a function of elemental concentration for ICP–MS

[ICP–MS, inductively coupled plasma–mass spectrometry, the percent relative standard deviations are based on three sequential determinations in a 0.4-percent solution of concentrated nitric acid in deionized water; µg/L, micrograms per liter; <MDL, less than the method detection limit; nd, not determined]

Elemental concentration, in µg/L	Percent relative standard deviation							
	0.05	0.1	0.5	1.0	10	50	100	250
Arsenic	<MDL	30	3	4	2	nd	1	0.6
Boron	nd	10	nd	5	2	0.4	0.2	nd
Lithium	0.9	8	2	2	0.7	nd	0.7	0.6
Selenium	<MDL	60	5	3	2	nd	2	0.7
Strontium	0.4	2	2	0.8	0.3	nd	0.7	0.6
Thallium	2	2	0.2	0.9	2	nd	0.8	1
Vanadium	8	10	2	0.6	0.7	nd	0.5	0.5

Table 6. Average percent spike recoveries in reagent-water, surface-water and ground-water matrices by inductively coupled plasma–mass spectrometry

[µg/L, micrograms per liter; number following the plus or minus symbol (±) is the standard deviation on the basis of seven determinations accrued on separate days; high spike, 75 µg/L for all elements; na, not applicable because the difference between the spike concentration and ambient concentration was greater than a factor of 10; <MDL, concentration is less than the method detection limit; %, percent]

Element	Low-spike, in µg/L	Reagent-water matrix			Surface-water matrix		
		Ambient concentration, in µg/L	Low-spike recovery, in %	High-spike recovery, in %	Ambient concentration, in µg/L	Low-spike recovery, in %	High-spike recovery, in %
Arsenic	0.5	<MDL	102±4	101±1	1	112±6	105±2
Boron	1	<MDL	77±9	103±2	40	88±52	102±4
Lithium	0.2	<MDL	94±8	105±3	30	na	99±7
Selenium	0.9	<MDL	110±10	99±2	2	126±7	104±3
Strontium	0.2	<MDL	93±4	104±3	390	na	114±8
Thallium	0.05	<MDL	97±11	100±6	60	na	103±5
Vanadium	0.6	<MDL	98±4	105±4	60	na	104±4
		Ground-water matrix					
Arsenic	0.5	1	75±8	68±4			
Boron	1	350	na	70±20			
Lithium	0.2	220	na	72±12			
Selenium	0.9	1	80±10	73±4			
Strontium	0.2	6,000	na	na			
Thallium	0.05	<MDL	60±20	97±5			
Vanadium	0.6	<MDL	64±20	64±10			

Comparison of Inductively Coupled Plasma–Mass Spectrometry to Former Methods of Analysis

The ICP–MS results are compared to former methods of analysis such as hydride generation–atomic absorption spectrophotometry (HG–AAS) and inductively coupled plasma–atomic emission spectrometry (ICP–AES). Up to 64 filtered, acidified natural-water samples were selected from the population of such samples submitted to the NWQL. The samples have a wide range of elemental concentrations and specific conductance. Surface-water and ground-water samples are included in the sample set; the number of each type is about proportional to its fraction of the total submitted for analysis during an average year. Other chemical characteristics that often influence the performance of analytical methods, such as sulfate and chloride concentrations, were also considered in the selection process (see table 7 for the chemical characteristics of the samples).

Results were evaluated by using several different approaches. Because the data extend over a wide concentration range, it is inappropriate to use the paired Student *t*-test to evaluate the null hypothesis because errors, whether random or systematic, are independent of the concentration. Consequently, linear regression analysis is used to calculate the slope, *y*-intercept, and coefficient of determination (R^2) for the equation that describes the relation between ICP–MS and a former USGS method. A slope coefficient of one and a *y*-intercept of zero indicate exact correlation. The corresponding p-values indicate the degree of confidence in each coefficient. Box plots are also provided to show the distribution of the results from each method. The non-parametric Wilcoxon Signed Rank Test is used as another test to determine whether there is a significant difference between results from the ICP–MS and former USGS methods. Data that were less than the highest MDL or MRL were omitted from the data set prior to statistical analysis. Statistical analysis results are summarized in the following paragraphs and are listed in table 8. Illustrations for the statistical tests are provided in figures 11 through 16.

Table 7. Chemical characteristics of natural-water samples used to evaluate ICP–MS

[ICP–MS, inductively coupled plasma–mass spectrometry; µg/L, microgram per liter; mg/L, milligram per liter; <MDL, less than the method detection limit; SC, specific conductance]

Element, in µg/L	25th percentile	Median	75th percentile	Maximum
Arsenic	1.7	2.9	7.4	104
Boron	52	120	210	1,700
Lithium	22	39	69	1,600
Selenium	2.9	4.5	8.2	34
Strontium	350	630	1,900	20,800
Thallium	All results were <MDL for former methods			
Vanadium	1.8	4.7	9.7	1,530
Constituent				
Chloride, in mg/L	9.8	68	501	9,176
SC, in µS/cm[1]	455	944	2,490	52,600
Sulfate, in mg/L	108	309	1,306	16,832

[1]Specific conductance in microsiemens per centimeter at 25 °C (µS/cm).

Results of the linear regression analyses show that there is strong correlation between ICP–MS and former USGS methods for the analysis of filtered, acidified natural water. The slope coefficients range from 0.90 to 1.1 (see table 8). The corresponding p-values indicate with 95-percent confidence that all the slopes are not significantly different from 1.0. Thallium concentrations were less than the GF–AAS and ICP–MS method detection limits in all samples. With the exception of lithium and vanadium, the p-values for the y-intercepts indicate a significant nonzero intercept that might result from differences in sensitivity or from the wide range of sample concentrations. For example, boron and strontium concentrations range from the MDLs to about 1,700 and 21,000 µg/L, respectively (see table 7).

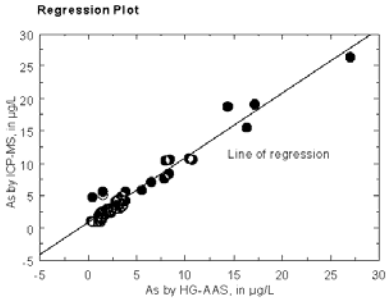
The p-values of the Wilcoxon Signed Rank Test indicate with 95-percent confidence that there is no significant difference between ICP–MS and ICP–AES results for dissolved boron and vanadium. In general, the descriptive statistics and figures 11 through 16 indicate that differences among results for the other elements are analytically insignificant considering the range of sample concentrations in the data set.

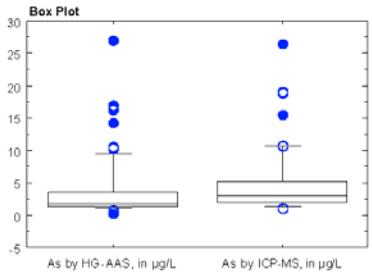
Table 8. Statistical analysis summary of inductively coupled plasma–mass spectrometry and former methods of analysis

[Coef., either the slope or the y-intercept regression coefficient; p-value, level of significance; R^2, coefficient of determination; HG–AAS, hydride generation–atomic absorption spectrophotometry; ICP–AES, inductively coupled plasma–atomic emission spectrometry; GF–AAS, stabilized temperature graphite furnace–atomic absorption spectrophotometry; nd, not detected; <, less than. All thallium results were less than the method detection limit]

Element	Former method	Slope		*y*-intercept		R^2	Wilcoxon signed rank test p-value[c]
		Coef.	p-value[a]	Coef.	p-value[b]		
Arsenic	HG–AAS	1.0	<0.0001	0.95	<0.0001	0.945	<0.0001
Boron	ICP–AES	0.90	<0.0001	13	0.0002	0.995	0.3714
Lithium	ICP–AES	0.92	<0.0001	1.0	0.2197	0.999	<0.0001
Selenium	HG–AAS	1.1	<0.0001	1.7	0.0002	0.864	<0.0001
Strontium	ICP–AES	0.94	<0.0001	96	0.0044	0.996	0.0030
Thallium	GF–AAS	nd	nd	nd	nd	nd	nd
Vanadium	ICP–AES	0.96	<0.0001	0.10	0.9242	0.919	0.1340

[a]The null hypothesis: slope is not equal to one.
[b]The null hypothesis: y-intercept is equal to zero.
[c]The null hypothesis: the difference in concentration between the new inductively coupled plasma–mass spectrometric method and the former USGS method is equal to zero.





Descriptive Statistics

	As by HG-AAS, in µg/L	As by ICP-MS, in µg/L
Mean	3.82	4.76
Std. Dev.	4.87	4.99
Std. Error	0.63	0.65
Count	59	59
Minimum	0.20	0.93
Maximum	27.00	26.48
# Missing	0	0

Wilcoxon Signed Rank Test for As by HG-AAS, in µg/L, As by ICP-MS, in µg/L

# 0 Differences	0
# Ties	1
Z-Value	-6.01
P-Value	<0.0001
Tied Z-Value	-6.01
Tied P-Value	<0.0001

Wilcoxon Rank Info for As by HG-AAS, in µg/L, As by ICP-MS, in µg/L

	Count	Sum Ranks	Mean Rank
# Ranks < 0	53	1681.50	31.73
# Ranks > 0	6	88.50	14.75

EXPLANATION

Hydride generation-atomic absorption spectrophotometry (HG-AAS); Inductively coupled plasma-mass spectrometry (ICP-MS); micrograms per liter (µg/L); Std. Dev., standard deviation; Std. Error, standard error; <, less than; >, greater than; #, number

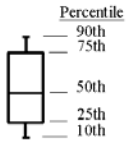
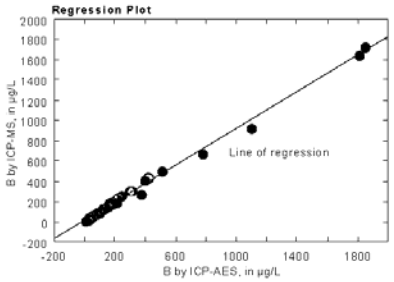
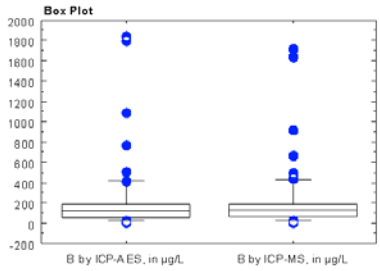


Figure 11. Statistical analysis of **arsenic (As)** results for 59 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and hydride generation–atomic absorption spectrophotometry.





Descriptive Statistics

	B by ICP-AES, in µg/L	B by ICP-MS, in µg/L
Mean	218.74	210.77
Std. Dev.	347.35	315.04
Std. Error	44.11	40.01
Count	62	62
Minimum	9.15	10.31
Maximum	1849.43	1721.82
# Missing	0	0

Wilcoxon Signed Rank Test for B by ICP-AES, in µg/L, B by ICP-MS, in µg/L

# 0 Differences	0
# Ties	0
Z-Value	-0.89
P-Value	0.3714
Tied Z-Value	-0.89
Tied P-Value	0.3714

Wilcoxon Rank Info for B by ICP-AES, in µg/L, B by ICP-MS, in µg/L

	Count	Sum Ranks	Mean Rank
# Ranks < 0	34	1104.00	32.47
# Ranks > 0	28	849.00	30.32

EXPLANATION

Inductively coupled plasma-atomic emission spectrometry (ICP-AES); Inductively coupled plasma-mass spectrometry (ICP-MS); micrograms per liter (µg/L); Std. Dev., standard deviation; Std. Error, standard error; <, less than; >, greater than; #, number

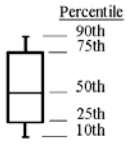
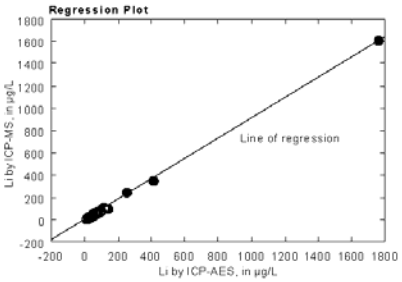
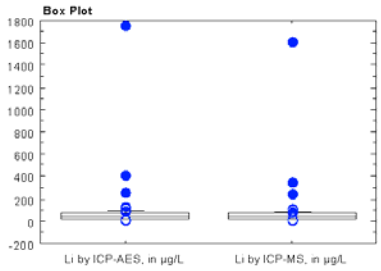


Figure 12. Statistical analysis of **boron (B)** results for 62 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and inductively coupled plasma–atomic emission spectrophotometry.





Descriptive Statistics

	Li by ICP-AES, in µg/L	Li by ICP-MS, in µg/L
Mean	81.33	75.39
Std. Dev.	223.15	204.17
Std. Error	28.11	25.72
Count	63	63
Minimum	5.20	4.01
Maximum	1760.38	1615.30
# Missing	0	0

Wilcoxon Signed Rank Test for Li by ICP-AES, in µg/L, Li by ICP-MS, in µg/L

# 0 Differences	0
# Ties	0
Z-Value	-3.90
P-Value	<0.0001
Tied Z-Value	-3.90
Tied P-Value	<0.0001

Wilcoxon Rank Info for Li by ICP-AES, in µg/L, Li by ICP-MS, in µg/L

	Count	Sum Ranks	Mean Rank
# Ranks < 0	19	439.00	23.11
# Ranks > 0	44	1577.00	35.84

EXPLANATION

Inductively coupled plasma-atomic emission spectrometry (ICP-AES); Inductively coupled plasma-mass spectrometry (ICP-MS); micrograms per liter (µg/L); Std. Dev., standard deviation; Std. Error, standard error; <, less than; >, greater than; #, number

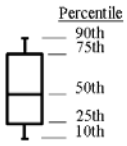
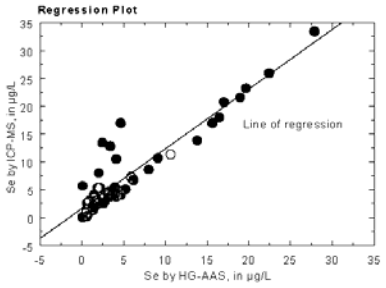
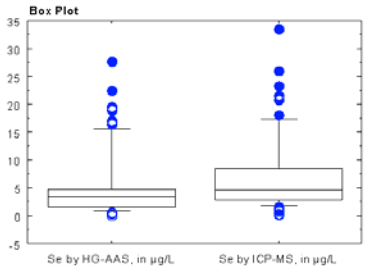


Figure 13. Statistical analysis of **lithium (Li)** results for 63 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry inductively coupled plasma–atomic emission spectrophotometry.





Descriptive Statistics

	Se by HG-AAS, in µg/L	Se by ICP-MS, in µg/L
Mean	5.07	7.09
Std. Dev.	5.91	6.80
Std. Error	0.74	0.85
Count	64	64
Minimum	0	0.22
Maximum	27.80	33.62
# Missing	0	0

Wilcoxon Signed Rank Test for Se by HG-AAS, in µg/L, Se by ICP-MS, in µg/L

# 0 Differences	0
# Ties	0
Z-Value	-6.86
P-Value	<0.0001
Tied Z-Value	-6.86
Tied P-Value	<0.0001

Wilcoxon Rank Info for Se by HG-AAS, in µg/L, Se by ICP-MS, in µg/L

	Count	Sum Ranks	Mean Rank
# Ranks < 0	62	2066.00	33.32
# Ranks > 0	2	14.00	7.00

EXPLANATION

Hydride generation-atomic absorption spectrophotometry (HG-AAS); Inductively coupled plasma-mass spectrometry (ICP-MS); micrograms per liter (µg/L); Std. Dev., standard deviation; Std. Error, standard error; <, less than; >, greater than; #, number

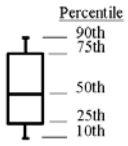
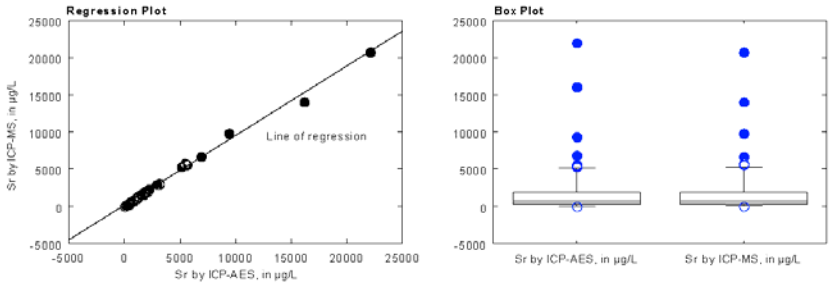


Figure 14. Statistical analysis of **selenium (Se)** results for 64 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and hydride generation–atomic absorption spectrophotometry.



Descriptive Statistics

	Sr by ICP-AES, in µg/L	Sr by ICP-MS, in µg/L
Mean	1891.95	1870.69
Std. Dev.	3644.10	3425.62
Std. Error	459.11	431.59
Count	63	63
Minimum	31.02	30.53
Maximum	22068.32	20823.39
# Missing	1	1

Wilcoxon Signed Rank Test for Sr by ICP-AES, in µg/L, Sr by ICP-MS, in µg/L

# 0 Differences	0
# Ties	0
Z-Value	-2.96
P-Value	0.0030
Tied Z-Value	-2.96
Tied P-Value	0.0030

One case was omitted due to missing values.

Wilcoxon Rank Info for Sr by ICP-AES, in µg/L, Sr by ICP-MS, in µg/L

	Count	Sum Ranks	Mean Rank
# Ranks < 0	43	1441.00	33.51
# Ranks > 0	20	575.00	28.75

One case was omitted due to missing values.

EXPLANATION

Inductively coupled plasma-atomic emission spectrometry (ICP-AES); Inductively coupled plasma-mass spectrometry (ICP-MS); micrograms per liter (µg/L); Std. Dev., standard deviation; Std. Error, standard error; <, less than; >, greater than; #, number

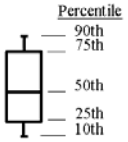
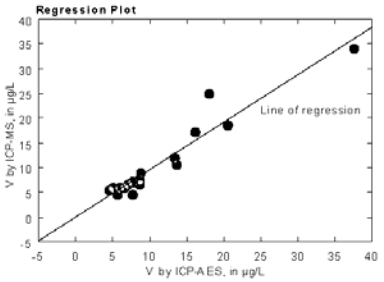
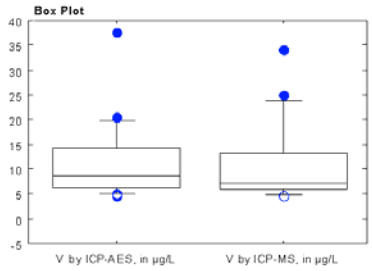


Figure 15. Statistical analysis of **strontium (Sr)** results for 63 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and inductively coupled plasma–atomic emission spectrophotometry.





Descriptive Statistics

	V by ICP-AES, in µg/L	V by ICP-MS, in µg/L
Mean	11.46	11.06
Std. Dev.	8.23	8.21
Std. Error	2.00	1.99
Count	17	17
Minimum	4.60	4.64
Maximum	37.54	34.10
# Missing	0	0

Wilcoxon Signed Rank Test for V by ICP-AES, in µg/L, V by ICP-MS, in µg/L

# 0 Differences	0
# Ties	0
Z-Value	-1.49
P-Value	0.1359
Tied Z-Value	-1.49
Tied P-Value	0.1359

Wilcoxon Rank Info for V by ICP-AES, in µg/L, V by ICP-MS, in µg/L

	Count	Sum Ranks	Mean Rank
# Ranks < 0	6	45.00	7.50
# Ranks > 0	11	108.00	9.82

EXPLANATION

Inductively coupled plasma-atomic emission spectrometry (ICP-AES); Inductively coupled plasma-mass spectrometry (ICP-MS); micrograms per liter (µg/L); Std. Dev., standard deviation; Std. Error, standard error; <, less than; >, greater than; #, number

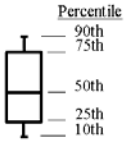


Figure 16. Statistical analysis of **vanadium (V)** results for 17 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and inductively coupled plasma–atomic emission spectrophotometry.

CONCLUSIONS

Results from reference material, spike recoveries, and the analysis of natural-water samples were used to evaluate the overall bias and variability of the determination of dissolved arsenic, boron, lithium, selenium, strontium, thallium, and vanadium by inductively coupled plasma–mass spectrometry (ICP–MS). All test results provide an accurate estimate of the expected analytical performance. The following list outlines the major conclusions of this report. In addition to analytical performance comparisons, suggestions are provided for selecting appropriate methodology and the potential effects of the use of ICP–MS on long-term trend analysis in water-quality studies.

- Method detection limits (MDLs) for ICP–MS are between about 10 and 200 times lower than hydride generation–atomic absorption spectrophotometry (HG–AAS) and inductively coupled plasma–atomic emission spectrometry (ICP–AES) methods.

- The short- and long-term accuracy for the determination of the new elements by ICP–MS were acceptable; all the elements were within one standard deviation of the most probable value.

- Data from up to 64 surface- and ground-water samples indicated that there was no significant method bias for the determination of dissolved arsenic, boron, lithium, selenium, strontium, and vanadium by ICP–MS.

- Determination of dissolved arsenic and selenium by ICP–MS does not require any digestion procedure as does the HG–AAS method.

- Matrix interferences from high concentrations of concomitant metals can affect the determination of lighter elements (< 60 amu). The concomitant concentrations that cause significant interference, however, are generally above the 99th percentile of elemental concentrations in samples submitted to the National Water Quality Laboratory (NWQL).

- ICP–MS is a state-of-the-art multielement technique that is more efficient and cost effective than former USGS single-element methods, such as HG–AAS. ICP–MS is the method of choice whenever multiple elements must be determined or whenever elemental concentrations are less than 10 µg/L.

- Use of the ICP–MS method for the determination of arsenic and selenium reduces the amount of chemical reagents and chemical waste when compared to HG–AAS.

- Data from ICP–MS will affect long-term trends in water-quality studies because of the reduced bias and variability at elemental concentrations less than 10 µg/L.

REFERENCES CITED

American Society for Testing and Materials, 1995, Annual book of ASTM standards, Section 11, Water (D1193, Standard specification for reagent water): Philadelphia, v. 11.01, p. 122–124.

Douglas, D.J., 1992, Fundamental aspects on inductively coupled plasma–mass spectrometry *in* Montaser, Akbar, and Golightly,

D.W., eds., Inductively coupled plasmas in analytical atomic spectrometry (2d ed.): New York, VCH Publishers, Inc., p. 614.

Faires, L.M., 1993, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of metals in water by inductively coupled plasma–mass spectrometry: U.S. Geological Survey Open-File Report 92-634, 28 p.

Fishman, M.J., and Friedman, L.C., eds., 1989, Methods for determination of inorganic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, 545 p.

Fishman, M.J., ed., 1993, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of inorganic and organic constituents in water and fluvial sediments: U.S. Geological Survey Open-File Report 93-125, 217 p.

Garbarino, J.R., and Struzeski, T.M., 1998, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of elements in whole-water digests using inductively coupled plasma–optical emission spectrometry and inductively coupled plasma–mass spectrometry: U.S. Geological Survey Open-File Report 98-165, 101 p.

Garbarino, J.R., and Taylor, H.E., 1994, Inductively coupled plasma–mass spectrometric determination of dissolved trace elements in natural water: U.S. Geological Survey Open-File Report 94-358, 28 p.

Horlick, Gary, and Shao, Youbin, 1992, Inductively coupled plasma–mass spectrometry for elemental analysis *in* Montaser, Akbar, and Golightly, D.W., eds., Inductively coupled plasmas in analytical atomic spectrometry (2d ed.): New York, VCH Publishers, Inc., p. 551.

Horowitz, A.J., Demas, C.R., Fitzgerald, K.K., Miller, T.L., and Rickert, D.A., 1994, U.S. Geological Survey protocol for the collection and processing of surface-water samples for the subsequent determination of inorganic constituents in filtered water: U.S. Geological Survey Open-File Report 94-539, 57 p.

Krushevska, Antoaneta, Waheed, Shahida, Nóbrega, Joaquim, Amarisiriwardena, Dulasiri, and Barnes, R.M., 1998, Reducing polyatomic interferences in the ICP–MS determination of chromium and vanadium in biofluids and tissues: Applied Spectroscopy, v. 52, no. 2, p. 205–211.

Perkin-Elmer, 1997a, Elan 6000 inductively coupled plasma–mass spectrometer software guide: Norwalk, Connecticut, Perkin-Elmer part number 0993-8968 Rev. F.

_____1997b, Elan 6000 inductively coupled plasma–mass spectrometer hardware guide: Norwalk, Connecticut, Perkin-Elmer part number 0993-8969 Rev. E.

U.S. Environmental Protection Agency, 1997, Guidelines for establishing test procedures for the analysis of pollutants (Part 136, Appendix B. Definition and procedure for the determination of the method detection limit—Revision 1.11): U.S. Code of Federal Regulations, Title 40, revised as of July 1, 1997, p. 265–267.

11. Appendix I. Glossary of terms from USGS National Water Quality Laboratory

AA - Atomic Absorption

AFD - Atomic Fluorescence detection

ASF - Automated-segmented flow, colorimetric

AES - Atomic Emission Spectrometry

BNA - Base neutral acid

BTM - Bottom material

CARB - Carbon

Che-X - Chelation extraction

CVM - Cold-vapor method

DBCP - Dibromochloropropane

DCP - Direct-current plasma

DIS - Dissolved, Pertains to the constituents in a representative water sample that pass through a 0.45-micrometer membrane filter for inorganic analysis or a 0.7-micrometer glass fiber filter for organic analysis. This is a convenient operational definition used by Federal agencies that collect water data. Determinations of "dissolved" constituents are made on subsamples of the filtrate.

GFAA - Graphite Furnace Atomic Absorption

DW - Drinking water

EDB - 1,2-Dibromoethane

EPA - Environmental Protection Agency

False negative - (also called type II error or beta error) - A statement that a substance is not present (was not found) in a sample when the substance was present (Keith, 1992).

False positive - (also called type I error or alpha error) - A statement that a substance is present in a sample when it is not (Keith, 1992).

FIL - Filtered (in the past known as dissolved (DIS)), Pertains to the constituents in a representative water sample that pass through a 0.45-micrometer membrane filter for inorganic analysis or a 0.7-micrometer glass fiber filter for organic analysis. This is a convenient operational definition used by Federal agencies that collect water data. Determinations of "dissolved" constituents are made on subsamples of the filtrate.

GFAA - Graphite Furnace Atomic Absorption

GC - Gas chromatography

GC/ECD - Gas chromatography/electron capture detector

GC/FID - Gas chromatography/flame ionization detector

GC/FPD - Gas chromatography/flame photometric detector

GC/MS - Gas chromatography/Mass Spectrometry

GC/MS/SIM - Gas chromatography/Mass Spectrometry/selected ion monitoring

GC/NPD - Gas chromatography/Nitrogen Phosphorous detector

HPLC - High-performance liquid chromatography

IC - Ion Chromatography

ICP - Inductively Coupled Plasma

ICP/MS - Inductively Coupled Plasma/Mass Spectrometry

Information-rich methods - Classified as organic methods that use either mass spectrometric or photodiode array ultraviolet/visible spectroscopic detection. These methods have additional qualifying information that allows enhanced analyte identification.

LC - Lab Code

LT-MDL - Long Term Method Detection Level - A detection level derived by determining the standard deviation of a minimum of 24 MDL spike sample measurements over an extended period of time. LT-MDL data are collected on a continuous basis to assess year-to-year variations in the LT-MDL. The LT-MDL controls false positive error. The chance of falsely reporting a concentration at or greater than the LT-MDL for a sample that did not contain the analyte is predicted to be less than or equal to 1 percent.

LRL - Laboratory Reporting Level - Generally equal to twice the yearly determined LT-MDL. The LRL controls false negative error. The probability of falsely reporting a non-detection for a sample that contained an analyte at a concentration equal to or greater that the LRL is predicted to be less than or equal to 1 percent. The value of the LRL will be reported with a "less than" remark code for samples in which the analyte was not detected. The National Water Quality Laboratory collects quality-control data from selected analytical methods on a continuing basis to determine long-term method detection levels (LT-MDL's) and establish laboratory reporting levels (LRL's). These values are re-evaluated annually based on the most current quality-control data and may, therefore, change. [Note: In several previous NWQL documents (Connor and others, 1998; NWQL Technical Memorandum 98.07, 1998), the LRL was called the non-detection value or NDV - a term that is no longer used.]

LS - Laboratory Schedule

LSD - Least Significant Digit - The first real number (nonzero) to the right of the decimal point in the standard deviation of the LT-MDL spike samples.

MDL - Method detection limit - Minimum concentration of a substance that can be measured and reported with 99-percent confidence that the analyte concentration is greater than zero. It is determined from the analysis of a sample in a given matrix containing the analyte (U.S. Environmental Protection Agency, 1997). At the MDL concentration, the risk of a false positive is predicted to be less than or equal to 1 percent.

mg/L - milligram per liter

MRL - Minimum reporting level - Smallest measured concentration of a constituent that may be reliably reported by using a given analytical method (Timme, 1995).

NA - Not applicable.

NAWQA - National Water-Quality Assessment Program.

Non-quantitative result - Unable to report a concentration. Either not detected or detected in a region of high uncertainty (high probability of false positive) and outside the calibration range.

NTU - Nephelometric turbidity unit

Nutrients - The Nutrients Unit analyzes for nitrogen and phosphorus in various forms, as well as chloride, sulfate, and some other minor analytes.

NWQL - National Water Quality Laboratory

OI - Oceanographic institute

PCB - Polychlorinated Biphenyls

PCN - Polychlorinated Naphthalene

Plasma - The Plasma Unit analyzes for trace metals by ICP.

Pt - Co - Platinum-cobalt units

Quantitative result - Concentration reported. Value is within range of instrument calibration and, thus, of higher certainty.

ROE - Residue on Evaporation

RL - Reporting level

Semi-quantitative result - Estimated concentration reported because it is outside the calibration range.

SPE - Solid phase extraction

SUS - Suspended solids

TCE - trichloroethene

THM - trihalomethane

TOT - Total

ug/L - microgram per liter

uS/cm - microsiemens per centimeter at 25 degrees Celsius

USGS - U.S. Geological Survey

VOC - Volatile Organic Compound

VOI - Volatile on Ignition

WWR - Whole water, recoverable.

WY - Water Year

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #13

Laboratory Analysis Methods for Water Quality, Utah Units

Version 1.00 (10/30/06)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

This SOP presents the laboratory methods used to analyze water samples collected from NCPN units in National Parks in the state of Utah. Most of the material in this SOP is from the UT Division of Epidemiology and Laboratory Services (DELS) operating manual version 7.003 revised June 2004 and standard reference sources for water analysis.

Table of Contents

List of Tables **4**
1. Introduction **5**
2. Methods **5**
3. Laboratory Quality Assurance **8**
3.1. Sample receiving 8
4. Laboratory reporting levels (LRL) **11**
5. Appendix A: Sample receipt checklist **12**
6. Appendix B: Utah Department of Water Quality Chemical Analysis Suites **15**
6.1. Suite 2 chemistry 15
6.2. Suite 4 chemistry 15
6.3. Suite 3 chemistry 15
7. Appendix C: UTDWQ Laboratory Reporting Limits **16**
8. Appendix D: Analytical Error Control **19**
9. Appendix E: Alkalinity, Method SM2320B **20**
10. Appendix F: EPA Method 350.1, Determination of Ammonia Nitrogen by Semi-automated Colorimetry **21**
11. Appendix G: EPA Method 300.0, Determination of Inorganic Anions by Ion Chromatography **24**
12. Appendix H: EPA Method 531.1, Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection High Performance Liquid Chromatography with Post Column Derivitization **28**
13. Appendix I: EPA Method 410.4, Chemical Oxygen Demand (Colorimetric, Automated; Manual) **32**
14. Appendix J: EPA Method 323.3, Chloride **35**
15. Appendix K: EPA Method 515.1, Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector **36**
16. Appendix L: Standards Methods Number SM3500 CD Chromium-VI **41**
17. Appendix M: EPA Method 110.2, Color (Colorimetric-Platinum-Cobalt) **42**
18. Appendix N: EPA Method 120.1, Conductance (Specific Conductance, umhos at 25°C) **45**
19. Appendix O: EPA Method 1623, Cryptosporidium and Giardia in Water by Filtration/IMS/FA **48**
20. Appendix P: EPA Method 335.4, Determination of Total Cyanide by Semi-Automated Colorimetry **52**
21. Appendix Q: Standard Methods Number SM4500C: Fluoride **55**
22. Appendix R: EPA Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry **56**
23. Appendix S: EPA Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry **61**
24. Appendix T: EPA Method 200.9, Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption **67**
25. Appendix U: EPA Method 245.1, Mercury (Manual Cold Vapor Technique) **72**
26. Appendix V: EPA Method 353.2, Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction) **75**
27. Appendix W: EPA Method 365.1, Phosphorus, All Forms (Colorimetric, Automated, Ascorbic Acid) **79**
28. Appendix X: EPA Method 370.1, Silica, Dissolved (Colorimetric) **83**
29. Appendix Y: EPA Method 150.1, pH (Electrometric Method) **86**
30. Appendix Z: EPA Method 160.1, Residue, Filterable (Gravimetric, Dried at 180 ^oC) **89**
31. Appendix AA: EPA Method 160.2, Residue, Non-Filterable (Gravimetric, Dried at 103-105 ^oC) **92**
32. Appendix AB: EPA Method 160.4, Residue, Volatile (Gravimetric, Ignition at 550 ^oC) **95**
33. Appendix AC: EPA Method 160.5, Settleable Matter (Volumetric, Imhoff Cone) **98**

34. Appendix AD: EPA Method 375.2, Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II) 100
35. Appendix AE: EPA Method 376.2, Sulfide (Colorimetric, Methylene Blue) 103

List of Tables

Table 2.1. Monthly analysis suite (February, March, May, June, August, September, November, December) 6
Table 2.2. Quarterly analyses suite (January, April, July, October) 6
Table 2.3. Sample handling, documentation, and acceptance policy. Highlighted methods are included in either monthly or quarterly analysis suites. 6

1. Introduction

This Water Quality Monitoring Standard Operating Procedure (SOP) describes laboratory analysis methods for water samples collected in NCPN Units in the state of Utah. All samples collected in these parks are sent to the Utah Department of Water Quality Laboratory, 46 North Medical Drive, Salt Lake City, UT 84113 for chemical analysis. Samples are collected and processed as described in SOP 10 prior to shipping to the laboratory.

2. Methods

Once samples are received by laboratory staff, they are checked to ensure samples are properly labeled, arrive chilled and fall within acceptable pH range . If samples do not meet acceptance criteria they are flagged and the customer is notified. A sample receipt check list used by Utah Department of Health Division of Laboratory Services is presented in Appendix A.

The suite of analyses conducted on samples sent to the laboratory is of two basic types, monthly or quarterly. The correct suite is automatically requested in Field_forms.xls based on the sampling date. The constituents in each analysis suite are presented in Appendix B. The laboratory reporting limits for each constituent are presented in Appendix C.

The following parameters are measured monthly, for every sample sent to the Utah Department of Health Laboratory:

1) Alkalinity, CaCO3, mg/l
2) Bicarbonate, mg/l
3) Calcium, mg/l
4) Carbon Dioxide, mg/l
5) Carbonate Ion, mg/l
6) Chloride, mg/l
7) Tot. Diss. Solids @180C, mg/l
8) Hardness, Ca+Mg, mg/l
9) Hydroxide, mg/l
10) Magnesium, ug/l
11) NO2+NO3 as N, mg/l
12) pH (lab)
13) Phosphorus as P, Total, mg/l
14) Potassium, mg/l
15) Sodium, mg/l
16) Spec. Conductivity, umhos/cm
17) Sulfate, mg/l
18) Total suspended solids, mg/l
19) Turbidity, NTU

The following parameters are measured quarterly, in addition to the above monthly parameters, by the Utah Department of Health Laboratory:

1) Aluminum, ug/l
2) Arsenic, ug/l
3) Barium, ug/l
4) Cadmium, ug/l
5) Chromium, ug/l
6) Copper, ug/l
7) Iron, Diss., ug/l
8) Lead, ug/l
9) Manganese, ug/l
10) Mercury, ug/l
11) Selenium, ug/l
12) Silver, ug/l
13) Zinc, ug/l

Table 2.1. Monthly analysis suite (February, March, May, June, August, September, November, December)

Bottle label	Analysis suite
Total Chemistry	2
Filtered metals	4
Nutrients	6

Table 2.2. Quarterly analyses suite (January, April, July, October)

Bottle label	Analysis suite
Total Chemistry	2
Filtered metals	3
Nutrients	6

Methods of analysis used by the Water Quality Lab are presented in table 2-1. This list contains all laboratory methods including some not currently used on samples submitted by NCPN. The laboratory methods used to analyze samples are presented as Appendices E through AB without alteration.

The Division of Epidemiology and Laboratory Services (DELS) will assist organizations by providing sample containers and instructions for proper sample handling. The following table represents available testing methods, respective containers, preservative and proper handling conditions, total holding times, and sample acceptance criteria.

Table 2.3. Sample handling, documentation, and acceptance policy. Highlighted methods are included in either monthly or quarterly analysis suites.

Testing Methods	Container Type	Volume	Preservative and Sample Handling	Holding Time	Sample Acceptance
Alkalinity: Method SM2320B Appendix E	Plastic[1]	125 ml	Store at 4 ^{o}C	14 Days	
Ammonia: Method EPA 350.1 Appendix F	Plastic[1]	500 mL	H_2SO_4 to pH < 2 Store at 4 ^{o}C	28 days	
Bromide and Chloride: Method EPA 300.0 Appendix G	Plastic[1]	125 mL	Store at 4 ^{o}C	28 days	
Bromate: Method EPA 300.0 Appendix G	Plastic[1]	125 ml	Ethylenediamine Store at 4 ^{o}C	14 days	
Fluoride: Method SM4500C Appendix Q	Plastic[1]	125 mL	None	28 days	

Testing Methods	Container Type	Volume	Preservative and Sample Handling	Holding Time	Sample Acceptance
Lead and Copper: Method EPA 200.8 Appendix R	Plastic[1]	1 L	4 mL HNO_3 to pH <2 add on arrival at the lab	6 months	
Metals, Total: Methods EPA 200.7, EPA 200.8, EPA 200.9, EPA 245.1 Appendices S,R,T,U	Plastic[1]	250 mL	HNO_3 to pH <2	6 months (Metals) 28 days (Mercury)	
Metals, Dissolved: *(Filtered in the Field) Methods EPA 200.7, EPA 200.8, EPA 200.9, EPA 245.1 Appendices S,R,T,U	Plastic[1]	250 mL	HNO_3 to pH <2	6 months (Metals) 28 days (Mercury)	
Mercury: Method EPA 245.1 Appendix U	Plastic[1]	250 mL	HNO_3 to pH <2	28 days	
Nitrate and Nitrite: Method EPA 353.2 Appendix V	Plastic[1]	120 mL	H_2SO_4 to pH <2 Store at 4 ^oC	28 days	pH <2
Nitrite: Method EPA 353.2 Appendix V	Plastic[1]	125 mL	No preservative, Store at 4 ^oC	48 hours	
Nutrients, Total: Methods 365.1 (Phosphate), Appendix W EPA 353.2 (Nitrate/Nitrite) Appendix V	Plastic[1]	500 mL	H_2SO_4 to pH <2 Store at 4 ^oC	28 days	pH <2
Nutrients, Dissolved: *(Filtered in the Field) Methods 365.1 Appendix W (Phosphate), EPA 353.2 (Nitrate/Nitrite) Appendix V	Plastic[1]	250 mL	H_2SO_4 to pH <2 Store at 4 ^oC	28 days	pH <2
pH: Method EPA 150.1 Appendix Y	Plastic[1]	1 L	None	None	
Phosphate: Method EPA 365.1 Appendix W	Plastic[1]	500 mL	H_2SO_4 to pH <2 Store at 4 ^oC	28 days	
Silica: Method EPA 370.1 Appendix X	Plastic[1]	1 L	Store at 4 ^oC	28 days	
Solids, Total Dissolved – TDS: (Filterable residues) Method SM2540C, EPA 160.1 Appendix Z	Plastic[1]	1 L	Store at 4 ^oC	7 days	

Testing Methods	Container Type	Volume	Preservative and Sample Handling	Holding Time	Sample Acceptance
Solids, Total Suspended – TSS: (Non-filterable residues) Method EPA 160.2 Appendix AA	Plastic[1]	1 L	Store at 4 ^{o}C	7 days	
Solids, Total Volatile – TVS: Method EPA 160.4 Appendix AB	Plastic[1]	1 L	Store at 4 ^{o}C	7 days	
Solids, Total Settable – RSS: Method EPA 160.5 Appendix AC	Plastic[1]	1 L	Store at 4 ^{o}C	48 hours	
Sulfate: (Surface Water) Method EPA 375.2 Appendix AD	Plastic[1]	125 mL	Store at 4 ^{o}C	28 days	
Sulfide: (Water) Method EPA 376.2 Appendix AE	Plastic[1]	125 mL	3 Drops Zinc Acetate and NaOH to pH >9	7 days	
Volatile Organic Compounds – VOCs: (Hazardous Soil) Method EPA 8260	Wide Mouth Glass[2,3] with Teflon lined lid	4 oz	Store at 4 ^{o}C	14 days to extract, 14 days to analyze	

[1] All plastic containers will be new with the proper preservative added for the type of sample to be collected.

[2] All glass containers will be washed with soap and water, rinsed with deionized water, rinsed with distilled water, and oven dried.

[3] The above sample containers assume that the sample is 100% solids and uniform particle size. If the sample is less than 100% solid a larger sample volume is required.

3. Laboratory Quality Assurance

The state of Utah Water Quality Laboratory is responsible for laboratory operations and the quality of the results produced notwithstanding poor sample collection technique or improper handling prior to arrival at the lab. The text below is from the UT Division of Epidemiology and Laboratory Services (DELS) operating manual version 7.003 revised June 2004. For consistency, the section numbering has been retained to match the original document.

3.1. Sample receiving

1. **DELS staff will follow practices listed in the Sample Receiving Procedure Manual in order to consistently record sample arrival conditions and that acceptance criteria are met. Briefly, pH (if method required) and sample temperature will be recorded at time of receipt with temperatures measured by an infrared scanning thermometer. Receiving staff will also document the sample handling conditions; specifically, cooling preservation, proper container used with valid date, and received date**

and time for checking method prescribed holding times.

2. **Sample receiving staff will assign a laboratory accession number to each sample received, followed by entry into the DELS Laboratory Information Management System (LIMS) with corresponding sample information and test request(s). A second staff will review data entry in the LIMS to minimize error from data entry processing. Sample receiving staff will also ensure any samples not meeting acceptance criteria are flagged for supervisor/lead chemist follow-up. The Sample Receipt Checklist will be used to record these processes as well as track resulting activities for any acceptance criteria deviations.**

   When there is doubt as to the suitability of a sample for environmental testing, or when a sample does not conform to the acceptance criteria provided, or the required sample information is not listed in sufficient detail, laboratory staff will contact the using organization for further instructions before proceeding, and the discussion result will be recorded on the Sample Receipt Checklist.

3. **Samples will be checked at time of receipt and documentation reviewed for meeting acceptance criteria. For each individual sample, the following is applicable:**

   a. Sample ID that unambiguously matches the identification on each container of the physical sample, e.g. a field identification code and/or site description. Currently this is being recorded as the sample ascension number in combination with a SOURCE code, e.g. the Department of Environmental Quality Department of Water Quality Storet code.

      i. Sample ID for each sample container must be unique when multiple containers are provided for one test, e.g. VOC analysis, where each container is assigned an additional identifier such as A, B, C, etc.
      ii. The sample ID code is to be placed on the sample container as a durable label.
      iii. The sample ID system will include identification for all samples, subsamples, and subsequent extracts and/or digestates.

   b. Client ID for tracking and reporting information.

   c. Location, date, and time of collection.

   d. Sample matrix description, e.g. drinking water, solid, non-aqueous liquid, aqueous, saline/estuarine, chemical waste, biological tissue, is recorded as Sample Type.

e. Regulatory programs requiring compliance, if any. Currently, this is being indicated by the DELS cost code, e.g. CWA (CC 350), SDWA (CC 361), RCRA (CC 365), etc.

f. Requested analysis according to available methods, e.g. EPA 525.1.

g. Use of appropriate containers in satisfactory condition (e.g., no leaks, cracks, etc.) with valid date and proper preservative. Documentation of QC checks on containers supplied by DELS is maintained by Tech Services, in a lot specific manner. Currently, information for chemical preservatives added to sample containers is printed on the container labels with chemical amount and corresponding CAS registry number.

h. Method specified holding times.

i. Adequate sample volume for requested testing.

j. Receipt pH and temperatures compliant with method program ranges.

    i. In the event samples are hand delivered to the laboratory the same day as collection, and do not meet method specified temperature acceptance criteria, then the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

k. If utilized, a complete chain of custody record form shall be maintained and custody seals checked to be intact on each individual container cap at time of receipt.

l. Documentation for field QC samples required by certain using organizations to supplement the basic DELS QAPP QC Samples e.g., trip blanks, field blanks, equipment blanks, duplicates or other field-submitted quality control measures.

4. Non-routine acceptance of a sample may occur under the following conditions:

a. The supervisor and/or lead chemist is in contact with the sampler or customer and is able to complete the requirements listed in section 7.3.

b. If the project manager, in contact with the sampler or customer, is unable to complete the requirements listed in section 7.3, the sample may be accepted when authorized by the customer. However, all test results associated with the non-compliant containers must be flagged indicating that the sample did not meet established acceptance criteria and a report comment must be added describing how the sample was deficient.

c. Any requested adjustments to original documentation must be lined through with a single-line and initialed by the DELS staff making the change.

5. **All samples will be stored in designated storage areas, meeting method specified holding requirements, and delivered to analysts according to the sample delivery and storage policy listed in the Sample Receiving Procedure Manual.**

    a. Samples which require thermal preservation will be stored under refrigeration which is ± 2 °C of the specified preservation temperature, unless other method specific criteria exist. For samples with a specified storage temperature of 4 °C, storage at a temperature above the freezing point of water to 6 °C will be the acceptable range.

        i. Sample fractions, extracts, leachates, and other sample preparation products are to be stored according to method specified holding requirements.

    b. Samples will be stored away from all standards, reagents, food and other potentially contaminating sources. Samples will also be stored in such a manner as to prevent cross contamination.

6. **Upon completion of analysis; samples, digestates, leachates, extracts, or other sample preparation products will be disposed according to method specific requirements found in the safety and/or waste management sections of individual method SOPs.**

4. Laboratory reporting levels (LRL)

The LRL controls false negative error (Appendix C). The probability of falsely reporting a non-detection **(a false negative)** for a sample that contained an analyte at a concentration equal to or greater that the LRL is predicted to be less than or equal to 1 percent. The value of the LRL will be reported with a "less than" remark code for samples in which the analyte was not detected. No Long Term-Method Detection Levels are reported for the state of UT water quality laboratory. However, personal communication with the laboratory director (Sanwat Cahudhuri 801-584-8400) indicated this data is being collected and can be shared in the future.

5. Appendix A: Sample receipt checklist

Utah Department of Health, Division of Laboratory Services

Date: _____

Sample Receipt Checklist

Sample number(s): _____

Received by: _____

Accession reviewed by: _____

All samples documentation/information complete? ☐ Yes ☐ No

If no, sample number(s):

_____

_____

_____

All samples at proper holding temperature? ☐ Yes ☐ No

If no, sample number(s):

_____

_____

_____

All samples received within holding time? ☐ Yes ☐ No

If no, sample number(s):

_____

_____

_____

All samples in proper containers with valid date? ☐ Yes ☐ No

If no, sample number(s):

_____

_____

_____

All sample containers intact? ☐ Yes ☐ No

If no, sample number(s):

_____

_____

_____

Sample pH checks acceptable upon receipt? ☐ N/A ☐ Yes ☐ No

If no, sample number(s):

_____

_____

_____

All chain of custody seals intact? ☐ N/A ☐ Yes ☐ No

If no, sample number(s):

_____

_____

_____

For samples with acceptance criteria not met:

Supervisor and/or lead chemist(s) notified? ☐ Yes ☐ No

Staff name/how contacted (sample #s):_____/_____

Date: _____ Initials: _____

Sample flagged (code #): _____

Date: _____ Initials: _____

Customer(s) notified for re-sampling? ☐ Yes ☐ No

Staff name/who contacted & how:_____/_____

Date: _____ Initials: _____

Result: _____

_____

_____

6. Appendix B: Utah Department of Water Quality Chemical Analysis Suites

6.1. Suite 2 chemistry

T. Alklalinty
Bicarbonate
Carbonate
carbon dioxide
hydroxide
Total Dissolved Solids
Specific Conductivity
Total Suspended Solids
Turbidity
Chloride
Sulfate

6.2. Suite 4 chemistry

Filtered metals suite 4 is a subset of a filtered metals suite 3. Suite 4 contains:

Calcium
Magnesium
Sodium
Potassium
Total Hardness

6.3. Suite 3 chemistry

Filtered metals suite 3 contains the five constituents in a suite 3 and the following 15 heavy metals:

Aluminum
Arsenic
Barium
Boron
Cadmium
Chromium
Copper
Iron
Lead
Manganese
Mercury
Nickel
Selenium
Silver
Zinc

A unfiltered nutrient 6 contains:
NO2+NO3 as N
Total Phosphorus

7. Appendix C: UTDWQ Laboratory Reporting Limits

Type 2	Group No 2	Short Name 3	Param No	Param Desc	Short Name 1	Short Name 2	Report Limit[1]
I	2	ALK	62	T. Alk/CaCO3, mg/l	Tot. Alk.	mg/l	4
I	4	BOD5	15	B.O.D. 5, mg/l	B.O.D. 5	mg/l	3
I	4	CH-A	104	Chlorophyll-A, ug/l	Chlor-A	ug/l	0.7
I	4	CH-B	105	Chlorophyll-B, ug/l	Chlor-B	ug/l	0.1
I	4	CH-C	106	Chlorophyll-C, ug/l	Chlor-C	ug/l	0.1
I	4	CL	53	Chloride, mg/l	Chloride	mg/l	10
I	4	CN	22	Cyanide, mg/l	Cyanide	mg/l	0.01
I	4	CNCL	218	Cyanide (Cl), mg/l	Cyan. (Cl)	mg/l	0.01
I	4	CNH+	267	Cyanide H+, mg/l	Cyanide H+	mg/l	0.01
I	4	COD	21	C.O.D., mg/l	C.O.D.	mg/l	15
I	4	COND	67	Sp. Cond. umhos/cm.	L-Sp. Cond	umhos/cm	2
I	4	DOC	197	D.O.C., mg/l	D.O.C.	mg/l	0.5
I	4	DOC2	797	D.O.C. 2, mg/l	D.O.C. 2	mg/l	0.5
I	4	F	54	Fluoride, mg/l	Fluoride	mg/l	0.05
I	4	O/G	19	Oil/Grease, mg/l	O&G	mg/l	5
I	4	PHEN	23	Phenolics, mg/l	Phenolics	mg/l	0.008
I	4	S	24	Sulfide, mg/l	Sulfide	mg/l	0.01
I	4	SBOD	101	Soluble BOD 5, mg/l	Sol. BOD5	mg/l	3
I	4	SCBO	103	SOL/CARB BOD5, mg/l	SlCarbBOD5	ug/l	3
I	4	TDS	68	TDS @ 180 C, mg/l	TDS @ 180C	mg/l	10
I	4	TKN	18	T.K.N., mg/l	T.K.N.	mg/l	0.5
I	4	TOC	20	T. O. C., mg/l	T.O.C.	mg/l	0.5
I	4	TOCD	798	T. O. C. Dup, mg/l	T.O.C. Dup	mg/l	0.5
I	4	TSS	16	Tot.Sus. Solids, mg/l	T.Sus.Sol	mg/l	4
I	4	TURB	65	Turbidity, NTU	Turbidity	NTU	0.1
I	4	TVS	100	T. Vol. Solids, mg/l	T Vol Sol	mg/l	9
I	9	CR+6	38	Chromium Hex/Cr ug/l	Chrom Hex	ug/l	5
I	9	D-TP	461	Dis. Tot. Phos. mg/l	D-T Phos.	mg/l	0.02
I	9	NH3	31	Ammonia as N, mg/l	Ammonia N	mg/l	0.05
I	9	NO2	57	Nitrite as N	Nitrite, N	mg/l	0.1
I	9	NO2+	17	NO2+NO3, N	NO2+NO3, N	mg/l	0.1
I	9	NO23	221	D-NO2+NO3 as N	DNO2+NO3,N	mg/l	0.1
I	9	NO2D	248	D-Nitrite as N	D-NO2	mg/l	0.1
I	9	NO3	56	Nitrate as N	Nitrate, N	mg/l	0.1
I	9	NO3D	247	D-Nitrate as N	D-NO3 as N	mg/l	0.1
I	9	OPO4	58	Phos. Ortho, mg/l	Ortho Phos	mg/l	0.02
I	9	PO4D	246	OPO4, Diss., mg/l	D-OPO4	mg/l	0.02
I	9	SIO2	59	Silica Diss. mg/l	SIO2	mg/l	5
I	9	SO4	60	Sulfate, mg/l	Sulfate	mg/l	20
I	9	TPO4	61	T. Phosphorus, mg/l	T. Phos.	mg/l	0.02
I	10	BRIC	652	Bromide by IC	Bromide IC	mg/l	0.02
I	10	BRO3	651	Bromate by IC	Bromate	mg/l	0.002
I	10	CLO3	653	Chlorate by IC	ChlorateIC	mg/l	0.02

I	10	CLO4	782	Perchlorate by IC (ug/L)	Perchlorat	ug/L	4
I	10	NO2C	658	Nitrite by IC, N	Nitrite, N	mg/l	0.1
I	10	NO3C	657	Nitrate by IC, N	Nitrate, N	mg/l	0.1
I	10	SO4C	660	Sulfate by IC	Sulfate IC	mg/l	1
M	3	D-AG	47	D-Silver, ug/l	D-Silver	ug/l	2
M	3	D-AL	113	D-Aluminum, ug/l	D-Aluminum	ug/l	30
M	3	D-AS	32	D-Arsenic, ug/l	D-Arsenic	ug/l	1
M	3	D-B	34	D-Boron, ug/l	D-Boron	ug/l	30
M	3	D-BA	33	D-Barium, ug/l	D-Barium	ug/l	100
M	3	D-BE	119	D-Beryllium, ug/l	D-Berylium	ug/l	1
M	3	D-CA	36	D-Calcium, mg/l	D-Calcium	mg/l	1
M	3	D-CD	35	D-Cadmium, ug/l	D-Cadmium	ug/l	1
M	3	D-CO	125	D-Cobalt, ug/l	D-Cobalt	ug/l	30
M	3	D-CR	37	D-Chromium, ug/l	D-Chromium	ug/l	5
M	3	D-CU	39	D-Copper, ug/l	D-Copper	ug/l	12
M	3	D-FE	40	Iron, Diss., ug/l	D-Iron	ug/l	20
M	3	D-HG	223	D-Mercury, ug/l	D-Mercury	ug/l	0.2
M	3	D-K	45	D-Potassium, mg/l	D-Potassum	mg/l	1
M	3	D-MG	42	D-Magnesium, mg/l	D-Magnesum	mg/l	1
M	3	D-MN	43	D-Manganese, ug/l	D-Mangan	ug/l	5
M	3	D-MO	129	D-Molybdenum, ug/l	D-Molybdum	ug/l	1
M	3	D-NA	48	D-Sodium, mg/l	D-Sodium	mg/l	1
M	3	D-NI	44	D-Nickel, ug/l	D-Nickel	ug/l	10
M	3	D-PB	41	D-Lead, ug/l	D-Lead	ug/l	3
M	3	D-SB	194	D-Antimony, ug/l	D-Antimony	ug/l	3
M	3	D-SE	46	D-Selenium, ug/l	D-Selenium	ug/l	1
M	3	D-TL	190	D-Thallium, ug/l	D-Thallium	ug/l	1
M	3	D-V	135	D-Vanadium, ug/l	D-Vanadium	ug/l	30
M	3	D-ZN	49	D-Zinc, ug/l	D-Zinc	ug/l	30
M	3	D-ZR	796	Dissolved Zirconium	D-Zirconiu		10
M	3	DAS	794	Dissolved Arsenic ug/l	As Dissolv	ug/l	0.5
M	3	DAS3	792	Dis. Arsenic +3 Valence ug/l	Dis As+3	ug/l	0.5
M	3	DAS5	793	Dis. Arsenic +5 Valence ug/l	Dis As+5	ug/l	0.5
M	3	DHSE	820	Diss. Selenium by Hydride ug/l	DHSelenium	ug/l	1
M	3	UFMS	789	U238 by ICPMS Filtered ug/L	U238MS Fil	ug/l	1
M	5	T-AG	154	T-Silver, ug/l	T-Silver	ug/l	2
M	5	T-AL	136	T-Aluminum, ug/l	T-Aluminum	ug/l	30
M	5	T-AS	137	T-Arsenic, ug/l	T-Arsenic	ug/l	1
M	5	T-AU	144	T-Gold, ug/l	T-Gold	ug/l	0.02
M	5	T-B	160	T-Boron, ug/l	T-Boron	ug/l	30
M	5	T-BA	138	T-Barium, mg/l	T-Barium	mg/l	0.1
M	5	T-BE	139	T-Beryllium, ug/l	T-Berylium	ug/l	1
M	5	T-CA	263	T-Calcium, mg/l	T-Calcium	mg/l	1
M	5	T-CD	140	T-Cadmium, ug/l	T-Cadmium	ug/l	1
M	5	T-CO	142	T-Cobalt, ug/l	T-Cobalt	ug/l	30
M	5	T-CR	141	T-Chromium, ug/l	T-Chromium	ug/l	5
M	5	T-CU	143	T-Copper, ug/l	T-Copper	ug/l	12
M	5	T-FE	145	T-Iron, mg/l	T-Iron	mg/l	0.02
M	5	T-HG	150	T-Mercury, ug/l	T-Mercury	ug/l	0.2
M	5	T-K	265	T-Potassium, mg/l	T-Potassum	mg/l	1

M	5	T-MG	264	T-Magnesium, mg/l	T-Magnesum	mg/l	1
M	5	T-MN	149	T-Manganese, ug/l	T-Mangan	ug/l	5
M	5	T-MO	151	T-Molybdenum, ug/l	T-Molybdum	ug/l	1
M	5	T-NA	266	T-Sodium, mg/l	T-Sodium	mg/l	1
M	5	T-NI	152	T-Nickel, ug/l	T-Nickel	ug/l	10
M	5	T-PB	147	T-Lead, ug/l	T-Lead	ug/l	3
M	5	T-SB	164	T-Antimony, ug/l	T-Antimony	ug/l	3
M	5	T-SE	153	T-Selenium, ug/l	T-Selenium	ug/l	1
M	5	T-TL	168	T-Thallium, ug/l	T-Thallium	ug/l	1
M	5	T-V	158	T-Vanadium, ug/l	T-Vanadium	ug/l	3
M	5	T-ZN	157	T-Zinc, ug/l	T-Zinc	ug/l	30
M	5	T-ZR	799	Total Zirconium	Zirconium		10
M	5	TAS	791	Total Arsenic ug/l	As Total	ug/l	0.5
M	5	THSE	823	Tot. Selenium by Hydride ug/l	THSelenium	ug/l	1
M	5	UUMS	790	U238 by ICPMS Unfiltered ug/L	U238MS Unf	ug/l	1
M	7	HGHW	236	Mercury (TCLP)	Mercury HW	ppm	0.2
R	6	226R	92	Radium 226, pc/l	Radium 226	pc/l	0.5
R	6	228R	93	Radium 228, pc/l	Radium 228	pc/l	1
R	6	ALPG	89	Alpha, gross, pc/l	Alpha, grs	pc/l	2
R	6	BETG	90	Beta, gross, pc/l	Beta gross	pc/l	10
R	6	D-U	131	D-Uranium, ug/l	D-Uranium	ug/l	1
R	6	DALG	777	Dissolved Gross Alpha	Diss Alpha		2
R	6	DBET	778	Dissolved Gross Beta	Diss Beta		10
R	6	DR26	779	Dissolved Radium 226	Diss Ra226		0.5
R	6	DR28	780	Dissolved Radium 228	Diss Ra228		1
R	6	T-U	155	T-Uranium	T-Uranium	ug/l	1

[1 Reporting limit – This is the …find out exactly from Sanwat Chaudhuri called and e-mailed late October 2006]

8. Appendix D: Analytical Error Control

General description of UT DWQ laboratory analytical error control (Personal communication March 2006 Sanwat Chaudhuri laboratory director)

“With sample analysis batch we (at the lab) analyze calibration standards, blank, duplicate sample every ten analyses, laboratory fortified blank and laboratory fortified samples every 20 samples. All sample analysis data are reviewed by the analyst and supervisor, QA officer or peer.”

9. Appendix E: Alkalinity, Method SM2320B

2320 ALKALINITY*

2320 A. Introduction

1. Discussion

Alkalinity of a water is its acid-neutralizing capacity. It is the sum of all the titratable bases. The measured value may vary significantly with the end-point pH used. Alkalinity is a measure of an aggregate property of water and can be interpreted in terms of specific substances only when the chemical composition of the sample is known.

Alkalinity is significant in many uses and treatments of natural waters and wastewaters. Because the alkalinity of many surface waters is primarily a function of carbonate, bicarbonate, and hydroxide content, it is taken as an indication of the concentration of these constituents. The measured values also may include contributions from borates, phosphates, silicates, or other bases if these are present. Alkalinity in excess of alkaline earth metal concentrations is significant in determining the suitability of a water for irrigation. Alkalinity measurements are used in the interpre-

* Approved by Standard Methods Committee, 1997.

10. Appendix F: EPA Method 350.1, Determination of Ammonia Nitrogen by Semi-automated Colorimetry

National Environmental Methods Index

NEMI Method Summary

Method Number:	350.1
Method Name:	**Descriptive Name:** Ammonia by Automated Colorimetry **Official Name:** Nitrogen, Ammonia (Colorimetric, Automated Phenate)
Revision:	Revision 2.0. August 1993
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)**
Method Download:	**Download full method now (PDF file)** (255251 Byte file)
Brief Method Summary:	Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color is intensified with sodium nitroprusside.

Scope And Application:	This method determines ammonia in drinking, surface, and saline waters; domestic and industrial wastes.
Applicable Conc Range:	0.01 - 2.0 mg/L (ammonia as N)
Interferences:	(A) Calcium and Magnesium: Ions of these metals can cause errors by precipitating during analyses. Use EDTA solution (for river and industrial waters) or potassium tartrate solution (for sea water) to reduce this interference. (B) Turbidity: Sample turbidity should be removed by filtration. (C) Color: Color absorbing in the range of the indophenol blue complex can interfere.
QC Requirements:	None.
Sample Handling:	Cool to 4^oC, add H₂SO₄ until sample pH < 2.
Max Holding Time:	28 days (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Ammonia by Automated Colorimetry (350.1)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Ammonia (7664-41-7)	.01 mg/L	101 % Rec (ML)	7 RSD (ML)	

METHOD 350.1

DETERMINATION OF AMMONIA NITROGEN BY SEMI-AUTOMATED COLORIMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

11. Appendix G: EPA Method 300.0, Determination of Inorganic Anions by Ion Chromatography

National Environmental Methods Index

NEMI Method Summary

Method Number:	300.0
Method Name:	**Descriptive Name:** Inorganic Anions by Ion Chromatography **Official Name:** Determination of Inorganic Anions by Ion Chromatography
Revision:	Revision 2.1, August 1993
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)**
Method Download:	**Download full method now (PDF file)** (2811143 Byte file)
Brief Method Summary:	A small volume of sample, typically 50-100 uL, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor device, and conductivity detector.
Scope And	Part A of the test method covers the determination of common

Application:	inorganic anions in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction), leachates (when no acetic acid is used). Part B of the test method covers the determination of bromate, chlorate, and chlorite in drinking and reagent waters.
Applicable Conc Range:	DL - * (* Upper detection limit is defined as the highest calibration point, as determined by analyst.)
Interferences:	(1) Coeluting species (e.g., low-molecular-weight organic acids). (2) High concentration of anions can effect resolution of peaks and elution times. (3) Fluoride is especially sensitive to coeluting species, and "water dip" (corresponding to the elution of water) at low concentrations.
QC Requirements:	Calibration Blank (CB), Quality Control Sample (QCS), Performance Evaluation Sample (PE), Instrument Performance Check Solution (IPC), Laboratory Duplicate (LD), Field Duplicate (FD), Laboratory Fortified Matrix (LFM), Laboratory Fortified Blank (LFB), Laboratory Reagent Blank (LRB), Linear Calibration Range (LCR)
Sample Handling:	Collect enough volume to ensure a representative sample and to allow for replicates. Either glass or plastic bottles may be used. For Part A Analytes: No preservation is required for bromide, chloride, or fluoride. Refrigerate samples at 4 degrees C for nitrate-N, nitrite-N, orthophosphate-P, and sulfate. Samples being tested for nitrate/nitrite should be adjusted to pH < 2 with concentrated H₂SO4 and refrigerated at 4 degrees C until analysis. Analyze samples being tested for bromide, chloride, fluoride, nitrate/nitrite, and sulfate within 28 days. Analyze samples being tested for nitrate-N, nitrite-N, and orthophosphate-P within 48 hours. For Part B Analytes: Samples analyzed for bromate and chlorate may be held for 28 days without preservation. Chlorite should be analyzed for immediately (within 10 minutes). However, if the sample cannot be analyzed immediately, it may be preserved for up to 14 days if treated with ethylenediamine (ClO_2 should be removed prior to this step) and chilled to 4^oC.
Max Holding Time:	Part A: 48 Hours (NO_2-N, NO_3-N, orthophosphate-P) 28 days (others) Part B: Bromate and Chlorate (28 days) and chlorite (14 days with preservation -- otherwise analyze immediately).

Relative Cost:	$51 to $200

Method *Inorganic Anions by Ion Chromatography (300.0)* has 10 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Phosphate (14265-44-2)	.003 mg/L	99 % Rec (SL)	2 RSD (SL)	10 mg/L
Nitrate (14797-55-8)	.002 mg/L	103 % Rec (SL)	2 RSD (SL)	10 mg/L
Nitrite (14797-65-0)	.004 mg/L	97 % Rec (SL)	2 RSD (SL)	10 mg/L
Sulfate (14808-79-8)	.02 mg/L	99 % Rec (SL)	2 RSD (SL)	20 mg/L
Chlorate (14866-68-3)	.003 mg/L	110 % Rec (SL)	10 RSD (SL)	.1 mg/L
Chlorite (14998-27-7)	.01 mg/L	94 % Rec (SL)	20 RSD (SL)	.05 mg/L
Bromate (15541-45-4)	.02 mg/L	N/A	N/A	
Chloride (16887-00-6)	.02 mg/L	96 % Rec (SL)	2 RSD (SL)	20 mg/L
Fluoride (16984-48-8)	.01 mg/L	91 % Rec (SL)	3 RSD (SL)	2 mg/L
Bromide (24959-67-9)	.01 mg/L	99 % Rec (SL)	2 RSD (SL)	5 mg/L

METHOD 300.0

DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY

John D. Pfaff
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.1
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

12. Appendix H: EPA Method 531.1, Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection High Performance Liquid Chromatography with Post Column Derivitization

NEMI Method Summary

Method Number:	531.1
Method Name:	**Descriptive Name:** Carbamates in Water Using HPLC w/ Post-Column Derivitization **Official Name:** Measurement of N-Methylcarbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection High Performance Liquid Chromatography with Post Column Derivitization
Revision:	Revision 3.1, 1995
Media:	WATER
Subcategory:	Organic
Source:	U.S. EPA Office of Ground Water and Drinking Water Technical Support Center 26 West Martin Luther King Drive Cincinnati, OH 45268-1320 U.S.EPA National Exposure Research Laboratory (NERL) 26 West Martin Luther King Drive Cincinnati, OH, 45268-0001 Phone: 513-569-7931 Fax: 513-569-7757
Citation:	**Methods for the Determination of Organic Compounds in Drinking Water - Supplement III (EPA/600/R-95-131)**
Method Download:	**Download full method now (PDF file)** (2619948 Byte file)
Brief Method Summary:	An aliquot of a filtered water sample is injected into a reverse phase high performance liquid chromatography (HPLC) column. Separation of the analytes is achieved using gradient elution chromatography. After elution from the HPLC column, the analytes are hydrolyzed with sodium hydroxide. The methyl amine formed during hydrolysis is reacted with o-phthalaldehyde (OPA) and 2-mercaptoethanol to form a highly fluorescent derivative (post-column derivitization). The concentrations of analytes eluted from the HPLC column are measured by detecting

	the fluorescence of derivatives with a fluoresence detector.
Scope And Application:	This method determines certain N-methylcarbamoyloximes and N-methylcarbamates (analytes) in ground water and finished drinking water.
Applicable Conc Range:	Not Provided.
Interferences:	(A) Glassware contamination: Thoroughly clean glassware, including baking or solvent rinse. (B) Reagent contamination: Use high purity reagents. (C) Contamination from sample carryover: Rinsing apparatus with water and purging equipment between analyses can minimize contamination. (D) Matrix interferences: Matrix interferences may be mitigated by using a different detector that operates on different chemical/physical principles than the original detector, or by using different chromatography columns. Alternative chromatography columns are described in the method.
QC Requirements:	Initial demonstration of laboratory capability, monitoring internal standard peak area or height in each sample and blank (when internal standard calibration procedures are being employed), followed by analysis of laboratory reagent blanks (LRBs), laboratory fortified samples, laboratory fortified blanks (LFBs), and QC samples. A MDL for each analyte must also be determined.
Sample Handling:	<P>Grab samples must be collected in glass containers following conventional sampling practices without prerinsing the bottle before collection. Samples targeted for the analysis of oxamyl, 3-hydroxycarbofuran, aldicarb sulfoxide, and carbaryl must be preserved at pH 3. Add 1.8 mL of monochloroacetic acid buffer to the 60 mL sample bottle. Dechlorinate with 80 mg of sodium thiosulfate per liter of sample if residual chlorine is present. After collection, seal the sample bottle and shake vigorously for 1 minute. Store samples at 4^oC until analysis.</P>
Max Holding Time:	28 days.
Relative Cost:	$201 to $400

Method *Carbamates in Water Using HPLC w/ Post-Column Derivitization (531.1)* has 11 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Propoxur (114-26-1)	1 ug/L	101 % Rec (SL)	32 RSD (SL)	1 ug/L
Aldicarb (116-06-3)	.22 ug/L	107 % Rec (SL)	7 RSD (SL)	1 ug/L
Carbofuran (1563-66-2)	.52 ug/L	90 % Rec (SL)	12 RSD (SL)	1.5 ug/L
Aldicarb sulfoxide (1646-87-3)	.59 ug/L	47 % Rec (SL)	21 RSD (SL)	2 ug/L
Aldicarb sulfone (1646-88-4)	1 ug/L	83 % Rec (SL)	20 RSD (SL)	2 ug/L
3-Hydroxycarbofuran (16655-82-6)	1.9 ug/L	108 % Rec (SL)	29 RSD (SL)	2 ug/L
Methomyl (16752-77-5)	.29 ug/L	102 % Rec (SL)	18 RSD (SL)	.5 ug/L
Methiocarb (2032-65-7)	1.9 ug/L	82 % Rec (SL)	19 RSD (SL)	4 ug/L
null (null)	.86 ug/L	82 % Rec (SL)	17 RSD (SL)	2 ug/L
Oxamyl (23135-22-0)	.86 ug/L	82 % Rec (SL)	17 RSD (SL)	2 ug/L
Carbaryl (63-25-2)	1.3 ug/L	97 % Rec (SL)	23 RSD (SL)	2 ug/L

13. Appendix I: EPA Method 410.4, Chemical Oxygen Demand (Colorimetric, Automated; Manual)

National Environmental Methods Index

NEMI Method Summary

Method Number:	410.4
Method Name:	**Descriptive Name:** Chemical Oxygen Demand by Colorimetry **Official Name:** Chemical Oxygen Demand (Colorimetric, Automated; Manual)
Revision:	Issued 1978
Media:	WATER
Subcategory:	Organic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (27806 Byte file)
Brief Method Summary:	Sample, blanks, and standards in sealed tubes are heated in an oven or block digestor in the presence of dichromate at 150^oC. After two hours, the tubes are removed from the oven or digestor, cooled, and measured spectrophotometrically at 600 nm.

Scope And Application:	This method covers the determination of COD in surface waters; and domestic and industrial wastes.
Applicable Conc Range:	3 - 900 mg/L (automated); 20 - 900 mg/L (manual)
Interferences:	(1) Chloride ion, and other halides that precipitate silver. (2) Incomplete oxidation of nitrogen-containing organic matter. (3) Nitrite ion. (4) Reduced inorganic species (e.g., ferrous iron, sulfide, manganous manganese).
QC Requirements:	not included
Sample Handling:	Collect 50 mL samples in either plastic or glass bottles. Preserve samples by adjusting the pH to < 2 with H₂SO₄ and refrigerate at 4^oC until analysis. Analyze samples within 28 days.
Max Holding Time:	28 days (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Chemical Oxygen Demand by Colorimetry (410.4)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
COD (E-10117)	3 mg/L	98 % Rec (ML)	22 RSD (ML)	20 mg/L

METHOD #: 410.4	Pending Approval for Sect. 304(h), CWA (Issued 1978)
TITLE:	Chemical Oxygen Demand (Colorimetric, Automated; Manual)
ANALYTE:	COD Chemical Oxygen Demand
INSTRUMENTATION:	Spectrophotometer, Autoanalyzer
STORET No.	00340

1.0 Scope and Application

1.1 This method covers the determination of COD in surface waters, domestic and industrial wastes.
1.2 The applicable range of the automated method is 3-900 mg/L and the range of the manual method is 20 to 900 mg/L.

2.0 Summary of Method

2.1 Sample, blanks and standards in sealed tubes are heated in an oven or block digestor in the presence of dichromate at 150°C. After two hours, the tubes are removed from the oven or digestor, cooled and measured spectrophotometrically at 600 nm.

3.0 Sample Handling and Preservation

3.1 Collect the samples in glass bottles if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.
3.2 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4°C until analysis.

4.0 Interferences

4.1 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion tubes to complex the chlorides.

5.0 Apparatus

5.1 Drying oven or block digestor, 150°C
5.2 Corning culture tubes, 16 x 100 mm or 25 x 150 mm with Teflon lined screw cap
5.3 Spectrophotometer or Technicon AutoAnalyzer
5.4 Muffle furnace, 500°C.

14. Appendix J: EPA Method 323.3, Chloride

Method not posted online

15. Appendix K: EPA Method 515.1, Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector

National Environmental Methods Index

NEMI Method Summary

Method Number:	515.1
Method Name:	**Descriptive Name:** Chlorinated Acids in Water by GC/ECD **Official Name:** Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector
Revision:	Revision 4.1, 1995
Media:	WATER
Subcategory:	Organic
Source:	U.S. EPA Office of Ground Water and Drinking Water Technical Support Center 26 West Martin Luther King Drive Cincinnati, OH 45268-1320 U.S.EPA National Exposure Research Laboratory (NERL) 26 West Martin Luther King Drive Cincinnati, OH, 45268-0001 Phone: 513-569-7931 Fax: 513-569-7757
Citation:	**Methods for the Determination of Organic Compounds in Drinking Water - Supplement II (EPA/600/R-92-129)**
Method Download:	**Download full method now (PDF file)** (133991 Byte file)
Brief Method Summary:	A measured volume of sample of approximately 1 L is adjusted to pH 12 with 6 N sodium hydroxide and shaken for one hour to hydrolyze derivatives. Extraneous organic material is removed by

	a solvent wash. The sample is acidified, and the chlorinated acids are extracted with ethyl ether by shaking in a separatory funnel or mechanical tumbling in a bottle. The acids are converted to their methyl esters using diazomethane as the derivatizing agent or alternatively trimethylsilyldiazomethane (TMSD). Excess derivatizing reagent is removed, and the esters are determined by capillary column/GC using an electron capture detector (ECD). Note: Method 515.1 calls for a solvent wash step after hydrolysis and prior to acidification and derivatization. Dacthal, the parent di-ester, does not hydrolyze, and is extracted in the wash step, leaving the mono- and di-acid metabolites to be measured.
Scope And Application:	This is a gas chromatographic (GC) method applicable to the determination of certain chlorinated acids in ground water and finished drinking water. This method is also applicable to the determination of salts and esters of analyte acids. This method should be used by or under the supervision of analysts experienced in the use of GC and in the interpretation of gas chromatograms.
Applicable Conc Range:	Ranges differ for each analyte depending on matrix and instrumentation.
Interferences:	(1) Contaminants in solvents, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or elevated baselines in gas chromatograms. (2) Acid forms of the analytes are strong organic acids which react readily with alkaline substances and can be lost during sample preparation. (3) Organic acids and phenols, especially chlorinated compounds. (4) Interferences by phthalate esters can pose a major problem in pesticide analysis when using the ECD. These compounds generally appear in the chromatogram as large peaks. (5) Matrix interferences may be caused by contaminants that are coextracted from the sample. (6) Rinsing apparatus with MTBE between analyses can minimize contamination from sample carryover. (7) Use the same solvent for each analysis to reduce the interferences from variable solvents.
QC Requirements:	Laboratory Duplicate (LD), Field Duplicate (FD), Laboratory Reagent Blank (LRB), Field Reagent Blank (FRB), Laboratory Performance Check Solution (LPC), Laboratory Fortified Blank (LFB), Laboratory Fortified Matrix (LFM), Quality Control Sample (QCS), Performance Evaluation Sample (PE), Surrogate Analyte, Internal Standard

Sample Handling:	Grab samples must be collected in glass containers which must not be prerinsed with sample prior to collection. Prior to collection, add 1 mL of 10 mg/L mercuric chloride to the bottle to produce a concentration of 10 mg/L. If residual chlorine is present, also add 80 mg/L of sodium thiosulfate prior to collection. After collection, shake sample containing preservative(s) vigorously for one minute. Keep samples iced or refrigerated at 4^oC and away from light until extraction.
Max Holding Time:	Analytes present in samples (prior to extraction) are stable for 14 days when properly stored. However, analyte stability may be affected by sample matrix. Extracts should be stored at 4^oC and away from light. Most extracted analytes are stable for 28 days.
Relative Cost:	$201 to $400

Method *Chlorinated Acids in Water by GC/ECD (515.1)* has 19 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
4-Nitrophenol (100-02-7)	.14 ug/L	148 % Rec (SL)	15 RSD (SL)	.2 ug/L
Dichlorprop (120-36-5)	.22 ug/L	110 % Rec (SL)	16 RSD (SL)	.4 ug/L
Chloramben (133-90-4)	.097 ug/L	111 % Rec (SL)	13 RSD (SL)	.4 ug/L
Dicamba (1918-00-9)	.085 ug/L	135 % Rec (SL)	24 RSD (SL)	.4 ug/L
Picloram (1918-02-1)	.15 ug/L	91 % Rec (SL)	17 RSD (SL)	.6 ug/L
DCPA Diacid	.067 ug/L	74 % Rec	13 RSD	.2 ug/L

(2136-79-0)		(SL)	(SL)	
Bentazon (25057-89-0)	.11 ug/L	92 % Rec (SL)	19 RSD (SL)	.2 ug/L
Acifluorfen (50594-66-6)	.026 ug/L	88 % Rec (SL)	23 RSD (SL)	.04 ug/L
3,5-Dichlorobenzoic acid (51-36-5)	.065 ug/L	102 % Rec (SL)	16 RSD (SL)	.6 ug/L
Dalapon (75-99-0)	1.3 ug/L	100 % Rec (SL)	20 RSD (SL)	10 ug/L
5-Hydroxydicamba (7600-50-2)	.017 ug/L	49 % Rec (SL)	28 RSD (SL)	.04 ug/L
Pentachlorophenol (87-86-5)	.032 ug/L	130 % Rec (SL)	31 RSD (SL)	.04 ug/L
Dinoseb (88-85-7)	.33 ug/L	42 % Rec (SL)	34 RSD (SL)	.4 ug/L
Silvex (93-72-1)	.21 ug/L	134 % Rec (SL)	23 RSD (SL)	.2 ug/L
2,4,5-T (93-76-5)	.045 ug/L	87 % Rec (SL)	21 RSD (SL)	.08 ug/L
2,4-D (94-75-7)	.078 ug/L	90 % Rec (SL)	14 RSD (SL)	.2 ug/L
2,4-DB (94-82-6)	1.7 ug/L	87 % Rec (SL)	15 RSD (SL)	4 ug/L
DCPA mono-acid degradate (887-54-7)	N/A	N/A	N/A	
Dacthal Acid Metabolites	N/A	N/A	N/A	

(N-55765)

METHOD 515.1. DETERMINATION OF CHLORINATED ACIDS IN WATER BY GAS CHROMATOGRAPHY WITH AN ELECTRON CAPTURE DETECTOR

Revision 4.1

Edited by J.W. Munch (1995)

R.C. Dressman and J.J. Lichtenberg - EPA 600/4-81-053, Revision 1.0 (1981)

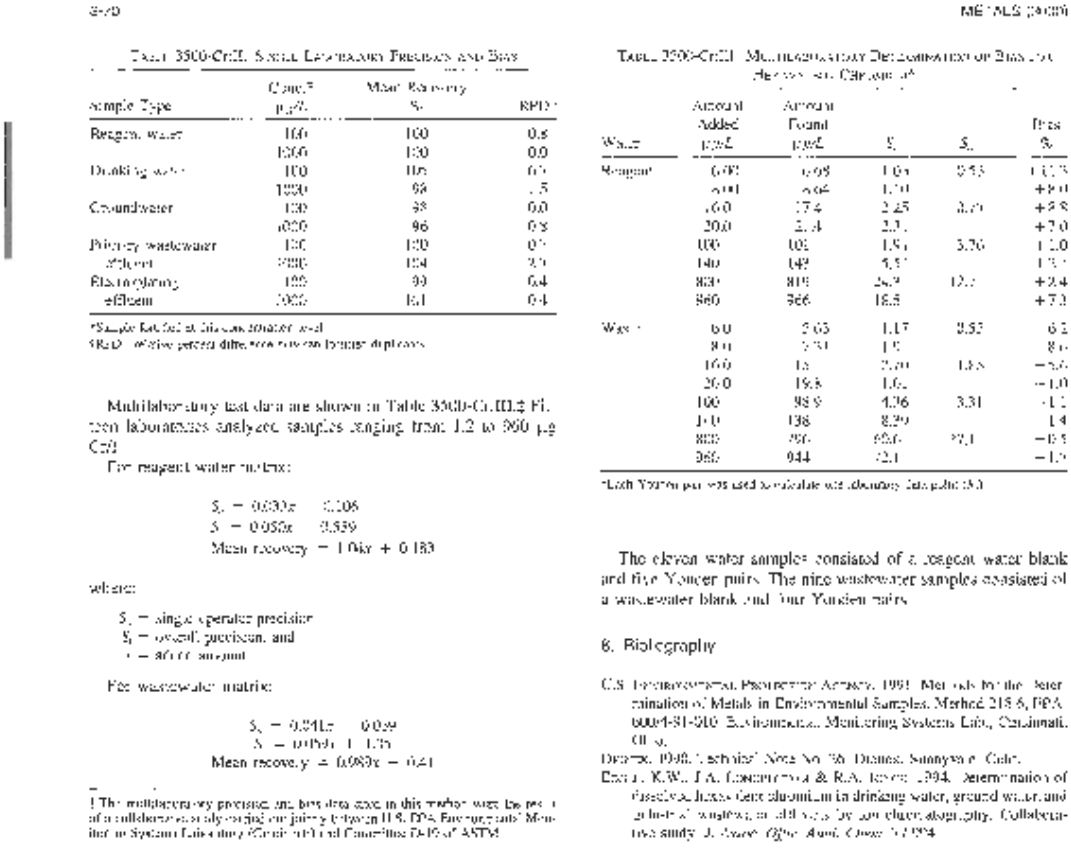
J.W. Hodgeson - Method 515, Revision 2.0 (1986)

D. J. Munch (USEPA, Office of Water) and T. Engel (Battelle Columbus Laboratories) - National Pesticide Survey Method 3, Revision 3.0 (1987)

R.L. Graves - Method 515.1, Revision 4.0 (1989)

NATIONAL EXPOSURE RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

16. Appendix L: Standards Methods Number SM3500 CD Chromium-VI



3-70 METALS (3500)

TABLE 3500-Cr.II. SINGLE-LABORATORY PRECISION AND BIAS

Sample Type	Conc.[a] µg/L	Mean Recovery %	RPD[b]
Reagent water	100	100	0.8
	1000	100	0.0
Drinking water	100	105	0.0
	1000	99	1.5
Groundwater	100	98	0.0
	1000	96	0.8
Primary wastewater effluent	100	100	0.0
	1000	104	3.9
Biotreated wastewater effluent	100	99	0.4
	1000	101	0.4

[a] Sample fortified at this concentration level.
[b] RPD = relative percent difference between laboratory duplicates.

Multilaboratory test data are shown in Table 3500-Cr.III.[1] Fifteen laboratories analyzed samples ranging from 1.2 to 900 µg Cr/l.

For reagent water matrix:

$$S_r = 0.030x + 0.106$$
$$S = 0.050x + 0.539$$
$$\text{Mean recovery} = 1.00x + 0.183$$

where:

S_r = single operator precision,
S = overall precision, and
x = added amount.

For wastewater matrix:

$$S_r = 0.241x + 0.059$$
$$S = 0.090x + 1.139$$
$$\text{Mean recovery} = 0.980x - 0.41$$

[1] The multilaboratory precision and bias data cited in this method were the result of a collaborative study carried out jointly between U.S. EPA Environmental Monitoring Systems Laboratory (Cincinnati, OH) and Committee D19 of ASTM.

TABLE 3500-Cr.III. MULTILABORATORY DETERMINATION OF BIAS AND PRECISION OF CHROMIUM[a]

Water	Amount Added µg/L	Amount Found µg/L	S_r	S	Bias %
Reagent	0.99	0.95	1.04	0.93	−4.0
	9.91	9.64	1.70		−2.7
	49.6	47.4	2.45	3.41	−4.4
	20.0	21.4	2.31		+7.0
	100	101	1.91	3.26	+1.0
	140	142	4.57		+1.4
	830	819	24.9	12.2	−1.3
	860	866	18.8		+0.7
Wastewater	6.0	5.65	1.17	0.52	−5.8
	8.0	7.51	1.5		−6.1
	16.0	14	2.70	1.35	−12.5
	20.0	19.8	1.62		−1.0
	100	98.9	4.36	3.31	−1.1
	140	138	8.29		−1.4
	800	790	66.6	22.1	−1.3
	960	944	42.1		−1.7

[a] Each Youden pair was used to calculate one laboratory data point (N = 15).

The eleven water samples consisted of a reagent water blank and five Youden pairs. The nine wastewater samples consisted of a wastewater blank and four Youden pairs.

6. Bibliography

U.S. ENVIRONMENTAL PROTECTION AGENCY. 1991. Methods for the Determination of Metals in Environmental Samples. Method 218.6, EPA 600/4-91-010. Environmental Monitoring Systems Lab., Cincinnati, OH.

DIONEX. 1998. Technical Note No. 36. Dionex, Sunnyvale, Calif.

EATON, K.W., J.A. CAMPBELL & R.A. JONES. 1994. Determination of dissolved hexavalent chromium in drinking water, ground water and industrial wastewater, at sub-µg/L levels by ion chromatography. Collaborative study. *J. Assoc. Offic. Anal. Chem.* 77:1294.

17. Appendix M: EPA Method 110.2, Color (Colorimetric-Platinum-Cobalt)

National Environmental Methods Index

NEMI Method Summary

Method Number:	110.2
Method Name:	**Descriptive Name:** Color by Spectrophotometry **Official Name:** Color (Colorimetric-Platinum-Cobalt)
Revision:	Issued 1971
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (21344 Byte file)
Brief Method Summary:	Color is measured by visual comparison of the sample with platinum-cobalt standards. One unit of color is that produced by 1 mg/L platinum in the form of the chloroplatinate ion.
Scope And Application:	The Platinum-Cobalt method is useful for measuring color of water derived from naturally occurring materials, i.e., vegetable residues such as leaves, barks, roots, humus, and peat materials.

	The method is not applicable to color measurement on waters containing highly colored industrial wastes.
Applicable Conc Range:	5 to 70 chloroplatinate units
Interferences:	(A) Turbidity: Since very slight amounts of turbidity interfere with the determination, samples showing visible turbidity should be clarified by centrifugation. (B) pH: Method is pH dependent.
QC Requirements:	None.
Sample Handling:	Representative samples shall be taken in scrupulously clean glassware. Refrigerate at 4^oC.
Max Holding Time:	48 hours (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Color by Spectrophotometry (110.2)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Color (E-11712)	5 Pt-Co Color	N/A	N/A	

METHOD #: 110.2	Approved for NPDES (Issued 1971)
TITLE:	Color (Colorimetric-Platinum-Cobalt)
ANALYTE:	Color
INSTRUMENTATION:	Spectrophotometer
STORET No.	TRUE COLOR 00080 APPARENT COLOR 00081

1.0 Scope and Application

1.1 The Platinum-Cobalt method is useful for measuring color of water derived from naturally occurring materials, i.e., vegetable residues such as leaves, barks, roots, humus and peat materials. The method is not applicable to color measurement on waters containing highly colored industrial wastes. See ADMI color method in this manual.
NOTE 1: The Spectrophotometric and Tristimulus methods are useful for detecting specific color problems. The use of these methods, however, is laborious and unless determination of the hue, purity, and luminance is desired, they are of limited value.

2.0 Summary of Method

2.1 Color is measured by visual comparison of the sample with platinum-cobalt standards. One unit of color is that produced by 1 mg/L platinum in the form of the chloroplatinate ion.

3.0 Interferences

3.1 Since very slight amounts of turbidity interfere with the determination, samples showing visible turbidity should be clarified by centrifugation.
3.2 Method is pH dependent.

4.0 Sample Handling and Preservation

4.1 Representative samples shall be taken in scrupulously clean glassware.
4.2 Since biological activity may change the color characteristics of a sample, the determination should be made as soon as possible. Refrigeration at 4°C is recommended.

5.0 Apparatus

5.1 Nessler tubes: Matched, tall form, 50 mL capacity.

6.0 Reagents

18. Appendix N: EPA Method 120.1, Conductance (Specific Conductance, umhos at 25°C)

National Environmental Methods Index

NEMI Method Summary

Method Number:	120.1
Method Name:	**Descriptive Name:** Conductance by Conductivity Meter **Official Name:** Conductance (Specific Conductance, umhos at 25^oC)
Revision:	Issued 1971; Editorial Revision 1982
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (15284 Byte file)
Brief Method Summary:	The specific conductance of a sample is measured by use of a self-contained conductivity meter, Wheatstone bridge-type, or equivalent. Samples are preferable analyzed at 25^oC. If not, temperature corrections are made

	and results reported at 25^oC.
Scope And Application:	This method determines conductance of drinking, surface, and saline water, domestic and industrial wastes and acid rain (atmospheric deposition).
Applicable Conc Range:	Not Available.
Interferences:	Turbidity: Since very slight amounts of turbidity interfere with the determination, samples must be filtered before analysis.
QC Requirements:	None.
Sample Handling:	Analyses can be performed either in the field or laboratory. If analysis is not completed within 24 hours of sample collection, sample should be filtered through a 0.45 micron filter and stored at 4^oC. Filter and apparatus must be washed with high quality distilled water and pre-rinsed with sample before use.
Max Holding Time:	24 hours, unpreserved. 28 days, preserved (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Conductance by Conductivity Meter (120.1)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Specific conductivity (E-10184)	N/A	100 % Rec (ML)	4 RSD (ML)	100 umhos/cm

METHOD #: 120.1	Approved for NPDES (Editorial Revision 1982)
TITLE:	Conductance (Specific Conductance, umhos at 25°C)
ANALYTE:	Conductance
INSTRUMENTATION:	Conductivity Meter
STORET No.	00095

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline water, domestic and industrial wastes and acid rain (atmospheric deposition).

2.0 Summary of Method

2.1 The specific conductance of a sample is measured by use of a self-contained conductivity meter, Wheatstone bridge-type, or equivalent.
2.2 Samples are preferable analyzed at 25°C. If not, temperature corrections are made and results reported at 25°C.

3.0 Comments

3.1 Instrument must be standardized with KCl solution before daily use.
3.2 Conductivity cell must be kept clean.
3.3 Field measurements with comparable instruments are reliable.
3.4 Temperature variations and corrections represent the largest source of potential error.

4.0 Sample Handling and Preservation

4.1 Analyses can be performed either in the field or laboratory.
4.2 If analysis is not completed within 24 hours of sample collection, sample should be filtered through a 0.45 micron filter and stored at 4°C. Filter and apparatus must be washed with high quality distilled water and pre-rinsed with sample before use.

5.0 Apparatus

5.1 Conductivity bridge, range 1 to 1000 umho per centimeter.
5.2 Conductivity cell, cell constant 1.0 or micro dipping type cell with 1.0 constant. YSI #3403 or equivalent.
5.3 Thermometer

6.0 Reagents

19. Appendix O: EPA Method 1623, Cryptosporidium and Giardia in Water by Filtration/IMS/FA

National Environmental Methods Index

NEMI Method Summary

Method Number:	1623
Method Name:	**Descriptive Name:** Cryptosporidium and giardia by filtration/IMS/FA microscopy **Official Name:** Cryptosporidium and Giardia in Water by Filtration/IMS/FA
Revision:	1-Apr
Media:	WATER
Subcategory:	Microbiological
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Cryptosporidium and Giardia in Water by Filtration/IMS/FA (EPA-821-R-01-025)**
Method Download:	**Download full method now (PDF file)** (1232957 Byte file)
Brief Method Summary:	The method is a performance based method--if equivalent or better performance can be shown, modifications of the method may be used. A 10-L or greater sample is put through a 1-micron membrane filter, the materials on the filter are eluted with an aqueous buffered salt and detergent solution, and the eluate is concentrated. Magnetic beads conjugated to an antibody are

	added to the concentrate, and the magnetized oocysts and cysts are separated from the extraneous materials using a magnet. The magnetic bead complex is detached from the oocysts and cysts and the purified sample is applied to a well slide. The oocysts and cysts are stained on well slides with fluorescently labeled monoclonal antibodies and 4',6-diamidino-2-phenylindole (DAPI). The stained sample is examined using fluorescence and differential interference contrast (DIC) microscopy.
Scope And Application:	Ambient, compliance monitoring: ambient surface waters. EPA Fed Reg (Aug 2001) for Giardia and Cryptosporidium, ambient only: fresh, marine, or estuarine surface waters; applicability must be demonstrated for other matrices. USEPA. 1996 (May 14). National primary drinking water regulations: monitoring requirements for public drinking water supplies - Cryptosporidium, Giardia, viruses, disinfection byproducts, water treatment plant data and other information requirements. Fed. Reg. 61(94):24354-24388. USEPA. 2001 (August 30). Guidelines establishing test procedures for the analysis of pollutants; Analytical methods for biological pollutants in ambient water; proposed rule. Fed. Reg. 66(169)45811-45829. Clean Water Act section 401. 40 CFR 136.1(c). (state certification, licenses) for compliance monitoring in programs 303(c), 304(a), and 501(a). 136.3 Identification of test procedures.
Applicable Conc Range:	40 to 500 oocysts/10 L and 40 to 500 cysts/10 L
Interferences:	Inorganic and organic debris, clays and algae, and iron and alum coagulants may interfere with concentration, separation, and examination of the sample. Organisms that autofluoresce may contribute to false positives. Solvents, reagents, labware, and other hardware may yield artifacts that interfere with microscopic examinations. Freezing samples, filters, eluates, concentrates, or slides may interfere with detection of oocysts and cysts.
QC Requirements:	Lab must operate a formal QA program. Method blank (negative control sample) should be run initially and a minimum of once per week or after changes in source of reagent water. Initial precision and recovery test (IPR). Analysis of spiked samples to evaluate and document data quality (matrix spike, MS). Analysis of standards and blanks as tests of continued performance: ongoing precision and recovery tests (OPR) and positive and negative staining control. All equipment should be thoroughly cleaned after use.
Sample	Sample preservation: Samples are chilled to 8^oC; must not freeze. Techniques for collection: Collect minimum 10 L

Handling:	of sample in plastic carboy or filter in the field. Must be shipped to the lab the same day as collected; see Standard Methods, 20th ed., L. Clesceri, A. Greenberg, and A. Eaton (editors). APHA: Washington, DC. 1998. Section 9060A and 9213B.2b Sample processing time 16-20 h. USEPA. 2001. Implementation and Results of the Information Collection Rule Supplemental Surveys. EPA-815-R-01-003. Office of Water, Office of Ground Water and Drinking Water, Standards and Risk Management Division, Washington, DC.
Max Holding Time:	96 hours between sample collection/filtration and initiation of elution. Sample to slide preparation must be completed in 1 working day. 72 hours between application of the purified sample to the slide to staining. 7 days between sample staining and examination.
Relative Cost:	Greater than $400

Method *Cryptosporidium and giardia by filtration/IMS/FA microscopy (1623)* has 2 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Giardia (137259-49-5)	40 oocysts/10 L	53 % Rec (ML)	43 RSD (ML)	138 oocysts/10 L
Cryptosporidium parvum (ITIS - 553105)	40 oocysts/10 L	43 % Rec (ML)	47 RSD (ML)	131 oocysts/10

United States Environmental Protection Agency
Office of Water 4603
EPA-821-R-01-025 April 2001

EPA

Method 1623: *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA



20. Appendix P: EPA Method 335.4, Determination of Total Cyanide by Semi-Automated Colorimetry

National Environmental Methods Index

NEMI Method Summary

Method Number:	335.4
Method Name:	**Descriptive Name:** Cyanide, total, by Colorimetry **Official Name:** Determination of Total Cyanide by Semi-Automated Colorimetry
Revision:	Revision 1.0, August 1993
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)**
Method Download:	**Download full method now (PDF file)** (202822 Byte file)
Brief Method Summary:	The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and is absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion is determined using an automated colorimeter with pyridine-barbituric acid chemistry.
Scope And	This method is determines cyanide in drinking, surface and saline

Application:	waters; and domestic and industrial wastes.
Applicable Conc Range:	0.005 to 0.5 mg/L
Interferences:	(1) Oxidizing agents can destroy cyanides during storage. (2) Sulfide can complex with cyanide in sample or distillate. (3) Fatty acids cause interference during distillation. (4) Carbonate causes interference during distillation. (5) Aldehydes cause interference during distillation. (6) Nitrate/Nitrate causes interference during distillation (they can generate cyanides when combined with certain organic compounds). (7) Sulfur-containing compound causes interference during distillation by forming free sulfide that is captured in distillate.
QC Requirements:	The minimum requirements consist of an initial demonstration of laboratory capability, the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance.
Sample Handling:	Test for and remove sulfides using lead acetate paper and cadmium carbonate, respectively. Test sample for the presence of oxidizing agents using KI-starch paper. If oxidizers are detected, add ascorbic acid until a drop test is negative. Preserve samples by adjusting to a pH > 12 with NaOH. Refrigerate samples at 4 degrees C until analysis. Analyze samples as soon as possible.
Max Holding Time:	14 days
Relative Cost:	Less than $50

Method *Cyanide, total, by Colorimetry (335.4)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Cyanides (57-12-5)	.005 mg/L	N/A	N/A	

METHOD 335.4

DETERMINATION OF TOTAL CYANIDE BY SEMI-AUTOMATED COLORIMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 1.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

21. Appendix Q: Standard Methods Number SM4500C: Fluoride

4500-F⁻ FLUORIDE*

4500-F⁻ A. Introduction

A fluoride concentration of approximately 1.0 mg/L in drinking water effectively reduces dental caries without harmful effects on health. Fluoride may occur naturally in water or it may be added in controlled amounts. Some fluorosis may occur when the fluoride level exceeds the recommended limits. In rare instances the naturally occurring fluoride concentration may approach 10 mg/L; such waters should be defluoridated.

Accurate determination of fluoride has increased in importance with the growth of the practice of fluoridation of water supplies as a public health measure. Maintenance of an optimal fluoride concentration is essential in maintaining effectiveness and safety of the fluoridation procedure.

1. Preliminary Treatment

Among the methods suggested for determining fluoride ion (F^-) in water, the electrode and colorimetric methods are the most satisfactory. Because both methods are subject to errors due to interfering ions (Table 4500-F⁻:I), it may be necessary to distill the sample as directed in Section 4500-F⁻.B before making fluoride determination. When interfering ions are not present in excess of the tolerance of the method, the fluoride determination may be made directly without distillation.

2. Selection of Method

The electrode methods (C and D) are suitable for fluoride concentrations from 0.1 to more than 10 mg/L. Adding the prescribed buffer frees the electrode method from most interferences that adversely affect the SPADNS colorimetric method and necessitate preliminary distillation. Some substances in industrial wastes, such as aluminum, may be sufficiently concentrated to present problems in electrode measurements and will not be measured without a preliminary distillation. Fluoride measurements can be made with an ion-selective electrode and either an expanded-scale pH meter or a specific ion meter, usually without distillation. In the time necessary for electrode equilibration,

The SPADNS method (D) has a linear analytical range of 0 to 1.40 mg F^-/L. Use of a nonlinear calibration can extend the range to 1.5 mg F^-/L. Color development is virtually instantaneous. Color

Table 4500-F⁻:I. Concentrations of Some Substances Causing Less than 10% Error in 1.0 mg F^-/L in Fluoride Methods

Substance	Method C (Electrode)[a]		Method D (SPADNS)	
	Conc. mg/L	Type of Error[b]	Conc. mg/L	Type of Error[b]
Alkalinity ($CaCO_3$)	5 000	+	5 000	-
Aluminum (Al^{3+})	3.0	−	0.1	−
Chloride (Cl^-)	30 000		7 000	+
Iron (Fe^{3+})	1 000			Reagent reacts with ascorbic
Color & turbidity				Reagent reacts with ascorbic
Iron	200	-	10	−
Ethylenediaminetetraacetate ($[NaO_2CCH_2]_2NCH_2CH_2N[CH_2CO_2Na]_2$)	20 000		1.0	+
Phosphate (PO_4^{3-})	30 000		15	+
Sulfate (SO_4^{2-})	20 000		300	

[a] Buffer added according to procedure.
[b] + denotes positive error
− denotes negative error
Blank denotes no measurable error.
[c] No interference noted. Titration reagent reacts with [illegible]

determinations are made photometrically, using either a filter photometer or a spectrophotometer. A curve developed from standards is used for determining the fluoride concentration of a sample.

Fluoride also may be determined by the automated complexone method, Method F.

Ion chromatography (Section 4110) is an acceptable method if weaker eluents are used to separate fluoride from interfering peaks or fluoride can be determined by capillary ion electrophoresis (Section 4140).

The flow injection method (G) is a convenient automated technique for analyzing large numbers of samples.

* Approved by Standard Methods Committee, 1997.

22. Appendix R: EPA Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry

National Environmental Methods Index

NEMI Method Summary

Method Number:	200.8
Method Name:	**Descriptive Name:** Metals in Waters by ICP/MS **Official Name:** Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry
Revision:	Revision 5.4, 1994
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111)**
Method Download:	**Download full method now (PDF file)** (371133 Byte file)
Brief Method Summary:	Except for the determination of dissolved analytes aqueous samples are acid preserved prior to sample processing. For the analysis of dissolved analytes, an acidified portion the filtrate is analyzed directly. For the determination of total-recoverable

	analytes in aqueous samples containing particulate material as well as solid type samples, samples are subjected to acid pretreatment with nitric and hydrochloric acids and gentle refluxing prior to analysis. The method involves multi-element determination by inductively coupled plasma-mass spectrometry. Sample solutions are pneumatically nebulized into a radio-frequency plasma where ionization occurs. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadruple mass spectrometer. Separated ions are detected by an electron multiplier or Faraday detector and the ion information processed by a data handling system. Isobaric and polyatomic interferences relating to the technique must be recognized and corrected. Instrument drift and matrix effects must be corrected with the use of internal standards.
Scope And Application:	This method determines 21 elements as dissolved elements in ground waters, surface waters, and drinking water, and total recoverable elements in these waters as well as wastewaters, sludges, and soils samples. Total-recoverable determination data should be reported as "total" (dissolved + suspended fractions) metal data.
Applicable Conc Range:	The analytical range depends on the type of detector utilized (electron multiplier or Faraday cup) and extends from the laboratory determined MDL to the upper limit of the linear dynamic range.
Interferences:	(1) Isobaric elemental interferences caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and cannot be resolved by the mass spectrometer in use. (2) Signals from relatively abundant isotopes can coalesce at the wings of relatively less abundant isotopes leading to loss of resolution and poorer quantitation. (3) Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom with the same nominal charge-to-mass ratio of the isotope of interest. (4) Physical interferences that hinder transport of the sample into the plasma (e.g., viscosity effects, high levels of solids). (5) Memory interferences (carry-over) of isotopes from previous sample runs.
QC Requirements:	Calibration Blanks (CBs), Laboratory Reagent Blanks (LRBs), Laboratory Duplicates (LDs), Laboratory Fortified Sample Matrix (LFM), Quality Control Samples (QCSs)
Sample	For dissolved elements, filter aqueous sample through a 0.45-um

Handling:	pore membrane filter. Adjust the pH of the filtrate to < 2 with HNO₃. For total recoverable elements, adjust the pH of the aqueous sample to < 2 with HNO₃.
Max Holding Time:	6 months (All metals except Hg - 28 days)
Relative Cost:	$201 to $400

Method *Metals in Waters by ICP/MS (200.8)* has 21 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Aluminum (7429-90-5)	1 ug/L	125 % Rec (ML)	17 RSD (ML)	8 ug/L
Lead (7439-92-1)	.6 ug/L	100 % Rec (ML)	41 RSD (ML)	4 ug/L
Manganese (7439-96-5)	.1 ug/L	108 % Rec (ML)	11 RSD (ML)	.8 ug/L
Mercury (7439-97-6)	.2 ug/L	86 % Rec (SL)	13 RSD (SL)	1 ug/L
Molybdenum (7439-98-7)	.3 ug/L	94 % Rec (ML)	6 RSD (ML)	2.8 ug/L
Nickel (7440-02-0)	.5 ug/L	101 % Rec (ML)	13 RSD (ML)	4 ug/L
Silver (7440-22-4)	.1 ug/L	116 % Rec (ML)	15 RSD (ML)	.8 ug/L
Thallium (7440-28-0)	.3 ug/L	103 % Rec (ML)	8 RSD (ML)	2.8 ug/L

Thorium-232 (7440-29-1)	.1 ug/L	116 % Rec (ML)	10 RSD (ML)	.8 ug/L
Antimony (7440-36-0)	.4 ug/L	98 % Rec (ML)	10 RSD (ML)	2.8 ug/L
Arsenic (7440-38-2)	1.4 ug/L	108 % Rec (ML)	35 RSD (ML)	8 ug/L
Barium (7440-39-3)	.8 ug/L	95 % Rec (ML)	6 RSD (ML)	8.01 ug/L
Beryllium (7440-41-7)	.3 ug/L	118 % Rec (ML)	8 RSD (ML)	2.8 ug/L
Cadmium (7440-43-9)	.5 ug/L	100 % Rec (ML)	5 RSD (ML)	4 ug/L
Chromium (7440-47-3)	.9 ug/L	103 % Rec (ML)	19 RSD (ML)	8 ug/L
Cobalt (7440-48-4)	.09 ug/L	110 % Rec (ML)	6 RSD (ML)	.8 ug/L
Copper (7440-50-8)	.5 ug/L	97 % Rec (ML)	15 RSD (ML)	4 ug/L
Uranium (7440-61-1)	.1 ug/L	108 % Rec (ML)	10 RSD (ML)	.8 ug/L
Vanadium (7440-62-2)	2.5 ug/L	97 % Rec (ML)	7 RSD (ML)	32 ug/L
Zinc (7440-66-6)	1.8 ug/L	104 % Rec (ML)	22 RSD (ML)	8 ug/L
Selenium (7782-49-2)	7.9 ug/L	105 % Rec (ML)	5 RSD (ML)	32 ug/L

METHOD 200.8

DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES BY INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY

Revision 5.4
EMMC Version

S.E. Long (Technology Applications Inc.), T.D. Martin, and E.R. Martin - Method 200.8, Revisions 4.2 and 4.3 (1990)

S.E. Long (Technology Applications Inc.) and T.D. Martin - Method 200.8, Revision 4.4 (1991)

J.T. Creed, C.A. Brockhoff, and T.D. Martin - Method 200.8, Revision 5.4 (1994)

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268
METHOD 200.8

23. Appendix S: EPA Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry

National Environmental Methods Index

NEMI Method Summary

Method Number:	200.7
Method Name:	**Descriptive Name:** Metals in Water by ICP-AES **Official Name:** Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry
Revision:	Revision 4.4, 1994
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111)**
Method Download:	**Download full method now (PDF file)** (293576 Byte file)
Brief Method Summary:	Except for the determination of dissolved analytes, aqueous samples are acid preserved prior to sample processing. For the analysis of dissolved analytes, an acidified portion of the filtrate is analyzed directly. For the determination of total recoverable

	analytes in aqueous samples containing particulate material as well as solid wastes, samples are subjected to acid pretreatment with nitric and hydrochloric acids and gentle refluxing prior to analysis. The method involves multi-element determination using sequential or simultaneous instruments. The instruments measure characteristic atomic-line emission spectra by optical spectrometry. Sample solutions are nebulized and the resulting aerosol is transported to the plasma torch. Element specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored at specific wavelengths by a photosensitive device. Photocurrents from the photosensitive device are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of the analytes. Background must be measured adjacent to analyte lines on samples during analysis. Various interferences are discussed and must be considered and addressed appropriately.
Scope And Application:	This method determines 31 analytes, in the dissolved fraction of aqueous samples and for the measurement of total-recoverable analytes in water, wastewater, and solid wastes. Total-recoverable determination data for aqueous samples should be reported as total (dissolved + suspended fractions) metal data.
Applicable Conc Range:	Unless otherwise noted, the analytical range extends from the laboratory-determined MDL to the upper limit of the linear dynamic range.
Interferences:	I. SPECTRAL INTERFERENCES: (1) Background emmission or stray light. (2) Spectral overlap of emissions. II. PHYSICAL INTERFERENCES: High viscosity or high particulate levels of sample can clog nebulizer. III. CHEMICAL INTERFERENCES: (1) Compound formation. (2) Ionization. (3) Solute-vaporization. IV. MEMORY INTERFERENCES: Carry-over from sample.
QC Requirements:	The minimum QC requirements consist of an initial demonstration of laboratory capability and on-going checks. The initial demonstration includes determining the linear dynamic range (LDR) for each wavelength utilized, determining the method detection limit (MDL) for each analyte, and analyzing a quality control sample (QCS). On-going checks include periodic analysis of laboratory reagent blanks (LRB), laboratory fortified blanks (LFB), instrument performance check solutions (IPC), calibration blanks (CB), spectral interference check solutions (SIC), and laboratory fortified matrices (LFM).

Sample Handling:	For dissolved elements, filter aqueous sample through a 0.45-um pore membrane filter. Adjust the pH of the filtrate to < 2 with (1+1) HNO₃. For total recoverable elements, adjust the pH of the sample to < 2 with (1+1)HNO₃. When determining boron and silica in aqueous samples, only plastic, polytetrafluoroethylene (PTFE) or quartz labware is used from time of sample collection to completion of analysis. If acid preservation in the field is not feasabile, preservation may take place at the laboratory if the sample is received by the laboratory within two weeks and is acidified at pH < 2 for 16 hours.
Max Holding Time:	6 Months
Relative Cost:	$201 to $400

Method *Metals in Water by ICP-AES (200.7)* has 31 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Aluminum (7429-90-5)	20 ug/L	105 % Rec (ML)	24 RSD (ML)	200 ug/L
Iron (7439-89-6)	30 ug/L	110 % Rec (ML)	34 RSD (ML)	50 ug/L
Lead (7439-92-1)	10 ug/L	109 % Rec (ML)	16 RSD (ML)	50 ug/L
Lithium (7439-93-2)	1 ug/L	N/A	N/A	
Magnesium (7439-95-4)	20 ug/L	103 % Rec (ML)	31 RSD (ML)	50 ug/L
Manganese (7439-96-5)	1 ug/L	98 % Rec (ML)	13 RSD (ML)	10 ug/L

Mercury (7439-97-6)	7 ug/L	N/A	N/A	
Molybdenum (7439-98-7)	4 ug/L	86 % Rec (ML)	30 RSD (ML)	20 ug/L
Nickel (7440-02-0)	5 ug/L	106 % Rec (ML)	31 RSD (ML)	20 ug/L
Potassium (7440-09-7)	.3 mg/L	57 % Rec (ML)	67 RSD (ML)	.5 mg/L
Silver (7440-22-4)	2 ug/L	48 % Rec (ML)	47 RSD (ML)	100 ug/L
Sodium (7440-23-5)	30 ug/L	119 % Rec (ML)	26 RSD (ML)	200 ug/L
Strontium (7440-24-6)	.3 ug/L	N/A	N/A	
Thallium (7440-28-0)	1 ug/L	96 % Rec (ML)	25 RSD (ML)	200 ug/L
Tin (7440-31-5)	7 ug/L	N/A	N/A	
Antimony (7440-36-0)	8 ug/L	90 % Rec (ML)	19 RSD (ML)	100 ug/L
Arsenic (7440-38-2)	8 ug/L	106 % Rec (ML)	19 RSD (ML)	100 ug/L
Barium (7440-39-3)	1 ug/L	92 % Rec (ML)	27 RSD (ML)	20 ug/L
Beryllium (7440-41-7)	.3 ug/L	91 % Rec (ML)	23 RSD (ML)	5 ug/L
Boron (7440-42-8)	3 ug/L	115 % Rec (ML)	27 RSD (ML)	100 ug/L

Cadmium (7440-43-9)	1 ug/L	98 % Rec (ML)	12 RSD (ML)	20 ug/L
Cerium (7440-45-1)	20 ug/L	N/A	N/A	
Chromium (7440-47-3)	4 ug/L	94 % Rec (ML)	22 RSD (ML)	20 ug/L
Cobalt (7440-48-4)	2 ug/L	85 % Rec (ML)	20 RSD (ML)	20 ug/L
Copper (7440-50-8)	3 ug/L	60 % Rec (ML)	43 RSD (ML)	10 ug/L
Vanadium (7440-62-2)	3 ug/L	98 % Rec (ML)	17 RSD (ML)	20 ug/L
Zinc (7440-66-6)	2 ug/L	99 % Rec (ML)	19 RSD (ML)	50 ug/L
Calcium (7440-70-2)	10 ug/L	98 % Rec (ML)	23 RSD (ML)	50 ug/L
Silica (7631-86-9)	20 ug/L	67 % Rec (ML)	67 RSD (ML)	200 ug/L
Phosphorus (7723-14-0)	60 ug/L	N/A	N/A	
Selenium (7782-49-2)	20 ug/L	96 % Rec (ML)	23 RSD (ML)	100 ug/L

METHOD 200.7

DETERMINATION OF METALS AND TRACE ELEMENTS IN WATER AND WASTES BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

Revision 4.4
EMMC Version

USEPA-ICP Users Group (Edited by T.D. Martin and J.F. Kopp) - Method 200.7, Revision 1.0, (Printed 1979, Published 1982)

T.D. Martin and E.R. Martin - Method 200.7, Revision 3.0 (1990)

T.D. Martin, C.A. Brockhoff, J.T. Creed, and S.E. Long (Technology Applications Inc.) - Method 200.7, Revision 3.3 (1991)

T.D. Martin, C.A. Brockhoff, J.T. Creed, and EMMC Methods Work Group - Method 200.7, Revision 4.4 (1994)

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U. S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

24. Appendix T: EPA Method 200.9, Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption

National Environmental Methods Index

NEMI Method Summary

Method Number:	200.9
Method Name:	**Descriptive Name:** Trace Elements in Water by GFAA **Official Name:** Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption
Revision:	Revision 2.2, 1994
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111)**
Method Download:	**Download full method now (PDF file)** (223855 Byte file)
Brief Method Summary:	For total recoverable elements in a solid or a particulate-containing aqueous samples, the solid/particulate is solubilized by gentle refluxing with nitric and hydrochloric acids. After cooling, the sample is brought up to volume and particulate is separated from the sample using a centrifuge or overnight settling. For dissolved elements or total recoverable elements where sample

	turbidity (particulate concentration) is low, the sample is prepared for analysis by adding nitric acid and diluting to a predetermined volume. The concentrations of elements in the prepared sample are measured using stabilized temperature platform (STP) graphite furnace atomic absorption (GFAA) spectroscopy.
Scope And Application:	This method determines dissolved and total recoverable elements in ground water, surface water, drinking water, storm runoff, industrial and domestic wastewater. This method also determines total recoverable elements in sediment, sludges, and soil. Total-recoverable determination data for aqueous samples should be reported as total (dissolved + suspended fractions) metal data.
Applicable Conc Range:	Ranges differ for each analyte depending on matrix, instrumentation, and selected operating conditions. Unless otherwise noted, the analytical range extends from the laboratory determined MDL to the upper limit of the linear dynamic range.
Interferences:	Interferences can be classified as spectral, matrix, elemental, and memory. Please see the method for details on reducing the levels of these interferences. (1) Spectral interferences: Caused by the resulting absorbance of light by a molecule or atom that is not the analyte of interest or emission from black body radiation coming from the carbon platform. (2) Matrix interferences: Caused by sample components that inhibit formation of free atomic analyte atoms during the atomization cycle- can be chemical or physical. (3) Elemental interferences: Antimony, aluminum, arsenic, cadmium, lead, selenium, silver, and thallium. (4) Memory Interferences: Caused from analyzing a sample containing a high concentration of an element which cannot be removed quantitatively in one complete set of furnace steps.
QC Requirements:	Method detection limit (MDL), initial precision and recovery (IPR), linear dynamic range (LDR), quality control sample (QCS), calibration verification solutions and blanks (CVs & CBs), method blanks (MBs), field blanks (FBs), equipment blanks (EBs), sampler check blanks, matrix spikes and matrix spike duplicates (MS/MSDs), and ongoing precision and recovery (OPR)
Sample Handling:	<P>For dissolved metals: Filter through a 0.45 um capsule membrane filter at time of collection. Acidify filtrate to pH of <2 with 1:1 HNO₃.</P><P>For total recoverable metals: Acidify sample with 1:1 HNO₃ to pH of <2, hold for 16 hours, verify pH, and add additional acid, if necessary.</P><P>Solid samples usually require no preservation

	other than storage at 4^oC.
Max Holding Time:	6 months for aqueous samples. No holding time limitation for solid samples.
Relative Cost:	$201 to $400

Method *Trace Elements in Water by GFAA (200.9)* has 16 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Aluminum (7429-90-5)	7.8 ug/L	112 % Rec (SL)	6 RSD (SL)	150 ug/L
Iron (7439-89-6)	N/A	108 % Rec (SL)	6 RSD (SL)	150 ug/L
Lead (7439-92-1)	.7 ug/L	102 % Rec (SL)	4 RSD (SL)	10 ug/L
Manganese (7439-96-5)	.3 ug/L	97 % Rec (SL)	<1 RSD (SL)	2.5 ug/L
Nickel (7440-02-0)	.6 ug/L	104 % Rec (SL)	4 RSD (SL)	20 ug/L
Silver (7440-22-4)	.5 ug/L	95 % Rec (SL)	6 RSD (SL)	1.25 ug/L
Thallium (7440-28-0)	.7 ug/L	95 % Rec (SL)	3 RSD (SL)	20 ug/L
Tin (7440-31-5)	1.7 ug/L	101 % Rec (SL)	<1 RSD (SL)	50 ug/L
Antimony (7440-36-0)	.8 ug/L	101 % Rec (SL)	15 RSD (SL)	15 ug/L

Arsenic (7440-38-2)	.5 ug/L	88 % Rec (SL)	<1 RSD (SL)	10 ug/L
Beryllium (7440-41-7)	.02 ug/L	106 % Rec (SL)	9 RSD (SL)	2.5 ug/L
Cadmium (7440-43-9)	.05 ug/L	105 % Rec (SL)	6 RSD (SL)	.5 ug/L
Chromium (7440-47-3)	.1 ug/L	106 % Rec (SL)	3 RSD (SL)	2.5 ug/L
Cobalt (7440-48-4)	.7 ug/L	89 % Rec (SL)	4 RSD (SL)	10 ug/L
Copper (7440-50-8)	.7 ug/L	112 % Rec (SL)	1 RSD (SL)	10 ug/L
Selenium (7782-49-2)	.6 ug/L	89 % Rec (SL)	2 RSD (SL)	25 ug/L

METHOD 200.9

DETERMINATION OF TRACE ELEMENTS BY STABILIZED TEMPERATURE GRAPHITE FURNACE ATOMIC ABSORPTION

Revision 2.2
EMMC Version

J.T. Creed, T.D. Martin, L.B. Lobring, and J.W. O'Dell - Method 200.9, Revision 1.2 (1991)

J.T. Creed, T.D. Martin, and J.W. O'Dell - Method 200.9, Revision 2.2 (1994)

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

25. Appendix U: EPA Method 245.1, Mercury (Manual Cold Vapor Technique)

National Environmental Methods Index

NEMI Method Summary

Method Number:	245.1
Method Name:	**Descriptive Name:** Mercury by CVAA **Official Name:** Mercury (Manual Cold Vapor Technique)
Revision:	Revision 3.0, 1994
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111)**
Method Download:	**Download full method now (PDF file)** (161331 Byte file)
Brief Method Summary:	A sample is digested in a glass bottle for 2 hours with a persulfate/permanganate solution under heating. After digestion, the mercury in the sample is reduced to its elemental form with stannous chloride. The concentration of mercury in the sample is determined using a cold vapor atomic absorption (CVAA) spectrometer system.
Scope And	This method allows for determination of total mercury in drinking,

Application:	surface, ground, sea, and brackish waters, and industrial and domestic wastewaters.
Applicable Conc Range:	above 0.2 ug/L
Interferences:	(A) Ions and metals: Sulfide, chloride, copper, and tellurium are reported interferences. (B) Volatile materials: Chlorine and other volatile compounds which absorb in the range of mercury should be purged from the head space of the digestion vessel before the addition of stannous chloride.
QC Requirements:	Quality control requirements include an initial demonstration of laboratory capability through analysis of laboratory reagent blanks (LRB), fortified blanks (LFB), quality control samples (QCS), and an MDL study. Ongoing quality control checks include analysis of laboratory fortified matrices (LFM), (laboratory reagent blanks) LRB, and instrument performance check (IPC) samples.
Sample Handling:	For total mercury, preserve samples with (1+1) nitric acid, and hold for 16 hours at a pH < 2 prior to analysis. Samples may be preserved in the field or upon receipt at the laboratory (the latter may minimize the chance of contamination). Use extreme care to minimize sample contamination by checking equipment, and avoiding exposure of the sample to airborne mercury in the laboratory.
Max Holding Time:	28 days.
Relative Cost:	$51 to $200

Method *Mercury by CVAA (245.1)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Mercury (7439-97-6)	.2 ug/L	166 % Rec (ML)	89 RSD (ML)	.21 ug/L

METHOD 245.1

DETERMINATION OF MERCURY IN WATER BY COLD VAPOR ATOMIC ABSORPTION SPECTROMETRY

Revision 3.0
EMMC Version

J.F. Kopp, M.C. Longbottom, and L.B. Lobring - Mercury in Water (Cold Vapor Technique), Revision 1.0, (1972)

J.F. Kopp and L.B. Lobring - Method 245.1, Revision 2.0 (1979)

L.B. Lobring and B.B. Potter - Method 245.1, Revision 2.3 (1991)

J.W. O'Dell, B.B. Potter, L.B. Lobring, and T.D. Martin - Method 245.1, Revision 3.0 (1994)

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268
METHOD 245.1

26. Appendix V: EPA Method 353.2, Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction)

National Environmental Methods Index

NEMI Method Summary

Method Number:	353.2
Method Name:	**Descriptive Name:** Nitrate-Nitrite Nitrogen by Colorimetry **Official Name:** Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction)
Revision:	Revision 2.0, August 1993
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)**
Method Download:	**Download full method now (PDF file)** (214130 Byte file)
Brief Method Summary:	A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus that reduced to nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined nitrate-nitrite, values are readily obtained by carrying out the procedure first with, and then without, the

	Cu-Cd reduction step.
Scope And Application:	This method pertains to the determination of nitrite singly, or nitrite and nitrate combined in surface and saline waters; and domestic and industrial wastes.
Applicable Conc Range:	0.05 - 10 mg/L
Interferences:	(1) Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered. (2) High concentrations of iron, copper, or other metals. (3) Large concentrations of oil and grease will coat the surface of the cadmium.
QC Requirements:	Not included.
Sample Handling:	If analysis can be made within 24 hours, refrigerating samples at 4^oC is sufficient. If samples are kept more than 24 hours, preserve with 2 mL of sulfuric acid per liter of sample and refrigerate. Do not preserve sample with mercuric chloride if they will be run through the reduction column.
Max Holding Time:	28 Days (nitrate+nitrite) 48 hours (nitrate or nitrite, singly) (MCAWW, Table 1).
Relative Cost:	$51 to $200

Method *Nitrate-Nitrite Nitrogen by Colorimetry (353.2)* has 3 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Nitrate (14797-55-8)	N/A	N/A	N/A	
Nitrite	N/A	N/A	N/A	

(14797-65-0)				
Nitrate/nitrite (E-10128)	.05 mg/L	99 % Rec (ML)	12 RSD (ML)	1 mg/L

METHOD 353.2

DETERMINATION OF NITRATE-NITRITE NITROGEN BY AUTOMATED COLORIMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

27. Appendix W: EPA Method 365.1, Phosphorus, All Forms (Colorimetric, Automated, Ascorbic Acid)

National Environmental Methods Index

NEMI Method Summary

Method Number:	365.1
Method Name:	**Descriptive Name:** Phosphorus (all forms) by Semi-Automated Colorimetry **Official Name:** Phosphorus, All Forms (Colorimetric, Automated, Ascorbic Acid)
Revision:	Revision 2.0, August 1993
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)**
Method Download:	**Download full method now (PDF file)** (552045 Byte file)
Brief Method Summary:	A sample is appropriately treated to convert all phosphorus of interest to reactive orthophosphate. Ammonium molybdate and antimony potassium tartrate are added to the treated sample reacting with orthophosphate in an acidic medium to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The concentration of the orthophosphate is measured by detecting the

	absorbance of the complex with a spectrophotometer. Note: In Section 7.7 of the method (preparation of 11 N sulfuric acid solution), "600 mL reagent water" should read "300 mL reagent water."
Scope And Application:	This method determines total phosphorus, hydrolyzable phosphorus, and orthophosphate in drinking, ground, and surface waters; and domestic and industrial wastes.
Applicable Conc Range:	0.01 - 1.0 mg/L
Interferences:	(A) Metals and silica: Copper, iron, and silica do not interfere at the levels reported in sea water, but excessively high concentrations of iron can cause precipitation and loss of phosphorus. (B) Salt error: Salt error for 5%-20% salt samples was less than 1%. (C) Arsenate: Arsenate can cause a positive interference, but is often at low concentrations. (D)Turbidity: Remove turbidity via filtration.
QC Requirements:	The minimum recommended quality control requirements include an initial demonstration of laboratory capability (determining the linear calibration range and method detection limit, and analyzing a quality control sample; LCR and MDL, and QCS), and the periodic analysis of laboratory reagent blanks.
Sample Handling:	<P>Samples should be collected in throughly clean plastic or glass bottles and should be of sufficient volume to ensure a representative sample.</P><P>Samples must be preserved with H₂SO₄ to a pH < 2 and cooled to 4^oC at the time of collection.</P>
Max Holding Time:	48 hours (orthophosphate, dissolved) 28 days (Hydrolyzable and Total) 24 hours (Total Dissolved). (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Phosphorus (all forms) by Semi-Automated Colorimetry (365.1)* has 2 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Phosphate (14265-44-2)	.01 mg/L	102 % Rec (ML)	9 RSD (ML)	
Phosphorus (7723-14-0)	.01 mg/L	99 % Rec (ML)	8 RSD (ML)	

Errata Sheet – March 5, 2004

- In Section 7.7 of the method (preparation of 11 N sulfuric acid solution), "600 mL reagent water" should read "300 mL reagent water."

28. Appendix X: EPA Method 370.1, Silica, Dissolved (Colorimetric)

National Environmental Methods Index

NEMI Method Summary

Method Number:	370.1
Method Name:	**Descriptive Name:** Silica by Colorimetry **Official Name:** Silica, Dissolved (Colorimetric)
Revision:	Issued 1971; Editorial Revision 1978
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (67827 Byte file)
Brief Method Summary:	A well-mixed sample is filtered through a 0.45 um filter. The filtrate, upon addition of the molybdate ion in acidic solution, forms a greenish-yellow complex proportional to the dissolved silica concentration, and its absorbance is measured spectrophotometrically. If concentrations of silica are exceptionally low, the greenish-yellow complex is reduced to a more intense heteropoly blue complex using 1-amino-2-naphthol-

	4-sulfonic acid.
Scope And Application:	This method determines silica in drinking, surface, and saline waters; domestic and industrial wastes.
Applicable Conc Range:	2 to 25 mg silica/L (standard). Less than 1 mg/L with reducing agent modification.
Interferences:	(A) Color/Turbidity: Correct for color or turbidity error by running blanks prepared without addition of the ammonium molybdate color reagent solution. (B) Tannin and phosphates: Interferences can be reduced or eliminated with oxalic acid. (C) Iron and sulfide: Iron and sulfide will interfere. (D) Contamination: Use silica-free reagents and minimize contact with glass. A blank also should be run.
QC Requirements:	None.
Sample Handling:	None.
Max Holding Time:	28 days (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Silica by Colorimetry (370.1)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Silica (7631-86-9)	2 mg/L	8 % Rec (ML)	14 RSD (ML)	5 mg/L

Close Window

METHOD #: 370.1	Approved for NPDES (Editorial Revision 1978)
TITLE:	Silica, Dissolved (Colorimetric)
ANALYTE:	Silica, SiO_2
INSTRUMENTATION:	Spectrophotometer
STORET No.	Dissolved 00955

1.0 Scope and Application

1.1 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.
1.2 The working range of the method is approximately 2 to 25 mg silica/L. The upper range can be extended by taking suitable aliquots; the lower range can be extended by the addition of amino-naphthol-sulfonic acid solution, as described in (6.8).

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a 0.45 μ membrane filter. The filtrate, upon the addition of molybdate ion in acidic solution, forms a greenish-yellow color complex proportional to the dissolved silica in the sample. The color complex is then measured spectrophotometrically.
2.2 In the low concentration modification the yellow (410 nm) molybdosilicic acid color is reduced by 1-amino-2-naphthol-4-sulfonic acid to a more intense heteropoly blue (815 nm or 650 nm).

3.0 Interferences

3.1 Excessive color and/or turbidity interfere. Correct by running blanks prepared without addition of the ammonium molybdate solution. See (6.7).
3.2 Tannin interference may be eliminated and phosphate interferences may be decreased with oxalic acid.
3.3 Large amounts of iron and sulfide interfere.
3.4 Contact with glass should be minimized, silica free reagents should be used as much as possible. A blank should be run.

4.0 Apparatus

4.1 Platinum dishes, 100 mL.
4.2 Colorimetric equipment-one of the following:
4.2.1 Spectrophotometer for use at 410 nm, 650 nm and/or 815 nm with a 1 cm or longer cell.
4.2.2 Filter photometer with a violet filter having maximum transmittance as near 410 nm as possible and a 1 cm or longer cell.
4.2.3 Nessler tubes, matched, 50 mL, tall form.

5.0 Reagents

29. Appendix Y: EPA Method 150.1, pH (Electrometric Method)

National Environmental Methods Index

NEMI Method Summary

Method Number:	150.1
Method Name:	**Descriptive Name:** pH in Water by Electrometric Method **Official Name:** pH, Electrometric Method
Revision:	Issued 1971; Editorial Revision 1978 and 1982
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (16262 Byte file)
Brief Method Summary:	The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.
Scope And	This method determines the pH of drinking, surface and saline water; and domestic and industrial wastes and acid rain

Application:	(atmospheric deposition).
Applicable Conc Range:	None given.
Interferences:	(1) Sodium error may bias results at pH > 10 unless a "low sodium error" electrode is used. (2) Coatings of oily material can impair electrode. (3) Temperature effects.
QC Requirements:	not included
Sample Handling:	Collect a volume of 25 mL in glass or plastic bottles. No preservation is required. The samples should be analyzed as soon as possible after collection, preferably in the field.
Max Holding Time:	ASAP
Relative Cost:	Less than $50

Method *pH in Water by Electrometric Method (150.1)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
pH (E-10139)				

METHOD #: 150.1	Approved for NPDES (Editorial Revision 1978, 1982)
TITLE:	pH (Electrometric)
ANALYTE:	pH
INSTRUMENTATION:	pH Meter
STORET No.	Determined on site 00400 Laboratory 00403

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition).

2.0 Summary of Method

2.1 The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

3.0 Sample Handling and Preservation

3.1 Samples should be analyzed as soon as possible preferably in the field at the time of sampling.

3.2 High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

4.0 Interferences

4.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

4.2 Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1 + 9) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent and cannot be controlled, it should therefore be noted by reporting both the pH and temperature at the time of analysis.

30. Appendix Z: EPA Method 160.1, Residue, Filterable (Gravimetric, Dried at 180 ^oC)

National Environmental Methods Index

NEMI Method Summary

Method Number:	160.1
Method Name:	**Descriptive Name:** Filterable Residue by Drying Oven **Official Name:** Residue, Filterable (Gravimetric, Dried at 180 ^oC)
Revision:	Issued 1971
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (28919 Byte file)
Brief Method Summary:	A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180^oC.
Scope And Application:	This method determines filterable residue in drinking, surface, and saline waters; domestic and industrial wastes.

Applicable Conc Range:	10 - 20,000 mg/L
Interferences:	(A) Mineral Waters: Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing. (B) Bicarbonate: Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate. (C) High Residue Levels: Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.
QC Requirements:	None.
Sample Handling:	Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration to 4^oC is recommended to reduce microbiological decomposition of solids.
Max Holding Time:	7 days (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Filterable Residue by Drying Oven (160.1)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Total Dissolved Solids (E-10173)	10 mg/L	101 % Rec (ML)	10 RSD (ML)	

METHOD #: 160.1	Approved for NPDES (Issued 1971)
TITLE:	Residue, Filterable (Gravimetric, Dried at 180°C)
ANALYTE:	Residue, Filterable
INSTRUMENTATION:	Drying Oven
STORET No.	70300

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
1.2 The practical range of the determination is 10 mg/L to 20,000 mg/L

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.
2.2 If Residue, Non-Filterable is being determined, the filtrate from that method may be used for Residue, Filterable.

3.0 Definitions

3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.

4.0 Sample Handling and Preservation

4.1 Preservation of the sample is not practical; analysis should begin as possible. Refrigeration or icing to 4°C, to minimize micro-biological decomposition of solids, is recommended.

5.0 Interferences

5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.
5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

6.0 Apparatus

31. Appendix AA: EPA Method 160.2, Residue, Non-Filterable (Gravimetric, Dried at 103-105 ^oC)

National Environmental Methods Index

NEMI Method Summary

Method Number:	160.2
Method Name:	**Descriptive Name:** Non-filterable Residue by Drying Oven **Official Name:** Residue, Non-Filterable (Gravimetric, Dried at 103-105 ^oC)
Revision:	Issued 1971
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (32131 Byte file)
Brief Method Summary:	A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to a constant weight at 103-105^oC.
Scope And	This method determines non-filterable residue in drinking,

Application:	surface, and saline waters; domestic and industrial wastes.
Applicable Conc Range:	4 - 20,000 mg/L
Interferences:	(A) Procedure: Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results. (B) High Residue Levels: Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Filters: Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.
QC Requirements:	None.
Sample Handling:	Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4^oC is recommended.
Max Holding Time:	7 days (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Non-filterable Residue by Drying Oven (160.2)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Total Suspended Solids (E-10162)	4 mg/L	91 % Rec (ML)	6 RSD (ML)	50 mg/L

METHOD #: 160.2	Approved for NPDES (Issued 1971)
TITLE:	Residue, Non-Filterable (Gravimetric, Dried at 103-105°C)
ANALYTE:	Residue ,Non-Filterable
INSTRUMENTATION:	Drying Oven
STORET No.	00530

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
1.2 The practical range of the determination is 4 mg/L to 20,000 mg/L.

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C.
2.2 The filtrate from this method may be used for Residue, Filterable.

3.0 Definitions

3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103-105°C.

4.0 Sample Handling and Preservation

4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.

32. Appendix AB: EPA Method 160.4, Residue, Volatile (Gravimetric, Ignition at 550 ^oC)

National Environmental Methods Index

NEMI Method Summary

Method Number:	160.4
Method Name:	**Descriptive Name:** Volatile Residue by Muffle Furnace **Official Name:** Residue, Volatile (Gravimetric, Ignition at 550 ^oC)
Revision:	Issued 1971
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (10122 Byte file)
Brief Method Summary:	The residue obtained from the determination of total, filterable or non-filterable residue is ignited at 550^oC in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile residue.
Scope And	This method determines the weight of solid material combustible at 550^oC. The test is useful in obtaining a rough

Application:	approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.
Applicable Conc Range:	Not Available.
Interferences:	None.
QC Requirements:	None.
Sample Handling:	Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4^oC is recommended.
Max Holding Time:	7 days (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Volatile Residue by Muffle Furnace (160.4)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Volatile Residue (E-11927)	N/A	N/A	7	170 mg/L

METHOD #: 160.4	Approved for NPDES (Issued 1971)
TITLE:	Residue, Volatile (Gravimetric, Ignition at 550°C)
ANALYTE:	Residue ,Volatile
INSTRUMENTATION:	Muffle Furnace
STORET No.	Total - 00505 Non-Filterable - 00535 Filterable - 00520

1.0 Scope and Application

1.1 This method determines the weight of solid material combustible at 550°C.
1.2 The test is useful in obtaining a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.

2.0 Summary of Method

2.1 The residue obtained from the determination of total, filterable or non-filterable residue is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile residue.

3.0 Comments

3.1 The test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion.
3.2 The results should not be considered an accurate measure of organic carbon in the sample, but may be useful in the control of plant operations.
3.3 The principal source of error in the determination is failure to obtain a representative sample.

4.0 Sample Handling and Preservation

4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids is recommended.

5.0 Precision and Accuracy

5.1 A collaborative study involving three laboratories examining four samples by means of ten replicates showed a standard deviation of ± 11 mg/L at 170 mg/L volatile residue concentration.

33. Appendix AC: EPA Method 160.5, Settleable Matter (Volumetric, Imhoff Cone)

National Environmental Methods Index

NEMI Method Summary

Method Number:	160.5
Method Name:	**Descriptive Name:** Determination Settleable Matter in Water Using an Imhoff Cone **Official Name:** Settleable Matter (Volumetric, Imhoff Cone)
Revision:	Issued 1974
Media:	WATER
Subcategory:	Physical
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	(No information is available)
Brief Method Summary:	Settleable matter is measured volumetrically with an Imhoff cone. The procedure to be used for this determination is found in: Standard Methods for the Examination of Water and Wastewater, 14th Edition, p. 95 Method 208F, Procedure 3a (1975).
Scope And	This method determines settleable matter in surface and saline

Application:	waters, domestic and industrial wastes.
Applicable Conc Range:	Above 0.2 ml/l/hr.
Interferences:	None Provided.
QC Requirements:	None.
Sample Handling:	Refrigerate at 4^oC.
Max Holding Time:	48 hours (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Determination Settleable Matter in Water Using an Imhoff Cone (160.5)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Settleable Solids (E-11949)	.2 mL/L/hr	N/A	N/A	

No method download available.

34. Appendix AD: EPA Method 375.2, Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)

National Environmental Methods Index

NEMI Method Summary

Method Number:	375.2
Method Name:	**Descriptive Name:** Sulfate by Colorimetry **Official Name:** Sulfate (Colorimetric, Automated, Methylthymol Blue, AA II)
Revision:	Revision 2.0, August 1993
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)**
Method Download:	**Download full method now (PDF file)** (312675 Byte file)
Brief Method Summary:	A sample is first passed through a sodium form cation-exchange column to remove multivalent metal ions. The sample containing sulfate is then reacted with an alcohol solution of barium chloride and methylthymol blue (MTB) at an acidic pH to form barium sulfate. The combined solution is raised to a basic pH so that excess barium reacts with MTB. The uncomplexed MTB color is gray; if it is all chelated with barium, the color is blue. Initially, the barium and MTB are equimolar and equivalent to 300-mg

	SO₄/L; thus the amount of uncomplexed MTB is equal to the sulfate present. The amount of sulfate in the sample is measured by measuring the absorbance of uncomplexed MTB using a spectrophotometer.
Scope And Application:	This method determines sulfate in drinking, ground and surface water; domestic and industrial wastes.
Applicable Conc Range:	3 - 300 mg/L and 0.5 - 30 mg/L (with minor modification)
Interferences:	(A) Ion exchange column breakdown: Run sulfate/Ca^{+2}standard to ensure proper column operation. (B) Turbidity: Filter or centrifuge turbid samples. (C) Contamination: Reagents or glassware can be contaminated.
QC Requirements:	The minimum recommended quality control requirements include an initial demonstration of laboratory capability (determining the linear calibration range and method detection limit, and analyzing a quality control sample; LCR and MDL, and QCS), and the periodic analysis of a laboratory reagent blank.
Sample Handling:	<P>Samples should be collected in thoroughly clean plastic or glass bottles and should be of sufficient volume to ensure a representative sample.</P><P>Samples must be preserved with H₂SO₄ to a pH < 2 and cooled to 4^oC at the time of collection.</P>
Max Holding Time:	28 days (MCAWW, Table 1).
Relative Cost:	Less than $50

Method *Sulfate by Colorimetry (375.2)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Sulfate (14808-79-8)	.5 mg/L	N/A	N/A	

METHOD 375.2

DETERMINATION OF SULFATE BY AUTOMATED COLORIMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

35. Appendix AE: EPA Method 376.2, Sulfide (Colorimetric, Methylene Blue)

National Environmental Methods Index

NEMI Method Summary

Method Number:	376.2
Method Name:	**Descriptive Name:** Sulfide by Colorimetry **Official Name:** Sulfide (Colorimetric, Methylene Blue)
Revision:	Issued 1978
Media:	WATER
Subcategory:	Inorganic
Source:	U.S.EPA National Exposure Research Laboratory (NERL) Microbiological and Chemical Exposure Assessment Research Division (MCEARD) [formerly the Environmental Monitoring Systems Laboratory (EMSL), Cincinnati, OH] 26 West Martin Luther King Drive Cincinnati, Ohio 45268-0001 Fax: 513-569-7757 email: **DWmethods.help@epa.gov**
Citation:	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Method Download:	**Download full method now (PDF file)** (16223 Byte file)
Brief Method Summary:	Sulfide in a sample reacts with dimethyl-p-phenylenediamine (p-aminodimethyl aniline) in the presence of ferric chloride to produce methylene blue, a dye which is measured with a colorimeter.
Scope And Application:	This method determines total and dissolved sulfides in drinking, surface and saline waters; domestic and industrial wastes.

Applicable Conc Range:	Sulfide concentrations below 20 mg/L.
Interferences:	(A) Aeration: Aeration can result in loss of sulfide due to volatilization or reaction with oxygen.(B) Color/Turbidity: Color or turbidity in the sample may interfere with the measurements.
QC Requirements:	None.
Sample Handling:	Analyze immediately.
Max Holding Time:	Analyze immediately.
Relative Cost:	Less than $50

Method *Sulfide by Colorimetry (376.2)* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Spiking Level
Sulfide (18496-25-8)	N/A	N/A	N/A	

METHOD # 376.2	Approved for NPDES (Issued 1978)
TITLE:	Sulfide (Colorimetric, Methylene Blue)
ANALYTE:	Sulfide
INSTRUMENTATION:	Spectrophotometer
STORET No.	Total 00745 Dissolved 00746

1.0 Scope and Application

1.1 This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.
1.2 Acid insoluble sulfides are not measured by this method. Copper sulfide is the only common sulfide in this class.
1.3 The method is suitable for the measurement of sulfide in concentrations up to 20 mg/L.

2.0 Summary of Method

2.1 Sulfide reacts with dimethyl-p-phenylenediamine (p-aminodimethyl aniline) in the presence of ferric chloride to produce methylene blue, a dye which is measured at a wavelength maximum of 625 nm.

3.0 Comments

3.1 Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form. Dissolved oxygen should not be present in any water used to dilute standards.
3.2 The analysis must be started immediately.
3.3 Color and turbidity may interfere with observations of color or with photometric readings.

4. Apparatus

4.1 Matched test tubes, approximately 125 mm long and 15 mm O.D.
4.2 Droppers, delivering 20 drops/mL. To obtain uniform drops, hold dropper in vertical position and allow drops to form slowly.
4.3 Photometer, use either 4.3.1 or 4.3.2.
4.3.1 Spectrophotometer, for use at 625 nm with cells of 1 cm and 10 cm light path.
4.3.2 Filter photometer, with filter providing transmittance near 625\nm.

5.0 Reagents

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #**14**

Data Management

Version 1.0 (04/11/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

This Standard Operating Procedure (SOP) describes data management procedures for all data derived from water quality monitoring in Northern Colorado Plateau Network (NCPN) park units. This SOP also describes and documents the NCPN Water Quality database and provides instructions for the development, maintenance and distribution of monitoring data associated with the NCPN Water Quality Monitoring Protocol. Procedures for data handling and quality assurance/quality control for all monitoring protocols implemented by the Northern Colorado Plateau Network are detailed in the network's Data Management Plan posted on the NCPN website.

For convenience and simplification purposes, the following definitions and acronyms are used throughout this document:

SEUG: Southeast Utah Group, which consists of ARCH, CANY, HOVE, and NABR.

STORET: STOrage and RETrieval database maintained by EPA, also referred to as **EPA-STORET**.

WRD-STORET: STORET database maintained by the NPS Water Resources Division.

NPSTORET: Microsoft Access database used to store data locally in a STORET-compatible format.

NWIS: National Water Information System database maintained by USGS.

PCFF: Personal Computer Field Forms, a database application developed by USGS designed for entering and storing field data.

NWQL: USGS National Water Quality Lab.

UT DWQ: Utah Division of Water Quality.

Utah Parks: Parks in which samples are collected by NPS staff and analysis is done by the Utah state lab (BRCA, CARE, ZION, and SEUG parks),

Colorado Parks: Parks in which samples are collected by NPS staff and analysis is done by USGS (BLCA/CURE), and

USGS-Sampled Parks: Parks in which USGS handles sample collection and analysis (DINO).

Table of Contents

1. Overview 5
1.1. DINO 5
1.2. BLCA/CURE 5
1.3. CARE/BRCA/ZION 6
1.4. SEUG Parks 6
1.5. Upload to WRD-STORET 6
2. Data Model 8
2.1. NCPN-water 8
2.2. NPSTORET 9
3. Populating Water Quality Databases 10
3.1. Field Data Entry 10
3.2. Data Upload to NCPN-water 11
3.2.1. Utah Parks 11
3.2.2. USGS-Sampled Parks 11
3.2.3. Colorado Parks 11
3.3. Upload from NCPN-water to NPSTORET 11
4. Data Verification and Validation 12
4.1. Manual Verification 12
4.2. Automated Verification at Data Entry 12
4.3. Automated Verification after Data Entry 12
4.4. Data Quality Review and Communication 13
4.5. Bias Control 13
5. Version Control 13
5.1. Working Backup Folder 13
5.2. Consistency in Directory Structure 14
5.3. Active and Archive Project Folders 14
6. Data Archiving 15
6.1. Data Archiving for Utah Parks 15
6.1.1. Field Forms 15
6.1.2. Photos 15
6.1.3. Raw Field Data 15
6.1.4. Reports 16
6.1.5. Database Formats 16
6.1.6. Instrument Log Books 16
6.2. Data Archiving for Colorado Parks 26
6.3. Data Archiving for USGS-Sampled Parks 33
7. Metadata 33
8. Data Management Calendar 33
9. Citations 35
10. Appendix A: Documentation of NCPN-water Database Tables 36
10.1. Table Name: CategoryRanks 36
10.2. Table Name: CONSOLIDATED_SITES 37
10.3. Table Name: COUNTY_CODES 38
10.4. Table Name: EPACODES 38
10.5. Table Name: HUC_CODES 38
10.6. Table Name: ORPHANED_SITES 39
10.7. Table Name: PARAMETER 40
10.8. Table Name: PARKS 41
10.9. Table Name: QW_RESULT 41
10.10. Table Name: SAMPLE 42
10.11. Table Name: SAMPLE_TYPES 43
10.12. Table Name: SITE 44
10.13. Table Name: SITE_TYPES 46
10.14. Table Name: SOURCE_AGENCIES 46

10.15. Table Name: tblCodeEquivalents 46
10.16. Table Name: tbl_DB_Revisions 47
10.17. Table Name: tbl_master_version 47
10.18. Table Name: tbl_SOP_version 47
10.19. Table Name: tlu_projects 48
10.20. Table Name: USGS_parmfile_master 48
10.21. Table Name: USGS_SWDV_SITES 49
10.22. Table Name: VALUE_REMARKS 50

List of Tables

Table 1. Summary of Archiving Information for Digital Data - UT Parks 24
Table 2. Summary of Archiving Information for Hard Copy Data - UT Parks 25
Table 3. Summary of Archiving Information for Digital Data - CO Parks 32
Table 4. Summary of Archiving Information for Hard Copy Data - CO Parks 32
Table 5 Calendar of data management events that must be performed on a routine basis. 34

List of Figures

Figure 1. NCPN data flow from generation to legacy storage. 7
Figure 2. Data Model for NCPN-water Database. 9
Figure 3. Data Model for NPSTORET Database. 10
Figure 4. Field Data - UT Parks 18
Figure 5. Lab Data - UT Parks 19
Figure 6. Calibration Data - UT Parks 20
Figure 7. Bacteria Data - UT Parks 21
Figure 8. Site Selection Data - All Parks 22
Figure 9. Record of cumulative bias – all park units. 23
Figure 10. Field Data - CO Parks 27
Figure 11. Lab Data - CO Parks 28
Figure 12. Calibration Data - CO Parks 29
Figure 13. Bacteria Data - CO Parks 30
Figure 14. Reservoir Profile Data - CO Parks 31

1. Overview

Due to the involvement of several different organizations in the NCPN water quality monitoring program, there is a complicated flow of data and multiple water quality databases are involved in the management of NCPN water quality data (Figure 1.)

The water quality component of the Natural Resource Challenge requires that networks archive all water quality data collected as part of the monitoring program in a STOrage and RETrieval (STORET) database maintained by the NPS Water Resources Division (WRD-STORET). Thus, all NCPN water quality monitoring data, regardless of which other databases it may reside in temporarily or permanently, will ultimately make its way to WRD-STORET.

The primary 'working' database for NCPN is called NCPN-water. It is the main repository for water quality monitoring data, including all historic and future data generated in NCPN units. It will be maintained as the working data base for several reasons including:

1. It has a complete historic record of water quality data for sites in and near NCPN parks that should be maintained and built upon;
2. NCPN has complete version control;
3. It has built-in functionality for quality control and reporting;
4. NCPN staff can add quality control and reporting functionality over time;
5. It can be integrated with national databases for upload.

Details associated with the transfer of data between the above-mentioned databases will be provided in Section 3.0 (Populating Water Quality Databases). The following sections briefly outline the flow of data for geographic park clusters where monitoring is overseen by individual hydrologists.

1.1. DINO

For the USGS-sampled park (DINO), field data are entered into Personal Computer Field Forms (PCFF, SOP 8 section 14), and lab data are entered into and stored in the USGS National Water Information System (NWIS) database. They will be periodically downloaded into NCPN-water, which serves as a link to WRD-STORET.

1.2. BLCA/CURE

Field data collected in BLCA/CURE are entered into PCFF but will flow into NCPN-water either directly via import templates (under construction) or via a link to NWIS (under construction). In the past, these data had a mixed history of custody because they were collected by NPS staff using the USGS protocol. For this reason, some data were stored in the USGS NWIS database, but recently funding has not been available for USGS quality control checks and thus, they have not made their way into NWIS. Because of future uncertainty in funding, it has been decided that new data from these parks will be pushed through the NCPN-water archive and quality control procedure. This will ensure timely upload to WRD-STORET and ensure their availability for analysis and reporting.

1.3. CARE/BRCA/ZION

For parks in which sample collection is done by NCPN staff (BRCA, CARE, and ZION), field data are loaded directly into NCPN-water via the “site table”, ”sample table” and “QW result” tabs in the Field_forms.xls spreadsheet. Lab data provided on-request from the UT DWQ will be formatted (under construction) and uploaded independently from field data. Both lab and field data in NCPN-water will then be uploaded to WRD-STORET by import into NPSTORET and then migration to WRD-STORET.

1.4. SEUG Parks

For parks in which sample collection is done by SEUG staff (ARCH, CANY, HOVE, NABR), data are stored in the EPA-STORET database via upload performed annually by the UT DWQ. A monthly synchronization of the data between EPA-STORET and WRD-STORET (performed by the NPS WRD) ensures that this data is ultimately stored in WRD-STORET. An alternate path that includes NCPN-water (under construction) will make these data available on a timelier basis.

1.5. Upload to WRD-STORET

NPSTORET is an Access template for uploading data to WRD-STORET. NCPN-water database fields will be mapped to appropriate fields in NPSTORET. NPSTORET will be passed electronically to WRD who will then upload data to WRD-STORET. The NCPN hydrologist will send the NPSTORET back-end database to the Water Resources Division annually who then uploads the data to WRD-STORET. While it is true that no one outside Fort Collins can access WRD-STORET, all the data in WRD-STORET are replicated at the EPA STORET Data Warehouse (http://www.epa.gov/storet/dw_home.html) where networks and the public can download their data and data belonging to states and other entities. You can, however, get WRD-STORET (EPA-STORET) data into NPSTORET by using the query tools at the EPA STORET Data Warehouse, downloading the resulting file, and importing it into NPSTORET.

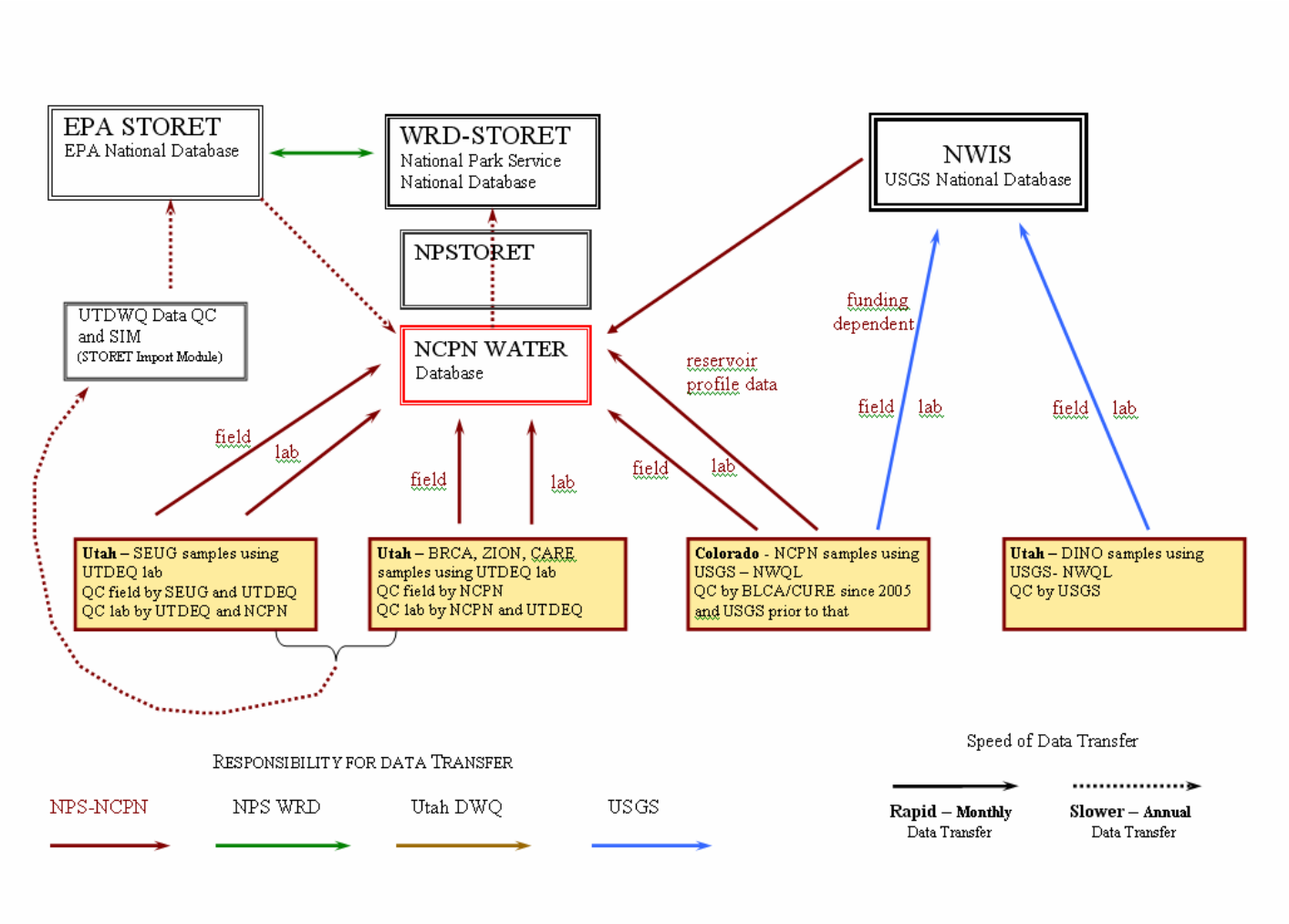


Figure 1. NCPN data flow from generation to legacy storage.

2. Data Model

The focus of this section is on the data models for the two local databases that will store NCPN water quality monitoring data: **NCPN-water** and **NPSTORET**.

2.1. NCPN-water

The relationships among the primary tables in the NCPN-water are shown in Figure 2 (all tables are not shown). The *Site* table stores information describing each site or station, including location information. Each site can have one or more associated samples, which are stored in the *Sample* table. Associated with each sample are one or more results, which are stored in the *QW_Result* table. Each result record has an associated parameter, which is stored in the *Parameter* table. Descriptive information for parameters includes parameter name, code, reporting units, and sample matrix. Lookup tables include *Parks*, *HUC_Codes*, *County_Codes*, *Sample_Types*, *Source_Agencies*, and *Value_Remarks*. Each sample is linked to tables *tbl_master_version* and *tbl_SOP_version* (not shown in the figure), which together define the narrative and suite of SOP versions in effect at the time of the sample. Documentation of all tables and attributes in NCPN-water is provided in Appendix A.

Modifications are planned for NCPN-water that will impact the data model. For example, new tables will be added to store calibration data and data associated with photographs.

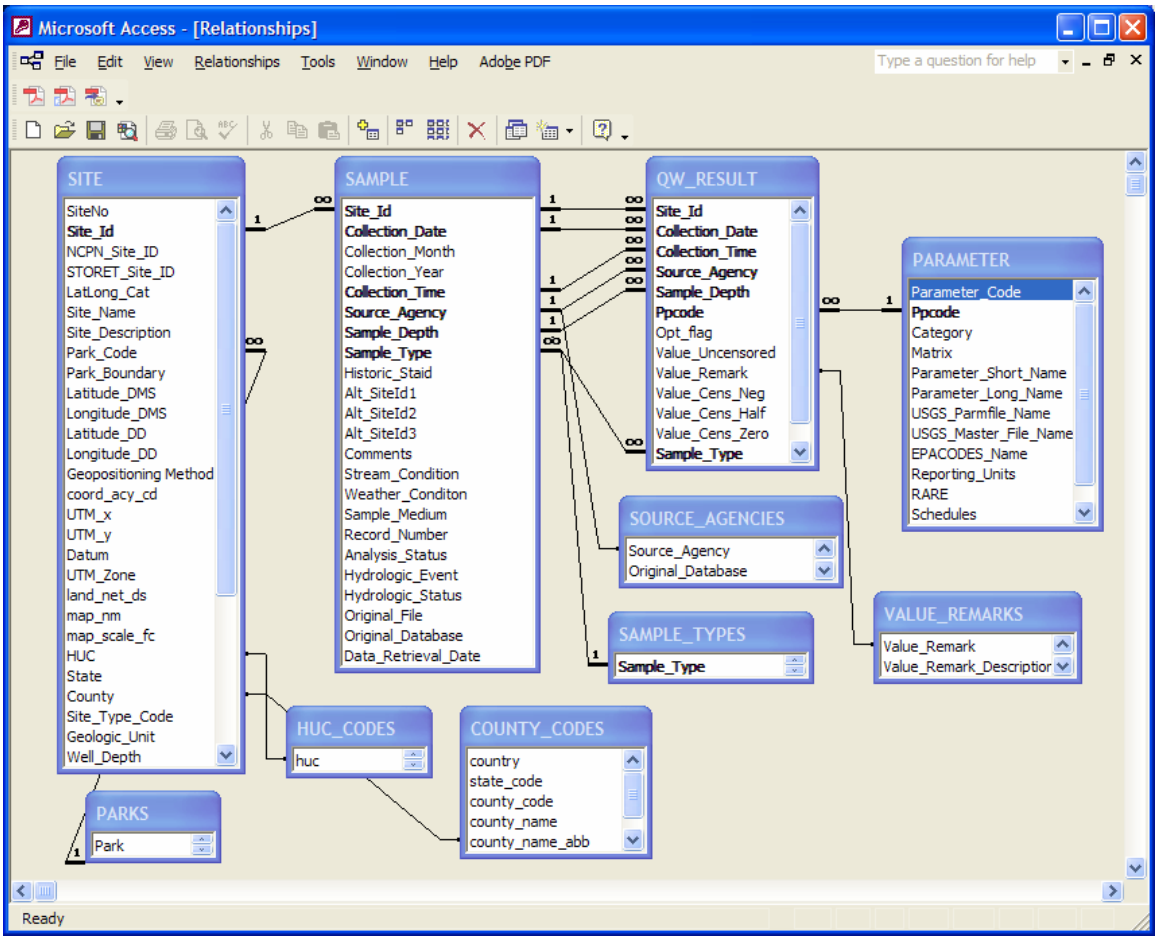


Figure 2. Data Model for NCPN-water Database.

2.2. NPSTORET

Figure 3 shows the relationships among the primary tables in NPSTORET (note that only 6 of the 39 tables are shown). Information describing monitoring projects is stored in *tblProjects*, while attributes of the monitoring locations/stations are stored in *tblLocations*. Station visits are recorded in *tblVisits*. A visit to a station in support of a project can have one or more activities (samples/events/data groupings). Information describing the activities that transpired during the Station Visit is stored in *tblActivities*. An activity can have one or more results, i.e., data documenting the field measurements, observations, and/or lab analyses for each activity. Results are stored in *tblResults*. Associated with each result record is metadata documenting the characteristic definitions - those parameters and analyses for which results were obtained. Characteristic definitions are stored in *tblCharacteristics*. Documentation of all tables and attributes in NPSTORET is provided in the NPSTORET Data Dictionary contained in and installed by the NPSTORET installation program (available at http://www.nature.nps.gov/water/infoanddata/index.cfm#NPSTORET).

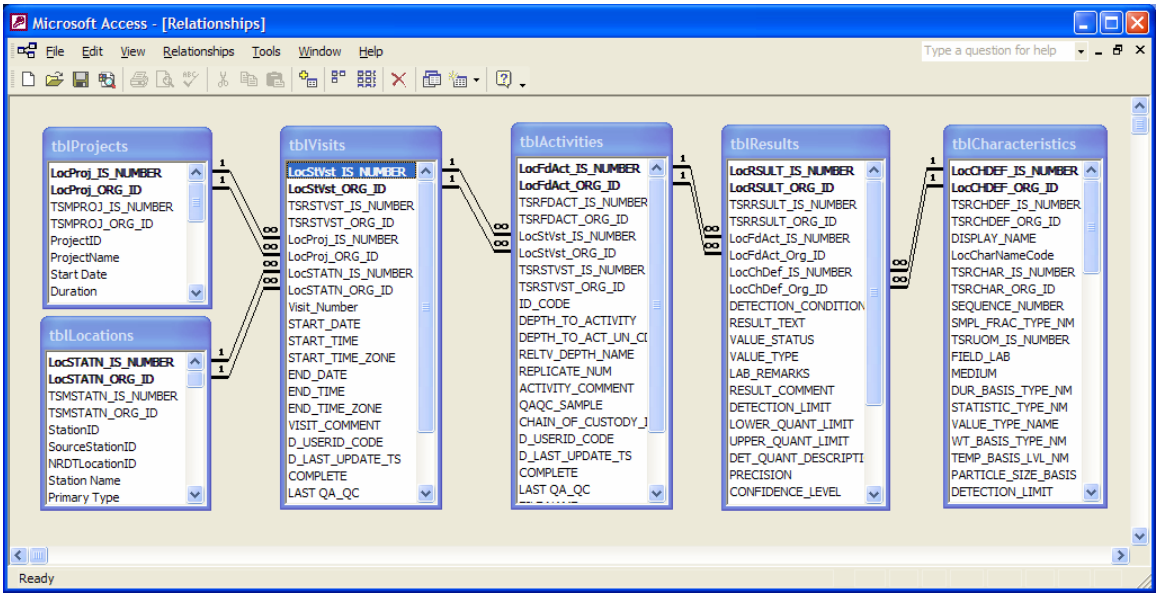


Figure 3. Data Model for NPSTORET Database.

3. Populating Water Quality Databases

Due to large geographic distances between parks, pre-existing programs and cooperative agreements, water quality data generated for NCPN parks takes a circuitous route to its ultimate destination in the WRD and EPA-STORET national databases (Figure 1). In some cases, separate data streams exist for field and laboratory-generated data due to the lag time between sample collection and laboratory analysis. Field data can be loaded into databases and quality control checked immediately, whereas lab data may take a few weeks (NWQL lab) to a year (UT DWQ) before it is available. The remainder of this section describes how the water quality databases are populated, including the procedures for field data entry, data upload to NCPN-water, and data upload from NCPN-water to NPSTORET.

3.1. Field Data Entry

Field data for cooperating groups in the NCPN are recorded on hard copy field forms in the field, and then subsequently entered into the appropriate software application: the *Field_forms.xls* spreadsheet for Utah parks, or *Personal Computer Field Forms* (PCFF) for Colorado parks. PCFF is a database application developed by USGS designed for entering and storing field data. Specific instructions for data entry are provided in SOP 8 section 14. Both Field_forms.xls and PCFF have utilities for generating reports that ship with physical water samples to the laboratory. For this reason, field data are always entered before shipping samples. To help ensure the accuracy of the data, data entry should occur as soon as possible after data collection is completed and by the person who collected the data or someone who is familiar with the data. The primary goal of data entry is *to transcribe the data from paper records into the computer with 100% accuracy*.

3.2. Data Upload to NCPN-water

Data are uploaded to NCPN-water via different mechanisms for the 3 monitoring groups: (i) **Utah parks**, (ii) **USGS-sampled parks**, and (iii) **Colorado parks**.

3.2.1. Utah Parks

All lab and field data will be uploaded to the NCPN-water database. A template for uploading field data from Field_forms.xls to NCPN-water has been built for Utah parks. This template allows monthly data to be uploaded immediately after sampling run and data entry are completed. A similar template has been built to accommodate the uploading of lab data for SEUG Utah parks. However, because of the usual delays in receiving lab results from the UT DWQ, lab data in most cases will not be uploaded immediately. Even though field and lab data will be uploaded to NCPN-water at different times, they will still be linked in the database via the site ID and site visit date fields.

The above-described field and lab templates for Utah parks are being deployed initially in BRCA, CARE, and ZION. Deployment of these templates to SEUG parks will occur after SEUG staff have been trained to use this new system. Until that time, SEUG data will continue to follow the path shown in Figure 1. These data will be obtained from the state of Utah by WRD, with network assistance, on an annual basis during transmission/submission of all network data to WRD.

3.2.2. USGS-Sampled Parks

All field and lab data for DINO are entered into NWIS because the field and lab work are done via contract with USGS. These data have to be downloaded from NWIS and formatted for import to NCPN-water. A data import module will be built to perform this data transfer (under construction).

3.2.3. Colorado Parks

All field and lab data for Colorado parks will be uploaded from PCFF using a customized template (under construction). Colorado parks also collect *reservoir profile data*, which consists of basic parameters (temperature, pH, conductivity, and DO) taken at 1meter intervals throughout the water column of each reservoir. These data are collected using a Hydrolab and imported into an Access database. Reservoir profile data will be uploaded from the Access database to NCPN-water using a customized template (under construction).

3.3. Upload from NCPN-water to NPSTORET

Uploading data from NCPN-water to NPSTORET will occur via the NPSTORET import module designed and maintained by NPS WRD. To use this import module, the appropriate fields in NCPN-water will be mapped to NPSTORET's importable fields and the appropriate file structures/templates defined. Tools will be developed to automate as much of this process as possible (under construction).

4. Data Verification and Validation

This section highlights some of the important data verification and validation procedures. A more comprehensive coverage of this subject is provided in the Quality Assurance Project Plan (QAPP) SOP (SOP 7).

4.1. Manual Verification

Data verification immediately follows data entry and involves checking the accuracy of the computerized records against the hard copy field records and identifying and correcting any errors. All records entered will be verified against their original source in the following order:

1. Visual review at data entry. The person doing data entry verifies each record after input and immediately corrects any errors.
2. Visual review after data entry. A pseudo-random selection of approximately five percent of all records entered during a data entry session are verified, preferably by a second person who did not perform the data entry. Errors are clearly marked and corrected as soon after data entry as possible.
3. Summary queries and tallies. Queries are run that detect broad errors such as duplicate or omitted records.

4.2. Automated Verification at Data Entry

Several QA/QC checks are incorporated in the Field Form spreadsheets that automatically validate data upon data entry. Such checks include auto-filled fields, range limits, and pick lists. For example, in each site page of the Field_forms.xls spreadsheet, column J automatically reports completeness for field data collection, and column K is a special column to indicate quality control checks. Additional QA/QC checks will be incorporated in both the spreadsheets and NCPN-water in the future.

4.3. Automated Verification after Data Entry

While many errors are prevented by including validation routines that function during data entry, additional review of the data is needed to detect generic and specific errors that fall outside the scope of automated validation.

Invalid data commonly consist of slightly misspelled names, the wrong date, or out-of-range errors in parameters with well-defined limits (e.g., elevation). Other errors consist of unreasonable metrics or associations. Validation queries will be of two types: one type will present tabular data in ways that records can be reviewed for content and context; the other type will perform specific calculations or routines to detect logic or range errors.

Corrections to errors detected during validation require notations in the *original paper field records* about how and why the data were changed. Modifications to the field records should be clear and concise while preserving the original data entries or notes. Validation efforts should also include a check for the completeness of a data set since field sheets or other sources of data could easily be overlooked.

4.4. Data Quality Review and Communication

The NCPN hydrologist and other NPS staff involved in data collection or entry will meet several times during the year to discuss data quality problems and issues. Procedures may need revision if verification and validation processes reveal an unacceptable level of data quality. Quality checks may also reveal the need to modify field or computer forms if data transcription or entry errors are frequent. The NCPN hydrologist will use data documentation and metadata to notify end users and other project personnel of project data quality.

4.5. Bias Control

The NCPN hydrologist will oversee change in staff, instruments or methods that could result in measurement bias. To help mitigate bias from such change a separate SOP (#6) has been written to help evaluate and control long term bias. Every effort will be made to eliminate bias by modifying measurement SOP's. However, in cases where no reasonable effort can be employed to control bias mathematical adjustments to data may be made. A record of why and how these adjustments were made is stored in the Record of bias SOP (#6) and in the Record_of_bias.xls spreadsheet (Figure 9). Adjustments to original data may be made to make data backwards compatible once it is extracted from the data base, but no changes will be made to the original data in the database.

5. Version Control

Version control is the process of managing copies of changing files over the course of a project. Change includes any alteration to the structure or content of the files, which should not be made without the ability to fully recover a data set as it existed before changes were made. Sound data archiving practices will often provide this ability to recover a given data set if needed (refer to Section 6.0 for details on data archiving). However, in certain cases, especially when editing a database, it is good practice to make a working backup of a file before any major changes are made. For example, if a result value were recorded erroneously in the Field_forms.xls spreadsheet and not discovered until after the record was uploaded to NCPN-water, the record would need to be edited in the database. In this case, a working backup of the NCPN-water database should be made before editing this record.

5.1. Working Backup Folder

NCPN recommends creating a working backup folder in the working project folder to store these backups. For example, suppose the following working folder contains the current version of the NCPN-water database:
X:\Active_Monitoring_Projects\Water_Quality\Data\Database\2006\NCPN_WQ.mdb

A *working_backup* folder would be added as follows:
X:\Active_Monitoring_Projects\Water_Quality\Data\Database\2006\working_backup\NCPN_WQ.mdb

Establishing a separate folder for working backups helps keep the working folder cleaner and serves as a reminder to make such backups. Files copied into the working backup folder will need to be renamed if multiple backups exist for the same file. The current

date can usually be appended to the file name to ensure a unique file name. Since working backups do not need to be retained long-term, these backup files should be reviewed periodically to determine which ones can be deleted.

5.2. Consistency in Directory Structure

In general, the use of logical and consistent directory structure and file/folder naming conventions can help simplify version control. NCPN recommends that, where possible, the different monitoring groups follow the guidelines and conventions used by NCPN to store water quality monitoring data. These guidelines and conventions, described below, build upon both the NCPN hydrologist's existing directory structure and the (draft) NCPN Directory Structure Guidance Document (NCPN-NPS 2007).

5.3. Active and Archive Project Folders

The NCPN will maintain two project folders for water quality monitoring data: (i) an *active* project folder (*X:\Active_Monitoring_Projects\Water_Quality*), which contains all current (i.e., non-archived) files, and (ii) an *archive* project folder (*X:\Archive\Monitoring_Archive\Water_Quality*), which contains all archived files. The need to have entirely different directory paths for these two folders is specific to the NCPN because of its backup infrastructure (which allows the top-level folders to have different backup schedules). Such a complete separation may not be necessary in all cases. What is important is that a separate folder is designated for archived files and that it is marked as "read-only".

The general structure of the active project folder is as follows (all possible folders are not shown):

- **Data**: Contains all current year data, which may include databases, spreadsheets, and/or text files. Includes the following subfolders (where applicable) to distinguish different types of data:
    - **Aquacalc**: Contains raw flow data.
    - **Data_lab**: Contains lab results data.
    - **Field_forms**: Contains Field Form spreadsheets.
    - **Field_site_descriptions**: Contains field site descriptions.
    - **Troll**: Contains raw core parameter data.
    - **Photos**: Contains photos stored by month and named by site- see section 6.1.2
- **Reports**: Stores the current annual report and any other reports produced during the current year. Includes the following sub-folders:
    - **PDF**: Contains PDF version of report(s).
    - **Native_Format**: Contains source files (e.g., Word documents) used to create report(s).
- **Protocol**: Stores the current version of the protocol, including narrative and SOP files.

The general structure of the archive project folder is similar to the active project folder, except that the archive folder contains files for all previous years. Thus, the folders listed above will have subfolders labeled by year (and in some cases, month, depending on the granularity of the data). Details of this structure are given in Table 1, while additional details associated with archiving are given in Section 6.0 (Data Archiving).

Finally, before leaving the subject of version control, it is important to mention that all water quality monitoring data should be stored on a secure machine that is backed up regularly. The NCPN shared drive (X:\) provides a secure location with a regular backup routine that includes off-site storage rotation.

6. Data Archiving

All NCPN water quality monitoring data, whether digital or hard copy, will be archived to help ensure the long-term preservation of this data. Since water quality monitoring procedures differ somewhat depending on state requirements and USGS involvement, the data archiving procedures also differ in some cases. Thus, data archiving will be discussed separately for the 3 monitoring groups: (i) **Utah Parks**, (ii) **Colorado Parks**, and (iii) **USGS-sampled Parks**.

6.1. Data Archiving for Utah Parks

Figure 4 illustrates the flow of field data for Utah parks. The intent of this figure is to identify all the different forms of data that require archiving, not to show all processing that is performed on the data. Thus, critical processing steps such as QA/QC checks do not appear in this figure.

6.1.1. Field Forms

Blank field forms are printed from an Excel spreadsheet and are taken into the field, where certain critical measurements are recorded manually on the hard copy forms (see shaded box labeled “Field Form Original”). Data on these hard copy forms are later entered into the Field Form spreadsheet (see box labeled “Field Form (xls)”), while data from the field instruments (see boxes labeled “Troll Data Report (bin)” and “Aquacalc Flow Data (txt)”) are imported into the same spreadsheet.

6.1.2. Photos

For any photos that were taken during the site visit, the photos are downloaded to a new folder for each monthly site visit. Photos are renamed and the photo name for each site is recorded in the Field_forms.xls spreadsheet, thus providing links to the original photos. Copies of the photos are subsequently uploaded to the NCPN Photo database, which will serve as an archive for photos. The photo database renames the archived photos according to NCPN photo file naming conventions. The newly assigned filenames are then recorded in the NCPN-water database (via an automated process), thus providing links to the archived photos in the photo database (this link is not shown in Figure 4).

6.1.3. Raw Field Data

Raw field data are archived digitally, as indicated by the “Troll Data Report Archive” and “Aquacalc Flow Data Archive” boxes. Completed field forms are archived in both digital (see box labeled “Field Form Archive (xls, txt)”) and hard copy (see shaded box labeled “Field Form Archive”) formats. The “(xls, txt)” in the digital Field Form Archive box indicates that the field form data is archived in both native (xls) and ASCII text (txt) formats.

6.1.4. Reports

Field form data are uploaded to the NCPN-water database, where they may be accessed to generate reports. Reports may be created using a variety of different source file types (e.g., Word, Excel) and are usually delivered in PDF format. A printed copy of each report will be archived (see shaded box labeled "Report Archive"), while all associated digital files will be archived as well (see box labeled "Report Archive"). The arrow labeled "analyze (real time)" between Field Form (xls) and Report (pdf, doc, etc.) indicates that reports may also be generated directly from the Field Form spreadsheets. Such reporting may be done, for instance, for QA/QC purposes or other reporting needs that cannot wait until the data are uploaded to the database.

6.1.5. Database Formats

The NCPN-water database is archived in both native (mdb) and ASCII text (txt) digital formats. Archiving of the NCPN Photo and WRD-STORET databases is not included here because it is outside the scope of the NCPN Water Quality Data Management realm.

6.1.6. Instrument Log Books

As shown in the separate data flow in the lower right hand corner, instrument logbooks (which exist only in hard copy format) are archived (see shaded box labeled "Instrument Logbooks Archive"). The Aquacalc, Troll, and turbidity meter instruments each have such logbooks.

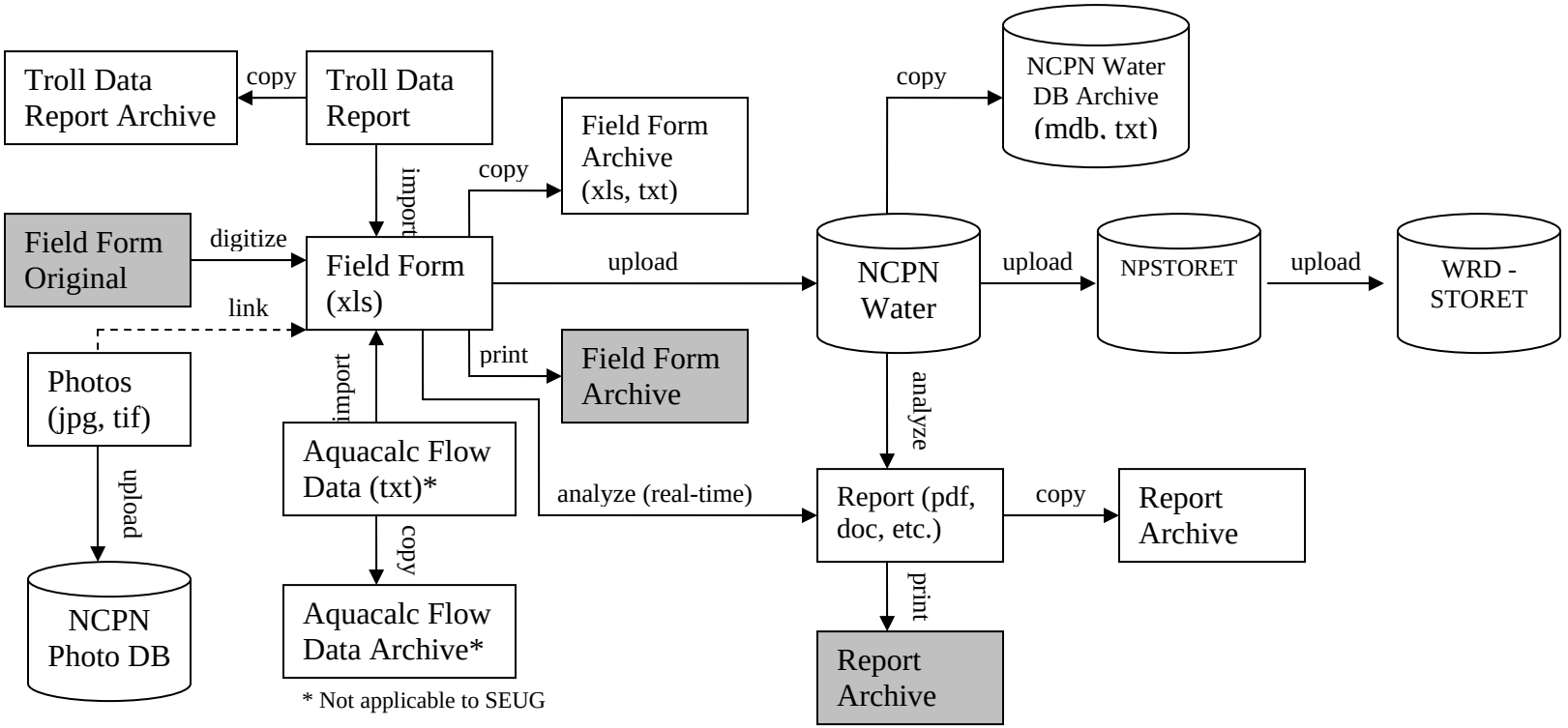
A similar diagram is provided for each of the other types of data: lab (Figure 5), calibration (Figure 6), bacteria (Figure 7), and site selection (Figure 8). The captions below each figure highlight the main points.

To help ensure that all relevant data items are archived appropriately, archiving procedures for Utah parks are summarized below for digital and hard copy data:

- Digital Data: Table 1 summarizes the archiving information for digital data for Utah parks, including file formats and archive locations. All digital data will be archived on the NCPN shared drive (X:) in the water quality archive folder (*X:\Archive\Monitoring_Archive\Water_Quality*). Digital file archives will be stored in both native and ASCII text formats. At least once per year, SEUG and NCPN will transfer the applicable files listed in Table 1 to the NCPN data manager, who will archive them accordingly.

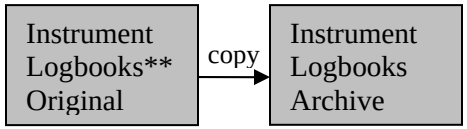
- Hard Copy Data: Table 2 summarizes the archiving information for hard copy data for Utah parks. Original hard copy data items produced as part of water quality monitoring are a high priority to maintain under archival conditions. NCPN will maintain archival-quality copies of the data items listed in Table 2 in the NCPN central files. At least once per year, SEUG and NCPN will provide archival-quality copies of the applicable data items listed in Table 2 to the NCPN data manager. Archival-quality materials (e.g., acid-free paper) can be obtained from Light Impressions (www.lightimpressionsdirect.com). Existing park procedures should be followed for archival of the original hard copies and any other archival-related requirements. Park curators should be consulted for specific details on these procedures. Alternatively,

SEUG and NCPN may send the original hard copies to the NCPN data manager (at least once per year), who will make archival copies. Such arrangements will need to be approved by the appropriate park staff (e.g., curators) to ensure the timely return of originals to the park.



General Assumptions/Notes (apply to all diagrams):

- Diagrams are intended to identify different forms of data that need archiving and thus do not show QA/QC processes.
- UT diagrams reflect current data flows for BRCA/ZION/CARE and "proposed" flows for SEUG.
- Reporting (labeled as "Report" which represents annual, triennial, or other reports) can be done on any type of data stored in the NCPN Water Database.



** Logbooks exist for the Aquacalc, Troll, and Turbidity meter instruments.



Figure 4. Field Data - UT Parks

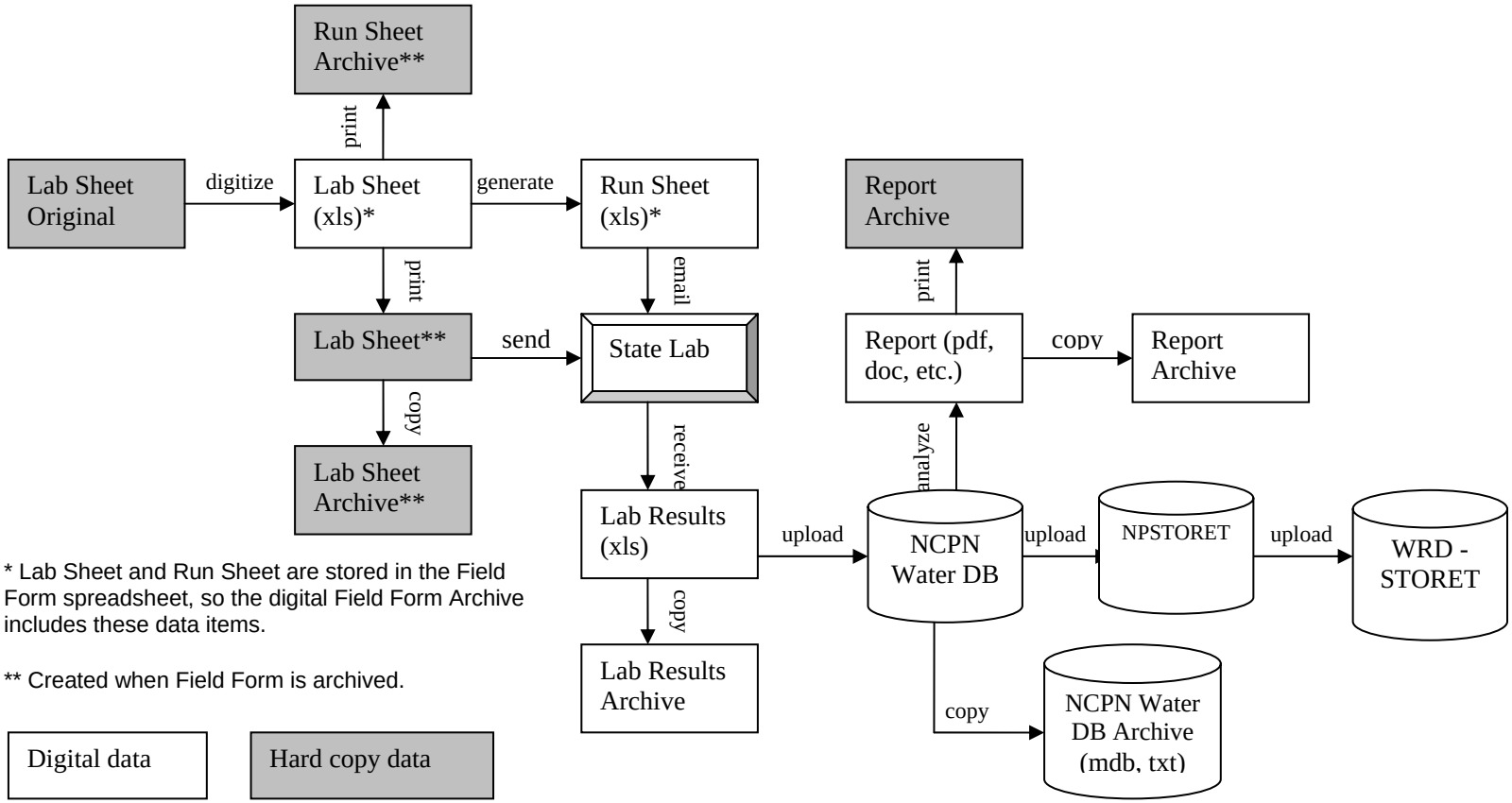
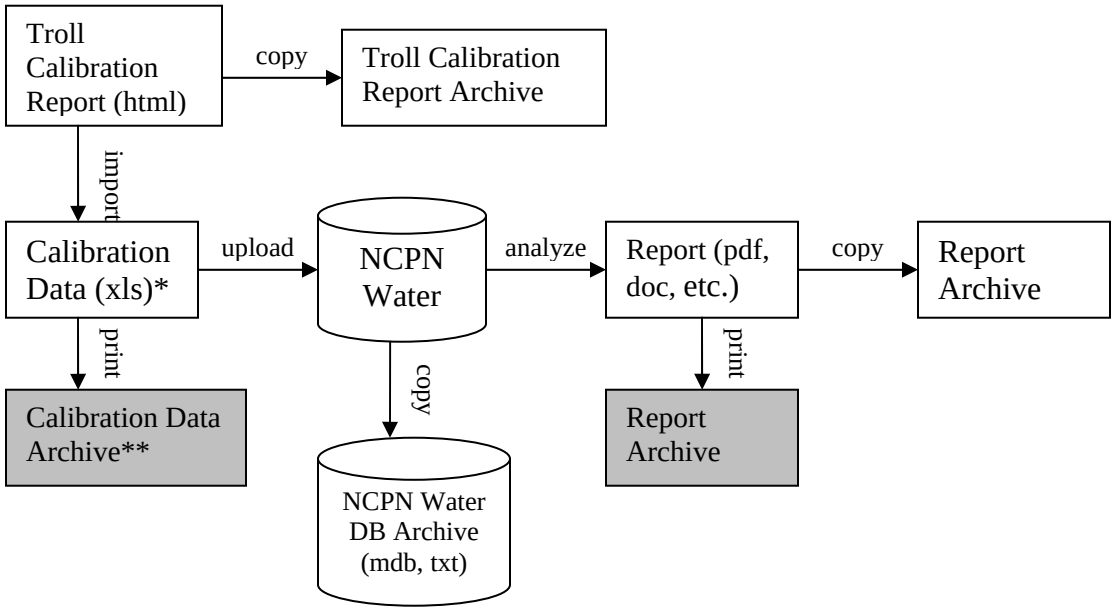


Figure 5. Lab Data - UT Parks

Blank lab sheets are printed from the Field_forms.xls spreadsheet and taken into the field, where data are recorded on the hard copy. These data are then entered manually into the lab sheet portion of Field_forms.xls, which generates both the lab sheet and run sheet that are sent to the state lab along with the samples. Hard copy archives of both the lab sheet and run sheet are created. Lab results received from the lab are archived digitally and uploaded to the NCPN-water database, where they are matched with the field data. Subsequently, lab results are transferred to WRD-STORET.



* Calibration data are of two types: *pre-mobilization* or *on-site*, both of which are stored in the Field Form spreadsheet. Thus, the digital Field Form Archive includes these data items.
** Created when Field Form is archived.

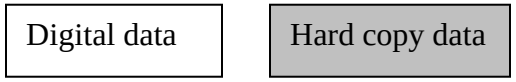


Figure 6. Calibration Data - UT Parks

There are two types of calibration data: *pre-mobilization*, performed prior to the site visit, and *on-site*, performed at the site. Since both types of data are recorded in Field_forms.xls, the digital archive of these data items is included in the Field Form Archive. A hard copy archive is made of the calibration data in the spreadsheet, and a digital archive of the Troll Calibration Report is made. While calibration data are uploaded to the NCPN-water database, these data are not uploaded to WRD-STORET since WRD-STORET does not currently support the storage of such data.

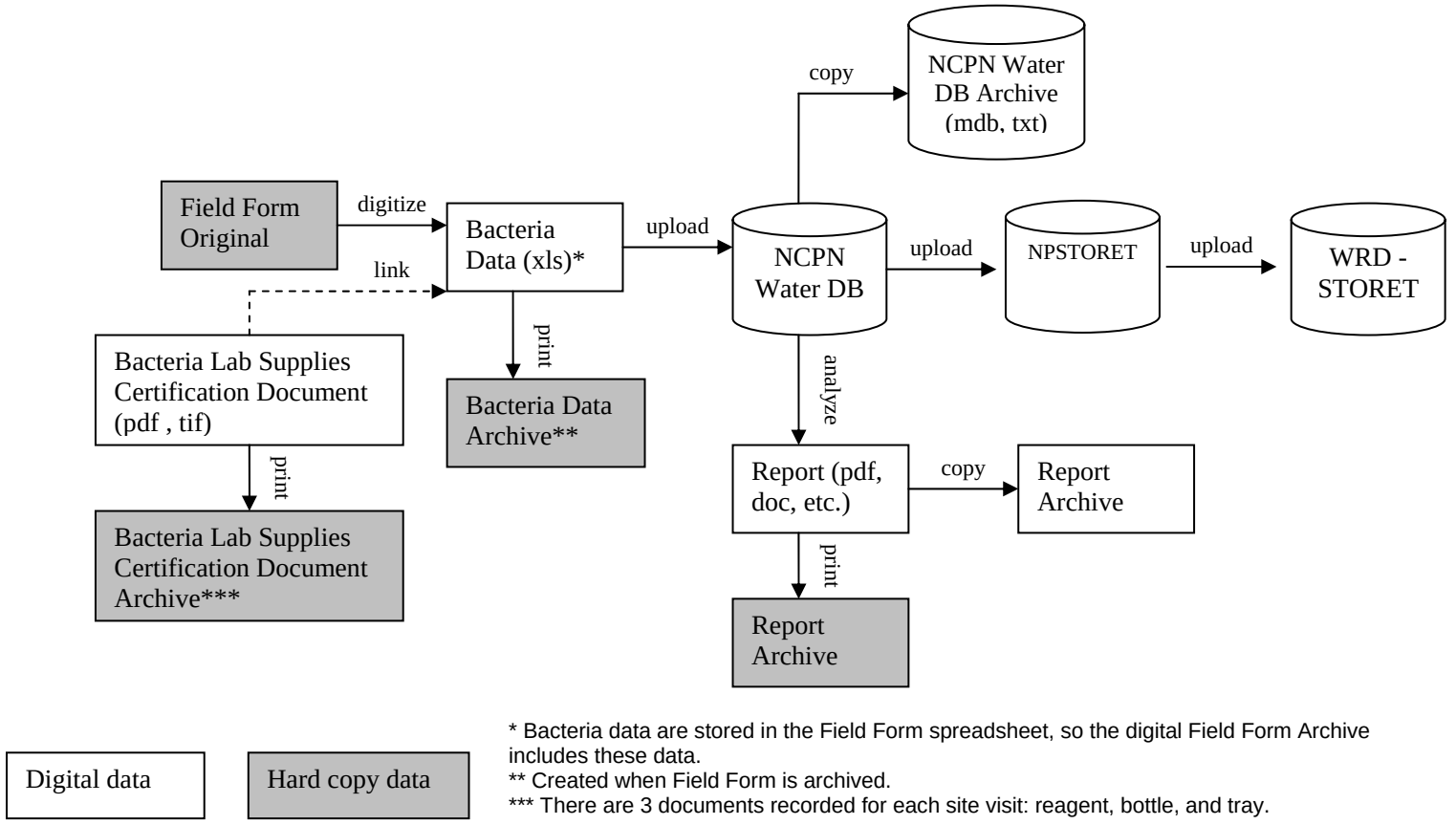


Figure 7. Bacteria Data - UT Parks

In the field, measurements are recorded on hard copy field forms (the same field forms discussed previously). These data, along with the digital file locations of the associated bacteria lab supplies certification documents, are then entered manually into the bacteria data portion of Field_forms.xls. Hard copy archives are made of the bacteria data and the bacteria supplies certification documents.

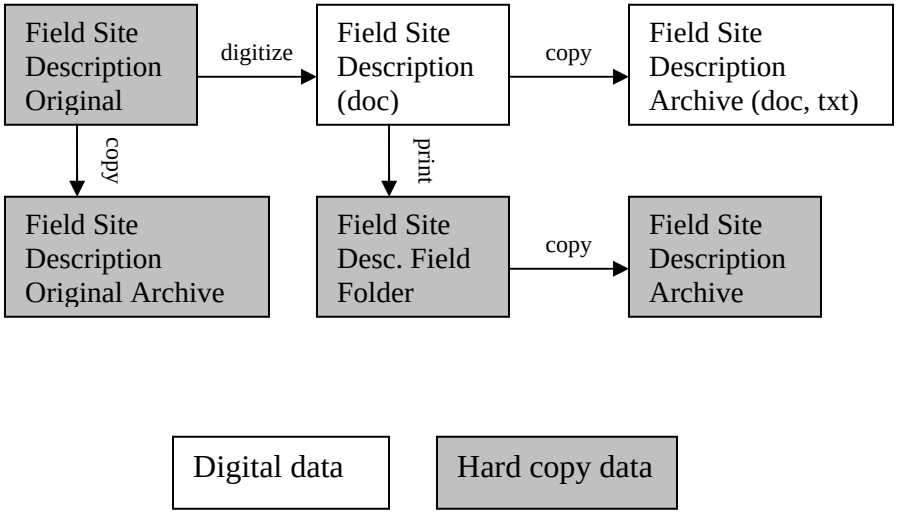


Figure 8. Site Selection Data - All Parks

In the field, data are recorded on a hard copy Field Site Description form. These data are subsequently entered into a Word document and a hard copy is printed for the Field Folder. Hard copy archives are made of the original Field Site Description form and the digital Field Site Description form. In addition, digital archives of the Field Site Descriptions are also made in both native (doc) and ASCII text (txt) formats.

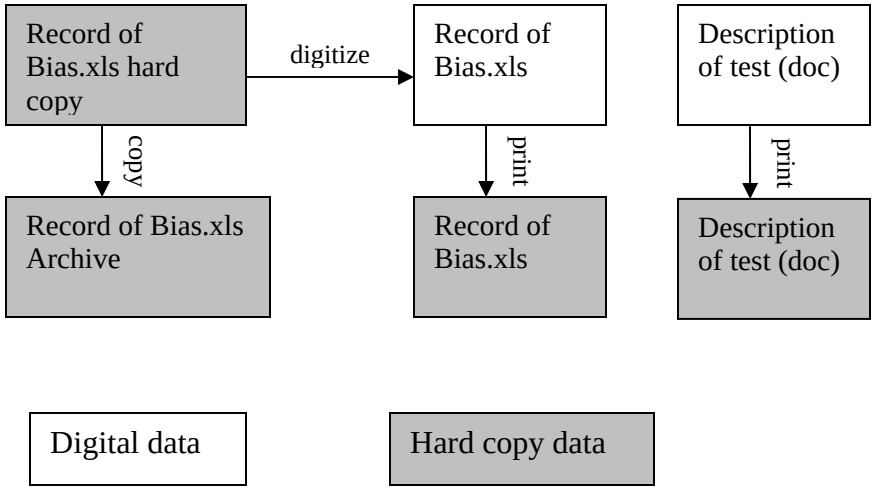


Figure 9. Record of cumulative bias – all park units.

Table 1. Summary of Archiving Information for Digital Data - UT Parks

Data Category	Data Item	File Format	Archive Location[1]	Comments
Field	Field Form	xls	Data\Field_Forms\<*year*>[2]\<*park_unit*>[3]\	Contains field data, lab sheet, run sheet, calibration data, and bacteria data.
	Troll Data Report	bin	Data\Troll\Data\<*year*>\<*month*>[4]\<*park_unit*>\	
	Aquacalc Flow Data	txt	Data\Aquacalc\<*year*>\<*month*>\	Not applicable to SEUG.
	Photographs	jpg, tif	R:\Archive\NCPN_Photos_Archive\NCPN Photos\Monitoring\Water_Quality\	Archive location is an absolute path.
Lab	Lab Sheet	xls	See Field Form archive location.	Contained in Field Form xls file.
	Run Sheet	xls	See Field Form archive location.	Contained in Field Form xls file.
	Lab Results	xls	\Data\Data_Lab\<*year*>\<*park_unit*>\	
Calibration	Calibration Data	xls	See Field Form archive location.	Contained in Field Form xls file.
	Troll Calibration Report	html	Data\Troll\Calibration Reports\<*year*>\<*month*>\<*park_unit*>\	
Bacteria	Bacteria Data	xls	See Field Form archive location.	Contained in Field Form xls file.
	Bacteria Lab Supplies Certification Document	pdf, tif	Data\Data_Lab\Certificates\<*year*>\<*park_unit*>\	
Site Selection	Field Site Description	doc	Data\Field_Site_Descriptions\<*year*>\<*park_unit*>\	
Consolidated	NCPN Water Quality Database	mdb	Data\Database\<*year*>\	Not applicable to SEUG, since database is maintained by NCPN.
	Annual Report	pdf, doc, etc.	Reports\<*year*>\	Not applicable to SEUG, since consolidated reports are the responsibility of NCPN. Any other consolidated reports (e.g., triennial report) will also be archived in this location.

[1] Unless stated otherwise, archive location is given relative to the Water Quality archive path on the NCPN shared drive (*X:\Archive\Monitoring_Archive\Water_Quality*).

[2] <*year*> indicates year in yyyy format.

[3] <*park_unit*> indicates park or managing unit (e.g., SEUG, NCPN).

[4] <*month*> indicates month name.

Table 2. Summary of Archiving Information for Hard Copy Data - UT Parks

Data Category	Data Item	Comments
Field	Field Forms	
	Instrument Logbook	Includes logbooks for all instruments used (Aquacalc, Troll, and turbidity meter).
Lab	Lab Sheet	Generated from Field Forms xls file.
	Run Sheet	Generated from Field Forms xls file.
Calibration	Calibration Data	Generated from Field Forms xls file. Includes both pre-mobilization and on-site calibration data.
Bacteria	Bacteria Data	Generated from Field Forms xls file.
	Bacteria Lab Supplies Certification Document	Includes 3 certification documents (reagent, bottle, and tray).
Site Selection	Field Site Description - Original	
	Field Site Description - Field Folder Copy	
Consolidated	Annual Report	Not applicable to SEUG, since consolidated reports are the responsibility of NCPN. Hard copies of any other consolidated reports (e.g., triennial report) will also be archived in the NCPN central files.

6.2. Data Archiving for Colorado Parks

For Colorado parks, flow diagrams are presented for each of the types of data collected: field (Figure 10), lab (Figure 11), calibration (Figure 12), bacteria (Figure 13), and reservoir profile (Figure 14). Since most of the data flows are similar to those described in the previous section for Utah parks, only the main points are highlighted in the captions below each figure. Site selection data is also applicable to Colorado parks, but it is not discussed in this section since it is the same for all parks (refer to Figure 8 in the previous section).

To help ensure that all relevant data items are archived appropriately, archiving procedures for Colorado parks are summarized below for digital and hard copy data:

- Digital Data: Table 3 summarizes the archiving information for digital data for Colorado parks, including file formats and archive locations. All digital data will be archived on the NCPN shared drive (X:) in the water quality archive folder (*X:\Archive\Monitoring_Archive\Water_Quality*). Digital file archives will be stored in both native and ASCII text formats. At least once per year, the BLCA/CURE hydrologist will transfer the applicable files listed in Table 3 to the NCPN data manager, who will archive them accordingly.

- Hard Copy Data: Table 4 summarizes the archiving information for hard copy data for Colorado parks. NCPN will maintain archival-quality copies of the data items listed in Table 4 in the NCPN central files. At least once per year, the BLCA/CURE hydrologist will provide archival-quality copies (i.e., on acid-free paper) of the applicable data items listed in Table 4 to the NCPN data manager. Existing park procedures should be followed for archival of the original hard copies and any other archival-related requirements. Park curators should be consulted for specific details on these procedures. Alternatively, the BLCA/CURE hydrologist may send the original hard copies to the NCPN data manager (at least once per year), who will make archival copies. Such arrangements will need to be approved by the appropriate park staff (e.g., curators) to ensure the timely return of originals to the park.

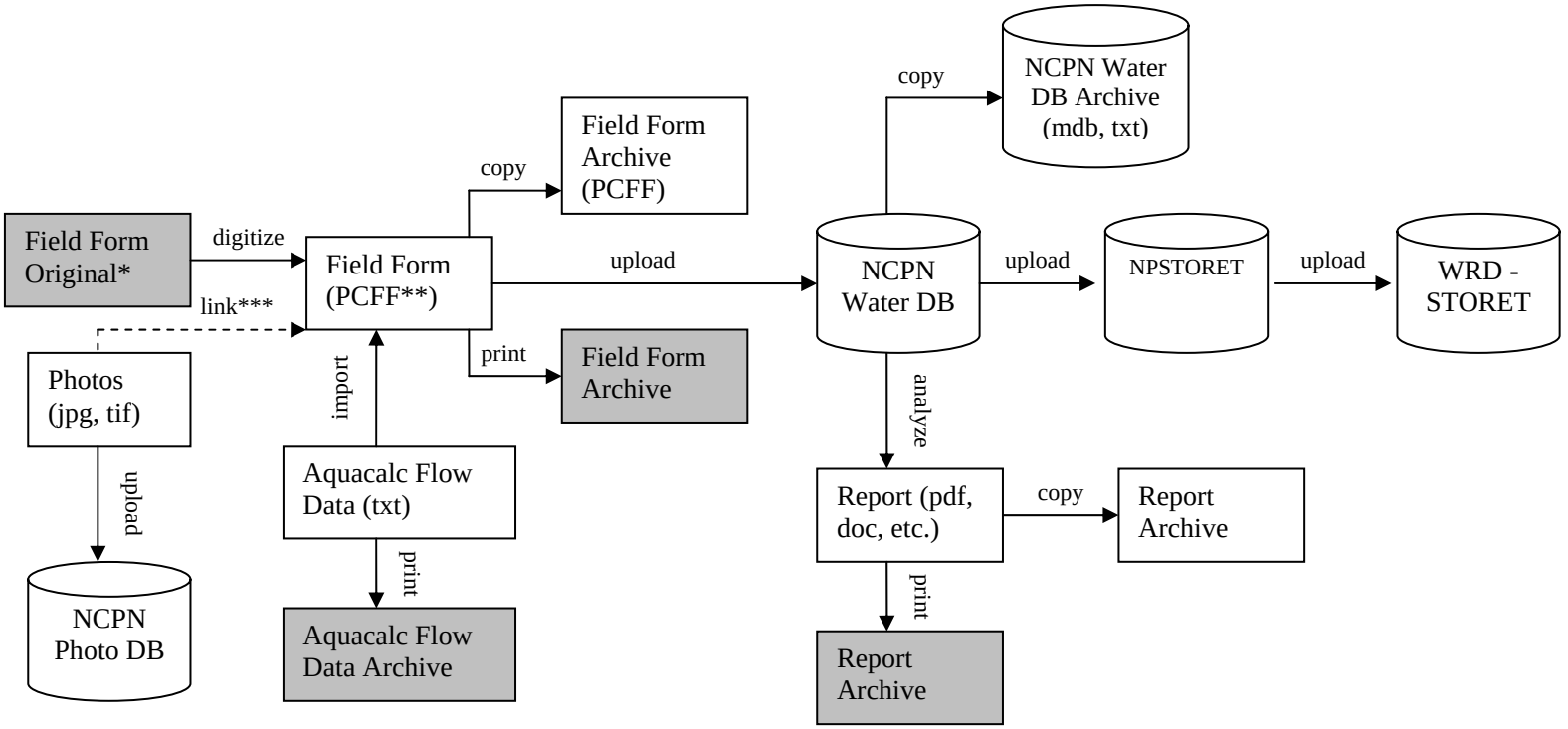


Figure 10. Field Data - CO Parks

Blank field forms are printed from PCFF and used to manually record measurements in the field. These data are then entered into PCFF, while Aquacalc flow data are imported into PCFF. A hard copy archive of the Aquacalc flow data is created. Field data stored in PCFF are archived in both digital and hard copy formats, and uploaded into the NCPN-water database. For any photos taken during the site visit, archive copies of the photos will be uploaded to the NCPN Photo database. The photo database renames the archived photos according to NCPN photo file naming conventions. The newly assigned filenames are then recorded in the NCPN-water database (via an automated process), thus providing links to the archived photos in the photo database (this link is not shown).

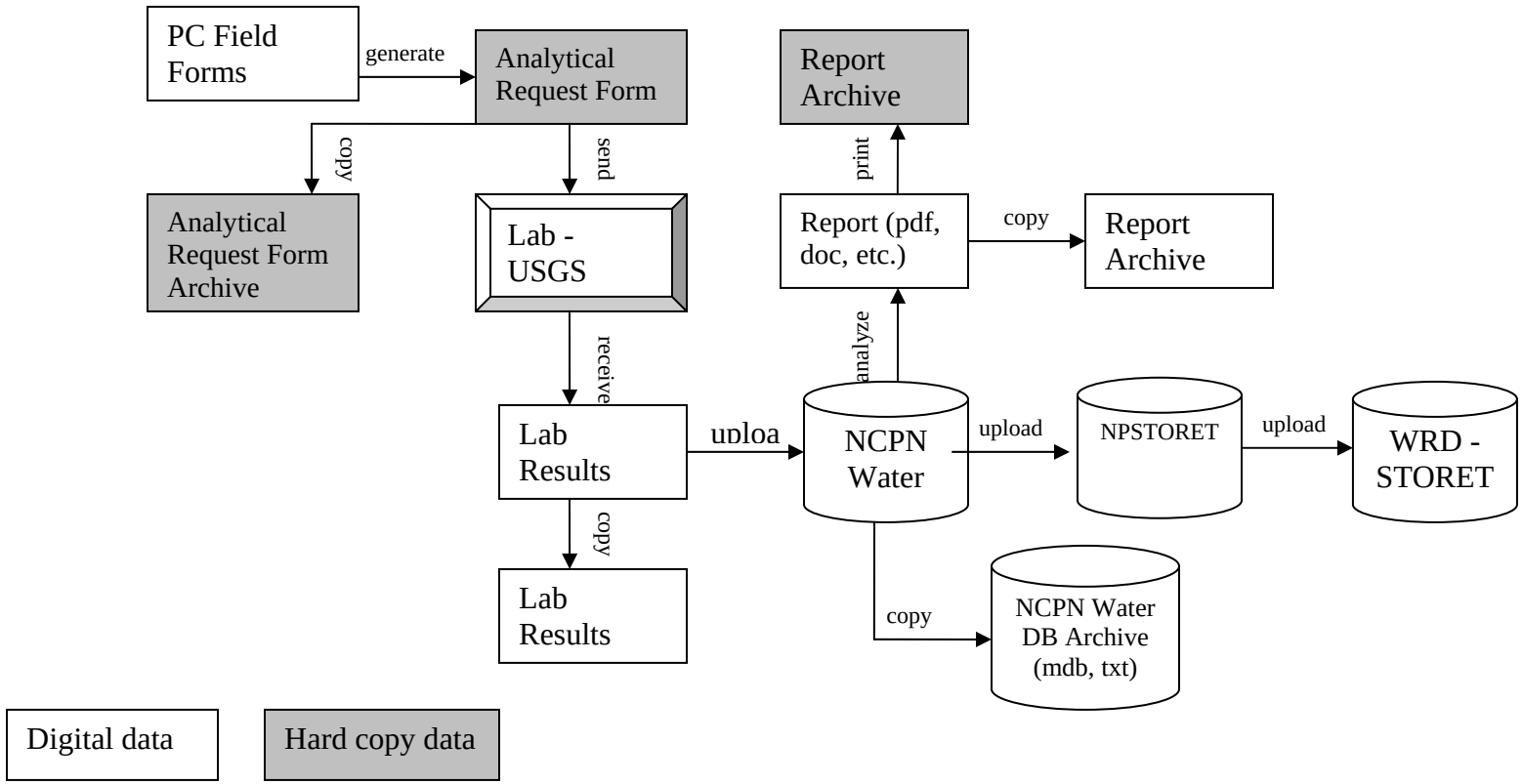


Figure 11. Lab Data - CO Parks

PCFF generates a hard copy Analytical Request Form, which is sent to the lab (USGS NWQL) along with the samples. A hard copy archive of the Analytical Request Form is created. Lab results received from the lab are archived digitally and uploaded to the NCPN-water database, where they are matched with the field data.

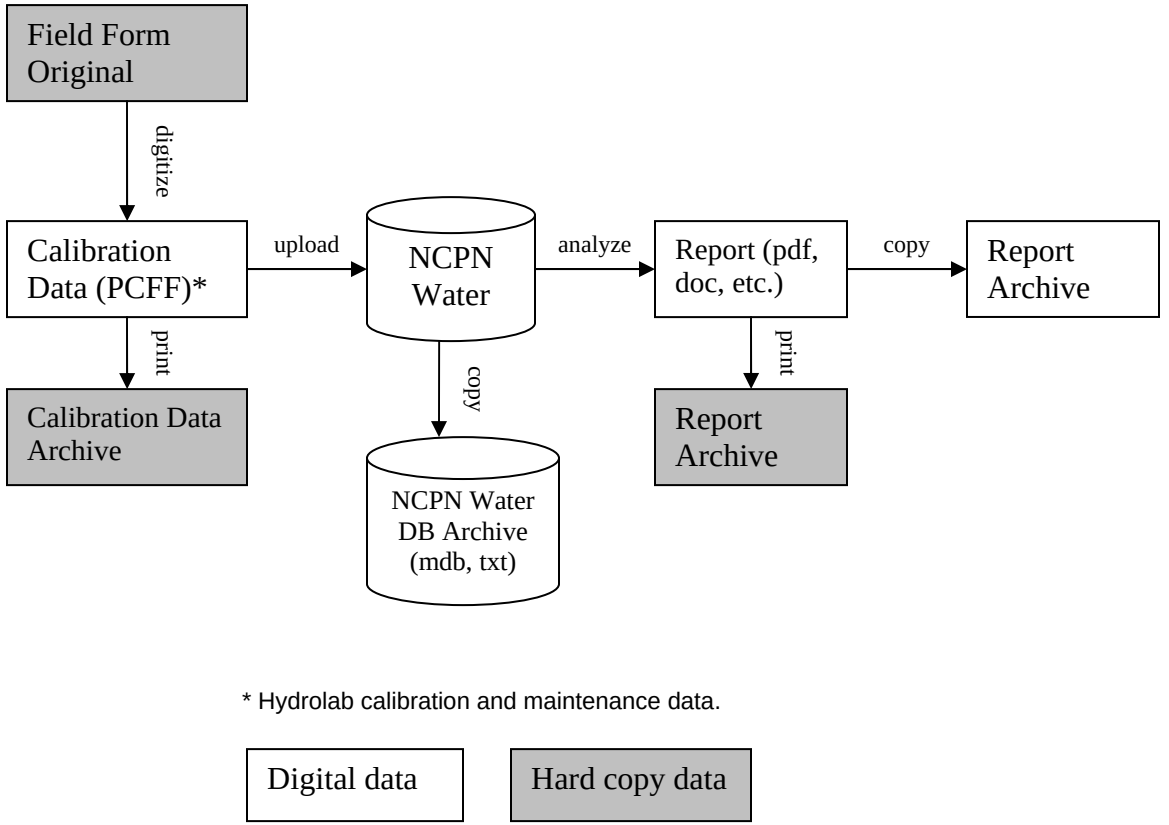


Figure 12. Calibration Data - CO Parks

Calibration data from the Hydrolab are manually entered into PCFF from the hard copy field forms. Maintenance data for the Hydrolab are also entered into PCFF. A hard copy archive of the calibration data is created from PCFF. Calibration data are uploaded from PCFF to the NCPN-water database.

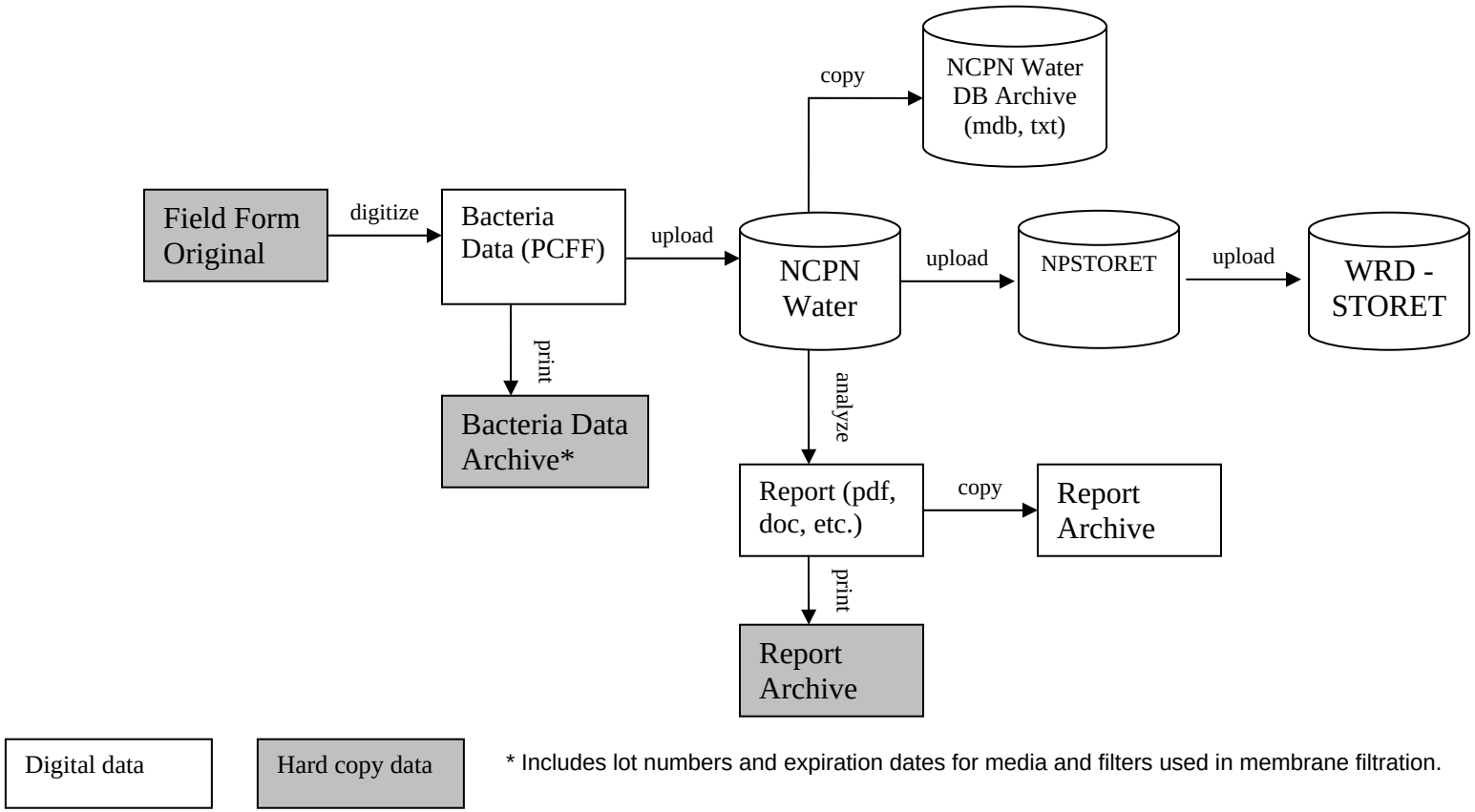


Figure 13. Bacteria Data - CO Parks

Bacteria data are manually entered into PCFF from the hard copy field forms. A hard copy archive of the bacteria data is created from PCFF. The data are uploaded from PCFF to the NCPN-water database.

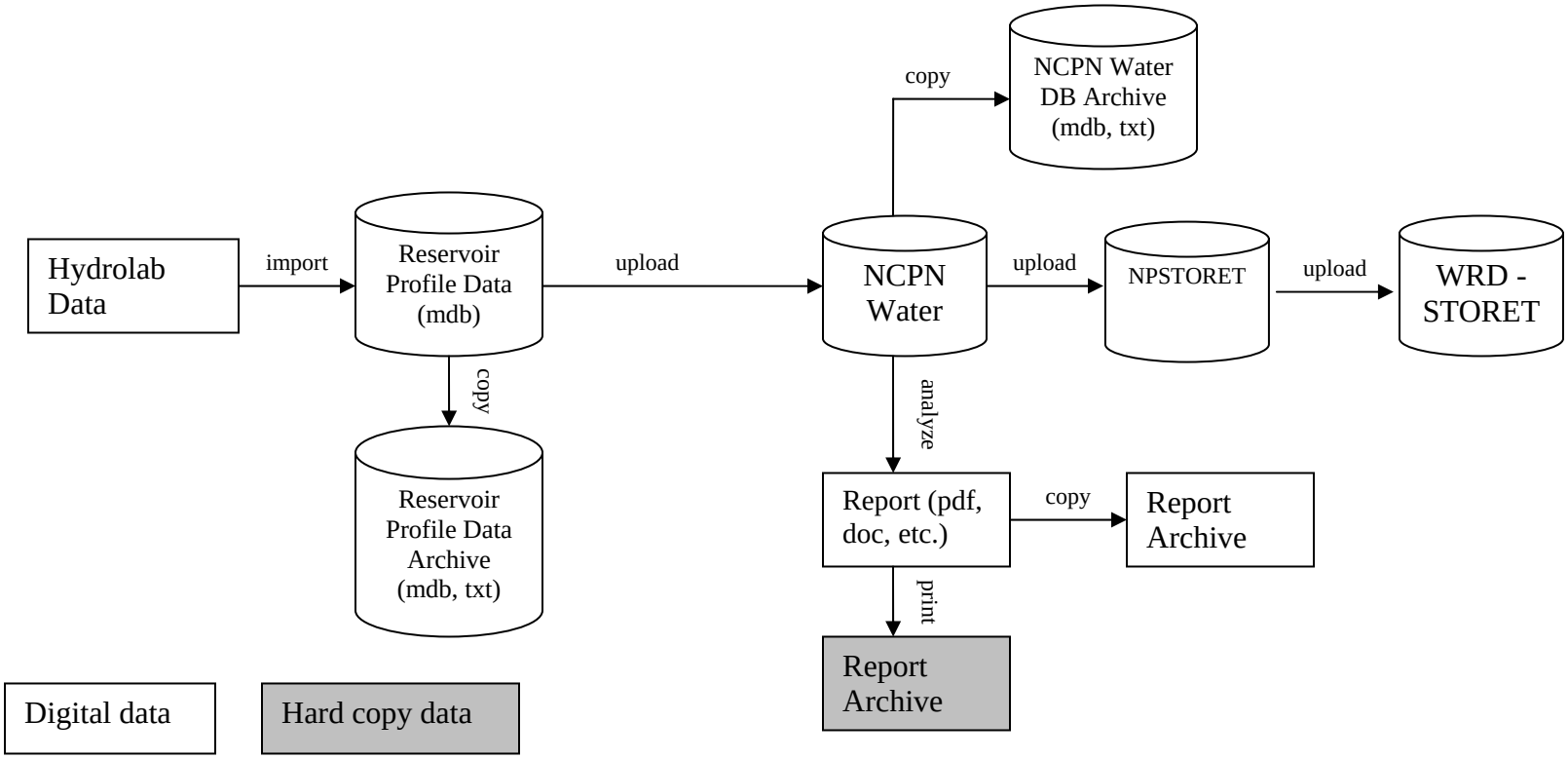


Figure 14. Reservoir Profile Data - CO Parks

Reservoir profile data are recorded using the Hydrolab, and then imported into a Microsoft Access database. Digital archives of this database are created in both native (mdb) and ASCII (txt) formats. The reservoir profile data are uploaded from the database to the NCPN-water database.

Table 3. Summary of Archiving Information for Digital Data - CO Parks

Data Category	Data Item	File Format	Archive Location[1]	Comments
Field	Field Form	PCFF	Data\Field_Forms\<*year*>[2]\<*park_unit*>[3]\	Contains field, calibration, and bacteria data.
	Photographs	jpg, tif	R:\Archive\NCPN_Photos_Archive\NCPN Photos\Monitoring\Water_Quality\	Archive location is an absolute path.
Lab	Lab Results	xls	\Data\Data_Lab\<*year*>\<*park_unit*>\	
Calibration	Calibration Data	PCFF	See Field Form archive location.	Contained in PCFF.
Bacteria	Bacteria Data	PCFF	See Field Form archive location.	Contained in PCFF.
Site Selection	Field Site Description	doc	Data\Field_Site_Descriptions\<*year*>\<*park_unit*>\	
Reservoir Profile	Reservoir Profile Data	mdb	Data\Reservoir_Profile\<*year*>\<*park_unit*>\	

[1] Unless stated otherwise, archive location is given relative to the Water Quality archive path on the NCPN shared drive (*X:\Archive\Monitoring_Archive\Water_Quality*).

[2] <*year*> indicates year in yyyy format.

[3] <*park_unit*> indicates park or managing unit (e.g., BLCA_CURE).

Table 4. Summary of Archiving Information for Hard Copy Data - CO Parks

Data Category	Data Item	Comments
Field	Field Forms	Generated from PCFF.
	Aquacalc Flow Data	
Lab	Analytical Request Form	Generated from PCFF.
Calibration	Calibration Data	Generated from PCFF.
Bacteria	Bacteria Data	Generated from PCFF.
Site Selection	Field Site Description - Original	
	Field Site Description - Field Folder Copy	

6.3. Data Archiving for USGS-Sampled Parks

DINO is currently the only park for which all sample collection and analysis work is handled by USGS. At least once per year, NCPN staff will download this data from NWIS. The raw data downloaded from NWIS (in ASCII text format) will be archived in the following location on the NCPN shared drive: *X:\Archive\Monitoring_Archive\Water_Quality\Data\USGS_Data\<year>*.

Since this data will be uploaded to the NCPN-water database, it will also be archived as part of the NCPN-water database archive.

7. Metadata

Each table and field in the NCPN-water database will be defined and documented. Documentation of the tables and fields in the STORET and NWIS databases will be maintained by the respective organizations responsible for those databases.

NCPN will create and maintain a Dataset Catalog record for the water quality monitoring data stored in NCPN-water. This Dataset Catalog record will be converted to the standard XML format and uploaded to the NPS Data Store.

WRD-STORET requires that water quality metadata be stored along with the actual results data. Thus, NCPN will create WRD-STORET metadata as part of the data transfer process (using NPSTORET). NCPN will also create and maintain a metadata record in the NPS Data Store for the WRD-STORET data/metadata.

Some NCPN parks have active Dataset Catalog records for their water quality monitoring programs. NCPN will update these records on an annual basis.

The complete protocol for this project (Protocol Narrative and SOP #s 1-17) is an integral component of the project metadata. All narrative and SOP version changes are noted in the Master Version Table (MVT), which is maintained in SOP #16. Any time the narrative or SOP versions change, a new Version Key number (VK#) must be created and recorded in the MVT, along with the date of the change and the versions of the narrative and SOPs in effect. The Version Key number is essential for project information to be properly interpreted and analyzed. *The protocol narrative, SOPs, and data should not be distributed independently of this table*.

8. Data Management Calendar

Due to the complexity of managing the different data flows in this program a calendar (Table 5) has been developed to help track steps in data management. The steps correspond to tasks outlined in previous sections and for the most part logically occur in a fixed chronological order. Some tasks and associated data management issues are necessarily ad hoc such as documenting bias when staff, instruments or methods change unpredictably.

Table 5 Calendar of data management events that must be performed on a routine basis.

Task (responsibility)	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	TBD	Comments
Field and lab data review for all but DINO (regional hydrologists)	√	√	√	√	√	√	√	√	√	√	√	√		Monthly within 3 days of sampling run or receipt from lab
Field and lab data review for DINO (NCPN hydrologist)	√	√	√	√	√	√	√	√	√	√	√	√		Monthly within 3 days of sampling run or download from NWIS
Field and lab data upload to NCPN-water (NCPN hydrologist / data management staff)	√	√	√	√	√	√	√	√	√	√	√	√		Monthly after data review
Quality Control checks on NCPN-water (NCPN hydrologist)	√	√	√	√	√	√	√	√	√	√	√	√		Monthly after upload of field and lab data
Photo archive (regional hydrologists)	√	√	√	√	√	√	√	√	√	√	√	√		Monthly within 3 days of sampling run
Manual verification Section 4.1 (NCPN hydrologist)		√												Annually spot check data entry
Summary queries in NCPN-water Section 4.1 (NCPN hydrologist)		√												Annually after spot checks of data entry
NCPN-water to NPSTORET (NCPN hydrologist / data management staff)			√											Annually after QC in NCPN-water
Data quality meeting (all hydrologists)			√											Annually after QC in NCPN-water
Data quality report (NCPN hydrologist)			√											Annually after QC in NCPN-water
Equipment log book archive		√												Photocopy log book entries annually
Version control (NCPN hydrologist)				√										Annually after updates to protocol made
Bias control													√	As needed when staff, methods or equipment changes
Working backup of NCPN-water													√	Monthly or as needed when changes are made to NCPN-water
Create/update metadata (NCPN hydrologist / data management staff)			√											Annually after QC in NCPN-water
Archive of NCPN-water (data management staff)			√											Annually after QC and metadata
Site selection and site description (regional hydrologists)													√	As needed when sites are added or locations change

9. Citations

NCPN-NPS 2007. Directory Structure Guidance Document, Version 1.0 (draft). National Park Service, Inventory and Monitoring Program, Northern Colorado Plateau Network, Moab, UT.

10. Appendix A: Documentation of NCPN-water Database Tables

This appendix provides a description of each field for each table in the NCPN-water database. The tables appear in alphabetical order. This documentation does not include the tables and fields that will be added as part of the modifications mentioned previously. These new tables and attributes will be included in the final version of this document.

10.1. Table Name: CategoryRanks

Description: Use with ExportToSPlus form to rank categories

Field Name	Field Description	Field Type	Field Width
Category	Constituent grouping (e.g., nutrient, trace elements, inorganics,organics,pesticides,etc.); includes matrix (from PARAMETER table)	dbText	50
Rank	Rank used by SPlusExporter to order categories for selection in form (same rank can be used more than once; alphabetical order default)	dbInteger	2

10.2. Table Name: CONSOLIDATED_SITES

Description: Record of ALL sites affected by site consolidation process (updated as of 12/24/03)

Field Name	Field Description	Field Type	Field width
Site_Id	Unique identifier for specific sites (default is USGS Site ID, when more than one ID available)	dbText	255
LatLong_Cat	Concatenated DMS latitude/longitude (used for site consolidation site_ids where USGS site not available)	dbText	50
Site_Name	Name of sample site (default is USGS Site Name, when more than one name available)	dbText	255
Site_Description	Description of site location	dbText	200
Park_Code	NCPN 4-digit acronym for NPS units (predominant park_code will be used for sites upstream of more than one park)	dbText	4
Park_Boundary	Identifies which sites are within the park boundaries ("yes" or "no"); based on review of updated boundary coverage downloaded from NPS GIS web site in 12/2003	dbBoolean	1
Latitude_DMS	Latitude (in degrees, minutes, seconds)	dbText	6
Longitude_DMS	Longitude (in degrees, minutes, seconds)	dbText	7
Latitude_DD	Latitude (decimal degrees)	dbDouble	8
Longitude_DD	Longitude (decimal degrees)	dbDouble	8
UTM_x	NAD 1983	dbDouble	8
UTM_y	NAD 1983	dbDouble	8
Datum	Datum	dbText	10
UTM_Zone	UTM Zone (limited to zones 12 or 13 for the NCPN)	dbText	20
HUC	Hydrologic Unit Code	dbText	8
State	State	dbText	2
County	County (based on FIPS County Code)	dbText	50
Site_Type_Code	Site Type Code (DV = diversion; SW= surface water (streams, rivers); GW = ground water; SP = spring; SEEP = seep; MINE = mine; OF= outfall (sewage, other point discharges); LK = Reservoir/Lake; PL = Place of water use (intakes, etc.); UNK = unknown)	dbText	4
Geologic_Unit	geologic unit	dbText	50
Well_Depth	total depth recorded for well (in feet)	dbDouble	8
Elevation	elevation of site location (in feet)	dbLong	4
Drainage_Basin_Area	area of drainage basin above site (in square miles)	dbLong	4

10.3. Table Name: COUNTY_CODES

Description: National FIPS county and state code reference file (downloaded from USGS website 12/9/03)

Field Name	Field Description	Field Type	Field Width
country	Country abbreviation	dbText	255
state_code	FIPS state code	dbText	255
county_code	FIPS county code	dbText	255
county_name	County name	dbText	255
county_name_abb	County name abbreviation	dbText	50
state_abb	State abbreviation	dbText	255

10.4. Table Name: EPACODES

Description: List of EPA parameter codes, used for updating PARAMETER table for new Ppcodes

Field Name	Field Description	Field Type	Field Width
Ppcode	Parameter code	dbText	255
ParameterName	Parameter name	dbText	255
ReportUnits	Reporting units for Ppcode	dbText	255
rptlev	Reporting level for Ppcode	dbDouble	8
medium	Sample medium for Ppcode	dbText	255
psname	Parameter short name (as given by USEPA)	dbText	255

10.5. Table Name: HUC_CODES

Description: National HUC and basin reference file (downloaded from USGS website 12/9/03)

Field Name	Field Description	Field Type	Field Width
huc	Hydrologic Unit Code	dbText	255
basin	River basin name	dbText	255

10.6. Table Name: ORPHANED_SITES

Description: Record of orphaned sites from site consolidation process - Deleted from SITE on 12/24/03

Field Name	Field Description	Field Type	Field Width
Site_Id	Unique identifier for specific sites (default is USGS Site ID, when more than one ID available)	dbText	255
LatLong_Cat	Concatenated DMS latitude/longitude (used for site consolidation site_ids where USGS site not available)	dbText	50
Site_Name	Name of sample site (default is USGS Site Name, when more than one name available)	dbText	255
Site_Description	Description of site location	dbText	200
Park_Code	NCPN 4-digit acronym for NPS units (predominant park_code will be used for sites upstream of more than one park)	dbText	4
Park_Boundary	Identifies which sites are within the park boundaries ("yes" or "no"); based on review of updated boundary coverage downloaded from NPS GIS web site in 12/2003	dbBoolean	1
Latitude_DMS	Latitude (in degrees, minutes, seconds)	dbText	6
Longitude_DMS	Longitude (in degrees, minutes, seconds)	dbText	7
Latitude_DD	Latitude (decimal degrees)	dbDouble	8
Longitude_DD	Longitude (decimal degrees)	dbDouble	8
UTM_x	NAD 1983	dbDouble	8
UTM_y	NAD 1983	dbDouble	8
Datum	Datum	dbText	10
UTM_Zone	UTM Zone (limited to zones 12 or 13 for the NCPN)	dbText	20
HUC	Hydrologic Unit Code	dbText	8
State	State	dbText	2
County	County (based on FIPS County Code)	dbText	50
Site_Type_Code	Site Type Code (DV = diversion; SW= surface water (streams, rivers); GW = ground water; SP = spring; SEEP = seep; MINE = mine; OF= outfall (sewage, other point discharges); LK = Reservoir/Lake; PL = Place of water use (intakes, etc.); UNK = unknown)	dbText	4
Geologic_Unit	geologic unit	dbText	50
Well_Depth	total depth recorded for well (in feet)	dbDouble	8
Elevation	elevation of site location (in feet)	dbLong	4
Drainage_Basin_Area	area of drainage basin above site (in square miles)	dbLong	4

10.7. Table Name: PARAMETER

Description: Table of parameters, codes, report units, categories,and matrices

Field Name	Field Description	Field Type	Field Width
Parameter_Code	Numeric parameter code	dbText	8
Ppcode	Numeric parameter code, Booker convention (Precede with P, for constituent value code)	dbText	8
Category	Constituent grouping (e.g., nutrient, trace elements, inorganics,organics,pesticides,etc.); includes matrix	dbText	50
Matrix	Sample matrix (water, waste, sediment, tissue)	dbText	35
Parameter_Short_Name	Most complete list of concise constituent parameter names (use as column heading for queries)	dbText	25
Parameter_Long_Name	Full parameter description	dbText	255
USGS_Parmfile_Name	Full parameter description from USGS NWIS qwdata parmfile.master table (updated 0ct 2003)	dbText	255
USGS_Master_File_Name	Outdated USGS parm names (from qwdata parm code dictionary dump, pulled spring 2003)	dbText	255
EPACODES_Name	Available EPA parm codes dictionary	dbText	255
Reporting_Units	Reporting units for parameter	dbText	25
RARE	<100 result occurrences	dbBoolean	1
Schedules	USGS laboratory analysis schedules used for parameter(s) at the National Water Quality Laboratory	dbText	50
Parameter_Source	Source of associated parameter code information in PARAMETER table. "USEPA" or "USGS" or "created' (to identify constituent code created as existing Pcodes not applicable)	dbText	50

10.8. Table Name: PARKS

Description: Lookup Table of Park Codes

Field Name	Field Description	Field Type	Field Width
Park	Name of park	dbText	50
Park_Code	4-digit park code (established for NCPN)	dbText	4

10.9. Table Name: QW_RESULT

Description: Table of Results

Field Name	Field Description	Field Type	Field Width
Site_Id	Site identification number (must be unique for each site)	dbText	255
Collection_Date	Sample collection date	dbDate	8
Collection_Time	Sample collection time (default value is 2359)	dbLong	4
Source_Agency	Person, agency or organization supplying original file appended to database (see SOURCE_AGENCIES lookup table for definition of all agencies used)	dbText	255
Sample_Depth	Depth of sample collection (zero may not be meaningful; default value = -999 when no depth provided)	dbLong	4
Ppcode	Parameter code assigned by agency or organization (unique for each parameter based on sample type, units and analysis)	dbText	8
Opt_flag	Flag to indicate the order of Ppcodes used to aggregate selected constituents (see category "Aggregated Parameters" in PARAMETER table for Ppcodes and order used)	dbText	50
Value_Uncensored	Results from original databases	dbDouble	8
Value_Remark	Results remarks (see VALUE_REMARKS lookup table for definition of all remarks used)	dbText	1
Value_Cens_Neg	Results where censored values (<, K, M, T, and U value remarks) are flagged by multiplying original (i.e., uncensored) value by negative one	dbDouble	8
Value_Cens_Half	Results where censored values (<, K, M, T, and U value remarks) are multiplied by one-half, a common calculation applied for selected statistical analyses	dbDouble	8
Value_Cens_Zero	Results where censored values (<, K, M, T, and U value remarks) are set to zero; the practice used by some states (e.g., CO) for selected standard analyses	dbDouble	8
Sample_Type	Type of sample collected (see SAMPLE_TYPES lookup table for definition of all types used)	dbText	50
PSource	Flag to indicate original Ppcode used to optimize selected constituents obtained with modCalcBestVariables module (see also tblCodeEquivalents)	dbText	10

10.10. Table Name: SAMPLE

Description: Table of Sample Information

Field Name	Field Description	Field Type	Field Width
Site_Id	Site identification number (must be unique for each site)	dbText	255
Collection_Date	Sample collection date	dbDate	8
Collection_Month	Month of sample (extracted from Date)	dbText	50
Collection_Year	Year of sample (extracted from Date)	dbText	50
Collection_Time	Sample collection time (default value for unknown times is 2359)	dbLong	4
Source_Agency	Person, agency or organization supplying original file appended to database	dbText	255
Sample_Depth	Sample depth (UNITS in FT; zero values may not be meaningful; default value for unspecified depths is -999)	dbLong	4
Sample_Type	Type of sample collected (see SAMPLE_TYPES look-up table for options)	dbText	50
Historic_Staid	Historic station identifier (i.e., site id) for merged sites	dbText	255
Alt_SiteId1	First alternative site identification provided with original database retrieval	dbText	255
Alt_SiteId2	Second alternative site identification provided with original database retrieval	dbText	255
Alt_SiteId3	Third alternative site identification provided with original database retrieval	dbText	50
Comments	Comments regarding sample	dbText	255
Stream_Condition	Stream condition	dbText	255
Weather_Conditon	Weather condition	dbText	255
Sample_Medium	Sample Medium Code (USGS NWIS parameter MEDIM; e.g., R = surface water)	dbText	1
Record_Number	Unique Sample Identifier (USGS NWIS parameter SAMPL)	dbText	50
Analysis_Status	Analysis Status Code (USGS NWIS parameter ASTAT)	dbText	50
Hydrologic_Event	Hydrologic Event Code (USGS NWIS parameter EVENT)	dbText	1
Hydrologic_Status	Hydrologic Status Code (USGS NWIS parameter HSTAT)	dbText	50
Original_File	Name of source file of information received and appended to database	dbText	255
Original_Database	Name of source database for information retrieved and appended to NCPN database (e.g., Legacy STORET, Modern STORET, NWIS)	dbText	50
Data_Retrieval_Date	Date of data retrieval (or date of receipt of data from outside source)	dbDate	8

10.11. Table Name: SAMPLE_TYPES

Description: Lookup Table of Sample Type Codes

Field Name	Field Description	Field Type	Field Width
Sample_Type	Sample type code based on USGS sample types (Exceptions: TS = Time Series data and PR = partial replicates are not a USGS sample type code; note that USGS sample type 7 is from NWIS DB1 and represents actual sample that has a dup, not a duplicate result)	dbText	50
Sample_Type_Description	Descriptions based on USGS sample types. All other (non-USGS) data in database coded to these USGS sample types	dbText	255

10.12. Table Name: SITE

Description: Table of Site/Station Information

Field Name	Field Description	Field Type	Field Width
SiteNo	Sequentially assigned site number that serves no discernable purpose in this database.	dbLong	4
Site_Id	Unique identifier for specific sites (default was USGS Site ID, when more than one ID available). Will be the STORET_Site_ID on new sites (1/26/07).	dbText	255
NCPN_Site_ID	This column corresponds to the NCPN_Site_ID column in table 10 of the NCPN Water Quality Protocol Narrative. Must be unique, nulls ok. 1/26/07	dbInteger	2
STORET_Site_ID	NPSTORET ID number for this park. Must be unique, nulls ok. On new sites (1/26/07), this will be the same value as Site_Id.	dbLong	4
LatLong_Cat	Concatenated DMS latitude/longitude (used for site consolidation site_ids where USGS site not available)	dbText	50
Site_Name	Name of sample site (default is USGS Site Name, when more than one name available)	dbText	255
Site_Description	Description of site location	dbText	200
Park_Code	NCPN 4-digit acronym for NPS units (predominant park_code will be used for sites upstream of more than one park)	dbText	4
Park_Boundary	Identifies which sites are within the park boundaries ("yes" or "no"); based on review of updated boundary coverage downloaded from NPS GIS web site in 12/2003	dbBoolean	1
Latitude_DMS	Latitude (in degrees, minutes, seconds)	dbText	6
Longitude_DMS	Longitude (in degrees, minutes, seconds)	dbText	7
Latitude_DD	Latitude (decimal degrees)	dbDouble	8
Longitude_DD	Longitude (decimal degrees)	dbDouble	8
Geopositioning Method	Method used to obtain latitude, longitude location (not available for Legacy STORET sites; see COORDINATE_METHOD_CODES lookup table for code descriptions)	dbText	255
coord_acy_cd	Lat/Long accuracy (see COORDINATE_ACCURACY_CODES lookup table for code descriptions)	dbText	50
UTM_x	NAD 1983	dbDouble	8
UTM_y	NAD 1983	dbDouble	8
Datum	Datum - field added 1/2007; Horizontal Datum applicable to latitude, longitude location (not available for Legecy STORET sites; NAD27 = North American Datum of 1927, NAD83 = North American Datum of 1983)	dbText	10
UTM_Zone	UTM Zone (limited to zones 12 or 13 for the NCPN)	dbText	20
land_net_ds	Land-net location (NE, NW, SE, and SW are used for the quarter, quarter-quarter, and quarter-quarter-quarter descriptions; smallest	dbText	23

Field Name	Field Description	Field Type	Field Width
	subdivision is listed first)		
map_nm	Name of location map	dbText	20
map_scale_fc	Scale of location map	dbText	7
HUC	Hydrologic Unit Code	dbText	8
State	State	dbText	2
County	County (based on FIPS County Code)	dbText	50
Site_Type_Code	Site Type Code (DV = diversion; SW= surface water (streams, rivers); GW = ground water; SP = spring; SEEP = seep; MINE = mine; OF= outfall (sewage, other point discharges); LK = Reservoir/Lake; PL = Place of water use (intakes, etc.); UNK = unknown)	dbText	4
Geologic_Unit	geologic unit	dbText	50
Well_Depth	total depth recorded for well (in feet)	dbDouble	8
Elevation	elevation of site location (in feet)	dbLong	4
alt_meth_cd	Altitude method code (not available for Legacy STORETand Modern STORET sites; see ALTITUDE_METHOD_CODES table for code descriptions)	dbText	1
alt_acy_va	Altitude accuracy code (not available for Legacy STORETand Modern STORET sites; accuracy given indicates +/- given number in feet)	dbText	3
alt_datum_cd	Altitude datum (NGVD29 = National Geodetic Vertical Datum of 1929; NGVD88 = North American Vertical Datum of 1988)	dbText	10
Drainage_Basin_Area	area of drainage basin above site (in square miles)	dbLong	4
WaterSource	Identifies water source associated with site as identified in Phase II Vital Signs report or from subsequent analyses (tribs NOT labeled with mainstem rivers)	dbText	50
WaterSource_PhaseII	Identifies if water source for site was identified in Phase II Vital Signs report (yes = identified in report)	dbBoolean	1
WBIDSEG	Waterbody segment ID	dbText	25
X_PHARD_15YR	Mean Hardness computed from data collected between 01/01/1988 and 12/31/2002 for use with TVS calculations (for sites with available hardness data using aggregated PHARD data)	dbDouble	8
AdjX_PHard_15YR	Adjusted Mean Hardness computed from data collected between 01/01/1988 and 12/31/2002 for use with TVS calculations; all values >400 mg/L set to 400.	dbDouble	8
X_PHARD_SampleCount	Count of samples used for Mean Hardness calculation	dbLong	4

10.13. Table Name: SITE_TYPES

Description: Lookup Table of Site Type Codes

Field Name	Field Description	Field Type	Field Width
Site_Type_Code	Site Type Code (DV = diversion; SW= surface water (streams, rivers); GW = ground water; SP = spring; SEEP = seep; MINE = mine; OF= outfall (sewage, other point discharges); LK = Reservoir/Lake; PL = Place of water use (intakes, etc.); UNK = unknown)	dbText	4
Site_Type_Description	Description of site type	dbText	100

10.14. Table Name: SOURCE_AGENCIES

Description: Lookup Table of Source Agency Codes

Field Name	Field Description	Field Type	Field Width
Source_Agency	Source agency code (as used in original database, if applicable)	dbText	255
Original_Database	Original source database for source agency code	dbText	50
Source_Agency_Description	Description of source agency code	dbText	255

10.15. Table Name: tblCodeEquivalents

Description: Table of Ppcode group names, prioritization order and factors used to aggregate constituents

Field Name	Field Description	Field Type	Field Width
Ppcode	Numeric parameter code, Booker convention (Precede with P, for constituent value code)	dbText	8
ParameterFullName	Full parameter description	dbText	175
Name	New name to be given to aggregated parameter (exclude suffix "P")	dbText	50
Order	Order of aggregation for Ppcodes with same "Name"	dbInteger	2
Factor	Multiplication factor applied to Ppcodes during aggregation process (must fill-in with "1" if not factor needs applied)	dbDouble	8

10.16. Table Name: tbl_DB_Revisions

Description: Table of Database revisions

Field Name	Field Description	Field Type	Field Width
Revision_ID	Database revision number or code - PK.	dbGUID	16
DB_Meta_ID	Foreign key to database metadata globally unique ID.	dbGUID	16
DB_Version	Database version number.	dbSingle	4
Revision_Date	Database revision date.	dbDate	8
Revision_By	Person doing revision.	dbText	50
Revision_Reason	Reason for the database revision.	dbMemo	0
Revision_Desc	Description of revision.	dbMemo	0

10.17. Table Name: tbl_master_version

Description: Table for tracking protocol versions.

Field Name	Field Description	Field Type	Field Width
project_ID	Project ID number to ensure uniqueness across all projects	dbLong	4
version_key_number	Protocol version key number (maintained in SOP #10)	dbLong	4
version_key_date	Date of protocol version key number	dbDate	8
narrative_version	Version of protocol narrative	dbDecimal	16
version_comments	Comments regarding version, if any	dbMemo	0

10.18. Table Name: tbl_SOP_version

Description: Table for tracking SOP versions.

Field Name	Field Description	Field Type	Field Width
version_key_number	Protocol version key number (maintained in SOP #16)	dbLong	4
SOP_number	SOP number	dbLong	4
SOP_version_number	SOP version number	dbDecimal	16
active_flag	Yes indicates SOP is active	dbBoolean	1

10.19. Table Name: tlu_projects

Description: Lookup table for projects for protocol version tracking purposes.

Field Name	Field Description	Field Type	Field Width
project_ID	Project ID number to ensure uniqueness across all projects	dbLong	4
project_name	Project name	dbText	50
project_linkfile	Path and file name of project database	dbText	255
project_include_flag	True indicates project is to be included in query results	dbBoolean	1
project_comments	Comments regarding project, if any	dbMemo	0

10.20. Table Name: USGS_parmfile_master

Description: USGS parameter code master file (reflects Oct 2003 update in NWIS QWDATA)

Field Name	Field Description	Field Type	Field Width
PPCODE	Numeric parameter code, Booker convention (Precede with P, for constituent value code)	dbText	255
USGS_SHORT_NAME	Parameter short name	dbText	255
REPORTING_UNITS	Parameter reporting units	dbText	255
USGS_LONG_NAMES	Parameter long name	dbText	255

10.21. Table Name: USGS_SWDV_SITES

Description: SITE table of USGS SW and DV sites for use with NCPN_GAGES.mdb

Field Name	Field Description	Field Type	Field width
Site_Id	Unique identifier for specific sites (default is USGS Site ID, when more than one ID available)	dbText	255
LatLong_Cat	Concatenated DMS latitude/longitude (used for site consolidation site_ids where USGS site not available)	dbText	50
Site_Name	Name of sample site (default is USGS Site Name, when more than one name available)	dbText	255
Site_Description	Description of site location	dbText	200
Park_Code	NCPN 4-digit acronym for NPS units (predominant park_code will be used for sites upstream of more than one park)	dbText	4
Park_Boundary	Identifies which sites are within the park boundaries ("yes" or "no"); based on review of updated boundary coverage downloaded from NPS GIS web site in 12/2003	dbBoolean	1
Latitude_DMS	Latitude (in degrees, minutes, seconds)	dbText	6
Longitude_DMS	Longitude (in degrees, minutes, seconds)	dbText	7
Latitude_DD	Latitude (decimal degrees)	dbDouble	8
Longitude_DD	Longitude (decimal degrees)	dbDouble	8
UTM_x	NAD 1983	dbDouble	8
UTM_y	NAD 1983	dbDouble	8
Datum	Datum	dbText	10
UTM_Zone	UTM Zone (limited to zones 12 or 13 for the NCPN)	dbText	20
HUC	Hydrologic Unit Code	dbText	8
State	State	dbText	2
County	County (based on FIPS County Code)	dbText	50
Site_Type_Code	Site Type Code (DV = diversion; SW= surface water (streams, rivers); GW = ground water; SP = spring; SEEP = seep; MINE = mine; OF= outfall (sewage, other point discharges); LK = Reservoir/Lake; PL = Place of water use (intakes, etc.); UNK = unknown)	dbText	4
Geologic_Unit	geologic unit	dbText	50
Well_Depth	total depth recorded for well (in feet)	dbDouble	8
Elevation	elevation of site location (in feet)	dbLong	4
Drainage_Basin_Area	area of drainage basin above site (in square miles)	dbLong	4

10.22. Table Name: VALUE_REMARKS

Description: Lookup Table of Value Remark Codes

Field Name	Field Description	Field Type	Field Width
Value_Remark	Value remark code (as used on original database)	dbText	1
Value_Remark_Description	Description of value remark code	dbText	255
Original_Database	Original source database for value remark code	dbText	50

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #15

Data Analysis and Reporting, All Units

Version 1.00 (3/13/06)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

Table of Contents

List of Tables **3**
1. Introduction **4**
2. Preparing Raw Data for Analysis **4**
2.1. Censored data and outliers 4
2.2. Replicates and data transformations 5
2.2.1. Replicates *5*
2.2.2. Transformations *5*
3. Data Analysis **6**
3.1. Before data analysis: 6
3.2. Summary statistics and tabular data presentation 6
3.2.1. Calculating precision *8*
3.2.2. Statistics for fecal coliform data *9*
3.2.3. Criteria comparison on annual basis *9*
3.3. Graphical data presentation 10
3.4. Long-term trend analysis 13
3.4.1. Basic trend analysis: Graphing and other useful tools *13*
3.4.2. Trend detection *13*
4. Reporting **16**
4.1. Annual report (proposed completion date February 2007 for Water Year 2006): 16
4.2. Analysis and synthesis report 17
4.3. Program and protocol reviews 17
4.4. Quality assurance report 17
5. Report Format **17**
6. Review Procedure **19**
6.1. Internal review 19
6.2. Park review 19
6.3. External review 19
7. Distribution Procedure **19**
7.1. Identifying stakeholders 19
7.2. Distributing the report 19
8. References **20**
9. Appendix A: State of Utah 2006 303(d) Report **22**
10. Appendix B: Colorado Water Quality Standards **23**
11. Appendix C: Utah Water Quality Standards **24**
12. Appendix D: Wyoming Water Quality Standards **25**
13. Appendix E: Arizona Water Quality Standards **26**

List of Tables

Table 3-1. NCPN I&M Water Year 2006 Water Quality Data - Station ID 7
Table 3-2. Data for 2006 water year, *site name*. 10

List of Figures

Figure 3.1. Example data summary chart from Stafford and Horne, 2004 11
Figure 3.2. Example box and whiskers plot, quantiles, mean and standard deviation summaries by month, from Stafford and Horne, 2004. 12
Figure 3.3. Options for testing monotonic trends in uncensored data 15
Figure 3.4. Options for testing step trends in uncensored data 15
Figure 3.5. Options for testing for monotonic trends in censored data. 15
Figure 3.6. Options for testing for step trends in censored data. 16

1. Introduction

This SOP is entirely drawn from Cooprider (2005a,b) and O'Ney (2005) with edits that reflect conditions and issues specific to the NCPN Park units. At this point in program development before significant data collection has occurred, it will be difficult to outline specific procedures for data analysis and interpretation. For this reason at this point we approach data analysis and reporting as a set of options that will be narrowed as data compilation and descriptive analysis progresses. After several years of data collection a more definitive SOP can be written that explicitly states the approach and steps required for data analysis collected by this program.

Water quality data typically has a non-normal distribution due to a lower bound of zero, the presence of outliers, and positive skewness. Seasonality and autocorrelation are also common as well as covariance with other variables such as discharge (Helsel and Hirsch, 1992). Water quality data is usually highly variable, both temporally and spatially. Data characteristics often used for water quality data include: a measure of the center of the data, a measure of spread or variability, a measure of the symmetry of data distribution, and possibly estimates of extremes (Helsel and Hirsch, 1992). This SOP provides guidance on how to prepare and analyze data given these characteristics.

2. Preparing Raw Data for Analysis

2.1. Censored data and outliers

Water quality data is often "censored" or reported as less than the detection limit. In some cases, there are also instances of data values greater than or equal to the upper detection limit. This occurs most frequently with fecal coliform data since the proper dilution, based on the expected range, is sometimes difficult to predict particularly during storm events. Censored data is considered outside the range of quantitation (i.e., it cannot be quantified and a number cannot be assigned to it) and generally should not be statistically analyzed. However, censored data are presented as less than or greater than the ML in order to compare it to water quality criteria (Irwin, 2004). Therefore, although these data should not be included in statistical analysis, they are still useful for water quality assessment.

More advanced methods for dealing with censored data are outlined in Ch. 13 in Helsel and Hirsch (1992). This chapter describes how observed data may be combined with censored data in order to calculate estimates of summary statistics. Also refer to the recent publication *Nondetects and Data Analysis: Statistics for Censored Environmental Data* (Helsel, 2004).

The NCPN Water Quality Assurance Project Plan (SOP#7) describes the details of data quality objectives and measurement quality objectives in relation to data reporting. A summary of how data should be reported is as follows:

1. Values below the Method Detection Limit (MDL) are to be reported as a (<) sign followed by the actual MDL value, and flagged with a ND = not detected.

2. Values between the MDL and the PQL (or practical quantitation limit) should be reported as the actual measured value, with a flag that is carried all the way through data storage, handling, and reporting. The flag is DNQ = detected, not quantifiable. These data are considered semi-quantitative.

3. Values above the PQL are deemed as acceptable values without reservation, and are shown as the actual measured value, and assigned a QA code of A (acceptable without reservation).

4. Do not immediately remove outliers from a data set because they appear unusual. It is important to first verify that no human errors have been made such as incorrectly copying a number or putting a decimal point in the wrong place. Rather than eliminating possibly important data in order to use standard statistical analyses (e.g., tests requiring normally distributed data), methods that are resistant to outliers should be utilized. (Helsel and Hirsch, 1992). Some of the summary statistics that are resistant to outliers are discussed in Section 3.0 of this SOP.

2.2. Replicates and data transformations

2.2.1. Replicates

For NCPN reporting the median value of replicates is used for analysis. The main reason is to avoid strong influence of outliers. In well-mixed streams outliers are not expected and may result from inappropriate use of the multiparameter probe.

The standard deviation or range of the replicates provides an estimate of the variability in the measurement technique. A better way of estimating this for core parameters measured in the field is the alternative measurement sensitivity (AMS) outlined in section 7.4.2 in SOP 7.

2.2.2. Transformations

1. The goal of data transformations is to “make data more symmetric, to make data more linear, and to make data more constant in variance” (Helsel and Hirsch, 2002). Some examples include logarithmic transformations and adjusting data for flow.

2. Use logarithmic transformations on data when there is a broad range in data values. Log transformations are useful with fecal indicator bacteria (FIB) data since transforming allows for a more simple data analysis and graphical display of data with a range that often spans several orders of magnitude. Log transformations are also commonly used with discharge measurements and sometimes with nutrient data. It is helpful to display both the transformed data and non-transformed data to understand how transformations affect the data.

3. Flow adjusted or flow-weighted data is simply the concentration (C) of the analyte divided by the discharge (Q). Transformations (either logarithmic or flow-adjusted) can make the data more "normal" (symmetric) and increase the possibility of using parametric statistics which are slightly more powerful at determining statistical differences. Transforming the data does not change the median and interquartile range (IQR). However, transforming does change the mean and standard deviation (Helsel and Hirsch, 1992). This is why the mean and standard deviation as well as the median and IQR are reported for water quality data or any other data that is typically non-parametric.

3. Data Analysis

3.1. Before data analysis:

1. Review data promptly to detect potential outliers or errors.
2. Conduct log transformations on bacteria data and calculate flow-weighted data.
3. Use the median of replicate samples for statistical analysis.
4. Don't conduct statistical analyses on censored data but use all data for overall comparisons against water quality criteria.
5. Export data from NPSTORET to Microsoft Excel to conduct analysis. Further analysis can be conducted with other statistical software. However, NPSTORET does have several statistical and graphical functions that could be used as they become available.
6. Use graphs before data analysis to learn more about the data set. A plot of raw data values (for one site) against time is an important preliminary tool to assist in visualizing the data distribution and to provide a check for temporal patterns and extreme values (outliers).

3.2. Summary statistics and tabular data presentation

General guidelines to keep in mind when examining data for analysis.

1. Use all data (including censored data) for comparison against water quality criteria.
2. Analyze reference or control sites (if available) separately to determine a baseline for specific streams.
3. To account for seasonal variability, conduct statistical tests on each of the different seasons.
4. Summarize data for each site and for each parameter seasonally and annually
5. Use flow (discharge) weighted data for consistency.
6. Compare data from stations upstream and downstream of a suspected pollution source or tributary (when such data are available).

7. Discrete and continuous data should be analyzed separately. However, data from the same days may be compared for quality control and to obtain a relationship between the datalogger readings and instantaneous monthly/weekly data.
8. Determine the inherent variability of a sampling technique by calculating the relative percent difference (RPD) explained below in section 3.1.1 eq. 1, and the measurement precision expanded uncertainty (MPEU) as in eq. 2.
9. The other statistics listed in the following table are standard output for most statistical software packages.

Present data in tabular form for each site visit as in Table 3-1:

Table 3-1. NCPN I&M Water Year 2006 Water Quality Data - Station ID.

DATE	pH	TEMP	DO	COND	TURB	others….	……
Nov-06							
Dec-06							
Jan-06							
Feb-06							
Mar-06							
Apr-06							
May-06							
Jun-06							
Jul-06							
Aug-06							
Sep-06							
Oct-06							
Statistics							
Mean							
Std. Error							
Median							
Std. Dev.							
Variance							
RPD[1]							
AMS+[2]							
Kurtosis[3]							
Skewness[4]							
Range							
Min							
Max							
Sum							
Count							
95% CI							

[1]RPD is Relative Percent Difference

$$\text{RPD} = \left[\frac{S_1 - S_2}{(S_1 + S_2)/2} \right] \times 100$$

[2] AMS+ is Alternative Measurement Sensitivity

AMS+ = t*sd,

where

s = sample standard deviation of measurement values obtained in situ

t = 3.708, the 99% confidence middle (two-sided) t-value for sample size 7

[3] Kurtosis is a measure of the "peakedness" of the **probability distribution** of a **real**-valued **random variable**. Higher kurtosis means more of the **variance** is due to infrequent extreme deviations, as opposed to frequent modestly-sized deviations.

[4] Skewness is a measure of the asymmytery of a probability distribution of a random variable.

3.2.1. Calculating precision

The following is an explanation for calculating precision of field duplicates. Other QA/QC measures are discussed in SOP #7. Precision indicates the degree of agreement between sequential independent samples at a site, collected by applying the same collection method. If the sample is representative and the sampling methods are consistent, two or more measurements made consecutively with a field instrument usually agree very closely (less than 10 per cent difference). Estimates of precision are also known as sampling error. Precision should be calculated as soon as results from duplicate analyses are available, no later than 7 days after receipt from laboratory.

When only two measurements are made on the same sample, the precision measurement is calculated using the relative percent difference (RPD) between duplicate sample results.

$$RPD = \left[\frac{S_1 - S_2}{(S_1 + S_2)/2} \right] \times 100 \qquad \text{eq.1}$$

where,

S_1 = the larger test result value,

S_2 = the smaller test result value.

For precision results, not only should RPDs be reported, but also raw numbers. This will allow for calculation of uncertainty statistics later, should this be needed.

When more than 2 measurements are made on / in the same sample medium use the MPEU which is calculated as,

$$MPEU = 3.708 \times sd_7 \qquad \text{eq. 2}$$

where 3.708 = the 99% confidence middle t value for sample size 7,

sd_7 = standard deviation of 7 replicate samples on a normal environmental sample.

The MPEU calculation is functionally analogous to a PQL but uses the two-sided t value because we are not only interested in one side (distinguishing a value from zero) but in the two-sided issue of how large of a difference between two individual values we can

justify as actually being a true difference from one another and not due to measurement process noise (Irwin 2004).

3.2.2. Statistics for fecal coliform data

In addition to the above summary statistics, a geometric mean should be calculated for fecal coliform and *E. coli* data. To calculate the estimated geometric mean (*In* O'Ney, 2005; adapted from WY-DEQ 2004):

1. convert each CFU count/100ml to its log
2. add the logs
3. divide the total of the logs by the number of samples to get the mean
4. take the antilog of (3); that number is the geometric mean (in CFU/100mL)

    Example: test results are 760, 3100, 300, 632 and 805
    Arithmetic mean = 760 + 3100 + 300 + 632 + 805 = 5597 / 5 = 1119.4
    Arithmetic median = 760
    Geometric mean = 816.58
    $\log_{10}$ 760 = 2.88
    $\log_{10}$ 3100 = 3.49
    $\log_{10}$ 300 = 2.48
    $\log_{10}$ 632 = 2.80
    $\log_{10}$ 805 = 2.91
    sum of the logs = 14.56
    mean of the logs = 14.56/5= 2.912
    antilog 2.912 = 816.58

An alternative method is to multiply the CFU counts/100ml together and take the nth root, where n = number of samples (5 in this example). Using the test results above:

760 x 3100 x 300 x 632 x 805 = 3.6 x 1014

(3.6 x 1014)1/5 or (3.6 x 1014).2 = 815.19

Results from field blanks should also be reviewed to establish that a sample is not being contaminated by conditions associated with the collection or custody of a sample or by crosscontamination during sampling or shipping. Another way to determine whether field methods are adequate is to calculate precision from duplicates. Fecal coliform duplicate precision is calculated for the Number of Colonies /100 ml value (not the log transformation) and is typically set at ±50%. See section 3.1.2 in this document for guidance on calculating precision.

3.2.3. Criteria comparison on annual basis

Next, summarize all data and compare to water quality criteria using the following example in Table 3-2 modified from (from Rugg, 2000). The water quality criteria for UT can be found in the 303(d) report as a series of tables that guide the interpretation of data to assess the attainment of designated uses. These tables are available in Section II-9 of Utah's 2006 303(d) report Appendix A. In some cases UT-DWQ uses "best professional judgment" to determine designated beneficial use support.

Table 3-2. Data for 2006 water year, *site name*.

	Dissolved Oxygen (mg/L)	Total Ammonia (mg/L)	Un-ionized Ammonia (mg/L)	Conductivity (mhos/cm)	Others (units)
Sample size					
Average					
Range					
Criteria					
Number of exceedances					
Percent exceedance					

3.3. Graphical data presentation

Important graphical features or comparisons to utilize include:

1. Location (line) showing detection limit on graph
2. Distance from suspected source or distance from source
3. Display pH, D.O., and temperature on one chart to show relationships
4. Parameter against time (shows seasonal changes) for each station
5. Parameter against time for all stations in a watershed
6. Site comparisons for each parameter
7. Display all data for one station on one page
8. For continuous data, graph daily, monthly, and seasonally
9. Relationships of conductivity to fecal coliforms
10. Relationship of flow to all other variables

Chapter 2 (Graphical Data Analysis) and Chapter 16 (Presentation Graphics) of *Statistical Methods in Water Resources* (Helsel and Hirsch, 1992) should be followed for graphical data analysis. Tables can be used in association with graphs but presentation of data should not rely solely on tables. Graphics should generally include:

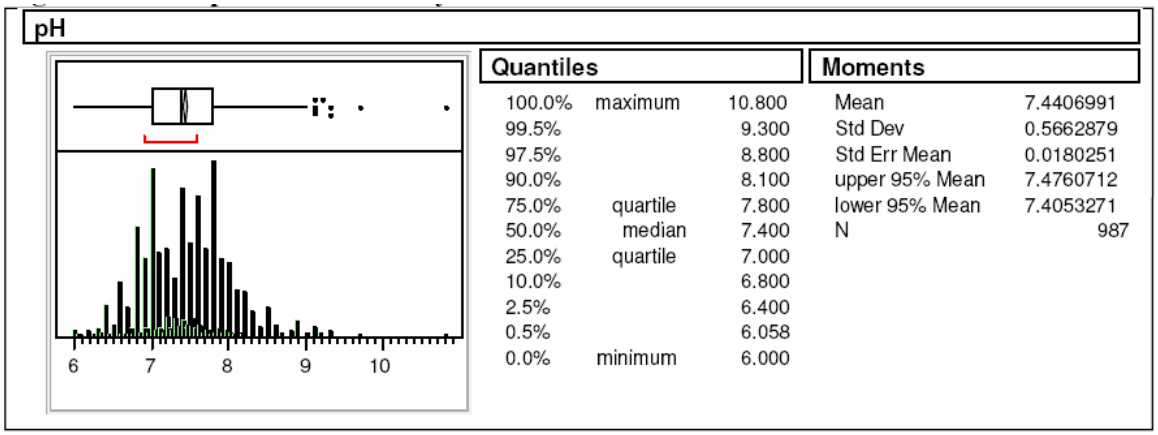
1. histograms (e.g., streamflow vs. number of occurrences): useful for depicting large differences in shape or symmetry. They are better for data that have natural categories or groupings; they are not as good with continuous data since it is difficult to depict this type of data accurately in a discrete group. It would work well for the number of sites exceeding different levels of water quality criteria (e.g., non-contact recreation and contact recreation).

2. simple box plots (box and whiskers plot) Whiskers are drawn to the points of maximum and minimum data, a box depicting the 25th and 75th percentile is drawn, and a horizontal line through the box depicts the median. These can be used for reviewing one set of data or for comparing multiple data sets. “They are valuable guides in determining whether central values, spread, and symmetry differ among groups of data.” They can be used to determine whether tests based on the assumption of normality can be used (Helsel and Hirsch, 1992).

3. scatterplots – relationship between two variables (e.g., flow vs. fecal coliforms); a “smooth” may be used to help determine the relationship of x to y. The preferred procedure for this is LOWESS (LOcally WEighted Scatterplot Smoothing) (Helsel and Hirsch, 1992).

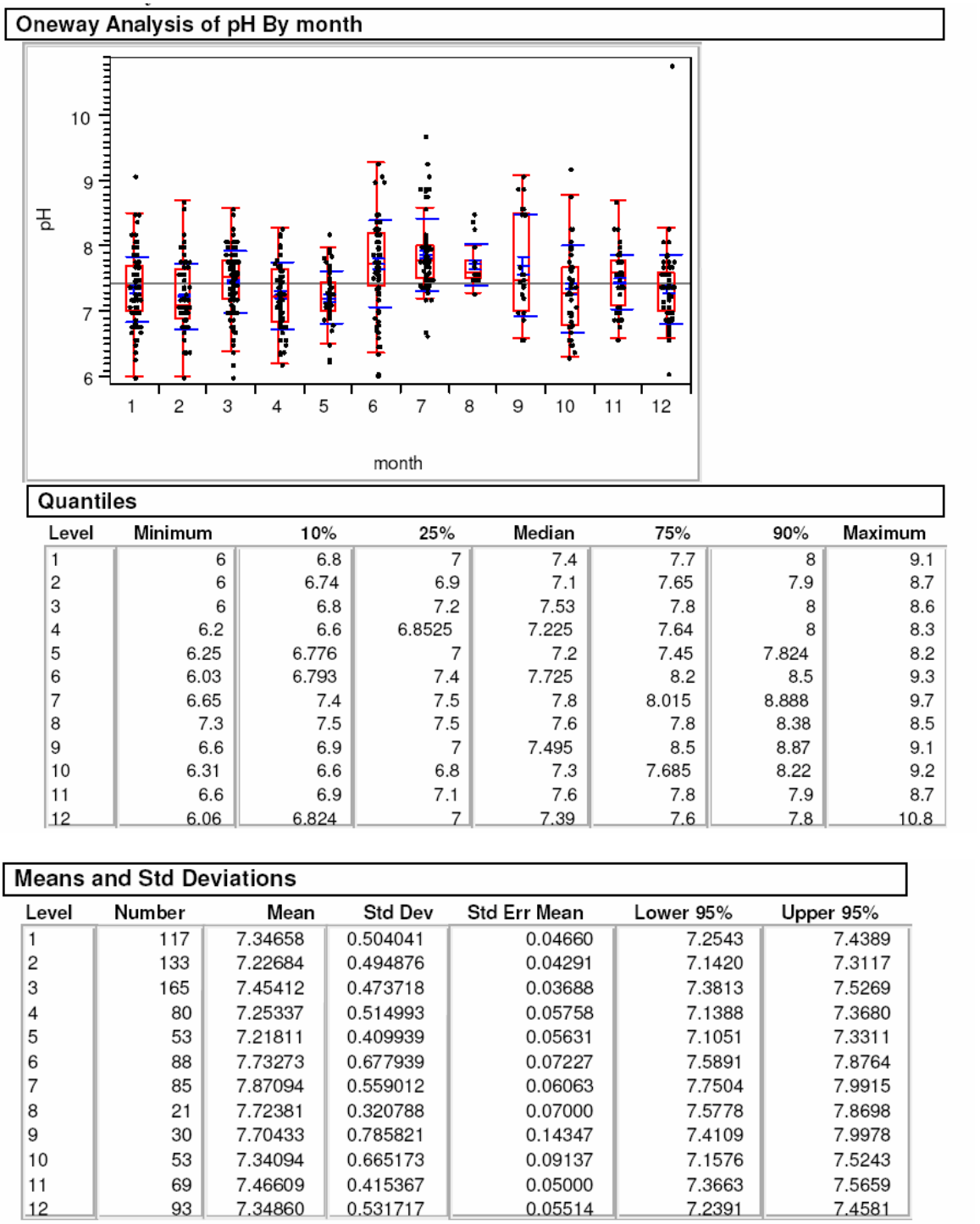
Summary tables, histograms, and box and whisker plots can be used to show median and interquartile ranges (non-parametric), mean and standard deviation (parametric), and 95% confidence intervals for means and medians. Example histograms and box plots are illustrated in Figures 3.1 and 3.2, respectively (from Stafford and Horne, 2004).

Figure 3.1. Example data summary chart from Stafford and Horne, 2004.



The heights of the bars in the histogram represent the number of times an observation was recorded. The outlier box-plot above the histogram shows the interquartile range within the box. The line across the middle of the box identifies the median sample value. The diamond represents the mean and 95% confidence interval. The lines extending from each end of the box, or the whiskers, encompass the quartiles +/- 1.5x (interquartile range). Points beyond the whiskers indicate extreme values that are possible outliers. The bracket along the edge of the box identifies the shortest half, or the densest 50% of the observations. To the right of the histogram, the quantiles and moments are displayed. The total number of observations is listed as N.

Figure 3.2. Example box and whiskers plot, quantiles, mean and standard deviation summaries by month, from Stafford and Horne, 2004.



Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
1	6	6.8	7	7.4	7.7	8	9.1
2	6	6.74	6.9	7.1	7.65	7.9	8.7
3	6	6.8	7.2	7.53	7.8	8	8.6
4	6.2	6.6	6.8525	7.225	7.64	8	8.3
5	6.25	6.776	7	7.2	7.45	7.824	8.2
6	6.03	6.793	7.4	7.725	8.2	8.5	9.3
7	6.65	7.4	7.5	7.8	8.015	8.888	9.7
8	7.3	7.5	7.5	7.6	7.8	8.38	8.5
9	6.6	6.9	7	7.495	8.5	8.87	9.1
10	6.31	6.6	6.8	7.3	7.685	8.22	9.2
11	6.6	6.9	7.1	7.6	7.8	7.9	8.7
12	6.06	6.824	7	7.39	7.6	7.8	10.8

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
1	117	7.34658	0.504041	0.04660	7.2543	7.4389
2	133	7.22684	0.494876	0.04291	7.1420	7.3117
3	165	7.45412	0.473718	0.03688	7.3813	7.5269
4	80	7.25337	0.514993	0.05758	7.1388	7.3680
5	53	7.21811	0.409939	0.05631	7.1051	7.3311
6	88	7.73273	0.677939	0.07227	7.5891	7.8764
7	85	7.87094	0.559012	0.06063	7.7504	7.9915
8	21	7.72381	0.320788	0.07000	7.5778	7.8698
9	30	7.70433	0.785821	0.14347	7.4109	7.9978
10	53	7.34094	0.665173	0.09137	7.1576	7.5243
11	69	7.46609	0.415367	0.05000	7.3663	7.5659
12	93	7.34860	0.531717	0.05514	7.2391	7.4581

In the example in Figure 3.2, pH data from all stations is grouped together for each month. “Level” is referring to month. One drawback of using maximum and minimum values is that as the data set grows, the most extreme values just keep extending and don’t necessarily reflect common conditions. They are useful for immediate management

decisions, since they often indicate something is wrong, but are not as useful for an overall sense of the water quality conditions.

3.4. Long-term trend analysis

Long-term trend analysis is generally conducted on five to ten years of data or more. However, data for most NCPN watersheds will be collected on two-year intervals. Trend analysis can be conducted on 4, 6, and 8 years of data and so on. The basic question in trend detection is "What is the effect of time on the given parameters?" If time is shown to have an affect, then we to ask "Are changes sudden or gradual?" and "What is the extent of the change?" Trend analysis should account for flow in order to be meaningful. Trend analysis should also account for seasonal differences. The ability to detect trends is dependent upon the variability of the data, as well as the responsiveness of the indicators (parameters), and sample size (Irwin, 2004).

3.4.1. Basic trend analysis: Graphing and other useful tools

For trend analysis, at a minimum, produce histograms displaying data from multiple years. Combine all data from all stations and display the maximums, means, range and number of water quality criteria exceedences (if applicable).

When graphing data for more than one year, seasonal patterns may be readily apparent. Each seasonal effect (strata) should be partitioned and graphed alone such that trends that develop over the long-term become visually clear. Examples of partitioned graphic representations are:

1. the concentration of a particular variable (y-axis) during low-flow periods (x-axis),
2. suspended sediments during winter storm events (refer to stream hydrograph),
3. nutrient values during the spring and summer (high productivity)
4. dissolved oxygen during peak temperature periods (summer)

Tips:

1. If the same data is used for long-term trends and short-term exceedences measured values can be averaged over each quarter, so that there is just one value per quarter.
2. The above approach can also be used for analysis of large (past) data sets with varying sampling frequencies
3. Maps displaying water quality trends are also a useful tool. Water quality stations can be identified by an increasing or decreasing trend or no trend. Another possibility is to utilize (or create) a water quality index to present NCPN data to a wide audience. One good example of a WQI was developed by the Washington Department of Ecology (Hallock, 2002).

3.4.2. Trend detection

Other routine trend analyses can be done according to Helsel's Internet Published Text Book (Helsel and Hirsch 1992). Consult Ch. 12 of the Helsel and Hirsch (1992) text for

techniques for trend analysis. This includes accounting for the affect of flow, other seasonal affects, and addresses both parametric and nonparametric statistics.

Stafford and Horne (2004), suggest the use of monotonic trends to look for gradual changes in water quality (Figure 3.3). The Helsel and Hirsch (1992) text covers various data analysis scenarios based on the distribution of the data (parametric, nonparametric, or mixed) and whether the data is flow-weighted. Figures 3.3–3.6 summarize recommendations for monotonic and step trend detection, depending on the type of data under analysis. Monotonic trends are to be used for gradual changes, and step trends are to be used before and after a change at a specific point in time. The monotonic trend hypothesis is more commonly used for general monitoring unless there is a reason to test for a step trend. The step trend hypothesis may be used after implementation of best management practice if there is expected to be a detectable change. The parameters classified as "mixed" in Figures 3.3 and 3.4 have both parametric and nonparametric components that are typically executed in separate steps.

Regression on season uses a periodic function of time of year, as does Tobit regression on season. Tobit regression is a type of linear regression that considers both censored and non (un?)censored values of the response variable, and uses maximum-likelihood estimation for determining slope and intercept of the modeled trend line (Hoppe, 2003).

Deseasonalizing is done by subtracting seasonal medians from each of the values to be regressed. The Seasonal Kendall test is the Mann-Kendall test for trend done for each season, with the Seasonal Kendall test statistic being the sum of the several Mann-Kendall test statistics. The seasonal Kendall trend test accounts for seasonal variations in concentrations by comparing ranks of data from the same recurring time intervals; for example, in a four-season year, springtime values are compared only to other springtime values, summer values to summer values, and so forth.

LOWESS is locally weighted scatterplot smoothing. The LOWESS curve represents a nonlinear, smoothed relation between two variables (instantaneous discharge and each water quality parameter). The method uses a series of weighted least squares regressions; observations are weighted by both distance from the fitted line and the magnitude of residuals from the previous regression. LOWESS is more desirable than simple regression because it makes no assumptions of data linearity or normality (Hoppe, 2003). Flow may be replaced by a transformation of flow in any of these analyses.

The Seasonal Rank Sum test is the Rank Sum test (also known as the Mann-Whitney “U” test (Kirchner, 2003), done for each season, with the Seasonal Rank Sum test statistic being the sum of the several test statistics.

Figure 3.3. Options for testing monotonic trends in uncensored data.

Options for testing monotonic trends in uncensored water quality data		
	Not Flow Adjusted	Flow Adjusted
Fully parametric	Regressions of concentration on time and season	Regression of concentration on time, season, and flow
Mixed	Regression of deseasonalized concentration on time	Seasonal Kendall on residuals from regression of concentration on flow
Nonparametric	Seasonal Kendall	Seasonal Kendall on residuals from LOWESS of concentration on flow

Figure 3.4. Options for testing step trends in uncensored data.

Options for testing step trends in uncensored water quality data		
	Not Flow Adjusted	Flow Adjusted
Fully parametric	Analysis of covariance of concentration on season and group (before and after)	Analysis of covariance concentration on season, flow and group
Mixed	Two-sample t test on deseasonalized concentration	Seasonal Rank Sum on residuals from regression of concentration on flow
Nonparametric	Seasonal Rank Sum	Seasonal Rank Sum on residuals from LOWESS of concentration on flow

Figure 3.5. Options for testing for monotonic trends in censored data.

Options for testing for monotonic trends in censored water quality data		
	Not Flow Adjusted	Flow Adjusted
Fully parametric	TOBIT regression of concentration on time and season	TOBIT regression of concentration on time, season and flow
Nonparametric	Seasonal Kendall	no test available

Figure 3.6. Options for testing for step trends in censored data.

Options for testing for step trends in censored water quality data		
	Not Flow Adjusted	Flow Adjusted
Fully parametric	TOBIT analysis of covariance of concentration on season and group	TOBIT analysis of variance of concentration on season, flow and group
Nonparametric	Seasonal Rank Sum	no test available

4. Reporting

Clear, concise, and consistent reporting of monitoring results is essential to the NCPN Water Quality Monitoring Program and is a primary form of information dissemination between the I&M program and the Parks. This SOP is adapted from SOP #11 in *Fish Community Monitoring in Prairie Park Streams with Emphasis on the Topeka Shiner (Notropsis Topeka)* (Peitz and Rowell, 2004).

A requirement for WRD is to provide a report that includes a paragraph summary for each parameter plus summary graphs of each site. The report is submitted with a copy of NPSTORET updated with the data from the past year. In addition, summary paragraphs will be provided for each watershed, including any proposed management activities related to water quality improvements. Recommendations for revising the protocol (changing monitoring intervals and timing, moving/adding sites, etc.) will also be proposed. These annual reports will also be provided to the NCPN parks.

Several types of reports will be made for the water quality monitoring program. These reports and their purposes are listed below (*from* Press, 2005):

4.1. Annual report (proposed completion date February 2007 for Water Year 2006):

1. Summary of water year data and document monitoring activities
2. Document current condition of the resources
3. Relative to State criteria
    a. UT Criteria State of UT water quality standards can be found at: **http://www.epa.gov/waterscience/standards/wqslibrary/ut/ut.pdf**. The designated uses for all water bodies of the state are listed in the above pdf, but a more user friendly searchable data base can be found at: **http://oaspub.epa.gov/wqsdatabase/wqsi_wb_du.rep_parameter?p_state=UT**
    b. CO criteria. State of CO water quality standards can be found at: http://www.epa.gov/waterscience/standards/wqslibrary/co/co_8_no31.pdf

4. Relative to anti-degradation concept in CWA
5. Document changes in the monitoring protocol
6. Document completeness of data collection
7. Document achievement of measurement objectives
8. Provides basis for communication within the park and network

4.2. Analysis and synthesis report

1. A comprehensive data analysis and synthesis every 3-5 years for 2-4 years of data. Having this extra time allows for more thorough data analysis and review of protocols and may give greater opportunity for adaptive management.
2. Document longer term condition of the resources
3. Relative to State criteria
4. Relative to anti-degradation concept in CWA
5. Determine patterns and trends
6. Discover correlations among resources being monitored
7. Analyze data to determine the level of change that can be detected using the existing sampling scheme
8. Recommend changes to management practices
9. Provides context to interpret data for the park within a multi-park, regional, or national context

4.3. Program and protocol reviews

1. Periodic formal reviews of operations and results
2. Review of protocol design and product to determine if changes are needed
3. Part of the quality assurance – peer review process
4. Provides structure for adaptive management of program

4.4. Quality assurance report

1. Produced biannually to demonstrate achievement of DQO's (SOP 7 section 7)
2. Representativeness, target population, and completeness
3. Data comparability (internal/NPS and external)
4. Measurement sensitivity (usually MDL and PQL) detection limits
5. Measurement precision as repeatability
6. Measurement systematic error/bias
7. Blank control bias (usually for chemical lab work only)

5. Report Format

Reports should be standardized with other I&M reports but will generally be written in 12 point Times New Roman text. Tables, figures, and photographs are encouraged to present data and site conditions. The following is the suggested outline by Peitz and Rowell (2004):

Title Page
- Title
- Author(s)
- Institutions
- Prepared for
- Date

Table of Contents (optional)
Executive summary (abstract)
Acknowledgements
1 Introduction
- 1.1 Background
- 1.2 Justification for study
- 1.3 Objectives

2 Methods
- 2.1 Study area(s)
- 2.2 Field method(s)
- 2.3 Analytical method(s)

3 Results
- 3.1 Measurement quality objectives
  - 3.1.1 Target population (SOP 7 section 7.1.1)
  - 3.1.2 Representativeness (SOP 7 section 7.1.2)
  - 3.1.3 Completeness (SOP 7 section 7.1.3)
  - 3.1.4 Data comparability (internal/NPS and external SOP 7 section 7.1.4)
  - 3.1.5 Measurement sensitivity and detection limits (SOP 7 section 7.1.5)
  - 3.1.6 Measurement precision as repeatability (SOP 7 section 7.1.6)
  - 3.1.7 Measurement systematic error/bias (SOP 7 section 7.1.7)
- 3.2 Summary statistics and charts
- 3.3 Exceedance reports

4 Discussion
This section includes documentation of antecedent hydrologic, climatic, and/or watershed conditions (if information is available) that may influence this year's data; and a brief narrative that places this year's monitoring efforts within the context of previous years (e.g. annual precipitation was far below average, resulting in drought conditions).

5 Management Implications
This section includes a narrative describing the current condition of aquatic habitat, and how it compares to conditions in previous years. To the extent possible this section should discuss how relevant management activities and concerns relate to monitoring results. This is where the I&M program has the opportunity to point out 'early warning' signs that may become significant.

6 References

6. Review Procedure

6.1. Internal review

One or more editorial reviews will be sought before submitting the report for review by staff in the park(s) where monitoring occurred and before external review. Internal review by person(s) skilled in technical writing for clarity and conciseness should fulfill this review requirement. Internal reviews will be conducted by the NCPN staff or cooperating hydrologists in network parks.

If reports are written to update findings only and they do not deviate significantly from previously reviewed and distributed reports, then the review process may stop here. However, review by park staff and subsequent external reviews must be sought for new reports or those that deviate significantly from previously reviewed and distributed reports.

6.2. Park review

Park staff, generally the Resource Managers, are in a unique position since they can supply details about management activities that may influence findings presented in a report. Also, they will most likely be directly involved in applying management recommendations to their respective parks. Therefore, review by park staff is vital to the interpretation of findings and the assessment of proposed management implications. Review by park staff should be conducted before a report is submitted for external review.

6.3. External review

External review by two or more experts in water quality monitoring should be sought for the first report in a series of annual reports. In addition, analytical methods employed on data presented in the report need to be reviewed by one or more statisticians. If a report updates a previously reviewed and distributed report, then external review is not required. However, external reviews must be sought for new reports or those that deviate significantly from previously reviewed and distributed reports. In order to conserve reviewer time, external reviews must follow the internal and park review process.

7. Distribution Procedure

7.1. Identifying stakeholders

The primary stakeholders in our Water Quality Monitoring efforts are park staff. Additional stakeholders include the NCPN I&M program and the State Water Quality Control Boards in UT and CO. Other potential stakeholders include any of the national water quality monitoring programs, universities and the general public.

7.2. Distributing the report

Reports will be provided to the respective parks where water quality monitoring was conducted. Additionally, a copy will be kept on file with the NCPN office of the National Park Service, Moab, UT and will be made available to all interested parties upon request.

All data collected by the NCPN is public property and is subject to requests under the Freedom of Information Act (FOIA). The data management plan for NCPN (Beer et al., 2005) describes appropriate procedures to respond to FOIA requests, including the protection of sensitive data such as endangered species locations. In the future, reports containing nonsensitive data will be disseminated through a website. Through the website, those requesting data will be asked to provide information to document by whom and for what purpose the report is being used. By documenting requests, users can be informed when updated reports are available. Users requesting paper copies will be documented also.

8. References

Beer, M., E. Nance, A. Wight, M. Powell, and R. DenBleyker. 2005. Northern Colorado Plateau Network, data management plan. National Park Service, Inventory and Monitoring Program, Northern Colorado Plateau Network, Moab, UT. 86 p. plus appendices.

Cooprider, MA. 2005a. Data Analysis, Version 1.01, Standard Operating Procedure #10. *In* San Francisco Area Network Freshwater Quality Monitoring Protocol, Version 2.0, Appendix H-SOPs, National Park Service, San Francisco Bay Area Network, CA. 51 pp. Plus appendices.

Cooprider, MA. 2005b. Data Reporting, Version 1.0, Standard Operating Procedure #10. *In* San Francisco Area Network Freshwater Quality Monitoring Protocol, Version 2.01, Appendix H-SOPs, National Park Service, San Francisco Bay Area Network, CA. 51 pp. Plus appendices.

Hallock, D. 2002. A Water Quality Index for Ecology's Stream Monitoring Program. Washington Department of Ecology. 17pp. + appendices.

Helsel, D.R. and Hirsch R.M. 1992. Statistical Methods in Water Resources. Studies in Environmental Science 49, Elsevier Publishing, NY. **http://water.usgs.gov/pubs/twri/twri4a3/pdf/twri4a3.pdf** Accessed 1/31/06

Helsel D. 2004. Nondetects and Data Analysis: Statistics for Censored Environmental Data. Wiley. 288 pp. **http://www.wiley.com/WileyCDA/WileyTitle/productCd-0471671738.html** Accessed 1/31/06

Hoppe, M. C. 2003. Ground and surface water quality. New York-New Jersey Highlands Regional Study Technical Report. U.S. Forest Service. Accessed 1/31/06 at http://www.fs.fed.us/na/highlands/atechnical_report/index.html

Irwin, R. 2004. Vital Signs Long-Term Aquatic Monitoring Projects: Part B – Planning Process Steps: Issues to Consider and then to Document in a Detailed Study Plan that Includes a Quality Assurance Project Plan (QAPP) and Monitoring "Protocols" (Standard Operating Procedures). National Park Service, Water Resources Division

Kirchner, J. 2003. EPS 120 Reader: Analysis of environmental data. Berkeley.

O'Ney, S. 2004. Data Analysis Procedures. Version 1.0, Standard Operating Procedure #9. *In* Regulatory Water Quality Monitoring Protocol, Version 1.0, Appendix E-SOPs, National Park Service, Greater Yellowstone Network. Bozeman, MT. 37 pp. plus appendices.

Peitz DG, Rowell GA. 2004. Fish community monitoring in prairie park streams with emphasis on Topeka Shiner (Notropis Topeka). Prepared for: Prairie Cluster Prototype Long-Term Ecological Monitoring Program. Version 1.00 (February 2, 2004).

Press, D. 2005. DRAFT San Francisco Bay Area Network Data Management Plan *In* Appendix 7 of the SFAN Draft Phase III Vital Signs Monitoring Plan. U.S. Department of Interior, National Park Service.

Rugg, M. E. 2000. Marin-Sonoma Counties agricultural runoff influence investigation – 1999- 2000 summary. California Department of Fish and Game

Stafford, S. and A. Horne. 2004. A review of the water quality monitoring programs in the National Parks in central coastal California. Report to the National Park Service. University of California, Berkeley.

WY-DEQ/WQD, 2004. Manual of Standard Operating Procedures for Sample Collection and Analysis. Wyoming Department of Environmental Quality, Water Quality Division, Watershed Program, Cheyenne, WY. http://deq.state.wy.us/wqd/watershed/Downloads/QA/4-1089.pdf Accessed 1/31/06

9. Appendix A: State of Utah 2006 303(d) Report

Utah's 2006 Integrated Report
Volume II - 303(d) List of Impaired Waters



Little Deer Creek, Wasatch County

Department of Environmental Quality
Division of Water Quality

10. Appendix B: Colorado Water Quality Standards

COLORADO DEPARTMENT OF PUBLIC HEALTH AND ENVIRONMENT
WATER QUALITY CONTROL COMMISSION

REGULATION NO. 31

THE BASIC STANDARDS AND METHODOLOGIES FOR SURFACE WATER
(5 CCR 1002-31)

ADOPTED: May 22, 1979	EFFECTIVE: July 10, 1979
AMENDED: December 12, 1983	EFFECTIVE: January 30, 1984
AMENDED: June 2, 1987	EFFECTIVE: July 31, 1988
AMENDED: June 6, 1988	EFFECTIVE: July 31, 1988
AMENDED: August 1, 1988	EFFECTIVE: September 30, 1988
AMENDED: August 7, 1989	EFFECTIVE: September 30, 1989
AMENDED: October 8, 1991	EFFECTIVE: November 30, 1991
AMENDED: May 4, 1993	EFFECTIVE: June 30, 1993
AMENDED: August 2, 1993	EFFECTIVE: September 30, 1993
AMENDED: October 4, 1993	EFFECTIVE: November 30, 1993
AMENDED: December 6, 1993	EFFECTIVE: January 31, 1994
AMENDED: January 10, 1995	EFFECTIVE: March 2, 1995
AMENDED: January 8, 1996	EFFECTIVE: March 1, 1996
AMENDED: January 13, 1997	EFFECTIVE: March 3, 1997
AMENDED: July 14, 1997	EFFECTIVE: August 30, 1997
AMENDED: January 12, 1998	EFFECTIVE: March 2, 1998
AMENDED: January 11, 1999	EFFECTIVE: March 2, 1999
AMENDED: August 15, 2000	EFFECTIVE: December 22, 2000
AMENDED: November 7, 2000	EFFECTIVE: March 20, 2001
EMERGENCY AMENDMENT: November 8, 2000	EFFECTIVE: November 8, 2001
AMENDED: February 13, 2001	EFFECTIVE: March 30, 2001
EMERGENCY AMENDMENT: May 14, 2001	EFFECTIVE: May 14, 2001
AMENDED: September 10, 2001	EFFECTIVE: October 30, 2001

11. Appendix C: Utah Water Quality Standards

STATE OF UTAH
OFFICE OF THE ATTORNEY GENERAL

JAN GRAHAM
ATTORNEY GENERAL



JAMES R. SOPER
Solicitor General

FELIX RICHARDS
Chief Deputy Attorney General

August 9, 2000

Don Ostler
Executive Secretary
Utah Water Quality Board
288 North 1460 West
Salt Lake City, Utah 84114-4870

Dear Don:

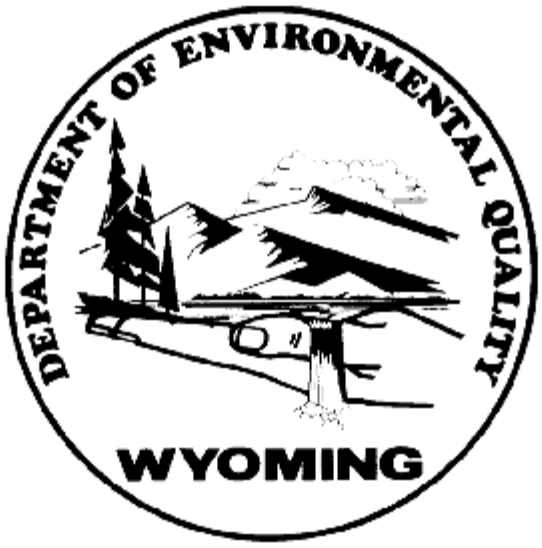
In accordance with the requirements of 40 C.F.R. § 131.6(e), I have reviewed the materials submitted by your office, and hereby certify as legal counsel to the Utah Water Quality Board, that based on that information, the revised water quality standards (R317-2, Utah Administrative Code) which were adopted by the Board on July 17, 2000, were duly adopted pursuant to state law.

Sincerely,

Fred G Nelson
Assistant Attorney General
Counsel to
Utah Water Quality Board

160 East 300 South, 5th Floor, P.O. Box 140873, Salt Lake City, Utah 84114-0873
Telephone: (801) 366-0290 Facsimile: (801) 366-0292

12. Appendix D: Wyoming Water Quality Standards



WATER QUALITY
RULES AND REGULATIONS

Chapter 1

13. Appendix E: Arizona Water Quality Standards



C:\David\NPS_WQ\
protocols\SOP15_ana

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #16

Protocol revision

Version 1.02 (8/12/08)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.0	2/4/08	H. Thomas	Added and updated MVT	Changes to SOP #5	1.01
1.01	8/12/08	H. Thomas	Added and updated MVT	Changes to SOP #8	1.02

This Standard Operating Procedure explains how to make changes to the Freshwater Quality Protocol, narrative and accompanying SOPs, and explains procedures for tracking these changes. NCPN or park staff editing the Protocol *Narrative* or any SOP need to follow this procedure to eliminate confusion in data collection and analysis methods. All NCPN aquatic resources staff should be familiar with this SOP in order to identify and use the most current methodologies. This SOP is adapted from the SAFN Protocol #1. That SOP can be accessed at: http://www1.nature.nps.gov/im/units/sfan/reports/WQ/SOP1_RevisingProtocol_V1_0.pdf

Table of Contents

1. Introduction **3**
2. Review process **3**
3. Revision history log **3**
4. Notification **4**
5. Posting new versions **4**
6. What constitutes significant change? **4**
7. Formatting **5**
8. References **5**

1. Introduction

The Water Quality Monitoring Protocol Narrative and accompanying SOPs have incorporated the most sound methodologies for collecting and analyzing water quality data. However, all protocols require editing as new and different sample collection, analysis, and data management information becomes available. Required edits should be made as soon as they are deemed necessary and appropriate reviews conducted.

2. Review process

1. All edits will be reviewed for grammatical and technical accuracy and overall clarity. Minor changes or additions to existing methods will be reviewed "in-house" by the NCPN hydrologist and other appropriate network staff. However, if a complete change in methods is anticipated, then an outside review is required. Regional (Intermountain Region) and National staff (Water Resources Division) of the National Park Service familiar with water quality monitoring and data analysis will be utilized as reviewers.
2. Also, local and state experts in water quality monitoring and statistical methodologies outside of the Park Service will be utilized in the review process. A group of technical experts was utilized (SOP 7 section Table 3-1) for the external peer review of the initial Freshwater Quality Protocol; these individuals would be called upon again to provide input related to any significant revisions in methodology.

3. Revision history log

1. Edits and protocol revisions will be documented in the Revision History Log that accompanies the Protocol Narrative and each SOP.
2. Only changes in the Protocol Narrative or specific SOP that has been edited will be logged. Version numbers increase incrementally by hundredths (e.g. version 1.01, version 1.02, …etc) for minor changes.
3. Major revisions should be designated with the next whole number (e.g., version 2.0, 3.0, 4.0 …). Record the previous version number, date of revision, author of the revision, identify paragraphs and pages where changes are made, and the reason for making the changes along with the new version number.
4. The NCPN tracks the narrative and SOP version numbers in a Master Version Table (MVT), which is maintained in this document (SOP #16). Any time a narrative or an SOP version change occurs, a new Version Key number (VK#) must be created and recorded in the MVT, along with the date of the change and the versions of the narrative and SOPs in effect. The VK number increments by whole integers (e.g., 1, 2, 3, 4, 5). Updates to the MVT also must be provided to the NCPN Data Manager for inclusion in the master version table database. *The protocol narrative, SOPs, and data should not be distributed independently of this table*.

Water Quality Monitoring, Northern Colorado Plateau Network
Master Version Table

Version Key #	Date of Change	Narrative	SOP 1	SOP 2	SOP 3	SOP 4	SOP 5	SOP 6	SOP 7	SOP 8	SOP 9	SOP 10
VK1	4/7/2007	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
VK2	2/4/2008	1.00	1.00	1.00	1.00	1.00	1.01	1.00	1.00	1.00	1.00	1.00
VK3	8/12/2008	1.00	1.00	1.00	1.00	1.00	1.01	1.00	1.00	1.10	1.00	1.00

Version Key #	Date of Change	SOP 11	SOP 12	SOP 13	SOP 14	SOP 15	SOP 16	SOP 17
VK1	4/7/2007	1.00	1.00	1.00	1.00	1.00	1.00	1.00
VK2	2/4/2008	1.00	1.00	1.00	1.00	1.00	1.01	1.00
VK3	8/12/2008	1.00	1.00	1.00	1.00	1.00	1.02	1.00

4. Notification

1. Notify the NCPN Lead Data Manager of any changes to the Protocol Narrative or SOP so that the new version number can be incorporated in the Metadata of the NPSTORET database.
2. The Data Manager will then edit the database per any changes to the Protocol Narrative and SOPs.

5. Posting new versions

Post new versions on the internet and forward copies to all individuals with a previous version of the Protocol Narrative or SOP.

6. What constitutes significant change?

When any significant changes in the sampling protocol occur such as changes in sample collection techniques or equipment, a change in analytical laboratory, or changes in staff, there should be an “overlap” of methods and personnel (Oakley et al., 2003). This requires using both the old and new techniques on a given sample as well as having both the outgoing and new staff sample concurrently. The National Park Service Water

Resources Division (NPS-WRD) recommends an overlap of at least seven sampling events (Irwin, 2005).

7. Formatting

To keep the protocols looking readable, always assign one of the following styles to all new text:

- Caption
- Endnotes
- Heading 1
- Heading 2
- Heading 3
- Indent
- Normal
- Table header
- Table note
- Table text
- Table title

Customized formatting can subsequently be added to the text, but it's best to start with these styles.

8. References

Irwin, R. 2005. Personal Communication. National Park Service, Water Resources Division, Fort Collins, Colorado.

Peitz, D.G., S. G. Fancy, L. P. Thomas, and B. Witcher. 2002. Bird monitoring protocol for Agate Fossil Beds National Monument, Nebraska and Tallgrass Prairie National Preserve, Kansas. Prairie Cluster prototype monitoring program. Version 1.00, September 6, 2002.

Oakley, K.L, L.P. Thomas, and S.G. Fancy. 2003. Monitoring Protocol Guidelines. Wildlife Society Bulletin 31(4): 1000-1003.

Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #17

Annual Project Review

Version 1.00 (1/08/07)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #

Table of Contents

1. Introduction **3**
2. Overview **3**
3. Review topics **3**
3.1. Equipment list / ownership / location / loan 3
3.2. Equipment maintenance 3
3.3. Supplies and consumables 3
3.4. Review safety contact numbers 4
3.5. Review and update field folders 4
3.6. Evaluate equipment calibration reports 4
3.7. Database updates 4
3.8. Evaluate measurement quality objectives 4
3.9. Field protocol 4
4. Reporting **5**

1. Introduction

This Standard Operating Procedure explains the type of annual project review necessary to achieve and demonstrate a high standard for water quality monitoring from the standpoint of efficiency and safety. It provides an opportunity for refining protocols based on quantitative evaluation of monitoring activities and the possibility for course correction if necessary.

2. Overview

The Water Quality Monitoring Protocol Narrative and accompanying SOPs have incorporated the soundest methodologies for collecting and analyzing water quality data. However, all aspects of the monitoring need a holistic review to ensure all tasks are accomplished and objectives met.

3. Review topics

The following topics deserve careful attention on an annual basis so that needs are anticipated before needs arise and to ensure overall smooth program operation.

3.1. Equipment list / ownership / location / loan

Some of the equipment used by NCPN is on loan from other parks. This equipment needs to be inventoried and its need for loan evaluated. If a piece of equipment is routinely used perhaps it should be purchased rather than borrowed. Also, equipment loaned out by NCPN should be accounted for so that it can be retrieved if necessary. An equipment list and ownership can be found in SOP 5 Appendix O.

3.2. Equipment maintenance

Although all water testing equipment is evaluated monthly via premobilization procedures described in SOP 5, an annual evaluation of equipment performance should be made by evaluating trends in calibration reports. Do this by plotting calibration coefficients stored in Field_forms.xls versus time.

3.3. Supplies and consumables

Evaluate the supply of all consumables and place orders as necessary for the following items. Records of purchased items can be found in the NCPN hydrologist equipment folder. Purchase identical supplies from the same supplier to minimize bias that may result from variability in equipment manufacturer. The list of lab chemicals and supplies can be found in SOP 5 Appendix N.

1. pH, conductivity and DO standards
2. Deionized water
3. Filters and pump hose
4. Turbidity standards and vials
5. Plastic carry bottles
6. Batteries for camera, troll, and turbidity meter

3.4. Review safety contact numbers

Individuals and contact numbers for them may change in the course of a year. Make sure contact information is current in case of emergency and for routine site visits. Be sure each is updated in the Field forms folder site descriptions.

3.5. Review and update field folders

Update field folders with new site description files when sites change on a rotating panel basis. Also update annual ranges in parameter values for each site.

A list of field folder contents can be found in SOP 4 section 7.1.

3.6. Evaluate equipment calibration reports

Sensors degrade over time and eventually become unreliable. Review calibration reports stored in field forms to evaluate the potential for slow sensor degradation. This can be accomplished relatively easily by plotting sensor calibration constants versus time with an eye toward acceptable sensor calibration limits that can be found in user manuals.

3.7. Database updates

Evaluate the NCPN water and NPStoret databases for completeness. Water quality data are entered into databases in staggered and non-periodic intervals due to complexities in managing field and laboratory data that may require re-runs. For this reason it is a good practice to annually evaluate the completeness of data entry and quality control checks.

3.8. Evaluate measurement quality objectives

A number of quality assurance metrics are discussed in SOP 7 section 7. These objectives are discussed in detail there but in general are used to quantify the quality of data collected in this monitoring effort. At a minimum each of the following need to be considered or calculated.

1. Target population (SOP 7 section 7.1.1)
2. Representativeness (SOP 7 section 7.1.2)
3. Completeness (SOP 7 section 7.1.3)
4. Data comparability (internal/NPS and external SOP 7 section 7.1.4)
5. Measurement sensitivity and detection limits (SOP 7 section 7.1.5)
6. Measurement precision as repeatability (SOP 7 section 7.1.6)
7. Measurement systematic error/bias (SOP 7 section 7.1.7)

3.9. Field protocol

At least once annually each hydrologist collecting water quality data should be accompanied by a peer or supervisor to ensure all standard operating procedures are being carried out completely and faithfully. Each hydrologist in NCPN has slightly different field protocols due to the unique field environments where they work. However, a new set of eyes may see potential for improvement or sources of potential contamination not readily apparent. This will ensure maximum uniformity in data collection, minimize observer induced bias and will help share techniques developed in our unique working environments.

4. Reporting

Results from all of the above considerations should be included in the annual report and summarized over longer duration in the triennial reports (SOP 15).