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 2.01 (April 2016)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.0	06/26/08	K. Lawrence	Added seatbelts to JHA	Forgot to include in earlier draft	1.01
1.01	5/26/08	D. Thoma	No cell phone while driving	New park safety policy	1.02
1.02	3/15/10	D. Thoma	Inform park if accident occurs	Not in earlier version	1.03
1.03	1/25/11	M. Van Grinsven	Sections 9.8-9.10, and updated JHA	Not in previous version	1.04
1.04	5/14/12	C. Hackbarth	Use GLCA dispatch for backcountry plans, not by individual park	Beneficial to use one main contact for backcountry travel	1.05
1.05	2/13/14	R. Weissinger, C. Hackbarth	Incorporate programmatic field safety protocol	Maintain safety information in one location for NCPN	2.00
2.00	3/30/16	C. Hackbarth	Added info about Backcountry Travel Plan; updated chemical list	Not in previous version; some chemicals no longer used	2.01

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 Health and Safety.

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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.

When in the field, each NCPN crew carries a well-stocked first aid kit and a satellite phone and/or a personal locator beacon (PLB). Crew members should be trained in how to use items in the first aid kits and the satellite phone/PLB and should know where these emergency materials are stored during fieldwork. Permanent technicians will have Wilderness First Aid and CPR training at a minimum.

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 four 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**.** NCPN uses the rule of 10 as a guideline for safe wading: if the water depth (ft) multiplied by the velocity of the water (ft/sec) is greater than 10, do not enter the stream. **If there is any doubt in regard to whether conditions are safe, the correct decision is to forgo sampling until another day.** If discharge is too high for wading, estimate the discharge. For qualitative flow estimates, use the STORET Flow Severity Index and hydrograph limb codes in the Field_forms.xls spreadsheet page "field codes".

Excerpts from USGS Field Manual section 9.4 Appendix C 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:

- An example JHA for Water Quality Field Work is presented in Appendix A.
- An example JHA for Water Quality Laboratory Sample Processing is presented in Appendix B.
- The USGS National Field Manual includes a Chapter for Safety in Field Activities. Pertinent sections are included as a useful reference in Appendix C.

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

At the beginning of the field season, each crew member should review the NCPN Field Safety Protocol, which is updated annually and is available on the NCPN network drive. The Field Safety Protocol provides detailed safety procedures including setting up a Backcountry Travel Plan with check-ins with Glen Canyon (GLCA) dispatch. Refer to the NCPN Field Safety Protocol for a blank form of the Backcountry Travel Plan and additional information.

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, program leaders, and GLCA dispatch to notify them of the time and location of work in each park (Figure 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.

6. Reporting Procedures

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. Vehicle Accident reporting forms can be found in Appendix D.

In the event of personal injury the injured party must notify the superintendent of the park where the injury occurred, the personnel office representing the park where injury occurred, and their immediate supervisor. Human resources staff can help the injured party navigate the reporting, documentation and claims paperwork.

1) Any **potentially** debilitating incident that could eventually result in lost time from work or assignment of alternative duties must be reported at the time of incident. There are no repercussions that result from reporting a **potential** injury. Even if a claim is not immediately filed this reporting is necessary to prove the incident leading to debilitation occurred while performing official duties.

2) Any debilitating injury that results in lost time from work or alternative duties while recovering must be reported to the **superintendent and human resources staff** of the park where the injury occurred. Superintendents are required to report lost time injuries to the Regional Director within 24 hours of an incident.

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 water quality supply closet.

A list of all chemicals (**Error! Reference source not found.**) 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.

Table 1. Chemical list.

Water Quality Monitoring Chemical List	
Item	**Company**
Colilert	IDEXX
Colilert Comparator	IDEXX
Conductivity Standard, 147 umhos	Aqua Solutions
Conductivity Standard, 1413 umhos	Aqua Solutions
Conductivity Standard, 12900 umhos	Aqua Solutions
pH Electrode Filling Solution	In-Situ
pH Electrode Storage Solution	In-Situ
pH 4 Buffer Solution	Aqua Solutions
pH 7 Buffer Solution	Aqua Solutions
pH 10 Buffer Solution	Aqua Solutions
Quat-128 Disinfectant	Waxie, Inc
Sample Bottle with Nitric Acid Preservative	Utah Public Health Laboratory
Sample Bottle with Sulfuric Acid Preservative	Utah Public Health Laboratory
Sodium Sulfite, Anhydrous	Hach Company
Turbidity StablCal Calibration Set	Hach Company

8. Appendix A: 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 d) Phone/mobile device use	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. Always wear seatbelt. Both hands on steering wheel at all times. No cell phone use while driving.
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. Always wear seatbelt. Both hands on steering wheel at all times. Be wary of washboards and corners.
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 boots 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 (ft) and velocity (ft/sec) is >). Be wary of slippery and loose stream beds and dangerous animals. Do not cross ice over flowing water that is more than 2 ft deep. Be aware of weather conditions and changing stream conditions.
6. Deal with environmental conditions	a) Temperature and sun exposure b) Adverse weather (rain, snow, lightning, and wind) c) Rockfall d) Wildfire	Wear sun protection and have appropriate extra clothing in case weather changes. Keep to low areas during lightning. Watch for flash floods. Maintain good hydration and salt levels. Be aware of potential rockfall areas, particularly during storms. Be aware of wildfires near where you are working and keep a safe distance in case you need to

		evacuate.
7. Dangerous fauna and flora	a) Insects: Scorpions and Spiders b) Snakes: Rattlesnakes c) Wildlife: Mountain Lions and Bears d) Plants: poison ivy, stinging nettle, thorny/spiny plants	Avoid placing extremities in locations such as rock crevices and overhangs, behind shrubs, and over fallen logs without inspecting the locations first. Be aware of your surroundings. Walk carefully, listen for rattles, and be able to identify bear and mountain lion tracks. If tracks appear fresh, return to vehicle. Wear appropriate clothing and shoes to protect from insects and plants. Check for ticks regularly, especially when working where they are common. Know how to administer first aid for bites and stings.

9. Appendix B: 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 Sodium Sulfite, pH and conductivity standards c) Inhalation of acid fumes	Wear eye protection, rubber gloves, apron, know clean up procedure for 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.

10. Appendix C: 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

9.8.1 Temperature and Sun Exposure

Field work often is necessary under adverse atmospheric and other environmental conditions. Unpredictable occurrences, such as earthquakes, require event-response planning. Prepare for extreme conditions that might be experienced in your area of the country. Before leaving for the field, check the weather forecast using one or more of these options: the local television station, the national weather channel, the local land/marine weather band channel that constantly repeats current and future weather conditions, or computer networks such as the World Wide Web at **http://www.intellicast.com**. Extremes of air temperature occur in all parts of the country. The ideal comfort range for humans is between 16 to 32ºC (60 to 90ºF). Hypothermia and hyperthermia normally occur in temperatures outside this range.

Hypothermia is a condition of reduced body temperature caused by exposure to cold, and aggravated by wet clothes, wind, hunger, and exhaustion. Hypothermia in extremities can lead to frostbite. Hypothermia can occur with air temperature above 16ºC (60ºF) under wet and (or) windy conditions. The best way to avoid hypothermia is to dress warm and stay dry. Refer to Denner (Revised 1993) for information on coldweather clothing. The warning signals of hypothermia are uncontrollable fits of shivering, incoherence, listlessness, fumbling hands, frequent stumbling, drowsiness, and inability to get up after resting. Victims of hypothermia must be treated immediately by removing them from exposure to the elements, replacing wet clothes with dry ones, and giving them warm, non-alcoholic drinks. Seek emergency facilities as soon as possible.

To prevent hypothermia:
1. Put on rain gear before it starts to rain or snow.
2. Put on additional clothes before starting to shiver.
3. Seek shelter immediately if conditions become severe.

Hyperthermia is a condition of increased body temperature caused by exposure to excessive heat. Contributing factors are physical exertion, clothing, humidity, lack of air movement, and temperature, but the most important factor is body hydration. The normal body requirement for fluids in temperate regions is 2 1/2 quarts per day; desert conditions require more fluid. Early warning symptoms of hyperthermia are chilling, a throbbing pressure in the head, unsteadiness, dizziness, nausea, dry skin (either hot and red or cool and pale), rapid pulse, and muscle pains and spasms. Persons suffering from hyperthermia should seek medical attention immediately. First aid involves cooling down and rehydrating.

To avoid hyperthermia:
1. Drink water in moderate amounts on a scheduled basis—do not wait until you are thirsty.
2. Avoid alcohol, caffeine, and soda—these liquids are not water substitutes.
3. Wear lightweight clothing and a wide-brimmed hat.
4. Schedule activities that require the most exertion in early morning or late afternoon, if possible, and not when air temperature is at its highest.

Sun exposure can have painful and dangerous short term and long-term effects. Regardless of the region in which you are working, take the proper precautions to protect your skin and eyes from excessive sun exposure.

To prevent excessive sun exposure:
1. Wear sunscreen on all exposed skin to avoid burning and skin cancer.
2. Wear sunglasses with polarized lenses to protect eyes, reduce glare, and improve vision, especially when working on water or snow.

9.8.2 THUNDERSTORMS, TORNADOES, AND HURRICANES

Thunderstorms, which can be accompanied by hail, are common throughout the United States. Some are predicted by weather forecasters. Others can move into an area with almost no advance warning. Watch the sky for signs of thunderstorms, and seek shelter before the weather deteriorates. Lightning is extremely dangerous and should be respected.

To protect yourself during thunderstorms, heed the following advice from Lockhart (1988):
- Seek shelter inside a vehicle or building; keep away from open doors and windows, plugged in appliances, and metal. Avoid contact with metal objects in a vehicle.
- Do not use a telephone.
- If outside, do not congregate. In case of a lightning strike, someone must be able to begin revival techniques immediately, such as cardiopulmonary resuscitation (CPR).
- Put on rubber boots or rubber-soled shoes.
- Do not work on electrical lines, pipes, cableways, or steel structures.
- Do not use metal objects such as wading rods, bridge cranes, and well-logging equipment.

- If caught in the open, crouch down low, but do not lie flat on the ground. Avoid standing near isolated trees.
- Avoid working on streams and lakes.
- Seek lower elevations such as valleys or canyons—avoid being on peaks or ridges.
- If you feel your hair standing on end and your skin tingling, this is a sign that lightning might be about to strike—crouch immediately (feet together, hands on knees).

Tornadoes sometimes accompany thunderstorms. Tornadoes are violently rotating columns of air that descend from the clouds in a funnel formation. A weather channel or weather-band radio will sometimes provide advance warning of possible tornadoes.

To protect yourself during a tornado, heed the following advice from Lockhart (1988):

- Seek shelter immediately if there is a sudden, violent change in weather involving wind, rain, hail, or funnel-shaped clouds.
- Avoid occupying vehicles or mobile homes.
- If you are caught outside, find a ravine, ditch, or culvert and lie flat.
- If inside, go to the basement or lowest interior reinforced part of the structure, such as a closet or bathroom. Stay away from windows.

9.8.3 FLOODS

Hurricanes and Monsoonal Rain Events are severe can contain heavy rain. Stay informed as to the projected path of an approaching storm system. Sampling activities should not be conducted until the system has moved out of the area.

Rain can fall at a rate of several inches per hour and rapidly create dangerous flash flood conditions, either in the area where you are working or several miles away. Weather forecasts will be helpful in planning your activities accordingly to ensure your safety. Maintain an updated copy of your district floodplan. Always be aware of rapidly rising stages in rivers and creeks. Beware of dry creekbeds that can become raging rivers in a short period of time. Although earthquakes occur more frequently in tectonically active areas, an earthquake can occur anywhere and without warning.

9.8.4 EARTHQUAKES

Do not panic and run during an earthquake. The greatest danger is from falling objects and walls (includingrock formations). If possible, take cover under a solid structure, such as a table, to protect yourself from falling objects until the shaking stops. If you are out in the open, sit down to avoid losing your balance.

- If an earthquake occurs while driving, slow down and pull over to the side of the road, avoiding overhead structures such as bridges and signs. If on a bridge, try to drive onto solid ground.

9.8.5 FIRE

Fire can spread out of control rapidly—call 911 if you notice a brush fire or other type of threatening fire or smoke. Working inside your field vehicle or outside at your field site requires fire prevention measures. Do not smoke. Keep matches stored in a metal container. Keep fire extinguishers visible and accessible.

- Know how to operate fire extinguishers.
- Know the type of fire for which an extinguisher is designed (extinguishers are different for ordinary combustibles, flammable liquids, and electrical equipment).
- Never point an extinguisher at a person's face.

- **Recharge fire extinguishers according to the schedule provided with the extinguisher.**

9.8.6 SNOW AND ICE

Snow and ice are dynamic mediums that change quickly in structure and strength. Snow and ice can accumulate rapidly, hiding hazards, and creating slippery conditions. Heavy snowfall (white-outs) can be disorienting and can produce avalanche conditions in steep terrain. Working on ice requires experience, training, and knowledge of the water body over which the ice has formed. Wear layers of appropriate clothing and work in teams. Refer to USGS Handbook 445-1-H, chapters 14 and 15, for detailed information.

9.9 ANIMALS

Most sampling activities take place in and around areas inhabited by animals. Before a field trip, try to find out which species inhabit the area and how to deal with them. Table 9-1 summarizes guidelines on what to do if faced with dangerous animals. Note that such guidelines can vary from expert to expert; the right way to deal with encounters could well be what works at the time.

- Most animals will vigorously protect their young and should not be approached or disturbed.
- If a threat or an injury occurs, the most important action is to remain calm and focus on taking the appropriate steps to relieve, remedy, or rescue yourself or another victim. Call for medical advice before transporting the victim, if possible. The most common remedy for bites and stings usually involves basic first aid, followed by immediate medical attention if symptoms warrant. If a member of the field team is allergic to insect bites or stings, this should be made known to all members of the team and noted in the emergency information section of the field folder. Treatment procedures should be reviewed before field activity begins. Persons with known allergic reactions to insects should wear or carry on their person medical alert identification, and carry sting kits for use in emergencies. Arachnids such as scorpions, spiders, and ticks are cause for caution by the field team. Spiders and scorpions are known to inhabit enclosed, dark spaces; for example, inside shoes or the corners of well houses and shelters.

9.9.1 ARACHNIDS AND INSECTS

Scorpions, spiders, and ticks

Scorpions. Scorpions are known to frequent the desert, but also have been found frozen in ice. Scorpions are not easily seen in the wild. They are nocturnal creatures that are sensitive to vibrations, either in the air or on the ground. When humans are stomping around, scorpions usually run for cover. Scorpion stings often involve an encounter between a big toe and a scorpion that has crawled into a shoe. Check shoes and boots left in the field vehicle before putting them on. **Beware of putting your hands and feet into small, dark spaces.**

Spiders. Although few spiders in North America bite people, and the venom of most is harmless (Audubon Society, 1980), exceptions include the black widow and the brown recluse. The black widow (*Latrodectus mactans*) has a fairly large geographical range. Red and brown widow spiders are found mainly in the Gulf Coast region of the United States. The brown recluse (*Loxosceles reclusa*) frequents areas of human habitation and prefers dark spaces such as equipment shelters, as well as areas in the wild. It is advisable to be familiar with the area in which you are working and take care when walking and when reaching into small spaces.

Ticks. Ticks are found nearly everywhere in North America, and can transmit diseases such as Rocky Mountain spotted fever, Lyme disease, human ehrlichiosis, and human granulocytic ehrlichiosis (HGE). To reduce your chances of being exposed to ticks, wear long pants and tuck the pants legs into your socks; use a repellant containing the compound DEET (N-diethyl-meta-toluamide) on exposed skin, except for the face; check your body regularly for ticks, including inspection of the neck and scalp. Remove attached ticks immediately (table 9-1).

Bees and wasps. Venomous insects of common concern include bees and wasps. Bee stingers are equipped with barbs that should be scraped off if a person is stung—using forceps or tweezers to pull the stinger out can force more venom into the wound (table 9-1). A wasp stinger has no barb; the venom is injected and the stinger usually slides out. The yellow jacket (a vespid wasp) nests in the ground and will aggressively defend the nest if disturbed, particularly in the late summer and early fall. Stings in the mouth or throat sometimes result when a bee or wasp has flown into a can of soda. Seek medical attention if symptoms warrant.

9.9.2 SNAKES

Snakes deserve our respect, but they do not have to be feared. Only about 10 percent of the approximately 3,000 species of snakes in the world are poisonous. Differences between poisonous and nonpoisonous snakes of North America are illustrated in figure 9-4. In the event of snakebite, take the victim to the nearest medical facility as soon as possible. Call the medical facility first if time allows. Even a person who has been bitten by a nonpoisonous snake should be treated by medical personnel, because some people are allergic to the foreign protein in snake saliva. Refer to table 9-1 for procedures to follow if bitten. The best advice regarding snake bites is to prevent them in the first place.

Snake expert Maynard Cox (1994) recommends:

- Do not put your hands or feet where you cannot see.
- Never handle a snake unnecessarily, dead or alive, poisonous or nonpoisonous.
- If you come upon a poisonous snake, turn and run. A snake normally can strike up to one-half or two-thirds of its body length, but if provoked it can strike up to its full body length. A common symptom of a poisonous snakebite from pit vipers (copperheads, cottonmouths or water moccasins, and rattlesnakes) is a burning, fiery, stinging pain at the bite site. Other symptoms could include swelling; skin discoloration; nausea and vomiting; a minty, metallic, rubbery taste in the mouth; sweating and chills. If the pain does not get any worse and remains localized, venom probably was not passed. If the pain becomes severe, venom was probably injected.

Copperheads (*Agkistrodon contortix*) have a wide distribution throughout the central, mid-Atlantic, and southern United States. They can be found on wooded hillsides or in areas near water. Although the bite of a copperhead can be painful, it is unlikely to result in an adult human death.

Cottonmouths or water moccasins (*Agkistrodon piscivoros*) are found in the southeastern United States and are never far from water. Cottonmouths usually swim with their entire body on top of the water (Huegel and Cook, undated). Cottonmouths might be seen in the daytime, but they are more active at night. They are an extremely aggressive snake and should not be approached.

Rattlesnakes have been found in every state except Alaska, Delaware, Hawaii, and Maine. All other states have at least one species of rattlesnake, and many have three or four. Arizona, for example, has 17 species or subspecies of rattlesnakes (Kauffeld, 1970). A rattling sound usually alerts that a nearby rattlesnake has been disturbed and can be preparing to strike, but if the snake is sufficiently disturbed, it might not rattle at all.

Coral Snakes. Symptoms are different for bites from coral snakes. The coral snake's venom is extremely toxic, but little or no pain may occur from the bite. Look for teeth marks at the puncture wound. Other symptoms could include euphoria, excess salivation, convulsions, weakness, and *paralysis (Cox, 1994).*

***The Eastern coral* snake** (*Micrurus fulvuis*) is found in the southeastern United States. It is identified by wide red and black bands separated by a narrow, bright yellow band. The red and black bands never touch.

The Arizona coral snake (*Micruroides euryxanthus*) is located in the southwestern United States. The red and black bands also never touch.

9.9.3 BEARS

Bear behavior is unpredictable. According to *Guidelines for Safe Geologic Fieldwork in Alaska* (U.S. Geological Survey, May 1978), bears fiercely defend any carcasses and will often bury or partially cover such prizes for later. Contrary to popular belief, bears can see almost as well as people, but trust their noses much more than their eyes or ears.

Avoiding contact with a bear is the best defense:

- Avoid game trails that bears might use.
- Avoid carrion (dead and decaying flesh), fresh kill, or gut piles.
- Avoid berry patches, or other areas abundant with plants that bears use for food.
- Avoid willow and dry grass patches; bears sometimes use these areas for daybeds.
- Avoid areas with fresh bear tracks.
- Make noise or wear bells when moving through the woods so as not to surprise bears.
- Avoid carrying food that a bear can smell. Always keep food sealed and in a backpack, not in a pants pocket.
- Work in teams of two or larger groups to help deter a bear from attacking.

If you meet a bear on the trail, effective methods of defense can vary:

- If you see the bear before it sees you, decide on your route of escape and leave the area at once.
- If you find yourself close to a bear, give the bear all the room you can.
- Let the bear know you are human—talk in a normal voice and wave your arms. Try to back away slowly, but if the bear follows, stop and hold your ground.
- **Do not run!** Bears often make bluff charges, sometimes within 10 feet of their adversary, without making contact.
- If a bear actually makes contact, surrender! Fall to the ground and play dead. Typically, a bear will break off its attack once it feels the threat has been eliminated. If the bear continues to bite after you assume a defensive posture, the attack is predatory and you should fight back vigorously.

9.9.4 Mountain Lions

Mountain lions (*Felis concolor*) are a species of larger cats found in North America (also called cougar, puma, and panther). The primary habitat for these members of the cat family is west of the Rocky Mountains and south of the Yukon, although Florida has a small population. Mountain lions are active during the day and night, and search a wide territorial range for food. The main food source is deer and smaller animals. Be alert to reports of mountain lion attacks or sightings. Healthy mountain lions do not usually attack humans, but when this occurs, it is usually because the person panicked and ran. Turning and running can evoke a predatory response from mountain lions.

Table 9-1. Guidelines on potentially dangerous animals

[mm, millimeter; in., inch; cm, centimeter; ft, foot; lb, pound; mph, mile per hour]

Animals	Description/ Characteristics	Procedure
Arachnids and Other Insects		
Black widow spiders (Black, brown and red widow spiders are illustrated in figure 9-3.)	Female (only one that bites) is black with abdomen almost spherical, usually with red hourglass mark below or with 2 transverse red marks separated by black. Spiderling is orange, brown, and white, gaining more black at each molt. Habitat among fallen branches and under objects, such as well shelters, furniture, and trash.	**If bitten, seek medical attention as soon as possible.**
Brown recluse spiders	Orange-yellow thorax with dark violin pattern. Bases of legs orange-yellow, rest of legs grayish to dark brown. Abdomen grayish to dark brown with no obvious pattern. Habitat outdoors in sheltered corners, among loose debris; indoors on the floor and behind furniture in houses and outbuildings.	
Scorpions	Nocturnal, sensitive to vibrations. Field boots are a favorite hiding place.	
Ticks	Small, less than 3 mm (<1/8 in.). Clamps to host using a dart-like anchor located just below the mouth.	**Do** • Check for ticks during and after field work. • Remove with tweezers within 24 hours. **Don't** • Leave the head imbedded. • Extract using matches or applying petroleum jelly or other coating.
Bees	Bees vary in size from 2 mm (0.08 in.) long to 4 cm (1.6 in.) long; divided into a number of family classifications which are determined by mouthparts and other characteristics that are difficult to see without dissection.	**Do** • Avoid all bee hives and wasp nests. • Scrape off the bee stinger with a knife or other flat object. • Use an over-the-counter sting ointment or a solution of baking soda, meat tenderizer, and ammonia. **Don't** • Use forceps or tweezers to pull the bee stinger out.
Wasps	Wasps vary in size from minute up to 5 cm (2 in.) long; adults distinguished by a narrow waist between the first and second abdominal segments.	
Snakes and Alligators		
Copperhead snake Cottonmouths or water moccasin snakes Rattlesnakes Eastern coral snakes and Arizona coral snakes	Elliptical eyes; short, stubby tail. Elliptical eyes; short, stubby tail. Usually swim with their entire body on top of the water. Never far from water. Most active at night. An extremely aggressive snake. Elliptical eyes; short, stubby tail. The rattle is a sign of fear, but if the snake is sufficiently disturbed, it might not rattle at all. Wide red and black bands separated by a narrow, bright yellow band; the red and black bands never touch; round pupils; short, stubby tail.	**Do not confront a snake—turn and run** **If bitten:** **Do** • Reassure victim. • Treat for shock. Keep victim lying down; elevate feet 10 to 12 in. • Seek medical attention as soon as possible. Call medical facility while en route, if possible. **Don't** • Cut and suck affected area. • Apply ice or a tourniquet. • Leave victim unattended.

Table 9-1. Guidelines on potentially dangerous animals–*Continued*

Animals	Description/ Characteristics	Procedure
Snakes and Alligators—*Continued*		
Alligators	Fairly inactive when water is cool. Most active during breeding season (mostly April and May). Alligators can run quickly for short distances.	**Don't** approach an alligator.
Mammals		
Polar bears	White, 8-10 ft in length, male 600-1,200 lb, female 400-700 lb, carnivores (primarily).	**Do** • Make your presence known (sing, talk, tie bells to pack). • Travel with a group. • Give bears plenty of room. • Play dead if attacked. Lie flat on stomach or curl up in a ball with hands behind neck. Remain motionless as long as possible, until the bear is gone. If bear continues attack long after you play dead, it is probably a predatory attack. FIGHT BACK VIGOROUSLY! **Don't** • Run. Bears can run up to 35 mph. • Imitate bear sounds or make a high-pitched squeal.
Black bears	Brown to black, white patch in front of chest, 5 ft in length, male 150-400 lb, female 125-250 lb, herbivores (primarily).	
Brown bears	Dark brown to blonde, 7-9 ft in length, male 400-1,100 lb, female 200-600 lb, herbivores (primarily).	
Mountain lions	Active during the day and night. Healthy mountain lions do not usually attack humans.	**Do** • Stand your ground. • Shout and wave your arms. • Throw rocks at the animal. **Don't** • Turn your back. • Panic and run.
Rodents and small mammals: mice, chipmunks, rats, skunks, squirrels, raccoons, bats, foxes, coyotes	Animals infected with hantavirus show no signs of illness. Virus is transmitted from being bitten, or when infective saliva or excreta are inhaled as aerosols produced directly from the infected rodent, or when dried or fresh material contaminated by rodent excreta are disturbed, directly introduced into broken skin, introduced onto the conjunctivae (mucous membrane covering the eyeball), or possibly ingested in contaminated food or water. Signs of an animal infected with rabies are nervousness, aggressiveness, excessive drooling and foaming at the mouth, abnormal behavior, such as wild animals losing their fear of humans or nocturnal animals being seen in the daytime.	**Do** • Wash the wound from a bite or scratch thoroughly with soap and water. • Seek medical attention. • Notify game warden or health department. **Don't** • Capture animal. • Pet or feed wild or domestic animals.
Domestic animals: cats, dogs, cows		

9.10 PLANTS

The most common poisonous plants are the *Toxicodendrons*, or poison ivies, and include climbing and nonclimbing poison ivy, eastern and western poison oak, and poison sumac (fig. 9-5). Resins and secretions from these species are capable of inflicting a mild to serious skin rash on susceptible individuals (Frankel, 1991). The best defense against *Toxicodendrons* and other poisonous plants is proper clothing. Long pants and sleeves will protect you in most cases, but plant poisons have been known to penetrate clothing. In the field, always wear shoes that protect your whole foot. The oily resin of the plant is only slightly soluble in water, and the best treatment is to flush the area of skin contact with copious amounts of cold water (table 9-2). A little water spreads the poison; lots of water washes it away. Soap can remove natural skin oils that protect against penetration of the resins; warm water can also hasten the absorption.

Table 9-2. Guidelines on poisonous plants

Plant	Description/Characteristics	Procedure
Poison ivy *(Toxicodendron rydbergii)*	• Climbing poison ivy has alternate, trifoliate leaves, tiny greenish-white flower clusters or white berries, and aerial roots that grow straight and are fuzzy. Ubiquitous in most environments (seldom found in deep, dark forests or at heights above 4,000 ft) (Frankel, 1991). • Non-climbing poison ivy lacks aerial roots. The leaves are larger and broader than the climbing variety, but the pattern is still alternate and trifoliate.	**In case of skin contact:** **Do** • Flood the affected area with copious amounts of **cold** water as soon as possible. **Don't** • Use soap. • Use warm water.
Eastern poison oak *(Toxicodendron toxicarium)*	Prefers the sandy soil of the Atlantic and Gulf coasts. Distinguished by trifoliate fuzzy leaves, fuzzy fruits, and leaflets with rounded tips.	
Western poison oak *(Toxicodendron diversilobum)*	Strictly a Pacific coast species. Usually grows as a short shrub but can grow as a vine, due to aerial roots. Trifoliate leaves.	
Poison sumac *(Toxicodendron vernix)*	A tree that prefers a moist habitat such as bogs or swamps. Ranges in height from 6 to 20 feet. The alternate leaves are compound with 7 to 13 lobeless, toothless leaflets arranged in a featherlike fashion with a single leaflet on the end. Can have white berries. The fruits of poisonous varieties of sumac droop downward.	

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

11. Appendix D: 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	31. IN WHICH VEHICLE	32. LOCATION IN VEHICLE	33. FIRST AID GIVEN BY
☐ KILLED ☐ DRIVER ☐ PASSENGER ☐ INJURED ☐ HELPER ☐ PEDESTRIAN	☐ FED ☐ OTHER (2)		

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	41. IN WHICH VEHICLE	42. LOCATION IN VEHICLE	43. FIRST AID GIVEN BY
☐ KILLED ☐ DRIVER ☐ PASSENGER ☐ INJURED ☐ HELPER ☐ PEDESTRIAN	☐ FED ☐ OTHER (2)		

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.)*		

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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.

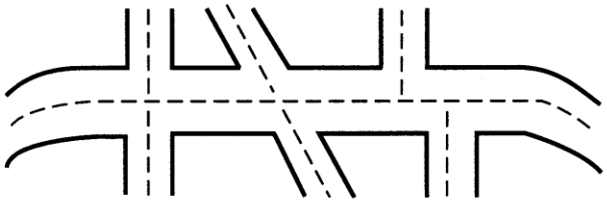
Example: → 1 2 ←

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)

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 ☐ YES ☐ NO	b. COMMENTS

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

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

12. Appendix E: Material Safety Data Sheets

MSDS List for Water Quality Monitoring	
Item	**Company**
Colilert	IDEXX
Colilert Comparator	IDEXX
Colilert Polystyrene Sample Vessels	IDEXX
Colilert Quanti-Tray	IDEXX
Conductivity Standard, 147 umhos	Aqua Solutions
Conductivity Standard, 1413 umhos	Aqua Solutions
Conductivity Standard, 12900 umhos	Aqua Solutions
Field Thermometer	H-B Instrument
High Vacuum Grease	Dow Corning
pH Electrode Filling Solution	In-Situ
pH Electrode Storage Solution	In-Situ
pH 4 Buffer Solution	Aqua Solutions
pH 7 Buffer Solution	Aqua Solutions
pH 10 Buffer Solution	Aqua Solutions
Quat-128 Disinfectant	Waxie, Inc
Sample Bottle with Nitric Acid Preservative	Utah Public Health Laboratory
Sample Bottle with Sulfuric Acid Preservative	Utah Public Health Laboratory
Sodium Sulfite, Anhydrous	Hach Company
Sodium Sulfite Solution	In-Situ
Turbidity StablCal Calibration Set	Hach Company

Colilert 98-12972-00, 98-12973-00, 98-14770-00, 98-14771-00
98-26016-00, 98-26017-00, 98-27163-00, 98-17506-00, 98-28942-00

1. IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING

Product name(s)	Colilert
Product code(s)	98-12972-00, 98-12973-00, 98-14770-00, 98-14771-00, 98-26016-00, 98-26017-00, 98-27163-00 98-17506-00, 98-28942-00 WP020I, WP200I, W100I, W200I, WB250-20I, WB250-100I, WP100I, WCHN200, WCPV-020I, WCPV-100I
Intended uses	Water microbiology.

Company	IDEXX Laboratories, Inc	IDEXX Laboratories Pty Ltd.	IDEXX Europe B.V.
	One IDEXX Drive	Metro Centre	Scorpius 60 Building F
	Westbrook ME 04092	Unit 20, 38-46 South Street	Hoofddorp 2132 LR
	United States	Rydalmere, NSW 2116 Australia	The Netherlands
Telephone	1-800-548-6733	1-800-655-978	00800 727 43399
Fax	1-207-556-4346	0-800-634-409	00800 433 99329

24 hour Emergency Phone # CHEMTREC 1-800-424-9300
Outside U.S +(00)-1-703-527-3887
Australia +(61)-290372994

2. HAZARDS IDENTIFICATION

GHS Classification

Skin Irritation, Category 3

Pictogram	No Symbol	
Signal Word	Warning	
Hazard statement(s)	H316	Causes mild skin irritation.
Precautionary statement(s)	P332+P313	If skin irritation occurs: Get medical advice/attention.

Classification according to Regulation (EC) No 1272/2008

Not a hazardous substance or mixture according to Regulation (EC) No 1272/2008.

Classification according to EU Directives 67/548/EEC or 1999/45/EC

This substance is not classified as dangerous according to Directive 67/548/EEC.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Substance	CAS-No.	EC-No.	Index-No.	RTECS-No.	Concentration
Ammonium sulfate	7783-20-2	231-984-1	-	BS4500000	-
Sodium chloride	7647-14-5	231-588-3	-	VZ4725000	-

4. FIRST AID MEASURES

IF INHALED:	Remove victim to fresh air and keep at rest in a position comfortable for breathing.
IF ON SKIN:	Wash with plenty of soap and water. If skin irritation occurs: Get medical advice/attention.
IF IN EYES:	Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. If eye irritation persists: Get medical advice/attention.
IF SWALLOWED:	Rinse mouth.

5. FIRE FIGHTING MEASURES

Suitable extinguishing media

Use extinguishing measures that are appropriate to the local circumstance and the surrounding environment.

Advice for firefighters

Wear self contained breathing apparatus for firefighting if necessary.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions, protective equipment and emergency procedures
Use personal protective equipment as required.

Environmental precautions
No special environmental precautions required.

Methods and materials for containment and cleaning up
Sweep up and shovel.

7. HANDLING AND STORAGE

Precautions for safe handling
Wash hands thoroughly after handling.

Conditions for safe storage
Keep container tightly closed.

8. EXPOSURE CONTROLS / PERSONNAL PROTECTION

Exposure Controls
Handle in accordance with good industrial hygiene and safety practice.

Personal protective equipment

Respiratory protection	No special protective equipment required.
Skin protection	Handle with gloves. Nitrile rubber.
Eye/Face protection	No special protective equipment required.
Body protection	No special protective equipment required.

9. PHYSICAL AND CHEMICAL PROPERTIES

Form	solid
Color (Colour)	white
Odor (Odour)	no data available
pH	not applicable
Melting point	no data available
Initial boiling point	no data available
Flash point	not applicable
Flammability	not applicable
Explosive properties	not applicable
Oxidizing properties	not applicable
Vapor (Vapour) pressure	no data available
Relative density	no data available
Water solubility	soluble

10. STABILITY AND REACTIVITY

Chemical stability	no data available.
Incompatible materials	Oxidizing agents.
Hazardous decomposition products	Carbon oxides, Nitrogen oxides.

11. TOXICOLOGICAL INFORMATION

Ammonium sulfate

Acute toxicity	LD50 Oral – rat – 2,840 mg/kg
Skin corrosion/irritation	Skin - rabbit - No skin irritation
Serious eye damage/eye irritation	Eyes - rabbit - No eye irritation
Respiratory or skin sensitization	no data available
Germ cell mutagenicity	no data available
Carcinogenicity	IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
Reproductive toxicity	no data available
Specific Target Organ Toxicity –Single Exposure	no data available
Specific Target Organ Toxicity –Repeated Exposure	no data available
Aspiration hazard	no data available

Sodium chloride

Acute toxicity	LC50 Inhalation - rat - 1 h - > 42,000 mg/m3 LD50 Dermal - rabbit - > 10,000 mg/kg
Skin corrosion/irritation	no data available
Serious eye damage/eye irritation	no data available
Respiratory or skin sensitization	no data available
Germ cell mutagenicity	no data available
Carcinogenicity	IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
Reproductive toxicity	no data available
Specific Target Organ Toxicity –Single Exposure	no data available
Specific Target Organ Toxicity –Repeated Exposure	no data available
Aspiration hazard	no data available

12. ECOLOGICAL INFORMATION

Ammonium sulfate

Toxicity to fish	LC50 - Oncorhynchus mykiss (rainbow trout) - 36.7 mg/l - 96 h
Toxicity to daphnia and other aquatic invertebrates	LC50 - Daphnia magna (Water flea) - 433 mg/l - 50 h
Toxicity to algae	no data available
Persistence and degradability	no data available
Bioaccumulative potential	no data available
Mobility in soil	no data available
PBT and vPvB assessment	no data available

Sodium chloride

Toxicity to fish	no data available
Toxicity to daphnia and other aquatic invertebrates	NOEC - Daphnia - 1,500 mg/l - 7 d LC50 - Daphnia magna (Water flea) - 1,661 mg/l - 48 h
Toxicity to algae	no data available

Persistence and degradability	no data available
Bioaccumulative potential	no data available
Mobility in soil	no data available
PBT and vPvB assessment	no data available

13. DISPOSAL CONSIDERATIONS

Dispose of contents in accordance with local/regional/national/international regulations.

14. TRANSPORT INFORMATION

DOT (US)	Not a dangerous good
ADR/RID	Not a dangerous good
IMDG	Not a dangerous good
ICAO/IATA	Not a dangerous good

15. REGULATORY INFORMATION

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

This material safety data sheet was prepared in accordance with NBR 14725 by Association of Brazil Technical Standards.

	Ammonium sulfate	Sodium chloride
Germany (WGK)	1	1

16. OTHER INFORMATION

HMIS Rating

Health hazard	1	Flammability	0	Physical hazard	0

1. IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING

Product name(s) Colilert Comparator
Product code(s) Presence/Absence - WP104, 98-11682-00
MPN Tube - W102, 98-20722-00
Quanti-Tray – WQTC, 98-09226-00
Quanti-Tray 2000 – WQT2KC, 98-09227-00
Recommended uses and restrictions Water Microbiology

Company	*IDEXX Laboratories, Inc.* *One IDEXX Drive* *Westbrook, ME 04092* *United States*	*IDEXX Laboratories Pty Ltd.* *Metro Centre* *Unit 20, 38-46 South Street* *Rydalmere, NSW 2116 Australia*	*IDEXX Europe B.V.* *Scorpius 60 Building F* *Hoofddorp 2132 LR* *The Netherlands*
Telephone	*1-800-548-6733*	*1-800-655-978*	*00800 727 43399*
Fax	*1-207-556-4346*	*1-800-634-409*	*00800 433 99329*

24 hour Emergency Phone # *CHEMTREC 1-800-424-9300*
Outside U.S 1-703-527-3887

2. HAZARDS IDENTIFICATION

According to Regulation (EC) No1272/2008

Not a hazardous substance or mixture according to Regulation (EC) No 1272/2008.

According to European Directive 67/548/EEC as amended.

This substance is not classified as dangerous according to Directive 67/548/EEC.

3. COMPOSITION/INFORMATION ON INGREDIENTS

This substance is not classified as dangerous.

4. FIRST AID MEASURES

IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
IF ON SKIN: Wash with plenty of soap and water.
IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
IF SWALLOWED: Rinse mouth.

5. FIRE FIGHTING MEASURES

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for firefighters
Wear self contained breathing apparatus for firefighting if necessary.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions
Use personal protective equipment as required. Wash thoroughly after handling.

Environmental precautions
Avoid release to the environment.

Methods and materials for containment and cleaning up
Wipe up with absorbent material.

7. HANDLING AND STORAGE

Precautions for safe handling
Use personal protective equipment as required. Wash thoroughly after handling.

Conditions for safe storage
Keep container tightly closed.

8. EXPOSURE CONTROLS / PERSONNAL PROTECTION

Personal protective equipment

Respiratory protection	*In case of inadequate ventilation, wear respiratory protection.*
Hand protection	*Handle with gloves.*
Eye protection	*Safety glasses.*
Skin protection	*Wear protective clothing.*
Hygiene measures	*Wash thoroughly after handling. Do not get in eyes, on skin, or on clothing.*

9. PHYSICAL AND CHEMICAL PROPERTIES

Form	*liquid*
Color (Colour)	*yellow*
Odor (Odour)	*odorless*
pH	*7.0 – 8.0*
Melting point	*no data available*
Boiling point	*no data available*
Flash point	*not applicable*
Flammability	*not applicable*
Explosive properties	*not applicable*
Oxidizing properties	*not applicable*
Vapor (Vapour) pressure	*no data available*
Density	*no data available*
Water solubility	*soluble*

10. STABILITY AND REACTIVITY

Chemical stability	*Stable under recommended storage conditions.*
Materials to avoid	*Oxidizing agents.*
Hazardous decomposition products	*Carbon oxides, Nitrogen oxides.*

11. TOXICOLOGICAL INFORMATION

Acute toxicity	*no data available*
Skin corrosion/irritation	*no data available*
Serious eye damage/eye irritation	*no data available*
Respiratory or skin sensitization	*no data available*
Germ cell mutagenicity	*no data available*
Carcinogenicity	*no data available*
Reproductive toxicity	*no data available*

Specific Target Organ Toxicity –Single Exposure	*no data available*
Specific Target Organ Toxicity –Repeated Exposure	*no data available*
Aspiration hazard	*no data available*

12. ECOLOGICAL INFORMATION

Toxicity to fish	*no data available*
Toxicity to daphnia and other aquatic invertebrates.	*no data available*
Toxicity to algae	*no data available*
Persistence and degradability	*no data available*
Bioaccumulative potential	*no data available*
Mobility in soil	*no data available*
PBT and vPvB assessment	*no data available*

13. DISPOSAL CONSIDERATIONS

Dispose of contents in accordance with local/regional/national/international regulations.

14. TRANSPORT INFORMATION

DOT (US) *Not a dangerous good*
ICAO/IATA *Not a dangerous good*
IMDG *Not a dangerous good*
ADR/RID *Not a dangerous good*

15. REGULATORY INFORMATION

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

16. OTHER INFORMATION

HMIS Rating

Health hazard 0 *Flammability* 0 *Physical hazard* 0

MATERIAL SAFETY DATA SHEET

Polystyrene Vessels 98-09220-00, 98-09204-00, 98-09221-00, 98-09222-00, 98-09223-00, 98-14079-00, 98-06161-00, 98-09588-00, 98-14269-00, 98-18739-00, 98-09203-00, 98-09228-00, 98-06159-00, 98-06160-00

1. IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING

Product name(s) Polystyrene Vessels

Product code(s) 98-09220-00, 98-09204-00, 98-09221-00, 98-09222-00, 98-09223-00, 98-14079-00, 98-06161-00, 98-09588-00, 98-14269-00, 98-18739-00, 98-09203-00, 98-09228-00, 98-06159-00, 98-06160-00
WV120-20, WV120-200, WV120SB-20, WV120SB-200, WV120BST-20, WV120BST-200, WV120ST-20, WV120ST-200, WV150SB-20, WV150SB-200, WV120SBAF-200, WV150SBST-200, WV290SBST-100, WV290SB-100

Recommended uses and restrictions Water Microbiology

Company	*IDEXX Laboratories, Inc.*	*IDEXX Laboratories Pty Ltd.*	*IDEXX Europe B.V.*
	One IDEXX Drive	*Metro Centre*	*Scorpius 60 Building F*
	Westbrook, ME 04092	*Unit 20, 38-46 South Street*	*Hoofddorp 2132 LR*
	United States	*Rydalmere, NSW 2116 Australia*	*The Netherlands*
Telephone	*1-800-548-6733*	*1-800-655-978*	*00800 727 43399*
Fax	*1-207-556-4346*	*1-800-634-409*	*00800 433 99329*

24 hour Emergency Phone # CHEMTREC 1-800-424-9300
Outside U.S 1-703-527-3887

2. HAZARDS IDENTIFICATION

According to Regulation (EC) No1272/2008

Not a hazardous substance or mixture according to Regulation (EC) No 1272/2008.

According to European Directive 67/548/EEC as amended.

This substance is not classified as dangerous according to Directive 67/548/EEC.

3. COMPOSITION/INFORMATION ON INGREDIENTS

This substance is not classified as dangerous.

4. FIRST AID MEASURES

IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
IF ON SKIN: Wash with plenty of soap and water.
IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
IF SWALLOWED: Rinse mouth.

5. FIRE FIGHTING MEASURES

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for firefighters
Wear self contained breathing apparatus for firefighting if necessary.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions
Use personal protective equipment as required. Wash thoroughly after handling.

Environmental precautions
Avoid release to the environment.

Methods and materials for containment and cleaning up
Sweep up and shovel.

7. HANDLING AND STORAGE

Precautions for safe handling
Use personal protective equipment as required. Wash thoroughly after handling.

Conditions for safe storage
Keep container tightly closed.

8. EXPOSURE CONTROLS / PERSONNAL PROTECTION

Personal protective equipment

Respiratory protection	*In case of inadequate ventilation, wear respiratory protection.*
Hand protection	*Handle with gloves.*
Eye protection	*Safety glasses.*
Skin protection	*Wear protective clothing.*
Hygiene measures	*Wash thoroughly after handling. Do not get in eyes, on skin, or on clothing.*

9. PHYSICAL AND CHEMICAL PROPERTIES

Form	*solid*
Color (Colour)	*no data available*
Odor (Odour)	*odorless*
pH	*not applicable*
Melting point	*no data available*
Boiling point	*no data available*
Flash point	*not applicable*
Flammability	*not applicable*
Explosive properties	*not applicable*
Oxidizing properties	*not applicable*
Vapor (Vapour) pressure	*no data available*
Density	*no data available*
Water solubility	*no data available*

10. STABILITY AND REACTIVITY

Chemical stability	*Stable under recommended storage conditions.*
Materials to avoid	*Oxidizing agents.*
Hazardous decomposition products	*Carbon oxides, Nitrogen oxides.*

11. TOXICOLOGICAL INFORMATION

Acute toxicity	*no data available*
Skin corrosion/irritation	*no data available*
Serious eye damage/eye irritation	*no data available*
Respiratory or skin sensitization	*no data available*
Germ cell mutagenicity	*no data available*
Carcinogenicity	*no data available*
Reproductive toxicity	*no data available*
Specific Target Organ Toxicity –Single Exposure	*no data available*
Specific Target Organ Toxicity –Repeated Exposure	*no data available*
Aspiration hazard	*no data available*

12. ECOLOGICAL INFORMATION

Toxicity to fish	*no data available*
Toxicity to daphnia and other aquatic invertebrates.	*no data available*
Toxicity to algae	*no data available*
Persistence and degradability	*no data available*
Bioaccumulative potential	*no data available*
Mobility in soil	*no data available*
PBT and vPvB assessment	*no data available*

13. DISPOSAL CONSIDERATIONS

Dispose of contents in accordance with local/regional/national/international regulations.

14. TRANSPORT INFORMATION

DOT (US) *Not a dangerous good*
ICAO/IATA *Not a dangerous good*
IMDG *Not a dangerous good*
ADR/RID *Not a dangerous good*

15. REGULATORY INFORMATION

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

16. OTHER INFORMATION

HMIS Rating

Health hazard 0 *Flammability* 0 *Physical hazard* 0

1. IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING

Product name(s) Quanti-Tray
Product code(s) 98-21378-00, 98-21675-00
WQT100, WQT2K
Recommended uses and restrictions Water Microbiology

Company	*IDEXX Laboratories, Inc.* *One IDEXX Drive* *Westbrook, ME 04092* *United States*	*IDEXX Laboratories Pty Ltd.* *Metro Centre* *Unit 20, 38-46 South Street* *Rydalmere, NSW 2116 Australia*	*IDEXX Europe B.V.* *Scorpius 60 Building F* *Hoofddorp 2132 LR* *The Netherlands*
Telephone	*1-800-548-6733*	*1-800-655-978*	*00800 727 43399*
Fax	*1-207-556-4346*	*1-800-634-409*	*00800 433 99329*

24 hour Emergency Phone # *CHEMTREC 1-800-424-9300*
Outside U.S 1-703-527-3887

2. HAZARDS IDENTIFICATION

According to Regulation (EC) No1272/2008

Not a hazardous substance or mixture according to Regulation (EC) No 1272/2008.

According to European Directive 67/548/EEC as amended.

This substance is not classified as dangerous according to Directive 67/548/EEC.

3. COMPOSITION/INFORMATION ON INGREDIENTS

This substance is not classified as dangerous.

4. FIRST AID MEASURES

IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
IF ON SKIN: Wash with plenty of soap and water.
IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
IF SWALLOWED: Rinse mouth.

5. FIRE FIGHTING MEASURES

Suitable extinguishing media
Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for firefighters
Wear self contained breathing apparatus for firefighting if necessary.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions
Use personal protective equipment as required. Wash thoroughly after handling.

Environmental precautions
Avoid release to the environment.

Methods and materials for containment and cleaning up
Wipe up with absorbent material.

7. HANDLING AND STORAGE

Precautions for safe handling
Use personal protective equipment as required. Wash thoroughly after handling.

Conditions for safe storage
Keep container tightly closed.

8. EXPOSURE CONTROLS / PERSONNAL PROTECTION

Personal protective equipment

Respiratory protection	*In case of inadequate ventilation, wear respiratory protection.*
Hand protection	*Handle with gloves.*
Eye protection	*Safety glasses.*
Skin protection	*Wear protective clothing.*
Hygiene measures	*Wash thoroughly after handling. Do not get in eyes, on skin, or on clothing.*

9. PHYSICAL AND CHEMICAL PROPERTIES

Form	*solid*
Color (Colour)	*no data available*
Odor (Odour)	*odorless*
pH	*not applicable*
Melting point	*no data available*
Boiling point	*no data available*
Flash point	*not applicable*
Flammability	*not applicable*
Explosive properties	*not applicable*
Oxidizing properties	*not applicable*
Vapor (Vapour) pressure	*no data available*
Density	*no data available*
Water solubility	*no data available*

10. STABILITY AND REACTIVITY

Chemical stability	*Stable under recommended storage conditions.*
Materials to avoid	*Oxidizing agents.*
Hazardous decomposition products	*Carbon oxides, Nitrogen oxides.*

11. TOXICOLOGICAL INFORMATION

Acute toxicity	*no data available*
Skin corrosion/irritation	*no data available*
Serious eye damage/eye irritation	*no data available*
Respiratory or skin sensitization	*no data available*
Germ cell mutagenicity	*no data available*
Carcinogenicity	*no data available*
Reproductive toxicity	*no data available*
Specific Target Organ Toxicity –Single Exposure	*no data available*

Specific Target Organ Toxicity –Repeated Exposure	*no data available*
Aspiration hazard	*no data available*

12. ECOLOGICAL INFORMATION

Toxicity to fish	*no data available*
Toxicity to daphnia and other aquatic invertebrates.	*no data available*
Toxicity to algae	*no data available*
Persistence and degradability	*no data available*
Bioaccumulative potential	*no data available*
Mobility in soil	*no data available*
PBT and vPvB assessment	*no data available*

13. DISPOSAL CONSIDERATIONS

Dispose of contents in accordance with local/regional/national/international regulations.

14. TRANSPORT INFORMATION

DOT (US) *Not a dangerous good*
ICAO/IATA *Not a dangerous good*
IMDG *Not a dangerous good*
ADR/RID *Not a dangerous good*

15. REGULATORY INFORMATION

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

16. OTHER INFORMATION

HMIS Rating

Health hazard 0 *Flammability* 0 *Physical hazard* 0

Aqua Solutions, Inc
6913 Hwy 225
Deer Park, TX 77536

MATERIAL SAFETY DATA SHEET

Phone: (281) 479-2569
Chemtrec 800-424-9300
24 Hour Emergency Assistance

SECTION 1 IDENTIFICATION

Product Number	6906
Product Name	Conductivity Standard 147 µmhos
Trade /Chemical Synonyms	
Formula	Not Applicable
RTECS	None
C.A.S. #	See below

Health:	1
Flammability:	0
Reactivity:	0

Hazard Rating:
Least Slight Moderate High Extreme
0 1 2 3 4

NA=Not Applicable NE=Not Established

SECTION 2 MIXTURE COMPONENTS

Sara 313	Component	CAS Numbe	%	Dim.	Exposure Limits
☐	Potassium Chloride	CAS# 7447-40-7	<3%	W/V	None Established
☐	Water, Deionized ASTM Type II	CAS# 7732-18-5	Balance	V/V	None Established

SECTION 3 HAZARD IDENTIFICATION (SEE ALSO SECTION 11)

Generally not hazardous in normal handling, however good laboratory practices should always be used. Avoid long term exposure to skin or by inhalation.

SECTION 4 FIRST AID MEASURES

FIRST AID: SKIN: Wash exposed area with soap and water. If irritation persists, seek medical attention.

EYES: Wash eyes with plenty of water for at least 15 minutes, lifting lids occasionally. Seek Medical Aid. INHALATION: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen

INGESTION: Give several glasses of milk or water. Vomiting may occur spontaneously, but DO NOT INDUCE! Never give anything by mouth to an unconscious person.

SECTION 5 FIRE FIGHTING MEASURES

Fire Extinguisher Type: Any means suitable for extinguishing surrounding fire

Fire / Explosion Hazards: None Known.

Fire Fighting Procedure:

Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and clothing.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Absorb spill with inert material. Flush excess down drain.

SECTION 7 HANDLING AND STORAGE

Store in a cool dry place. This material is not considered hazardous. Handle using safe laboratory practices.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

Respiratory Protection: None required

Ventilation: Local Exhaust ☑ Mechanical ☐

Protective Gloves: Wear appropriate gloves to prevent skin exposure

Eye Protection: Splash Goggles

Other Protective Equipment: Use safe laboratory handling procedures.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Melting Point:	Information not available	**Percent Volatile by Volume:**	>95
Boiling Point:	~100°C	**Evaporation Rate:**	Not Applicable
Vapor Pressure:	Not Applicable		
Vapor Density:	Information not available	**Evaporation Standard:**	
Solubility in Water:	Soluble	**Auto Ignition Temp:**	Not Applicable
Appearance /Odors:	Clear, colorless, odorless liquid	**Lower Flamm. Limit in Air:**	Not Applicable
Flash Point:	Not Applicable		
Specific Gravity:	~1	**Upper Flamm. Limit in Air:**	Not Applicable

SECTION 10 STABILITY AND REACTIVITY INFORMATION

Stability: Stable

Conditions to Avoid: None known

Materials to Avoid: None

Hazardous Decomposition Products: None known

Hazardous polymerization Will Not Occur

Conditions to Avoid: None known

SECTION 11 ADDITIONAL INFORMATION

Conditions aggravated/target organs: Persons with preexisting skin, eye or respiratory conditions may be more susceptible. Acute: Possible irritation to skin, eyes, lungs, mucous membranes and GI tract. Chronic: None known.

DOT Classification: Not Regulated

DOT Regulations may change from time to time. Please consult the most recent D.O.T. regulations.

Rev. No: 0.1 Date Entered: 3/4/2009 Approved: WPF Safety Dir.: WPF

The information herein is believed to be accurate and is offered in good faith for the user's consideration and investigation. No warranty either expressed or implied is made for the completeness or accuracy of the information whether originating from the above mentioned company or not. Users of this material should satisfy themselves by independent investigation of current scientific and medical knowledge that the material can be used safely.

Aqua Solutions, Inc
6913 Hwy 225
Deer Park, TX 77536

MATERIAL SAFETY DATA SHEET

Phone: (281) 479-2569
Chemtrec 800-424-9300
24 Hour Emergency Assistance

SECTION 1 IDENTIFICATION

Product Number	6920
Product Name	Conductivity Standard 0.01 Molar (1,413 µmhos)
Trade /Chemical Synonyms	
Formula	Not Applicable
RTECS	None
C.A.S. #	See below

Health:	1
Flammability:	0
Reactivity:	0

Hazard Rating:
Least Slight Moderate High Extreme
0 1 2 3 4

NA=Not Applicable NE=Not Established

SECTION 2 MIXTURE COMPONENTS

Sara 313	Component	CAS Numbe	%	Dim.	Exposure Limits
☐	Potassium Chloride	CAS# 7447-40-7	<1%	W/V	None Established
☐	Water, Deionized ASTM Type II	CAS# 7732-18-5	Balance	V/V	None Established

SECTION 3 HAZARD IDENTIFICATION (SEE ALSO SECTION 11)

Generally not hazardous in normal handling, however good laboratory practices should always be used. Avoid long term exposure to skin or by inhalation.

SECTION 4 FIRST AID MEASURES

FIRST AID: SKIN: Wash exposed area with soap and water. If irritation persists, seek medical attention.

EYES: Wash eyes with plenty of water for at least 15 minutes, lifting lids occasionally. Seek Medical Aid. INHALATION: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen

INGESTION: Give several glasses of milk or water. Vomiting may occur spontaneously, but DO NOT INDUCE! Never give anything by mouth to an unconscious person.

SECTION 5 FIRE FIGHTING MEASURES

Fire Extinguisher Type: Any means suitable for extinguishing surrounding fire

Fire / Explosion Hazards: None

Fire Fighting Procedure:

Use any means suitable for extinguishing surrounding fire. Use water spray cool fire exposed containers.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Absorb spill with inert material. Flush excess down drain.

SECTION 7 HANDLING AND STORAGE

This Material is not considered hazardous. Handle using safe laboratory practices.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

Respiratory Protection: None required

Ventilation: Local Exhaust ☑ Mechanical ☐

Protective Gloves: Wear appropriate gloves to prevent skin exposure

Eye Protection: Safety Glasses w/Side Shields

Other Protective Equipment: None required

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Melting Point:	~0° C	Percent Volatile by Volume:	>99%
Boiling Point:	~100° C	Evaporation Rate:	N/A
Vapor Pressure:	N/E	Evaporation Standard:	Not Applicable
Vapor Density:	information not available	Auto Ignition Temp:	N/A
Solubility in Water:	Soluble	Lower Flamm. Limit in Air:	N/A
Appearance /Odors:	Clear water white / no odor	Upper Flamm. Limit in Air:	N/A
Flash Point:	N/A		
Specific Gravity:	1		

SECTION 10 STABILITY AND REACTIVITY INFORMATION

Stability: Stable

Conditions to Avoid: Protect from freezing.

Materials to Avoid: None

Hazardous Decomposition Products: Not known to occur.

Hazardous polymerization Will Not Occur

Conditions to Avoid: None known

SECTION 11 ADDITIONAL INFORMATION

Conditions aggravated/target organs: Persons with preexisting skin, eye or respiratory disorders may be more susceptible. Acute: Essentially non-hazardous. Possible eye and digestive tract irritation. Chronic: None known.

DOT Classification: Not Regulated

DOT Regulations may change from time to time. Please consult the most recent D.O.T. regulations.

Rev. No: 0.1 Date Entered: 3/4/2009 Approved: WPF Safety Dir.: WPF

The information herein is believed to be accurate and is offered in good faith for the user's consideration and investigation. No warranty either expressed or implied is made for the completeness or accuracy of the information whether originating from the above mentioned company or not. Users of this material should satisfy themselves by independent investigation of current scientific and medical knowledge that the material can be used safely.

Aqua Solutions, Inc
6913 Hwy 225
Deer Park, TX 77536

MATERIAL SAFETY DATA SHEET

Phone: (281) 479-2569
Chemtrec 800-424-9300
24 Hour Emergency Assistance

SECTION 1 IDENTIFICATION

Product Number	6931
Product Name	Conductivity Standard 12,900 µmhos
Trade /Chemical Synonyms	
Formula	Not Applicable
RTECS	None
C.A.S. #	See below

Health:	1
Flammability:	0
Reactivity:	0

Hazard Rating:
Least Slight Moderate High Extreme
0 1 2 3 4

NA=Not Applicable NE=Not Established

SECTION 2 MIXTURE COMPONENTS

Sara 313	Component	CAS Numbe	%	Dim.	Exposure Limits
☐	Potassium Chloride	CAS# 7447-40-7	<2%	W/V	None Established
☐	Water, Deionized ASTM Type II	CAS# 7732-18-5	Balance	V/V	None Established

SECTION 3 HAZARD IDENTIFICATION (SEE ALSO SECTION 11)

Generally not hazardous in normal handling, however good laboratory practices should always be used. Avoid long term exposure to skin or by inhalation.

SECTION 4 FIRST AID MEASURES

FIRST AID: SKIN: Wash exposed area with soap and water. If irritation persists, seek medical attention.

EYES: Wash eyes with plenty of water for at least 15 minutes, lifting lids occasionally. Seek Medical Aid. INHALATION: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen

INGESTION: Give several glasses of milk or water. Vomiting may occur spontaneously, but DO NOT INDUCE! Never give anything by mouth to an unconscious person.

SECTION 5 FIRE FIGHTING MEASURES

Fire Extinguisher Type: Any means suitable for extinguishing surrounding fire

Fire / Explosion Hazards: None

Fire Fighting Procedure:

Use any means suitable for extinguishing surrounding fire. Use water spray cool fire exposed containers.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Absorb spill with inert material. Flush excess down drain.

SECTION 7 HANDLING AND STORAGE

This material is not considered hazardous. Handle using safe laboratory practices.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

Respiratory Protection: None required

Ventilation: Local Exhaust ☑ Mechanical ☐

Protective Gloves: Wear appropriate gloves to prevent skin exposure

Eye Protection: Splash Goggles

Other Protective Equipment: None required

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Melting Point:	~0°C	**Percent Volatile by Volume:**	>98
Boiling Point:	100°C	**Evaporation Rate:**	Information not available
Vapor Pressure:	Information not available		
Vapor Density:	Information not available	**Evaporation Standard:**	
Solubility in Water:	Soluble	**Auto Ignition Temp:**	Not applicable
Appearance /Odors:	Clear, colorless, odorless liquid	**Lower Flamm. Limit in Air:**	Not applicable
Flash Point:	None		
Specific Gravity:	1	**Upper Flamm. Limit in Air:**	Not applicable

SECTION 10 STABILITY AND REACTIVITY INFORMATION

Stability: Stable

Conditions to Avoid: None known

Materials to Avoid: Concentrated Acids and Bases, Water reactive materials.

Hazardous Decomposition Products: None known

Hazardous polymerization Will Not Occur

Conditions to Avoid: None known

SECTION 11 ADDITIONAL INFORMATION

Overexposure symptoms. Acute: Essentially non-hazardous. Possible irritation of eyes/stomach. Chronic: None known. Conditions aggravated/Target organs: none known.

DOT Classification: Not Regulated

DOT Regulations may change from time to time. Please consult the most recent D.O.T. regulations.

Rev. No: 0.1 Date Entered: 3/4/2009 Approved: WPF Safety Dir.: WPF

The information herein is believed to be accurate and is offered in good faith for the user's consideration and investigation. No warranty either expressed or implied is made for the completeness or accuracy of the information whether originating from the above mentioned company or not. Users of this material should satisfy themselves by independent investigation of current scientific and medical knowledge that the material can be used safely.

Material Safety Data Sheet
Easy-Read® Liquid Inside Thermometer

Manufacturer:
H-B Instrument Company
102 West Seventh Avenue
Trappe, PA 19426 USA

Date: March 10, 2007
Supersedes MSDS of February 10, 2004
13201484
13202512

EMERGENCY TELEPHONE
Chem-Tel, Inc.
1-800-255-3924

INFORMATION TELEPHONE
H-B Instrument Company
610-489-5500
1-800-4-TEST-LAB
Fax: 610-489-9100

I. PRODUCT INFORMATION

Identity: Easy-Read® Liquid Inside Thermometer
Chemical Name: Mixture of Biodegradable Technical White Oil, Natural Citrus Oils and Black Anthroquinone Dye
Chemical Family: Hydrocarbon and Monoazo–Anthroquinone Dye
Formula: Not Applicable
CAS Registry Number: N/A
DOT Shipping Name: N/A
DOT Hazard Class: N/A

Contents of thermometers less than 0.3 ml. This product is exempt in containers under 110 gallons.
DOT Reg. #49CFR 173.118A

II. HAZARDOUS INGREDIENTS

Component(s)	Hazard Information		
	OSHA PEL	ACGIH TLV	OTHERS
NONE KNOWN	N/A	N/A	N/A

No other hazards per OSHA 29 CFR1910.1200. None under Title III of Sara

III. PHYSICAL AND CHEMICAL CHARACTERISTICS

Vapor Pressure (mm HG):	Not Determined
Vapor Density (Air = 1):	Not Determined
Solubility in Water:	<0.03% By Weight
Appearance and Odor:	Black Liquid, Citrus Smell, Volatile
Specific Gravity (H20-1):	0.806 at 23°C (ASTM D-792)
Melting Point:	N/A
Evaporation Rate (Butyl Acetate = 1):	0.04

IV. FIRE AND EXPLOSION HAZARD DATA

Flash Point (Method Used):	73.3°C (T.C.C. ASTM D-56)
Flammability Limits in Air, LEL:	Not Known
UEL:	Not Known
Extinguishing Media for Fires:	Water Fog, Carbon Dioxide, Foam, Dry Chemical
Special Fire Fighting Procedures:	Do not use stream of water
Unusual Fire and Explosion Hazards:	May produce dense black smoke and potentially toxic fume if bulk quantity of liquid is allowed to combust.

Material Safety Data Sheet
Easy-Read® Liquid Inside Thermometer

V. REACTIVITY DATA

Stability:	Stable
Conditions to Avoid (Stability):	Away from heat or flame
Materials to Avoid:	Strong oxidizing or reducing agent
Hazardous Decomposition Products:	Oxides of carbon, nitrogen and sulfer on combustion
Hazardous Polymerization Product:	May Not Occur
Conditions to Avoid (Polymerization):	None Known

VI. HEALTH HAZARD DATA

Carcinogen or Potential Carcinogen	NTP:	IARC:	OSHA:
	N/A	N/A	N/A

Health Hazard (Acute of Chronic)

Skin:	Skin corrosive agent per USFHSA, NOT EXPECTED TO BE A HAZARD IN THE NORMAL INTENEDED USE OF THE THERMOMETER
Eyes:	NOT AN EYE IRRITANT PER USFHSA
Ingestion:	Acute oral LD50 > 5g/kg in rat, human ingestion hazard not known
Inhalation:	Not Known
Effects of Over-Exposure:	Corrosive to skin, NOT EXPECTED TO BE A HAZARD IN THE NORMAL INTENDED USE OF THE THERMOMETER
Medical conditions generally Aggravated by Exposure:	Not Known

EMERGENCY FIRST AID PROCEDURES

Skin: Wash immediately with water. Consult physician if irritation occurs
Eyes: Wash immediately with water. Consult physician if irritation occurs
Ingestion: Do not induct vomiting. Consult physician at once
Inhalation: If liquid enters into lung, consult physician at once

VII. PRECAUTIONS FOR SAFE HANDLING AND USE

Spill or Leak Procedures:	Absorb with dry sand or oil absorbent
Disposal Recommendations:	Comply with federal, state or local regulation
Storage & Handling Recommendations:	Keep away from heat or flame
Other precautions:	None Known

VIII. CONTROL MEASURES

Respiratory Protection:	None required under normal use condition
Ventilation:	Local
Protective Gloves:	None required under normal use conditions
Eye Protection:	None required under normal use conditions
Other Protective Equipment or Clothing:	Chemical-resistant protective clothing if handling bulk quantity of liquid
Work Hygiene Practice:	Standard Industrial Hygiene Practices Recommended in Handling Bulk Quantity of Liquid

IX. REGULATORY INFORMATION

OSHA Compliance:	Please see Section II
State Right-To-Know Compliance:	
Pennsylvania:	No known hazardous component
New Jersey:	No known hazardous component
Massachusetts:	No known hazardous component
California:	No known hazardous component
Federal Compliance:	May contain component(s) subjected to EPA TSCA Section 8(e) Status Report – May be regulated under other Federal Regulations

X. WARRANTY LIMITATION

H-B Instrument Company believes that the information contained herein (including data and statements) is accurate as of the date hereof. NO WARRANTY OF MERCHANTABILITY, FITNESS FOR ANY PARTICULAR PURPOSE, OR ANY OTHER WARRANTY, EXPRESS OR IMPLIED, IS MADE AS CONCERNS THAT INFORMATION HEREIN PROVIDED. The information provided herein relates only to the specific product designated and may not be valid where such product is used in combination with any other materials or in any process. Further, since the conditions and methods of use of the product and the information referred to herein are beyond the control of H-B Instrument Company, H-B Instrument Company expressly disclaims any and all liability as to any results obtained or arising from any use of the product or such information. No statement made herein shall be construed as a permission or recommendation for the use of any product in a manner that might infringe existing patents. Technical information included does contain information provided by vendors to H-B Instrument Company.

SECTION I — IDENTIFICATION

Emergency Phone Number:	480-929-0070
Effective Date:	12/30/1999
Revised Date:	01/01/2002
Chemical Name:	Solvent Black 000
Trade Name:	Calco Oil Black ZMM
Chemical Family:	Anthraquinone, Anthraquinone, Disazo
Chemical Formula:	Solvent Green 3 CAS# 128-80-3, Solvent Violet 13 CAS# 81-48-1, Solvent Yellow 72 CAS# 4645-07-2, Solvent Red 26 CAS #4477-79-6

SECTION II — HAZARDOUS INGREDIENTS

Hazardous Components	Hazardous %	TLV (Units)	Prod. CAS#
None as per 29CFR Part 1910.1200			Listed above
None under Title III of SARA			

SECTION III — PHYSICAL DATA

Boiling Point (°F):	N/A
Freezing Point (°F):	N/A
Volatility/Vol. (%):	N/A
Melting Point:	N/A
Vapor Pressure:	N/A
Vapor Density (Air=1):	N/A
Solubility in H_2O:	Soluble
Appearance/Odor:	Dark Powder/No odor
Specific Gravity (H_2O=1):	N/A
Evaporation Rate:	N/A
pH:	N/A

SECTION IV — FIRE AND EXPLOSION HAZARD DATA

Flash Point:	N/A
Lower Flame Limit:	N/A
Higher Flame Limit:	N/A
Extinguish Media:	In case of fire use water spray, foam, dry chemical or CO_2.
For Fire:	Dust may form explosive mixture with air.
Unusual Fire Hazard:	None

SECTION V — HEALTH HAZARD DATA

Threshold Limit Value: N/A
Overexposure Effects: LD50, Oral (ingestion) – greater than 5000 Mg/Kg; no further information was found concerning any adverse acute/chronic health effects resulting from overexposure to this product.

First Aid Procedures:
Inhalation: If large amounts are inhaled, remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult give oxygen and call a physician.
Skin Contact: Flush skin with plenty of water after excessive contact. Wash thoroughly with hand cream or hand cleaner.
Eye Contact: Flush eyes immediately with plenty of water for at least 15 minutes. Call a physician.
Ingestion: Induce vomiting immediately by giving two glasses of water. Get medical attention. Never give fluids to an unconscious person.

SECTION VI — REACTIVITY DATA

Chemical Stability: Stable
Conditions to Avoid: None
Incompatible Materials: Oxidizing and reducing agents may destroy color
Decomposition Products: CO, CO2, Oxides of Nitrogen, Sulfur, and other potentially toxic fumes
Hazardous Polymerization
Polymerization Avoid: None expected

SECTION VII — SPILL OR LEAK PROCEDURE

For Spill: Spills should be swept up and placed in containers. Spill areas can be washed with water. Collect wastewater for approved disposal.

Waste Disposal Method: Waste disposal should be in accordance with existing federal, state and local environmental regulations.

SECTION VII (A) — REGULATORY INFORMATION

MITI (Japanese): We do not have access to inventory
AICS (Australian): We do not have access to inventory
Calif. Prop. 65: None unless indicated in Sect. II
EPA (TSCA): All components are in compliance
EINECS/ELINCS: All components should be listed; see CAS number(s)
RCRA: Not regulated
CERLA: Not regulated
WHMIS: See component(s) – CAS numbers
USDA: Many products are on file. Please check with our registration department.
DSL/NDSL: All components should be listed – see CAS number(s)
Carcinogenicity: NTP, IARC, OSHA: None unless indicated in Section II of this MSDS.

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Easy-Read® Liquid Black Dye

SECTION VIII — SPECIAL PROTECTION

Respiratory Protection: Employees should avoid inhalation of dusts. Whenever potential for dusting exists, appropriate NIOSH/MSHA approved respirator with dust filter should be worn.
Ventilation: Use local ventilation if dusting is a problem
Protective Gloves: Wear suitable gloves
Eye Protection: Wear eye/face protection
Other Protective Equip.: None
Handling & Storage: In accordance with good industrial practices. Handle with care to avoid contact.

SECTION IX — SPECIAL PRECAUTIONS

Hazard Class: None
DOT Shipping Name: None
Reportable Quantity (RQ): None
UN Number: None
NA Number: None
Packaging Size: N/A

The data and information given in this Material Safety Data Sheet are accurate on the date of preparation. It does not indicate any warranty or representation. We disclaim all liability relating to use of material since this is beyond our control.

DOW CORNING(R) HIGH VACUUM GREASE

1. PRODUCT AND COMPANY IDENTIFICATION

Dow Corning Corporation
South Saginaw Road
Midland, Michigan 48686

24 Hour Emergency Telephone: (989) 496-5900
Customer Service: (989) 496-6000
Product Disposal Information: (989) 496-6315
CHEMTREC: (800) 424-9300

MSDS No.: 01018817 Revision Date: 2009/05/07

Generic Description: Silicone compound
Physical Form: Grease
Color: Translucent white
Odor: Odorless

NFPA Profile: Health 0 Flammability 1 Instability/Reactivity 0

Note: NFPA = National Fire Protection Association

2. HAZARDS IDENTIFICATION

POTENTIAL HEALTH EFFECTS

Acute Effects

Eye: Direct contact may cause temporary redness and discomfort.

Skin: No significant irritation expected from a single short-term exposure.

Inhalation: No significant effects expected from a single short-term exposure.

Oral: Low ingestion hazard in normal use.

Prolonged/Repeated Exposure Effects

Skin: No known applicable information.

Inhalation: No known applicable information.

Oral: No known applicable information.

Signs and Symptoms of Overexposure

No known applicable information.

Medical Conditions Aggravated by Exposure

No known applicable information.

The above listed potential effects of overexposure are based on actual data, results of studies performed upon similar compositions,

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DOW CORNING(R) HIGH VACUUM GREASE

component data and/or expert review of the product. Please refer to Section 11 for the detailed toxicology information.

3. COMPOSITION/INFORMATION ON INGREDIENTS

None present. This is not a hazardous material as defined in the OSHA Hazard Communication Standard.

4. FIRST AID MEASURES

Eye: Immediately flush with water.

Skin: No first aid should be needed.

Inhalation: No first aid should be needed.

Oral: No first aid should be needed.

Notes to Physician: Treat according to person's condition and specifics of exposure.

5. FIRE FIGHTING MEASURES

Flash Point: 212 °F / 100 °C (Closed Cup)

Autoignition Temperature: Not determined.

Flammability Limits in Air: Not determined.

Extinguishing Media: On large fires use dry chemical, foam or water spray. On small fires use carbon dioxide (CO2), dry chemical or water spray. Water can be used to cool fire exposed containers.

Fire Fighting Measures: Self-contained breathing apparatus and protective clothing should be worn in fighting large fires involving chemicals. Determine the need to evacuate or isolate the area according to your local emergency plan. Use water spray to keep fire exposed containers cool.

Unusual Fire Hazards: None.

6. ACCIDENTAL RELEASE MEASURES

Containment/Clean up:	Determine whether to evacuate or isolate the area according to your local emergency plan. Observe all personal protection equipment recommendations described in Sections 5 and 8. For large spills, provide diking or other appropriate containment to keep material from spreading. If diked material can be pumped, store recovered material in appropriate container. Clean up remaining materials from spill with suitable absorbant. Clean area as appropriate since spilled materials, even in small quantities, may present a slip hazard. Final cleaning may require use of steam, solvents or detergents. Dispose of saturated absorbant or cleaning materials appropriately, since spontaneous heating may occur. Local, state and federal laws and regulations may apply to releases and disposal of this material, as well as those materials and items employed in the cleanup of releases. You will need to determine which federal, state and local laws and regulations are applicable. Sections 13 and 15 of this MSDS provide information regarding certain federal and state requirements.

7. HANDLING AND STORAGE

Use with adequate ventilation. Avoid eye contact.

Use reasonable care and store away from oxidizing materials.

8. EXPOSURE CONTROLS / PERSONAL PROTECTION

Component Exposure Limits

There are no components with workplace exposure limits.

Engineering Controls

Local Ventilation:	None should be needed.
General Ventilation:	Recommended.

Personal Protective Equipment for Routine Handling

Eyes:	Use proper protection - safety glasses as a minimum.
Skin:	Washing at mealtime and end of shift is adequate.
Suitable Gloves:	Handle in accordance with good industrial hygiene and safety practices.
Inhalation:	No respiratory protection should be needed.
Suitable Respirator:	None should be needed.

Personal Protective Equipment for Spills

Eyes:	Use proper protection - safety glasses as a minimum.
Skin:	Washing at mealtime and end of shift is adequate.
Inhalation/Suitable Respirator:	No respiratory protection should be needed.
Precautionary Measures:	Avoid eye contact. Use reasonable care.

Note: These precautions are for room temperature handling. Use at elevated temperature or aerosol/spray applications may require added precautions.

9. PHYSICAL AND CHEMICAL PROPERTIES

Physical Form:	Grease
Color:	Translucent white
Odor:	Odorless
Specific Gravity @ 25°C:	1.1
Viscosity:	2000000 cSt
Freezing/Melting Point:	Not determined.
Boiling Point:	Not determined.
Vapor Pressure @ 25°C:	Not determined.
Vapor Density:	Not determined.
Solubility in Water:	Not determined.
pH:	Not determined.
Volatile Content:	Not determined.
Flash Point:	212 °F / 100 °C (Closed Cup)
Autoignition Temperature:	Not determined.
Flammability Limits in Air:	Not determined.

Note: The above information is not intended for use in preparing product specifications. Contact Dow Corning before writing specifications.

10. STABILITY AND REACTIVITY

Chemical Stability:	Stable.
Hazardous Polymerization:	Hazardous polymerization will not occur.
Conditions to Avoid:	None.
Materials to Avoid:	Oxidizing material can cause a reaction.

Hazardous Decomposition Products

Thermal breakdown of this product during fire or very high heat conditions may evolve the following decomposition products: Carbon oxides and traces of incompletely burned carbon compounds. Silicon dioxide. Formaldehyde.

11. TOXICOLOGICAL INFORMATION

Special Hazard Information on Components

No known applicable information.

12. ECOLOGICAL INFORMATION

Environmental Fate and Distribution

Complete information is not yet available.

Environmental Effects

Complete information is not yet available.

Fate and Effects in Waste Water Treatment Plants

Complete information is not yet available.

Ecotoxicity Classification Criteria

Hazard Parameters (LC50 or EC50)	High	Medium	Low
Acute Aquatic Toxicity (mg/L)	<=1	>1 and <=100	>100
Acute Terrestrial Toxicity	<=100	>100 and <= 2000	>2000

This table is adapted from "Environmental Toxicology and Risk Assessment", ASTM STP 1179, p.34, 1993.

This table can be used to classify the ecotoxicity of this product when ecotoxicity data is listed above. Please read the other information presented in the section concerning the overall ecological safety of this material.

13. DISPOSAL CONSIDERATIONS

RCRA Hazard Class (40 CFR 261)

When a decision is made to discard this material, as received, is it classified as a hazardous waste? No

State or local laws may impose additional regulatory requirements regarding disposal.

14. TRANSPORT INFORMATION

DOT Road Shipment Information (49 CFR 172.101)

Not subject to DOT.

Ocean Shipment (IMDG)

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Not subject to IMDG code.

Air Shipment (IATA)

Not subject to IATA regulations.

15. REGULATORY INFORMATION

Contents of this MSDS comply with the OSHA Hazard Communication Standard 29 CFR 1910.1200.

TSCA Status: All chemical substances in this material are included on or exempted from listing on the TSCA Inventory of Chemical Substances.

EPA SARA Title III Chemical Listings

Section 302 Extremely Hazardous Substances (40 CFR 355):
None.

Section 304 CERCLA Hazardous Substances (40 CFR 302):
None.

Section 311/312 Hazard Class (40 CFR 370):
Acute: No
Chronic: No
Fire: No
Pressure: No
Reactive: No

Section 313 Toxic Chemicals (40 CFR 372):
None present or none present in regulated quantities.

Note: Chemicals are listed under the 313 Toxic Chemicals section only if they meet or exceed a reporting threshold.

Supplemental State Compliance Information

California

Warning: This product contains the following chemical(s) listed by the State of California under the Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65) as being known to cause cancer, birth defects or other reproductive harm.

None known.

Massachusetts

CAS Number	Wt %	Component Name
7631-86-9	7.0 - 13.0	Silica, amorphous

New Jersey

CAS Number	Wt %	Component Name
63148-62-9	> 60.0	Polydimethylsiloxane
7631-86-9	7.0 - 13.0	Silica, amorphous
70131-67-8	5.0 - 10.0	Dimethyl siloxane, hydroxy-terminated

Pennsylvania

CAS Number	Wt %	Component Name
63148-62-9	> 60.0	Polydimethylsiloxane
7631-86-9	7.0 - 13.0	Silica, amorphous
70131-67-8	5.0 - 10.0	Dimethyl siloxane, hydroxy-terminated

16. OTHER INFORMATION

Prepared by: Dow Corning Corporation

These data are offered in good faith as typical values and not as product specifications. No warranty, either expressed or implied, is hereby made. The recommended industrial hygiene and safe handling procedures are believed to be generally applicable. However, each user should review these recommendations in the specific context of the intended use and determine whether they are appropriate.

(R) indicates Registered Trademark

In-Situ Inc. Innovations in Water Monitoring

Material Safety and Data Sheet

I. Chemical Product and Company Identification

Product Name:	Manufacturer:	Emergency Contact:
pH Reference Fill Solution (4M Potassium Chloride, Saturated with Silver Chloride)	Aqua Phoenix Scientific, Inc. 320 Maple Ave. Hanover, PA 17331 Telephone: 866 632 1291 Fax: 717 633 1285	INFOTRAC Emergency Response Hotline: 1-800-535-5053 (in the U.S. and Canada) 1-352-323-3500 www.infotrac.net

II. Composition, Information on Ingredients

Hazardous Components Specific Chemical Identity: Common Names	CAS NO.	%	OSHA PEL	ACGIH TLV
Potassium Chloride	7447-40-7	30% w/v	N/A	N/A
Water, purified	7732-18-5	>70% w/v	N/A	N/A

III. Hazard Identification

Emergency Overview: May cause irritation to the eyes and skin. Wash areas of contact. Get medical attention if irritation develops.

Target Organs: Eyes, Skin

Eyes	May cause irritation, redness, pain, tearing
Skin	May cause irritation, redness, pain
Ingestion	May cause upset stomach
Inhalation	Not likely to be a hazard by inhalation
Chronic Effect /Carcinogenicity	None (IARC, NTP, OSHA)

IV. First Aid

Eyes	Immediately flush eyes with water for at least 15 minutes. Immediately get medical assistance.
Skin	Flush with water for 15 minutes. Get medical assistance if irritation develops.
Ingestion	Dilute with water or milk. Get medical assistance.
Inhalation	Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

V. Fire Fighting Measures

Flash Point	N/A
Extinguishing Media	Any means suitable for extinguishing fire.
Fire and Explosion Hazards	Not considered to be a fire or explosion hazard.
Fire Fighting Instructions/Equipment	Use normal procedures. Use protective clothing. Use NIOSH-approved breathing equipment.
NFPA Rating	(estimated) Health: 1; Flammable: 0; Reactivity: 0

VI. Accidental Release Measures

Absorb with suitable material. Always obey local regulations.

VII. Handling and Storage

Handling	Wash hands after handling. Avoid contact with skin and eyes.
Storage	Protect from freezing and physical damage. Store above 20°C to prevent crystallization. If crystallization does occur, gently warm in hot water bath and occasionally agitating to re-dissolve.

VIII. Exposure Controls, Personal Protection

Engineering Controls	Normal ventilation is adequate
Respiratory Controls	Normal ventilation is adequate
Skin Protection	Chemical resistant gloves
Eye Protection	Safety Glasses or goggles

IX. Physical and Chemical Properties

Appearance	Clear, colorless liquid	**Odor**	Odorless
pH @ 25°C	N/A	**Solubility in Water**	Infinite
Boiling Point	Approx 100°C	**Specific Gravity**	Approx 1.13-1.17
Melting point	Approx 0°C	**Vapor Pressure**	N/A

X. Stability and Reactivity

Chemical Stability	Stable under normal conditions of use and storage
Incompatibility	Bromine trifluoride and potassium permanganate with sulfuric acid
Hazardous Decomposition Products	Oxides of potassium
Hazardous Polymerization	Does not occur

XI. Toxicological Information

LD50 orl-rat	2600 mg/kg (Potassium Chloride)
LC50 inhalation-rat	N/A

XII. Ecological Information

Ecotoxicity	N/A

XIII. Disposal Considerations

Absorb with suitable material.
All chemical waster generators must determine whether a discarded chemical is classified as hazardous waste. Comply with all local, state, and federal regulations.

XIV. Transport Information

DOT	Not Regulated

XV. Regulatory Information (not meant to be all inclusive)

OSHA Status	These chemicals are not considered highly hazardous by OSHA.
TSCA	The components of this solution are listed on the TSCA Inventory.
SARA Title III Section 313	N/A
RCRA Status	N/A
CERCLA Reportable Quality	N/A
WHMIS	N/A

XVI. Additional Information

Issue Date: 3/9/07
Revision Date: 3/8/10, Rev. 002
Document: 0059630

* N/A – Not Applicable/Not Available

Disclaimer: The information, data and recommendations contained herein were provided to In-Situ Inc. by the manufacturer named on this Material Safety Data Sheet. In-Situ Inc. makes no warranty of any kind whatsoever with respect thereto and disclaims all liability from reliance thereon. In-Situ Inc. reserves the right to revise this Material Safety Data Sheet as new information is provided to it by the manufacturer.

In-Situ Inc.

For more information contact In-Situ Inc.
221 East Lincoln Avenue, Fort Collins, CO 80524
1-800-446-7488 (toll-free in U.S. & Canada
1-970-498-1500 (international & domestic)
www.in-situ.com

In-Situ Inc. Innovations in Water Monitoring

Material Safety and Data Sheet

I. Chemical Product and Company Identification

Product Name:	Manufacturer:	Emergency Contact:
Electrode Storage Solution	Aqua Phoenix Scientific, Inc. 320 Maple Ave. Hanover, PA 17331 Telephone: 866 632 1291 Fax: 717 633 1285	INFOTRAC Emergency Response Hotline: 1-800-535-5053 (in the U.S. and Canada) 1-352-323-3500 www.infotrac.net

II. Composition, Information on Ingredients

Hazardous Components Specific Chemical Identity: Common Names	CAS NO.	%	OSHA PEL	ACGIH TLV
Potassium Acid Phthalate	877-24-7	<1% w/v	N/A	N/A
Potassium Chloride	7447-40-7	<8% w/v	N/A	N/A
Water, purified	7732-18-5	>91% w/v	N/A	N/A

III. Hazard Identification

Emergency Overview: May cause eye, skin, respiratory, and digestive tract irritation. Wash areas of contact with water.
Target Organs: Eyes, Skin

Eyes	May cause slight irritation.
Skin	May cause slight irritation.
Ingestion	May cause gastrointestinal irritation, nausea, diarrhea, and vomiting.
Inhalation	May cause respiratory tract irritation.
Chronic Effect /Carcinogenicity	None (IARC, NTP, OSHA).

IV. First Aid

Eyes	Immediately flush eyes with water for at least 15 minutes. Immediately get medical assistance.
Skin	Flush with water for 15 minutes. Get medical assistance if irritation develops.
Ingestion	DO NOT induce vomiting. Dilute with water or milk. Get medical assistance.
Inhalation	Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

V. Fire Fighting Measures

Flash Point	N/A
Extinguishing Media	Use means suitable to extinguishing surrounding fire.
Fire and Explosion Hazards	Not considered to be a fire or explosion hazard.
Fire Fighting Instructions/Equipment	Use normal procedures. Poisonous gases may be produced in fire. Use protective clothing. Use NIOSH-approved breathing equipment.
NFPA Rating	(estimated) Health: 1; Flammable: 0; Reactivity: 0

VI. Accidental Release Measures

Absorb with suitable material. Always obey local regulations.

VII. Handling and Storage

Handling	Wash hands after handling. Avoid contact with skin and eyes.
Storage	Protect from freezing and physical damage. Store in cool, dry place.

VIII. Exposure Controls, Personal Protection

Engineering Controls	Normal ventilation is adequate
Respiratory Controls	Normal ventilation is adequate
Skin Protection	Chemical resistant gloves
Eye Protection	Safety Glasses or goggles

IX. Physical and Chemical Properties

Appearance	Clear, colorless liquid	**Odor**	Odorless
pH @ 25°C	N/A	**Solubility in Water**	Miscible
Boiling Point	Approx 100°C	**Specific Gravity**	Approx 1
Melting point	Approx 0°C	**Vapor Pressure**	14mm Hg at 20°C

X. Stability and Reactivity

Chemical Stability	Stable under normal conditions of use and storage
Incompatibility	N/A
Hazardous Decomposition Products	N/A
Hazardous Polymerization	Does not occur

XI. Toxicological Information

LD50 orl-rat	>90 mL/kg
LC50 inhalation-rat	N/A

XII. Ecological Information

Ecotoxicity	N/A

XIII. Disposal Considerations

Absorb with suitable material. Flush to sewer with large quantities of water.
All chemical waster generators must determine whether a discarded chemical is classified as hazardous waste. Comply with all local, state, and federal regulations.

XIV. Transport Information

DOT	Not Regulated

XV. Regulatory Information (not meant to be all inclusive)

OSHA Status	These chemicals are not considered hazardous by OSHA.
TSCA	The components of this solution are listed on the TSCA Inventory.
SARA Title III Section 313	N/A
RCRA Status	N/A
CERCLA Reportable Quality	N/A
WHMIS	N/A

XVI. Additional Information

Issue Date: 9/07
Revision Date: 3/8/10, Rev. 001
Document: 0032850

* N/A – Not Applicable/Not Available

Disclaimer: The information, data and recommendations contained herein were provided to In-Situ Inc. by the manufacturer named on this Material Safety Data Sheet. In-Situ Inc. makes no warranty of any kind whatsoever with respect thereto and disclaims all liability from reliance thereon. In-Situ Inc. reserves the right to revise this Material Safety Data Sheet as new information is provided to it by the manufacturer.

In-Situ Inc.

For more information contact In-Situ Inc.
221 East Lincoln Avenue, Fort Collins, CO 80524
1-800-446-7488 (toll-free in U.S. & Canada
1-970-498-1500 (international & domestic)
www.in-situ.com

Aqua Solutions, Inc
6913 Hwy 225
Deer Park, TX 77536

MATERIAL SAFETY DATA SHEET

Phone: (281) 479-2569
Chemtrec 800-424-9300
24 Hour Emergency Assistance

SECTION 1 IDENTIFICATION

Product Number	1460
Product Name	Buffer Solution, pH 4 (Red)
Trade /Chemical Synonyms	
Formula	Not Applicable
RTECS	None
C.A.S. #	See below

Health:	1
Flammability:	0
Reactivity:	0

Hazard Rating:
Least Slight Moderate High Extreme
0 1 2 3 4

NA=Not Applicable NE=Not Established

SECTION 2 MIXTURE COMPONENTS

Sara 313	Component	CAS Numbe	%	Dim.	Exposure Limits
☐	Methyl Red Sodium Salt	CAS# 845-10-3	< 0.01	W/V	None Established
☐	Potassium Hydrogen Phthalate	CAS# 877-24-7	1.02%	W/V	TXDS: orl-rat LD•□: 3200 r
☐	Thymol Crystal	CAS# 89-83-8	0.01%	W/V	TXDS: orl-rat LD•□: 640 m
☐	Water, Deionized ASTM Type II	CAS# 7732-18-5	Balance	V/V	None Established

SECTION 3 HAZARD IDENTIFICATION (SEE ALSO SECTION 11)

May be harmful if swallowed. May cause irritation. Avoid breathing vapors, or dusts. Use with adequate ventilation. Avoid contact with eyes, skin, and clothes. Wash thoroughly after handling. Keep container closed.

SECTION 4 FIRST AID MEASURES

FIRST AID: SKIN: Wash exposed area with soap and water. If irritation persists, seek medical attention.

EYES: Wash eyes with plenty of water for at least 15 minutes, lifting lids occasionally. Seek Medical Aid. INHALATION: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen

INGESTION: If swallowed, induce vomiting immediately after giving two glasses of water. Never give anything by mouth to an unconscious person.

SECTION 5 FIRE FIGHTING MEASURES

Fire Extinguisher Type: Any means suitable for extinguishing surrounding fire

Fire / Explosion Hazards: None

Fire Fighting Procedure:

Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and clothing.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Absorb spill with inert material. Flush excess down drain.

SECTION 7 HANDLING AND STORAGE

Store in a cool dry place. This Material is not considered hazardous. Handle using safe laboratory practices.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

Respiratory Protection: None required

Ventilation: Local Exhaust ☑ Mechanical ☐

Protective Gloves: Gloves to prevent skin exposure as latex or vinyl

Eye Protection: Safety Glasses w/Side Shields

Other Protective Equipment: Use safe laboratory handling procedures.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Melting Point:	~0° C	**Percent Volatile by Volume:**	> 99%
Boiling Point:	~100° C	**Evaporation Rate:**	Information not available
Vapor Pressure:	Information not available	**Evaporation Standard:**	
Vapor Density:	Information not available	**Auto Ignition Temp:**	Not applicable
Solubility in Water:	Soluble	**Lower Flamm. Limit in Air:**	Not applicable
Appearance /Odors:	Clear red solution/Slight benzoic acid odor	**Upper Flamm. Limit in Air:**	Not applicable
Flash Point:	not flammable		
Specific Gravity:	~1		

SECTION 10 STABILITY AND REACTIVITY INFORMATION

Stability: Stable

Conditions to Avoid: High temperature, air

Materials to Avoid: Strong acids and bases

Hazardous Decomposition Products: Not known to occur

Hazardous polymerization Will Not Occur

Conditions to Avoid: None known

SECTION 11 ADDITIONAL INFORMATION

Overexposure symptoms. Acute: Essentially non-hazardous. Possible irritation of eyes/stomach. Chronic: None known. Conditions aggravated/Target organs: none known.

DOT Classification: Not Regulated

DOT Regulations may change from time to time. Please consult the most recent D.O.T. regulations.

Rev. No: 0.3 Date Entered: 3/4/2009 Approved: WPF Safety Dir.: WPF

The information herein is believed to be accurate and is offered in good faith for the user's consideration and investigation. No warranty either expressed or implied is made for the completeness or accuracy of the information whether originating from the above mentioned company or not. Users of this material should satisfy themselves by independent investigation of current scientific and medical knowledge that the material can be used safely.

Aqua Solutions, Inc
6913 Hwy 225
Deer Park, TX 77536

MATERIAL SAFETY DATA SHEET

Phone: (281) 479-2569
Chemtrec 800-424-9300
24 Hour Emergency Assistance

SECTION 1 IDENTIFICATION

Product Number	1500
Product Name	Buffer Solution, pH 7 (Yellow)
Trade /Chemical Synonyms	
Formula	Not Applicable
RTECS	None
C.A.S. #	See below

Health:	1
Flammability:	0
Reactivity:	0

Hazard Rating:
Least Slight Moderate High Extreme
0 1 2 3 4

NA=Not Applicable NE=Not Established

SECTION 2 MIXTURE COMPONENTS

Sara 313	Component	CAS Numbe	%	Dim.	Exposure Limits
☐	Potassium Phosphate Monobasic	CAS# 7778-77-0	0.68%	W/V	None Established
☑	Sodium Hydroxide	CAS# 1310-73-2	0.01%	W/V	OSHA PEL 2 mg/m*f* ACGI 2mg/m*f*
☐	Water, Deionized ASTM Type II	CAS# 7732-18-5	Balance	V/V	None Established

SECTION 3 HAZARD IDENTIFICATION (SEE ALSO SECTION 11)

Generally not hazardous in normal handling, however good laboratory practices should always be used. Avoid long term exposure to skin or by inhalation.

SECTION 4 FIRST AID MEASURES

FIRST AID: SKIN: Wash exposed area with soap and water. If irritation persists, seek medical attention.

EYES: Wash eyes with plenty of water for at least 15 minutes, lifting lids occasionally. Seek Medical Aid. INHALATION: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen

INGESTION: If swallowed, induce vomiting immediately after giving two glasses of water. Never give anything by mouth to an unconscious person.

SECTION 5 FIRE FIGHTING MEASURES

Fire Extinguisher Type: Any means suitable for extinguishing surrounding fire

Fire / Explosion Hazards: None

Fire Fighting Procedure:

Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and clothing.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Absorb spill with inert material. Flush excess down drain.

SECTION 7 HANDLING AND STORAGE

Store in a cool dry place. This Material is not considered hazardous. Handle using safe laboratory practices.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

Respiratory Protection: None required

Ventilation: Local Exhaust ☑ Mechanical ☐

Protective Gloves: Gloves to prevent skin exposure as latex or vinyl

Eye Protection: Splash Goggles

Other Protective Equipment: Use safe laboratory handling procedures.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Melting Point:	~0°C	**Percent Volatile by Volume:**	>98
Boiling Point:	~100°C	**Evaporation Rate:**	Information not available
Vapor Pressure:	Information not available		
Vapor Density:	Information not available	**Evaporation Standard:**	
Solubility in Water:	Soluble	**Auto Ignition Temp:**	Not applicable
Appearance /Odors:	Clear, yellow, odorless liquid	**Lower Flamm. Limit in Air:**	Not applicable
Flash Point:	Not flammable		
Specific Gravity:	~1	**Upper Flamm. Limit in Air:**	Not applicable

SECTION 10 STABILITY AND REACTIVITY INFORMATION

Stability: Stable

Conditions to Avoid: High temperatures

Materials to Avoid: Concentrated Acids and Bases, Water reactive materials.

Hazardous Decomposition Products: None known

Hazardous polymerization Will Not Occur

Conditions to Avoid: None known

SECTION 11 ADDITIONAL INFORMATION

Conditions aggravated/target organs: None known. Acute: Essentially non-hazardous. Possible irritation of eyes/stomach. Chronic: none known.

DOT Classification: Not Regulated

DOT Regulations may change from time to time. Please consult the most recent D.O.T. regulations.

Rev. No: 0.1 Date Entered: 8/1/2007 Approved: WPF Safety Dir.: WPF

The information herein is believed to be accurate and is offered in good faith for the user's consideration and investigation. No warranty either expressed or implied is made for the completeness or accuracy of the information whether originating from the above mentioned company or not. Users of this material should satisfy themselves by independent investigation of current scientific and medical knowledge that the material can be used safely.

Aqua Solutions, Inc
6913 Hwy 225
Deer Park, TX 77536

MATERIAL SAFETY DATA SHEET

Phone: (281) 479-2569
Chemtrec 800-424-9300
24 Hour Emergency Assistance

SECTION 1 IDENTIFICATION

Product Number	1525
Product Name	Buffer Solution, pH 10.0 (Blue)
Trade /Chemical Synonyms	
Formula	Not Applicable
RTECS	None
C.A.S. #	See below

Health:	1
Flammability:	0
Reactivity:	0

Hazard Rating:
Least Slight Moderate High Extreme
0 1 2 3 4

NA=Not Applicable NE=Not Established

SECTION 2 MIXTURE COMPONENTS

Sara 313	Component	CAS Numbe	%	Dim.	Exposure Limits
☐	Bromothymol Blue Sodium Salt	CAS# 34722-90-	<0.01%	W/V	None established
☐	Sodium Bicarbonate	CAS# 144-55-8	0.21%	W/V	None Established
☐	Sodium Carbonate Anhydrous	CAS# 497-19-8	0.26%	W/V	None Established
☐	Water, Deionized ASTM Type II	CAS# 7732-18-5	Balance	V/V	None Established

SECTION 3 HAZARD IDENTIFICATION (SEE ALSO SECTION 11)

Generally not hazardous in normal handling, however good laboratory practices should always be used. Avoid long term exposure to skin or by inhalation.

SECTION 4 FIRST AID MEASURES

FIRST AID: SKIN: Wash exposed area with soap and water. If irritation persists, seek medical attention.

EYES: Wash eyes with plenty of water for at least 15 minutes, lifting lids occasionally. Seek Medical Aid. INHALATION: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen

INGESTION: Give several glasses of water. Vomiting may occur, but it is not necessary to induce.

SECTION 5 FIRE FIGHTING MEASURES

Fire Extinguisher Type: Any means suitable for extinguishing surrounding fire

Fire / Explosion Hazards: None

Fire Fighting Procedure:

Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and clothing.

SECTION 6 ACCIDENTAL RELEASE MEASURES

Absorb spill with inert material. Flush excess down drain.

SECTION 7 HANDLING AND STORAGE

Store in a cool dry place. This Material is not considered hazardous. Handle using safe laboratory practices.

SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

Respiratory Protection: None required

Ventilation: Local Exhaust ☑ Mechanical ☐

Protective Gloves: Gloves to prevent skin exposure as latex or vinyl

Eye Protection: Splash Goggles

Other Protective Equipment: Use safe laboratory handling procedures.

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Melting Point:	~0°C	Percent Volatile by Volume:	>98
Boiling Point:	~100°C	Evaporation Rate:	Information not available
Vapor Pressure:	Information not available	Evaporation Standard:	
Vapor Density:	Information not available		
Solubility in Water:	Soluble	Auto Ignition Temp:	Not applicable
Appearance /Odors:	Clear, blue, odorless liquid	Lower Flamm. Limit in Air:	Not applicable
Flash Point:	Not flammable		
Specific Gravity:	~1	Upper Flamm. Limit in Air:	Not applicable

SECTION 10 STABILITY AND REACTIVITY INFORMATION

Stability: Stable

Conditions to Avoid: High Temperature

Materials to Avoid: None known

Hazardous Decomposition Products: None known

Hazardous polymerization Will Not Occur

Conditions to Avoid: None known

SECTION 11 ADDITIONAL INFORMATION

Overexposure symptoms. Acute: Essentially non-hazardous. Possible irritation of eyes/stomach. Chronic: None known. Conditions aggravated/Target organs: none known.

DOT Classification: Not regulated

DOT Regulations may change from time to time. Please consult the most recent D.O.T. regulations.

Rev. No: 0.1 Date Entered: 8/1/2007 Approved: WPF Safety Dir.: WPF

The information herein is believed to be accurate and is offered in good faith for the user's consideration and investigation. No warranty either expressed or implied is made for the completeness or accuracy of the information whether originating from the above mentioned company or not. Users of this material should satisfy themselves by independent investigation of current scientific and medical knowledge that the material can be used safely.

WAXIE SANITARY SUPPLY

Material Safety Data Sheet
OSHA's Hazard Communication Standard
U.S. Department of Labor

29 CFR 1910.1200 OMB No. 1218-0072

Identity # 170300, 170302, 170304 Name #700 Disinfectant Cleaner, Quat-128

Section 1
Manufactured For - Waxie Sanitary Supply
Address - 9353 Waxie Way Emergency Phone (Chem-Tel) 1-(800) 255-3924
City - San Diego State - CA 92123-1036 Phone - (800) 995-4466

Date Prepared- 11/21/2007

Section 2 - Hazardous Ingredients/Identity Information

Components	CAS#	OSHA PEL	ACGIH TLV	Weight% (Optional)
Alkyl Dimethyl Benzyl Ammonium Chloride (C12-16)	68424-85-1	N/A	N/A	4.34%
Di-(C8-10)-Alkyl Dimethyl Ammonium Chloride	68424-95-3	N/A	N/A	3.254%
Dioctyldimethylammonium Chloride	5538-94-3	N/A	N/A	1.628%
Didecyl Dimethyl Ammonium Chloride	7173-51-5	N/A	N/A	1.628%
Ethanol	64-17-5	1000PPM	1000PPM	< 3%

Note:

VOC Content- 25.2 g/L

Section 3 - Physical/Chemical Characteristics

Boiling Point- >212 F
Vapor Pressure - Not Tested
Vapor Density - Not Tested
Solubility in Water- 100%
Appearance and Odor- BLUE COLOR LIQUID WITH PLEASANT ODOR.

pH- 6 TO 8
Specific Gravity- 1.011
Melting Point - Not Tested
Evaporation Rate - Not Tested

Section 4 - Fire and Explosion Hazard Data

Flash Point- 201F PMCC
Flammable Limits - Not Tested
LEL- Not Tested
UEL- Not Tested
DOT# NONE

NFPA Hazard Rating -	H	F	R	S	HMIS Hazard Rating -	H	F	R
(0- Least, 4- Extreme)	3	1	0	0		3	1	0

Extinguishing Media- CO2, DRY FOAM, WATER FOG
Special Fire Fighting Procedures- Vapor may form explosive mixture with air.

Unusual Fire and Explosion Hazards- Should wear full fire-fighting gear with NIOSH approved self contained breathing apparatus with full facepiece in the pressure demand or other positive pressure mode.

Section 5 - Reactivity Data

Stability - Stable
Conditions to Avoid - None

Incompatible Materials to Avoid- Anionic Materials, Strong Oxidizing agents
Hazardous Decomposition or Byproducts- CO, CO2
Hazardous Polymerization- NONE

Conditions to Avoid- Strong Oxidizers

Section 6 - Health Hazard Data

Routes of Entry
Inhalation? - Possible
Skin? - Possible
Ingestion? - Possible
Health Hazards - Same as Signs and Symptoms of Exposures
Note - All Chemicals in this Product are Found on the TSCA Inventory List.
OSHA Carcinogenicity? - None; NTP? - None; IARC Monographs? - None.

170300, 17302, 170304

Signs and symptons of over exposure:

Eyes - Burning Sensation

Skin- Slight Irritation and may cause corrosion to the skin.

Ingestion-May be harmful. Ingestion in large amount may cause gastrointestinal disturbances, irritation, nausea, diarrhea

Inhalation- Slight Irritation, headache, dizziness, nausea.

Medical Conditions Generally Aggravated by Exposure - Same as Signs and Symptoms of Over Exposure.

Emergency and First Aid Procedures

Eyes - Flush With Water For 15 Minutes. If Irritation Persists, Call Physician.

Skin- Wash Off With Soap & Water, If Irritation Persists, Call Physician

Ingestion- Drink Large Amounts of Water, Call Physician

Inhalation- Get Person to Fresh Air If Unconscious, Call Physician

Section 7 - Precautions For Safe Handling and Use.
Steps to be taken in case material is released or spilled - Contain with an inert, absorbent material. Collect in an approved container and place in a proper waste disposal container.

Waste Disposal Method - Dispose of in accordance with state and local regulations.
Precautions To Be Taken In Handling and Storing - Store in a cool, dry place, out of direct sunlight. Do not freeze, less than or equal to 32 F, or heat above 110 F.

Other Precautions - Keep out of reach of children. Follow directions on the container for proper use of this product.

Section 8 - Control Measures
Respiratory Protection - Open all doors and windows. "If" there is an exposure, and it is above the TLV or PEL, a NIOSH approved respirator equipped for the exposure or suitable respiratory protection per 29 CRF 1910.134, is required.
Ventilation Local Exhaust - If Available Special - None
Mechanical - If Available Other - None

Protective Gloves- Plastic or Rubber, Chemical Resistant

Eye Protection- Glasses, Goggles

Other Protective Clothing or Equipment- NONE

Hygienic Work Practices - Use common sense and care around chemicals. Never mix chemicals. Consult your supervisor for other practices. All practices depend on your specific business. Directions for use are normally found on label which will dictate engineering and control measures.

Other Special Requirements NONE

Note: Waxie believes the data set forth are accurate, Waxie makes no warranty with respects thereto and disclaims all liability for reliance thereon. Such data are offered solely for consideration, investigation and verification. Also, the data set forth is for the concentrated finished product. All lab samples are for experimental purposes only and used at the customers discretion

ISO9001 - 2000 certified

MSDS7386

Material Safety Data Sheet

Nitric acid solutions, 0.4% - 50% v/v, 0.1N-6.0N

Section 1 - Chemical Product and Company Identification

MSDS Name:

Nitric acid solutions, , 0.4% - 50% v/v, 0.1N-6.0N

Catalog Numbers:

LC17730, LC17750, LC17770, LC17800, LC17840, LC17850, LC17870

Synonyms:

Company Identification:

LabChem, Inc.
200 William Pitt Way
Pittsburgh, PA 15238

Company Phone Number:

(412) 826-5230

Emergency Phone Number:

(800) 424-9300

CHEMTREC Phone Number:

(800) 424-9300

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name:	Percent
7732-18-5	Water	balance
7697-37-2	Nitric acid	0.04-50

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Appearance: colorless
Caution! Corrosive. May cause severe eye and skin irritation with possible burns.
Target Organs: none known.

Potential Health Effects

Eye:

Causes pain, photophobia, tearing, edema, corneal ulceration, severe burns, necrosis of deep tissue with permanent damage and blindness.

Skin:

Skin may turn brown-yellow. Deep burns are slow to heal and scarring may occur.

Ingestion:

Causes pain in mouth, throat, stomach followed by vomiting, bloody diarrhea, hypotension, oliguria, anuria, possible fatal circulatory collapse, asphyxia from glottal edema. Perforation burns of gastrointestinal tract possible with fever and peritonitis.

Inhalation:

May cause coughing, dizziness, weakness, dry throat/chest, chest pain, dyspnea, frothy sputum, hypotension, cyanosis, pneumonitis, fatal pulmonary edema.

Chronic:

Dermatitis, conjunctivitis, dental erosion, jaw necrosis, chronic cough, bronchitis, chemical pneumonitis, gastrointestinal disturbances.

Section 4 - First Aid Measures

Eyes:

Flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower lids until no evidence of chemical remains. Get medical aid at once. Cover burns with loose sterile non-medicated bandages.

Skin:

Get medical aid. Immediately flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes. Remove contaminated clothing and shoes. Cover burns with a dry sterile bandage (secure, not tight).

Ingestion:

Get medical aid at once. Give 1 ounce of milk of magnesia. Give conscious victim large quantities of water to dilute acid. Give oxygen if respiration is depressed.

Inhalation:

Give artificial respiration if necessary. Get medical aid. Keep victim warm, at rest. Move victim to fresh air. Maintain airway and blood pressure.

Notes to Physician:

Treat symptomatically and supportively.

Section 5 - Fire Fighting Measures

General Information:

Move container if possible, cool with flooding amounts of water. Avoid breathing corrosive vapors. Negligible fire and explosion hazard for solutions <5%. At higher concentration, increased flammability of combustibles, readily oxidizable materials. Severe explosion hazard by reaction with incombatibles (metallic powders, carbides, hydrogen sulfide, turpentine). In or near fire material emits toxic and reactive nitrogen oxides of gases.

Extinguishing Media:

For small fires, use dry chemical, carbon dioxide, water spray or alcohol-resistant foam.

Autoignition Temperature:

No information found.

Flash Point:

No information found.

NFPA Rating:

CAS# 7732-18-5: Not published.
CAS# 7697-37-2: Not published.

Explosion Limits:

Lower: Upper:

Section 6 - Accidental Release Measures

General Information:

Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks:

Absorb spill with an alkaline material such as soda ash or lime. Keep out of sewers/drains. Ventilate and wear protective clothing. Scoop material into suitable (plastic or glass) container, label for disposal as "corrosive".

Section 7 - Handling and Storage

Handling:

Avoid breathing dust, vapor, mist, or gas. Avoid contact with eyes, skin, and clothing. Handle as corrosive liquid, dispense with care; wear protective clothing, gloves, goggles.

Storage:

Store at room temperature. Store in glass or approved plastic containers only, keep capped. Protect from heat and incompatibles.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls:

Local exhaust ventilation may be necessary to control any air contaminants to within their TLVs during the use of this product.

Exposure Limits

Chemical Name:	ACGIH	NIOSH	OSHA
Water	None of the components are on this list.	None of the components are on this list.	None of the components are on this list.
Nitric acid	2 ppm TWA;4 ppm STEL	2 ppm TWA; 5 mg/m3 TWA	2 ppm TWA; 5 mg/m3 TWA;

OSHA Vacated PELs

Nitric acid: 2 ppm TWA; 5 mg/m3 TWA

Personal Protective Equipment

Eyes:

Do not wear contact lenses when working with chemicals. An eye wash fountain should be available in the immediate work area. Wear gloves, splash proof goggles and faceshield.

Skin:

Wear impervious gloves (viton, saranex).

Clothing:

Wear appropriate protective clothing to prevent skin exposure.

Respirators:

PEL to 50ppm - SAF/SCBAF/SAF:PD,PP,CF.
>50ppm - SCBAF:PD,PP.
Firefighting - SCBAF:PD,PP. NOTE: DO NOT USE OXIDIZABLE SORBENTS IN RESPIRATORS.

Section 9 - Physical and Chemical Properties

Physical State:	Clear liquid
Color:	colorless
Odor:	acrid odor
pH:	acidic
Vapor Pressure:	14 mm Hg @25c
Vapor Density:	0.7 - 2.2
Evaporation Rate:	>1 (ether=1)
Viscosity:	No information found.
Boiling Point:	181-212°F
Freezing/Melting Point:	-44 - 32°F
Decomposition Temperature:	No information found.
Solubility in water:	Soluble.
Specific Gravity/Density:	1.0-1.5
Molecular Formula:	No information found.
Molecular Weight	No information found.

Section 10 - Stability and Reactivity

Chemical Stability:

Stable under normal temperatures and pressures up to boiling point. Nitric oxides quietly evolved - sunlight catalyses oxide formation (yellow color, aging).

Conditions to Avoid:

Incompatible materials, easily oxidized materials.

Incompatibilities with Other Materials

Metals, acetic acid, acetic anhydride, acrylonitrile, alcohols, anhydrides, fluorine, organic acids, perchlorates, aldehydes (e.g. acetaldehyde, acrolein, chloral hydrate, formaldehyde), ketones (e.g. acetone, acetophenone, MEK, MIBK), metals as powders (e.g. hafnium, raney nickel), organics, carbides, acetonitrile, acetone, arsine, phosphides, dioxides, thiocyanates, inorganic acids, chlorine, cyclic compounds, halides.

Hazardous Decomposition Products

Nitrogen oxides, nitric acid vapors, catalyzed by sunlight.

Hazardous Polymerization

Has not been reported

Section 11 - Toxicological Information

RTECS:

CAS# 7732-18-5: ZC0110000.
CAS# 7697-37-2: QU5775000; QU5900000.

LD50/LC50:

CAS# 7732-18-5:
Oral, rat: LD50 = >90 mL/kg.
CAS# 7697-37-2:
Inhalation, rat: LC50 =67 ppm(NO2)/4H.

Carcinogenicity:

CAS# 7732-18-5: Not listed as a carcinogen by ACGIH, IARC, NIOSH, NTP, OSHA, or CA Prop 65.
CAS# 7697-37-2: Not listed as a carcinogen by ACGIH, IARC, NIOSH, NTP, OSHA, or CA Prop 65.

Epidemiology:

Severe eye, mucous membrane, and skin irritant.

Teratogenicity:

Reproductive:

Mutagenicity

Neurotoxicity

Section 12 - Ecological Information

No information found.

Section 13 - Disposal Considerations

Dispose of in accordance with federal, state, and local regulations.

Section 14 - Transport Information

US DOT

	Nitric acid >2% W/W	Nitric acid <2% W/W
Shipping Name:	Nitric acid, Not more than 70%	Not regulated.
Hazard Class:	8	
UN Number:	UN2031	
Packing Group:	PG II	

Section 15 - Regulatory Information

US Federal

TSCA

CAS# 7732-18-5 is listed on the TSCA Inventory.
CAS# 7697-37-2 is listed on the TSCA Inventory.

SARA Reportable Quantities (RQ)

CAS# 7697-37-2: final RQ = 1000 pounds (454 kg)

CERCLA/SARA Section 313

This material contains Nitric acid (CAS# 7697-37-2, 0.04-50%),which is subject to the reporting requirements of Section 313 of SARA Title III and 40 CFR Part 373.

OSHA - Highly Hazardous

CAS# 7697-37-2 is considered highly hazardous by OSHA.

US State

State Right to Know

Nitric acid can be found on the following state Right-to-Know lists: California, New Jersey, Florida, Pennsylvania, Minnesota, Massachusetts.

California Regulations

European/International Regulations

Canadian DSL/NDSL

CAS# 7732-18-5 is listed on Canada's DSL List.
CAS# 7697-37-2 is listed on Canada's DSL List.

Canada Ingredient Disclosure List

CAS# 7732-18-5 is not listed on Canada's Ingredient Disclosure List.
CAS# 7697-37-2 is listed on Canada's Ingredient Disclosure List.

Section 16 - Other Information

MSDS Creation Date: November 17, 1997
Revision Date: August 12, 2004

Information in this MSDS is from available published sources and is believed to be accurate. No warranty, express or implied, is made and LabChem Inc. assumes no liability resulting from the use of this MSDS. The user must determine suitability of this information for his application.

ISO9001 - 2000 certified

MSDS7459

Material Safety Data Sheet

Sulfuric Acid Solutions 0.03-93%, Sulfuric Acid Solutions 0.01N - 12N

Section 1 - Chemical Product and Company Identification

MSDS Name:

Sulfuric Acid Solutions 0.03-93%, Sulfuric Acid Solutions 0.01N - 12N

Catalog Numbers:

LC25570, LC25580, LC25590, LC25600, LC25605, LC25607, LC25610, LC25620, LC25640, LC25645, LC25650, LC25655, LC25660, LC25670, LC25675, LC25680, LC25685, LC25700, LC25720, LC25730, LC25740, LC25770, LC25790, LC25800, LC25810, LC25830, LC25840, LC25850, LC25870, LC25880

Synonyms:

Company Identification:

LabChem, Inc.
200 William Pitt Way
Pittsburgh, PA 15238

Company Phone Number:

(412) 826-5230

Emergency Phone Number:

(800) 424-9300

CHEMTREC Phone Number:

(800) 424-9300

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name:	Percent
7732-18-5	Water	balance
7664-93-9	Sulfuric acid	0.03-93

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Appearance: colorless
Danger! Corrosive. Causes eye and skin burns. Causes digestive and respiratory tract burns.
Target Organs: None.

Potential Health Effects

Eye:

Causes severe eye burns. Eye contact can result in blindness; exposure to mist leads to watering, irritation.

Skin:

Skin contact may result in severe burns, blistering, pain.

Ingestion:

May cause severe and permanent damage to the digestive tract. Causes gastrointestinal tract burns. Vomiting and diarrhea of dark blood may occur; asphyxia from throat swelling. Stomach and esophagus may become perforated.

Inhalation:

May cause severe irritation of the respiratory tract with sore throat, coughing, shortness of breath and delayed lung edema. Causes chemical burns to the respiratory tract. At 5mg/M3 concentrations, nose and throat irritation occurs, with headache, cough, increased respiratory rate, impairment of lung to ventilate.

Chronic:

Delayed symptoms include tight chest, fluid in lungs, cyanosis (blue color), hypotension, bronchitis or emphysema. tracheobronchitis, dental erosion/discoloration, pneumonia, gastrointestinal disturbances may occur. Skin irritation/dermatitis, conjunctivitis and lacrimation of the eye can occur.

Section 4 - First Aid Measures

Eyes:

Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower lids until chemical is gone. Get medical aid at once.

Skin:

Get medical aid at once. Immediately flush skin with plenty of soap and water for at least 15 minutes while removing contaminated clothing and shoes.

Ingestion:

Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Get medical aid at once. Give one ounce (30ml) of milk of magnesia. Give conscious victim large quantities of water to dilute acid.

Inhalation:

Get medical aid at once. Move victim to fresh air immediately. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

Notes to Physician:

Treat symptomatically and supportively.

Section 5 - Fire Fighting Measures

General Information:

As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. Contact with metals may evolve flammable hydrogen gas. Avoid breathing toxic and corrosive vapors - keep upwind.

Extinguishing Media:

Use extinguishing media most appropriate for the surrounding fire.

Autoignition Temperature:

No information found.

Flash Point:

No information found.

NFPA Rating:

CAS# 7732-18-5: Not published.
CAS# 7664-93-9: health - 3; flammability - 0; reactivity - 2.

Explosion Limits:

Lower: Upper:

Section 6 - Accidental Release Measures

General Information:

Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks:

May be neutralized with slaked lime, limestone, or sodium bicarbonate to a pH 7. Place in labeled plastic containers for disposal, wash area down with water.

Section 7 - Handling and Storage

Handling:

Wash thoroughly after handling. Do not get in eyes, on skin, or on clothing. Do not ingest or inhale. Use with adequate ventilation.

Storage:

Do not store near alkaline substances. Store in labeled non-reactive containers (glass, plastic) protected from heat and incompatible substances.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls:

Use adequate general or local exhaust ventilation to keep airborne concentrations below the permissible exposure limits.

Exposure Limits

Chemical Name:	ACGIH	NIOSH	OSHA
Water	None of the components are on this list.	None of the components are on this list.	None of the components are on this list.
Sulfuric acid	1 mg/m3 TWA;3 mg/m3 STEL	1 mg/m3 TWA	1 mg/m3 TWA;

OSHA Vacated PELs

Sulfuric acid: 1 mg/m3 TWA

Personal Protective Equipment

Eyes:

Wear appropriate protective eyeglasses or chemical safety goggles as described by OSHA's eye and face protection regulations in 29 CFR 1910.133. Provide an eye-wash fountain in the immediate work area. Do not wear contact lenses when working with chemicals.

Skin:

Wear appropriate protective gloves to prevent skin exposure.

Clothing:

Wear acid protective clothing and gloves.

Respirators:

50mg/M3 -GMAGHiEP/HiEPF/SAF/SCBAF.
100mg/M3 - SAF:PD,PP,CF.
Escape - GMAGHiEP/SCBA. Firefighting - SCBAF:PD,PP.
(Respirator Codes: (NIOSH) PUblication No. 78-210)

Material Safety Data Sheet

Sulfuric Acid Solutions 0.03-93%, Sulfuric Acid Solutions 0.01N - 12N

Section 9 - Physical and Chemical Properties

Physical State: Liquid
Color: colorless
Odor: odorless
pH: acidic
Vapor Pressure: No information found.
Vapor Density: No information found.
Evaporation Rate: <ether
Viscosity: No information found.
Boiling Point: No information found.
Freezing/Melting Point: No information found.
Decomposition Temperature: No information found.
Solubility in water: Soluble.
Specific Gravity/Density: 1.0 - 1.7
Molecular Formula: No information found.
Molecular Weight No information found.

Section 10 - Stability and Reactivity

Chemical Stability:

Stable under normal temperatures and pressures.

Conditions to Avoid:

Incompatible materials, excess heat, combustible materials, organic materials, oxidizers, amines, bases.

Incompatibilities with Other Materials

Explosive or violent reactions with acetone cyanohydrin, acetone and nitric acid or potassium dichromate, acrylonitrile, alcohols, hydrogen peroxide, allyl chloride, bromates and metals, bromine pentafluoride, carbides, all chlorates, chlorine trifluoride, cuprous nitride, ethylene cyanohydrin, fulminates, indane and nitric acid, iron, mercuric nitride, nitric acid and glycerides, p-nitrotoluene, pentasilver trihydroxydiaminophosphate, perchlorates, phosphorus isocyanate picrates, sliver permanganate, sodium, sodium carbonate, toluene and nitric acid. Dangerous temperatures and pressures occur with other substances, especially organic combinations. Explosive hydrogen gas is evolved from contact with steel, other metals.

Hazardous Decomposition Products

Oxides of sulfur, hydrogen gas.

Hazardous Polymerization

Has not been reported.

Section 11 - Toxicological Information

RTECS:

CAS# 7732-18-5: ZC0110000.
CAS# 7664-93-9: WS5600000.

LD50/LC50:

CAS# 7732-18-5:
Oral, rat: LD50 = >90 mL/kg.
CAS# 7664-93-9:
Inhalation, mouse: LC50 =320 mg/m3/2H
Inhalation, rat: LC50 =510 mg/m3/2H
Oral, rat: LD50 = 2140 mg/kg.

Carcinogenicity:

CAS# 7732-18-5: Not listed as a carcinogen by ACGIH, IARC, NIOSH, NTP, OSHA, or CA Prop 65.
CAS# 7664-93-9
ACGIH: A2 - Suspected Human Carcinogen (contained in strong inorganic acid mists)
California: Not listed.
NIOSH: Not listed.
NTP: Not listed.
OSHA: Select carcinogen
IARC: Group 1 carcinogen

Epidemiology:

Teratogenicity:

Reproductive:

Mutagenicity

Neurotoxicity

Section 12 - Ecological Information

No information found.

Section 13 - Disposal Considerations

Dispose of in accordance with federal, state, and local regulations.

Section 14 - Transport Information

US DOT

	> 51% W/W	0.5% to 51% W/W	< 0.5% W/W
Shipping Name:	Sulfuric acid	Sulfuric Acid	Not regulated.
Hazard Class:	8	8	
UN Number:	UN1830	UN2796	
Packing Group:	PG II	PG II	

Section 15 - Regulatory Information

US Federal

TSCA

CAS# 7732-18-5 is listed on the TSCA Inventory.
CAS# 7664-93-9 is listed on the TSCA Inventory.

SARA Reportable Quantities (RQ)

CAS# 7664-93-9: final RQ = 1000 pounds (454 kg)

CERCLA/SARA Section 313

This material contains Sulfuric acid (CAS# 7664-93-9, 0.03-93%),which is subject to the reporting requirements of Section 313 of SARA Title III and 40 CFR Part 373.

OSHA - Highly Hazardous

None of the components are on this list.

US State

State Right to Know

Sulfuric acid can be found on the following state Right-to-Know lists: California, New Jersey, Florida, Pennsylvania, Minnesota, Massachusetts.

California Regulations

European/International Regulations

Canadian DSL/NDSL

CAS# 7732-18-5 is listed on Canada's DSL List.
CAS# 7664-93-9 is listed on Canada's DSL List.

Canada Ingredient Disclosure List

CAS# 7732-18-5 is not listed on Canada's Ingredient Disclosure List.
CAS# 7664-93-9 is listed on Canada's Ingredient Disclosure List.

Section 16 - Other Information

MSDS Creation Date: November 9, 1997
Revision Date: November 11, 2004

Information in this MSDS is from available published sources and is believed to be accurate. No warranty, express or implied, is made and LabChem Inc. assumes no liability resulting from the use of this MSDS. The user must determine suitability of this information for his application.

In-Situ Inc. Innovations in Water Monitoring

Material Safety and Data Sheet

I. Chemical Product and Company Identification

Product Name:	Manufacturer:	Emergency Contact:
Sodium Sulfite **2 Molar Standard Solution**	Aqua Phoenix Scientific, Inc. 320 Maple Ave. Hanover, PA 17331 Telephone: 866 632 1291 Fax: 717 633 1285	INFOTRAC Emergency Response Hotline: 1-800-535-5053 (in the U.S. and Canada) 1-352-323-3500 www.infotrac.net

II. Composition, Information on Ingredients

Hazardous Components Specific Chemical Identity: Common Names	CAS NO.	%	OSHA PEL	ACGIH TLV
Sodium Sulfite	7757-83-7	5% w/v	Not listed	Not Established
Cobalt Chloride Hexahydrate	7791-13-1	<0.01% w/v	Not listed	0.1 mg/m^3

III. Hazard Identification

Emergency Overview: Non-flammable, non-corrosive. Does not present significant health hazards. Wash areas of contact with water.

Target Organs: Eyes, Skin

Eyes	May cause slight irritation
Skin	May cause slight irritation
Ingestion	Large doses may cause upset stomach
Inhalation	Not likely to be a hazard
Chronic Effect /Carcinogenicity	None (IARC, NTP, OSHA)

IV. First Aid

Eyes	Immediately flush eyes with water for at least 15 minutes. Immediately get medical assistance.
Skin	Flush with water for 15 minutes. Get medical assistance if irritation develops.
Ingestion	DO NOT induce vomiting. Dilute with water or milk. Get medical assistance.
Inhalation	Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

V. Fire Fighting Measures

Flash Point	N/A
Extinguishing Media	Use means suitable to extinguishing surrounding fire.
Fire and Explosion Hazards	Not considered to be a fire or explosion hazard.
Fire Fighting Instructions/Equipment	Use normal procedures. Poisonous gases may be produced in fire. Use protective clothing. Use NIOSH-approved breathing equipment.
NFPA Rating	(estimated) Health: 1, Flammable: 0, Reactivity: 0

VI. Accidental Release Measures

Absorb with suitable material. Always obey local regulations.

VII. Handling and Storage

Handling	Wash hands after handling. Avoid contact with skin and eyes.
Storage	Protect from freezing and physical damage.

VIII. Exposure Controls, Personal Protection

Engineering Controls	Normal ventilation is adequate
Respiratory Controls	Normal ventilation is adequate
Skin Protection	Chemical resistant gloves
Eye Protection	Safety Glasses or goggles

IX. Physical and Chemical Properties

Appearance	Clear, colorless liquid	**Odor**	Odorless
pH @ 25°C	N/A	**Solubility in Water**	Infinite
Boiling Point	Approx 100.1°C	**Specific Gravity**	1.00-1.01
Melting point	Approx (-6)-0°C	**Vapor Pressure**	N/A

X. Stability and Reactivity

Chemical Stability	Stable under normal conditions of use and storage
Incompatibility	Strong Oxidizing agents, Lithium, Bromine, Trifluoride
Hazardous Decomposition Products	Oxides of Sodium and fumes of Chloride
Hazardous Polymerization	Does not occur

XI. Toxicological Information

LD50 orl-rat	N/A
LC50 inhalation-rat	N/A

XII. Ecological Information

Ecotoxicity	N/A

XIII. Disposal Considerations

Dilute with water.
All chemical waster generators must determine whether a discarded chemical is classified as hazardous waste. Comply with all local, state, and federal regulations.

XIV. Transport Information

DOT	Not Regulated

XV. Regulatory Information (not meant to be all inclusive)

OSHA Status	These chemicals are not considered hazardous by OSHA.
TSCA	The components of this solution are listed on the TSCA Inventory.
SARA Title III Section 313	N/A
RCRA Status	N/A
CERCLA Reportable Quality	N/A
WHMIS	N/A

XVI. Additional Information

Issue Date: 9/28/07
Revision Date: 3/8/10, Rev. 004
Document: 0017690

* N/A – Not Applicable/Not Available

Disclaimer: The information, data and recommendations contained herein were provided to In-Situ Inc. by the manufacturer named on this Material Safety Data Sheet. In-Situ Inc. makes no warranty of any kind whatsoever with respect thereto and disclaims all liability from reliance thereon. In-Situ Inc. reserves the right to revise this Material Safety Data Sheet as new information is provided to it by the manufacturer.

In-Situ Inc.

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221 East Lincoln Avenue, Fort Collins, CO 80524
1-800-446-7488 (toll-free in U.S. & Canada
1-970-498-1500 (international & domestic)
www.in-situ.com

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

MSDS No: M00002

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: Sodium Sulfite, Anhydrous
Catalog Number: 19501H

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M00002
Chemical Name: Sulfurous acid, disodium salt
CAS No.: 7757-83-7
Chemical Formula: Na_2SO_3
Chemical Family: Inorganic Salt
Hazard: May cause allergic reaction. May cause irritation.
Date of MSDS Preparation:
Day: 03
Month: October
Year: 2013

2. COMPOSITION / INFORMATION ON INGREDIENTS

Sodium Sulfate, ACS, Anhyd
CAS No.: 7757-83-7
TSCA CAS Number: 7757-83-7
Percent Range: 100.0
Percent Range Units: weight / weight
LD50: Oral mouse LD50 = 820 mg/kg
LC50: LC50 Inhalation - rat - 4h - >5,500 mg/m3
TLV: 5 mg/m³
PEL: Not established
Hazard: May cause allergic reaction. May cause irritation.

3. HAZARDS IDENTIFICATION

Emergency Overview:
Appearance: White crystals
Odor: None
MAY CAUSE EYE, SKIN AND RESPIRATORY TRACT IRRITATION
MAY CAUSE ALLERGIC RESPIRATORY REACTION IF SWALLOWED OR INHALED
HMIS:
Health: 1
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment, Section 8.
NFPA:
Health: 1
Flammability: 0
Reactivity: 0
Symbol: Not applicable

Potential Health Effects:
Eye Contact: May cause irritation
Skin Contact: May cause irritation
Skin Absorption: May be harmful if absorbed through the skin
Target Organs: None reported
Ingestion: May cause: abdominal pain diarrhea colic circulatory disturbances central nervous system depression allergic respiratory reaction
Target Organs: Central nervous system
Inhalation: May cause: respiratory tract irritation allergic respiratory reaction
Target Organs: None reported
Medical Conditions Aggravated: Sulfites are strong sensitizers. Inhalation and ingestion may cause allergic respiratory reactions in asthmatics. Persons with respiratory conditions should take special care when working with products that contain sulfites.
Chronic Effects: None reported
Cancer / Reproductive Toxicity Information:
O.S.H.A. Listed: No
IARC Group 3: Non-classifiable
Sulfites
NTP Listed: No
Additional Cancer / Reproductive Toxicity Information: an experimental mutagen.
Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.
Skin Contact (First Aid): Wash skin with soap and plenty of water. Call physician if irritation develops.
Ingestion (First Aid): Give large quantities of water. Call physician immediately.
Inhalation: Remove to fresh air. Give artificial respiration if necessary. Call physician.

5. FIRE FIGHTING MEASURES

Flammable Properties: During a fire, corrosive and toxic gases may be generated by thermal decomposition.
Flash Point: Not applicable
Method: Not applicable
Flammability Limits:
Lower Explosion Limits: Not applicable
Upper Explosion Limits: Not applicable
Autoignition Temperature: Not applicable
Hazardous Combustion Products: Toxic fumes of: sulfur oxides. sodium monoxide
Fire / Explosion Hazards: None reported
Static Discharge: None reported.
Mechanical Impact: None reported
Extinguishing Media: Use media appropriate to surrounding fire conditions
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13, Special Instructions for disposal assistance. Outside of the US, only persons properly qualified according to state or local regulations should respond to a spill involving chemicals.
Containment Technique: Stop spilled material from being released to the environment.
Clean-up Technique: If permitted by regulation, Sweep up material. Dilute with a large excess of water. Adjust to a pH between 6 and 9 with an acid, such as sulfuric or citric. Flush reacted material to the drain with a large excess of water. Decontaminate the area of the spill with a soap solution. Otherwise, Pick up spill for disposal and place in a closed container Dispose of in accordance with local, state and federal regulations or laws.
Evacuation Procedure: Evacuate local area (15 foot radius or as directed by your facility's emergency response plan) when: a pound or more of loose powder is spilled. If conditions warrant, increase the size of the evacuation.
Special Instructions (for accidental release): Not applicable

304 EHS RQ (40 CFR 355): Not applicable
D.O.T. Emergency Response Guide Number: None

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin Do not breathe dust. Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Keep away from: oxidizers acids Store at 10 - 30°C.
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves In the EU, the selected gloves must satisfy the specifications of EU Directive 89/686/EEC and standard EN 374 derived from it. lab coat
Inhalation Protection: adequate ventilation
Precautionary Measures: Keep away from: acids/acid fumes oxidizers
TLV: 5 mg/m³
PEL: Not established

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: White crystals
Physical State: Solid
Molecular Weight: 126.04
Odor: None
pH: 1% solution = 9.8
Vapor Pressure: Not applicable
Vapor Density (air = 1): Not applicable
Boiling Point: Not applicable
Melting Point: Decomposes @ 600°C; 1112°F
Specific Gravity/ Relative Density (water = 1; air =1): 2.6
Evaporation Rate (water = 1): Not applicable
Volatile Organic Compounds Content: Not applicable
Partition Coefficient (n-octanol / water): Not available
Solubility:
Water: 13.9 g/ 100 g H_2O @ 0°C (32°F)
Acid: Not determined
Other: Soluble in glycerol; Insoluble in alcohol
Metal Corrosivity:
Steel: Not determined
Aluminum: Not determined

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Heating to decomposition.
Reactivity / Incompatibility: Incompatible with: acids oxidizers
Hazardous Decomposition: Heating to decomposition releases toxic and/or corrosive fumes of: sulfur oxides sodium monoxide
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: Oral mouse LD50 = 820 mg/kg
LC50: LC50 Inhalation - rat - 4h - >5,500 mg/m3
Dermal Toxicity Data: None reported
Skin and Eye Irritation Data: None reported
Mutation Data: Cytogenetic analysis, sperm morphology - mouse -cells - 25 mg/l; Mutation - human lymphocytes - 100 µmol/l
Reproductive Effects Data: None reported
Ingredient Toxicological Data: --
Not applicable

12. ECOLOGICAL INFORMATION

Product Ecological Information: Biological Oxygen Demand (BOD): 0.12 lb/lb; 2600 ppm/24.48 & 96 hr./ mosquito fish/TLm/fresh water
Ingredient Ecological Information: --
Not applicable

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: None
Special Instructions (Disposal): Work in an approved fume hood. Working in a large container, cautiously add small portions of the material to cold water with agitation. Adjust to a pH between 6 and 9 with an acid, such as sulfuric or citric. Open cold water tap completely, slowly pour the reacted material to the drain. Allow cold water to run for 5 minutes to completely flush the system.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--
DOT Hazard Class: NA
DOT Subsidiary Risk: NA
DOT ID Number: NA
DOT Packing Group: NA
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
ICAO Hazard Class: NA
ICAO Subsidiary Risk: NA
ICAO ID Number: NA
ICAO Packing Group: NA
I.M.O.:
I.M.O. Proper Shipping Name: Not Currently Regulated
--
I.M.O. Hazard Class: NA
I.M.O. Subsidiary Risk: NA
I.M.O. ID Number: NA
I.M.O. Packing Group: NA
Additional Information: There is a possibility that this product could be contained in a reagent set or kit composed of various compatible dangerous goods. If the item is NOT in a set or kit, the classification given above applies. If the item IS part of a set or kit, the classification would change to the following: UN3316 Chemical Kit, Class 9, PG II or III. If the item is not regulated, the Chemical Kit classification does not apply.

15. REGULATORY INFORMATION

U.S. Federal Regulations:
O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)
E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard
S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.
--
302 (EHS) TPQ (40 CFR 355): Not applicable
304 CERCLA RQ (40 CFR 302.4): Not applicable
304 EHS RQ (40 CFR 355): Not applicable
Clean Water Act (40 CFR 116.4): Not applicable
RCRA: Contains no RCRA regulated substances.
C.P.S.C.: Not applicable
State Regulations:
California Prop. 65: No Prop. 65 listed chemicals are present in this product.
Identification of Prop. 65 Ingredient(s): None
California Perchlorate Rule CCR Title 22 Chap 33: Not applicable
Trade Secret Registry: Not applicable
National Inventories:
U.S. Inventory Status: TSCA Listed: Yes
TSCA CAS Number: 7757-83-7

16. OTHER INFORMATION

Intended Use: Laboratory Use
References: 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). Air Contaminants, Federal Register, Vol. 54, No. 12. Thursday, January 19, 1989. pp. 2332-2983. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists, 1992. Sax, N. Irving. Dangerous Properties of Industrial Materials, 7th Ed. New York: Van Nostrand Reinhold Co., 1989. The Merck Index, 11th Ed. Rahway, New Jersey: Merck and Co., Inc., 1989. Gosselin, R. E. et al. Clinical Toxicology of Commercial Products, 5th Ed. Baltimore: The Williams and Wilkins Co., 1984. Vendor Information. Technical Judgment.
Revision Summary: Substantial revision to comply with EU Reg 1272/2008, Reg 1907/2006 and UN GHS (ST/SG/AC.10/36/Add.3).

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

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(970) 669-3050

Date Printed 4/21/11
MSDS No: M02701

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: <0.1 NTU Calibration Solution
Catalog Number: 2684701

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M02701
Chemical Name: Not Applicable
CAS No.: Not Applicable
Chemical Formula: Not Applicable
Chemical Family: Mixture
Hazard: May cause eye irritation.
Date of MSDS Preparation:
Day: 21
Month: August
Year: 2009

2. COMPOSITION / INFORMATION ON INGREDIENTS

Methyl Alcohol
CAS No.: 67-56-1
TSCA CAS Number: 67-56-1
Percent Range: < 0.1
Percent Range Units: weight / weight
LD50: Oral Human LDLo = 143 mg/kg: Oral rat LD50 = 5628 mg/kg
LC50: Inhalation rat LC50 = 64000 ppm/4H: Inhalation mouse LCLo = 50 g/m^3/2H
TLV: 200 ppm
PEL: 200 ppm
Hazard: Toxic. Cannot be made non-toxic. Flammable. Causes irritation.

Ethanol
CAS No.: 64-17-5
TSCA CAS Number: 64-17-5
Percent Range: < 0.5
Percent Range Units: weight / weight
LD50: Oral rat LD50 = 7060 mg/kg; Oral mouse LD50 = 3450 mg/kg; Oral human LDLo: 1400 mg/kg; Oral child LDLo: 2000 mg/kg
LC50: Inhhalation rat LC50 = 20000 ppm/10H; Inhalation mouse LC50 = 39 g/m^3/4H
TLV: 1000 ppm
PEL: 1000 ppm
Hazard: Flammable. May cause irritation.

Demineralized Water
CAS No.: 7732-18-5
TSCA CAS Number: 7732-18-5
Percent Range: >98
Percent Range Units: weight / weight
LD50: None reported
LC50: None reported
TLV: Not established
PEL: Not established
Hazard: No effects anticipated.

3. HAZARDS IDENTIFICATION

Emergency Overview:
Appearance: Clear, colorless liquid
Odor: Alcoholic
MAY CAUSE EYE IRRITATION

HMIS:
Health: 1
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment, Section 8.
NFPA:
Health: 1
Flammability: 0
Reactivity: 0
Symbol: Not applicable
Potential Health Effects:
Eye Contact: May cause mild irritation
Skin Contact: No effects are anticipated
Skin Absorption: No effects anticipated
Target Organs:
Ingestion: No Effects Anticipated
Target Organs:
Inhalation: No effects anticipated
Target Organs:
Medical Conditions Aggravated: Pre-existing: Eye conditions Skin conditions
Chronic Effects: Methanol is a cumulative poison. Methanol may cause visual impairment and possibly blindness by repeated or prolonged exposure.
Cancer / Reproductive Toxicity Information:
O.S.H.A. Listed: No

IARC Listed: No

NTP Listed: No

Additional Cancer / Reproductive Toxicity Information: Contains: an experimental teratogen. an experimental mutagen.
Toxicologically Synergistic Products: None reported

4. FIRST AID

Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.
Skin Contact (First Aid): Remove contaminated clothing. Call physician if irritation develops. Wash skin with plenty of water.
Ingestion (First Aid): If you feel unwell, contact a physician.
Inhalation: Remove to fresh air.

5. FIRE FIGHTING MEASURES

Flammable Properties: Material will not burn.
Flash Point: Not Applicable
Method: Not applicable
Flammability Limits:

Lower Explosion Limits: Not Applicable
Upper Explosion Limits: Not Applicable
Autoignition Temperature: Not Applicable
Hazardous Combustion Products: None
Fire / Explosion Hazards: None reported
Static Discharge: None reported.
Mechanical Impact: None reported
Extinguishing Media: Use media appropriate to surrounding fire conditions
Fire Fighting Instruction: Not applicable

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13. Special Instructions for disposal assistance.
Containment Technique: Absorb spilled liquid with non-reactive sorbent material.
Clean-up Technique: Absorb spilled liquid with non-reactive sorbent material. Decontaminate the area of the spill with a soap solution.
Evacuation Procedure: Evacuate as needed to perform spill clean-up.
Special Instructions (for accidental release): Mixture contains a component which is regulated in the US as a hazardous air pollutant.
304 EHS RQ (40 CFR 355): Not applicable
D.O.T. Emergency Response Guide Number: None

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin clothing Do not breathe mist or vapors. Wash thoroughly after handling. Use with adequate ventilation. Maintain general industrial hygiene practices when using this product.
Storage: Not applicable
Flammability Class: Class IIIA

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes skin clothing Do not breathe: mist/vapor Wash thoroughly after handling. Use with adequate ventilation.
TLV: Not established
PEL: Not established

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: Clear, colorless liquid
Physical State: Liquid
Molecular Weight: Not Applicable
Odor: Alcoholic
pH: Not Determined
Vapor Pressure: Not Determined
Vapor Density (air = 1): Not Determined
Boiling Point: Not Determined
Melting Point: Not Determined
Specific Gravity/ Relative Density (water = 1; air =1): Not Determined
Evaporation Rate (water = 1): Not Determined

Volatile Organic Compounds Content: Alcohols <1%
Partition Coefficient (n-octanol / water): Not Determined
Solubility:
Water: Soluble
Acid: Not Determined
Other: Not Applicable
Metal Corrosivity:
Steel: Not Determined
Aluminum: Not Determined

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Conditions to Avoid: Not applicable
Reactivity / Incompatibility: None reported
Hazardous Decomposition: Heating to decomposition releases toxic fumes of carbon monoxide and carbon dioxide.
Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Product Toxicological Data:
LD50: Not Determined
LC50: Not Determined
Dermal Toxicity Data: Not Determined
Skin and Eye Irritation Data: Not Determined
Mutation Data: Not Determined
Reproductive Effects Data: Not Determined
Ingredient Toxicological Data: Alcohol SDA 3A LD50: Orl Rat 7060 mg/kg

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: --
No ecological data available for the ingredients of this product.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: None
Special Instructions (Disposal): Dilute to 3 to 5 times the volume with cold water. Open cold water tap completely. slowly pour the material to the drain. Flush system with plenty of water.
Empty Containers: Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--
DOT Hazard Class: NA
DOT Subsidiary Risk: NA
DOT ID Number: NA

DOT Packing Group: NA
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
ICAO Hazard Class: NA
ICAO Subsidiary Risk: NA
ICAO ID Number: NA
ICAO Packing Group: NA
I.M.O.:
I.M.O. Proper Shipping Name: Not Currently Regulated
--
I.M.O. Hazard Class: NA
I.M.O. Subsidiary Risk: NA
I.M.O. ID Number: NA
I.M.O. Packing Group: NA
Additional Information: There is a possibility that this product could be contained in a reagent set or kit composed of various compatible dangerous goods. If the item is NOT in a set or kit. the classification given above applies. If the item IS part of a set or kit. the classification would change to the following: UN3316 Chemical Kit, Class 9, PG II or III. If the item is not regulated. the Chemical Kit classification does not apply.

15. REGULATORY INFORMATION

U.S. Federal Regulations:
O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)
E.P.A.:
S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard
S.A.R.A. Title III Section 313 (40 CFR 372): This product contains a chemical(s) subject to the reporting requirements of Section 313 of Title III of SARA.
Methanol
302 (EHS) TPQ (40 CFR 355): Not applicable
304 CERCLA RQ (40 CFR 302.4): Methanol 5000 lbs.
304 EHS RQ (40 CFR 355): Not applicable
Clean Water Act (40 CFR 116.4): Not applicable
RCRA: Not applicable
C.P.S.C.: Not applicable
State Regulations:
California Prop. 65: No Prop. 65 listed chemicals are present in this product.
Identification of Prop. 65 Ingredient(s): None
California Perchlorate Rule CCR Title 22 Chap 33: Not applicable
Trade Secret Registry: Not applicable
National Inventories:
U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).
TSCA CAS Number: Not applicable

16. OTHER INFORMATION

Intended Use: Laboratory Reagent
References: NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards. Cincinnati: Department of Health and Human Services. 1981. Gosselin. R. E. et al. Clinical Toxicology of Commercial Products, 5th Ed. Baltimore: The Williams and Wilkins Co., 1984. Vendor Information. Technical Judgment. Sax. N. Irving. Dangerous Properties of Industrial Materials. 7th Ed. New York: Van Nostrand Reinhold Co., 1989. NIOSH Registry of Toxic Effects of Chemical Substances, 1985-86. Cincinnati: U.S. Department of Health and Human Services, April. 1987. Air Contaminants. Federal Register, Vol. 54. No. 12. Thursday. January 19, 1989. pp. 2332-2983. The Merck Index, 11th Ed. Rahway. New Jersey: Merck and Co., Inc., 1989. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists. 1992. EU Occupational Exposure Limits On Line.

Revision Summary: Substantially Revised MSDS

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

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Date Printed 11/12/10
MSDS No: M01398

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: STABLCAL® FORMAZIN STANDARD 20 NTU
Catalog Number: 2684801

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M01398
Chemical Name: Not applicable
CAS Number: Not applicable
Additional CAS No. (for hydrated forms): Not applicable
Chemical Formula: Not applicable
Chemical Family: Not applicable
Intended Use: Laboratory Use Standard solution

2. HAZARDS IDENTIFICATION

GHS Classification:
Hazard categories: Respiratory or Skin Sensitization: Resp. Sens.1 Respiratory or Skin Sensitization: Skin Sens.1
GHS Label Elements:
DANGER



Hazard statements: May cause allergy or asthma symptoms or breathing difficulties if inhaled. May cause an allergic skin reaction.
Precautionary statements: Wear protective gloves / protective clothing / eye protection / face protection. Avoid breathing dust/fume/gas/mist/vapours/spray. IF ON SKIN: Wash with plenty of soap and water. If skin irritation or rash occurs: Get medical advice/attention. P272: Contaminated work clothing should not be allowed out of the workplace. Wash contaminated clothing before reuse.
HMIS:
Health: 2
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment. Section 8.
NFPA:
Health: 2
Flammability: 0
Reactivity: 0
Symbol: Not applicable

3. COMPOSITION / INFORMATION ON INGREDIENTS

Hazardous Components according to GHS: Yes
Hexamethylenetetramine

CAS Number: 100-97-0
Chemical Formula: $C_6H_{12}N_4$

GHS Classification: Flam Sol. 2, H228; Skin Sens 1. H317
Percent Range: 1.0 - 10.0
Percent Range Units: weight / weight
PEL: Not established
TLV: Not established

Hazardous Components according to GHS: ***No***

Demineralized Water

CAS Number: 7732-18-5
Chemical Formula: H_2O
GHS Classification: Not applicable
Percent Range: 90.0 - 100.0
Percent Range Units: volume / volume
PEL: Not established
TLV: Not established

Other components, each

CAS Number: Not applicable
Chemical Formula: Not applicable
GHS Classification: Not applicable
Percent Range: < 1.0
Percent Range Units: weight / weight
PEL: Not established
TLV: Not established

Formazin Polymer

CAS Number: Not applicable
Chemical Formula:
GHS Classification: Not applicable
Percent Range: < 1.0
Percent Range Units: weight / weight
PEL: Not established.
TLV: Not established.

4. FIRST AID

General Information:
Advice to doctor: Treat symptomatically.
Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.
Skin Contact (First Aid): Wash skin with soap and plenty of water. Call physician if irritation develops.
Inhalation: Remove to fresh air.
Ingestion (First Aid): Give 1-2 glasses of water under medical supervision. Never give anything by mouth to an unconscious person. Call physician immediately.

5. FIRE FIGHTING MEASURES

Flammable Properties: During a fire, this product decomposes to form toxic gases.
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear. Evacuate area and fight fire from a safe distance.
Extinguishing Media: Use media appropriate to surrounding fire conditions
Extinguishing Media NOT To Be Used: Not applicable
Fire / Explosion Hazards: None reported
Hazardous Combustion Products: Toxic fumes of: ammonia formaldehyde nitrogen oxides. carbon monoxide. carbon dioxide.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13. Special Instructions for disposal assistance.
Containment Technique: Absorb spilled liquid with non-reactive sorbent material. Dike large spills to keep spilled material from entering sewage and drainage systems or bodies of water.
Clean-up Technique: Absorb spilled liquid with non-reactive sorbent material. Sweep up material. Place material in a plastic bag. Mark bag 'Non-hazardous trash'. and dispose of as normal refuse. Decontaminate the area of the spill with a soap solution.
Evacuation Procedure: Evacuate as needed to perform spill clean-up. If conditions warrant, increase the size of the evacuation.
DOT Emergency Response Guide Number: Not applicable

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin Do not breathe mist or vapors. Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Store between 5 - 25 °C. Keep away from: direct sunlight Protect from: heat
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes skin Do not breathe: mist/vapor Wash thoroughly after handling.
TLV: Not established
PEL: Not established
For Occupational Exposure Limits (OEL) for ingredients, see section 3 - Composition/Information on Ingredients.:

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: Clear, colorless liquid
Physical State: Liquid
Molecular Weight: Not applicable
Odor: None
Odor Threshold: Odorless
pH: 8.26
Metal Corrosivity:
Corrosivity Classification: Not classified as corrosive to metals according to GHS criteria.
Steel: Not determined
Aluminum: Not determined
Specific Gravity/ Relative Density (water = 1; air =1): 1.01
Viscosity: Not determined
Solubility:
Water: Miscible
Acid: Miscible
Other: Not determined
Partition Coefficient (n-octanol / water): Not applicable
Coefficient of Water / Oil: Not applicable

Melting Point: Not determined
Decomposition Temperature: Not applicable
Boiling Point: 100°C; 212°F
Vapor Pressure: Not determined
Vapor Density (air = 1): Not determined
Evaporation Rate (water = 1): Not determined
Volatile Organic Compounds Content: Not determined
Flammable Properties: During a fire, this product decomposes to form toxic gases.
Flash Point: Not applicable
Method: Not applicable
Flammability Limits:
Lower Explosion Limits: Not applicable
Upper Explosion Limits: Not applicable
Autoignition Temperature: Not determined
Explosive Properties:
Not classified according to GHS criteria.
Oxidizing Properties:
Not classified according to GHS criteria.
Reactivity Properties:
Not classifed as self-reactive, pyrophoric, self-heating or emitting flammable gases in contact with water according to GHS criteria.
Gas under Pressure: Not classified as gas under pressure according to GHS.

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Mechanical Impact: None reported
Static Discharge: None reported.
Reactivity / Incompatibility: Incompatible with: oxidizers
Hazardous Decomposition: Heating to decomposition releases: ammonia carbon monoxide formaldehyde nitrogen oxides
Conditions to Avoid: Extreme temperatures

11. TOXICOLOGICAL INFORMATION

Toxicokinetics, Metabolism and Distribution: No information available for mixture.
Toxicologically Synergistic Products: None reported
Acute Toxicity: Based on classification principles, the classification criteria are not met.
Hexamethylenetetramine: LD 50 Orl Rat - 9200 mg/kg bw
Specific Target Organ Toxicity - Single Exposure (STOT-SE): Based on classification principles, the classification criteria are not met.
Specific Target Organ Toxicity - Repeat Exposure (STOT-RE): Based on classification principles, the classification criteria are not met.
Skin Corrosion/Irritation: Based on classification principles, the classification criteria are not met.
Eye Damage: Based on classification principles, the classification criteria are not met.
Sensitization: Contains a sensitizing compound. Respiratory Sensitizer Skin Sensitizer
Hexamethylenetetramine: skin sensitizer; in water, has been reported as a respiratory sensitizer.
CMR Effects/Properties (carcinogenic, mutagenic or toxic to reproduction): No germ cell mutagenicity, carcinogeniicty or reproductive toxicity data found.
This product does NOT contain any IARC listed chemicals.
This product does NOT contain any NTP listed chemicals.
This product does NOT contain any OSHA listed carcinogens.
Symptoms/Effects:
Ingestion: May cause: gastrointestinal tract irritation kidney damage
Inhalation: No effects anticipated
Skin Absorption: None Reported
Chronic Effects: Chronic overexposure may cause symptoms similar to acute exposure.
Medical Conditions Aggravated: Allergies or sensitivity to hexamethylenetetramine.

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: Hexamethylenetetramine: Water Pollution Factors: BOD_5: 0.015, 0.026 std. dil. sew.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number:
Special Instructions (Disposal): Dilute material with excess water making a weaker than 5% solution. Open cold water tap completely, slowly pour the material to the drain. Flush system with plenty of water.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information. In Europe: Chemical and analysis solutions must be disposed of in compliance with the respective national regulations. Product packaging must be disposed of in compliance with the country-specific regulations or must be passed to a packaging return system.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
T.D.G.:
Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
PIN: NA
Group: NA
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
I.M.O.:
I.M.O. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
Additional Information: There is a possibility that this product could be contained in a reagent set or kit composed of various compatible dangerous goods. If the item is NOT in a set or kit, the classification given above applies. If the item IS part of a set or kit, the classification would change to the following: UN3316 Chemical Kit, Class 9, PG II or III. If the item is not regulated, the Chemical Kit classification does not apply.

15. REGULATORY INFORMATION

U.S. Federal Regulations:

O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)

E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard Delayed (Chronic) Health Hazard

S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.

--

302 (EHS) TPQ (40 CFR 355): Not applicable

304 CERCLA RQ (40 CFR 302.4): Not applicable

304 EHS RQ (40 CFR 355): Not applicable

Clean Water Act (40 CFR 116.4): Not applicable

RCRA: Contains no RCRA regulated substances.

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.

Identification of Prop. 65 Ingredient(s): None

California Perchlorate Rule CCR Title 22 Chap 33:

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).

CAS Number: Not applicable

Canadian Inventory Status: All ingredients of this product are DSL Listed.

EEC Inventory Status: All ingredients used to make this product are listed on EINECS / ELINCS.

Australian Inventory (AICS) Status:

New Zealand Inventory (NZIoC) Status:

Korean Inventory (KECI) Status:

Japan (ENCS) Inventory Status:

China (PRC) Inventory (MEP) Status:

16. OTHER INFORMATION

References: 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). Air Contaminants. Federal Register. Vol. 54. No. 12. Thursday, January 19. 1989. pp. 2332-2983. CCINFO RTECS. Canadian Centre for Occupational Health and Safety. Hamilton, Ontario Canada: 30 June 1993. Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Fire Protection Guide on Hazardous Materials. 10th Ed. Quincy. MA: National Fire Protection Association, 1991. IARC Monographs on the Evaluation of the Carcinogenic Risks to Humans. World Health Organization (Volumes 1-42) Supplement 7. France: 1987. Lefevre. Marc J. First Aid Manual for Chemical Accidents, 2nd Ed. New York: Van Nostrand Reinhold Company. 1989. List of Dangerous Substances Classified in Annex I of the EEC Directive (67/548) - Classification. Packaging and Labeling of Dangerous Substances. Amended July 1992. Sixth Annual Report on Carcinogens. 1991. U.S. Department of Health and Human Services. Rockville. MD: Technical Resources, Inc. 1991. Technical Judgment. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists. 1992. Verschueren. Karel. Handbook of Environmental Data on Organic Chemicals. New York: Van Nostrand Reinhold Co.. 1977.

Complete Text of H phrases referred to in Section 3: H334 May cause allergy or asthma symptoms or breathing difficulties if inhaled. H317 May cause an allergic skin reaction. H228 Flammable solid.

Revision Summary: . Substantially Revised MSDS

Date of MSDS Preparation:

Day: 01

Month: October

Year: 2010

MSDS Prepared: MSDS prepared by Product Compliance Department extension 3350

CCOHS Evaluation Note: This product has been classified and labeled in accordance with the requirements of GHS (ST/SG/AC.10/36/Add.3). It is offered under exemption from WHMIS labeling as specified in the Controlled Products Regulation (CPR) Section 17. It is offered under the interim policy that was established by Health Canada permitting use of GHS-formatted safety data sheets in Canada prior to revision of CPR to GHS.

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

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Date Printed 11/12/10
MSDS No: M01360

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: STABLCAL® FORMAZIN STANDARD 100 NTU
Catalog Number: 2684901

Hach Company
P.O.Box 389
Loveland. CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M01360
Chemical Name: Not applicable
CAS Number: Not applicable
Additional CAS No. (for hydrated forms): Not applicable
Chemical Formula: Not applicable
Chemical Family: Not applicable
Intended Use: Laboratory Use Standard solution

2. HAZARDS IDENTIFICATION

GHS Classification:
Hazard categories: Respiratory or Skin Sensitization: Resp. Sens.1 Respiratory or Skin Sensitization: Skin Sens.1
GHS Label Elements:
DANGER



Hazard statements: May cause allergy or asthma symptoms or breathing difficulties if inhaled. May cause an allergic skin reaction.
Precautionary statements: Wear protective gloves / protective clothing / eye protection / face protection. . IF ON SKIN: Wash with plenty of soap and water. If skin irritation or rash occurs: Get medical advice/attention. P272: Contaminated work clothing should not be allowed out of the workplace. Wash contaminated clothing before reuse.
HMIS:
Health: 2
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment. Section 8.
NFPA:
Health: 2
Flammability: 0
Reactivity: 0
Symbol: Not applicable

3. COMPOSITION / INFORMATION ON INGREDIENTS

Hazardous Components according to GHS: ***Yes***
Hexamethylenetetramine

CAS Number: 100-97-0
Chemical Formula: $C_6H_{12}N_4$
GHS Classification: Flam Sol. 2. H228; Skin Sens 1. H317

Percent Range: 1.0 - 10.0
Percent Range Units: weight / weight
PEL: Not established
TLV: Not established

Hazardous Components according to GHS: *No*

Demineralized Water

CAS Number: 7732-18-5
Chemical Formula: H_2O
GHS Classification: Not applicable
Percent Range: 90.0 - 100.0
Percent Range Units: volume / volume
PEL: Not established
TLV: Not established

Other components, each

CAS Number: Not applicable
Chemical Formula: Not applicable
GHS Classification: Not applicable
Percent Range: < 1.0
Percent Range Units: weight / volume
PEL: Not established
TLV: Not established

Formazin Polymer

CAS Number: Not applicable
Chemical Formula:
GHS Classification: Not applicable
Percent Range: < 1.0
Percent Range Units: weight / weight
PEL: Not established.
TLV: Not established.

4. FIRST AID

General Information:
Advice to doctor: Treat symptomatically.
Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.
Skin Contact (First Aid): Wash skin with soap and plenty of water. Call physician if irritation develops.
Inhalation: Remove to fresh air.
Ingestion (First Aid): Give 1-2 glasses of water under medical supervision. Never give anything by mouth to an unconscious person. Call physician immediately.

5. FIRE FIGHTING MEASURES

Flammable Properties: During a fire, this product decomposes to form toxic gases.
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear. Evacuate area and fight fire from a safe distance.
Extinguishing Media: Use media appropriate to surrounding fire conditions
Extinguishing Media NOT To Be Used: Not applicable
Fire / Explosion Hazards: None reported
Hazardous Combustion Products: This material will not burn.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13. Special Instructions for disposal assistance.
Containment Technique: Absorb spilled liquid with non-reactive sorbent material. Dike large spills to keep spilled material from entering sewage and drainage systems or bodies of water.
Clean-up Technique: Absorb spilled liquid with non-reactive sorbent material. Sweep up material. Place material in a plastic bag. Mark bag 'Non-hazardous trash', and dispose of as normal refuse. Decontaminate the area of the spill with a soap solution.
Evacuation Procedure: Evacuate as needed to perform spill clean-up. If conditions warrant, increase the size of the evacuation.
DOT Emergency Response Guide Number: Not applicable

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin Do not breathe mist or vapors. Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Store between 5 - 25 °C. Keep away from: direct sunlight Protect from: heat
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes skin Do not breathe: mist/vapor Wash thoroughly after handling.
TLV: Not established
PEL: Not established
For Occupational Exposure Limits (OEL) for ingredients, see section 3 - Composition/Information on Ingredients.:

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: Turbid, milky suspension
Physical State: Liquid
Molecular Weight: Not applicable
Odor: None
Odor Threshold: Odorless
pH: 8.14
Metal Corrosivity:
Corrosivity Classification: Not classified as corrosive to metals according to GHS criteria.
Steel: Not determined
Aluminum: Not determined
Specific Gravity/ Relative Density (water = 1; air =1): 1.01
Viscosity: Not determined
Solubility:
Water: Miscible
Acid: Miscible
Other: Not determined
Partition Coefficient (n-octanol / water): Not applicable
Coefficient of Water / Oil: Not applicable
Melting Point: Not applicable

Decomposition Temperature: Not applicable
Boiling Point: 100°C
Vapor Pressure: Not determined
Vapor Density (air = 1): Not determined
Evaporation Rate (water = 1): Not determined
Volatile Organic Compounds Content: Not determined
Flammable Properties: During a fire, this product decomposes to form toxic gases.
Flash Point: Not applicable
Method: Not applicable
Flammability Limits:
Lower Explosion Limits: Not applicable
Upper Explosion Limits: Not applicable
Autoignition Temperature: Not determined
Explosive Properties:
Not classified according to GHS criteria.
Oxidizing Properties:
Not classified according to GHS criteria.
Reactivity Properties:
Not classifed as self-reactive, pyrophoric, self-heating or emitting flammable gases in contact with water according to GHS criteria.
Gas under Pressure: Not classified as gas under pressure according to GHS.

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Mechanical Impact: None reported
Static Discharge: None reported.
Reactivity / Incompatibility: Incompatible with: oxidizers
Hazardous Decomposition: Heating to decomposition releases: ammonia carbon monoxide formaldehyde nitrogen oxides
Conditions to Avoid: Extreme temperatures

11. TOXICOLOGICAL INFORMATION

Toxicokinetics, Metabolism and Distribution: No information available for mixture.
Toxicologically Synergistic Products: None reported
Acute Toxicity: Based on classification principles, the classification criteria are not met.
Hexamethylenetetramine: LD 50 Orl Rat - 9200 mg/kg bw
Specific Target Organ Toxicity - Single Exposure (STOT-SE): Based on classification principles, the classification criteria are not met.
Specific Target Organ Toxicity - Repeat Exposure (STOT-RE): Based on classification principles, the classification criteria are not met.
Skin Corrosion/Irritation: Based on classification principles, the classification criteria are not met.
Eye Damage: Based on classification principles, the classification criteria are not met.
Sensitization: Contains a sensitizing compound. Respiratory Sensitizer Skin Sensitizer
Hexamethylenetetramine: skin sensitizer; in water, has been reported as a respiratory sensitizer.
CMR Effects/Properties (carcinogenic, mutagenic or toxic to reproduction): No germ cell mutagenicity, carcinogeniicty or reproductive toxicity data found.
This product does NOT contain any IARC listed chemicals.
This product does NOT contain any NTP listed chemicals.
This product does NOT contain any OSHA listed carcinogens.
Symptoms/Effects:
Ingestion: May cause: gastrointestinal tract irritation kidney damage
Inhalation: No effects anticipated
Skin Absorption: None Reported
Chronic Effects: Chronic overexposure may cause symptoms similar to acute exposure.
Medical Conditions Aggravated: Allergies or sensitivity to hexamethylenetetramine.

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: Hexamethylenetetramine: Water Pollution Factors: BOD_5: 0.015, 0.026, std. dil. sew.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number: Not applicable
Special Instructions (Disposal): Dilute material with excess water making a weaker than 5% solution. Open cold water tap completely, slowly pour the material to the drain. Flush system with plenty of water.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information. In Europe: Chemical and analysis solutions must be disposed of in compliance with the respective national regulations. Product packaging must be disposed of in compliance with the country-specific regulations or must be passed to a packaging return system.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
T.D.G.:
Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
PIN: NA
Group: NA
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
I.M.O.:
I.M.O. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
Additional Information: There is a possibility that this product could be contained in a reagent set or kit composed of various compatible dangerous goods. If the item is NOT in a set or kit, the classification given above applies. If the item IS part of a set or kit, the classification would change to the following: UN3316 Chemical Kit, Class 9, PG II or III. If the item is not regulated, the Chemical Kit classification does not apply.

15. REGULATORY INFORMATION

U.S. Federal Regulations:

O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)

E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard Delayed (Chronic) Health Hazard

S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.

--

302 (EHS) TPQ (40 CFR 355): Not applicable

304 CERCLA RQ (40 CFR 302.4): Not applicable

304 EHS RQ (40 CFR 355): Not applicable

Clean Water Act (40 CFR 116.4): Not applicable

RCRA: Contains no RCRA regulated substances.

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.

Identification of Prop. 65 Ingredient(s): Not applicable

California Perchlorate Rule CCR Title 22 Chap 33:

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).

CAS Number: Not applicable

Canadian Inventory Status: All ingredients of this product are DSL Listed.

EEC Inventory Status: All ingredients used to make this product are listed on EINECS / ELINCS.

Australian Inventory (AICS) Status:

New Zealand Inventory (NZIoC) Status:

Korean Inventory (KECI) Status:

Japan (ENCS) Inventory Status:

China (PRC) Inventory (MEP) Status:

16. OTHER INFORMATION

References: 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). Air Contaminants, Federal Register, Vol. 54, No. 12. Thursday, January 19, 1989. pp. 2332-2983. CCINFO RTECS. Canadian Centre for Occupational Health and Safety. Hamilton, Ontario Canada: 30 June 1993. Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Fire Protection Guide on Hazardous Materials, 10th Ed. Quincy, MA: National Fire Protection Association, 1991. IARC Monographs on the Evaluation of the Carcinogenic Risks to Humans. World Health Organization (Volumes 1-42) Supplement 7. France: 1987. Lefevre, Marc J. First Aid Manual for Chemical Accidents, 2nd Ed. New York: Van Nostrand Reinhold Company, 1989. List of Dangerous Substances Classified in Annex I of the EEC Directive (67/548) - Classification, Packaging and Labeling of Dangerous Substances. Amended July 1992. Sixth Annual Report on Carcinogens, 1991. U.S. Department of Health and Human Services. Rockville, MD: Technical Resources, Inc. 1991. Technical Judgment. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists, 1992. Verschueren, Karel. Handbook of Environmental Data on Organic Chemicals. New York: Van Nostrand Reinhold Co., 1977.

Complete Text of H phrases referred to in Section 3: H334 May cause allergy or asthma symptoms or breathing difficulties if inhaled. H317 May cause an allergic skin reaction. H228 Flammable solid.

Revision Summary: . Substantially Revised MSDS

Date of MSDS Preparation:

Day: 01

Month: October

Year: 2010

MSDS Prepared: MSDS prepared by Product Compliance Department extension 3350

CCOHS Evaluation Note: This product has been classified and labeled in accordance with the requirements of GHS (ST/SG/AC.10/36/Add.3). It is offered under exemption from WHMIS labeling as specified in the Controlled Products Regulation (CPR) Section 17. It is offered under the interim policy that was established by Health Canada permitting use of GHS-formatted safety data sheets in Canada prior to revision of CPR to GHS.

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2010

World Headquarters
Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Date Printed 11/12/10
MSDS No: M01401

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: STABLCAL® FORMAZIN STANDARD 800 NTU
Catalog Number: 2660501

Hach Company
P.O.Box 389
Loveland, CO USA 80539
(970) 669-3050

Emergency Telephone Numbers:
(Medical and Transportation)
(303) 623-5716 24 Hour Service
(515)232-2533 8am - 4pm CST

MSDS Number: M01401
Chemical Name: Not applicable
CAS Number: Not applicable
Additional CAS No. (for hydrated forms): Not applicable
Chemical Formula: Not applicable
Chemical Family: Not applicable
Intended Use: Laboratory Use Standard solution

2. HAZARDS IDENTIFICATION

GHS Classification:
Hazard categories: Respiratory or Skin Sensitization: Resp. Sens.1 Respiratory or Skin Sensitization: Skin Sens.1
GHS Label Elements:
DANGER



Hazard statements: May cause allergy or asthma symptoms or breathing difficulties if inhaled. May cause an allergic skin reaction.
Precautionary statements: Wear protective gloves / protective clothing / eye protection / face protection. . IF ON SKIN: Wash with plenty of soap and water. If skin irritation or rash occurs: Get medical advice/attention. P272: Contaminated work clothing should not be allowed out of the workplace. Wash contaminated clothing before reuse.
HMIS:
Health: 2
Flammability: 0
Reactivity: 0
Protective Equipment: X - See protective equipment, Section 8.
NFPA:
Health: 2
Flammability: 0
Reactivity: 0
Symbol: Not applicable

3. COMPOSITION / INFORMATION ON INGREDIENTS

Hazardous Components according to GHS: ***Yes***
Hexamethylenetetramine

CAS Number: 100-97-0
Chemical Formula: $C_6H_{12}N_4$
GHS Classification: Flam Sol. 2, H228; Skin Sens 1, H317

Percent Range: 1.0 - 10.0
Percent Range Units: weight / weight
PEL: Not established
TLV: Not established

Hazardous Components according to GHS: ***No***

Demineralized Water

CAS Number: 7732-18-5
Chemical Formula: H_2O
GHS Classification: Not applicable
Percent Range: 90.0 - 100.0
Percent Range Units: volume / volume
PEL: Not established
TLV: Not established

Other components, each

CAS Number: Not applicable
Chemical Formula: Not applicable
GHS Classification: Not applicable
Percent Range: < 1.0
Percent Range Units: weight / weight
PEL: Not established
TLV: Not established

Formazin Polymer

CAS Number: Not applicable
Chemical Formula:
GHS Classification: Not applicable
Percent Range: < 1.0
Percent Range Units: weight / weight
PEL: Not established.
TLV: Not established.

4. FIRST AID

General Information:
Advice to doctor: Treat symptomatically.
Eye Contact: Immediately flush eyes with water for 15 minutes. Call physician.
Skin Contact (First Aid): Wash skin with soap and plenty of water. Call physician if irritation develops.
Inhalation: Remove to fresh air.
Ingestion (First Aid): Give 1-2 glasses of water under medical supervision. Never give anything by mouth to an unconscious person. Call physician immediately.

5. FIRE FIGHTING MEASURES

Flammable Properties: During a fire, this product decomposes to form toxic gases.
Fire Fighting Instruction: As in any fire, wear self-contained breathing apparatus pressure-demand and full protective gear. Evacuate area and fight fire from a safe distance.
Extinguishing Media: Use media appropriate to surrounding fire conditions
Extinguishing Media NOT To Be Used: Not applicable
Fire / Explosion Hazards: None reported
Hazardous Combustion Products: Toxic fumes of: ammonia formaldehyde nitrogen oxides. carbon monoxide. carbon dioxide.

6. ACCIDENTAL RELEASE MEASURES

Spill Response Notice:
Only persons properly qualified to respond to an emergency involving hazardous substances may respond to a spill according to federal regulations (OSHA 29 CFR 1910.120(a)(v)) and per your company's emergency response plan and guidelines/procedures. See Section 13. Special Instructions for disposal assistance.
Containment Technique: Absorb spilled liquid with non-reactive sorbent material. Dike large spills to keep spilled material from entering sewage and drainage systems or bodies of water.
Clean-up Technique: Absorb spilled liquid with non-reactive sorbent material. Sweep up material. Place material in a plastic bag. Mark bag 'Non-hazardous trash', and dispose of as normal refuse. Decontaminate the area of the spill with a soap solution.
Evacuation Procedure: Evacuate as needed to perform spill clean-up. If conditions warrant, increase the size of the evacuation.
DOT Emergency Response Guide Number: Not applicable

7. HANDLING / STORAGE

Handling: Avoid contact with eyes skin Do not breathe mist or vapors. Wash thoroughly after handling. Maintain general industrial hygiene practices when using this product.
Storage: Store between 5 - 25 °C. Keep away from: direct sunlight Protect from: heat
Flammability Class: Not applicable

8. EXPOSURE CONTROLS / PROTECTIVE EQUIPMENT

Engineering Controls: Have an eyewash station nearby. Maintain general industrial hygiene practices when using this product.
Personal Protective Equipment:
Eye Protection: safety glasses with top and side shields
Skin Protection: disposable latex gloves
Inhalation Protection: adequate ventilation
Precautionary Measures: Avoid contact with: eyes skin Do not breathe: mist/vapor Wash thoroughly after handling.
TLV: Not established
PEL: Not established
For Occupational Exposure Limits (OEL) for ingredients, see section 3 - Composition/Information on Ingredients.:

9. PHYSICAL / CHEMICAL PROPERTIES

Appearance: Turbid, milky suspension
Physical State: Liquid
Molecular Weight: Not applicable
Odor: None
Odor Threshold: Odorless
pH: 7.36
Metal Corrosivity:
Corrosivity Classification: Not classified as corrosive to metals according to GHS criteria.
Steel: Not determined
Aluminum: Not determined
Specific Gravity/ Relative Density (water = 1; air =1): 1.01
Viscosity: Not determined
Solubility:
Water: Miscible
Acid: Miscible
Other: Not determined
Partition Coefficient (n-octanol / water): Not applicable
Coefficient of Water / Oil: Not applicable

Melting Point: Not determined
Decomposition Temperature: Not applicable
Boiling Point: 100°C; 212°F
Vapor Pressure: Not determined
Vapor Density (air = 1): Not determined
Evaporation Rate (water = 1): Not determined
Volatile Organic Compounds Content: Not determined
Flammable Properties: During a fire, this product decomposes to form toxic gases.
Flash Point: Not applicable
Method: Not applicable
Flammability Limits:
Lower Explosion Limits: Not applicable
Upper Explosion Limits: Not applicable
Autoignition Temperature: Not determined
Explosive Properties:
Not classified according to GHS criteria.
Oxidizing Properties:
Not classified according to GHS criteria.
Reactivity Properties:
Not classifed as self-reactive, pyrophoric, self-heating or emitting flammable gases in contact with water according to GHS criteria.
Gas under Pressure: Not classified as gas under pressure according to GHS.

10. STABILITY / REACTIVITY

Chemical Stability: Stable when stored under proper conditions.
Mechanical Impact: None reported
Static Discharge: None reported.
Reactivity / Incompatibility: Incompatible with: oxidizers
Hazardous Decomposition: Heating to decomposition releases: ammonia carbon monoxide formaldehyde nitrogen oxides
Conditions to Avoid: Extreme temperatures

11. TOXICOLOGICAL INFORMATION

Toxicokinetics, Metabolism and Distribution: No information available for mixture.
Toxicologically Synergistic Products: None reported
Acute Toxicity: Based on classification principles, the classification criteria are not met.
Hexamethylenetetramine: LD 50 Orl Rat - 9200 mg/kg bw
Specific Target Organ Toxicity - Single Exposure (STOT-SE): Based on classification principles, the classification criteria are not met.
Specific Target Organ Toxicity - Repeat Exposure (STOT-RE): Based on classification principles, the classification criteria are not met.
Skin Corrosion/Irritation: Based on classification principles, the classification criteria are not met.
Eye Damage: Based on classification principles, the classification criteria are not met.
Sensitization: Contains a sensitizing compound. Respiratory Sensitizer Skin Sensitizer
Hexamethylenetetramine: skin sensitizer; in water, has been reported as a respiratory sensitizer.
CMR Effects/Properties (carcinogenic, mutagenic or toxic to reproduction): No germ cell mutagenicity, carcinogeniicty or reproductive toxicity data found.
This product does NOT contain any IARC listed chemicals.
This product does NOT contain any NTP listed chemicals.
This product does NOT contain any OSHA listed carcinogens.
Symptoms/Effects:
Ingestion: May cause: gastrointestinal tract irritation kidney damage
Inhalation: No effects anticipated
Skin Absorption: None Reported
Chronic Effects: Chronic overexposure may cause symptoms similar to acute exposure.
Medical Conditions Aggravated: Allergies or sensitivity to hexamethylenetetramine.

12. ECOLOGICAL INFORMATION

Product Ecological Information: --
No ecological data available for this product.
Ingredient Ecological Information: Hexamethylenetetramine: Water Pollution Factors: BOD_5: 0.015, 0.026, std. dil. sew.

13. DISPOSAL CONSIDERATIONS

EPA Waste ID Number:
Special Instructions (Disposal): Dilute material with excess water making a weaker than 5% solution. Open cold water tap completely, slowly pour the material to the drain. Flush system with plenty of water.
Empty Containers: Rinse three times with an appropriate solvent. Dispose of empty container as normal trash.
NOTICE (Disposal): These disposal guidelines are based on federal regulations and may be superseded by more stringent state or local requirements. Please consult your local environmental regulators for more information. In Europe: Chemical and analysis solutions must be disposed of in compliance with the respective national regulations. Product packaging must be disposed of in compliance with the country-specific regulations or must be passed to a packaging return system.

14. TRANSPORT INFORMATION

D.O.T.:
D.O.T. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
T.D.G.:
Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
PIN: NA
Group: NA
I.C.A.O.:
I.C.A.O. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
I.M.O.:
I.M.O. Proper Shipping Name: Not Currently Regulated
--
Hazard Class: NA
Subsidiary Risk: NA
ID Number: NA
Packing Group: NA
Additional Information: There is a possibility that this product could be contained in a reagent set or kit composed of various compatible dangerous goods. If the item is NOT in a set or kit, the classification given above applies. If the item IS part of a set or kit, the classification would change to the following: UN3316 Chemical Kit, Class 9, PG II or III. If the item is not regulated, the Chemical Kit classification does not apply.

15. REGULATORY INFORMATION

U.S. Federal Regulations:

O.S.H.A.: This product meets the criteria for a hazardous substance as defined in the Hazard Communication Standard. (29 CFR 1910.1200)

E.P.A.:

S.A.R.A. Title III Section 311/312 Categorization (40 CFR 370): Immediate (Acute) Health Hazard Delayed (Chronic) Health Hazard

S.A.R.A. Title III Section 313 (40 CFR 372): This product does NOT contain any chemical subject to the reporting requirements of Section 313 of Title III of SARA.

--

302 (EHS) TPQ (40 CFR 355): Not applicable

304 CERCLA RQ (40 CFR 302.4): Not applicable

304 EHS RQ (40 CFR 355): Not applicable

Clean Water Act (40 CFR 116.4): Not applicable

RCRA: Contains no RCRA regulated substances.

State Regulations:

California Prop. 65: No Prop. 65 listed chemicals are present in this product.

Identification of Prop. 65 Ingredient(s): None

California Perchlorate Rule CCR Title 22 Chap 33:

Trade Secret Registry: Not applicable

National Inventories:

U.S. Inventory Status: All ingredients in this product are listed on the TSCA 8(b) Inventory (40 CFR 710).

CAS Number: Not applicable

Canadian Inventory Status: All ingredients of this product are DSL Listed.

EEC Inventory Status: All ingredients used to make this product are listed on EINECS / ELINCS.

Australian Inventory (AICS) Status:

New Zealand Inventory (NZIoC) Status:

Korean Inventory (KECI) Status:

Japan (ENCS) Inventory Status:

China (PRC) Inventory (MEP) Status:

16. OTHER INFORMATION

References: 29 CFR 1900 - 1910 (Code of Federal Regulations - Labor). Air Contaminants. Federal Register. Vol. 54. No. 12. Thursday, January 19. 1989. pp. 2332-2983. CCINFO RTECS. Canadian Centre for Occupational Health and Safety. Hamilton. Ontario Canada: 30 June 1993. Fire Protection Guide on Hazardous Materials. 10th Ed. Quincy. MA: National Fire Protection Fire Protection Guide on Hazardous Materials. 10th Ed. Quincy. MA: National Fire Protection Association, 1991. IARC Monographs on the Evaluation of the Carcinogenic Risks to Humans. World Health Organization (Volumes 1-42) Supplement 7. France: 1987. Lefevre. Marc J. First Aid Manual for Chemical Accidents. 2nd Ed. New York: Van Nostrand Reinhold Company. 1989. List of Dangerous Substances Classified in Annex I of the EEC Directive (67/548) - Classification. Packaging and Labeling of Dangerous Substances. Amended July 1992. Sixth Annual Report on Carcinogens, 1991. U.S. Department of Health and Human Services. Rockville. MD: Technical Resources, Inc. 1991. Technical Judgment. TLV's Threshold Limit Values and Biological Exposure Indices for 1992-1993. American Conference of Governmental Industrial Hygienists. 1992. Verschueren. Karel. Handbook of Environmental Data on Organic Chemicals. New York: Van Nostrand Reinhold Co., 1977.

Complete Text of H phrases referred to in Section 3: H334 May cause allergy or asthma symptoms or breathing difficulties if inhaled. H317 May cause an allergic skin reaction. H228 Flammable solid.

Revision Summary: . Substantially Revised MSDS

Date of MSDS Preparation:

Day: 01

Month: October

Year: 2010

MSDS Prepared: MSDS prepared by Product Compliance Department extension 3350

CCOHS Evaluation Note: This product has been classified and labeled in accordance with the requirements of GHS (ST/SG/AC.10/36/Add.3). It is offered under exemption from WHMIS labeling as specified in the Controlled Products Regulation (CPR) Section 17. It is offered under the interim policy that was established by Health Canada permitting use of GHS-formatted safety data sheets in Canada prior to revision of CPR to GHS.

Legend:

NA - Not Applicable	w/w - weight/weight
ND - Not Determined	w/v - weight/volume
NV - Not Available	v/v - volume/volume

USER RESPONSIBILITY: Each user should read and understand this information and incorporate it in individual site safety programs in accordance with applicable hazard communication standards and regulations.

THE INFORMATION CONTAINED HEREIN IS BASED ON DATA CONSIDERED TO BE ACCURATE. HOWEVER, NO WARRANTY IS EXPRESSED OR IMPLIED REGARDING THE ACCURACY OF THESE DATA OR THE RESULTS TO BE OBTAINED FROM THE USE THEREOF.

HACH COMPANY ©2010

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.

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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 and Coordination with Utah Division of Water Quality, Utah Units

Version 1.01 (7/27/12)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.00	7/27/2012	R. Weissinger	UTDWQ Sampling and Analysis plan added	New State requirement	1.01

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.

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9. Appendix B: Example Sampling and Analysis Plan for the State of Utah **9**

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 monthly site visits, and lists the names and locations of supported sampling sites.

The location of sampling sites can change, but they must be approved by UTDWQ personnel. A Sampling and Analysis plan must be submitted annually prior to the start of the water year listing the sites, their location and sampling frequency (Appendix B). This table is updated annually as per Tables 6-8 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.

4. Information Received at the Training

4.1. Sampling schedule

The UTDWQ requests monthly samples for the water 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. 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.

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).
2. Keep a calibration log with each instrument.
    a. For the multiparameter probe this will be generated automatically and will be incorporated into the Field_forms.xls data sheet (electronic version).
    b. For the flow meter this will be generated automatically as part of the data file and will be incorporated into the Field_forms.xls data sheet (electronic version).
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.
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. UTDWQ 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. UTDWQ recalibrates their optical DO sensors using a bubbler in the office each day.
    b. Because of the remote nature of many NCPN sampling locations, NCPN calibrates the optical DO sensor in the office prior to each sampling run.
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 and the NCPN Backcountry Safety Plan. Discussion of field safety includes:

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. Satellite phone and/or personal locator beacon
    v. Backcountry travel plan filed with Glen Canyon dispatch

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 are acceptable or not.

8. 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 accepted by UTDWQ for the cooperative monitoring program. Panels are C = continuous, R = rotating, and QC = quality control samples.

Park	Storet ID	Station Name	Sampling	Panel
All Utah	5995202	NPSETAL Field Blank	NCPN/SEUG	QC
ARCH	5995200	FRESHWATER SPG IN SALT WASH .5MI AB RD XING SW-1	SEUG	R
ARCH	5995220	SALT WASH AT WOLFE RANCH RD XING SW-3	SEUG	C
ARCH	5995222	Salt Wash above Winter Camp Wash at road crossing	SEUG	QC
ARCH	5995240	COURTHOUSE WASH .5MI AB COLORADO R AT USGS CW-1	SEUG	C
ARCH	5995245	Upper Courthouse Wash @ NPS BNDY	SEUG	R
ARCH	5995246	Courthouse Wash at spring approx. 75m inside NPS west boundary	SEUG	R
ARCH	5995250	SLEEPY HOLLOW SPRING IN UPPER COURTHOUSE WASH SH-1	SEUG	R
ARCH	5995270	WILLOW SPRING WS-1	SEUG	R
ARCH	5995290	LOST SPRING	SEUG	R
BRCA	4951855	Sheep Ck bl Spring in Bryce Cyn NP	NCPN	C
BRCA	4951857	Yellow Ck bl Spring in Bryce Cyn NP	NCPN	R
BRCA	4951915	MOSSY CAVE SPRING NW OF TROPIC	NCPN	R
CANY	4930010	GREEN R AB CNFL / COLORADO R	SEUG	C
CANY	4930150	GREEN R AT MINERAL BOTTOMS	SEUG	C
CANY	4952380	COLORADO R. BL BIG DROP #3 RAPIDS	SEUG	C
CANY	4952400	COLORADO R AB CNFL / GREEN R	SEUG	C
CANY	4956290	COLORADO RIVER AT POTASH BOAT RAMP	SEUG	C
CANY	4956291	COLORADO RIVER AT POTASH BOAT RAMP	SEUG	QC
CANY	5995020	2.4 MILE LOOP SPRING BS-2	SEUG	R
CANY	5995050	CAVE SPRING SQ-3	SEUG	R
CANY	5995120	LITTLE SPRING CANYON CREEK LS2	SEUG	R
CANY	5995150	SALT CK NR CRESCENT ARCH SC-10	SEUG	
CANY	5995160	SALT CREEK NR PEEKABOO SPRING SC-12	SEUG	R

Park	Storet ID	Station Name	Sampling	Panel
CANY	5995420	CHOCOLATE DROPS SPG .5 MI S OF MAZE OVERLOOK SF-4	SEUG	R
CANY	5995500	MAZE OVERLOOK SPG .25MI SE OF MAZE OVERLOOK SF-2	SEUG	R
CANY	5995540	HORSESHOE CYN SPG .3MI AB CNFL/ WATER CYN LOWER HSC-2	SEUG	R
CARE	4954770	SULPHUR CK AB CNFL/ FREMONT R IN PICNIC AREA	NCPN	R
CARE	4954775	SULPHUR CK BL ROAD TO GOOSENECKS OVERLOOK IN CRNP	NCPN	R
CARE	4954780	PLEASANT CK S OF SLEEPING RAINBOW RANCH	NCPN	R
CARE	4954795	Oak Creek above Sands Ranch Dam	NCPN	R
CARE	4954812	Halls Ck Bl Narrows	NCPN	R
HOVE	5995700	SQUARE TOWER SPRING	SEUG	R
HOVE	5995710	CAJON SPRING	SEUG	R
HOVE	5995720	HACKBERRY HOUSE SPRING	SEUG	R
NABR	5995310	ARMSTRONG CANYON CK AT KACHINA NATURAL BRIDGE KB-1	SEUG	R
NABR	5995320	OWACHOMO BRIDGE SPRING IN TUWA CYN AB CNFL OB-1	SEUG	R
NABR	5995330	SIPAPU BRIDGE SPRING .25 MI AB BRIDGE SB-1	SEUG	R
TICA	4994970	HANSEN CAVE SPRING IN TIMPANOGOS CAVE NM	TICA	C
ZION	4950800	La Verkin Ck at Lee Pass Trail	NCPN	C
ZION	4950920	North Ck at Planned Gage in Park	NCPN	C
ZION	4951260	North Fk Virgin R at North Fork Rd	NCPN	C
ZION	4951261	North Fk Virgin ab Scenic Narrows	NCPN	QC
ZION	4951265	N FK VIRGIN R AT WSA BNDRY	NCPN	C
ZION	4951266	N FK VIRGIN R AB CNFL/ BAPTISM RIVER	NCPN	QC

9. Appendix B: Example Sampling and Analysis Plan for the State of Utah

WY2016 National Park Service Northern Colorado Plateau Network Water Quality Sampling and Analysis Plan

Prepared by: Rebecca Weissinger, NCPN and Mary Moran, SEUG
Date submitted:

Submitted to: Utah Department of Water Quality
Reviewed by:
Date reviewed:

Parent documents:

> Thoma, D., D. Sharrow, K. Wynn, J. Brown, M. Beer, and H. Thomas. 2009. Water quality vital signs monitoring protocol for park units in the Northern Colorado Plateau Network. National Park Service, Moab, UT.

The complete program documentation can be found at:
http://science.nature.nps.gov/im/units/ncpn/WQBrief.cfm

Project Overview

The Northern Colorado Plateau Network Inventory and Monitoring program (NCPN) conducts long-term monitoring of natural resources in sixteen national park units in Utah, western Colorado, northern Arizona and southern Wyoming. In cooperation with the Utah Department of Water Quality and partner staff at the Southeast Utah Group (SEUG) and Timpanogos Cave (TICA) of the National Park Service, the NCPN coordinates sample collection, data archiving, reporting and analysis for a selected set of waters in the following Utah parks:

Submitted as	Code	Park Unit	Sampled by
NPSCAN	ARCH	Arches National Park	SEUG
NPSCAN	HOVE	Hovenweep National Monument	SEUG
NPSCAN	NABR	Natural Bridges National Monument	SEUG
NPSCAN RIVER	CANY	Canyonlands National Park	SEUG
NPSETAL	BRCA	Bryce Canyon National Park	NCPN
NPSETAL	CARE	Capitol Reef National Park	NCPN
NPSETALTimp	TICA	Timpanogos Cave National Monument	TICA
NPSETAL	ZION	Zion National Park	NCPN

Project Objectives

NCPN Water Quality monitoring emphasizes the support of protected uses consistent with National Park Service directives through meeting or exceeding water quality standards as developed by the state of Utah.

1. Establish range and variability of water quality parameters under base flow conditions as they vary with seasonal and climatic conditions.
2. Determine status and trends in selected water quality parameters as a function of flow, season and climatic conditions.

3. Compare water quality data against state standards for chronic exceedance on a monthly and annual basis. Screen for possible *E. coli* exceedances.
4. Analyze long term data for designation as Outstanding National Resource Waters (or equivalent Utah Category 1 waters).
5. Determine point and non-point sources of pollution within watershed via association.

Program Staff

Code	Name	Email	Phone	Duties
NCPN	Carolyn Hackbarth	Carolyn_Hackbarth@nps.gov	435-719-2344	Sample collection and submittal
NCPN	Rebecca Weissinger	Rebecca_Weissinger@nps.gov	435-719-2356	Program coordination, data review, analysis, reporting
NCPN	Helen Thomas	Helen_Thomas@nps.gov	435-719-2340	Data archiving
SEUG	Mary Moran	Mary_Moran@nps.gov	435-719-2139	Sample collection and submittal, data review
TICA	Cami McKinney	Cami_McKinney@nps.gov	801-756-5239 x501	Sample collection and submittal

Water Year 2016 Sample Sites and Proposed Analytes

Core parameters of pH, specific conductance, dissolved oxygen, water temperature and discharge are collected during each visit except for Colorado River sites, where usually only water temperature and discharge are collected. Turbidity and fecal coliforms are also measured at some sites.

NCPN sampling- ***NPSETAL***

Sites highlighted in green are proposed sampling sites for WY16.

Storet #	Site Name	Park	Monthly parameters (10x/year)	Quarterly parameters (4x/year)	Visits in WY16	WQ Probe	Flow (units)	Threats	Past exceedances
4950800	LaVerkin Creek	ZION	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	visitor use	total P
4950920	North Creek	ZION	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	visitor use, fire effects, livestock	pH, TDS, dissolved oxygen, total P
4951265	North Fork Virgin River WSA	ZION	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	livestock, visitor use, housing development, oil and gas	*E. coli*, total P
4951266	North Fork Virgin River above Narrows	ZION	Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	5	In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	Lab duplicate sample – Quality control	

Storet #	Site Name	Park	Monthly parameters (10x/year)	Quarterly parameters (4x/year)	Visits in WY16	WQ Probe	Flow (units)	Threats	Past exceedances
4951855	Sheep Creek	BRCA	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Rickly v-notch weir	livestock, visitor use, road building	*E. coli*, TDS
4951857	Yellow Creek	BRCA	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)		In-Situ TROLL 9500	Baski 8" cutthroat flume (cfs)	livestock, visitor use, agriculture, road building	*E. coli*, sulfate, boron
4951915	Mossy Cave	BRCA	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Rickly v-notch weir	visitor use, tourism development	*E. coli*
4954770	Lower Sulphur Creek	CARE	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	agriculture	*E. coli*
4954775	Upper Sulphur Creek	CARE	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	livestock, agriculture	temperature, total P, unionized ammonia, TDS, TSS, *E. coli*
4954780	Pleasant Creek	CARE	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)		In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	livestock	pH, total P, unionized ammonia, temperature, TSS, *E. coli*

Storet #	Site Name	Park	Monthly parameters (10x/year)	Quarterly parameters (4x/year)	Visits in WY16	WQ Probe	Flow (units)	Threats	Past exceedances
4954795	Oak Creek	CARE	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)		In-Situ TROLL 9500	Sontek Flowtracker ADV (cfs)	livestock	TSS, DO, temperature
4954812	Hall's Creek	CARE	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	Baski 8" cutthroat flume (cfs)	visitor use, livestock, oil and gas	temperature, dissolved oxygen
4994970	Hansen Cave	TICA	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	10	In-Situ TROLL 9500	N/A	unknown watershed, visitor use	
5995202	NPSETAL field blank		Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 4)	Filtered Metals (Type 3)	5		N/A	Field blank sample - Quality control	

SEUG sampling

NPSCAN RIVER

Storet #	Site Name	Park	Monthly parameters (Apr - Oct)	Visits in WY16	WQ Probe	Flow (units)	Threats	Priority parameters (from threats & reported results)

Storet #	Site Name	Park	Monthly parameters (Apr - Oct)	Visits in WY16	WQ Probe	Flow (units)	Threats	Priority parameters (from threats & reported results)
4930010	Green River above the confluence with the Colorado River	CANY - River	Water Temp, Discharge, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 3)	6-8	N/A	USGS gage (cfs)	Yes, widespread and varied upstream land use	total P, dissolved solids, total suspended solids, Al; potentially radionuclides & associates?
4930150	Green River at Mineral Bottom boat ramp	CANY - River	Water Temp, Discharge, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 3)	6-8	N/A	USGS gage (cfs)	Yes, widespread and varied upstream land use	total P, dissolved solids, total suspended solids, Al; potentially radionuclides & associates?
4952380	Colorado River below Big Drop 3 rapid	CANY - River	Water Temp, Discharge, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 3)	6-8	N/A	USGS gage (cfs)	Yes, widespread and varied upstream land use.	total P, dissolved solids, total suspended solids, Al, sulfates; potentially radionuclides & associates?
4952400	Colorado River above the confluence with the Green River	CANY - River	Water Temp, Discharge, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered	6-8	N/A	USGS gage (cfs)	Yes, widespread and varied upstream land use	total P, dissolved solids, total suspended solids, sulfates, Al, Se; potentially radionuclides &

Storet #	Site Name	Park	Monthly parameters (Apr - Oct)	Visits in WY16	WQ Probe	Flow (units)	Threats	Priority parameters (from threats & reported results)
			Metals (Type 3)					associates?
4956290	Colorado River at Potash boat ramp	CANY - River	Water Temp, Discharge, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 3)	6-8	N/A	USGS gage (cfs)	Yes, widespread and varied upstream land use	total P, dissolved solids, total suspended solids, sulfates, Se; potentially radionuclides & associates?
4956291	Colorado River at Potash boat ramp (duplicate)	CANY - River	Water Temp, Discharge, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 3)	3-4	N/A	USGS gage (cfs)	QAQC - duplicate	
5995202	NPSETAL field blank		Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9), Filtered Metals (Type 3)	3-4	N/A	N/A	QAQC - field blank	

NPSCAN

Storet #	Site Name	Park	Monthly parameters (10x/yr)	Quarterly parameters (3x/yr)	Visits in WY16	WQ Probe	Flow (units)	Threats	Priority parameters (from threats & reported results)
5995700	Square Tower Spring	HOVE	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9)	Filtered Metals (Type 3)	10-11	In-Situ Troll 9500	Pool level	Yes, oil and gas	DO, sulfates, total P, Al: metals? hydrocarbons?
5995710	Cajon Spring	HOVE	Bacteria, Core, Chemistry (Type 2), Non-Filtered Nutrients (Type 2), Filtered Nutrients (Type 9)	Filtered Metals (Type 3)	10-11	In-Situ TROLL 9500	Pool level	Yes, oil and gas	DO, sulfates, total P; metals? hydrocarbons?

Justification of Proposed Analytes

NCPN primarily monitors for water quality exceedances with the end goals of improving impaired waters and designating pristine waters as Outstanding National Resource Waters or its equivalent of Utah Category 1 waters. Analytes can also serve as leading indicators of specific disturbances of concern to national park managers. Calculating the cation-anion balance of each analysis is an important part of assuring the quality of laboratory results (QA/QC).

				Leading Indicators			
Proposed Analyte	**Analysis**	**Source water Standard exists**	**QA/QC**	**Oil and gas**	**Mining**	**Livestock**	**Soil disturbance**
Dissolved Oxygen	NCPN	x					
pH	NCPN	x		x			
Specific conductance	NCPN			x	x		
Turbidity	NCPN			x	x	x	x
Coliforms	NCPN	x				x	
T. Alk/CaCO3	DWQ			x			
TDS	DWQ	x		x	x		x
TSS	DWQ			x			
Nitrate + Nitrite as N	DWQ	x				x	
Phosphate, Tot. Dig. (as P)	DWQ	x					x
Sulfate	DWQ		x	x	x		
Ammonia as N	DWQ	x					
Chloride	DWQ		x	x			
Total Nitrogen	DWQ						
Boron	DWQ	x					
Calcium	DWQ		x				
Iron	DWQ		x				
Magnesium	DWQ		x				
Potassium	DWQ		x				
Sodium	DWQ		x	x			
Hardness (from D-CA and D-MG)	DWQ	*					
Arsenic	DWQ	x		x	x		
Barium	DWQ			x			
Cadmium	DWQ	x					
Chromium	DWQ	x					
Copper	DWQ	x					
Lead	DWQ	x					
Manganese	DWQ		x				

Nickel	DWQ	x		
Selenium	DWQ	x		x
Silver	DWQ	x		
Zinc	DWQ	x		
Aluminum	DWQ	x		
Mercury	DWQ	x		
Bicarbonate	DWQ		x	

* Hardness is needed to interpret metals concentrations for standards

Field Methods

Field and laboratory methods employ standard methods of water quality sampling and analysis. Standard operating procedures (SOPs) found in Thoma et al. 2009 cover field season preparations and equipment, sequence of events in the field, details of taking measurements (including field forms and electronic data entry), post-collection processing of samples, lab analysis, data verification and end-of-season procedures. A Quality Assurance Project Plan (QAPP) describes Quality Assurance (QA) and Quality Control (QC) Procedures (attached). A brief description of each SOP is given in Appendix A.

Applicable SOPs include:
SOP 1: Safety for Water Quality Monitoring
SOP 3: Staff Training, Utah Units
SOP 4: Site Selection
SOP 5: Preparations and Equipment Calibration, Handling and Storage
SOP 6: Cumulative Bias
SOP 7: Quality Assurance Project Plan (QAPP), All Units
SOP 8: Field Methods for Core Parameters, Turbidity and Discharge, All Units
SOP 10: Field Sample Collection and Processing for Water Chemistry, Nutrients and TSS, Utah Units
SOP 11: Fecal Indicator Bacteria, All Units

Laboratory Analysis

Laboratory analyses include a standard suite of water chemistry parameters and metals analyzed by the Utah Department of Water Quality state laboratory. The applicable SOP is SOP 13: Laboratory Analysis, Utah parks.

Quality Control

Quality control is taken at several steps during sample collection and processing. Prior to sampling all equipment is calibrated using known standards in the office and then at each field site. Readings are compared to acceptable measurement ranges. Samples are collected using standard clean procedures. Holding times and requirements are followed. Duplicate samples and field blanks are collected at 10% of field samples for analysis by the state laboratory.

Data Archiving, Analysis, and Reporting

Samples are collected by NPS staff, and sent to the State Water Quality Laboratory for Analysis. Field measurements and notes are electronically transferred to the state using their template described in SOP 10 section 8.4. NPS data are stored in a relational database called NPStoret, and data from the state laboratory are separated from non-NPS data using the STORET Report function and electronically transferred to WRD-STORET on an annual basis by the NCPN hydrologist. Raw hardcopy and electronic

field forms are archived on the NCPN server and an off-site facility. Following QA/QC procedures, a certified copy of the database is also archived at the end of each water year.

NCPN summarizes data annually in water quality briefs that note exceedances at individual sites. Biannual reports detail exceedances and document potential sources for exceedances. A trend report every five years investigates trends in parameters through the use of graphical techniques and nonparametric analyses such as the seasonal Kendall tau test.

Schedule

NPSETAL sites are sampled monthly from October through September for a total of ten samples submitted to the state laboratory. Weather and access conditions preclude sampling throughout the winter, so only one sample is submitted during December, January and February.

NPSCAN sites are sampled monthly at least 10 times per water year from October through September.

NPSCAN RIVER sites are sampled monthly 6-8 times per year from April through October.

Appendix A. NCPN Standard Operating Procedures (SOP) for Water Quality Monitoring

SOP #	Title	Description
SOP 1	Safety	Each NPS employee performing field inventory and monitoring activities in the NCPN develops, in conjunction with their supervisor (and, if appropriate, other knowledgeable persons), a Job Hazard Analysis of the task to be performed. This is consistent with Director's Order #50B and Reference Manual #50B, Occupational Safety and Health Program. The Job Hazard Analysis will identify real and potential hazards associated with inventory and monitoring activities, and develop approaches that minimize or eliminate the hazards.
SOP 2	Staff Training, Colorado Units	Annual training will be provided by NPS staff that has attended USGS training in water quality field techniques. As a partner, USGS will provide quality reviews of NPS field and laboratory techniques.
SOP 3	Staff Training, Utah Units	Annual training is conducted by the state and required for all field staff. If staff is available, back-up staff will also attend this training. If new staff come on board outside of the regular training season (spring) arrangements will be made with UDEQ for a special training session.
SOP 4	Sample Site Selection and Documentation	A description of the criteria used to select sample sites. A process for documenting each site as part of the permanent record. In addition to location information, this will include access information such as road conditions and land ownership, known hazards, park contacts, special equipment requirements, the source of streamflow data, and parameters and parameter groups to be measured.
SOP 5	Preparations and Equipment Calibration, Handling, & Storage, All Units	This SOP will follow guidelines provided by the manufacturers (e.g., Oakton, In-SITU, Inc., Marsh-McBirney, Rickly Hydrologic, etc.) for equipment operation and maintenance as well as common sense. This includes calibration methods and frequency, cleaning, changing pH electrodes, and D.O. membranes. An equipment log will be established for each major piece of equipment where calibrations, servicing, problems and other notable events are recorded. In addition, a field equipment checklist is included in the protocol. This lists all required and optional equipment and supplies to be carried with the field crew (or in the field vehicle) at all times. The checklist is provided for review before leaving the base park/office for each sampling event.
SOP 6	Quality Assurance Project Plan (QAPP), all Units	Following guidance from Irwin (2004), the QAPP or QC SOP includes 1) QC objectives for measurement certainty (detection limits such as MDL (method detection limit) and ML (minimum quantitation limit), 2)QC objectives for measurement precision, 3) QC objectives for measurement systematic error (bias as percent recovery), 4)QC objectives for data completeness (including adequacy of planned sample sizes and statistical power), and 5) QC objectives for blank controls for lab measurements. Individual SOPs for parameters also include discussion related to data comparability and selection of laboratories and protocols. SOP is detailed (e.g., indicating how many duplicate samples will be collected for QC) so that other agencies can determine whether they can utilize NCPN data in conjunction with their own data.

SOP 8	Field Methods for Core Parameters, Turbidity and Discharge, All Units	This focuses primarily on the use of multiparameter probes for measuring basic physical and chemical characteristics of the water. 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. For moderate to large streams, the preferred method is to use a nearby stream gage as a source of flow data. Colorado - Specifically, the Hydrolab Minisonde 4a with a Surveyor 4 display will be used for determining dissolved oxygen concentration and percent saturation, specific conductance, pH and temperature in CO units. Details of this field SOP will focus on the actual in-situ measurement (e.g., location of probe within sample site, location of probe in water column, proximity to streambank, differences in measurement techniques in pools versus riffles.) Utah - Same except for use of TROLL 9000/9500 with a Rugged Reader display. The Hach 2100 P Turbidimeter will be used to measure turbidity.
SOP 9	Field Sample Collection and Sample Processing for Water Chemistry and Nutrients, Colorado Units	This field SOP focuses on the actual in-situ collection of samples (e.g., clean and sterile technique to avoid contaminating a sample, location of sample in the water column, proximity to streambank, and the use of composite samples). Details of sample bottle labeling, storage, and transport to laboratories (including chain of custody forms) are discussed. Laboratory methods are also discussed.
SOP 10	Field Sample Collection and Sample Processing for Water Chemistry, Nutrients and TSS, Utah Units	Similar to SOP 9, except specific to coordination with the Utah State Laboratory and UDWQ protocols.
SOP 11	Field and Laboratory Methods for Bacteria, All Units	Sampling frequency is at a screening level (monthly) as opposed to an enforcement level, such as for swimming beaches (5 samples/month).
SOP 12	Laboratory Analysis, Colorado Parks	Describes USGS National Water Quality Laboratory protocols. Lists EPA Method number and reporting limit for each parameter.
SOP 13	Laboratory Analysis, Utah Parks	Describes UDEQ laboratory protocols. Lists EPA Method number and reporting limit for each parameter.
SOP 14	Data Management	Discusses the handling of data from multiple sources and repositories of data. Includes discussion of NPS-WRD requirement for use of WRD-STORET and cooperators requirement for storage in STORET (Utah) and NWIS (USGS).
SOP 15	Data Analysis and Reporting	Describes techniques for annual reporting of results including notable hydrologic events, exceedances, and recommendations on modifications to the sampling Network or Protocols.

SOP 16	Revising the Protocol Narrative and SOPs	Describes the procedure for updating this protocol narrative and the SOP's to maintain a relevant reference source that serves to maintain high integrity in data collection and archive. Older versions of SOP's are kept on file so that in the future changes in the program can be tracked.
SOP 17	Annual Project Review	An annual review of procedures and needs. Will provide a schedule and list of what will be reviewed, including: Annual maintenance and upkeep Equipment maintenance Supplies and consumables Review safety contact numbers Review and update field folders Update field forms Equipment list / ownership / location / loan

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.

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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).

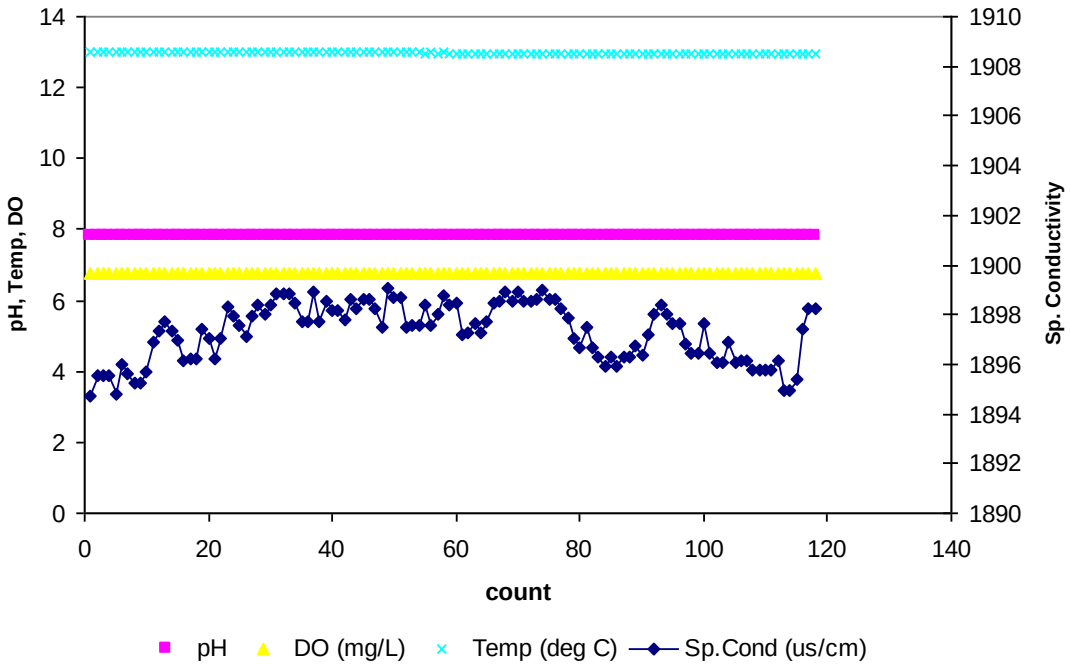


Figure 1. Example of base flow over-sampling to determine degree of stream mixing on Sheep Creek, Bryce Canyon National Park, UT. Sheep Creek has a high gradient and base flow less than 1.0 ft^3/s.

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.

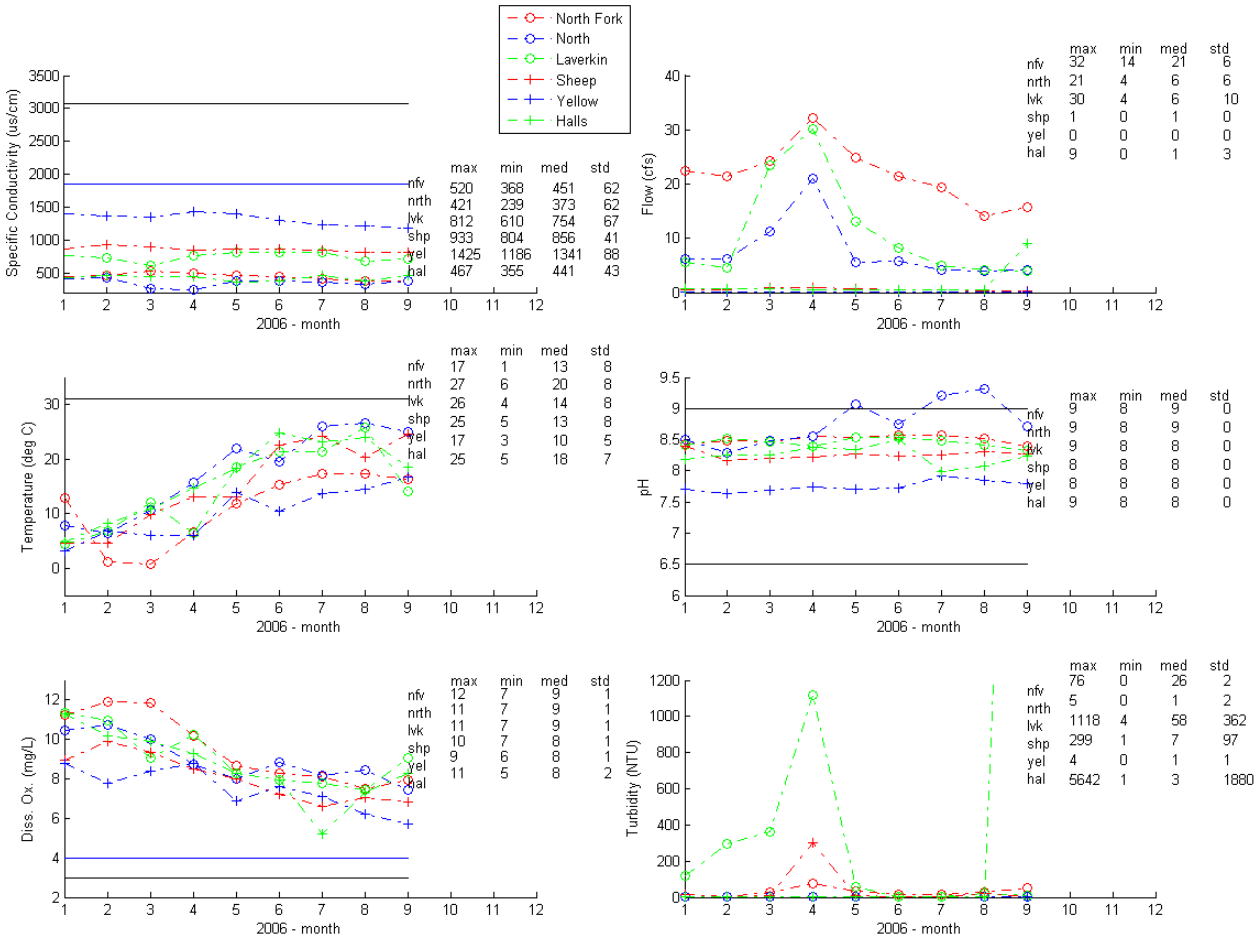
DISCHARGE MEASUREMENTS
Flow is measured with either the 1or 8 inch Baski flume, or V-notch weir. High-water Flow Measurement has not been made at this site, but the maximum stage based on high water marks could be waded and measured with a pygmy meter. Discharge will be estimated if too high for wading.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

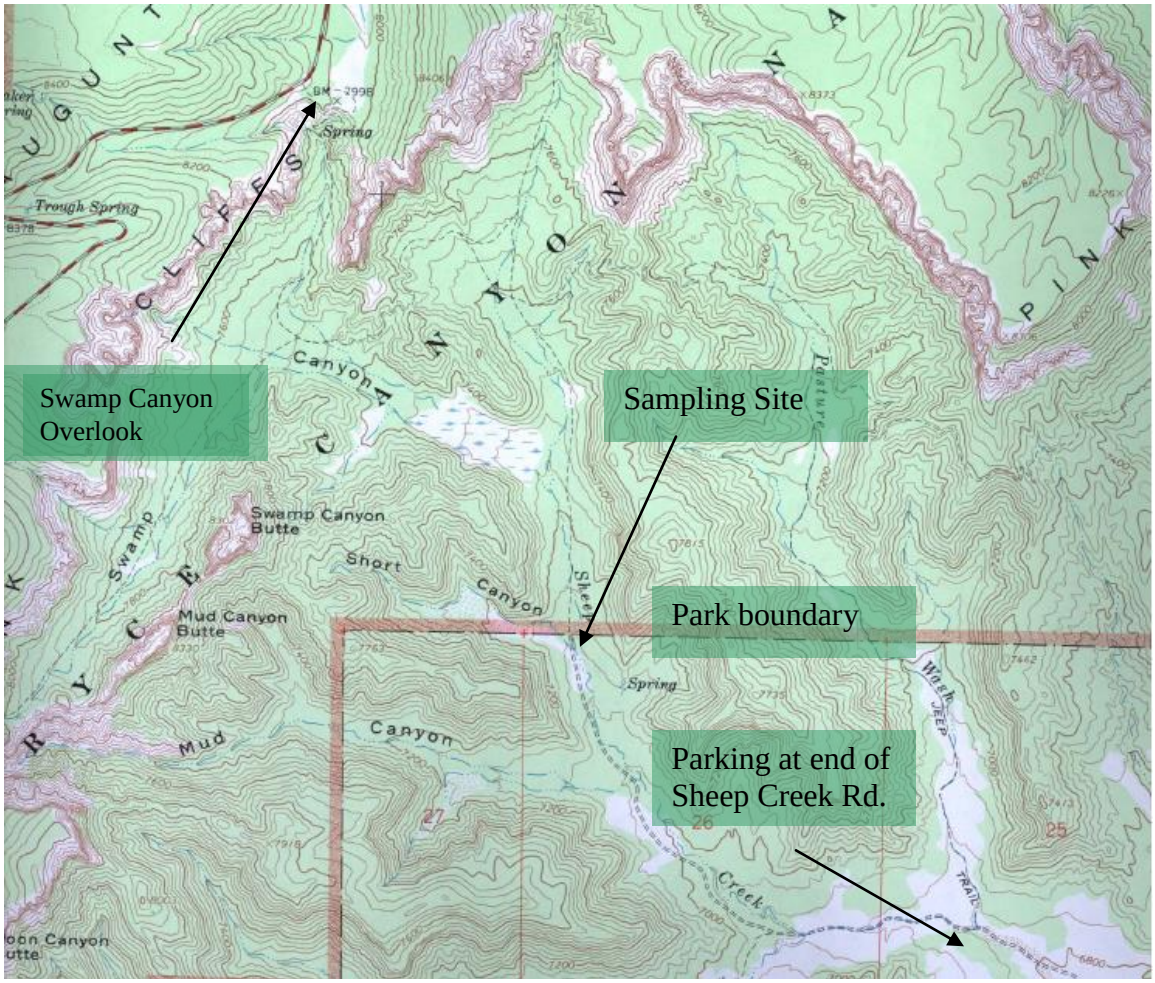
General Access – The 2.5 miles of dirt road from State route 9 is often rough and rutted. A high clearance vehicle is needed.

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.

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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 1.04

Revision History Log

Previous Version #	Revision date	Author	Change(s) made	Reason for change	New Version #
1.00	2/4/2008	D. Thoma	Add quick start guide to premob, update UT equip list	Make premob faster	1.01
1.01	3/16/2010	C. Hackbarth	Delete obsolete information, update methods for RDO sensor, grammar	Update to current methods	1.02
1.02	12/5/2012	C. Hackbarth	Added info for FlowTracker	New instrument for measuring	1.03
1.03	4/2016	C. Hackbarth	Updated all sections for clarity, deleted methods that are not in use	Update to current methods	1.04

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 multiparameter instrument and Rugged Reader handheld display in the measurement of core field parameters.

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1. Field Trip Preparations

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

1.1 Site visit preparation checklist, Utah

The following checklist contains an outline of steps to prepare for the monthly water quality monitoring field run.

Site Visit Preparation Checklist	
✓	Activity
2-3 weeks before trip	
	Sign out GOV vehicle
	Check quantity and expiration dates of calibration solutions (Conductivity, pH, turbidity, sodium sulfite for DO calibration), order more if necessary
	Check supply of filters and pump tubing, order more if necessary
	Check supply of sample bottles from UTDWQ, contact Utah Public Health Laboratory for additional bottles at 801-965-2400
1-2 weeks before trip	
	Create Travel Authorization if trip is more than a day
	Email relevant park staff schedule for site visit dates
	Email relevant park staff about campsite reservations or park housing
	Email relevant park staff to find interested individuals in hiking to water quality sites
1-7 days before trip	
	Check that equipment batteries are sufficiently charged for Rugged Reader, GPS, camera, UV lamp
	Make sure all data from previous sampling trips have been downloaded, saved, and then deleted from Rugged Reader, FlowTracker, and camera
	Conduct premobilization equipment calibration and error checks
	Locate all necessary equipment and load into GOV vehicle (see **Field Equipment Checklist** below)
	Print out site visit field forms for each site
	Fill out backcountry plan and email or fax copy to GLCA dispatch on day of trip

1.2 Field equipment checklist, Utah

The following checklist contains the equipment and supplies needed for a water quality monitoring field run.

Field Equipment Checklist	
✓	Item
	Troll with calibrated sensors and calibration cup attached
	Stainless steel sensor cover
	Sensor removal tool and grease
	Spare sensors (SpC, DO, pH)
	Rugged Reader with charged battery, spare battery, and car charger
	Troll Rugged Cable for connecting with Rugged Reader
	2 D-cell spare batteries
	Field thermometer
	Calibration solutions in mesh bag
	pH 7 buffer
	pH 10 buffer
	1413 Conductivity standard
	Sample bottles (quantity dependent on site visits)
	Total Chemistry bottle
	Total Nutrients bottle
	Dissolved Nutrients bottle
	Dissolved Metals bottle
	1 liter carry bottle for filtered samples
	Bacterial bottle
	Turbidity vial
	FlowTracker
	Mounting bracket
	Screwdriver
	8 AA spare batteries
	Wading rod
	Survey tape and clamps
	Baski Flume
	Flume spare parts bag (screwdriver, screws)
	V-notch weir
	Flume/Weir supplies
	Level
	Small shovel
	Bacteria supplies
	Quanti-Tray sealer with power cord
	Incubator and power cords
	Quanti-Trays
	Colilert
	Bacteria bottles

	Storage container with ice for field transport
	Rubber gloves
	Hand sanitizer
	Turbidimeter
	Turbidity vials
	Standard gelex vials for calibration checks
	4 AA spare batteries
	Geo Pump for sample filtration
	Tubing
	0.45 um disposable filters
	Rubber gloves
	Cooler(s)
	Ice
	Bubble wrap
	Ziplock for lab sheets
	Field Laptop
	USB connection cables for Rugged Reader, FlowTracker, camera
	Software loaded including Win-Situ and SonTek FlowTracker
	Digital camera with spare battery
	GPS unit with spare battery
	Clipboard
	Site visit field forms and pencil
	Permanent marker for labelling sample bottles
	Field binder
	Site descriptions and maps
	Safety information and emergency contacts
	Equipment manuals
	Satellite phone
	SPOT personal beacon
	Waders
	Ditch boots
	Neoprene gloves
	Personal Flotation Device (PFD)

1.3 Hiking Checklist, Utah

The following checklist includes items needed at water quality monitoring sites. Some of the items are specific to sites such as flow measurement equipment. When hiking to a sampling site it is important to ensure all the equipment necessary to complete the job makes it to the site.

Hiking Checklist	
✓	Item
	Troll with calibrated sensors and calibration cup
	Stainless steel sensor cover
	Rugged Reader with charged battery
	Troll Rugged Cable for connecting with Rugged Reader
	2 D cell spare batteries
	Field thermometer
	Calibration solutions in mesh bag
	pH 7 buffer
	pH 10 buffer
	1413 Conductivity standard
	Sample bottles
	Total Chemistry bottle
	Total Nutrients bottle
	Dissolved Nutrients bottle (leave at car)
	Dissolved Metals bottle (leave at car)
	1 liter carry bottle
	Bacterial bottle
	Bacterial storage container with ice for field transport
	Turbidity vial
	Flow Measurement – FlowTracker
	FlowTracker in carry bag
	Wading rod
	Mounting bracket
	Screwdriver
	8 AA spare batteries
	Survey tape and clamps
	Flow Measurement – Flume
	Baski Flume
	Level
	Small shovel
	Spare parts bag (screwdriver, screws)
	Flow Measurement – Weir
	V-notch weir
	Level
	Small shovel
	Digital camera and spare battery
	GPS unit (when necessary) and spare battery

	Clipboard
	Site visit field forms and pencil
	Permanent marker for labeling sample bottles
	Satellite phone and/or SPOT personal beacon
	First Aid Kit
	Clothing and personal gear:
	Winter (cold water) sampling: chest waders or ditch boots, neoprene gloves, snowshoes
	Leatherman
	Headlamp
	Sunglasses
	Sun hat
	Sunscreen
	Food
	Water
	Water filtering device (depending on length of hike)
	PFD (when necessary)

1.4 Vehicle equipment checklist

Check vehicle for safety equipment and supplies.

Vehicle Equipment Checklist	
✓	Item
	Jack
	Spare tire
	Shovel
	Jumper cables
	Tire chains
	Flashlight
	Warning cones
	First aid kit

1.5 Field trip preparations checklist, Colorado

Hydrologists in Colorado network parks follow the checklist outlined in the USGS National Field Manual, Chapter A1, Section 1.1.1, pages 12-14, http://water.usgs.gov/owq/FieldManual/. Items specific to bacteria sample collection are listed in SOP 11, Section 3.1.

2. Equipment log

An equipment log is stored electronically in the premobilization tab of the Field_forms spreadsheet. Equipment information includes serial number, date and description of problems, date when maintenance was performed and by whom, and date and results from calibration.

3. Premobilization instrument checks

3.1 In-Situ Troll multiparameter instrument calibration, Utah

In Utah park units, a premobilization instrument check is designed to minimize trouble in the field when using a multiparameter instrument such as the In-Situ Troll. Calibrations are performed using high quality standards for conductivity, pH, and optical dissolved oxygen. The instrument is checked for accuracy against solutions of known conductivity, pH, and dissolved oxygen. Temperature is checked for accuracy against a NIST-traceable liquid-in-glass or digital thermometer. Pre-calibration readings can indicate the tendency and degree of a particular instrument or sensor to go out of calibration between regular field use. These readings can be instructive of the remaining sensor life and if short term storage measures are adequate.

Calibration data are recorded in the premobilization page of the Field_forms spreadsheet and automatically compared against allowable ranges to determine if the calibration and accuracy are acceptable. If the calibration and accuracy check do not meet the NPS-WRD recommended guidelines (Table 1), the user must consult the appropriate chapter in the Troll multiparameter instrument user manual (Appendix C) and take action to fix the problem. The premobilization calibration procedure for the In-Situ Troll is outlined in Appendix A.

3.1.1 Dissolved oxygen calibration

A 2-point calibration for zero DO and 100% DO is performed in the lab. Refer to Appendix A for the calibration procedure.

3.1.2 Conductivity calibration

A 1-point calibration is performed using 1413 µS/cm standard with error checks at 147 µS/cm and 12900 µS/cm. Refer to Appendix A for the calibration procedure.

3.1.3 pH calibration

A 2-point calibration is performed (typically pH 7 and pH 10) meant to bracket typical source water pH. At times, a 3-point calibration is performed when a 2-point calibration is out of range or after sensor maintenance. Refer to Appendix A for the calibration procedure.

3.1.4 Temperature check

A temperature check is made in the lab prior to field mobilization by comparing the Troll thermistor reading to that of a NIST-certified liquid thermometer or NIST-traceable thermometer.

3.2 Turbidity calibration, Utah

Turbidity is measured at sites in Capitol Reef, Bryce Canyon, and Zion with a Hach 2100P Turbidimeter. The turbidimeter 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 spreadsheet. Refer to the calibration procedure outlined in the Hach Turbidimeter user manual in Appendix F.

3.3 Premobilization, Colorado

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

4. Data quality objective for core parameters

Quantitative ranges in allowable variation have been established to ensure data collected using multiparameter instruments are representative (**Error! Reference source not found.**). 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 insufficient time for probe equilibration with ambient water. The allowable ranges are drawn from USGS National Field Manual and NPS-WRD guidance. These are the values against which premobilization error checks are compared.

Table 1. Recommended Multiparameter Instrument Post-Calibration Check Error Limits (NPS Core Parameters). These are also the Data Quality Objectives (DQOs) discussed in the Quality Assurance standard operating procedure (SOP 7).

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

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

5. Field calibrations

5.1 In-Situ Troll multiparameter instrument field calibration, Utah

Hydrologists in the Utah park units of the NCPN use In-Situ Troll multiparameter instruments and follow the user manual for operation and calibration (Appendix C). Field calibrations of pH and conductivity are performed with standards in temperature equilibrium with ambient surface water before taking measurements at each site. If surface water temperature is < 6°C or >40°C, perform calibration indoors prior to visit. Another option is to calibrate with standards at air temperature at site as long as the air temperature is in range of 6°C to 40°C. Optical DO sensors do not require field calibration.

5.1.1 Conductivity calibration in field

In the field, a 1-point calibration is performed with a standard of 1413 µS/cm. Be sure to rinse sensors and calibration cup with standard before calibrating. Record calibration data on site visit field form. Refer to SOP 8 for the detailed field calibration procedure.

5.1.2 pH calibration in field

A 2-point calibration is performed in the field (typically pH 7 and 10) meant to bracket typical water body pH. Temperature adjusted values of pH buffers need to be determined before performing calibration. First, rinse sensors and calibration cup with first pH buffer. After filling calibration cup with first pH buffer, take a temperature reading. Use Table 2 “pH Buffer Values at Various Temperatures” to determine the pH values to input in the calibration screen. Assume that both pH buffers will be similar in temperature. Rinse with second pH buffer when prompted and proceed with calibration. Record calibration data on site visit field form. Refer to SOP 8 for the detailed field calibration procedure.

Table 2. pH Buffer Values at Various Temperatures. The values listed are specific to Aqua Solutions pH buffers.

Temp (°C)	pH 4 buffer	pH 7 buffer	pH 10 buffer	Temp (°C)	pH 4 buffer	pH 7 buffer	pH 10 buffer
6	4.00	7.09	10.25	24	4.01	7.01	10.01
7	4.00	7.09	10.23	25	4.01	7.00	10.00
8	4.00	7.08	10.22	26	4.01	7.00	9.99
9	4.00	7.08	10.20	27	4.01	7.00	9.98
10	4.00	7.07	10.19	28	4.01	6.99	9.96
11	4.00	7.06	10.17	29	4.01	6.99	9.95
12	4.00	7.06	10.16	30	4.02	6.99	9.94
13	4.00	7.05	10.15	31	4.02	6.98	9.93
14	4.00	7.05	10.13	32	4.02	6.98	9.92
15	4.00	7.04	10.12	33	4.02	6.98	9.91
16	4.00	7.04	10.11	34	4.02	6.98	9.90
17	4.00	7.03	10.09	35	4.02	6.98	9.89
18	4.00	7.03	10.08	36	4.03	6.97	9.88
19	4.00	7.03	10.07	37	4.03	6.97	9.87
20	4.00	7.02	10.06	38	4.03	6.97	9.86
21	4.00	7.02	10.04	39	4.03	6.97	9.86
22	4.01	7.01	10.03	40	4.03	6.97	9.85
23	4.01	7.01	10.02				

5.2 Hydrolab multiparameter instrument field calibration, Colorado

Hydrologists in Colorado park units of the NCPN use Hydrolab multiparameter instruments and follow the Hydrolab user manual (Appendix D) and Hydrolab display unit manual (Appendix E) for operation and the USGS National Field Manual (http://water.usgs.gov/owq/FieldManual/) for calibration procedures.

5.2.1 Conductivity calibration

Conductivity calibration is performed following the guidelines in the USGS National Field Manual, Chapter 6, Section 6.3. The only exception being that the first calibration point is performed using air (0 µs/cm) instead of a low standard.

5.2.2 pH calibration

Calibration of pH is performed following the guidelines in the USGS National Field Manual, Chapter 6, Section 6.4.

5.2.3 Dissolved oxygen calibration

Dissolved oxygen calibration is performed following the guidelines in the USGS National Field Manual, Chapter 6, Section 6.2.

6. Equipment maintenance and storage

6.1 In-Situ Troll multiparameter instrument, Utah

After calibration and during a field run, moisten a sponge or add a little water to the cal cup to maintain a moist environment for the sensors. Never use DI water. Attach the calibration cup to the probe body with all sensors installed and store the instrument in an upright position whenever possible. For long term storage between field runs some sensors will need to be removed from the instrument body.

- Conductivity sensor: store dry, can remain attached or can be removed.
- Optical DO sensor: store dry, can remain attached or can be removed.
- pH sensor: Short term storage (several days) – store attached with tap water in calibration cup. Long term storage (several weeks) - Remove the sensor and store it in the electrode storage bottle with 10-20 mL of storage solution. Tighten the cap to prevent drying. Prior to use, condition the sensor by rinsing with deionized or tap water and soaking for 15 minutes.

6.2 Hydrolab multiparameter instrument, Colorado

Refer to the Hydrolab user manual for instrument maintenance and storage (Appendix D).

7. Troubleshooting

7.1 In-Situ Troll multiparameter instrument, Utah

7.1.1 Conductivity sensor

The conductivity sensor rarely has any issues. Be sure to never use partially frozen conductivity solution. Freezing will unequally distribute the salts in solution and will not have the conductivity stated on the bottle.

7.1.2 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.

7.1.3 Optical DO sensor

If any problems arise with the optical DO sensor, first check to make sure that the DO cap is not damaged. Replace the cap if necessary.

7.2 Hydrolab multiparameter instrument, Colorado

Refer to the Hydrolab user manual for troubleshooting (Appendix D).

8. Software updates

Latest software versions of Win-Situ 4 for the In-Situ Troll and Pocket-Situ 4 for the Rugged Reader can be downloaded for free from the In-Situ website after the original software has been installed. Check for and install necessary updates at least once per year at https://in-situ.com/support/type/software/.

9. Post sampling activities, Utah

1. Enter data into monthly Field_forms.xls and check QA column for completeness.
2. Download data from FlowTracker and Rugged Reader and save files to appropriate folders on network drive. Refer to SOP 8 for downloading instructions.
3. Prepare samples for shipment to laboratory with plenty of ice and include lab sheets for each site sampled in cooler.
4. Download photos from camera and save images to appropriate photos folder on network drive.
5. File hard copies of completed Field_forms.xls pages.
6. Make sure that all equipment is clean and properly stored.
7. Prepare a list of items to be ordered.

10. Post sampling activities, Colorado

1. Update PCCF forms for each site visit.
2. Print archival copies of completed forms.
3. Prepare samples for shipping as per Wilde et al. (2004) in Chapter 5, Section 5 in USGS National Field Manual at http://water.usgs.gov/owq/FieldManual/

11. 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.)

Appendix A: Premobilization calibration procedure, Utah

The purpose of a premobilization procedure is to ensure the In-Situ Troll multiparameter instrument is calibrated, operating within acceptance criteria, and that this is documented prior to field work. The procedure involves the following:

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

1.1 Premobilization calibration procedure basic steps

1. Fill bubbler with tap water or deionized (DI) water. Allow to reach room temperature.
2. Turn on bubbler, place Troll sensors in bubbler, wait 5 minutes for stabilization, then take 7 snapshot pre-calibration readings.
3. Record NIST thermometer temperature of bubbler water.
4. Take SC (specific conductance) pre-calibration readings in 147, 1413, 12900 µS/cm conductivity standards.
5. Calibrate SC in 1413 µS/cm conductivity standard.
6. Take post-calibration SC readings in 147, 1413, 12900 µS/cm standards.
7. If applicable, take SC reading of 1413 field calibration standard.
8. Take pH pre-calibration readings in 4, 7, 10 buffers.
9. Calibrate pH in 7, 10 buffers.
10. Take post-calibration pH readings in 4, 7, 10 buffers.
11. If applicable, take readings for pH 4 and pH 7 field calibration standards.
12. Make sodium sulfite solution, if necessary. Take pre-calibration 0% DO reading.
13. Take pre-calibration 100% DO reading.
14. Calibrate 100% DO (and 0% DO when necessary).
15. Take post-calibration 100% DO reading.
16. Take post-calibration 0% DO reading.
17. Place sensors in bubbler, wait 5 minutes for stabilization, then take 7 snapshot post-calibration readings.
18. Record NIST thermometer temperature of bubbler water.

1.2 Premobilization calibration procedure detailed steps

1.2.1 Before beginning the premobilization procedure

1. Print out the premobilization sheet or enter directly into the Field_forms spreadsheet. This data sheet is located on the premob tab of the Field_forms xls file. Enter data into the shaded cells. The other cells contain formulas that will be filled in automatically. Refer to Appendix B for an example premobilization data sheet.
2. Locate equipment that will be needed (see list below).

3. Make sure the batteries for the Troll and Rugged Reader have sufficient power.
4. Check to see if any of the sensors need to be replaced.

Equipment List

- Premobilization sheet
- Troll multiparameter instrument with sensors (pH, conductivity, dissolved oxygen)
- Rugged Reader
- Cable to attach Troll to Rugged Reader
- Bubbler cup with battery-powered air pump
- Conductivity standards at room temperature (147, 1413, 12900 µS/cm)
- pH buffers at room temperature: pH 4, pH 7, pH 10
- 0% Dissolved oxygen solution, Sodium sulfite
- Cal Cup
- Lab stand
- NIST thermometer
- Field thermometer (if applicable)
- Deionized water and/or tap water

1.2.2 Setting up the Troll multiparameter instrument and Rugged Reader

1. Carefully insert the sensors in the correct ports of the Troll.
2. Connect the Troll to the Rugged Reader with the associated cable.
3. Turn on the Rugged Reader.
4. Select "Start" in the upper left of the screen and a drop-down menu will appear.
5. From the menu, select "Pocket-Situ 4" and wait for software to open.
6. Double-click (tap twice) on the "COM1-19200" option.
7. After a connection has been established, double-click on the "Parameters" option that will appear.
8. Select the desired parameter and the sensor information and readings will appear on the screen below the menu portion. Notice the "Read" and "Calibrate" commands at the bottom of the screen.
9. To set up or change unit preferences in Rugged Reader, tap the "Home" site in the Navigation tree, then tap "Setup" in the bar at the bottom of the screen. Select the units tab, then set the following:
   Barometric Pressure = mm Hg
   Conductivity = microSiemens/cm Specific Conductivity
   Dissolved Oxygen = milligrams/L
   Temperature = Celsius

1.2.3 Premobilization pre-calibration profiler mode readings

1. Connect Troll to Rugged Reader and open Pocket-Situ 4. Establish connection to Troll.
2. Fill bubbler ¾ full with DI or tap water and allow to equilibrate to room temperature.
3. Place Troll sensors in bubbler and turn on bubbler.
4. Place the NIST thermometer in bubbler. If using a separate field thermometer, place in bubbler.

5. Wait 5 minutes for the Troll and thermometer(s) to stabilize.
6. Record the NIST thermometer temperature. The acceptable range for the Troll thermistor temperature is ±0.3°C of NIST temperature. The acceptable range for the field thermometer temperature is ±1°C of NIST temperature.
7. In Pocket-Situ select "Parameters" and the "Profiler" tab should appear in the bar at the bottom of the screen.
8. Select "Profiler" to open a window that allows naming a file.
9. Select the keyboard icon to allow typing and enter the premobilization profiler file name according to the following convention, "precal_mmddyy" where mm is the 2-digit month, dd is the 2-digit year, and yy is the 2-digit year.
10. Record 7 profiler readings by tapping "snapshot" 7 times in succession.
11. Select "Close" to store the data and exit profiler mode.
12. This file will be downloaded and pasted into Field_forms.xls.

1.2.4 Calibration of specific conductivity (SC)

As a general rule, start with lowest standard and work up in conductivity when taking readings or when calibrating to minimize carry-over error. Also, rinse with DI or tap water, and then rinse 3 times with the next standard 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.

1. Conductivity pre-calibration readings for three standards (147, 1413 and 12900 µS/cm):
   a. Rinse sensors and cal cup 3 times with lowest conductivity standard (here 147 µS/cm).
   b. Fill cal cup to fill line with 147 µS/cm standard and attach to instrument. Make sure both the conductivity sensor and the temperature sensor are adequately immersed in the calibration standard. Gently tap the cal cup to release any bubbles that have collected on the conductivity sensor.
   c. On Rugged Reader, choose "Parameters" then "Conductivity". Select "Read" and record the conductivity reading on premobilization sheet.
   d. Before reading the next higher conductivity standard, rinse the sensors well. First, fill the cal cup with DI or tap water, attach to the instrument, and rotate back and forth to ensure a complete rinse. Then gently shake off the excess water. Rinse sensor 3 times with a small amount of the standard that will be tested next.
   e. Record the conductivity on the premobilization sheet for the next two standards, making sure to go in ascending order of conductivity standard.

2. Conductivity calibration: 1-point calibration using 1413 µS/cm standard
   a. Rinse sensor and cal cup 3 times with 1413 µS/cm standard.
   b. Fill cal cup to fill line with 1413 µS/cm standard and attach it to the instrument.
   c. From the "Parameters" menu, select "Conductivity" then "Calibrate" at the bottom of the screen. On the next screen (where 1413 is set as the default), select "Next" then "Run". The sensor will begin calibrating
   d. The screen first reads "Unstable", then "Nominal", then "Stable". When it reads "Stable", the final calibration screen will appear with a K-cell value.
   e. Check the K-cell value, the acceptable range is 0.30 to 0.39, select "Finish" to finalize calibration. If K-cell value is outside of range, check these conditions and recalibrate:

(1) Ensure that the temperature sensor is fully submerged during calibration
(2) Ensure the vent ports of the conductivity sensor are submerged and no bubbles are present. Tap cal cup gently to remove bubbles.

3. Conductivity post-calibration readings and calibration error check
   a. Take post-calibration readings of the conductivity standards (147, 1413 and 12900 µS/cm). The 1413 µS/cm standard can be read immediately following the calibration step. Then rinse well with water followed by a triple rinse with next standard to be tested. Proceed with 147 µS/cm standard, then 12900 µS/cm standard.
   b. Record post-calibration readings on the premobilization sheet.
   c. Acceptable range for post-calibration conductivity values:
   147 standard: 142 to 152 µS/cm (±5 µS/cm)
   1413 standard: 1408 to 1418 µS/cm (±5 µS/cm)
   12900 standard: 12513 to 13287 µS/cm (3% error)
   If any calibration standard readings are out of range, check values again using fresh solution. Sensor may need to be replaced if readings are still out of range.

4. Post-calibration reading of field conductivity standard
   a. Fill cal cup with 1413 µS/cm standard used in field.
   b. On the Rugged Reader, choose "Conductivity" then "Read". Record the measurement on the premobilization sheet.
   c. Acceptable range for 1413 µS/cm field conductivity standard is 1371 to 1455 µS/cm (3% error).

1.2.5 pH calibration

Start with the lowest pH buffer and work up in increasing pH when taking readings or when calibrating to minimize carry-over error. Also, rinse with DI or tap water, and then rinse 3 times with the next pH buffer in which the sensor will be submerged to eliminate potential for dilution or carry-over concentration effects.

1. pH pre-calibration readings for three pH buffers: pH 4, 7, 10
   a. Rinse 3 times with lowest pH (pH 4).
   b. Fill cal cup with pH 4 buffer.
   c. On the Rugged Reader, choose "Parameters" then "pH". Select "Read" then record the pH reading on the premobilization sheet.
   d. After proper rinsing, read pH of subsequent standards in ascending value of pH (pH 7 then pH 10) and record pH reading on the premobilization sheet.
   e. If pH 4 value is between 3.7 to 4.3 perform a 2-point calibration. If pH 4 value is outside this range, perform a 3-point calibration.

2. pH calibration: 2-point calibration with pH 4 and 7 or pH 7 and 10
   a. Choose solutions that bracket anticipated environmental waters. For neutral to acidic conditions use pH 4 and 7. For neutral to basic conditions use pH 7 and 10. These are room temperature calibrations.
   b. Rinse 3 times with lower pH buffer (here pH 4), fill cal cup to fill line and attach it to the instrument.

c. From the “Parameters” menu, select “pH” then “Calibrate”. On the next screen (where 7.00 and 10.01 are the default settings), select “Next” then “Run”. Wait for the screen to make the progression from “Unstable” to “Nominal” to “Stable”. Once the reading is stable the screen will display the next pH to be tested.
d. Rinse the sensors thoroughly with water and then 3 times with the next pH buffer (here pH 10) before loading the buffer in the cal cup. Select “Run” to begin stabilization for the second calibration point.
e. Once the reading is “Stable” a final screen will show the slope and offset.
f. Select “Finish” to complete the 2-point calibraiton.
g. Take a post-calibration reading of the current pH buffer (here pH 10) and record on the premobilization sheet.
h. Check slope and offset. Acceptable range for slope is -50 to -66 mV/pH. Acceptable range for offset is 350 to 450 mV. If slope or offset is outside of range, replace filling solution and/or junction, soak the sensor in tap water for 15 minutes and recalibrate.
i. Rinse the sensors and proceed to the post-calibration step.

3. pH calibration: 3-point calibration
   a. Rinse 3 times with pH 4 buffer, fill cal cup with pH 4 buffer and attach it to the instrument.
   b. From the “Parameters” menu, select “pH”, then “Calibrate”. On the next screen, choose 3 for number of calibration points where cal point 1 = 4.01, cal point 2 = 7.00 and cal point 3 = 10.01. Select “Next” to continue.
   c. In the next screen, select “Run” to begin stabilization. Wait for the screen to make the progression from “Unstable” to “Nominal” to “Stable”. Once the first reading is stable the screen will display the next pH to be tested.
   d. Rinse the sensors thoroughly with water and then 3 times with pH 7 buffer before loading pH 7 buffer in the cal cup. Select “Run” to begin the stabilization of the second calibration point. After the second reading is stable the screen will display the next pH to be tested.
   e. Rinse the sensors thoroughly with water and then 3 times with pH 10 buffer before loading pH 10 buffer in the cal cup. Select “Run” to begin the stabilization of the final calibration point.
   f. Once the reading is “Stable” a final calibration screen will show the slopes and offsets.
   g. Select “Finish” to complete the 3-point calibration.
   h. Take a post-calibration reading of the current pH buffer (here pH 10) and record on the premobilization sheet.
   i. Check slope and offset. Acceptable range for slope is -50 to -66 mV/pH. Acceptable range for offset is 350 to 450 mV. If slope or offset is outside of range, replace filling solution and/or junction, soak the sensor in tap water for 15 minutes and recalibrate.
   j. Rinse the sensors and proceed to the post-calibration step.

4. pH post-calibration readings
   a. Take post-calibration readings of the pH buffers (pH 4, pH 7, pH 10). The pH 10 buffer can be read immediately following the calibration step. Then rinse well and proceed with pH 4 buffer.
   b. Record post-calibration readings on the premobilization sheet.

c. Acceptable range for post-calibration values at room temperature:
   pH 4: 3.7 to 4.3
   pH 7: 6.7 to 7.3
   pH 10: 9.7 to 10.3
   If any of the post-calibration readings are out of range, check values again using fresh solution. If values are still out of range, replace filling solution.

5. Post-calibration reading of field pH buffers
   a. Measure pH 7 and pH 10 buffers used in the field and record the readings on the premobilization sheet. Acceptable range for field pH buffers:
      pH 7: 6.7 to 7.3
      pH 10: 9.7 to 10.3

1.2.6 Calibration of dissolved oxygen

The calibration of dissolved oxygen (DO) depends on the type of sensor. The two types of sensors that can be used with a multi-parameter Troll are the Clark cell polarographic DO sensor and the optical DO sensor (also called "RDO" for rugged dissolved oxygen). This updated version only contains instructions for the optical DO sensor.

1. Preparation of Zero DO Solution
   a. Add 1 gram sodium sulfite to 1 liter of deionized water (0.008M solution).
   b. Invert the container several times to achieve dissolution of the solid/crystal reagents and allow solution to stabilize for a few minutes.

2. Optical Dissolved Oxygen pre-calibration readings
   a. 0% Oxygen Saturation
      i. Dry the sensors with a Kimwipe.
      ii. Fill the cal cup to the fill line with sodium sulfite and place the sensors into the cup ensuring the sensors are completely submerged.
      iii. Wait about 15 minutes for oxygen to be consumed, then select "Rugged Dissolved Oxygen" from the "Parameters" menu, then select "Read". Record the pre-calibration reading on the premobilization sheet.
      iv. Sodium sulfite consumes oxygen aggressively, be sure to rinse the sensors with tap water thoroughly to avoid cross-contamination.

   b. 100% Oxygen Saturation
      i. Fill the bubbler about ¾ full with room temperature tap water or DI water.
      ii. Turn on the bubbler.
      iii. Place the sensors into the top opening of the bubbler. Ensure the DO sensor and the temperature sensor are completely submerged and the DO sensor cap is not in the direct path of the bubbles.
      iv. Allow 10 minutes for bubbler water to achieve oxygen saturation, then select "Rugged Dissolved Oxygen" from the "Parameters" menu, then select "Read". Record the pre-calibration reading on the premobilization sheet.

3. Optical Dissolved Oxygen calibration

a. Start calibration immediately following 100% DO pre-calibration procedure. The sensors need to be in bubbler for at least 10 minutes before proceeding with calibration.
b. Select “Rugged Dissolved Oxygen” and then “Calibrate”.
c. Choose “Calibrate Only” then “Next>”.
d. Click “Run”. Wait for the status to read “Stable” for 100% oxygen saturation.
e. Select “Next>” to continue to the Zero Oxygen Calibration Point screen. If performing a 2-point calibration, continue to next step. If performing a 1-point calibration, select “Next” again to finalize the calibration.
f. If performing a 2-point calibration, dry the sensors with a Kimwipe and place sensors in the cal cup with sodium sulfite.
g. Allow up to 15 minutes for oxygen to be consumed. Click “Run” in the Zero Oxygen Point screen. If the Zero DO reading is > 1 mg/l after clicking “Run”, immediately select “Stop” to delay the calibration. Wait 5 minutes longer for oxygen to be consumed or add slightly more sodium sulfite to the solution and wait 5 more minutes, then select “Run” to restart the zero point calibration.
h. Once the status of the zero oxygen point reads “Stable”, the finalize calibration screen will automatically pull up, choose “Finish” to program the sensors with new calibration coefficients.
i. In the “Rugged Dissolved Oxygen” screen, check the slope and offset.
    i. A slope between 0.8 and 1.2 is acceptable. Offset acceptable range is -0.1 to 0.1 mg/L.
    ii. Values outside these ranges can indicate problems with the sensor or sensor cap.

4. Optical Dissolved Oxygen post-calibration readings
    a. Take readings for 100% oxygen and 0% oxygen after calibration. Wait at least 5 minutes before each measurement to ensure stabilization. Rinse the sensors very thoroughly after using sodium sulfite solution to avoid cross-contamination.
    b. Record the post-calibration readings on the premobilization sheet.
    c. Go to the NOAA weather site to find the barometric pressure for the area. Record the value on the premobilization sheet.

5. Calibration Error Check
    a. Acceptance criteria for 100% DO is ±0.4 mg/L from **stimulus** value (and between 95% and 105% saturation value)
    Stimulus = (oxygen saturation calibration point) x (slope).
    To find values in Rugged Reader, scroll down while in the Rugged Dissolved Oxygen parameter screen.
    b. Acceptance criteria for 0% DO is <0.2 mg/L. Note that this value will read negative at times and this is acceptable.
    c. NOTE: If any values are out of range, check these conditions and recalibrate:
    (1) If using a bubbler, ensure that bubbles have not accumulated on the RDO sensor tip
    (2) Bubbler has not been on for enough time to achieve saturation.
    (3) Bubbler has been on for too long and conditions are now oversaturated.
    (4) If using sodium sulfite solution for 0% DO calibration and value is out of range, the solution might be bad.

*If values are still out of range after recalibrating, then the RDO cap or RDO sensor may need to be replaced.

1.2.7 Premobilization post-calibration profiler mode readings

1. Place Troll sensors in bubbler.
2. Turn on bubbler
3. Place the NIST thermometer in bubbler. If using a separate field thermometer, place in bubbler.
4. Wait 5 minutes for the Troll thermistor and thermometer(s) to stabilize.
5. Record the NIST thermometer temperature. The acceptable range for the Troll temperature is ±0.3°C of NIST temperature. The acceptable range for the field thermometer temperature is ±1°C of NIST temperature.
6. In Pocket-Situ select "Parameters" and the "Profiler" tab should appear in the lower right of the screen.
7. Select "Profiler" to open a window that allows naming a file.
8. Select the keyboard icon to allow typing and enter the premobilization profiler file name according to the following convention, "postcal_mmddyy" where mm is the 2-digit month, dd is the 2-digit year, and yy is the 2-digit year.
9. Record 7 profiler readings by tapping "snapshot" 7 times in succession.
10. Select "Close" to store the data and exit profiler mode.
11. This file will be downloaded and pasted into Field_forms.xls.

1.2.8 Complete premobilization instrument check

1. Download calibration reports and premobilization pre- and post-calibration 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.

Appendix B: Field_forms.xls premobilization worksheet example

Original data sheet created by D. Thoma (12/2005), data sheet updated 10/2013 and 3/2015 by C. Hackbarth.

NCPN Premobilization Calibration Log:												
Customized for Pocket Situ version 4.58.18 for Troll 9500			last updated 3/11/15		**IMPORTANT!! only enter data into cells that are highlighted**							
Date	3/7/2016											
Start time	14:39				**Sensor**		**Sensor ID**				**mfg date**	**Port**
End time	15:46				Temperature		platinum resistance thermistor					N/A
Operator	Carolyn Hackbarth				Conductivity		LC05709	Low	Range		10/30/2008	4
Instrument Mfr.	In-Situ				pH		PH11582				1/12/2009	1
Sonde serial number	50720				Dissolved Oxygen		382475				8/28/2014	3
Model	Troll 9500				Barometric Pressure		Piezoresistive silicon pressure sensor					
Display	Rugged Reader				vented cable		235513					
Display voltage (%)	70											
Sonde (v)	3.06				**Notes:**							
Sondepower (%)	100											
Temperature Check and Profiler Readings			****Water temperature acceptance criteria: NIST vs Troll is +/- 0.3°C and NIST vs field thermometer is +/- 1°C**									
Pre-Cal. Measurements		**precal file name**		precal_030716			**Post-Cal Measurements**		**postcal file name**		postcal_030716	
	°C	°F	**Error**	**Allowable?**	**Range**		°C	°F	**Error**	**Allowable?**	**Range**	
Troll temperature	23.11	73.60	0.01	USGS/NPS	22.8 - 23.4	Troll temperature	22.34	72.21	-0.06	USGS/NPS	22.1 - 22.7	
NIST Thermometer	23.1	73.58				NIST Thermometer	22.4	72.32				
Field Thermometer	22.5	72.50	-0.60	yes	22.1 - 24.1	Field Thermometer	22	71.60	-0.40	yes	21.4 - 23.4	
Profiler Precal Readings		**median**	**range**	**stable readings?**		**Profiler Postcal Readings**		**median**	**range**	**stable readings?**		
Specific Conductivity		321.38	0.08	yes		Specific Conductivity		332.83	0.09	yes		
pH		8.40	0.01	yes		pH		8.22	0.02	yes		
Dissolved Oxygen		7.09	0.00	yes		Dissolved Oxygen		7.39	0.02	yes		
Temperature (°C)		23.11	0.02	yes		Temperature (°C)		22.34	0.02	yes		
Barometric pressure		647.73				Barometric pressure		647.46				
Notes:						Important!! If readings are unstable, check conditions and repeat 7 snapshot readings.						
						If readings continue to be unstable, sensor(s) may need to be replaced.						

Specific Conductance Calibration (1413 µS/cm standard) & Error Check @ 147 and 12,900 µS/cm

Standard (µS/cm)	Pre-Cal. Reading (µS/cm)	lot #	Exp	maker	Cat #		Post-Cal. Reading (µS/cm)	Abs. diff	USGS or NPS Allowable?	Acceptable range	as of July 2008 Lab AMS
147	146	5061578	6/30/16	Aqua Solutions	6906-4L		150	3	USGS/NPS	142 - 152	2.56 us/cm
1413	1393	5032659	3/30/16	Aqua Solutions	6920-4L		1413	0	USGS/NPS	1408 - 1418	**Field AMS+**
12900	12769	5082101	8/30/16	Aqua Solutions	6931-4L		12953	53	USGS/NPS	12513 - 13287	3.00 us/cm

Type of sensor?	Low	Range	Post calibration error check on solutions used in field last month		
K-cell	0.324	**K-cell acceptable low range sensor = 0.30 - 0.39**	**Standard**	**reading**	**Abs. diff**	**Allowable?**	**Acceptable range**
K-cell check	ok		**1413**	1421	8	USGS/NPS	1371 - 1455

Important!! If the K-cell is out of range, check conditions and recalibrate. If after recalibration the K-cell is still out of range then the conductivity sensor may need to be replaced.

Notes:

pH Calibration: 2-point or 3-point. pH 4, 7, 10 standards

pH buffer value adjusted for temp	pH buffer temp	Pre-Cal. Reading	Temp (°C) during calibration	Post-Cal. Reading	Error	USGS or NPS Allowable?	Acceptable range	As of July 2008 Lab AMS
4.00	19.15	3.94	19.17	3.99	-0.01	USGS	3.70 - 4.30	0.02
7.03	18.59	7.12	18.93	7.08	0.05	USGS	6.73 - 7.33	**Field AMS+**
10.07	19.13	10.11	19.25	10.07	0	USGS	9.77 - 10.37	0.02

pH standard	lot #	Exp	maker	Cat #	Post calibration error check on solutions used in field last month					
					pH buffer temp	**pH (temp adj)**	**reading**	**Error**	**Allowable?**	**Acceptable range**
4	5060487	6/30/17	Aqua Solutions	1460-4L						
7	4120872	12/30/16	Aqua Solutions	1500-4L	20.27	7.02	7.05	0.03	yes	6.72 - 7.32
10	50119113	1/30/17	Aqua Solutions	1525-4L	20.12	10.06	10.06	0.00	yes	9.76 - 10.36

slope/offset check	**Slope 1 mV/pH**	-57.15	ok	**slope 1 acceptable range = -66 to -50 mV/pH**
	Offset 1 mV	405.75	ok	**offset 1 acceptable range = 350 to 450 mV**
If making a 3-point calibration	**Slope 2 mV/pH**	-53.54	ok	**slope 2 acceptable range = -66 to -50 mV/pH**
	Offset 2 mV	380.16	ok	**offset 2 acceptable range = 350 to 450 mV**
	maintenance needed?		no	

Important!! If the slope and/or offset are out of range, replace the filling solution and recalibrate
If the sensor fails to calibrate within range after replacing the filling solution, replace the reference junction

Notes:

Dissolved Oxygen (DO) Calibration		Sensor Type	Optical RDO								
					Pre-Cal	**Post-Cal**			**USGS or NPS**	**Acceptable**	**as of July 2008**
	Cal Method	**Lot #**	**Exp**	**stimulus**	**Reading**	**Reading**	**Error**	**Units**	**Allowable?**	**range**	**Lab AMS**
100% oxygen	bubbler			7.24	7.1	7.29	0.05	mg/L	USGS	6.84 - 7.64	0.17 mg/L
0% oxygen	sodium sulfite	A3322	11/1/2017	0	0.00	0.00	0.00	mg/L	yes	-0.20 - 0.20	**Field AMS+**
DO% saturation check	bubbler			100	97.77	100.43	0.43	% Saturation	NPS	95 - 105	0.08 mg/L
									*No USGS criteria for DO% Saturation		
If using Optical RDO, enter saturation point and slope to determine stimulus DO value											
oxygen saturation calibration point		7.03	mg/L								
slope		1.03									
stimulus DO value		7.24	mg/L								
acceptable range		6.84 - 7.64	**Make sure the post-cal reading for 100% DO is within this range. If out of range, check conditions and recalibrate.**								
Slope		1.03	(mg/L) / (mg/L)	**slope acceptable range for RDO is 0.8 to 1.2, 1-mil membrane is 30 to 67, 2-mil membrane is 15 to 34**							
Slope check		ok									
Offset		0.01	mg/L	**offset acceptable range for RDO is -0.1 to 0.1, Clark cell 1-point cal is 2 nA**							
Offset check		ok									
Important!! If the slope or offset is out of range, check conditions and recalibrate. If after recalibration the sensor is still out of range then the sensor is bad and may need to be replaced.											
Temperature during calibration		22.98	°C								
Barometric pressure during calibration		647.87	mmHg								
Nearest NOAA altimeter setting (inches Hg)		29.69	inches	sea level pressure							
Elevation of calibration		4085	ft								
Conversion for elevation from NOAA formula		649.20	mmHg								
Barometric pressure % error		0.21									
Barometer check		barometer OK	NOTE: If barometer needs maintenance, send to In-Situ for repair								
calibration good?		yes									
Notes:											

Turbidity Calibration: with primary formazin standard (At least every 3 months)										
					Turbidity calibration standards					
Date of Latest calibration and assignment of Gelex values			2/9/2016		**manufacturer**	**Cat #**	**NTU**	**Variation**	**Lot #**	**Exp**
Gelex	**0-10**	**10-100**	**100-1000**		Hach	2684701	<0.1	n/a	A4346	Mar-16
Assigned value	6.27	51.7	497		Hach	2684801	20	+/- 1ntu	A5029	May-16
Measured value	6.28	51.4	498		Hach	2684901	100	+/- 5ntu	A5034	May-16
Acceptable range	6.08 - 6.46	50.1 - 53.3	482 - 512		Hach	2660501	800	+/- 40ntu	A5029	Apr-16
% rel. diff.	0.159	0.580	0.201							
± 3% Y/N	Y	Y	Y		**Turbidity instrument**					
Recalibration necessary?	no				Hach 2100P					
					Conforms to USEPA method 180.1					

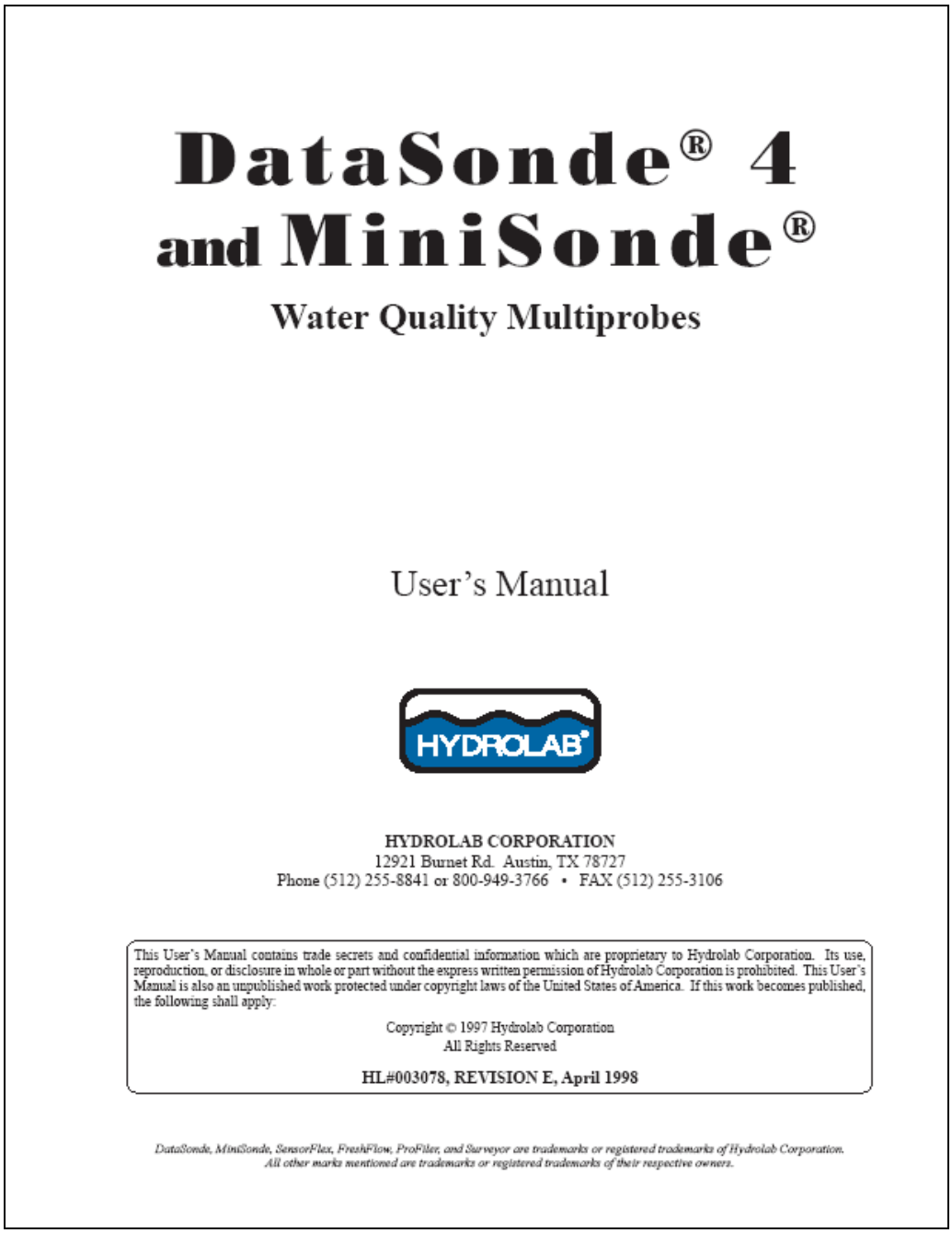
Appendix C: In-Situ Troll 9500 Operator's Manual

Double-click image to open pdf file in Adobe Acrobat Reader



Appendix D: Hydrolab User's Manual

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DataSonde® 4 and MiniSonde®

Water Quality Multiprobes

User's Manual

HYDROLAB

HYDROLAB CORPORATION
12921 Burnet Rd. Austin, TX 78727
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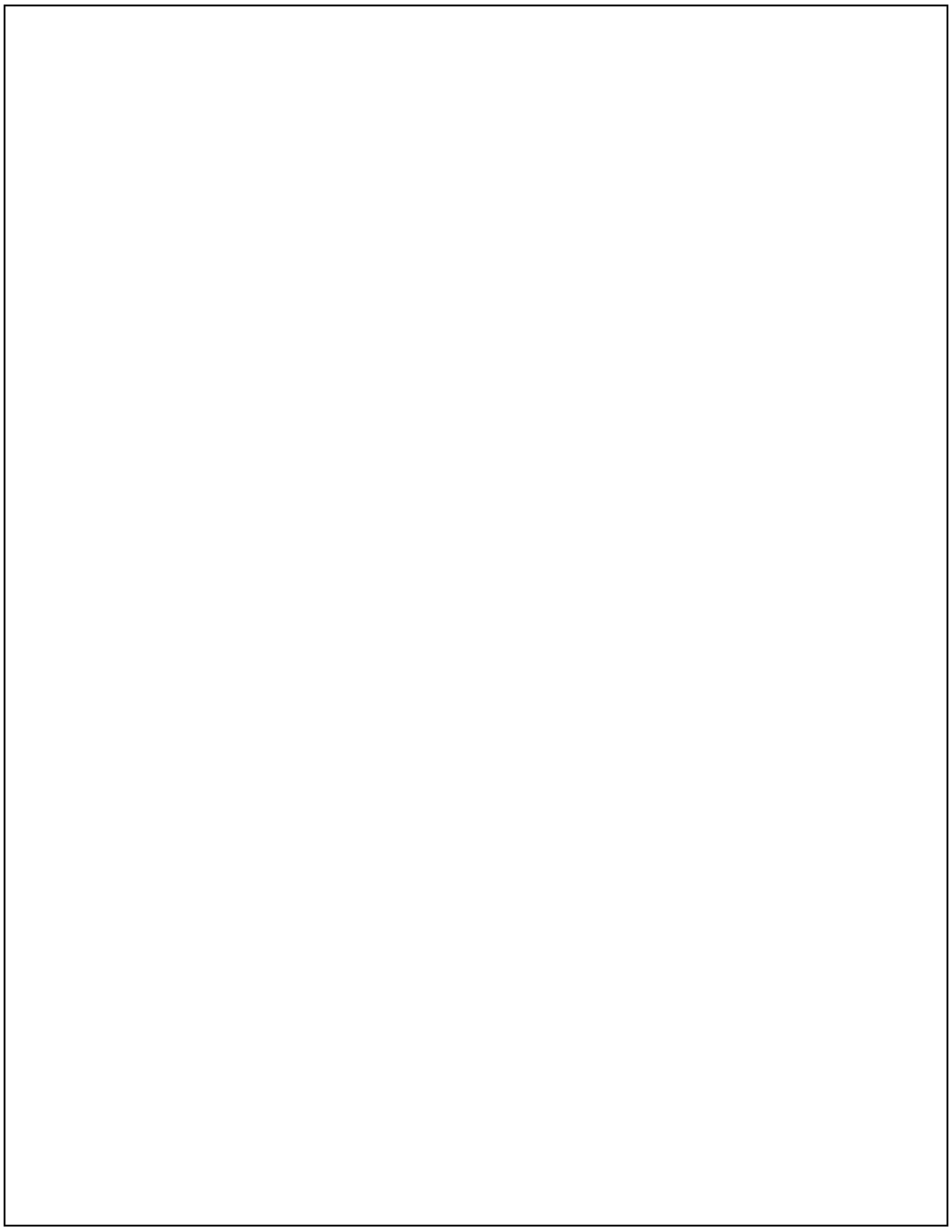
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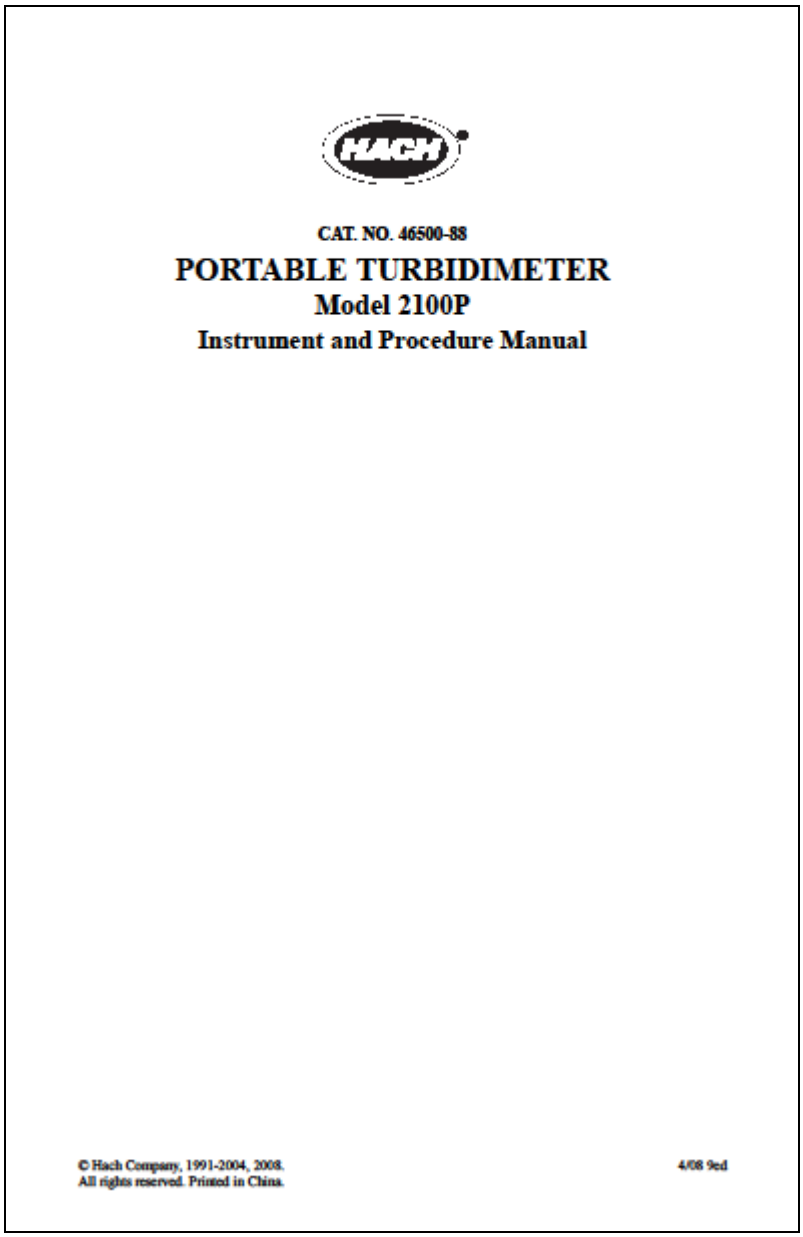
Appendix E: Hydrolab Display Unit Manual

Double-click image (box) to open pdf file in Adobe Acrobat Reader. Advance to page 2 to view file, the first page is blank.



Appendix F: Hach 2100P Turbidimeter Manual

Double-click image to open pdf file in Adobe Acrobat Reader



CAT. NO. 46500-88

PORTABLE TURBIDIMETER

Model 2100P

Instrument and Procedure Manual

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4/08 9ed

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 #

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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 paired 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:

$$\sqrt{\frac{\sum (x-\bar{x})^2}{(n-1)}}$$

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

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	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.

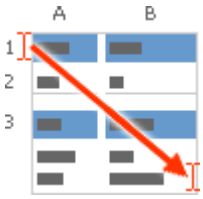
Example

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	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

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	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

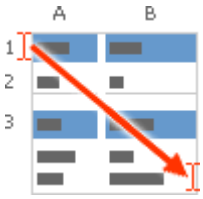
Suppose we observe that, in our sample of 50 commuters, the average length of travel to work is 30 minutes with a population standard deviation of 2.5. With alpha = .05, CONFIDENCE(.05, 2.5, 50) returns 0.692952. The corresponding confidence interval is then 30 ± 0.692952 = approximately [29.3, 30.7]. 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.

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	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.

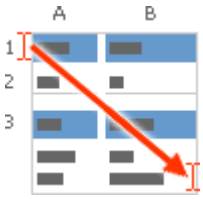
Example

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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)

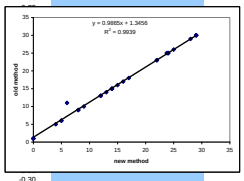
observation	observer 1 (old = y) pH	observer 2 (new = x) pH	percent difference	fraction of change old to new	observer 1 (old = y) specific conductance (us/cm)	observer 2 (new = x) specific conductance (us/cm)	percent difference	fraction of change old to new	observer 1 (old = y) temperature (deg C)	observer 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 Differe	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 1.01 (3/29/2016)

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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 UTDWQ 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.

Title	Park/Network
I&M Program Manager	NCPN
Water Quality Program Manager	NCPN
Hydrologist	ZION
Hydrologist	CURE
Chief of Resources	SEUG
Chief of Resources	CARE
Chief of Resources	TICA
Chief of Resources	DINO
Chief of Resources	BRCA

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 changes in equipment, laboratory methods, 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.

Title (Affiliation)	Responsibility
Water Quality Program Manager (NCPN)	Technical Lead - oversee collection of monitoring data and data management, coordinate report writing and protocol revision
Hydrologists (CURE, ZION)	Aquatic Professionals Team - technical working group
I&M Program Manager (NCPN)	Coordinate with water quality technical lead on reporting requirements, assist with peer review process, program oversight
I&M Data Manager (NCPN)	Assist with water quality database management, archive, and uploading to NPS and national data bases
Western Slope water quality program manager (USGS)	Provide technical oversight and coordination with cooperative agreements.
Hydrologists (WRD)	Technical oversight, peer review, and formal program approval
Cooperative Sampling Program Manager (UTDWQ)	Coordinate Utah samples with state laboratory, provide access to laboratory results

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 are superior to 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, and recreation. Fishing and other aquatic life classifications include cold water game fish, warm water game fish, non-game fish and other aquatic life such as waterfowl and shore birds. Waters may also be designated for irrigation and stock watering. A special class 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 must be verified by checking current state water quality guidelines. 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 compared to chronic state water quality standards for designated beneficial uses to provide advance warning to park managers of impending problems and to support management decisions for improving impaired waters.

2. The National Park Service does not have regulatory authority over waters in the U.S., or even the authority to make assessments for designated beneficial use. For that reason, comparisons of water quality data to designated beneficial-use criteria do not state whether a designated beneficial use was attained. Those designations are left to the states, in their triennial 305(b) reports to Congress. However, the NPS participates with states in collecting data used in the protection of water bodies under state jurisdiction.

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 compared to acute and chronic state water quality standards to look for spikes or long term near-exceedance conditions. 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. Investigate potential cause(s) of chronic or acute exceedances and near-exceedances. 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.

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 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 (e.g., 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 (i.e. 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 by the State's 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 meets 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. The rationale for the sampling design and site selection, water bodies exceeding state standards for intended uses, and the sampling sites and panel rotation for the sites are provided in the protocol narrative.

Site visits are coordinated to occur during periods of base 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 Utah Public Health Laboratory in Taylorsville, 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 in an MS Access data base, NPSTORET, for archive and data analysis (SOP 14).

Data analysis and reporting will track site conditions and trends in data range and variability. Emphasis will be placed on comparisons with state water quality standards.

The standards vary by state and intended use of the water body. State standards and designated uses must be updated periodically to ensure they remain current.

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 analysis

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.

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 ensure 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 providing data that can be used to assess attainment of beneficial use criteria and the 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 ensure large enough sample sizes to meet project goals outlined in the protocol narrative. 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 during the sampling season, 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 4 times annually due to cost constraints.

7.1.3.2. Statistics

The completeness of data required for long term trend analysis is related to parameter variability. 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. For many parameters, it may take longer than 2 years to detect significant trends (>30% change in mean parameter value) even at 100% completeness. Likewise more subtle change detection will require longer monitoring durations.

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 and duplicated in Table 6. 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.

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 are updated / reported for each result that falls below the reporting or detection limit 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 archived by the USGS NWQL laboratory. The reporting limits are the minimum detectable or quantifiable concentration reported by the USGS NWQL laboratory. These can change based on laboratory performance checks and on data use practices. 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 are associated with each result reported by the laboratory. 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 archived by the Utah Division of Water Quality. The reporting limits can change based on laboratory performance checks and on data use practices.

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 are included in user manuals appended to SOP 5. The only case where the 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 measurements 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 for CO parks and SOP 13 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 deionized or tap water. 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 without 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 4. 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 9500	+/- 30%	…..	95-105%	0.010	na	na
Dissolved Oxygen (mg/L)	Troll 9500 Optical	+/- 30%	…..	95-105%	0.148	na	0.02
Temperature (oC)	Troll 9500 Thermistor	+/- 30%	…..	95-105%	0.042	na	na
Specific Conductance (µs/cm)	Troll 9500 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 90-110% 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 9500 users manual (SOP 5). The same type of field calibration is performed by hydrologists in CO park units (SOP 8).

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 5. 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 (6). 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 and in site selection and timing SOP 4). Data variability and sample sizes necessary to achieve a high degree of power are discussed in 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 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. The original paper forms used in the field are stored with the NCPN water quality program lead until data are certified and are then archived at WACC.

6. Electronic copies of field forms and the project database are stored on NCPN's shared computer drive, which has an on-site server and regular off-site backups.

9.1.2. Laboratory data

1. Laboratory data are accessed via publicly available databases including NWIS for USGS data and AQWMS or EPA STORET for UTDWQ data.

2. These data are imported to a local NPSTORET database and linked to site visit core parameter data. After certification, the data are exported for upload to the NPS WRD centralized water quality database for upload to the publicly available WQX.

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. S/he does quality control checks and can then request re-runs on samples if necessary. S/he provides an electronic data deliverable to NCPN for upload to NPSTORET.

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 water quality program lead, 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 water quality program lead. After these quality control checks the NCPN hydrologist will finalize data in NPSTORET. 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 water quality program lead with the help of park hydrologists is responsible for 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. 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 monthly site visits. Therefore, completeness goals have been set as 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 6. 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 9500	Hydrolab mini sonde 4a	none	none	none	none
Conductivity	In situ Troll 9500	Hydrolab mini sonde 4a	none	none	none	none
Dissolved oxygen	In situ Troll 9500	Hydrolab mini sonde 4a	none	none	none	none
Temperature	In situ Troll 9500	Hydrolab mini sonde 4a	none	none	none	none
Flow	Baski cutthroat flume, Flowtracker ADV, 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 Utah Public Health laboratory, the laboratory 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 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 NPSTORET 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.

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 a water quality standards comparison report 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. 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 UTDWQ 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 UTDWQ 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.

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 reject 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.

24. Reconciliation with Data Quality Objectives

To ensure data collection, compilation and review meet the project objectives an important step in report preparation will include summarizing accuracy, precision, and completeness. Accuracy and precision will be calculated based on results from the premobilization instrument calibration or 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
$C = (U / P) \times 100$
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

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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

STATE OF UTAH DEPARTMENT OF ENVIRONMENTAL QUALITY DIVISION OF WATER QUALITY QUALITY ASSURANCE PROGRAM PLAN FOR WATER MONITORING PROGRAMS

Dianne R. Nielson, Ph.D.

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: _____

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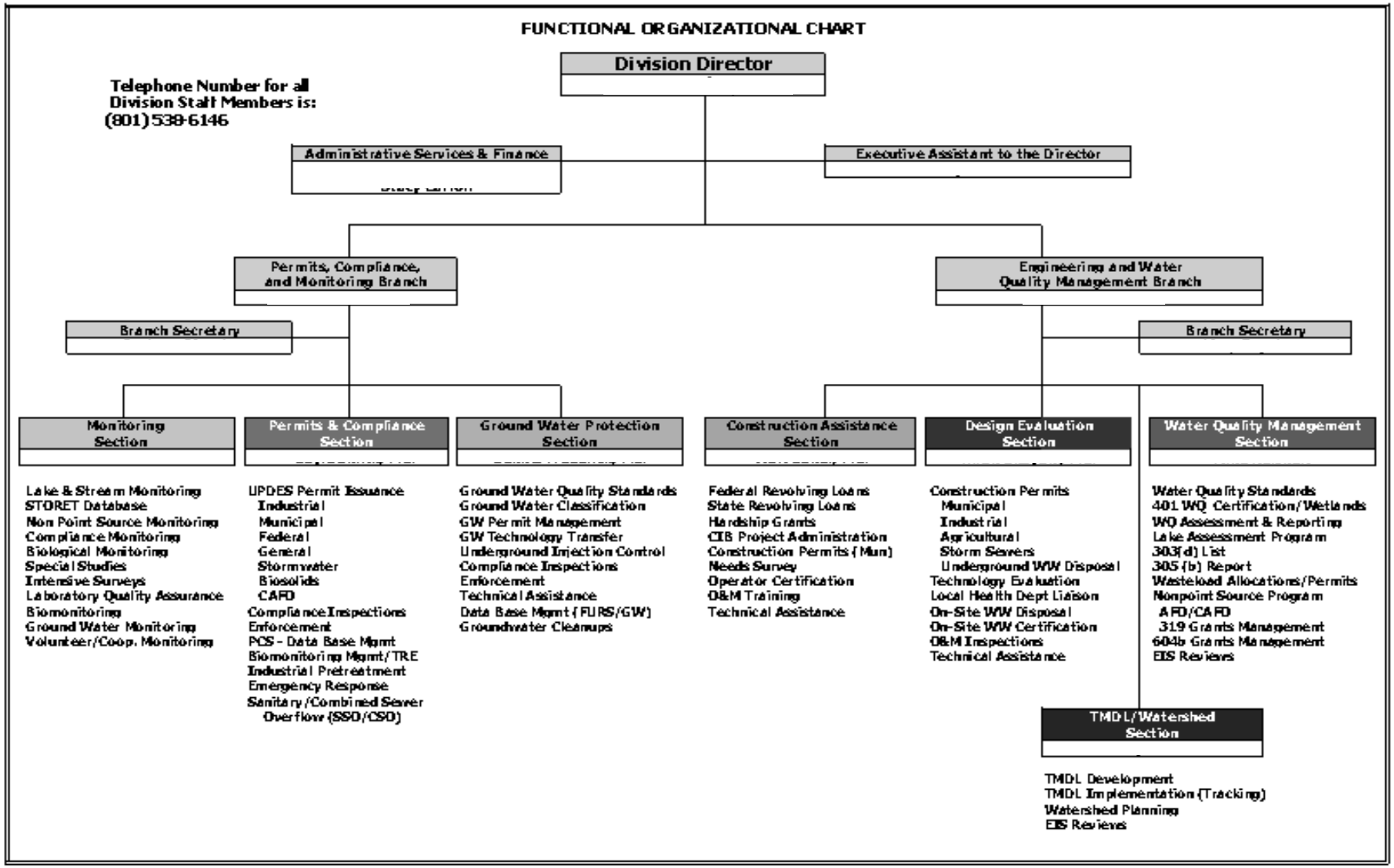
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DIVISION OF WATER QUALITY

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.

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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.

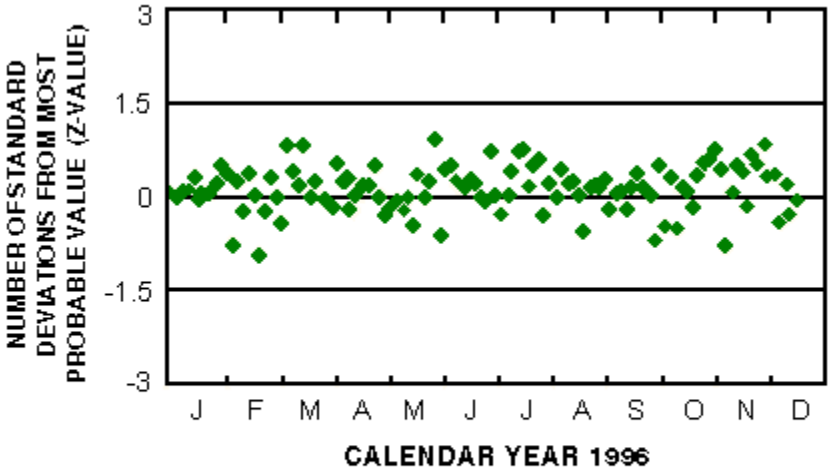


Figure 1. Control chart of cadmium determinations in standard reference material samples by graphite furnace atomic absorption spectrophotometry.

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.

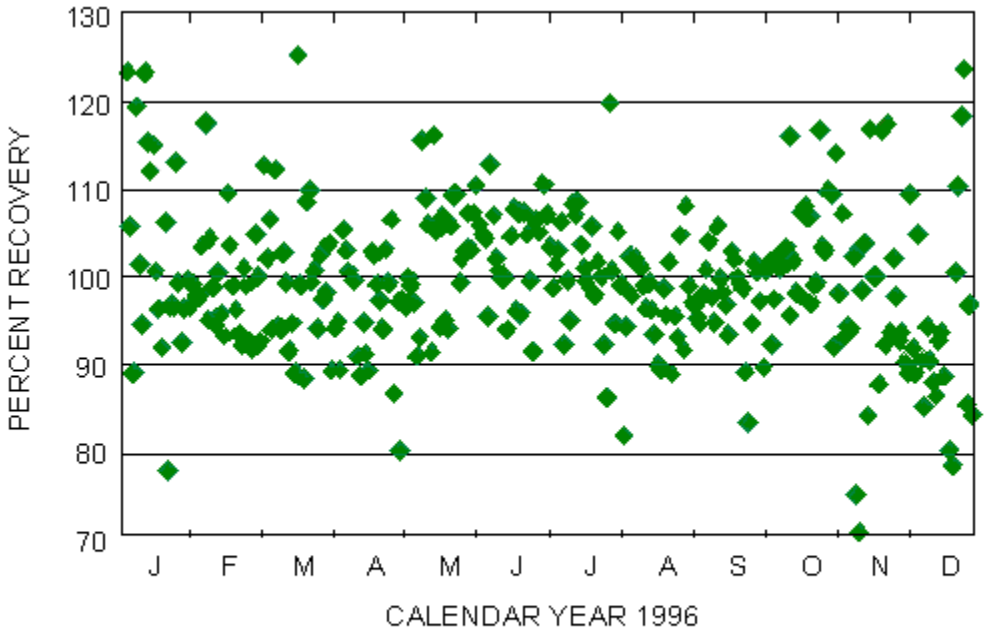


Figure 2. Percent recovery of the herbicide atrazine analyzed by solid-phase extraction, gas chromatography/mass spectrometry.

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.

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April 1998

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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

Reading #	**DO (mg/L)**	**Baro. Press. (mm Hg)**	**Spc. Cond (uS/cm@25 oC)**	**Water temp oC**	**pH 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

Reading #	**DO (mg/L)**	**Baro. Press. (mm Hg)**	**Spc. Cond (uS/cm@25 oC)**	**Water temp oC**	**pH 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 1.03 (April 2015)

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 appendix for decontamination procedure	Previously did not have a decontamination procedure	1.01
1.01	9/2012	Carolyn Hackbarth	Added FlowTracker procedure; updated methods	New instrument for measuring flow; outdated methods	1.02
1.02	4/2015	Carolyn Hackbarth	Updated field pH calibration procedure; updates to appendices	Use of temperature adjusted pH buffer values; outdated manuals and rating charts	1.03

This SOP focuses primarily on techniques and equipment for discharge measurement and the use of multiparameter instruments 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 Colorado park units follow the USGS National Field Manual.

Utah - The In-Situ Troll 9500 multiparameter instrument with a Rugged Reader display will be used for determining core parameters. The Hach 2100P Turbidimeter will be used to measure turbidity.

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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 of a portable weir or flume provides the greatest accuracy, though the impacts of repeated installations should be considered.

Table 1. Recommended approaches to discharge measurement.

Discharge				
Discharge magnitude	**Gallons/minute (gpm)**	**Cubic feet per second (cfs)**	**Metric mL/s, L/s or m^3/s**	**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	0.02 to 0.22	1.0 to 10 L/s	Weir, Flume
Fifth	100 to 448.8	0.22 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 to 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.
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.

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 backcountry 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 at a spring is low, the discharge measurement may take tens 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.

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 the Field_forms spreadsheet "field_codes" tab and described in Section 12.

2. Use of Established Stream Gages

The use of an established stream gage is the preferred method for 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/nwis/rt

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 where discharge is > 1 cfs. In Utah park units, a pygmy meter with Aquacalc and wading rod were used to measure flow from December 2005 to August 2012. Since September 2012, the type of meter used in Utah park units is the FlowTracker ADV (Acoustic Doppler Velocimeter). Operator manuals for the FlowTracker and wading rod are provided in the appendix. This updated version only contains instructions for the FlowTracker.

Text below is modified from USGS Discharge Measurements at Gaging Stations, Buchanan and Somers, 1969: p. 31-54.

6. The current meter procedure - general

Choose a cross section in a straight reach that has the following characteristics:

- Streambed is free of large rocks, weeds, and protruding obstructions that create eddies, slack water, and turbulence.
- Streambed has a flat profile to eliminate vertical components of velocity.
- All of the flow is directed downstream (i.e. there are no eddies) and lines of flow are as near parallel as possible.

After the cross section has been selected continue with the following steps:

1. String measuring tape across the channel with it perpendicular to the direction of flow. Pull the tape taut and secure it at each bank.
2. 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.
3. Measure the area and mean velocity of each section separately.
   a. The person wading in the channel should stand downstream of the velocity meter and at an arms-length distance so as not to disturb the flow path.
4. At each vertical, the following observations are recorded on the FlowTracker.
   a. The distance to a reference point on the bank along the measuring tape
   b. The six-tenths (0.6) of depth value
   c. The averaged 40 second velocity as indicated by the current meter. This velocity is measured at 0.6 of the depth below the water surface in the vertical.
5. 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 FlowTracker.

7. The current meter procedure - specific

7.1. FlowTracker Procedure

1. The FlowTracker User Manual can be found in Appendix B.
2. Turn on the FlowTracker by pressing the On/Off switch for 1 second until the LCD screen turns on.
3. Press Enter to display the Main Menu.
4. Press 1 for the Setup Parameters Menu and verify the following values:
   a. Units: English
   b. Avg Time: 40
   c. Mode: Discharge
5. To set date and time go to Main Menu, press 2 for the System Function Menu, then scroll through screens to select 9: Set System Clock.
6. From the Main Menu, press 3 to Start Data Run

7. Enter a file name
    a. Naming convention is 2 letter site abbreviation + 2-digit month + 2-digit day + 2-digit year.
    b. Example: NV110512 for North Fork Virgin November 5, 2012
8. Enter site and operator name (Optional)
9. When prompted to run the automatic QC test, Press 1 to run the test and follow the on-screen instructions. The QC test must be performed at each site to ensure that the FlowTracker is working properly.
10. In the Starting Edge screen, press Set Location and enter the starting edge location.
11. Press Set Depth to set starting edge of water depth.
12. Press LEW/REW to toggle the starting edge of water (Enter Right or Left *facing downstream*).
13. Press Next Station to continue
14. Press Set Location and enter the location of the next measurement point
15. Press Set Depth and enter the stream depth by reading increments on the wading rod, adjust the wading rod accordingly.
16. Situate the probe so it is perpendicular to the measuring tape and press Measure.
17. The screen will display a countdown from 40 seconds while velocity is being measured.
18. Once the 40 second measurement is complete, a summary of velocity and quality control data is shown.
    a. Press 1 to accept the measurement and move on to the next measurement.
    b. A warning will be issued if any data are outside expected values.
        i. If the warning is due to operator error, Press 2 to repeat the measurement.
        ii. If a warning is due to natural elements of the stream, then accept the measurement and move on to the next station.
19. Measure the velocity at 20-30 stations following the same procedure outlined above.
20. When all stations are completed, press the End Section key.
    a. The FlowTracker reviews all data using several quality control criteria.
    b. When the review is complete, the ending-edge screen is shown.
    c. Enter the location and depth of the ending-edge.
21. Press Calculate Disch. to complete discharge calculations. Press Enter to move between the different display screens.
22. Record discharge (Total Q) and velocity (V Mean) on the field form.
23. Press 0 to return to the main menu. It is important to return to the Main Menu before turning the system off to ensure all data is saved.
24. To power off the FlowTracker hold the On/Off switch for 4 seconds.

8. Data storage and download for Current Meters

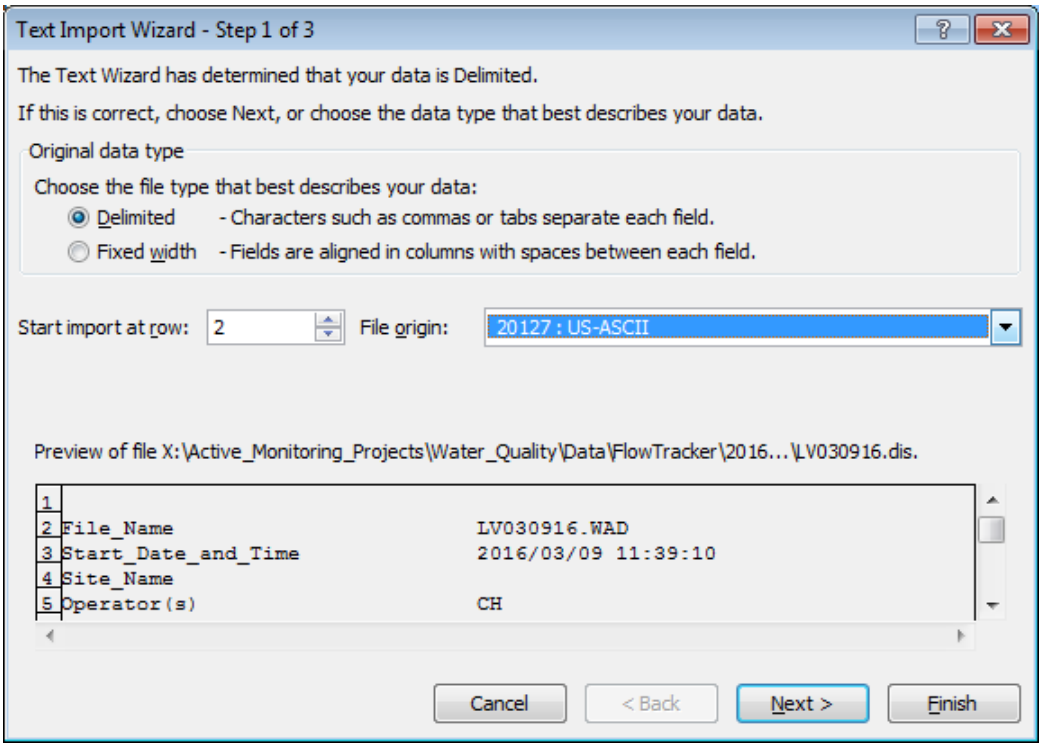
8.1. To view data files stored on FlowTracker

1. Turn on FlowTracker and press Enter to display the Main Menu.
2. Press 2 for Systems Functions Menu.
3. Press 1 to View Data File and select the desired file.
4. Nine screens of data are available
    a. Press Enter to move between the different display screens for discharge and velocity values.

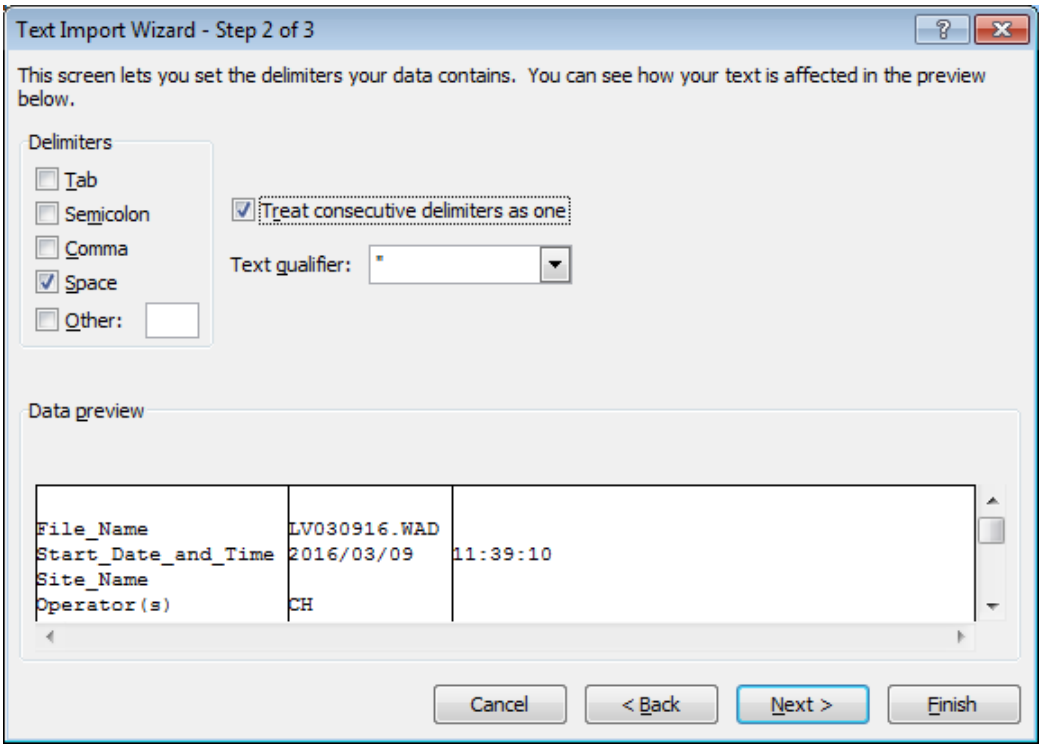
b. Press "Prev. Station" from any file summary screen to view the station data; scroll through all station data using the "Next Station" and "Prev. Station" keys.

8.2. To Download FlowTracker files to PC

1. Connect FlowTracker to serial port of computer or use USB converter to attach to USB drive of computer.
2. Open SonTek FlowTracker software.
3. Once the software is opened, click Connect to a FlowTracker to download data.
4. Choose the appropriate COM port and Baud rate (9600) and click Connect.
5. In the FlowTracker software a message should appear with "FlowTracker Found on COM", click OK. If the wrong COM port is selected then a message will appear with "Unable to wake up instrument", choose the correct COM port and try again.
6. Once connected, click Recorder to open a new window where files can be downloaded.
7. Select the files to be downloaded and then set the Baud rate (the default rate of 57600 can be used).
8. Specify the appropriate destination folder.
9. Click **Download** to copy the selected files to the destination folder.
10. Once the files have downloaded, click **Close.**
11. Click Disconnect after the selected files have been downloaded and the FlowTracker Handheld ADV can be disconnected from the computer.
12. In order to import the Flowtracker files into the Field_forms spreadsheet, they need to be exported into Excel.
13. First, open SonTek FlowTracker software if not already open.
14. Choose the export settings:
    - ✓Export ASCII Discharge file (DIS)
    - ✓Put headers on ASCII files
15. Click Program Settings and a new window will appear.
16. Specify English Units under Unit System, No Correction under Mounting Correction, and Export files to the same directory as the data file under Export Settings.
17. Then choose Open a FlowTracker file or Open many FlowTracker files/folders.
18. After selected files have been opened, export files will be created and can be located in the same directory as the data file.
19. Open Microsoft Excel, then go to File>Open and navigate to the folder with the exported FlowTracker files (DIS file extension). Select “All files” from the drop down list in the lower right to display the .DIS files.
20. Select the desired .DIS file to import.
21. The Text Import Wizard window will appear.
22. In the first window, choose **Delimited**, select **2** for “Start import at row:”, and scroll to **20127: US ASCII** for “File origin:”, then select “Next>”.



23. In the next window, select **Space** as the Delimiter, and make sure that the box is checked for "Treat consecutive delimiters as one". Select "Finish" and the file will open in Excel.



24. Save the file as type **Excel workbook** using the same name and folder as the original data file.
25. Copy and paste the data from the Excel file into the appropriate area in the Field_forms spreadsheet.

9. Use of Weirs and Flumes

Typically, weirs are used to measure discharge in channels which have second to fourth order magnitude where flow is less than 0.2 ft^3/s. Flumes are used to measure third to sixth order magnitude discharge where flow is at least 0.0285 ft^3/s and usually no more than 1 ft^3/s. Flumes and weirs may be heavy and difficult to pack into backcountry 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.

The Baski Cutthroat flume manual and corresponding rating tables are in Appendix A. The rating table for the 90 degree v-notch weir is located in the Field_forms.xls "rating_charts" tab.

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 of 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

This procedure is modified from USGS Discharge Measurements at Gaging Stations, Buchanan and Somers, 1969, p. 57-59.

1. Push the weir into a channel of loose material. Weirs will 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 stage height 3 to 5 times over a 3 to 5 minute interval.

7. Record % loss on the field form. Some % loss due to seepage around the weir plate is common and should be estimated (e.g. measured discharge is 6 gpm with an estimated % loss 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.
8. The mean value of the recorded stage heights will be used to compute the discharge.
9. The discharge (ft^3/s) is calculated using a standard equation specific to the weir plate, or a rating chart. The standard equation for flow over a weir with a 90-degree notch is
   $Q = Ch^{5/2}$
   Where Q is discharge in ft^3/s,
   C is the coefficient of discharge with a value of 2.5, and
   h is the static head or stage height, in ft
10. This equation is implemented automatically in Field_forms.xls when users enter stage height.
11. 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.

9.2. The portable flume procedure

This procedure is modified from USGS Discharge Measurements at Gaging Stations, Buchanan and Somers, 1969, p. 59-61

1. Install 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. Level the floor of the upstream section both longitudinally and transversely.
3. Fill in with available channel bed or bank material around the flume to prevent any water from bypassing it.
4. Allow flow to stabilize prior to reading the gage. For the Baski 8-inch cutthroat flume, stages lower than 0.10 feet and higher than 0.5 feet cannot be measured accurately. If the stage does not fall within the desired range, use a more appropriate method to measure flow.
5. After the flow stabilizes, record the upstream gage height and downstream gage height 3 to 5 times over a 3 to 5 minute interval.
6. Record a visual estimate of % total flow leaking around the flume. 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.
7. If the downstream gage height is less than 60% of the upstream gage height (stage*0.6), the flume has free flow. If the downstream stage is 60% of more of the upstream stage, the flume has submerged flow. Determine the correct rating curve to use and convert the recorded stage to discharge. The standard rating curve for the flume automatically converts gage height to discharge in the Field_forms.xls spreadsheet.

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.

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.

Each of the following procedures is modified from USGS Discharge Measurements at Gaging Stations, Buchanan and Somers, 1969, 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 field form.
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 field form.
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

This procedure is modified from USGS Discharge Measurements at Gaging Stations, Buchanan and Somers, 1969, 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. 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 field form.
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

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 In-Situ Multiparameter Instruments)

For use with the In-Situ Rugged Reader handheld PC and Troll multiparameter instrument, 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 one page is all that is needed for each site.

A hard copy of the field form 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. Parameter value readings before and after calibration

Some of the data will be over written when electronic files from the Troll unit and current meter are downloaded and pasted into Field_forms.xls. This eliminates transcription error and saves time once in the office. Download instructions for the electronic data can be found in this document.

An example of a field site form from the Field_forms.xls spreadsheet can be found in Appendix D. 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.

15. Field Calibration of In-Situ Troll

Field calibrations of pH and conductivity are performed with standards in temperature equilibrium with ambient surface water before taking measurements at each site. Optical DO sensors do not require field calibration.

1. Cool standards to ambient water temperature by immersing in stream for at least 15 minutes.
    a. If surface water temperature is < 6°C or >40°C, perform calibration indoors prior to visit. Another option is to calibrate with standards at air temperature at site as long as the air temperature is in range of 6°C to 40°C.
2. Attach Troll to Rugged Reader and establish connection with Pocket-Situ software.
3. Rinse Cal Cup and sensors with 1413 µS/cm conductivity standard, then fill to line with standard.
4. Take a pre-calibration reading of 1413 µS/cm conductivity standard and record on field form.
5. Conductivity calibration: In Pocket-Situ, select “Conductivity” from the “Parameters” menu, then tap “Calibrate” at the bottom of the screen. On the next screen (where 1413 is set as the default), select “Next” then “Run”. The sensor will begin calibrating. The screen first reads “Unstable”, then “Nominal”, then “Stable”. When it reads “Stable”, the final calibration screen will appear with a k-cell value.
6. Check the k-cell value, the acceptable range is 0.30 to 0.39. If k-cell value is outside of range, check conditions and recalibrate. If k-cell value is within acceptable limits, take a post-calibration reading and record on Field form. The post-calibration reading should be ±5 µS/cm (1408 -1418 µS/cm). If calibration reading is out of range, check conditions and recalibrate.
7. Rinse Cal Cup and sensors with stream water, then with pH 7 buffer. Fill Cal Cup to line with pH 7 buffer. Take a pre-calibration reading of pH 7 and record on field form.
8. Take a temperature reading of pH 7 buffer and record the temperature on field form. Use the table below “pH Buffer Values at Various Temperatures” to determine the pH values to input in calibration screen. Assume that both pH buffers will be similar in temperature.

Table 5. pH Buffer Values at Various Temperatures. The values listed are specific to Aqua Solutions pH buffers.

Temp (°C)	pH 4 buffer	pH 7 buffer	pH 10 buffer	Temp (°C)	pH 4 buffer	pH 7 buffer	pH 10 buffer
6	4.00	7.09	10.25	24	4.01	7.01	10.01
7	4.00	7.09	10.23	25	4.01	7.00	10.00
8	4.00	7.08	10.22	26	4.01	7.00	9.99
9	4.00	7.08	10.20	27	4.01	7.00	9.98
10	4.00	7.07	10.19	28	4.01	6.99	9.96
11	4.00	7.06	10.17	29	4.01	6.99	9.95
12	4.00	7.06	10.16	30	4.02	6.99	9.94
13	4.00	7.05	10.15	31	4.02	6.98	9.93
14	4.00	7.05	10.13	32	4.02	6.98	9.92
15	4.00	7.04	10.12	33	4.02	6.98	9.91
16	4.00	7.04	10.11	34	4.02	6.98	9.90
17	4.00	7.03	10.09	35	4.02	6.98	9.89
18	4.00	7.03	10.08	36	4.03	6.97	9.88
19	4.00	7.03	10.07	37	4.03	6.97	9.87
20	4.00	7.02	10.06	38	4.03	6.97	9.86
21	4.00	7.02	10.04	39	4.03	6.97	9.86
22	4.01	7.01	10.03	40	4.03	6.97	9.85
23	4.01	7.01	10.02				

9. pH calibration: In Pocket-Situ, select “pH” from the “Parameters” menu, then tap “Calibrate”. On the next screen, enter the temperature-adjusted pH values using the keyboard icon, then tap “Next”. On the next screen, tap “Run”. Once the reading is stable the screen will display the next pH to be tested. Rinse the sensors thoroughly with stream water and the next pH buffer (here pH 10) before loading the buffer in the Cal Cup. Select “Run” to begin stabilization for the second calibration point. Once the reading is “Stable” a final screen will show the slope and offset. Select “Finish” to complete the 2-point calibration. Take a post-calibration reading of pH 10 buffer and record on field form.

16. Using the In-Situ Rugged Reader for field data acquisition

16.1. Log core parameter data

1. Connect the Troll to the Rugged Reader with the associated cable.
2. Turn on Rugged Reader
3. Select Start at the upper left corner of the screen and a drop-down menu will appear.
4. Select Pocket-Situ 4 from the drop-down menu
   a. Double click COM1-19200 to find Troll
   b. Once the connection is established the Troll will appear in the Navigation tree.
   c. Double-click Troll 9000 Profiler
5. Select “Parameters” and then “Profiler” located at the bottom of the screen.
   a. Troll data files have the following naming convention:

SN34051 2005-09-28 192249 site_date

i. Digits 1-7 are the Troll serial number
ii. Digits 9-18 are the date
iii. Digits 20-25 are hours, min, sec in military time
iv. Digits >27 are the file name specified by user when file was opened

b. To specifiy the 'site_date' select the keyboard icon
   i. Site is a short abbreviation for the site name
   ii. Date is of format mmddyy

6. Click OK
7. Allow the Troll to equilibrate in the water. A Troll equipped with the optical DO sensor will take less than a minute to equilibrate to field conditions.
8. It can be helpful to monitor the status of equilibration by selecting graph on the bottom of the screen. A flat line means equilibrium is achieved.
9. 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.
10. Click "meter" then click "snapshot" 7 times to record 7 readings **without removing the sensors from the water**. The median value of each core parameter will be determined automatically in the Field_forms.xls spreadsheet.
11. Click "close" to end data logging session.
12. Before leaving the site view the data file by navigating to: \Home\Data Folder\Troll 9500 Profiler Data\SN_# of your unit\
13. Record the core parameters on the field form just in case the file cannot be recovered from the Rugged Reader. For each parameter make a quick estimate of the median from the 7 snapshot readings.
14. Close Pocket-Situ by selecting "X" in the upper right of the screen.

Table 6. 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 Liquid-in-glass Thermometer	 ± 0.2°C ± 0.5°C
[2]Conductivity (Specific Cond.) When ≤ 100 µS/cm When > 100 µS/cm	 ± 5 percent ± 3 percent
[2]pH: Meter displays to 0.01	 ± 0.1 to 0.2 pH unit
[3]Dissolved oxygen: Polarographic or optical	 ± 0.2 mg/L to ± 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 monthly

16.2. Troll data and calibration files stored on Rugged Reader

The calibration and data files recorded in the field are downloaded using a PC. Each of the files are copied and pasted into the appropriate section of the Field_form spreadsheet. Note: All of the following commands are typed or entered on the PC.

16.2.1. Troll data file download

1. Connect Rugged Reader to PC with USB cable. After connecting, Windows Mobile Device Center will automatically detect device.
2. After a connection is made go to "Connect without setting up your device", select "File Management", then click "Browse the contents of your device".
3. A new window will open labeled Pocket_PC, double-click on the backwards slash (\ ) folder.
4. Navigate to \Built-In Storage\PocketSitu\Data
   a. You can view file names here but cannot view data in the files.
5. Copy and paste BIN files from the Rugged Reader to the appropriate data folder on PC.
6. Open Win-Situ software on the PC
7. Open a BIN file in Win-Situ by clicking File > Open BIN file
8. Navigate to desired BIN file and double click to open
9. Click File > Export to Excel
10. Copy and paste from the temporary Excel file into appropriate location in Field_forms.xls

16.2.2. Troll calibration report download

1. Establish connection with Rugged Reader (follow Steps 1-3 of Download data reports to PC.
2. Navigate to \Built-In Storage\PocketSitu\calibration reports\
3. Select the Serial Number (SN) of the appropriate Troll to display calibration files
4. Troll calibration files have the following naming convention:
   SN34051 2005-09-29 083430 PH
   a. Digits 1-7 are the Troll serial number
   b. Digits 9-18 are the date
   c. Digits 20-25 are the hour, minutes, seconds in military time
   d. Digits 27-29 are the type of calibration: PH, Cond, DO or RDO
5. Copy and paste the calibration reports to the appropriate data folder on the PC
6. Double-click the calibration files to view the calibration results as a temporary internet file. Make sure to open the calibration files in Internet Explorer, otherwise the file will not paste properly in the Excel file.
7. Highlight the text then copy and paste the temporary internet file into the appropriate location in Field_form.xls spreadsheet.
8. In order for the file to be ready for data management and analysis, the file will need to be converted from "Text to columns"
9. Highlight the calibration file, then select Data > Text to columns.
10. Choose "Delimited" as the original data type, then select Next. Choose "Space" as the delimiter, then select Finish.
11. Make sure that the data does not overwrite any existing data in the spreadsheet.

17. Turbidity

Turbidity is measured and reported in nephelometric turbidity units (NTU) using the Hach 2100P portable turbidity meter. A description of the EPA method is provided in Appendix E. Instructions for calibration and measurement are in the Hach Turbidimeter manual in SOP 5: Preparations, Equipment Calibration, Handling and Storage.

18. Decontamination Procedures

Decontaminating equipment that comes in contact with water is essential to minimize the potential for spreading aquatic invasive species. In 2007 New Zealand mudsnails were found at a fish hatchery that flows in to the Fremont River. Quagga mussels are more likely to be introduced into reservoirs and streams 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 F) has been developed to prevent spread of aquatic invasive species.

19. References Cited

Buchanan T.J. and Somers, W.P. 1969, Discharge measurements at gaging stations: Techniques of Water-Resources Investigations of the United States Geological Survey, Book 3, Chapter A8, 65p.

20. Appendix A: Baski Cutthroat Flume User Manual

Cutthroat Flume Assembly

Open flume sidewalls and stand them upside down (see gauges or name plate for orientation) with long sections of sidewalls on one end and short sections, plus wing walls if desired, on the other end. The sidewalls should be bending in opposite directions at the throat (see Photo A). Place bottom pieces on, adjusting sides to proper throat distance. Secure by inserting bolt from inside of the flume into aligned hole (head fits into beveled hole), then placing washer over the bolt from the outside and hand tightening a nut on the bolt. Do this for each hole.

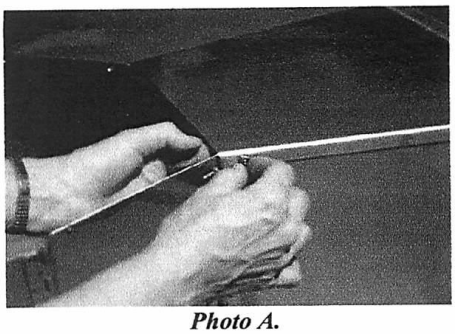


Photo A.

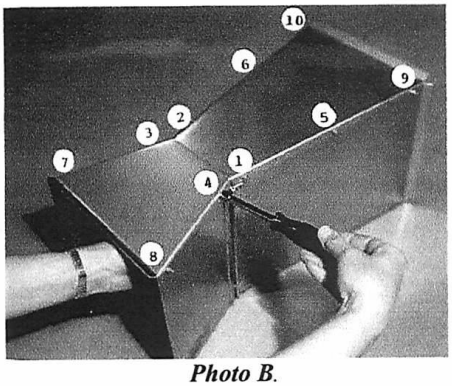


Photo B.

After all nuts are hand tightened on the two bottom pieces, tighten the nuts somewhat more (not completely) using the nut driver and working from the throat toward the flume ends, alternating from side to side and end to end as numbered in Photo B.

Turn the flume right side up and install center brace. The center brace for the 1", 2", and 4" throat is a flat piece and 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 nearer the inlet opening, thus, the holes will align. Insert bolt from the underside (these holes are not beveled), then place washer over the bolt from the topside and hand tighten the nut. Follow the same procedure for each hole.

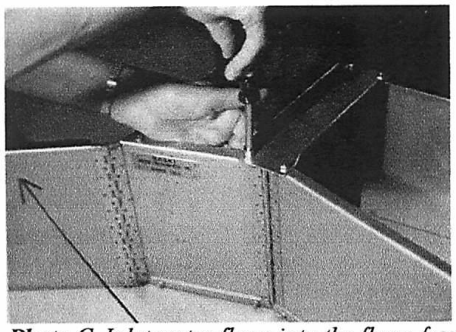


Photo C. *Inlet water flows into the flume from the end with the arrow.*

Baski Inc. *1586 So. Robb Way, Denver CO 80232 Ph: 800-55BASKI Email baski1@uswest.net*

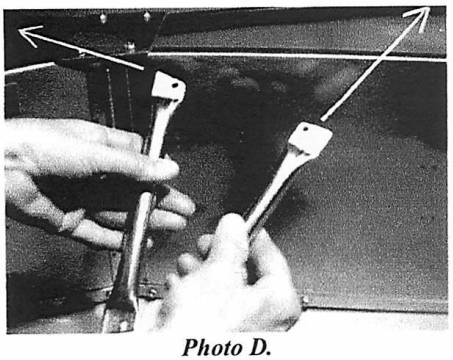


Photo D.

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.

Place handle under the bent portion (or lip) of the sidewall and secure by inserting bolt from the underside, then placing a washer over bolt from the topside and screwing on a nut. Do likewise for the other end of the handle. Follow same procedure for the second handle on opposite end of flume.

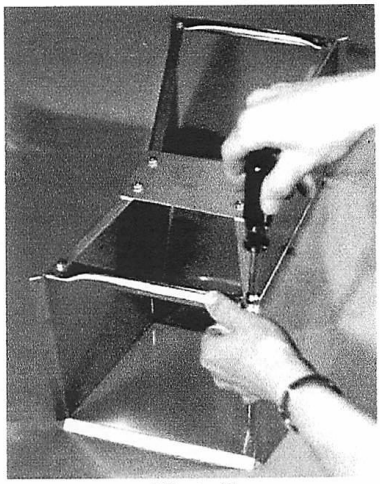


Photo E

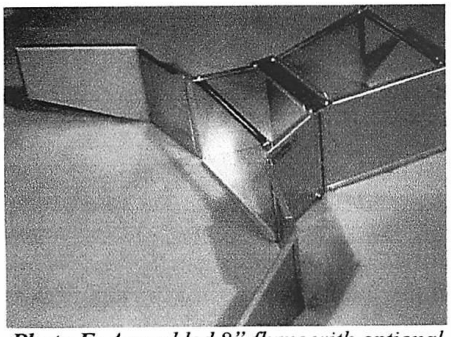


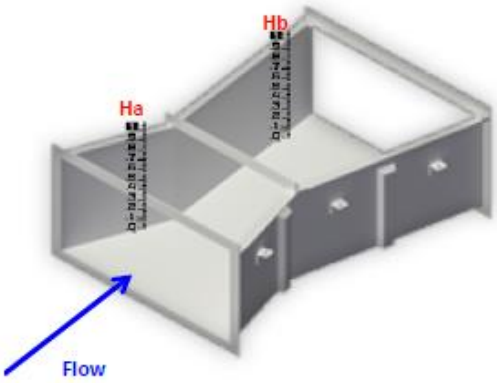
Photo F: *Assembled 8" flume with optional 19" wing walls.*

After all nuts are hand tightened on the three top pieces, use the nut driver to tighten them 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.

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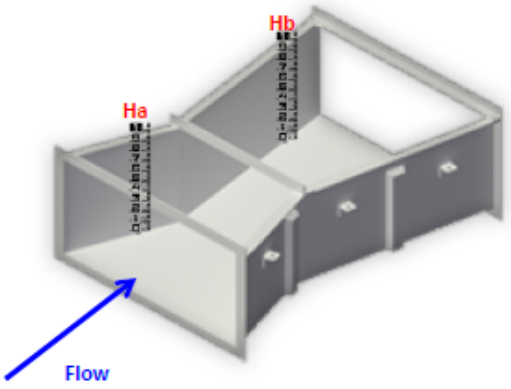
20.1. Baski Cutthroat Flume Rating Table

Rating Equations for 18 Inch Long Rectangular Cutthroat Flume



	Free Flow (cfs) Hb < 0.6*Ha		Submerged Flow (cfs) Hb > 0.6*Ha	
Throat Width	From Baski (do not use)	from Skogerboe and Others, 1972 (use)	From Baski (do not use)	from Skogerboe and Others, 1972 (use)
1 Inch	$0.50\ ha^2$	$0.494\ Ha^{2.15}$	None given	$0.261\ Ha^{1.741}$
2 Inch	$1.02\ ha^2$	$0.974\ Ha^{2.15}$	None given	$0.516\ Ha^{1.741}$
4 Inch	$2.08\ ha^2$	$1.975\ Ha^{2.15}$	None given	$1.048\ Ha^{1.741}$
8 Inch	$4.22\ ha^2$	$4.030\ Ha^{2.15}$	None given	$2.140\ Ha^{1.741}$

18-Inch L x 8-Inch W Rectangular Cutthroat Flume



Minimum Head (ft) (Ha)	0.10
Miniumum Flow Rate (cfs)	0.0285
Maximum Head (ft) (Ha)	0.50
Maximum Flow Rate (cfs)	0.9080
Accuracy	± 3%
Submergence Transition	60%

Installation and Operation Guidelines:

1) Install flume in a straight section of the channel.
2) Insure flume is level from front to back and side to side.
3) Minimize leakage from underneath and on the sides of the flume.
4) Always get an upstream stage (Ha) and downstream stage (Hb) so you can determine if you have "free-flow" or "submerged" conditions.
5) If the downstream stage is 60% or less than the upstream stage (Hb < 0.6*Ha) use the free flow rating . If the downstream stage is greater than 60% of the upstream stage (Hb > 0.6*Ha) use the submerged flow rating table.
6) The rating for free flow = 4.030 Ha^2.15. The rating for submerged flow = 2.14 Ha^1.741.
7) Discharge for stages <0.10 feet are inaccurate for this flume due to fluid flow properties and boundary conditions. Report any discharges for stages (Ha) <0.10 feet as "<0.0285 cfs" for free flow conditions and "<0.0389 cfs" for submerged flow conditions.

Reference:

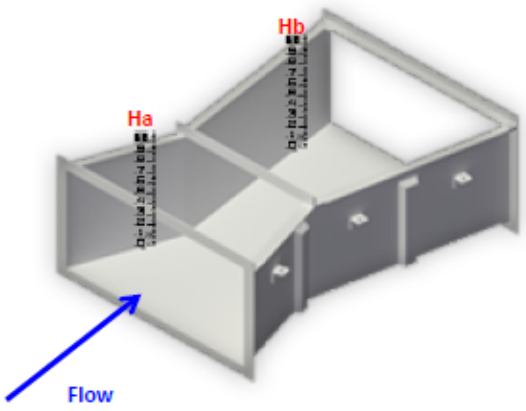
Skogerboe, G.V., Bennett, R. S. and Walker, W.R., 1972. Generalized discharge relations for Cutthroat flumes. Journal of the Irrigation and Drainage Division, ASCE, Vol. 98, No IR4, December, pp. 569-583.

Tables were revised from:
http://www.openchannelflow.com/products/flumes/cutthroat

Rating Table:

Stage (feet) Ha	Free Flow (cfs) (4.030 Ha ^2.15) Hb < 0.6*Ha	Submerged Flow (cfs) (2.14 Ha ^1.741) Hb > 0.6*Ha
0.01	< 0.0285	< 0.0389
0.02		
0.03		
0.04		
0.05		
0.06		
0.07		
0.08		
0.09		
0.10	0.0285	0.0389
0.11	0.0350	0.0459
0.12	0.0422	0.0534
0.13	0.0502	0.0613
0.14	0.0588	0.0698
0.15	0.0682	0.0787
0.16	0.0784	0.0881
0.17	0.0893	0.0979
0.18	0.1010	0.1081
0.19	0.1134	0.1188
0.20	0.1266	0.1299
0.21	0.1406	0.1414
0.22	0.1554	0.1533
0.23	0.1710	0.1656
0.24	0.1874	0.1784
0.25	0.2046	0.1915
0.26	0.2226	0.2051
0.27	0.2414	0.2190
0.28	0.2610	0.2333
0.29	0.2815	0.2480
0.30	0.3028	0.2631
0.31	0.3249	0.2785
0.32	0.3478	0.2944
0.33	0.3716	0.3106
0.34	0.3963	0.3271
0.35	0.4217	0.3441
0.36	0.4481	0.3614
0.37	0.4753	0.3790
0.38	0.5033	0.3970
0.39	0.5322	0.4154
0.40	0.5620	0.4341
0.41	0.5926	0.4532
0.42	0.6242	0.4726
0.43	0.6565	0.4924
0.44	0.6898	0.5125
0.45	0.7240	0.5329
0.46	0.7590	0.5537
0.47	0.7949	0.5748
0.48	0.8317	0.5963
0.49	0.8694	0.6181
0.50	0.9080	0.6402

18-Inch L x 4-Inch W Rectangular Cutthroat Flume



Minimum Head (ft) (Ha)	0.10
Miniumum Flow Rate (cfs)	0.014
Maximum Head (ft) (Ha)	0.50
Maximum Flow Rate (cfs)	0.445
Accuracy	± 3%
Submergence Transition	60%

Installation and Operation Guidelines:

1) Install flume in a straight section of the channel.
2) Insure flume is level from front to back and side to side.
3) Minimize leakage from underneath and on the sides of the flume.
4) Always get an upstream stage (Ha) and downstream stage (Hb) so you can determine if you have "free-flow" or "submerged" conditions.
5) If the downstream stage is 60% or less than the upstream stage (Hb < 0.6*Ha) use the free flow rating . If the downstream stage is greater than 60% of the upstream stage (Hb > 0.6*Ha) use the submerged flow rating table.
6) The rating for free flow = 1.975 Ha^2.15. The rating for submerged flow = 1.048 Ha^1.741.
7) Discharge for stages <0.10 feet are inaccurate for this flume due to fluid flow properties and boundary conditions. Report any discharges for stages (Ha) <0.10 feet as "<0.0140 cfs" for free flow conditions and "<0.0190 cfs" for submerged flow conditions.

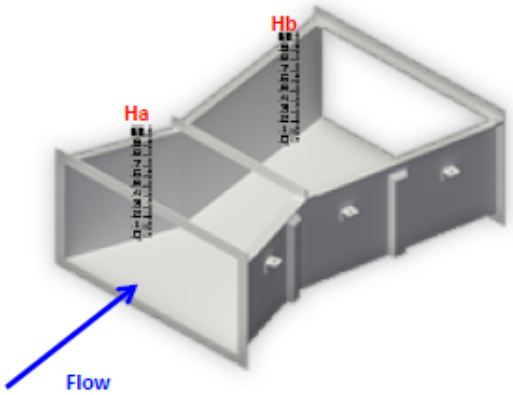
Reference:
Skogerboe, G.V., Bennett, R. S. and Walker, W.R., 1972. Generalized discharge relations for Cutthroat flumes. Journal of the Irrigation and Drainage Division, ASCE, Vol. 98, No IR4, December, pp. 569-583.

Tables were revised from:
http://www.openchannelflow.com/products/flumes/cutthroat

Rating Table:

Stage (feet) Ha	Free Flow (cfs) (1.975 Ha ^2.15) Hb < 0.6*Ha	Submerged Flow (cfs) (1.048 Ha ^1.741) Hb > 0.6*Ha
0.01	< 0.0140	< 0.0190
0.02		
0.03		
0.04		
0.05		
0.06		
0.07		
0.08		
0.09		
0.10	0.0140	0.0190
0.11	0.0172	0.0225
0.12	0.0207	0.0261
0.13	0.0246	0.0300
0.14	0.0288	0.0342
0.15	0.0334	0.0385
0.16	0.0384	0.0431
0.17	0.0438	0.0479
0.18	0.0495	0.0529
0.19	0.0556	0.0582
0.20	0.0621	0.0636
0.21	0.0689	0.0692
0.22	0.0762	0.0751
0.23	0.0838	0.0811
0.24	0.0918	0.0874
0.25	0.1003	0.0938
0.26	0.1091	0.1004
0.27	0.1183	0.1072
0.28	0.1279	0.1143
0.29	0.1380	0.1214
0.30	0.1484	0.1288
0.31	0.1592	0.1364
0.32	0.1705	0.1442
0.33	0.1821	0.1521
0.34	0.1942	0.1602
0.35	0.2067	0.1685
0.36	0.2196	0.1770
0.37	0.2329	0.1856
0.38	0.2467	0.1944
0.39	0.2608	0.2034
0.40	0.2754	0.2126
0.41	0.2904	0.2219
0.42	0.3059	0.2314
0.43	0.3218	0.2411
0.44	0.3381	0.2510
0.45	0.3548	0.2610
0.46	0.3720	0.2712
0.47	0.3896	0.2815
0.48	0.4076	0.2920
0.49	0.4261	0.3027
0.50	0.4450	0.3135

18-Inch L x 2-Inch W Rectangular Cutthroat Flume



Minimum Head (ft) (Ha)	0.05
Miniumum Flow Rate (cfs)	0.0016
Maximum Head (ft) (Ha)	0.50
Maximum Flow Rate (cfs)	0.2195
Accuracy	± 3%
Submergence Transition	60%

Installation and Operation Guidelines:

1) Install flume in a straight section of the channel.
2) Insure flume is level from front to back and side to side.
3) Minimize leakage from underneath and on the sides of the flume.
4) Always get an upstream stage (Ha) and downstream stage (Hb) so you can determine if you have "free-flow" or "submerged" conditions.
5) If the downstream stage is 60% or less than the upstream stage (Hb < 0.6*Ha) use the free flow rating . If the downstream stage is greater than 60% of the upstream stage (Hb > 0.6*Ha) use the submerged flow rating table.
6) The rating for free flow = 0.974 Ha^2.15. The rating for submerged flow = 0.516 Ha^1.741.
7) Discharge for stages <0.05 feet are inaccurate for this flume due to fluid flow properties and boundary conditions. Report any discharges for stages (Ha) <0.05 feet as "<0.0016 cfs" for free flow conditions and "<0.0028 cfs" for submerged flow conditions.

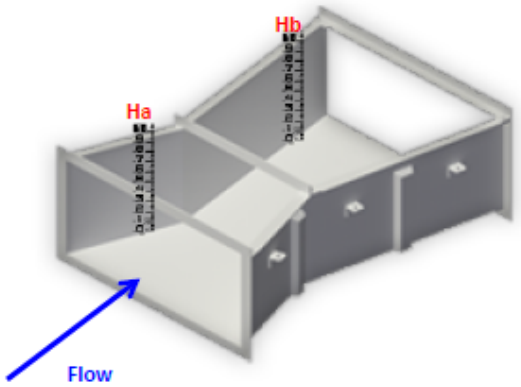
Reference:
Skogerboe, G.V., Bennett, R. S. and Walker, W.R., 1972. Generalized discharge relations for Cutthroat flumes. Journal of the Irrigation and Drainage Division, ASCE, Vol. 98, No IR4, December, pp. 569-583.

Tables were revised from:
http://www.openchannelflow.com/products/flumes/cutthroat

Rating Table:

Stage (feet) Ha	Free Flow (cfs) (0.974 Ha ^2.15) Hb < 0.6*Ha	Submerged Flow (cfs) (0.516 Ha ^1.741) Hb > 0.6*Ha
0.01	< 0.0016	< 0.0028
0.02		
0.03		
0.04		
0.05	0.0016	0.0028
0.06	0.0023	0.0038
0.07	0.0032	0.0050
0.08	0.0043	0.0064
0.09	0.0055	0.0078
0.10	0.0069	0.0094
0.11	0.0085	0.0111
0.12	0.0102	0.0129
0.13	0.0121	0.0148
0.14	0.0142	0.0168
0.15	0.0165	0.0190
0.16	0.0189	0.0212
0.17	0.0216	0.0236
0.18	0.0244	0.0261
0.19	0.0274	0.0286
0.20	0.0306	0.0313
0.21	0.0340	0.0341
0.22	0.0376	0.0370
0.23	0.0413	0.0399
0.24	0.0453	0.0430
0.25	0.0494	0.0462
0.26	0.0538	0.0494
0.27	0.0583	0.0528
0.28	0.0631	0.0563
0.29	0.0680	0.0598
0.30	0.0732	0.0634
0.31	0.0785	0.0672
0.32	0.0841	0.0710
0.33	0.0898	0.0749
0.34	0.0958	0.0789
0.35	0.1019	0.0830
0.36	0.1083	0.0871
0.37	0.1149	0.0914
0.38	0.1216	0.0957
0.39	0.1286	0.1002
0.40	0.1358	0.1047
0.41	0.1432	0.1093
0.42	0.1508	0.1140
0.43	0.1587	0.1187
0.44	0.1667	0.1236
0.45	0.1750	0.1285
0.46	0.1834	0.1335
0.47	0.1921	0.1386
0.48	0.2010	0.1438
0.49	0.2101	0.1490
0.50	0.2195	0.1544

18-Inch L x 1-Inch W Rectangular Cutthroat Flume



Minimum Head (ft) (Ha)	0.05
Miniumum Flow Rate (cfs)	0.0008
Maximum Head (ft) (Ha)	0.50
Maximum Flow Rate (cfs)	0.1113
Accuracy	± 3%
Submergence Transition	60%

Installation and Operation Guidelines:

1) Install flume in a straight section of the channel.
2) Insure flume is level from front to back and side to side.
3) Minimize leakage from underneath and on the sides of the flume.
4) Always get an upstream stage (Ha) and downstream stage (Hb) so you can determine if you have "free-flow" or "submerged" conditions.
5) If the downstream stage is 60% or less than the upstream stage (Hb < 0.6*Ha) use the free flow rating . If the downstream stage is greater than 60% of the upstream stage (Hb > 0.6*Ha) use the submerged flow rating table.
6) The rating for free flow = 0.494 Ha^2.15. The rating for submerged flow = 0.261 Ha^1.741.
7) Discharge for stages <0.05 feet are inaccurate for this flume due to fluid flow properties and boundary conditions. Report any discharges for stages (Ha) <0.05 feet as "<0.0008 cfs" for free flow conditions and "<0.0014 cfs" for submerged flow conditions.

Reference:
Skogerboe, G.V., Bennett, R. S. and Walker, W.R., 1972. Generalized discharge relations for Cutthroat flumes. Journal of the Irrigation and Drainage Division, ASCE, Vol. 98, No IR4, December, pp. 569-583.

Tables were revised from:
http://www.openchannelflow.com/products/flumes/cutthroat

Rating Table:

Stage (feet) Ha	Free Flow (cfs) (0.494 Ha ^2.15) Hb < 0.6*Ha	Submerged Flow (cfs) (0.261 Ha ^1.741) Hb > 0.6*Ha
0.01	< 0.0008	< 0.0014
0.02		
0.03		
0.04		
0.05	0.0008	0.0014
0.06	0.0012	0.0019
0.07	0.0016	0.0025
0.08	0.0022	0.0032
0.09	0.0028	0.0039
0.10	0.0035	0.0047
0.11	0.0043	0.0056
0.12	0.0052	0.0065
0.13	0.0061	0.0075
0.14	0.0072	0.0085
0.15	0.0084	0.0096
0.16	0.0096	0.0107
0.17	0.0109	0.0119
0.18	0.0124	0.0132
0.19	0.0139	0.0145
0.20	0.0155	0.0158
0.21	0.0172	0.0172
0.22	0.0191	0.0187
0.23	0.0210	0.0202
0.24	0.0230	0.0218
0.25	0.0251	0.0234
0.26	0.0273	0.0250
0.27	0.0296	0.0267
0.28	0.0320	0.0285
0.29	0.0345	0.0302
0.30	0.0371	0.0321
0.31	0.0398	0.0340
0.32	0.0426	0.0359
0.33	0.0456	0.0379
0.34	0.0486	0.0399
0.35	0.0517	0.0420
0.36	0.0549	0.0441
0.37	0.0583	0.0462
0.38	0.0617	0.0484
0.39	0.0652	0.0507
0.40	0.0689	0.0529
0.41	0.0726	0.0553
0.42	0.0765	0.0576
0.43	0.0805	0.0600
0.44	0.0846	0.0625
0.45	0.0887	0.0650
0.46	0.0930	0.0675
0.47	0.0974	0.0701
0.48	0.1020	0.0727
0.49	0.1066	0.0754
0.50	0.1113	0.0781

21. Appendix B: FlowTracker Technical Manual

SonTek/YSI
9940 Summers Ridge Road, San Diego, CA 92121-3091 USA
Telephone (858) 546-8327 • Fax (858) 546-8150
E-mail: inquiry@sontek.com • Internet: http://www.sontek.com

SonTek®
YSI *incorporated*

FlowTracker®
Handheld ADV®
User's Manual
Firmware Version 3.7

featuring

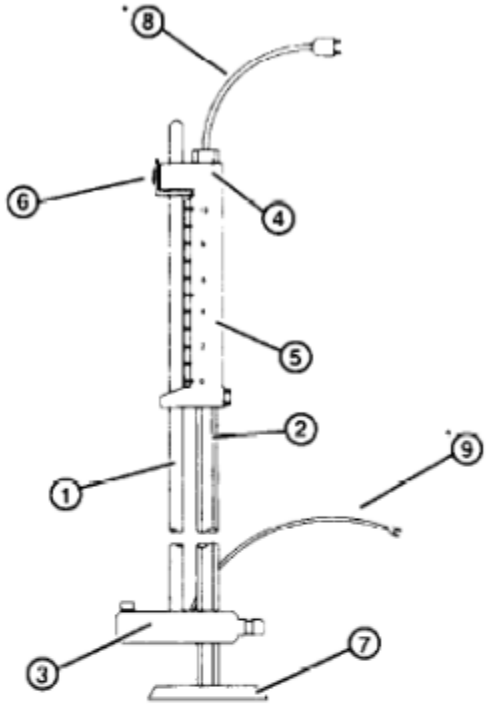
SmartQC™

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P/N 6054-60051–D

22. Appendix C: Top Setting Wading Rod Instructions

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.

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23. Appendix D: Example field site form from Field_forms.xls spreadsheet

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.

			NCPN I&M WATER QUALITY FIELD FORM					
			v1.08	9/30/2015				
Long Name	La Verkin Ck at Lee Pass Trail			**Sample team**			**Date(m/d/yr)**	3/9/2016
Station abbv.	laverk			Carolyn Hackbarth			**Start time**	11:30
Lat	37.40563	**map datum**	**UTM_E**	307070	**Zone 12**		**End time**	13:01
Long	113.17983	WGS84	**UTM_N**	4142102	**NAD83**		**Mean time**	12:15
altitude (ft)	4995					**Sample Collected at**		13:00
7.5' quad	Kolob Arch		**HUC code**	15010008				
Storet #	4950800		**State**	Utah				
Comments			**County**	Washington				
			Park	ZION				
			UT LABORATORY INFORMATION				All except carry bottle	
		Collect	**Collect**	**Collect**	**Collect**	**Collect**	are from	
Samples collected?:		**Total chem.**	**Filt. metals**	**Nutrients**	**Turbidity**	**Carry**	Utah Public Health Laboratory	
(y/n)……..		y	y	y	y	y	Attn CEL Sample Receiving	
		Microbiology	**Secchi disc**	**Chl. a**	**Algae**	**Inverts**	4431 S 2700 W	
(y/n)……..		y	n	n	n	n	Taylorsville, UT 84129	
							801-965-2405	
Bottles labeled		**Date & time**	**STORET #**	**Collector**	**Site name**			
(y/n)……..		y	y	y	y			**Filter packs**
Sample period (m / q)		m						Pall 12176
Sam Dpth (ft)	0	**Suite**	**Preservative**	**lot #**	**CAS #**	**Exp date**		**Lot #**
Total Chemistry		2	none	2015243	none	9/4/16		FA0669
Non-filtered Nutrients		2	H_2SO_4	2015153	7664-93-9	11/27/16		
Filtered nutrients		9	H_2SO_4	2015358	7664-93-9	11/27/16		
Filtered metals		4	$HN0_3$	2015015	7697-37-2	9/5/16		
Sample type	4		**Date shipped (m/d/yr)**		3/14/2016			
Source code	3		**UPS tracking #**		1ZB1T06C0392286034			
Project	306		**Date received (m/d/yr)**		3/15/2016			
			FIELD MEASUREMENTS					
measurable	**Water**	**Visual est.**						
dischar. (y/n)	**color**	**of mixing**						
y	turbid	well						
			units	**meas type**	**avg veloc**	**vtype**		
disch.method	**serial #**	**flow value**	(C,G,M,blank)	(M,E,blank)	(ft/s, blank)	(M,E,blank)		
meter	flow tracker	6.1594	C	M	0.9025	M		
Gage ht.(ft)								
Q inst.(cfs)	6.1594			**hydrograph**	**flow**			
current meter file name		LV030916.WAD		**stage**	**severity**			
current meter discharge(cfs)		6.1594		base	normal			
estimated discharge (cfs)		**estimate type**						
	7	visual						
Volumetric						estimated	adjusted flow	
measurement	vol.(mL)	time (s)	(mL/s)	(gpm)	(cfs)	% loss	(cfs)	
1								
2								
3								
4								
5								
		stable flow ?				**average**		
Weir					calc flow	estimated	adjusted flow	
measurement	stage (ft)				(cfs)	% loss	(cfs)	
1								
2								
3								
4								
5								
		stable flow ?				**average**		
Flume	upstream	dow nstream			calc flow	estimated	adjusted flow	
measurement	stage (ft)	stage (ft)			(cfs)	% loss	(cfs)	
1			Flow type:					
2								
3								
4								
5								
average				**average**				
		stable flow ?						
Discharge comments								

Troll 9500 file name			Core Parameters			Inc Min(°C)	Inc Max(°C)	held < 10°C?
lvk_030916	DO	Baro. Press.	Spc. Cond	Water	pH	35.0	35.0	y
Reading #	(mg/L)	(mm Hg)	(uS/cm@25°C)	temp °C	unitless		Date	Time
1	9.78	633.95	679.72	10.29	8.60	collect	3/9/16	13:00
2	9.76	633.94	679.71	10.29	8.60	start inc.	3/9/16	17:05
3	9.76	633.93	679.67	10.30	8.60	duration held (d)		0.170138889
4	9.77	633.96	679.59	10.31	8.60	read	3/10/16	17:45
5	9.77	633.95	679.63	10.31	8.60	duration inc (d)		1.0278
6	9.76	633.96	679.64	10.31	8.60		data credible?	y
7	9.79	633.97	679.62	10.31	8.60		big wells (+)	sm. well(+)
median	9.77	633.95	679.64	10.31	8.60	Tot. Coli	49	29
range	0.03	0.04	0.13	0.02	0.00	E. Coli	0	0
well mxd?	yes		yes	yes	yes	95L	MPN	95H
DO sat(%)	105.02			Tot. Coli (MPN/100mL)		379.1	579.4	847.2
Turbid(ntu)	171			E. Coli (MPN/100mL)		0	<1	3.7
%Cloud Cov.	0			reagent certificate		EL530	7/22/2016	Certificates\2016
Weather	1			bottle certificate		EL006	4/28/2018	Certificates\2015
Field condns	1			tray certificate		CL018	3/19/2018	Certificates\2015
			CALIBRATION INFORMATION					
		Temperature check						
Field thermometer			Troll 9500	Troll 9500		conversion formulas		
air temp °C	air temp °F		air temp °C	air temp °F		(F - 32) x 5/9 = C		
12.0	53.6					(C x 1.8) + 32 = F		
Field thermometer			Troll 9500	Troll 9500			enter temp	
H2O temp °C	H2O temp °F		H2O temp °C	H2O temp °F		C to F		
10.5	50.9		10.31	50.558		F to C		
Field thermometer:								
Enviro-safe liquid-in-glass thermometer, 0.5 °C increments					Internal (NCPN) Notes			
or digital (thermistor-type) thermometer								
Spin test	minutes	seconds	date	pass?				
pygmy			3/9/2016					
price AA								
			Signal avg					
Turbidity	date	time	(on/off)					
	3/9/2016	17:00	on					
most recent calibration		2/9/2016						
Secondary gelex standards (ntu)			acceptable range (+/- 3%)		%rel diff.			
6.27	reads	6.34	6.08 - 6.46		1.12			
51.7	reads	51.7	50.1 - 53.3		0.00			
497	reads	498	482 - 512		0.20			
			Is recalib. necessary?		no			
Photo Log	Photos taken?	y						
File Name	*Photo Type*	*View*	*Comments*					
P3090004	digital	upstream						
P3090006	digital	upstream						
P3090007	digital	downstream						
Conductivity calibration								
Cond of std used for calibration			1413					
Cond. reading prior to calibration			1429		Cond. post calibration		1413	
manufacturer			Aqua Solutions			abs. diff.	0.00	
lot #			5032659			calib good?	USGS/NPS	
expiration date			3/30/16					
catalog #			6920-4L					
CAS#			see MSDS					
pH Calibration		pH buffer temp (°C) =	11.46					
pH low			7.06		pH high		10.16	
pH prior to calibration			7.02		pH post calibration		10.17	
manufacturer			Aqua Solutions				Aqua Solutions	
lot #			4120872				50119113	
expiration date			12/30/16				1/30/17	
catalog #			1500-4L				1525-4L	
CAS#			see MSDS				see MSDS	
DO Field Calibration: 100% DO 1-point calibration						abs. diff.	0.01	
DO pre calib		mg/L				calib good?	USGS	
DO post calib		mg/L						
abs. diff		mg/L						
calib good?								
DO stimulus		mg/L						

24. Appendix E: 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

25. Appendix F: Decontamination Procedure

Decontamination Considerations During and After Field Activities

National Park Service
Northern Colorado Plateau Network Inventory and Monitoring Program
Water Quality

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 dependent on location and section 2 is meant as a general 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 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.

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, picnickers, 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 Procedure for Aquatic Invasive Species

Decontaminate all equipment, personal items, and clothing that have come into contact with water or mud to minimize the potential for spreading of exotic aquatic invasive species between streams.

A. Remove all mud and debris using a scrub brush.

B. Make a 5% disinfectant solution of quaternary ammonium compound. This is a common cleaning agent used in homes, swimming pools, and hospitals that is 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). Use one of these products and the appropriate amount of tap water to make the 5% disinfectant solution. Cost and effectiveness of the two brands are comparable.

Safety:

- Use protective, unlined rubber gloves and splash goggles or a face shield when handling the solution. Take extra precautions when handling undiluted chemicals. Have an eye wash and clean water available to treat accidental exposure.
- Consult the product label and Material Safety Data Sheet for additional information before working with these products.

Volume of tap water	Volume of *Quat128*	Volume of *Sparquat256*
1000 mL water	46 ml	30 ml
1 gallon water	0.8 cup	0.5 cup

C. Test the 5% solution before each use. The 5% solution needs to be diluted to about 600 parts per million (ppm) of ammonium compounds before it can be tested.

- Measure out **½ cup** of 5% solution made from *Quat128* , add **2 cups** of water and mix.
- Test the diluted solution with "Quat Chek 1000" Test Paper. Match the color of the paper with the ppm on the color chart. For optimal disinfection, the diluted solution should have a concentration between 600 and 800 ppm. **Do not use the diluted solution for decontamination. Dispose of the diluted test solution.**
- If the concentration is too weak, adjust the concentration or dispose of the solution properly and make a new solution.

D. Decontaminate sampling equipment. Place the 5% disinfectant solution in a backpack spray pump, and use this to apply the solution to equipment. The solution must be in contact with the surface being sanitized for at least 10 minutes.

E. Rinse sanitized equipment with water. When feasible, allow to dry completely before entering another body of water.

F. Dispose of used disinfectant solution properly. Disposal of used solution must be in compliance with all local, state, and federal regulations. Do not dump the solution into any stream or lake, or on areas where it can migrate into any storm drain, body of water, or sensitive habitat

G. Store unopened *Sparquat 256* and *Quat128* in a cool, dry place, out of direct sunlight. These products will keep for up to two years at temperatures ranging from 32º to 110º F without losing effectiveness.

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>. (http://www.usu.edu/buglab/Content/Files/2007NZMS.pdf, accessed 4/13/15)

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/>.

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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.

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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

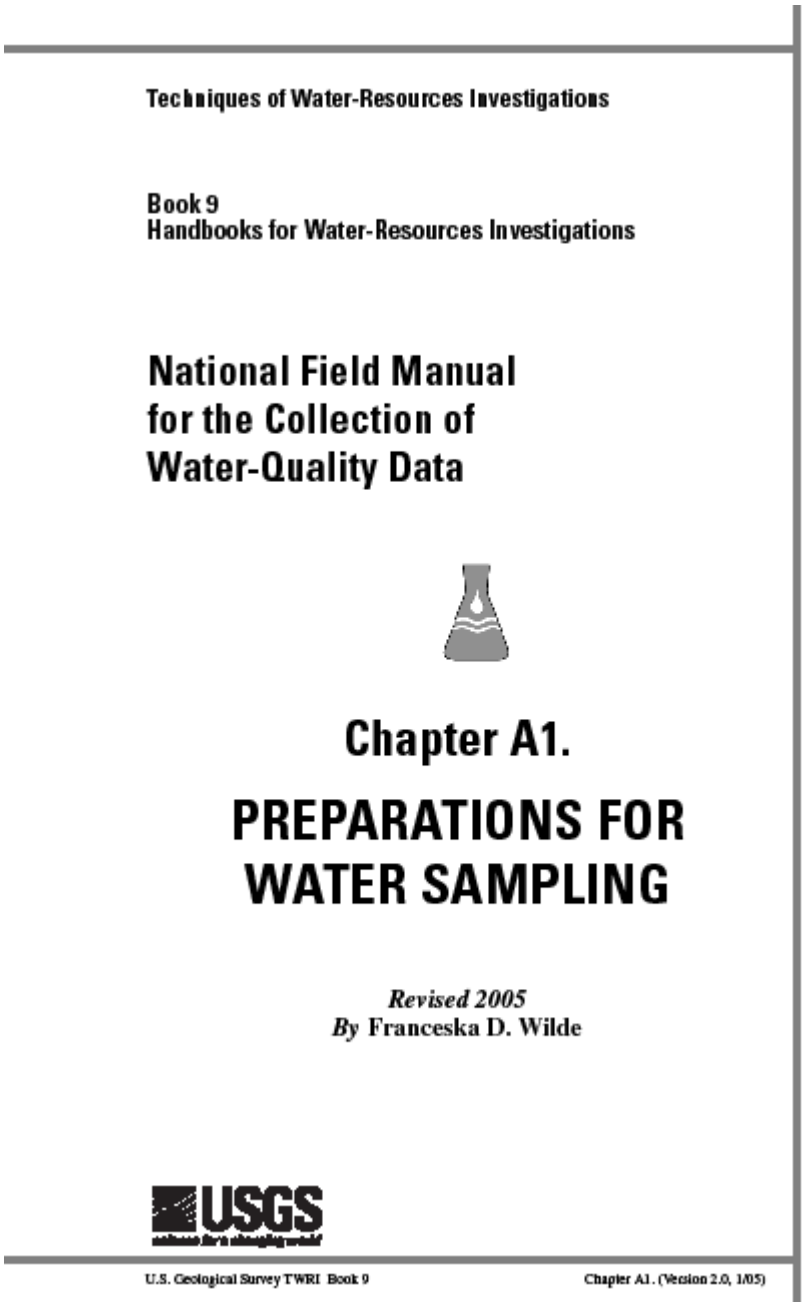
2. Citations

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

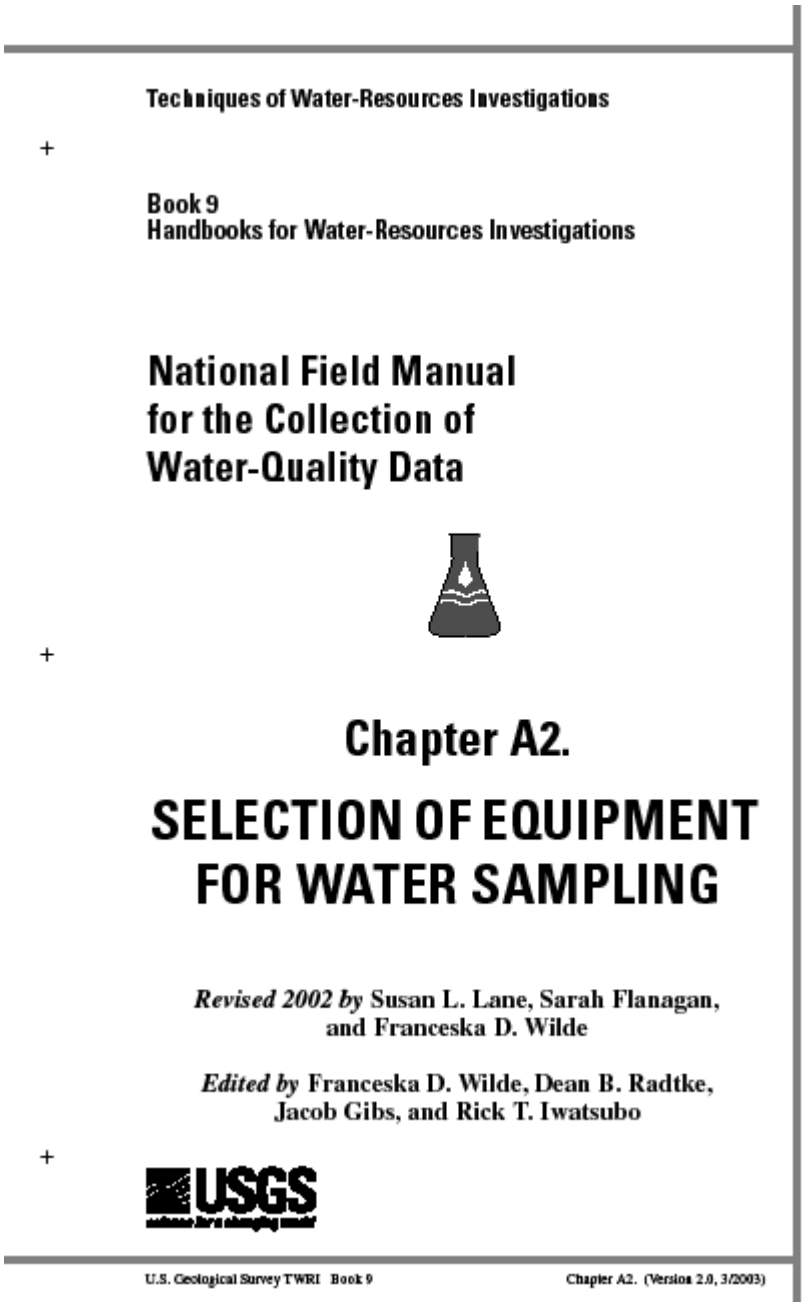
Revised 2005
By Franceska D. Wilde

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)

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Techniques of Water-Resources Investigations

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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

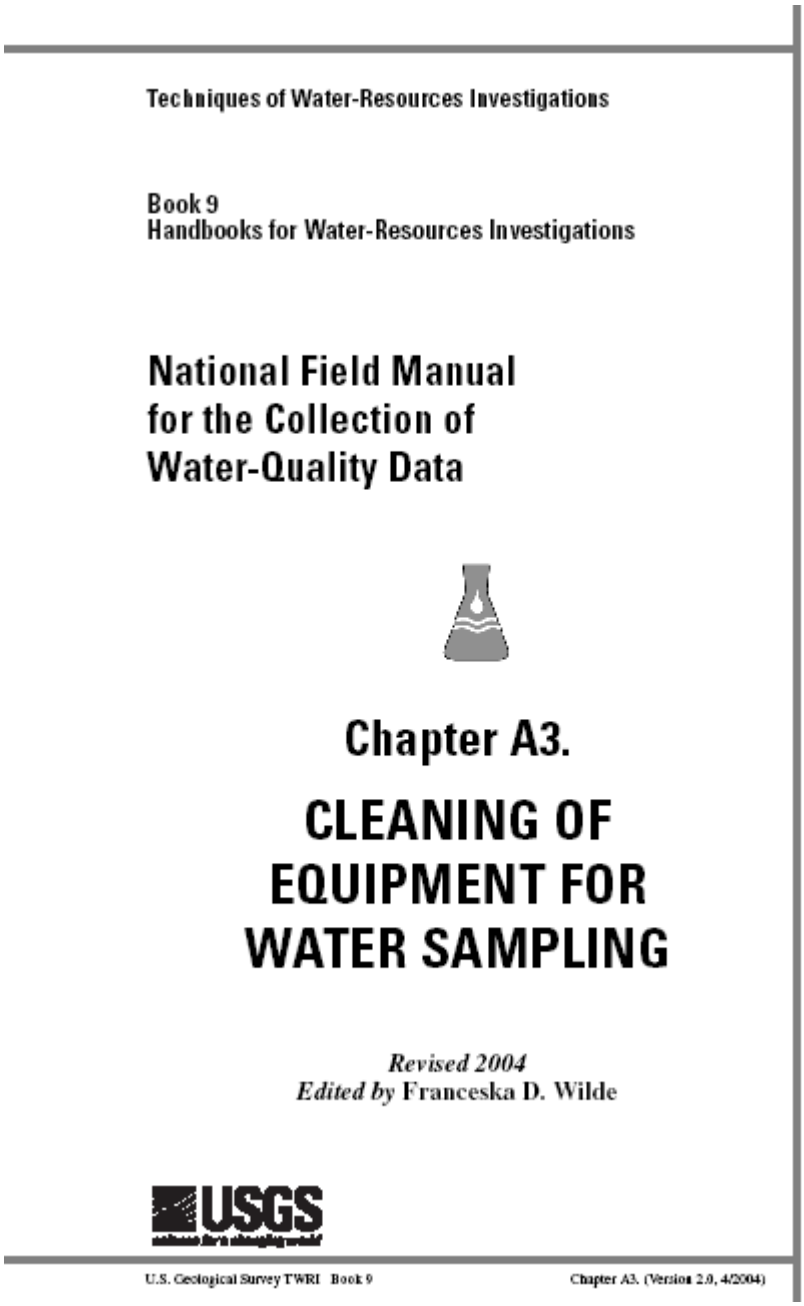
Edited by Franceska D. Wilde, Dean B. Radtke, Jacob Gibs, and Rick T. Iwatsubo

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)

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National Field Manual for the Collection of Water-Quality Data

Chapter A3.

CLEANING OF EQUIPMENT FOR WATER SAMPLING

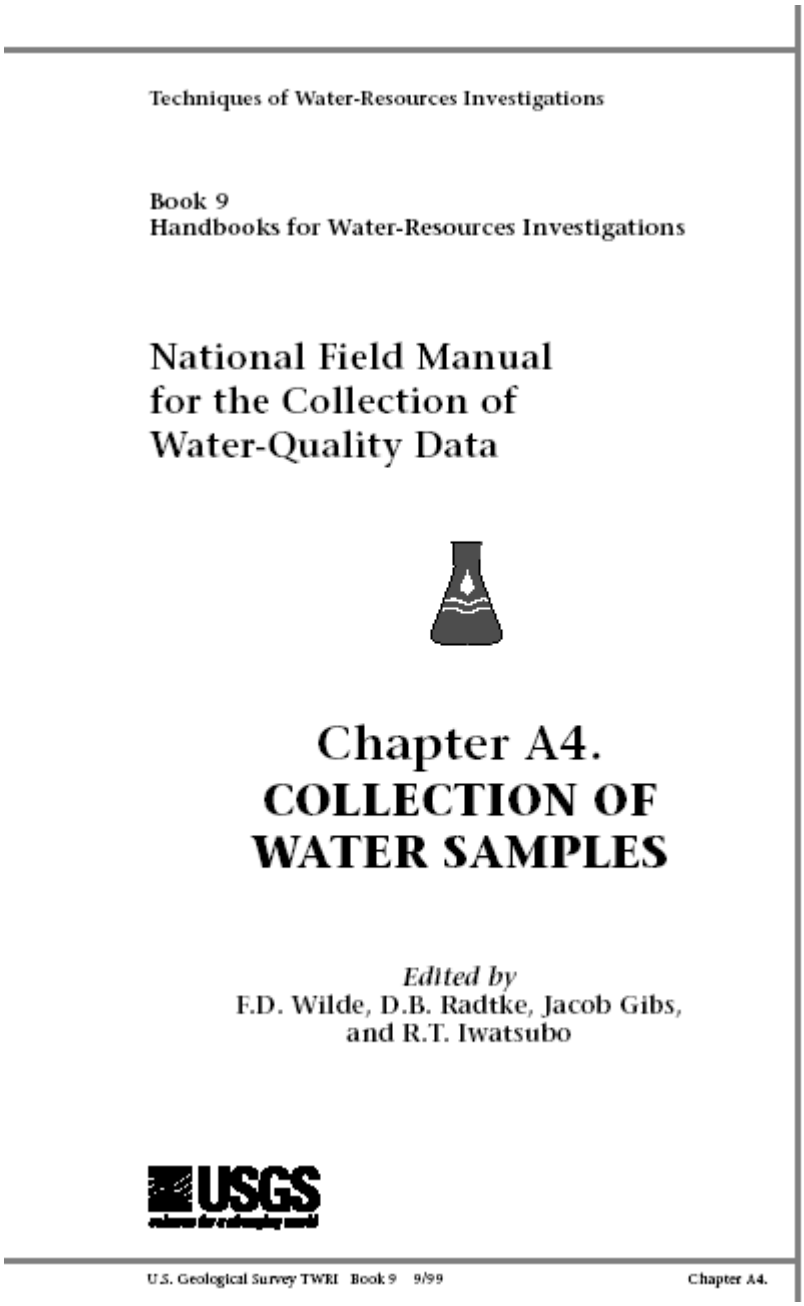
Revised 2004
Edited by Franceska D. Wilde

USGS

U.S. Geological Survey TWRI Book 9 Chapter A3. (Version 2.0, 4/2004)

3.4. Collection of Water Samples (9/99)

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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

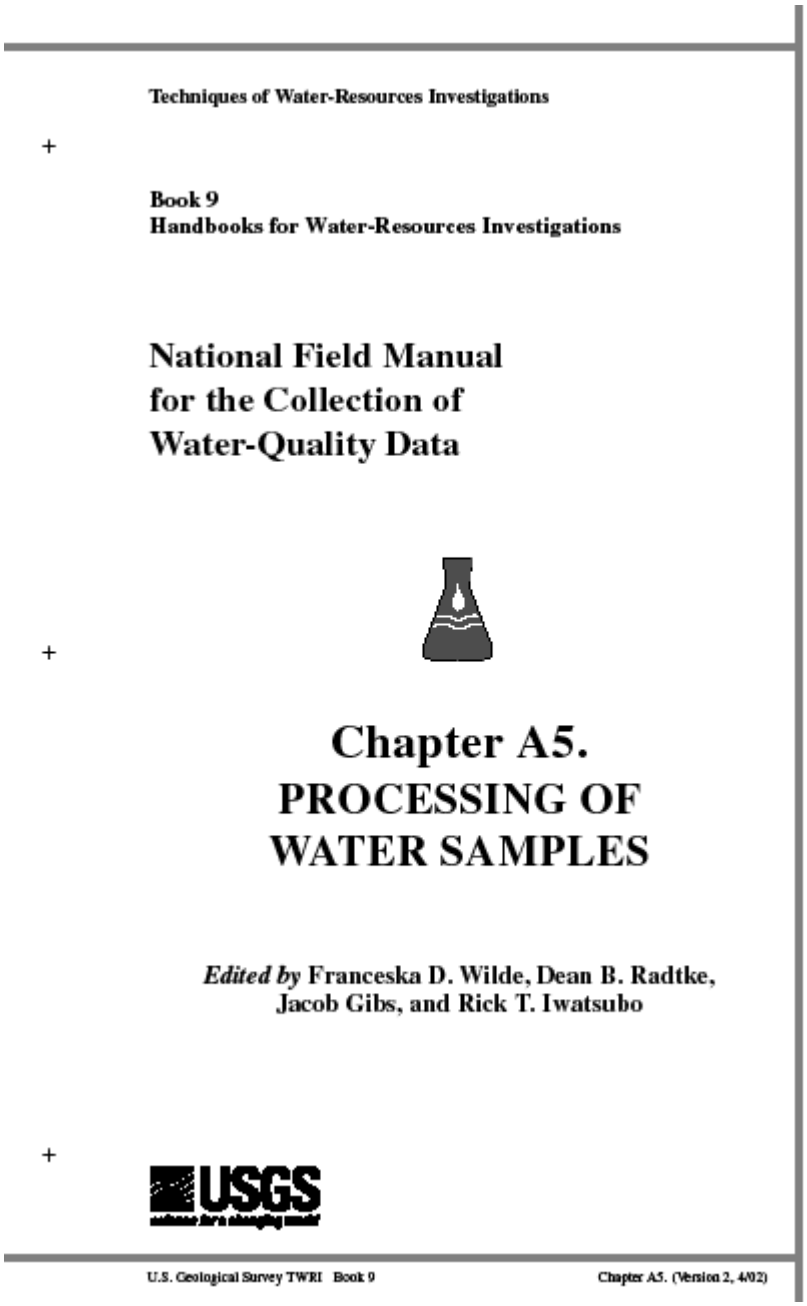
Edited by
F.D. Wilde, D.B. Radtke, Jacob Gibs, and R.T. Iwatsubo

USGS

U.S. Geological Survey TWRI Book 9 9/99 Chapter A4.

3.5. Processing of Water Samples, Version 2.1 (4/04)

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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

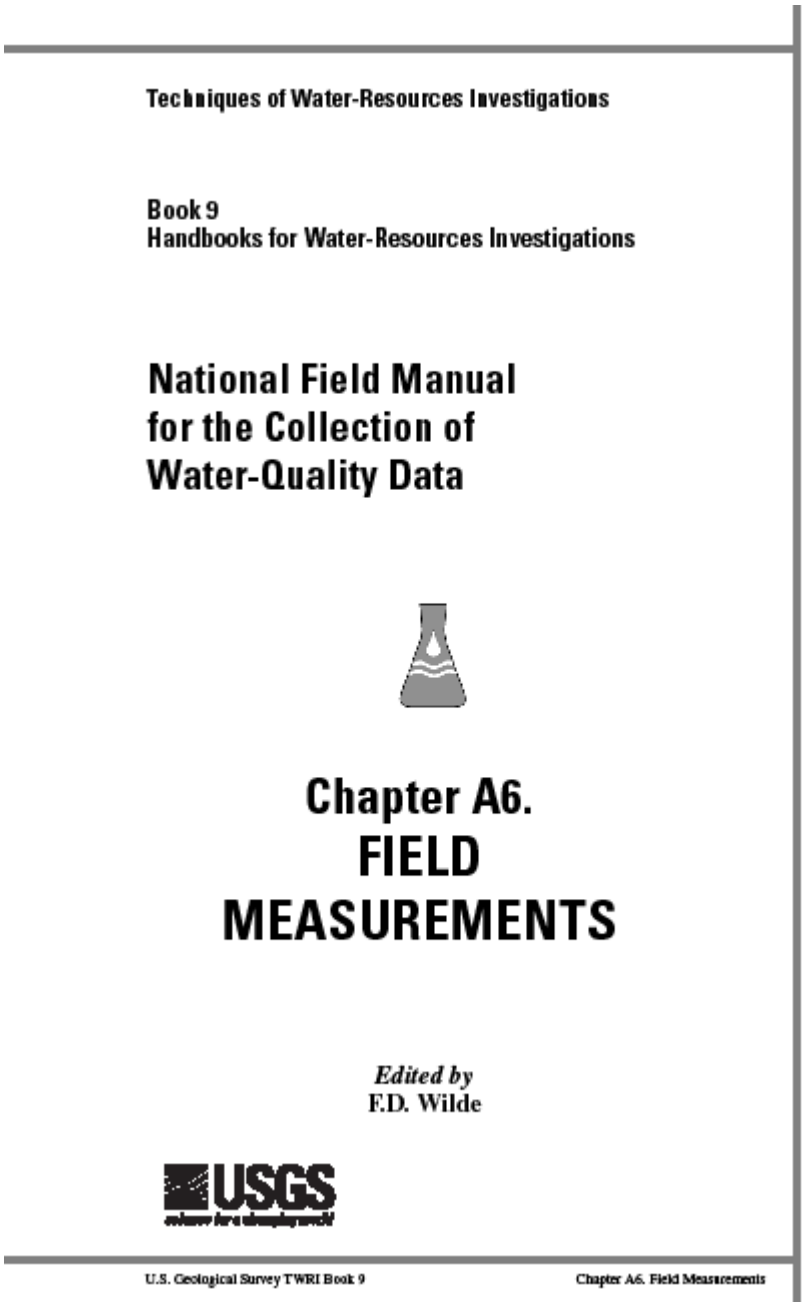
Edited by Franceska D. Wilde, Dean B. Radtke,
Jacob Gibs, and Rick T. Iwatsubo

USGS

U.S. Geological Survey TWRI Book 9 Chapter A5. (Version 2, 4/02)

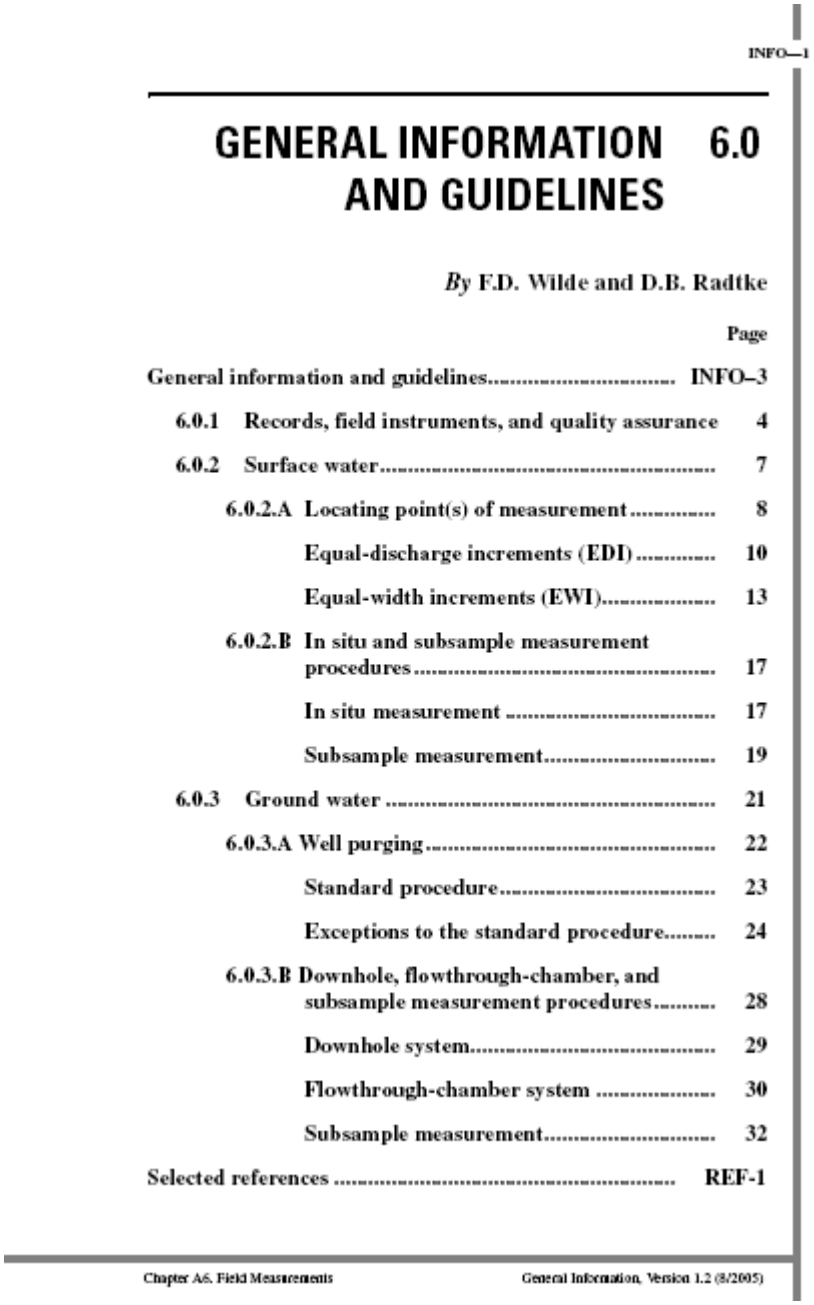
3.6. Field Measurements

CTRL+click the heading to read the chapter online; double-click the picture to open a pdf version.



3.6.1. General Information and Guidelines (Version 1.2, 8/2005)

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By F.D. Wilde and D.B. Radtke

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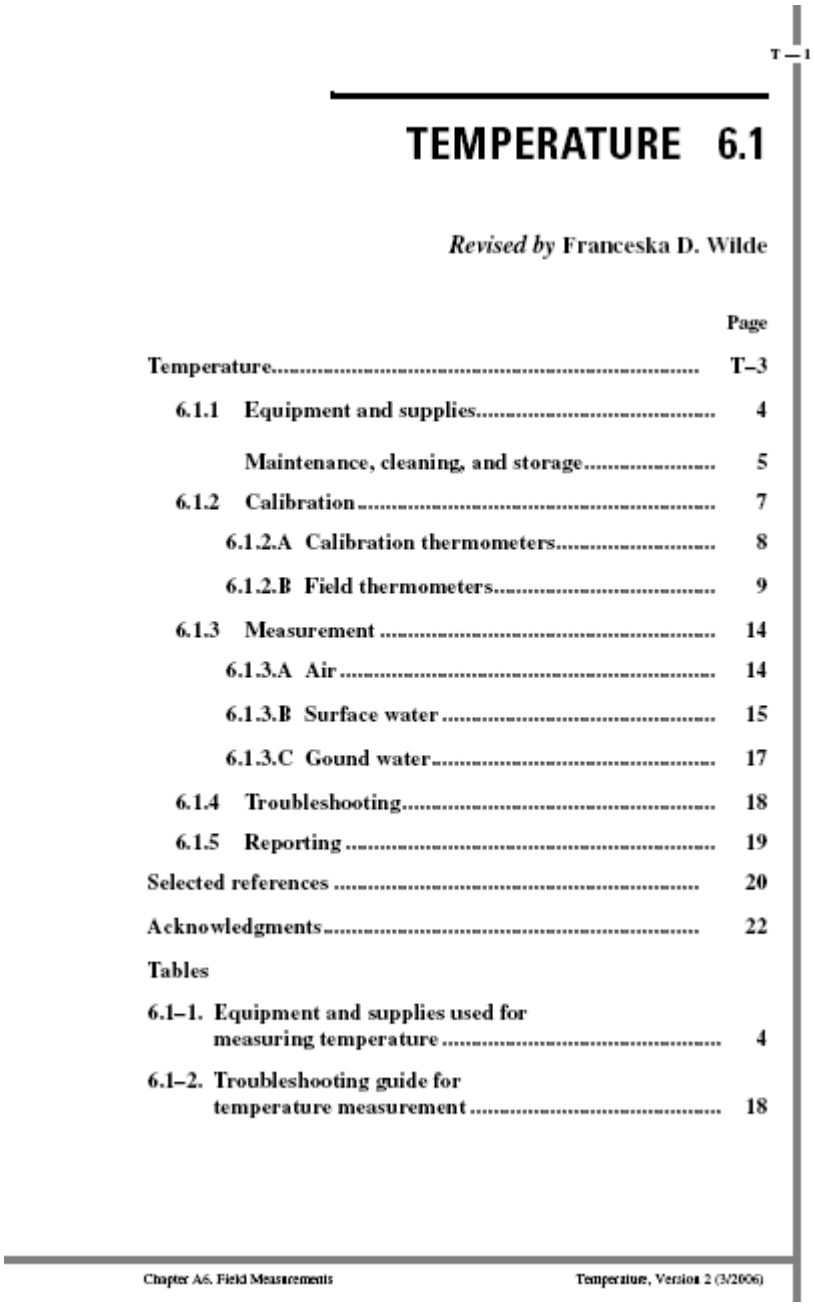
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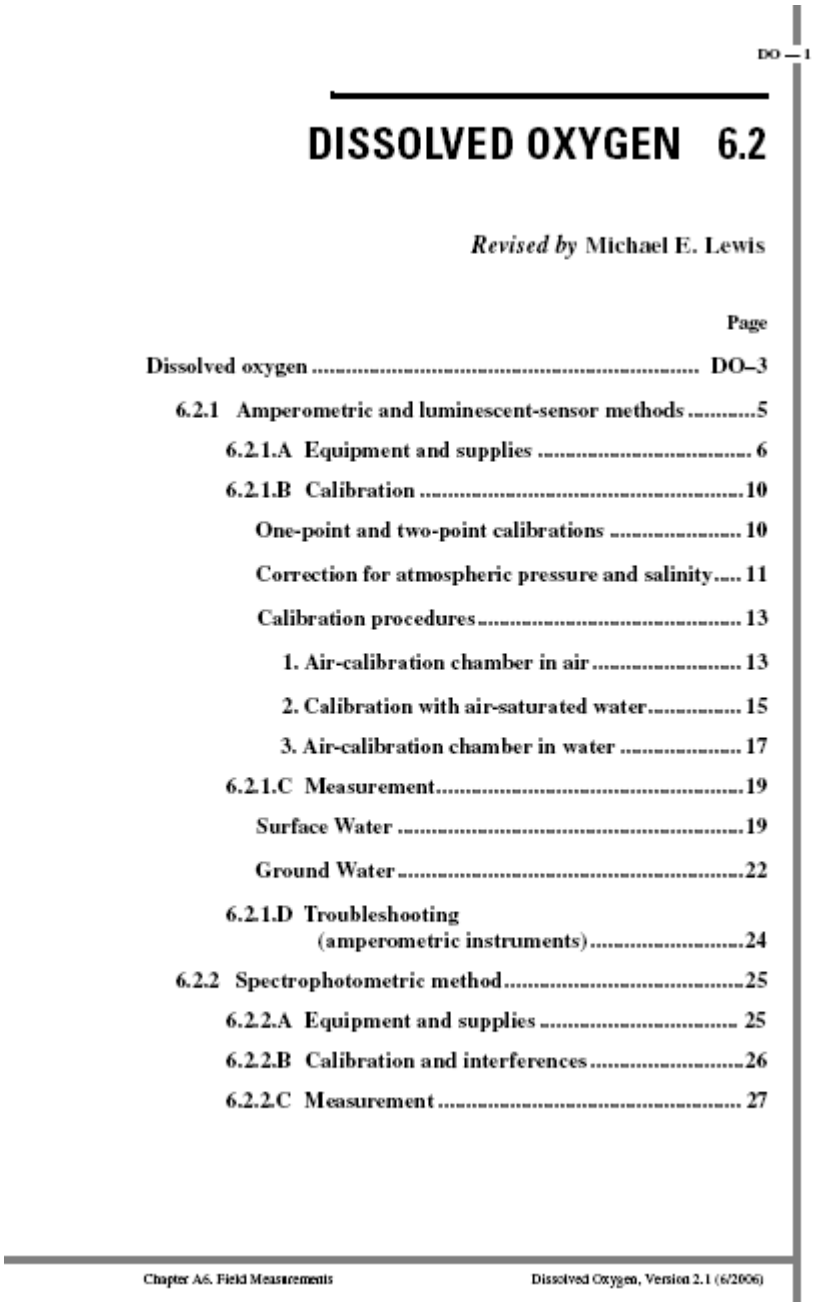
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Revised by Michael E. Lewis

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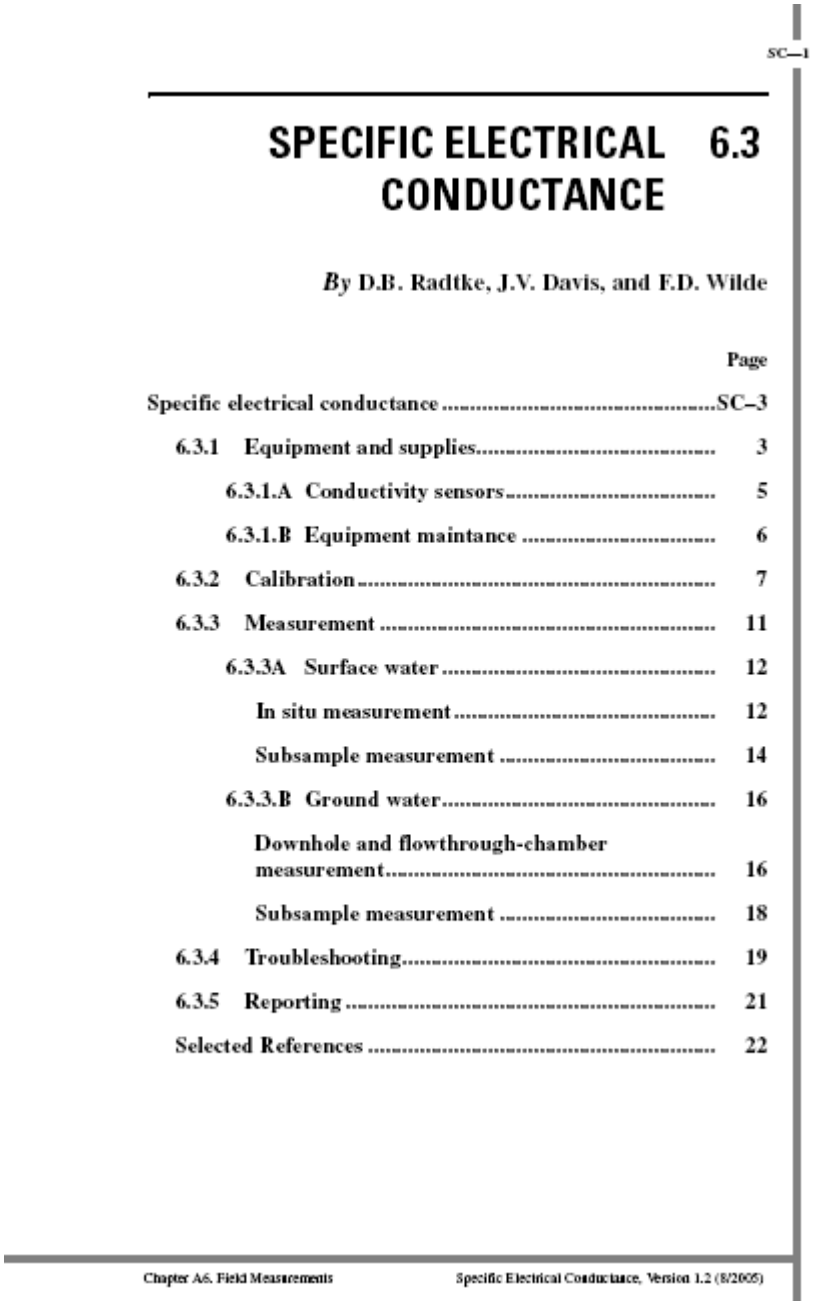
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By D.B. Radtke, J.V. Davis, and F.D. Wilde

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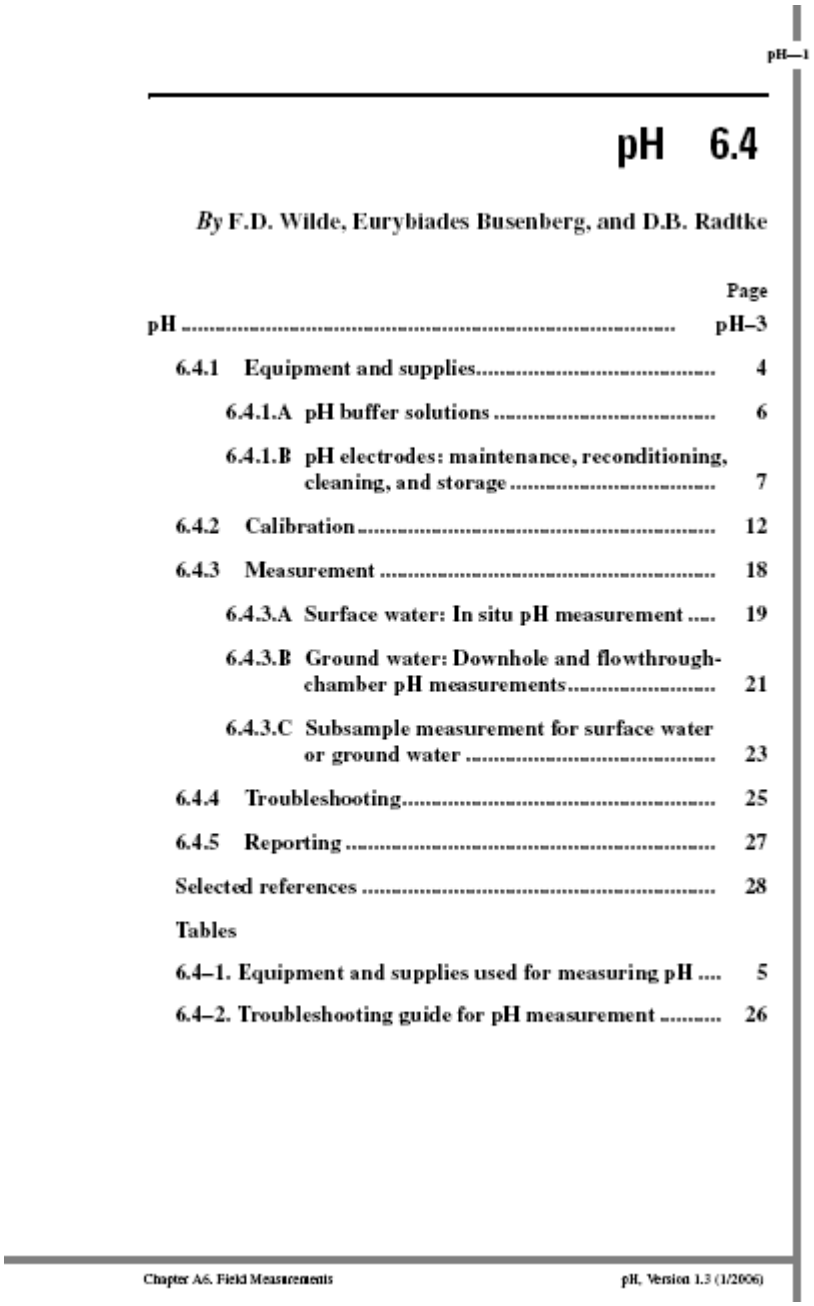
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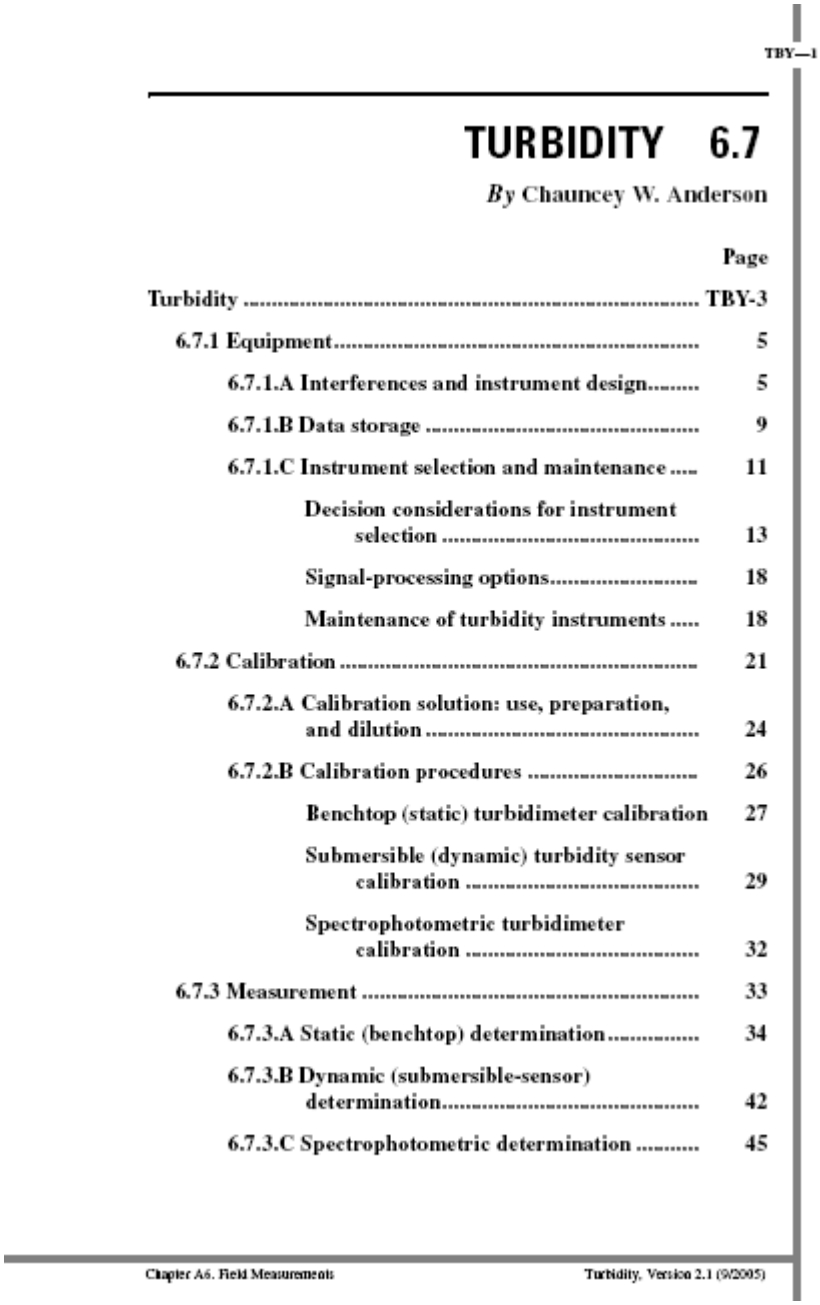
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By F.D. Wilde, Eurybiades Busenberg, and D.B. Radtke

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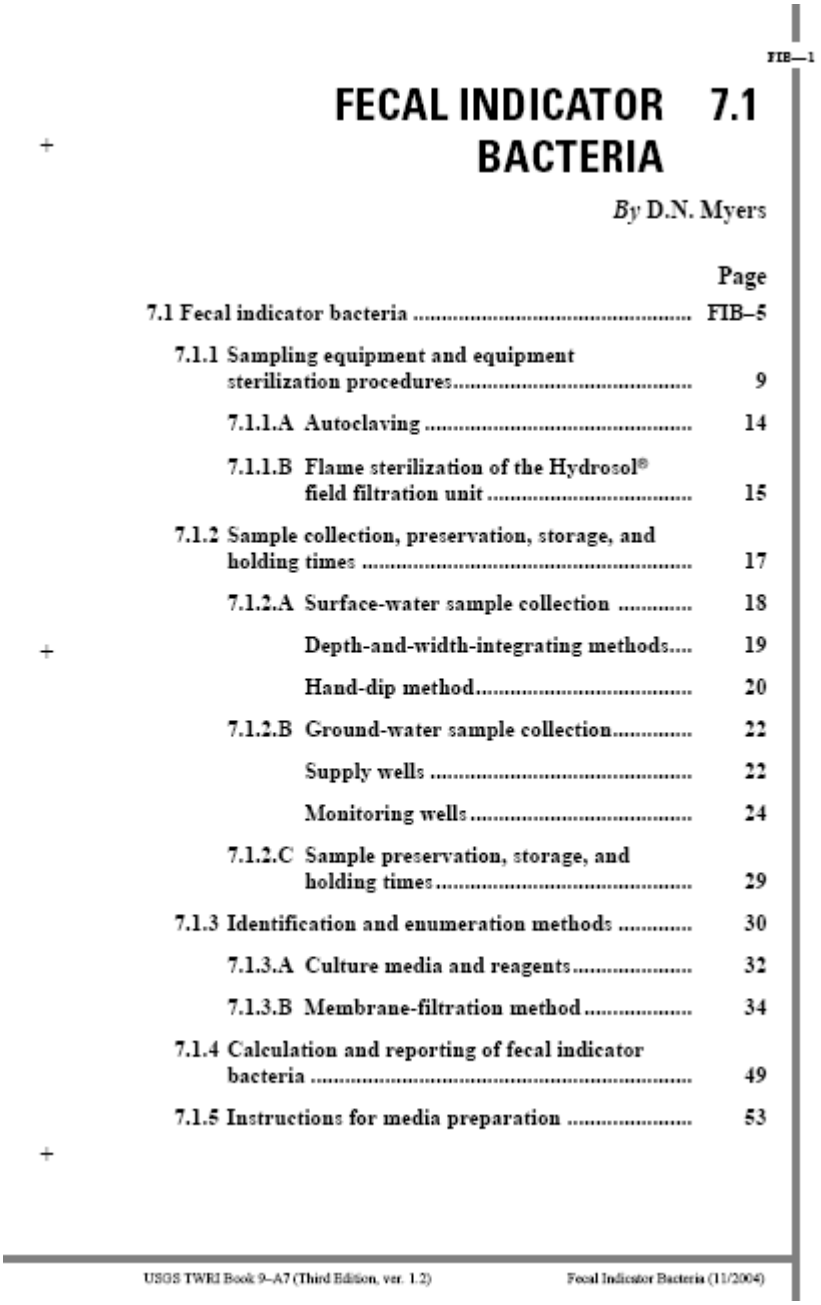
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By Chauncey W. Anderson

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3.6.7. Fecal Indicator Bacteria

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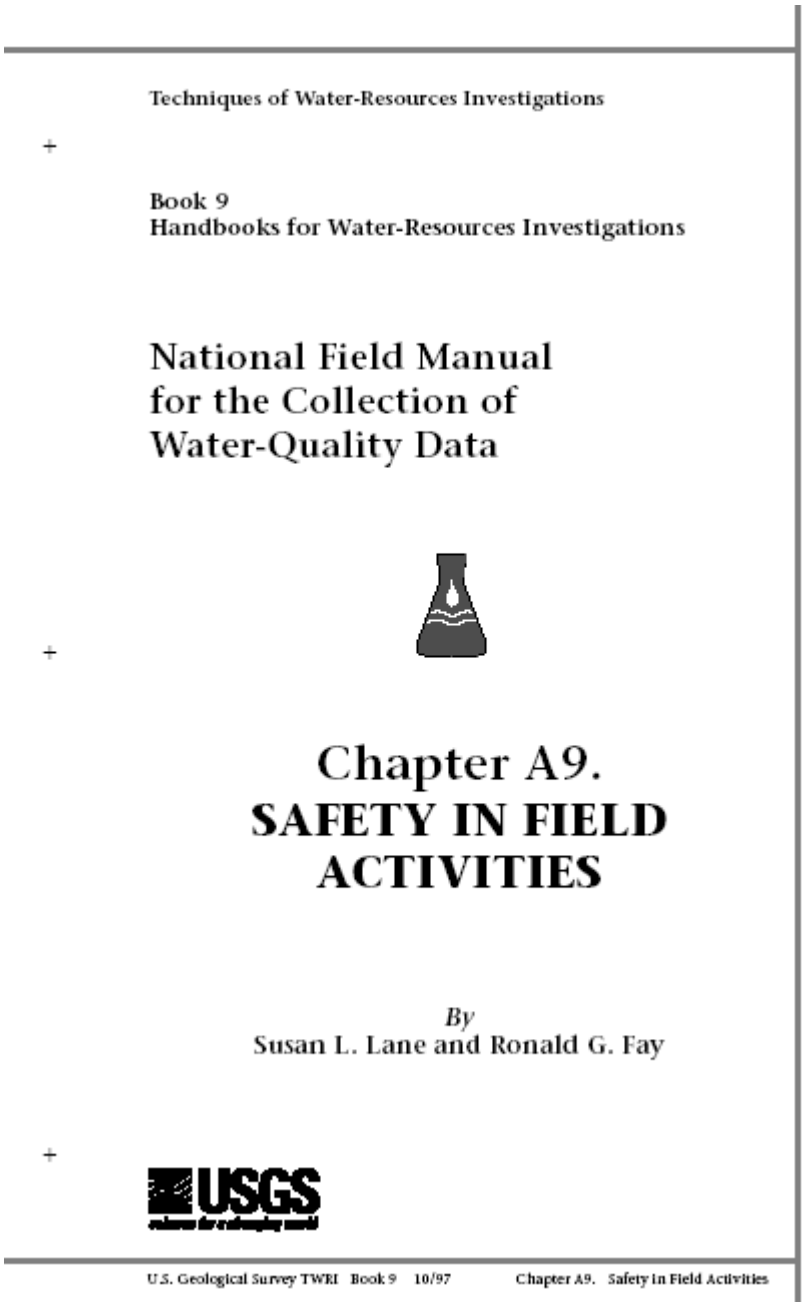
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By D.N. Myers

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3.6.8. Safety in Field Activities (10/97)

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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.

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	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

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	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.

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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 1.02 (4/26/2016)

Revision History Log

Previous Version #	Revision date	Author	Change(s) made	Reason for change	New Version #
1.00	10/2013	C. Hackbarth	Added information about field blanks	QA/QC	1.01
1.01	4/2016	C. Hackbarth / R. Weissinger	Delete obsolete information	Update to current methods	1.02

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.

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1. Introduction

Sample collection for Utah park units is fairly straightforward, but must follow a standard procedure to ensure samples are not contaminated, are stored properly and to ensure 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. Sample bottles:
   a. Chemistry bottle
   b. Filtered metals bottle
   c. Non-filtered nutrient bottle
   d. Filtered nutrient bottle
   Sample bottles have an expiration date printed on the label. Discard expired bottles and request new ones from Utah Public Health Laboratory 801-965-2405.
2. Carry bottle
3. Turbidity vial
4. Permanent marker
5. Field site forms
6. Lab sheets

2.1.2. At vehicle

1. 0.45µm filters
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 permanent marker:

1. STORET number
2. Date
3. Time
4. Sample point name
5. Sampler initials

Record the STORET number and date on each bottle somewhere **outside of the label** in case the label peels or wears off. Do not write on the lids or on shoulder of bottle, this space is for lab use.

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	250 ml	H_2SO_4

4. Sample Collection

After labeling each of the sample bottles:

1. In streams that are well-mixed, samples should be collected in the thalweg when possible. If this area is too deep, choose a point in the channel where water is flowing and appears to be well-mixed (avoid backflows and eddies).
2. Immerse bottle just below surface (if possible) to fill. Avoid introduction of scum or floating debris on the water surface by quickly submerging sample container underwater. Avoid touching the bottle to the stream bottom.
3. Streams that are not well-mixed or large rivers see Appendix A – Methods of sampling based on discharge.
4. Do not overfill as acid preservative may be lost.
5. To maintain acid preservative, do not rinse in stream. Fill acid preserved sample bottles from a carry bottle rinsed 3 times with stream water.
6. There is no requirement for composite samples, because most streams will be thoroughly mixed.
7. Do NOT fill the Filtered Metals or Filtered Nutrients 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 at each site.

1. Rinse the carry bottle 3 times with sample water before collecting sample.
2. Filter nutrients and metals as soon as possible after collection.
3. Use a new piece of tubing for each site.
4. Attach filter to outflow end of tubing with attention to flow direction labeled on the filter pack
5. Rinse inflow end of tubing exterior with sample water before inserting in carry bottle.
6. Insert inflow end into carry bottle
7. Invert filter pack to 45 degree angle and turn on peristaltic pump
8. Rinse tubing and filter with sample water for 10-15 seconds.

9. 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.
10. Filter directly into the filtered metals and or filtered nutrient sample bottle.

6. Replicate Samples

Two (or more) samples taken closely in time and space collected into the appropriate sample containers and preserved, stored, handled, and analyzed identically to regular samples. This sample evaluates cumulative effects of both field precision (natural variation in the stream, field sample collection and/or processing equipment, sample collection technique, sample container and preservative, sample handling, shipment or transport to the lab) and lab precision (sample handling and analysis at the laboratory).

7. Field Blanks

Field blanks are samples that are prepared in the field by pouring DI water into each appropriate sample container containing the appropriate preservative. The filtered field blanks are processed the same way as regular routine samples. These blanks are performed after equipment decontamination and before the next sample collection. The sample is stored, handled, and analyzed identically to the regular samples. Field blanks evaluate contamination resulting from airborne particulates or fumes, handling of the sample container by the sampler, sample container and preservative, sample handling and shipment or transport to the lab, as well as sample handling and analysis at the laboratory. The filtered field blanks also evaluate contamination resulting from field sample collection and/or processing equipment, and sample collection technique. It is important that *every* piece of equipment that normally touches a sample is incorporated into the equipment blank sampling process.

8. Bacteria

See SOP 11

9. Holding Samples

1. Hiking samples out without ice is okay 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.

10. Data Forms

10.1. Paper

Blank forms to send with samples to the laboratory can be found in Appendix B. A lab sheet must be sent to the lab for each site that is sampled. The trip sheet only needs to be sent with the last cooler of samples for that month. Remember the chemistry code is 4 for monthly sampling and 3 for quarterly sampling. Quarterly sheets are used in January, April, July, and October.

10.2. Digital

Both forms that accompany samples to the lab are automatically filled in using Field_forms.xls. Lab sheets can be found at the bottom of each site page. The trip sheet can be found in the "UT_master" tab. Both can be printed by highlighting the area of interest and printing "selected area".

The two data forms are described in the following sections.

10.2.1. Lab sheets

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.

10.2.2. Trip sheets

Trip sheets summarize lab sheet data and should be submitted with the last set of samples to the lab for the month. The trip sheet is also submitted electronically (see Run sheet digital summary below). Field parameters for each site sampled are reported.

10.3. State of Utah codes

Topic	Code	Instructions
Date	Mm/dd/yyyy	
Time	Military time	
Sample Type	4 Grab sample 10 No Flow or Discharge 11 No Access	
Organization	04 (NPS)	
Project Code	306 (Co-op)	
Weather Condition	1 Clear 2 Overcast 3 Partly Cloudy 4 Wind 5 Fog 6 Dust 7 Light Rain 8 Heavy Rain 9 Hail 10 Light Snow 11 Heavy Snow	
Field Conditions	1 Normal 2 Evidence of	

Topic	Code	Instructions
	Recent High Water 3 Flood 4 Shore Ice 5 Anchor Ice 6 Solid Ice 11 Clear Water 12 Milky Water 13 Cloudy Water 14 Opaque Water	
Velocity		1) Record as measured (**M**) if based on gage or current meter measurement. 2) Record as estimated (**E**) if visually estimated or floating stick method. 3) Leave blank if flume, weir, or volumetric measurement. 4) Bottom of page is for flow calculations if needed 5) 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 Flowtracker 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 <u>before</u> heading out to sample in order to ensure it is working. If not then a discharge measurement will be necessary.
Chemistry	(2) = ions (3) = minerals + metals (filtered) (4) = minerals only (filtered) (2) = nutrients, ammonia, Total P (nonfiltered) (9) = Dissolved P, NO_2+NO_3, dissolved N (filtered) Note: The "Temp, pH and Comment Fields" here are	

Topic	Code	Instructions
	filled in by the lab on arrival	

10.4. Run sheet digital summary

Digital field data summaries are submitted quarterly to UTDWQ using an electronic template. 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.

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.

11. Mailing

1. Samples must be stored on ice or refrigerated until delivery to the laboratory. Do not freeze samples.
2. Ship samples with ice. Do not hold or ship with "blue ice" as it will not chill effectively. Water can be frozen in milk jugs or similar containers for shipment.
3. In a large cooler, place samples upright along inside edge of cooler, pack remaining space with bagged ice. Place bubble wrap on top of ice and samples.
4. Enclose Lab sheets and Trip sheets in a large Ziploc baggie and place on top of ice and bubble wrap inside the cooler.
5. Write the return address with a permanent marker on the inside of the cooler lid.
6. Wrap the cooler circumference top to bottom with packing tape.
7. Ship UPS Ground from Zion or Moab and it will arrive in less than 24 hours. Shipping from Bryce Canyon takes 2 days and is not ideal.
8. Send samples on Monday, Tuesday, Wednesday or Thursday only (no Friday or weekend arrival to lab!) to:

   Utah Public Health Laboratory
   Attn: CEL Sample Receiving
   4431 South 2700 West
   Taylorsville, Utah 84129

9. Lab will return the cooler with a new set of sample bottles.
10. Request a tracking number and receipt from the person who ships the cooler.
11. On the Field_forms.xls file record
    a. Date samples shipped (mm/dd/yr)
    b. UPS tracking #
    c. Date samples received (mm/dd/yy)

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: Utah State Lab Forms

12.1. Lab Sheet – Blank Form

Utah Division of Water Quality								
Cooperative Monitoring Program								
Monitoring Run:								
Trip ID:							Agency	**4**
Collector:							Project Code	**306**
Sampler Contact Information (name and phone number):								
Monitoring Location ID:							Time:	
Description:							Date:	
								MM-DD-YY
Sample Type:	**4**							
Weather Conditions:								
Field Conditions:								
FIELD TESTS								
Sp Cond (µS/cm):								
TEST REQUESTS								
Chemistry:	**2**	Temp:		pH:		Comments:		
Filtered Metals:	**4**	Temp:		pH:		Comments:		
Filtered Metals are a code 3 for **QUARTERLY** trips								
Filtered Nutrients:	**9**	Temp:		pH:		Comments:		
Non-Filtered Nutrients:	**2**	Temp:		pH:		Comments:		
Field Comments:								

12.2. Lab Sheets – Example Form

Utah Division of Water Quality								
Cooperative Monitoring Program								
Monitoring Run:	**NPS ZION ET AL MONTHLY**							
Trip ID:	**NPSETAL 051115**						Agency	**4**
Collector:	**Carolyn Hackbarth**						Project Code	**306**
Sampler Contact Information (name and phone number):				Carolyn Hackbarth 435-719-2344				
Monitoring Location ID:	**4951265**						Time:	**13:30**
Description:	**N FK VIRGIN R AT WSA BNDRY**						Date:	**05-17-15**
								MM-DD-YY
Sample Type:	**4**							
Weather Conditions:	**1**							
Field Conditions:	**1**							
FIELD TESTS								
Sp Cond (µS/cm):	**503.3**							
TEST REQUESTS								
Chemistry:	**2**	Temp:		pH:		Comments:		
Filtered Metals:	**4**	Temp:		pH:		Comments:		
Filtered Nutrients:	**9**	Temp:		pH:		Comments:		
Non-Filtered Nutrients:	**2**	Temp:		pH:		Comments:		
Field Comments:								

Use **NPS ZION ET AL QUARTERLY** for months 1,4,7,10

Add date of 1st sample to Trip ID in format MMDDYY

12.3. Trip Sheets – Blank Form

Monitoring Run Sample Summary

Trip ID: NPSETAL **Samplers:**

Trip Description:

Sampler Contact Information (name and phone number):

CHEMISTRY = 10 **FILTERED NUTRIENT = 10**

NON-FILTERED NUTRIENT = 10 **FILTERED METALS = 10**

Seq. #	Project	Monitoring Location ID	Station Description	Date	Time	W temp (°C)	pH	SpCond µS/cm)	DO (mg/L)	DO % sat	Flow (cfs)	E/M
1	306	4951855	Sheep Ck bl Spring in Bryce Cyn NP									
2	306	4951857	Yellow Ck bl Spring in Bryce Cyn NP									
3	306	4951915	MOSSY CAVE SPRING NW OF TROPIC									
4	306	4950800	La Verkin Ck at Lee Pass Trail									
5	306	4951265	N FK VIRGIN R AT WSA BNDRY									
6	306	4951266	N FK VIRGIN R AB CNFL/BAPTISM RIVER									
7	306	4950920	North Ck at Planned Gage in Park									
8	306	4954795	Oak Creek above Sandy Ranch Dam									
9	306	4954780	PLEASANT CK S OF SLEEPING RAINBOW RANCH									
10	306	4954770	SULPHUR CK AB CNFL/FREMONT R IN PICNIC AREA									
11	306	4954775	SULPHUR CK BL ROAD TO GOOSENECKS OVERLOOK IN CR									
12	306	4954812	Halls Ck Bl Narrows									
13	306	5995202	NPSETAL FIELD BLANK									

12.4. Trip Sheets – Example Form

Monitoring Run Sample Summary

Trip ID: NPSETAL 051115 **Samplers:** Carolyn Hackbarth

Trip Description: NPS MONTHLY

Sampler Contact Information (name and phone number): Carolyn Hackbarth 435-719-2344

CHEMISTRY = 10 **FILTERED NUTRIENT = 10**

NON-FILTERED NUTRIENT = 10 **FILTERED METALS = 10**

Seq. #	Project	Monitoring Location ID	Station Description	Date	Time	W temp (°C)	pH	SpCond µS/cm)	DO (mg/L)	DO % sat	Flow (cfs)	E/M
1	306	4951855	Sheep Ck bl Spring in Bryce Cyn NP	5/14/2015	10:30	8.11	7.77	1252.11	8.46	94.02	0.0579	M
2	306	4951857	Yellow Ck bl Spring in Bryce Cyn NP									
3	306	4951915	MOSSY CAVE SPRING NW OF TROPIC	5/13/2015	17:30	10.72	8.13	448.84	8.40	98.04	0.0189	M
4	306	4950800	La Verkin Ck at Lee Pass Trail	5/16/2015	14:00	10.32	8.50	611.57	9.59	103.61	6.4552	M
5	306	4951265	N FK VIRGIN R AT WSA BNDRY	5/17/2015	13:30	17.35	8.52	503.34	8.07	103.22	10.1761	M
6	306	4951266	N FK VIRGIN R AB CNFL/BAPTISM RIVER									
7	306	4950920	North Ck at Planned Gage in Park	5/18/2015	14:00	15.13	8.75	354.78	9.15	105.90	5.8459	M
8	306	4954795	Oak Creek above Sandy Ranch Dam									
9	306	4954780	PLEASANT CK S OF SLEEPING RAINBOW RANCH									
10	306	4954770	SULPHUR CK AB CNFL/FREMONT R IN PICNIC AREA	5/12/2015	16:50	19.18	8.17	1121.63	7.37	98.51	8.3342	M
11	306	4954775	SULPHUR CK BL ROAD TO GOOSENECKS OVERLOOK IN CRI	5/12/2015	13:45	17.15	8.38	1135.33	7.87	102.55	7.0995	M
12	306	4954812	Halls Ck Bl Narrows	5/11/2015	15:00	25.09	8.45	354.95	7.96	111.78	0.3169	M
13	306	5995202	NPSETAL FIELD BLANK	5/17/2015	18:00							

13. Appendix C: Utah Excel Spreadsheet instructions

Instructions for Data Entry into Water Quality Spreadsheet

1. Make a working copy of the water quality spreadsheet. Choose any easy-to - remember name. Don't make changes to the original file.
2. Activity ID. For UTDWQ use only. Leave blank.
3. Trip ID. Agency initials, then MMDDYY, e.g., NPSetal021416 for the monitoring run.
4. Monitoring Location ID. Enter the seven digit STORET number.
5. Date. MM/DD/YY for sample date.
6. Time. HH:MM for sample time.
7. Project. Enter 306 for Co-op monitoring.
8. Sampling Type. Enter 4 (grab), 10 (no flow) or 11 (no access) as appropriate.
9. Organization. Enter 4 for NPS.
10. Weather 1 – 4. Enter up to 4 codes describing weather during the sampling event. Leave unused weather condition code cells blank. Do not repeat the same weather condition code.
11. Field 1 – 4. Enter up to 4 field condition codes. Leave unused field condition code cells blank. Do not repeat the same field condition code.
12. Comment. Enter any comment, e.g. "dead cow in stream".
13. Flow. Enter the numeric value of the flow. Leave this blank if the flow was not measured or estimated.
14. Units. Enter a single upper case letter to indicate type of 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.
15. Utype. Enter either an upper case M to indicate a measured flow value or an upper case E to indicate an estimated flow value. Leave this blank if the flow was not measured or estimated.
16. Velocity. Enter the average velocity of the flow in feet per second. Leave this blank if the velocity was not measured or estimated.
17. Vtype. Enter either an upper case M to indicate a measured velocity value or an upper case E to indicate an estimated velocity value. Leave this blank if the velocity was not measured or estimated.
18. Res_cl. Enter the numeric value of the residual chlorine. Leave this blank if the residual chlorine was not measured.
19. Temp. Enter the numeric value of the water temperature (degrees Celsius). Leave this blank if the water temperature was not measured.
20. pH. Enter the numeric value of the pH. Leave this blank if the pH was not measured.
21. Sp_cond. Enter the numeric value of the specific conductivity (microSiemens per centimeter). Leave this blank if the specific conductivity was not measured.
22. Salinity. Leave this blank.
23. percentDO. Enter the numeric value of the %DO saturation. Leave this blank if the %DO Saturation was not measured
24. DisOxy. Enter the numeric value of the dissolved oxygen (milligrams per liter). Leave this blank if the dissolved oxygen was not measured.

25. Depth. LAKE SAMPLES ONLY. Enter the depth the sample was. Leave blank if this is a stream sample.
26. Relative depth. LAKE SAMPLES ONLY. Leave blank if this is a stream sample.
27. Secchi. LAKE SAMPLES ONLY. Enter the numeric value of the secchi disk reading (meters). Leave this blank if a secchi disk reading was not taken.
28. Turbidity. Enter the numeric value of turbidity (NTU). Leave this blank if turbidity was not measured.
29. Save the File with the appropriate Trip ID.

13.1. Example hard copy of UT Excel spreadsheet

Activity ID	Trip ID	Monitoring Location ID	Date	Time	Project	Sampling Type	Organization	weath1	weath2	weath3	weath4	f
	NPSETAL030916	4950920	3/10/2016	12:00	306	4	4	1				
	NPSETAL030916	4950800	3/9/2016	11:30	306	4	4	1				
	NPSETAL030916	4951915	3/11/2016	11:15	306	4	4	2				

comments	flow	units	utype	velocity	vtype	res_cl	temp	pH	sp_cond	Salinity	percentDO	DisOxy	depth	Rel Dep
	7.02	C	M	3.03	M		16.37	8.57	349.85		102.82	8.65		
	6.16	C	M	0.90	M		10.31	8.60	679.64		105.02	9.77		
	0.057912	C	M				8.56	8.28	439.31		100.2248	9.07		

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 1.03 (April 2016)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.00	6/29/10	Matt Van Grinsven	TOC, Section 7 (Safety and Disposal), and Appendix C	Update disposal methods	1.01
1.01	8/1/12	R. Weissinger	Added Quality Assurance Section 4.4	Add QA measures for Utah sampling; new standard in Utah	1.02
1.02	4/28/16	C. Hackbarth	Added appendix for IDEXX Colilert and Quanti-Tray procedure	Not included in previous version	1.03

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.

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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.

In Colorado, 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.

The state of Utah uses the IDEXX method to enumerate *E. coli*. Unfiltered samples are fed a reagent that targets enzymes specific to coliforms and *E. coli*. They are sealed into a quantification tray and incubated for 24 hours. After that time, wells with total coliforms appear yellow in natural light and wells with *E. coli* fluoresce under ultraviolet light. Positive wells are counted and converted to a most probable number (MPN) per 100 ml of sample.

2. Procedures Used in Park Units

The fecal coliform procedures previously 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 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 has moved 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 IDEXX *E. coli* method used in **Utah** units is presented in a video on the IDEXX web site labeled “Learn how to use Colilert”. **https://www.idexx.com/water/training-videos.html**

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). 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 Quanti-Tray/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 and 1 L Nalgene storage bottle with ice (this will last up to 8 hours even in summer if kept in shade)
Quanti-Tray Sealer with power supply and inverter
Quanti-Trays
Colilert reagent
Deionized/distilled water for field blank
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 (automatically calculated in field form)
Yes or No if sample was kept < 10 deg C from time of collection
Minimum and maximum incubator temperatures
Date and time Quanti-Tray wells read
Number of large and small positive (+) wells under ambient and UV light
Most Probable Number (MPN) from look up table
Lot numbers and expiration dates of Colilert reagent, Quanti-Trays, and bottles

4.1.4. Label sample bottle with:

Site Name
Date of collection

4.2. Sample collection: IDEXX Quanti-Tray/2000 method

Preferences for the location of sample collection are presented in Table 2.

4.3. The IDEXX Quanti-Tray/2000 methodology

The basic steps are as follows:

1. Record date and location on sample bottle.
2. Record date and time on field form.
3. Collect 100 mL in sample bottle.

4. Place sample bottle in 1 L Nalgene storage bottle packed in ice and insulated with water bottle parka for hike to car. Process samples within 8 hours of collection.
5. At the car power up the Quanti-Tray sealer and incubator and allow each to warm up.
6. Remove sample from ice and add Colilert reagent and shake gently until dissolved.
7. Label a Quanti-Tray with date, location, and start time of incubation.
8. Pour sample into Quanti-Tray and run through sealer.
9. Place sealed Quanti-Tray in incubator for 24 hours before reading.

4.4. Quality Assurance

One replicate and one field blank will be analyzed for 10% of samples or once per month, whichever is more frequent. A replicate sample should be collected from source water at the same time and treated in the same way as a regular sample. A field blank uses distilled or deionized water treated in the same manner as a regular sample (kept on ice and processed with other samples).

Holding times (on ice) of samples may not exceed 8 hours prior to processing.

NCPN uses a portable incubator that does not maintain temperatures as consistently as a wall incubator. For this reason, an acceptable temperature range for our sampling is 34 – 36.5º C (IDEXX technical support 7/27/12). At temperatures below 34° the bacteria will not grow as expected, resulting in false negatives. At temperatures above 38° the cell walls of the bacteria begin to break down, also compromising results.

Samples must incubate for at least 24 hours (18 hours if using Colilert-18) prior to being read. If samples are pale yellow, put them back in the incubator for up to four hours prior to reading. Do not exceed the 4 hour limit.

If NCPN sampling is consistently above chronic or acute *E. coli* standards, contact the Utah Department of Environmental Quality to develop a "hotspot" sampling approach. For compliance purposes, Utah QA procedures are more stringent than NCPN's screening procedures and include analyzing field blanks every day of sampling, taking triplicate samples, and maintaining an incubator temperature of 34.5 – 35.5°C.

4.5. Computing reported values: IDEXX Quantitray/2000 method

4.5.1. Total coliforms

Count large and small wells that are positive for total coliforms under ambient light. Use these values to look up MPN for Total Coliforms. Positive wells for total coliforms will display yellow equal to or greater than the comparator. Note: the look up function is built into Field_forms.xls

4.5.2. E. coli

Count large and small wells that are positive for *E. coli* under ultraviolet light. Use these values to look up MPN for *E. coli*. Positive wells for *E. coli* will display yellow and fluorescence equal to or greater than the comparator. Note: the look up function is built into Field_forms.xls

4.5.3. Submit sample location, sample date, observer, total coliform MPN/100 ml, *E. coli* MPN/100 ml, weather and field conditions to the Utah Department of Environmental Quality.

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	Plates outside the Ideal Range are:	Compute Reported Value by:
(20-60 colonies for both FC and 20-80 colonies for E. coli)		(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:

$$cfu/100ml = \frac{total\ colonies\ counted\ x100}{total\ \ ml\ sample\ filtered}$$

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.

$$\frac{[(45+28)\times 100]}{(100+50)ml} = 49\,cfu/100ml$$

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.

$$\frac{[(89)\times 100]}{10ml} = 890\,cfu/100ml$$

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.

$$\frac{[(>60)\times 100]}{1ml} => 6{,}000 cfu/100ml$$

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.

State	Parameter	Domestic source	Recreation and Aesthetics	Recreation and Aesthetics	Agriculture
			----------Beneficial Use----------		
		1C	2A	2B	4
			----------(No.)/100 ML)----------		
UT	E. coli: 0-Day Geometric mean	206	126	206	
UT	E.coli: Maximum	940	576	940	
		CLASS 1a Existing Primary Contact	CLASS 1b Potential Primary Contact	CLASS Secondary Contact	Domestic Water Supply
			----------Beneficial Use----------		
			----------(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

Bacteria samples are processed using the Environmental Protection Agency (EPA) certified IDEXX Quanti-Tray/2000 system. This process involves growing bacteria for enumeration in sealed trays. NCPN processes between 15-30 bacteria samples per month. Each sample results in a tray that may contain hazardous microbes at the end of each test. The used trays are stored in isolated and biohazard labeled containers until properly sterilized. The incubation process may generate cultures of *E. coli*, and may also encourage the growth of colonies (or media) that may contain enteric pathogens, such as Salmonella, Shigella, and Legionella. Microbes generated during the incubation process are considered a biohazard, and require special handling and sterilization prior to disposal.

The NCPN sterilization methods were developed in consultation with the Utah Division of Epidemiology & Laboratory Services, the former NCPN program manager Thom O'Dell, a PhD mycologist, and are procedures certified by the World Health Organization. The sterilization method involves the use of a pressure cooker type autoclave to kill bacteria in a high pressure steam bath. Used bottles and trays are placed in biohazard autoclave bags, and heated in a pressure cooker for 20-30 minutes at 15 lbs PSI (121 ^{o}C). The autoclave bags have an indicator strip that reads "Contents have been AUTOCLAVED" when exposed to adequate sterilization conditions. Once this sterilization process is complete, all microbial (hazardous) contents within the biohazard bags have been completely destroyed, and are suitable for general waste disposal or recycling.

The World Health Organizations (WHO) report Standard Operating Procedures for Microbiology contains guidelines for disinfection and sterilization procedures. Chapter 3 in Section A of this report describes sterilization and disinfection protocol for a variety of clinical technology equipment, including equipment associated with the bacteriological examination of water (Chapter 22 in Section B). The methods used by NCPN to render biohazard materials non-infectious are described in detail in Chapter 3 Section A: of the WHO Microbiology SOP report (Appendix C).

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)**

World Health Organization Guidelines on Standard Operating Procedures for Microbiology. Web accessed on 6/3/2010. **http://www.searo.who.int/en/Section10/Section17/Section53/Section482.htm**

9. Appendix A. Standard Methods for the Examination of Water and Wastewater, Applicable Portions of Section 9000.

Extracted and reproduced 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 0.02*N* 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 boilover 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-

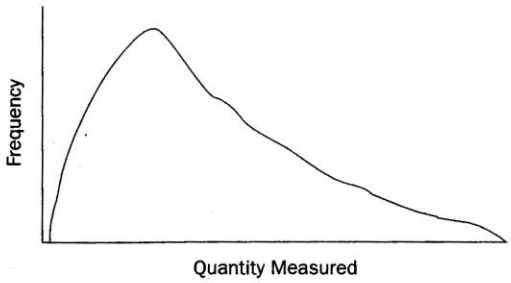


Figure 9020:1. Frequency curve (positively skewed distribution).

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

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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,[2] 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.

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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.

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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

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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.

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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.

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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

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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.

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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).

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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 grid-marked 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:

$$\text{Upper limit} = c + 2\sqrt{c} \quad \text{Lower limit} = c - 2\sqrt{c}$$

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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.

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10. Appendix B. EPA Method 1604: Total Coliforms and Escherichia coli in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)

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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
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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.

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Method 1604: Total Coliforms and *Escherichia coli* in Water by Membrane Filtration Using a Simultaneous Detection Technique (MI Medium)

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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

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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] $\bar{X}$, 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.

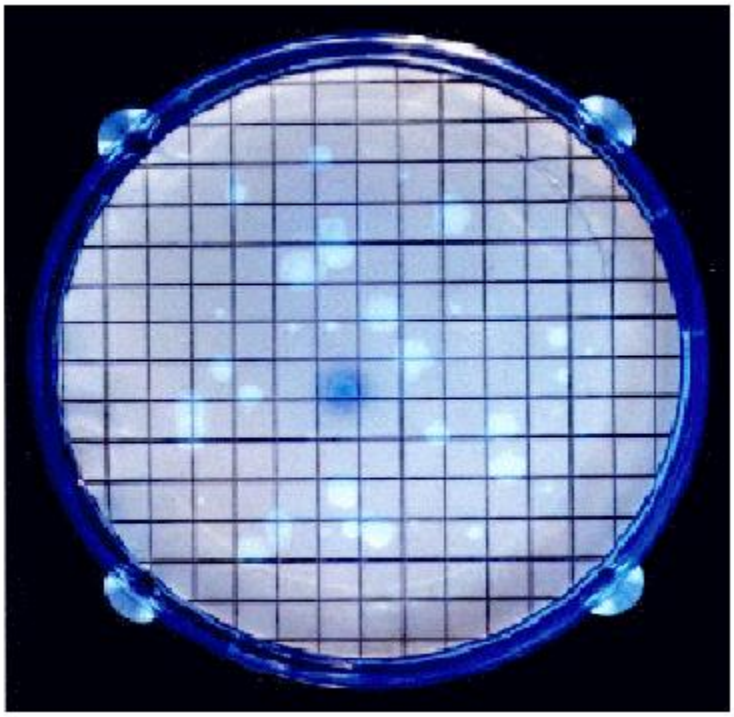


Figure 1. This photograph shows *Escherichia coli* (blue/green fluorescence) and total coliforms other than *E. coli* (blue/white fluorescence) on MI agar under longwave UV light (366 nm). The sample used was a wastewater-spiked Cincinnati, Ohio tap water.

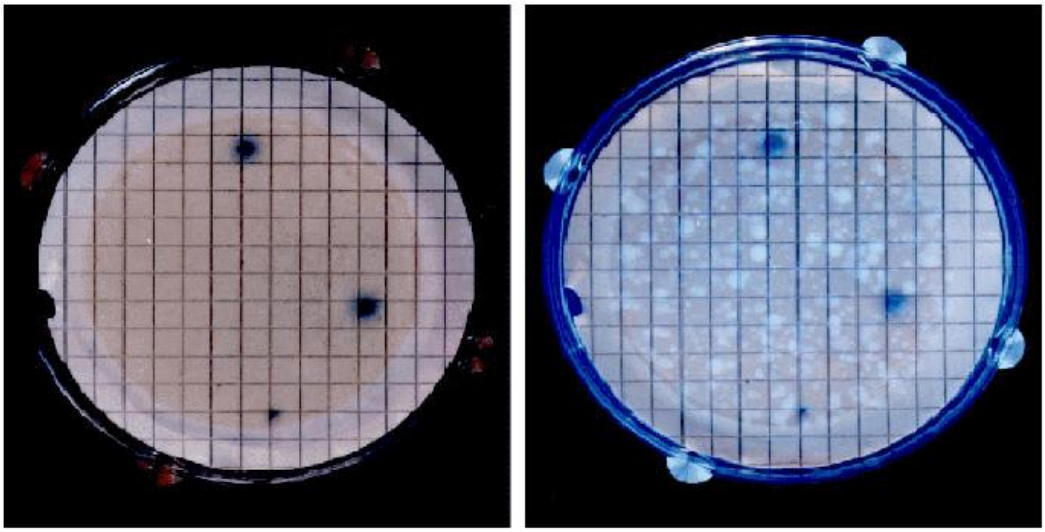


Figure 2. These photographs show *Escherichia coli* and total coliforms from cistern water on MI agar. The confluent plate was photographed under different lighting: ambient light on the left, and longwave UV light (366 nm) on the right. Under ambient light, *E. coli* are blue, and total coliforms other 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).

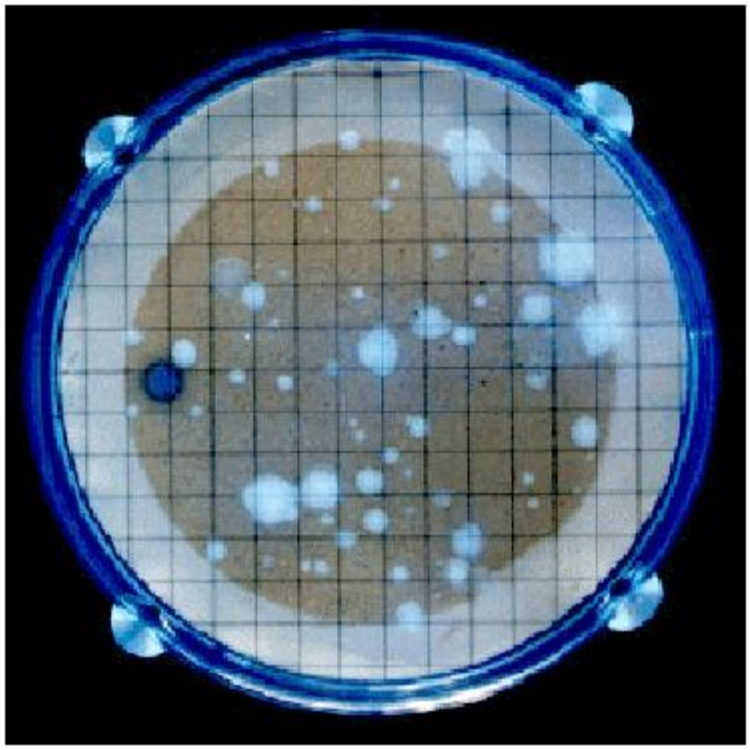


Figure 3. This photograph shows that *Escherichia coli* (blue/green fluorescence) and total coliforms other than *E. coli* (blue/white fluorescence) can easily be detected on MI agar plates from samples with high turbidity levels. The sample used was surface waterspiked Cincinnati, Ohio tap water.

11. Appendix C. Section A: Chapter 3 from the World Health Organization Report: Guidelines on Standard Operating Procedures for Microbiology

3. Sterilization

Sterilization is defined as the destruction or removal (by filtration) of all microorganisms and their spores, whereas **disinfection** is the destruction of many microorganisms but not usually the bacterial spores. Sterilization is usually achieved with the help of heat whereas chemical agents are employed to effect disinfection.

Sterilization and disinfection are part of the daily routine of microbiological laboratories and constitute a vital activity which ensures that cultures, containers, media and equipment are treated in such a way that only the inoculated organisms will grow while all others will be eliminated.

Sterilization by heat

This can be achieved by autoclaving, by exposing articles to dry heat in hot air ovens or boiling.

Autoclaving

Autoclaves can sterilize anything that can withstand a temperature of 121°C for 30 minutes. A pressure cooker used in homes for cooking purposes can also be used as a makeshift autoclave.

The containers having clinical material are subjected to heat treatment in the autoclave after which these are emptied and washed and put back into service.

Only autoclaves designed for laboratory work and capable of dealing with a mixed load should be used. Porous load and bottle fluid sterilizers are rarely satisfactory for laboratory work. There are two varieties of laboratory autoclaves:

- Pressure cooker type.
- Gravity displacement models with automatic air and condensate discharge.

Pressure–cooker type laboratory autoclaves

The most common type is a device for boiling water under pressure. It has a vertical metal chamber with a strong metal lid which can be fastened down and sealed with a rubber gasket. An air and steam discharge tap, pressure

gauge and a safety valve are fitted in the lid. Water in the bottom of the autoclave is heated by external gas burners, an electric immersion heater or a steam coil.

Operating instructions

- Ensure that there is sufficient water inside the chamber.
- Load the autoclave and fasten the lid keeping the discharge tap open.
- Adjust the safety valve to the required temperature and turn the heat on.
- Allow the mixture of air and steam to pass out freely till all air has been discharged.
- Close the air discharge tap and let the steam pressure rise within the chamber till it attains a temperature of 121°C (1.5 kg/cm²).
- Hold on the pressure for 15 minutes.
- Turn off the heat and let the autoclave cool.
- Slowly open the air and steam discharge taps after the pressure gauge has reached zero.
- Allow the material to cool before these are handled (usually agar bottles take hours before these become safe to handle).

Autoclave with air discharge by gravity displacement

These are usually rectangular in shape and arranged horizontally. These autoclaves have a jacket around the chamber.

Operating instructions

- Bring the jacket of the autoclave to operating temperature.
- Load the chamber, close the door and open the steam valve so that steam can freely enter the top of the chamber. Air and condensate shall automatically flow out through the drain at the bottom.
- When the drain thermometer reaches the required temperature, allow further period for the load to reach that temperature (this has to be determined initially and periodically for each autoclave).
- Continue the autoclave cycle for the holding period.
- Close the steam valve and let the autoclave cool till a temperature of 80°C is reached.

- Gradually and softly open the autoclave enabling the steam to escape and allow the load to cool further.

Hot air oven

A hot air oven is electrically operated and should be equipped with a fan to ensure uniform temperature inside. The required temperature for sterilization is generally 160°C for one hour.

Operating instructions

- Arrange the material to be sterilized loosely and evenly on the racks of the oven allowing free circulation of air and thereby even heating of the load.
- Do not pack the load tightly since air is a poor conductor of heat.
- Switch on the power supply and control the temperature of the oven by adjusting the thermostat.
- Note the time when the desired temperature is reached (heating-up time).
- Hold the load in the oven at this temperature for a definite period of time (holding period). This is usually 60 minutes at 160°C.
- Do not overheat since it would char the cotton plugs and paper wrappings.

Autoclaves and hot air ovens can be used for disinfection of infectious waste before it is discarded. In addition, waste can be disposed of by boiling in detergent or by burial.

Boiling in detergent

In the absence of an autoclave, most specimen containers can be boiled in water having detergents to decontaminate. This process kills the vegetative bacteria but fails to destroy the spores and certain viruses. The easiest way to get best results is to add washing powder or sodium carbonate crystals, 60 grams to one litre of water in a big container and boil specimen containers in it for a minimum of 30 minutes.

Disinfection

Disinfection can be undertaken either chemically or by boiling. Boiling is an effective method to disinfect equipment e.g. needles and syringes, if autoclaving facilities are not available. Equipment which has already been cleaned should be boiled for 20 minutes. Chemical disinfection is used for heat-sensitive equipment that is damaged at high temperatures. Commonly-

used chemical disinfectants include chlorine releasing compounds; ethyl and isopropyl alcohol, quaternary ammonium compounds and gluteraldehyde.

The synopsis of a few commonly-used disinfectants is given in Table 1.

Preferred methods of sterilization for common articles are given in Table 2.

Decontamination of some of the commonly reusable equipment has been briefly presented in Table 3.

Table 1: ***Disinfectants and their mode of application****

Target	Disinfectant	Strength to use (disinfectant/ material V/V)	Application	Time of exposure
Skin	Ethanol Iodine Povidone iodine Quaternary ammonium comp	70% 1% 1%	Direct Direct Direct Direct	2 minutes 2 minutes 2 minutes 2 minutes
Blood	Cresol (pH 9) Ca hypochlorite	5% 1%	2:1 2:1	6 hours 6 hours
Urine	Cresol (pH 9)	5%	1:1	4 hours
Sputum	Cresol (pH 9)	5%	1:1	4 hours
Faeces	Cresol (pH 9) Hypochlorite (Na/Ca) Ca hydroxide	5% 1% 20%	2:1 3:1 2:1	6 hours 6 hours 6 hours
Work benches	Lysol Cresol Hypochlorite Chloramine-T	5% 1% 5%	Direct Direct Direct Direct	4 hours 4 hours 4 hours 4 hours
Glassware	Hypochlorite	1%	Direct	4 hours
Lab instruments	Hypochlorite Isopropanol	0.1% 70%	Direct Direct	4 hours 4 hours

* Based upon: Basics of quality assurance: WHO/EMRO, 1992, page 162

Table 2: ***Preferred methods of sterilization for common-use articles***

Autoclaving Animal cages Sugar tubes Lab. coats Cotton Filters Instruments Culture media	***Hot air oven*** Glass ware Beakers Flasks Petridish Pipette Slides Glass syringes Test tubes Powders
Rubber Gloves, stopper, tubing	***Wood*** Tongue depressor, applicator

Glass Slides, syringes, test tubes Enamel metal trays Wire baskets	

Table 3: ***Disinfection of specific equipment***

Container/material	Method of choice for decontamination	Alternative method of decontamination
Reusable stool container	Autoclaving 121◦C for 30 minutes	Fill the jar having stool with 5% solution of phenol and keep for 24hours
	Empty into lavatory*	Empty into lavatory*
Reusable containers of CSF, pus, sputum	Autoclaving	Boiling in detergent
Urine bottles (after emptying in lavatory*)	Autoclaving	Fill with 2% phenol or 1% bleach, leave for 4 hours, clean with detergent
Blood containers	Autoclaving	Soak overnight in strong disinfectant(5% cresol; 1% Ca hypochlorite, 1:2 V/V)
Glass microscope slides**	Autoclaving	Soak overnight in 5% phenol

* If the lavatory is connected to a septic tank, phenol or other antiseptics should not be put into the lavatory.

** Glass microscope slides which have been used for the diagnosis of tuberculosis should be discarded after keeping them soaked in detergent overnight.

Biohazard waste management

Waste is defined as any solid, liquid or gaseous material that is no longer used and will either be recycled, disposed of or stored in anticipation of treatment and/or disposal.

Storage

Prior to disposal, all biohazardous waste should be maintained and stored separately from the general waste stream and from other hazardous wastes. The containers used to store biohazardous waste should be leak-proof, clearly labelled with a red or orange universal biohazard symbol and sealed tightly when transported. In certain cases, it may be necessary to double-bag the waste to prevent leakage. Any biohazardous sharps, such as infectious needles and scalpels, must be placed in containers that are puncture-resistant, leak-proof on all sides and the bottom, and close-able. These containers can then be placed in a standard biohazard bag.

Disposal options

There are three main disposal options:

- render the waste noninfectious by autoclaving and dispose it in the general waste stream. If autoclaving is not possible, decontaminate with chemical disinfectants or by boiling for 20 minutes before disposal.
- on-site incineration, if possible.
- transportation of locally-generated waste to a distant appropriate facility.

Incineration is the preferred disposal option. Not only does this method render the waste noninfectious but it also changes the form and shape of the waste. Sterilization is an effective method for decontaminating waste, but it does not alter the appearance of the waste. Steam sterilization in an autoclave at a temperature of 121°C for at least 15 minutes destroys all forms of microbial life, including high numbers of bacterial spores. This type of complete sterilization can also be accomplished using dry heat which requires a temperature of 160-170°C for 2-4 hours. However, it must be ensured that heat comes in contact with the material to be rendered sterile. Therefore, bottles containing liquid material should have loosened caps or cotton plug caps to allow for steam and heat exchange within the bottle. Biohazard bags containing waste should be tied loosely. Once sterilized, biohazardous waste should be sealed in appropriate containers, labelled as disinfected waste and disposed of in an approved facility.

Biological waste should be clearly labelled prior to disposal and complete records should be maintained.

Burial

It is not a decontaminating process *per se*. However, it does prevent the infectious material from becoming a reservoir of infection if properly buried. It requires digging a pit of almost 5 meters depth and 2 meters width and having a tightly fitted heavy lid on top. Disposable containers with clinical material are thrown daily into it and the lid is replaced immediately after throwing the specimens. Once a week, the refuse is covered with a layer of quicklime. If quicklime is not available, the refuse is covered with almost 10 cm thick layer of dried leaves once a week.

Further reading

El-Nageh MM et al. Basics of Quality Assurance for Intermediate and Peripheral Laboratories. WHO Regional Publication, Eastern Mediterranean Series No 2, 156-166, 1992.

12. Appendix D. IDEXX Method: Colilert and Quanti-Tray/2000 Procedures

Colilert* Test Kit

Introduction

Colilert* simultaneously detects total coliforms and *E. coli* in water. It is based on IDEXX's patented Defined Substrate Technology* (DST*). When total coliforms metabolize Colilert's nutrient-indicator, ONPG, the sample turns yellow. When *E. coli* metabolize Colilert's nutrient-indicator, MUG, the sample also fluoresces. Colilert can simultaneously detect these bacteria at 1 cfu/100 mL within 24 hours even with as many as 2 million heterotrophic bacteria per 100 mL present.

Storage

Store at 2–30°C away from light.



Presence/Absence (P/A) Procedure

1. Add contents of one pack to a 100 mL sample in a sterile, transparent, nonfluorescing vessel.
2. Cap vessel and shake.
3. Incubate at 35±0.5°C for 24 hours.
4. Read results according to Result Interpretation table below.



Quanti-Tray* Enumeration Procedure

1. Add contents of one pack to a 100 mL water sample in a sterile vessel.
2. Cap vessel and shake until dissolved.
3. Pour sample/reagent mixture into a Quanti-Tray* or Quanti-Tray*/2000 and seal in an IDEXX Quanti-Tray* Sealer.
4. Place the sealed tray in a 35±0.5°C incubator for 24 hours.
5. Read results according to the Result Interpretation table below. Count the number of positive wells and refer to the MPN table provided with the trays to obtain a Most Probable Number.



Result Interpretation

Appearance	Result
Less yellow than the comparator[1]	Negative for total coliforms and *E. coli*
Yellow equal to or greater than the comparator	Positive for total coliforms
Yellow and fluorescence equal to or greater than the comparator	Positive for *E. coli*

- Look for fluorescence with a 6-watt, 365-nm UV light within 5 inches of the sample in a dark environment. Face light away from your eyes and towards the sample.
- Colilert results are to be read after 24 hours of incubation.
- However, if the results are ambiguous to the analyst based on the initial reading, incubate up to an additional four hours (but not to exceed 28 hours total) to allow the color and/or fluorescence to intensify.
- Positives for both total coliforms and *E. coli* observed before 24 hours and negatives observed after 28 hours are also valid.
- In addition, laboratories may incubate samples for additional time (up to 28 hours total) for their convenience.

Procedural Notes

- This insert may not reflect your local regulations. For compliance testing, be sure to follow appropriate regulatory procedures. For example, samples run in other countries are incubated at 36±2°C for 24–28 hours.
- Colilert can be run in any multiple tube format. *Standard Methods for the Examination of Water and Wastewater*[2] MPN tables should be used to find Most Probable Numbers (MPNs).
- If a water sample has some background color, compare inoculated Colilert sample to a control blank of the same water sample.
- If sample dilutions are made, multiply the MPN value by the dilution factor to obtain the proper quantitative result.
- Use only sterile, nonbuffered, oxidant-free water for dilutions.

Quanti-Tray*/2000

English

Introduction

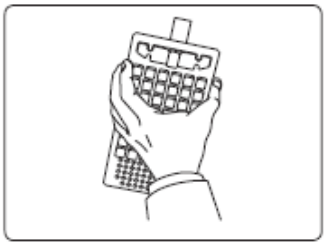
IDEXX Quanti-Tray*/2000 is designed to give quantitated bacterial counts of 100 mL samples using IDEXX reagent products. Add the reagent/sample mixture to a Quanti-Tray/2000, seal it in a Quanti-Tray* Sealer and incubate per the reagent instructions. Count the number of positive large and small wells and use the Most Probable Number (MPN) Table attached to determine the MPN.

Contents

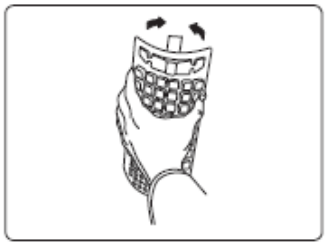
This package contains sterile Quanti-Tray/2000 trays.

User Instructions

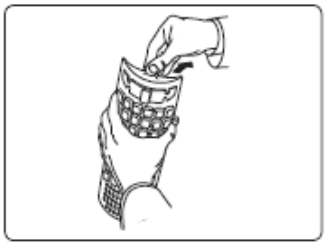
1. Use one hand to hold a Quanti-Tray* upright with the well side facing the palm.



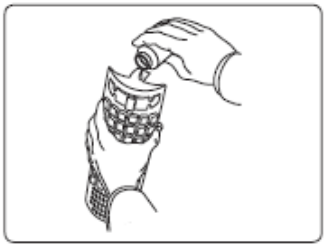
2. Squeeze the upper part of the Quanti-Tray so that the Quanti-Tray bends toward the palm.



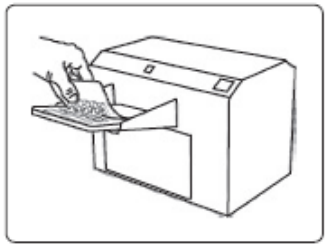
3. Gently pull foil tab to separate the foil from the tray. Avoid touching the inside of the foil or tray.



4. Pour the reagent/sample mixture directly into the Quanti-Tray, avoiding contact with the foil tab. Tap the small wells 2–3 times to release any air bubbles. Allow foam to settle.



5. Place the sample-filled Quanti-Tray onto the Quanti-Tray/2000 rubber insert of the Quanti-Tray Sealer with the well side (plastic) of the Quanti-Tray facing down.



6. Seal according to the Quanti-Tray Sealer instructions.
7. Incubate according to reagent instructions.
8. Count large and small positive wells and refer to the Quanti-Tray/2000 MPN table to find the MPN.†
9. Dispose of media in accordance with good laboratory practices.

For technical support, please call:

North/South America: +1 207 556 4496 or 1 800 321 0207
Europe: +00800 4339 9111
UK: +44 01638 676800
China: +86 21 61279528
Japan: +81 422 71 5921
Australia: +1800 443 399

idexx.com/water

†Download the IDEXX MPN generator software for automated Quanti-Tray results at idexx.com/mpngenerator.

*Quanti-Tray is a trademark or registered trademark of IDEXX Laboratories, Inc. in the United States and/or other countries.
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One IDEXX Drive
Westbrook, Maine 04092 USA

IDEXX Quanti-Tray®/2000 MPN Table

# Large Wells Positive	# Small Wells Positive																								
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
0	<1	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0	11.0	12.0	13.0	14.1	15.1	16.1	17.1	18.1	19.1	20.2	21.2	22.2	23.3	24.3
1	1.0	2.0	3.0	4.0	5.0	6.0	7.1	8.1	9.1	10.1	11.1	12.1	13.2	14.2	15.2	16.2	17.3	18.3	19.3	20.4	21.4	22.4	23.5	24.5	25.6
2	2.0	3.0	4.1	5.1	6.1	7.1	8.1	9.2	10.2	11.2	12.2	13.3	14.3	15.4	16.4	17.4	18.5	19.5	20.6	21.6	22.7	23.7	24.8	25.8	26.9
3	3.1	4.1	5.1	6.1	7.2	8.2	9.2	10.3	11.3	12.4	13.4	14.5	15.5	16.5	17.6	18.6	19.7	20.8	21.8	22.9	23.9	25.0	26.1	27.1	28.2
4	4.1	5.2	6.2	7.2	8.3	9.3	10.4	11.4	12.5	13.5	14.6	15.6	16.7	17.8	18.8	19.9	21.0	22.0	23.1	24.2	25.3	26.3	27.4	28.5	29.6
5	5.2	6.3	7.3	8.4	9.4	10.5	11.5	12.6	13.7	14.7	15.8	16.9	17.9	19.0	20.1	21.2	22.2	23.3	24.4	25.5	26.6	27.7	28.8	29.9	31.0
6	6.3	7.4	8.4	9.5	10.6	11.6	12.7	13.8	14.9	16.0	17.0	18.1	19.2	20.3	21.4	22.5	23.6	24.7	25.8	26.9	28.0	29.1	30.2	31.3	32.4
7	7.5	8.5	9.6	10.7	11.8	12.8	13.9	15.0	16.1	17.2	18.3	19.4	20.5	21.6	22.7	23.8	24.9	26.0	27.1	28.3	29.4	30.5	31.6	32.8	33.9
8	8.6	9.7	10.8	11.9	13.0	14.1	15.2	16.3	17.4	18.5	19.6	20.7	21.8	22.9	24.1	25.2	26.3	27.4	28.6	29.7	30.8	32.0	33.1	34.3	35.4
9	9.8	10.9	12.0	13.1	14.2	15.3	16.4	17.6	18.7	19.8	20.9	22.0	23.2	24.3	25.4	26.6	27.7	28.9	30.0	31.2	32.3	33.5	34.6	35.8	37.0
10	11.0	12.1	13.2	14.4	15.5	16.6	17.7	18.9	20.0	21.1	22.3	23.4	24.6	25.7	26.9	28.0	29.2	30.3	31.5	32.7	33.8	35.0	36.2	37.4	38.6
11	12.2	13.4	14.5	15.6	16.8	17.9	19.1	20.2	21.4	22.5	23.7	24.8	26.0	27.2	28.3	29.5	30.7	31.9	33.0	34.2	35.4	36.6	37.8	39.0	40.2
12	13.5	14.6	15.8	16.9	18.1	19.3	20.4	21.6	22.8	23.9	25.1	26.3	27.5	28.6	29.8	31.0	32.2	33.4	34.6	35.8	37.0	38.2	39.5	40.7	41.9
13	14.8	16.0	17.1	18.3	19.5	20.6	21.8	23.0	24.2	25.4	26.6	27.8	29.0	30.2	31.4	32.6	33.8	35.0	36.2	37.5	38.7	39.9	41.2	42.4	43.6
14	16.1	17.3	18.5	19.7	20.9	22.1	23.3	24.5	25.7	26.9	28.1	29.3	30.5	31.7	33.0	34.2	35.4	36.7	37.9	39.1	40.4	41.6	42.9	44.2	45.4
15	17.5	18.7	19.9	21.1	22.3	23.5	24.7	25.9	27.2	28.4	29.6	30.9	32.1	33.3	34.6	35.8	37.1	38.4	39.6	40.9	42.2	43.4	44.7	46.0	47.3
16	18.9	20.1	21.3	22.6	23.8	25.0	26.2	27.5	28.7	30.0	31.2	32.5	33.7	35.0	36.3	37.5	38.8	40.1	41.4	42.7	44.0	45.3	46.6	47.9	49.2
17	20.3	21.6	22.8	24.1	25.3	26.6	27.8	29.1	30.3	31.6	32.9	34.1	35.4	36.7	38.0	39.3	40.6	41.9	43.2	44.5	45.9	47.2	48.5	49.8	51.2
18	21.8	23.1	24.3	25.6	26.9	28.1	29.4	30.7	32.0	33.3	34.6	35.9	37.2	38.5	39.8	41.1	42.4	43.8	45.1	46.5	47.8	49.2	50.5	51.9	53.2
19	23.3	24.6	25.9	27.2	28.5	29.8	31.1	32.4	33.7	35.0	36.3	37.6	39.0	40.3	41.6	43.0	44.3	45.7	47.1	48.4	49.8	51.2	52.6	54.0	55.4
20	24.9	26.2	27.5	28.8	30.1	31.5	32.8	34.1	35.4	36.8	38.1	39.5	40.8	42.2	43.6	44.9	46.3	47.7	49.1	50.5	51.9	53.3	54.7	56.1	57.6
21	26.5	27.9	29.2	30.5	31.8	33.2	34.5	35.9	37.3	38.6	40.0	41.4	42.8	44.1	45.5	46.9	48.4	49.8	51.2	52.6	54.1	55.5	56.9	58.4	59.9
22	28.2	29.5	30.9	32.3	33.6	35.0	36.4	37.7	39.1	40.5	41.9	43.3	44.8	46.2	47.6	49.0	50.5	51.9	53.4	54.8	56.3	57.8	59.3	60.8	62.3
23	29.9	31.3	32.7	34.1	35.5	36.8	38.3	39.7	41.1	42.5	43.9	45.4	46.8	48.3	49.7	51.2	52.7	54.2	55.6	57.1	58.6	60.2	61.7	63.2	64.7
24	31.7	33.1	34.5	35.9	37.3	38.8	40.2	41.7	43.1	44.6	46.0	47.5	49.0	50.5	52.0	53.5	55.0	56.5	58.0	59.5	61.1	62.6	64.2	65.8	67.3
25	33.6	35.0	36.4	37.9	39.3	40.8	42.2	43.7	45.2	46.7	48.2	49.7	51.2	52.7	54.3	55.8	57.3	58.9	60.5	62.0	63.6	65.2	66.8	68.4	70.0
26	35.5	36.9	38.4	39.9	41.4	42.8	44.3	45.9	47.4	48.9	50.4	52.0	53.5	55.1	56.7	58.2	59.8	61.4	63.0	64.7	66.3	67.9	69.6	71.2	72.9
27	37.4	38.9	40.4	42.0	43.5	45.0	46.5	48.1	49.6	51.2	52.8	54.4	56.0	57.6	59.2	60.8	62.4	64.1	65.7	67.4	69.1	70.8	72.5	74.2	75.9
28	39.5	41.0	42.6	44.1	45.7	47.3	48.8	50.4	52.0	53.6	55.2	56.9	58.5	60.2	61.8	63.5	65.2	66.9	68.6	70.3	72.0	73.7	75.5	77.3	79.0
29	41.7	43.2	44.8	46.4	48.0	49.6	51.2	52.8	54.5	56.1	57.8	59.5	61.2	62.9	64.6	66.3	68.0	69.8	71.5	73.3	75.1	76.9	78.7	80.5	82.4
30	43.9	45.5	47.1	48.7	50.4	52.0	53.7	55.4	57.1	58.8	60.5	62.2	64.0	65.7	67.5	69.3	71.0	72.9	74.7	76.5	78.3	80.2	82.1	84.0	85.9
31	46.2	47.9	49.5	51.2	52.9	54.6	56.3	58.1	59.8	61.6	63.3	65.1	66.9	68.7	70.5	72.4	74.2	76.1	78.0	79.9	81.8	83.7	85.7	87.6	89.6
32	48.7	50.4	52.1	53.8	55.6	57.3	59.1	60.9	62.7	64.5	66.3	68.2	70.0	71.9	73.8	75.7	77.6	79.5	81.5	83.5	85.4	87.5	89.5	91.5	93.6
33	51.2	53.0	54.8	56.5	58.3	60.2	62.0	63.8	65.7	67.6	69.5	71.4	73.3	75.2	77.2	79.2	81.2	83.2	85.2	87.3	89.3	91.4	93.6	95.7	97.8
34	53.9	55.7	57.6	59.4	61.3	63.1	65.0	67.0	68.9	70.8	72.8	74.8	76.8	78.8	80.8	82.9	85.0	87.1	89.2	91.4	93.5	95.7	97.9	100.2	102.4
35	56.8	58.6	60.5	62.4	64.4	66.3	68.3	70.3	72.3	74.3	76.3	78.4	80.5	82.6	84.7	86.9	89.1	91.3	93.5	95.7	98.0	100.3	102.6	105.0	107.3
36	59.8	61.7	63.7	65.7	67.7	69.7	71.7	73.8	75.9	78.0	80.1	82.3	84.5	86.7	88.9	91.2	93.5	95.8	98.1	100.5	102.9	105.3	107.7	110.2	112.7
37	62.9	65.0	67.0	69.1	71.2	73.3	75.4	77.6	79.8	82.0	84.2	86.5	88.8	91.1	93.4	95.8	98.2	100.6	103.1	105.6	108.1	110.7	113.3	115.9	118.6
38	66.3	68.4	70.6	72.7	74.9	77.1	79.4	81.6	83.9	86.2	88.6	91.0	93.4	95.8	98.3	100.8	103.4	105.9	108.6	111.2	113.9	116.6	119.4	122.2	125.0
39	70.0	72.2	74.4	76.7	78.9	81.3	83.6	86.0	88.4	90.9	93.4	95.9	98.4	101.0	103.6	106.3	109.0	111.8	114.6	117.4	120.3	123.2	126.1	129.2	132.2
40	73.8	76.2	78.5	80.9	83.3	85.7	88.2	90.8	93.3	95.9	98.5	101.2	103.9	106.7	109.5	112.4	115.3	118.2	121.2	124.3	127.4	130.5	133.7	137.0	140.3
41	78.0	80.5	83.0	85.5	88.0	90.6	93.3	95.9	98.7	101.4	104.3	107.1	110.0	113.0	116.0	119.1	122.2	125.4	128.7	132.0	135.4	138.8	142.3	145.9	149.5
42	82.6	85.2	87.8	90.5	93.2	96.0	98.8	101.7	104.6	107.6	110.6	113.7	116.9	120.1	123.4	126.7	130.1	133.6	137.2	140.8	144.5	148.3	152.2	156.1	160.2
43	87.6	90.4	93.2	96.0	99.0	101.9	105.0	108.1	111.2	114.5	117.8	121.1	124.6	128.1	131.7	135.4	139.1	143.0	147.0	151.0	155.2	159.4	163.8	168.2	172.8
44	93.1	96.1	99.1	102.2	105.4	108.6	111.9	115.3	118.7	122.3	125.9	129.6	133.4	137.4	141.4	145.5	149.7	154.1	158.5	163.1	167.9	172.7	177.7	182.9	188.2
45	99.3	102.5	105.8	109.2	112.6	116.2	119.8	123.6	127.4	131.4	135.4	139.6	143.9	148.3	152.9	157.6	162.4	167.4	172.6	178.0	183.5	189.2	195.1	201.2	207.5
46	106.3	109.8	113.4	117.2	121.0	125.0	129.1	133.3	137.6	142.1	146.7	151.5	156.5	161.6	167.0	172.5	178.2	184.2	190.4	196.8	203.5	210.5	217.8	225.4	233.3
47	114.3	118.3	122.4	126.6	130.9	135.4	140.1	145.0	150.0	155.3	160.7	166.4	172.3	178.5	185.0	191.8	198.9	206.4	214.2	222.4	231.0	240.0	249.5	259.5	270.0
48	123.9	128.4	133.1	137.9	143.0	148.3	153.9	159.7	165.8	172.2	178.9	186.0	193.5	201.4	209.8	218.7	228.2	238.2	248.9	260.3	272.3	285.1	298.7	313.0	328.2
49	135.5	140.8	146.4	152.3	158.5	165.0	172.0	179.3	187.2	195.6	204.6	214.3	224.7	235.9	248.1	261.3	275.5	290.9	307.6	325.5	344.8	365.4	387.3	410.6	435.2

IDEXX Quanti-Tray®/2000 MPN Table

# Large Wells Positive	# Small Wells Positive																							
	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48
0	25.3	26.4	27.4	28.4	29.5	30.5	31.5	32.6	33.6	34.7	35.7	36.8	37.8	38.9	40.0	41.0	42.1	43.1	44.2	45.3	46.3	47.4	48.5	49.5
1	26.6	27.7	28.7	29.8	30.8	31.9	32.9	34.0	35.0	36.1	37.2	38.2	39.3	40.4	41.4	42.5	43.6	44.7	45.7	46.8	47.9	49.0	50.1	51.2
2	27.9	29.0	30.0	31.1	32.2	33.2	34.3	35.4	36.5	37.5	38.6	39.7	40.8	41.9	43.0	44.0	45.1	46.2	47.3	48.4	49.5	50.6	51.7	52.8
3	29.3	30.4	31.4	32.5	33.6	34.7	35.8	36.8	37.9	39.0	40.1	41.2	42.3	43.4	44.5	45.6	46.7	47.8	48.9	50.0	51.2	52.3	53.4	54.5
4	30.7	31.8	32.8	33.9	35.0	36.1	37.2	38.3	39.4	40.5	41.6	42.8	43.9	45.0	46.1	47.2	48.3	49.5	50.6	51.7	52.9	54.0	55.1	56.3
5	32.1	33.2	34.3	35.4	36.5	37.6	38.7	39.9	41.0	42.1	43.2	44.4	45.5	46.6	47.7	48.9	50.0	51.2	52.3	53.5	54.6	55.8	56.9	58.1
6	33.5	34.7	35.8	36.9	38.0	39.2	40.3	41.4	42.6	43.7	44.8	46.0	47.1	48.3	49.4	50.6	51.7	52.9	54.1	55.2	56.4	57.6	58.7	59.9
7	35.0	36.2	37.3	38.4	39.6	40.7	41.9	43.0	44.2	45.3	46.5	47.7	48.8	50.0	51.2	52.3	53.5	54.7	55.9	57.1	58.3	59.4	60.6	61.8
8	36.6	37.7	38.9	40.0	41.2	42.3	43.5	44.7	45.9	47.0	48.2	49.4	50.6	51.8	53.0	54.1	55.3	56.5	57.7	59.0	60.2	61.4	62.6	63.8
9	38.1	39.3	40.5	41.6	42.8	44.0	45.2	46.4	47.6	48.8	50.0	51.2	52.4	53.6	54.8	56.0	57.2	58.4	59.7	60.9	62.1	63.4	64.6	65.8
10	39.7	40.9	42.1	43.3	44.5	45.7	46.9	48.1	49.3	50.6	51.8	53.0	54.2	55.5	56.7	57.9	59.2	60.4	61.7	62.9	64.2	65.4	66.7	67.9
11	41.4	42.6	43.8	45.0	46.3	47.5	48.7	49.9	51.2	52.4	53.7	54.9	56.1	57.4	58.6	59.9	61.2	62.4	63.7	65.0	66.3	67.5	68.8	70.1
12	43.1	44.3	45.6	46.8	48.1	49.3	50.6	51.8	53.1	54.3	55.6	56.8	58.1	59.4	60.7	62.0	63.2	64.5	65.8	67.1	68.4	69.7	71.0	72.4
13	44.9	46.1	47.4	48.6	49.9	51.2	52.5	53.7	55.0	56.3	57.6	58.9	60.2	61.5	62.8	64.1	65.4	66.7	68.0	69.3	70.7	72.0	73.3	74.7
14	46.7	48.0	49.3	50.5	51.8	53.1	54.4	55.7	57.0	58.3	59.6	60.9	62.3	63.6	64.9	66.3	67.6	68.9	70.3	71.6	73.0	74.4	75.7	77.1
15	48.6	49.9	51.2	52.5	53.8	55.1	56.4	57.8	59.1	60.4	61.8	63.1	64.5	65.8	67.2	68.5	69.9	71.3	72.6	74.0	75.4	76.8	78.2	79.6
16	50.5	51.8	53.2	54.5	55.8	57.2	58.5	59.9	61.2	62.6	64.0	65.3	66.7	68.1	69.5	70.9	72.3	73.7	75.1	76.5	77.9	79.3	80.8	82.2
17	52.5	53.9	55.2	56.6	58.0	59.3	60.7	62.1	63.5	64.9	66.3	67.7	69.1	70.5	71.9	73.3	74.8	76.2	77.6	79.1	80.5	82.0	83.5	84.9
18	54.6	56.0	57.4	58.8	60.2	61.6	63.0	64.4	65.8	67.2	68.6	70.1	71.5	73.0	74.4	75.9	77.3	78.8	80.3	81.8	83.3	84.8	86.3	87.8
19	56.8	58.2	59.6	61.0	62.4	63.9	65.3	66.8	68.2	69.7	71.1	72.6	74.1	75.5	77.0	78.5	80.0	81.5	83.1	84.6	86.1	87.6	89.2	90.7
20	59.0	60.4	61.9	63.3	64.8	66.3	67.7	69.2	70.7	72.2	73.7	75.2	76.7	78.2	79.8	81.3	82.8	84.4	85.9	87.5	89.1	90.7	92.2	93.8
21	61.3	62.8	64.3	65.8	67.3	68.8	70.3	71.8	73.3	74.9	76.4	77.9	79.5	81.1	82.6	84.2	85.8	87.4	89.0	90.6	92.2	93.8	95.4	97.1
22	63.8	65.3	66.8	68.3	69.8	71.4	72.9	74.5	76.1	77.6	79.2	80.8	82.4	84.0	85.6	87.2	88.9	90.5	92.1	93.8	95.5	97.1	98.8	100.5
23	66.3	67.8	69.4	71.0	72.5	74.1	75.7	77.3	78.9	80.5	82.2	83.8	85.4	87.1	88.7	90.4	92.1	93.8	95.5	97.2	98.9	100.6	102.4	104.1
24	68.9	70.5	72.1	73.7	75.3	77.0	78.6	80.3	81.9	83.6	85.2	86.9	88.6	90.3	92.0	93.8	95.5	97.2	99.0	100.7	102.5	104.3	106.1	107.9
25	71.7	73.3	75.0	76.6	78.3	80.0	81.7	83.3	85.1	86.8	88.5	90.2	92.0	93.7	95.5	97.3	99.1	100.9	102.7	104.5	106.3	108.2	110.0	111.9
26	74.6	76.3	78.0	79.7	81.4	83.1	84.8	86.6	88.4	90.1	91.9	93.7	95.5	97.3	99.2	101.0	102.9	104.7	106.6	108.5	110.4	112.3	114.2	116.2
27	77.6	79.4	81.1	82.9	84.6	86.4	88.2	90.0	91.9	93.7	95.5	97.4	99.3	101.2	103.1	105.0	106.9	108.8	110.8	112.7	114.7	116.7	118.7	120.7
28	80.8	82.6	84.4	86.3	88.1	89.9	91.8	93.7	95.6	97.5	99.4	101.3	103.3	105.2	107.2	109.2	111.2	113.2	115.2	117.3	119.3	121.4	123.5	125.6
29	84.2	86.1	87.9	89.8	91.7	93.7	95.6	97.5	99.5	101.5	103.5	105.5	107.5	109.5	111.6	113.7	115.7	117.8	120.0	122.1	124.2	126.4	128.6	130.8
30	87.8	89.7	91.7	93.6	95.6	97.6	99.6	101.6	103.7	105.7	107.8	109.9	112.0	114.2	116.3	118.5	120.6	122.8	125.1	127.3	129.5	131.8	134.1	136.4
31	91.6	93.6	95.6	97.7	99.7	101.8	103.9	106.0	108.2	110.3	112.5	114.7	116.9	119.1	121.4	123.6	125.9	128.2	130.5	132.9	135.3	137.7	140.1	142.5
32	95.7	97.8	99.9	102.0	104.2	106.3	108.5	110.7	113.0	115.2	117.5	119.8	122.1	124.5	126.8	129.2	131.6	134.0	136.5	139.0	141.5	144.0	146.6	149.1
33	100.0	102.2	104.4	106.6	108.9	111.2	113.5	115.8	118.2	120.5	122.9	125.4	127.8	130.3	132.8	135.3	137.8	140.4	143.0	145.6	148.3	150.9	153.7	156.4
34	104.7	107.0	109.3	111.7	114.0	116.4	118.9	121.3	123.8	126.3	128.8	131.4	134.0	136.6	139.2	141.9	144.6	147.4	150.1	152.9	155.7	158.6	161.5	164.4
35	109.7	112.2	114.6	117.1	119.6	122.2	124.7	127.3	129.9	132.6	135.3	138.0	140.8	143.6	146.4	149.2	152.1	155.0	158.0	161.0	164.0	167.1	170.2	173.3
36	115.2	117.8	120.4	123.0	125.7	128.4	131.1	133.9	136.7	139.5	142.4	145.3	148.3	151.3	154.3	157.3	160.5	163.6	166.8	170.0	173.3	176.6	179.9	183.3
37	121.3	124.0	126.8	129.6	132.4	135.3	138.2	141.2	144.2	147.3	150.3	153.5	156.7	159.9	163.1	166.5	169.8	173.2	176.7	180.2	183.7	187.3	191.0	194.7
38	127.9	130.8	133.8	136.8	139.9	143.0	146.2	149.4	152.6	155.9	159.2	162.6	166.1	169.6	173.2	176.8	180.4	184.2	188.0	191.8	195.7	199.7	203.7	207.7
39	135.3	138.5	141.7	145.0	148.3	151.7	155.1	158.6	162.1	165.7	169.4	173.1	176.9	180.7	184.7	188.7	192.7	196.8	201.0	205.3	209.6	214.0	218.5	223.0
40	143.7	147.1	150.6	154.2	157.8	161.5	165.3	169.1	173.0	177.0	181.1	185.2	189.4	193.7	198.1	202.5	207.1	211.7	216.4	221.1	226.0	231.0	236.0	241.1
41	153.2	157.0	160.9	164.8	168.9	173.0	177.2	181.5	185.8	190.3	194.8	199.5	204.2	209.1	214.0	219.1	224.2	229.4	234.8	240.2	245.8	251.5	257.2	263.1
42	164.3	168.6	172.9	177.3	181.9	186.5	191.3	196.1	201.1	206.2	211.4	216.7	222.2	227.7	233.4	239.2	245.2	251.3	257.5	263.8	270.3	276.9	283.6	290.5
43	177.5	182.3	187.3	192.4	197.6	202.9	208.4	214.0	219.8	225.8	231.8	238.1	244.5	251.0	257.7	264.6	271.7	278.9	286.3	293.8	301.5	309.4	317.4	325.7
44	193.6	199.3	205.1	211.0	217.2	223.5	230.0	236.7	243.6	250.8	258.1	265.6	273.3	281.2	289.4	297.8	306.3	315.1	324.1	333.3	342.8	352.4	362.3	372.4
45	214.1	220.9	227.9	235.2	242.7	250.4	258.4	266.7	275.3	284.1	293.3	302.6	312.3	322.3	332.5	343.0	353.8	364.9	376.2	387.9	399.8	412.0	424.5	437.4
46	241.5	250.0	258.9	268.2	277.8	287.8	298.1	308.8	319.9	331.4	343.3	355.5	368.1	381.1	394.5	408.3	422.5	437.1	452.0	467.4	483.3	499.6	516.3	533.5
47	280.9	292.4	304.4	316.9	330.0	343.6	357.8	372.5	387.7	403.4	419.8	436.6	454.1	472.1	490.7	509.9	529.8	550.4	571.7	593.8	616.7	640.5	665.3	691.0
48	344.1	360.9	378.4	396.8	416.0	436.0	456.9	478.6	501.2	524.7	549.3	574.8	601.5	629.4	658.6	689.3	721.5	755.6	791.5	829.7	870.4	913.9	960.6	1011.2
49	461.1	488.4	517.2	547.5	579.4	613.1	648.8	686.7	727.0	770.1	816.4	866.4	920.8	980.4	1046.2	1119.9	1203.3	1299.7	1413.6	1553.1	1732.9	1986.3	2419.6	>2419.6

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.

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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

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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

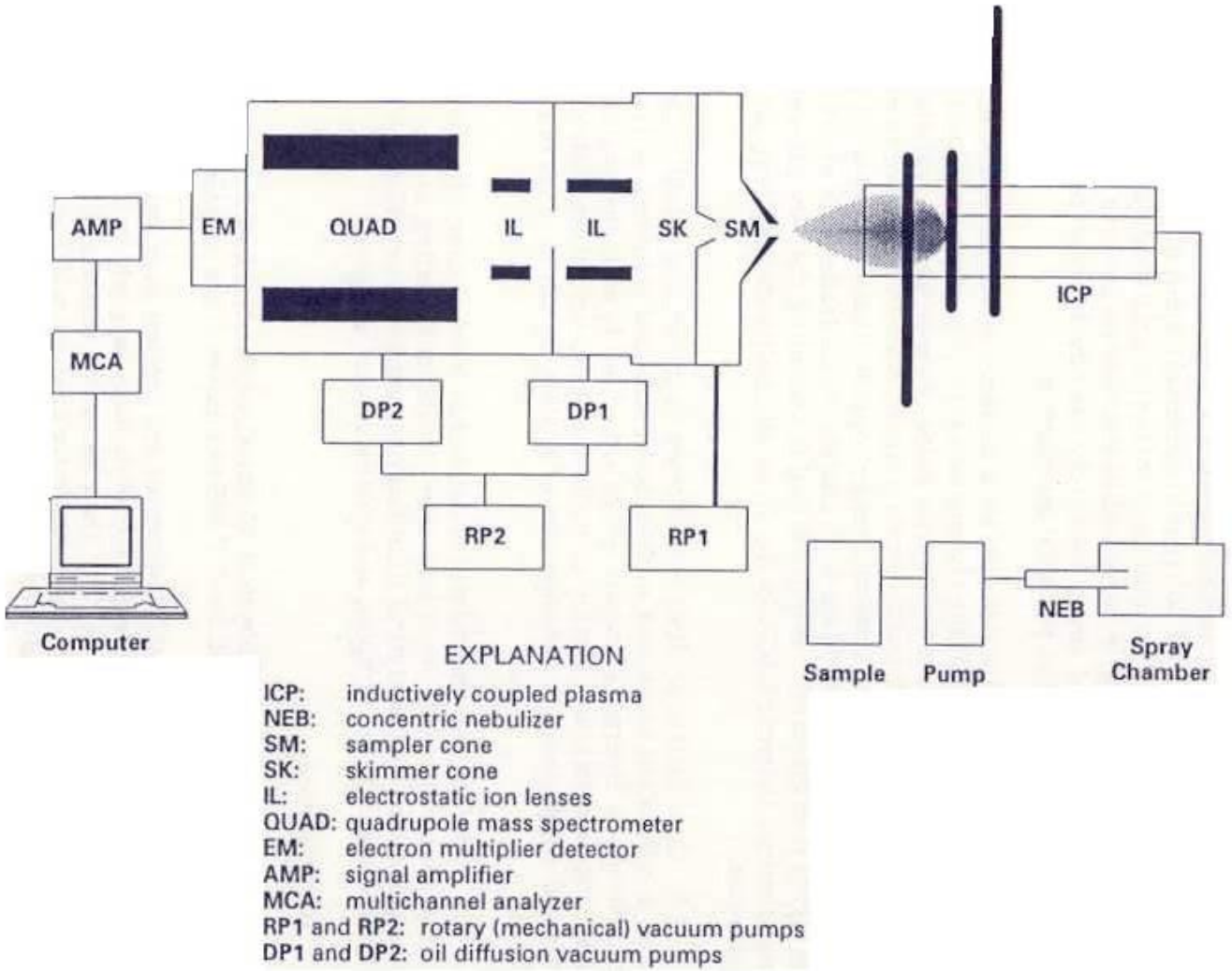


Figure 1.--Block diagram of inductively coupled plasma - mass spectrometer.

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.

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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.

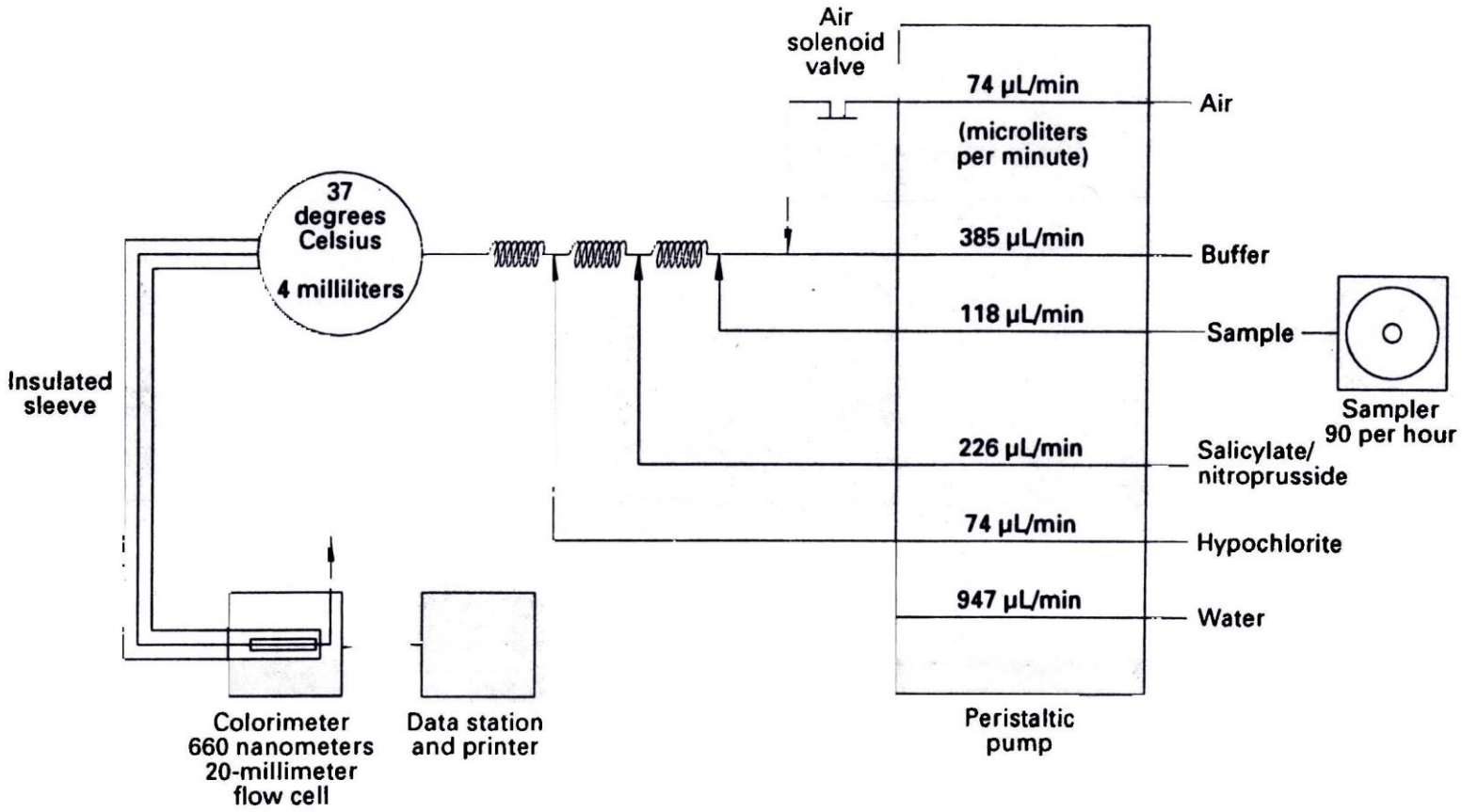


Figure 4.—Nitrogen, ammonia, salicylate-hypochlorite manifold.

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.

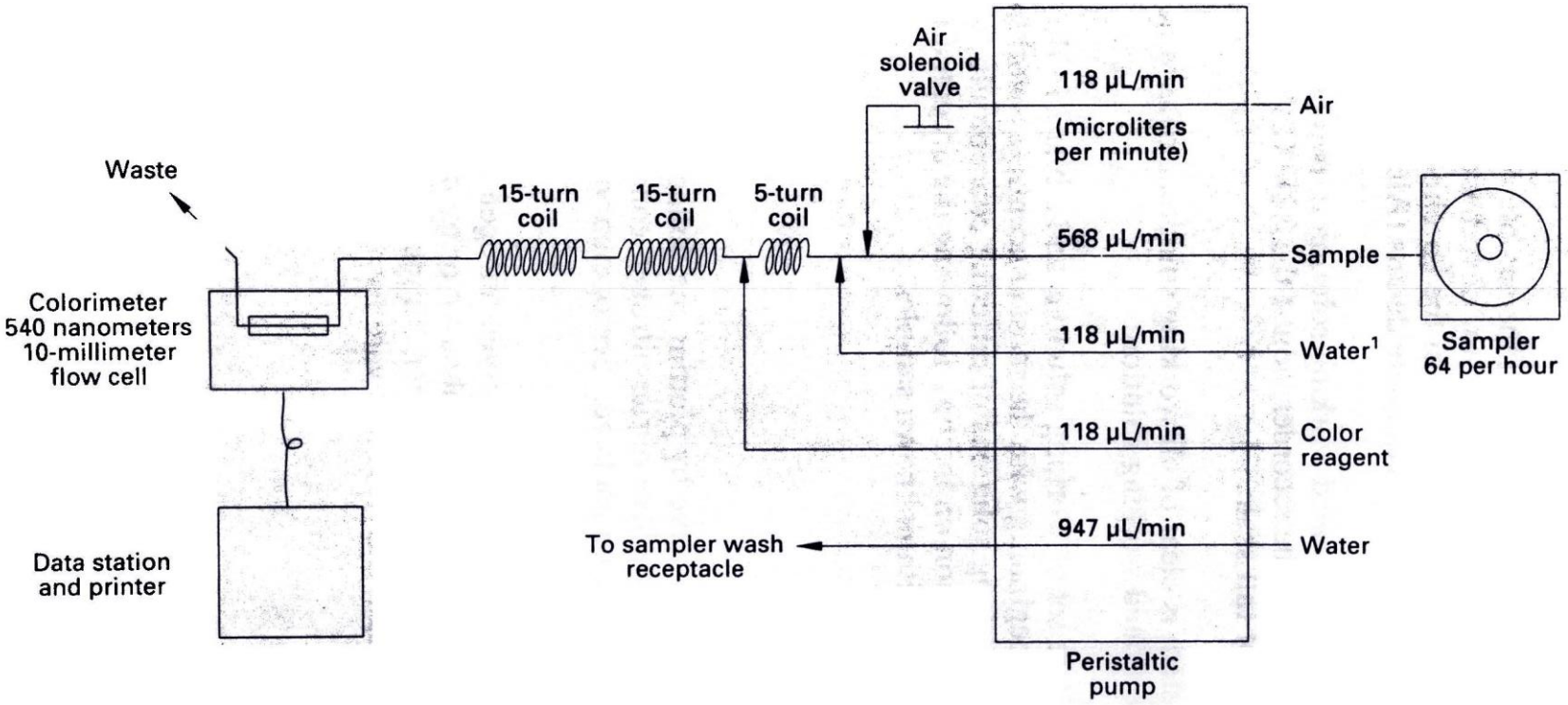
5.3 *Nitrite-nitrogen standard solution II*, 1.00 mL = 0.001 mg NO_2-N: Dilute 10.0 mL nitrite-nitrogen standard solution I to 1,000 mL with demineralized water.

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

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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.

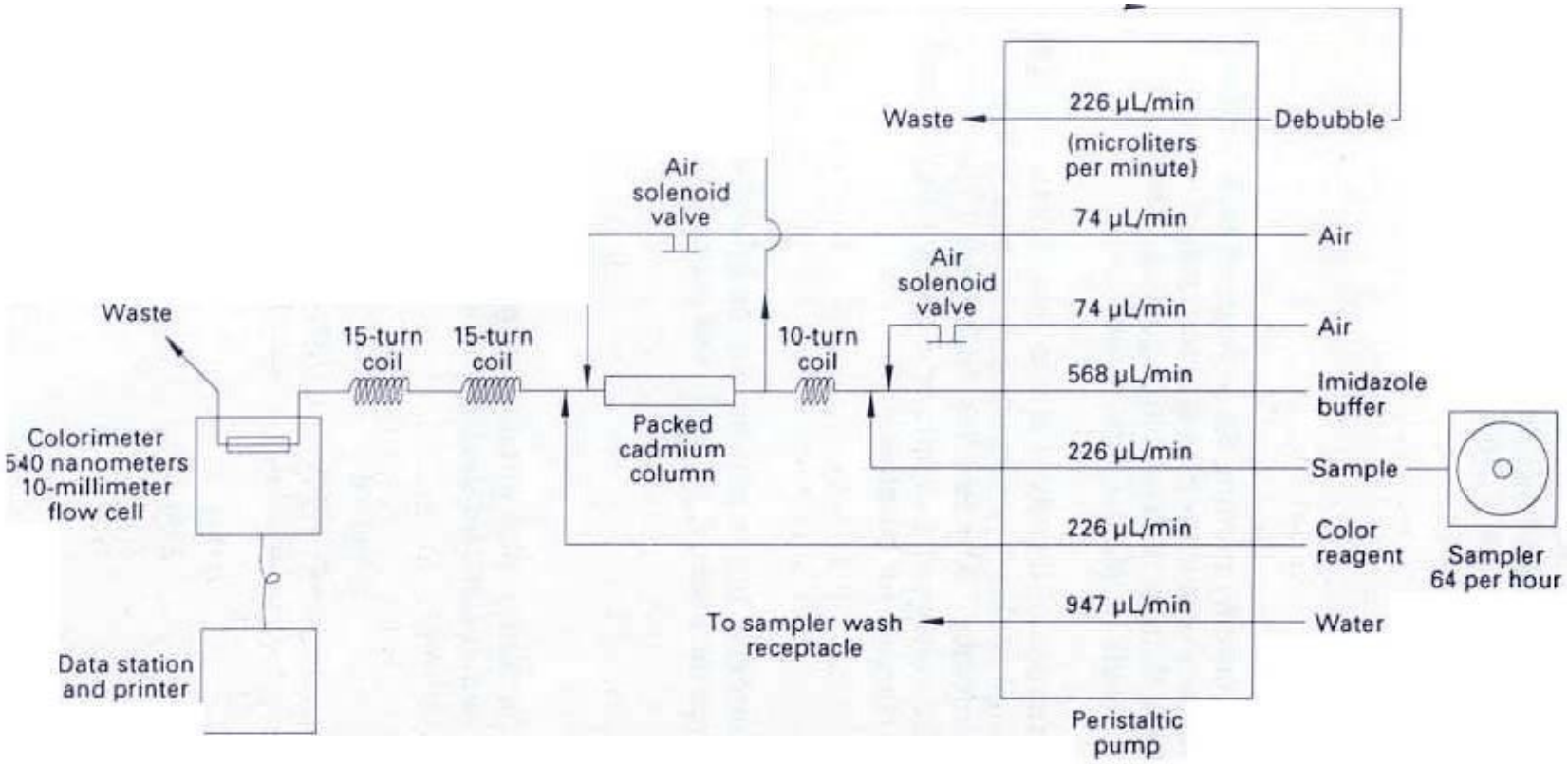


Figure 7.—Nitrogen, nitrite plus nitrate, low ionic-strength water, cadmium reduction-diazotization manifold.

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

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Alpkem Corp., 1986, Rapid flow analyzer operator's manual: ALPKEM, methodology section.

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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.

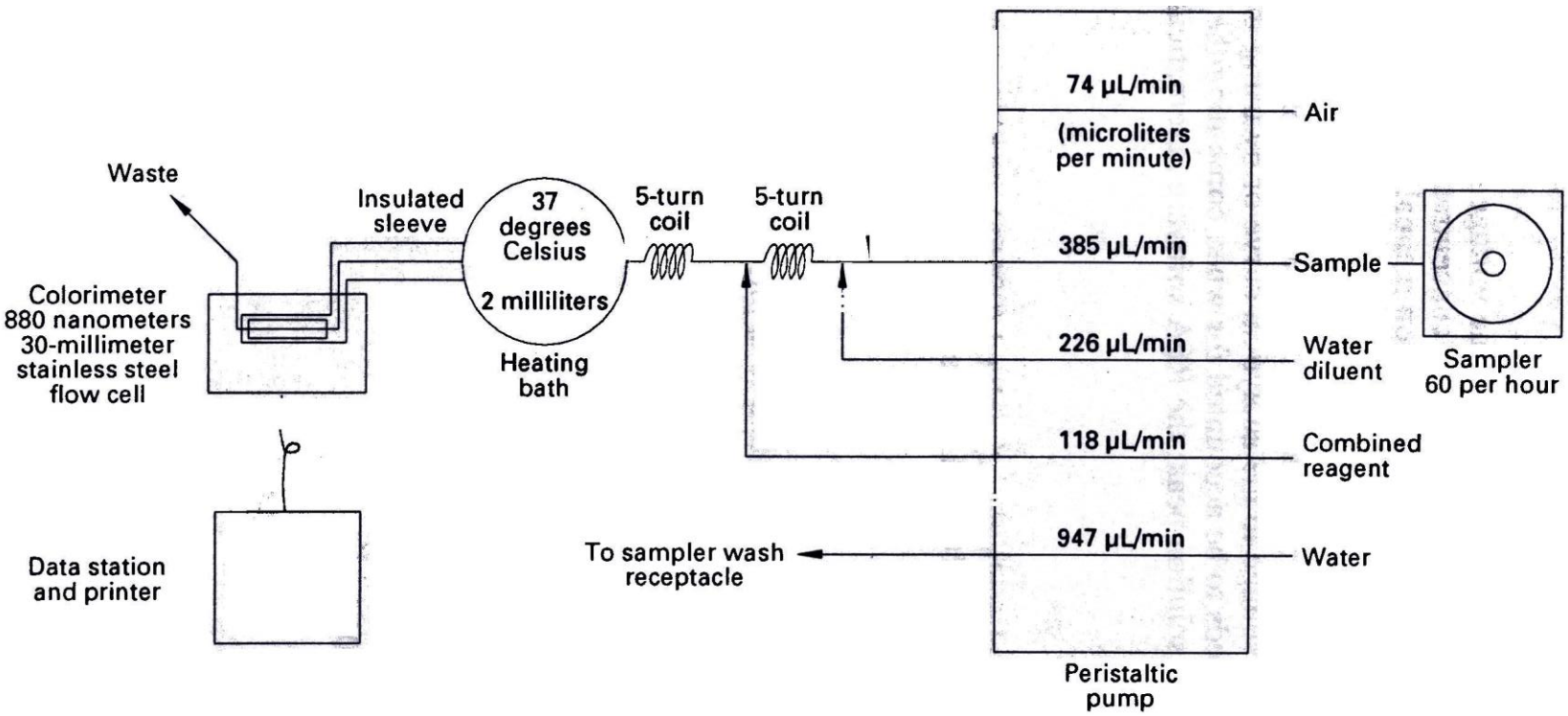


Figure 9.—Phosphorus, low ionic-strength water, phosphomolybdate manifold.

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

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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.

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Techniques of Water-Resources Investigations
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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.

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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

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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

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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

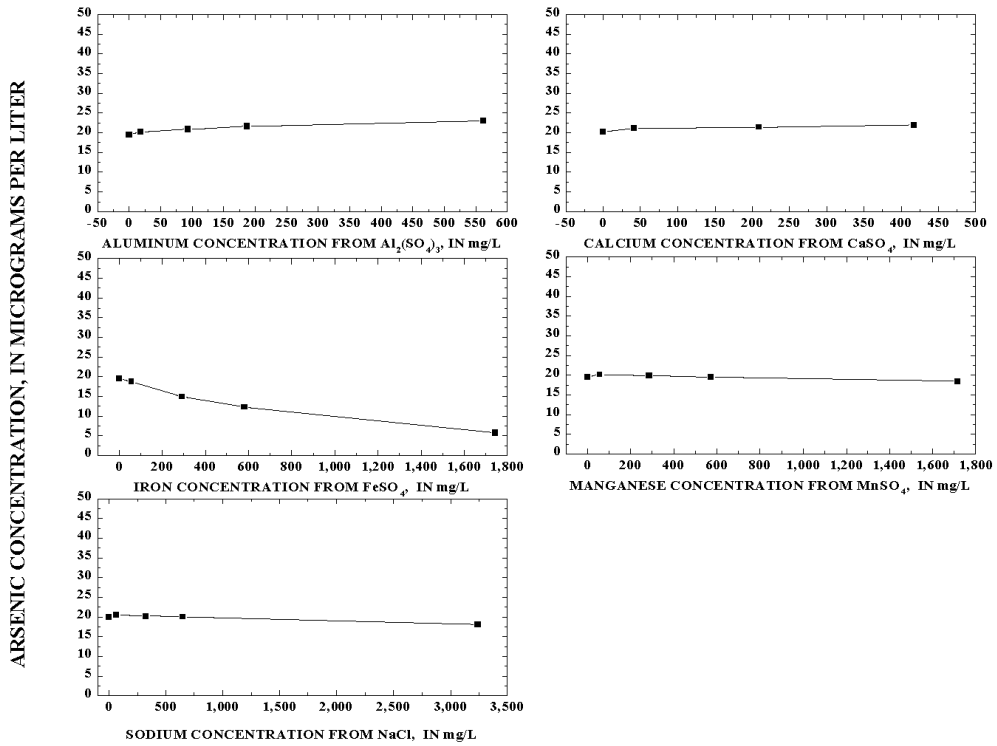


Figure 1. Relations among selected cations and anions and **arsenic** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

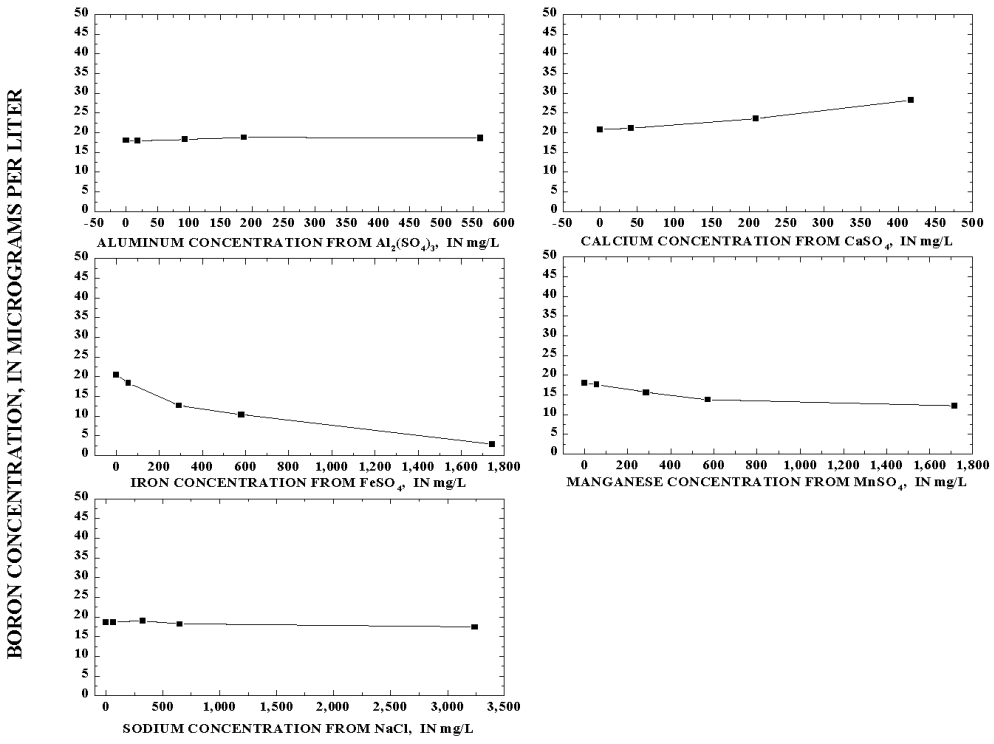


Figure 2. Relations among selected cations and anions and **boron** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

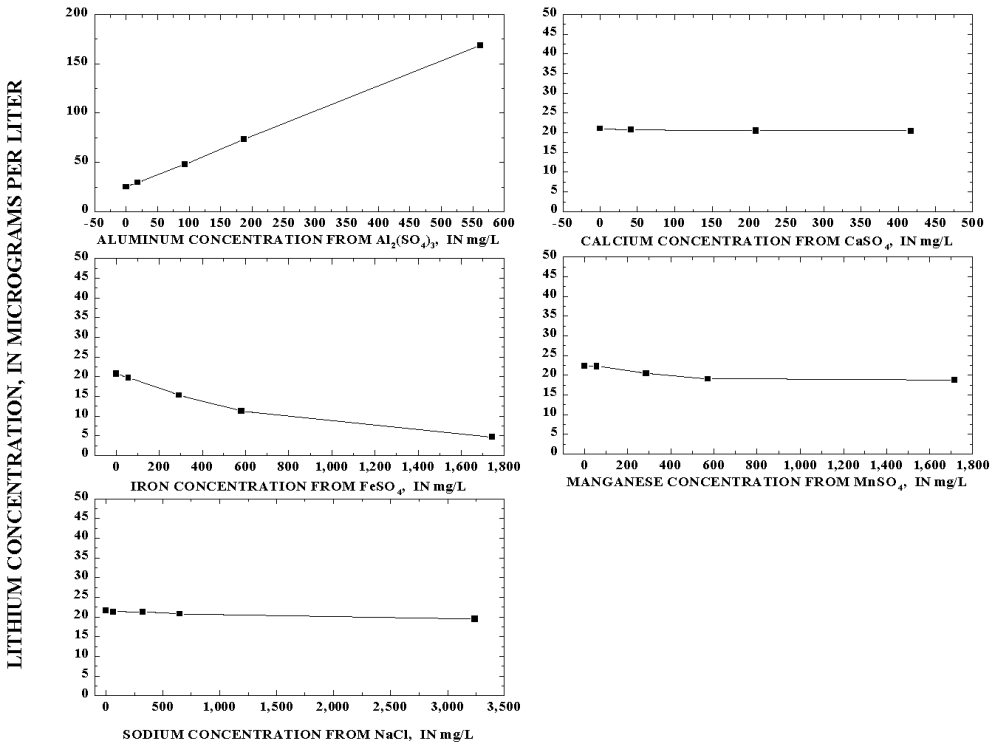


Figure 3. Relations among selected cations and anions and **lithium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

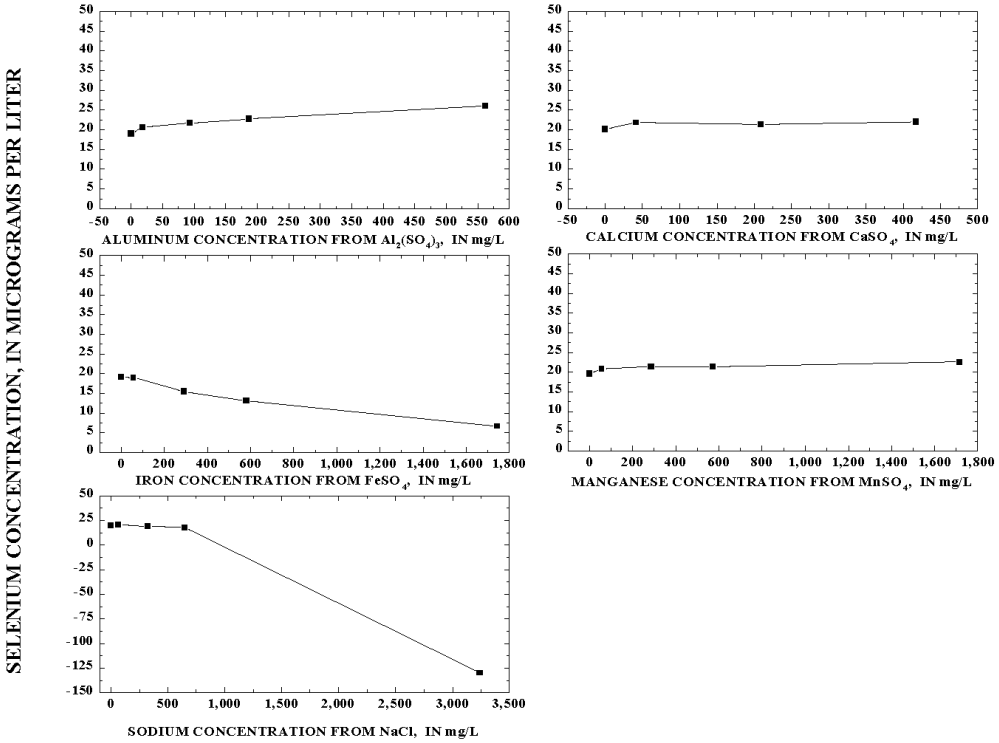


Figure 4. Relations among selected cations and anions and **selenium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

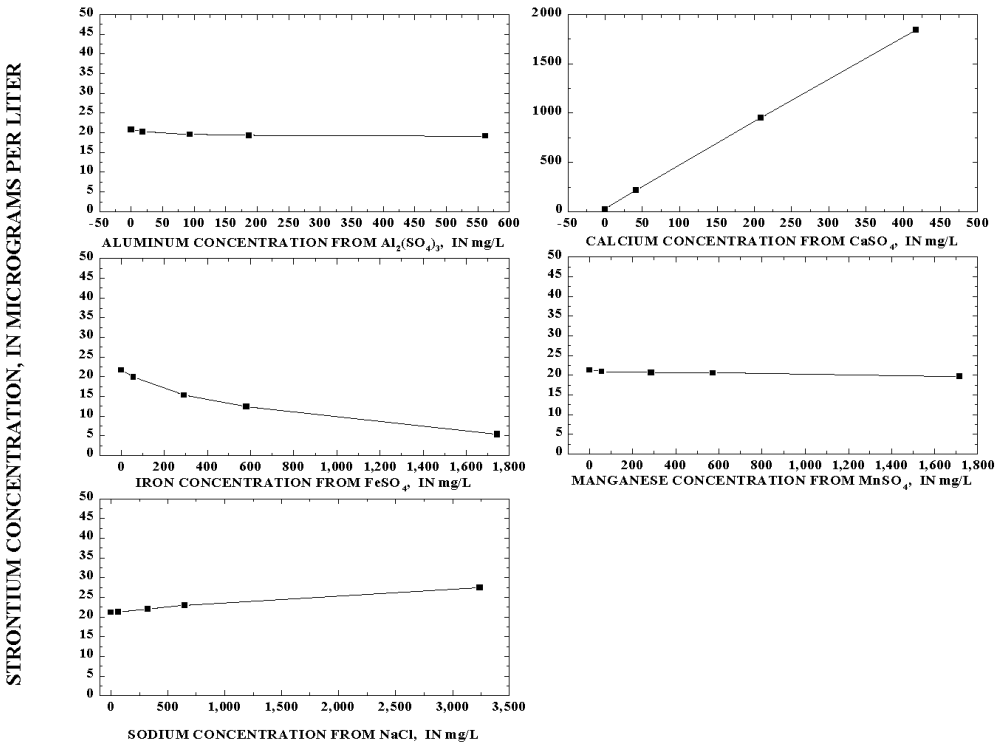


Figure 5. Relations among selected cations and anions and **strontium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

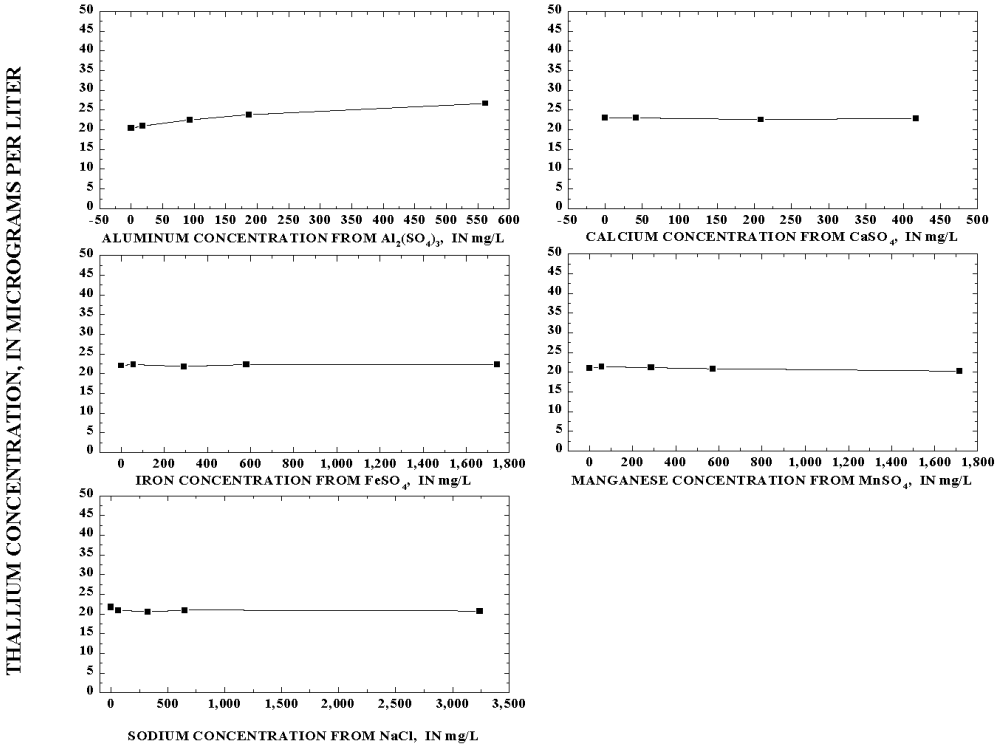


Figure 6. Relations among selected cations and anions and **thallium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

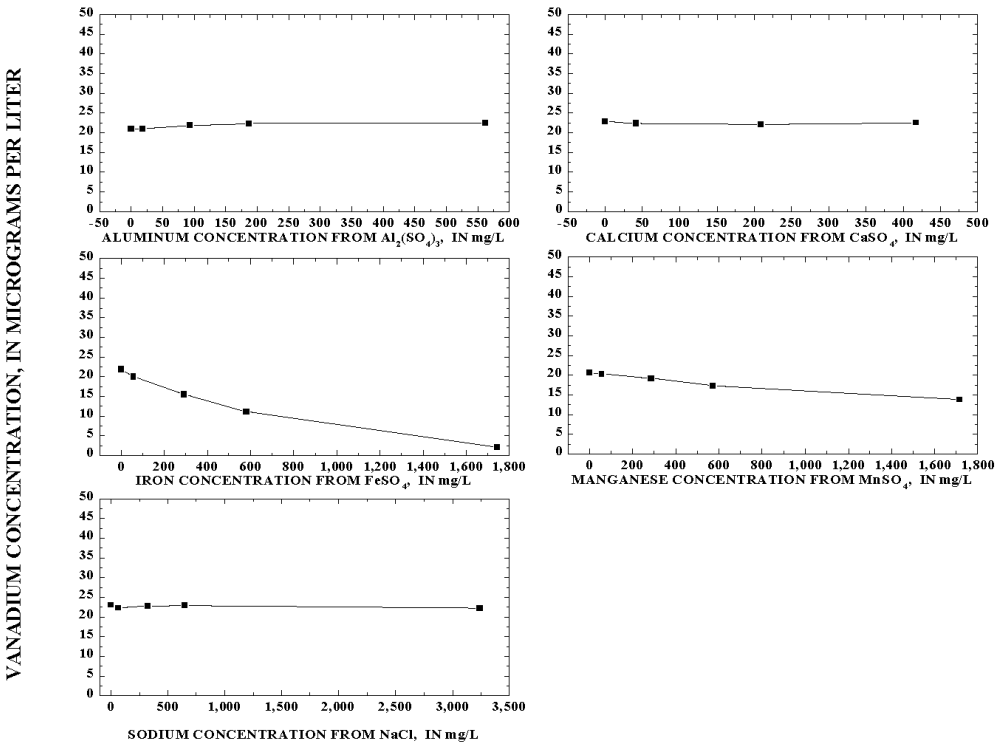


Figure 7. Relations among selected cations and anions and **vanadium** determined by inductively coupled plasma–mass spectrometry (mg/L, milligrams per liter).

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.

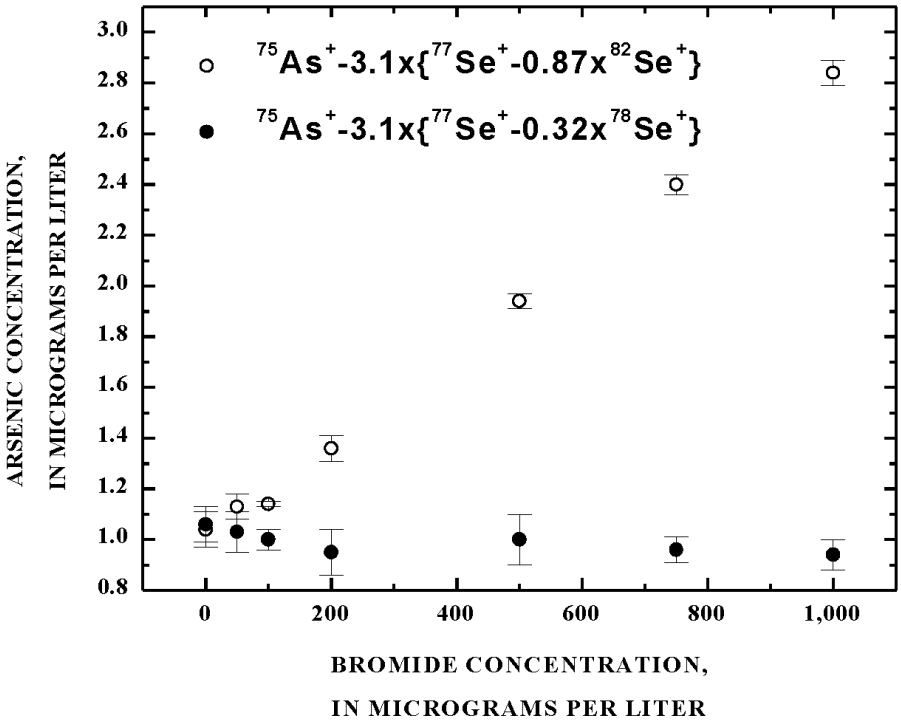


Figure 8. Accuracy of inductively coupled plasma–mass spectrometric arsenic determinations in the presence of bromide using different correction equations. The error bars correspond to one standard deviation based on three instrumental measurements.

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.

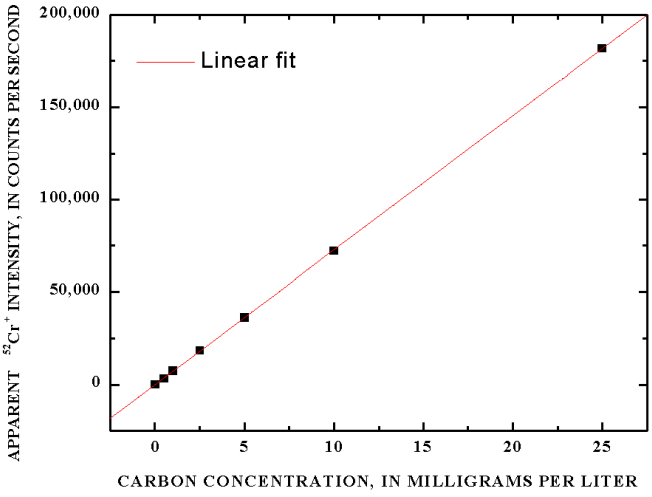


Figure 9. Relation of apparent chromium-52 signal from $^{40}Ar^{12}C^{+}$ as a function of increasing concentrations of carbon.

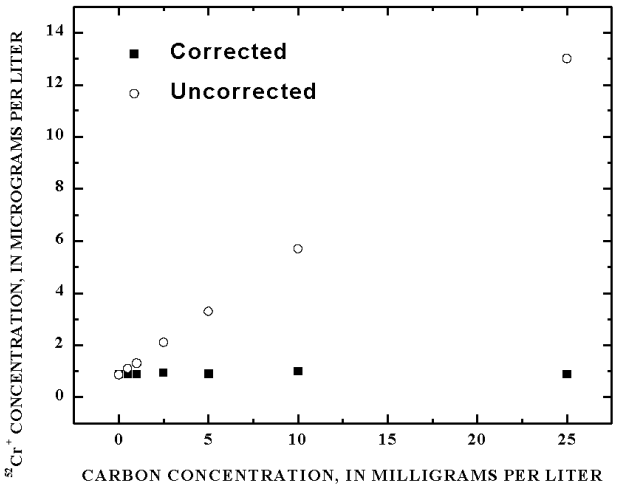


Figure 10. The accuracy of inductively coupled plasma–mass spectrometric chromium-52 determinations in the presence of carbon.

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).

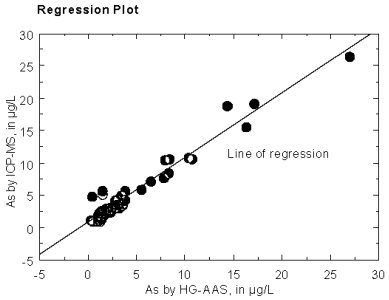
The p-values of the Wilcoxon Signed Rank Test indicate with 95-percent confidence that there is no significant difference between ICP–MS and ICP–AES results for dissolved boron and vanadium. In general, the descriptive statistics and figures 11 through 16 indicate that differences among results for the other elements are analytically insignificant considering the range of sample concentrations in the data set.

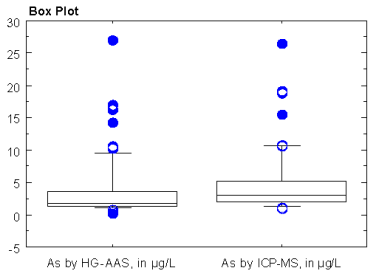
Table 8. Statistical analysis summary of inductively coupled plasma–mass spectrometry and former methods of analysis

[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

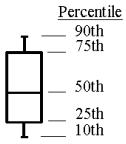
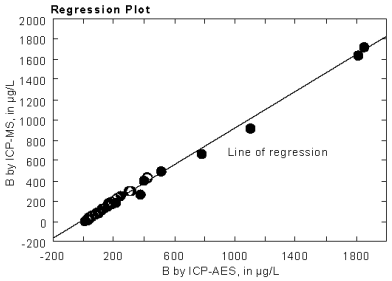
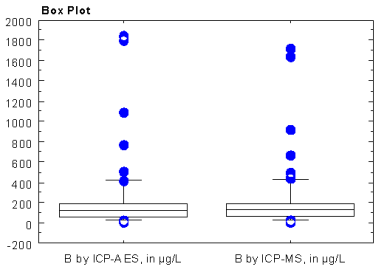


Figure 11. Statistical analysis of **arsenic (As)** results for 59 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and hydride generation–atomic absorption spectrophotometry.





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

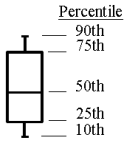
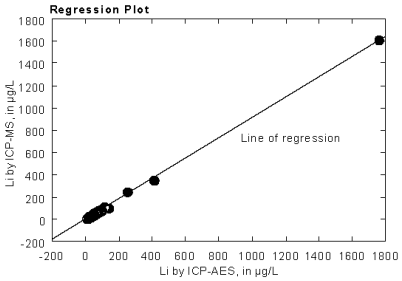
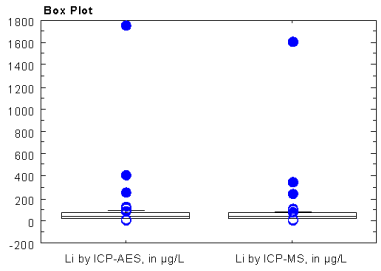


Figure 12. Statistical analysis of **boron (B)** results for 62 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and inductively coupled plasma–atomic emission spectrophotometry.





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

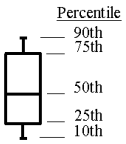
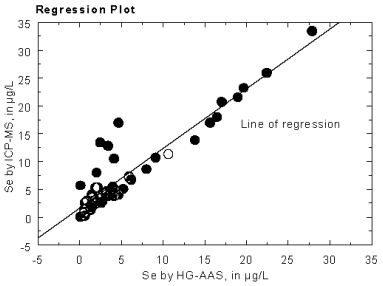
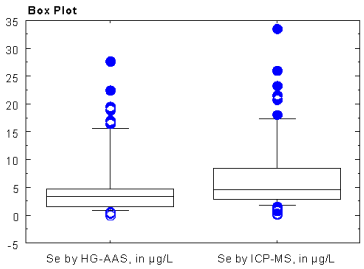


Figure 13. Statistical analysis of **lithium (Li)** results for 63 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry inductively coupled plasma–atomic emission spectrophotometry.





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

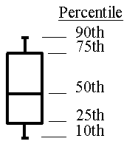
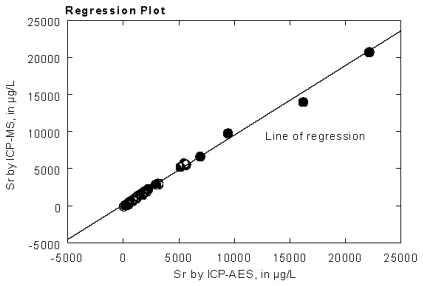
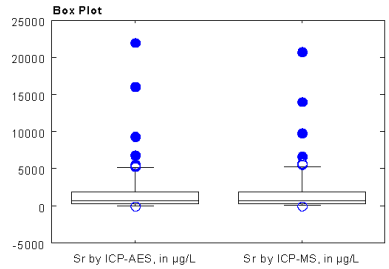


Figure 14. Statistical analysis of **selenium (Se)** results for 64 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and hydride generation–atomic absorption spectrophotometry.





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

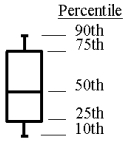
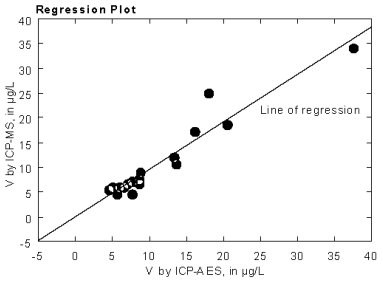
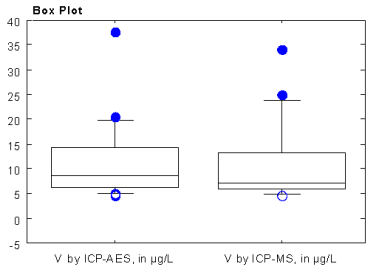


Figure 15. Statistical analysis of **strontium (Sr)** results for 63 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and inductively coupled plasma–atomic emission spectrophotometry.





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

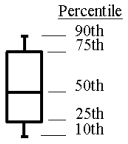


Figure 16. Statistical analysis of **vanadium (V)** results for 17 filtered, acidified, natural-water samples from inductively coupled plasma–mass spectrometry and inductively coupled plasma–atomic emission spectrophotometry.

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.

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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 1.02 (3/19/2010)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.00	7/2008	R. Weissinger	Added filtered nutrients	New analysis suite	1.01
1.01	3/19/2010	D.Thoma	Added new methods appendices	Methods used by AWAL labs not included in previous version	1.02

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.

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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 laboratory method is mapped to each individual record in the water quality database.

The following parameters are measured monthly, for every sample sent to the Utah Public Health Laboratory:

Alkalinity, CaCO3, mg/l
Ammonia as N, mg/l
Bicarbonate, mg/l
Calcium, Diss., mg/l
Carbon Dioxide, mg/l
Carbonate Ion, mg/l
Chloride, mg/l
Tot. Diss. Solids @180C, mg/l
Hardness, Ca+Mg, mg/l
Hydroxide, mg/l
Magnesium, Diss., mg/l
Nitrogen, Diss., mg/l
NO2+NO3 as N, mg/l
pH (lab)
Phosphorus as P, Total, mg/l
Phosphorus as P, Diss., mg/l
Potassium, Diss., mg/l
Sodium, Diss., mg/l
Spec. Conductivity, umhos/cm
Sulfate, mg/l
Total suspended solids, mg/l
Turbidity, NTU

The following parameters are measured quarterly, in addition to the above monthly parameters, by the Utah Department of Health Laboratory:

Aluminum, Diss., ug/l
Arsenic, Diss., ug/l
Barium, Diss., ug/l
Cadmium, Diss., ug/l
Chromium, Diss., ug/l
Copper, Diss., ug/l
Iron, Diss., ug/l
Lead, Diss., ug/l
Manganese, Diss., ug/l
Mercury, Diss., ug/l
Selenium, Diss., ug/l
Silver, Diss., ug/l

Zinc, Diss., 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
Total nutrients	2
Filtered nutrients	9

Table 2.2. Quarterly analyses suite (January, April, July, October)

Bottle label	Analysis suite
Total Chemistry	2
Filtered metals	3
Total nutrients	2
Filtered nutrients	9

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. Alkalinity
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 (ClO₂ should be removed prior to this step) and chilled to 4^oC.
Max Holding Time:	Part A: 48 Hours (NO₂-N, NO₃-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

National Environmental Methods Index

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)

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CINCINNATI, OHIO 45268

16. Appendix L: Standards Methods Number SM3500 CD Chromium-VI

TABLE 3500-Cr.II. SINGLE-LABORATORY PRECISION AND BIAS

Sample Type	Conc.[a] µg/L	Mean Recovery %	RPD[b]
Reagent water	10.0	100	0.8
	120.0	100	0.0
Drinking water	10.0	105	0.0
	100.0	99	1.5
Groundwater	10.0	98	0.0
	100.0	96	0.8
Primary wastewater effluent	10.0	120	0.0
	100.0	104	3.9
Biotreated wastewater effluent	10.0	90	0.4
	100.0	101	0.4

[a] Sample fortified at this concentration level.
[b] RPD = relative percent difference between 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:

$$S_r = 0.041x + 0.059$$
$$S = 0.090x + 1.139$$
$$\text{Mean recovery} = 0.989x - 0.41$$

[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) and Committee D19 of ASTM.

TABLE 3500-Cr.III. MULTILABORATORY DETERMINATION OF BIAS FOR ANALYSIS OF CHROMIUM

Water	Amount Added µg/L	Amount Found µg/L	S_r	S_t	Bias %
Reagent	0.00	0.00	1.04	0.53	[illegible]
	5.00	5.04	1.70		+0.8
	10.0	17.4	2.45	3.40	+9.8
	20.0	21.4	2.31		+7.0
	100	101	1.51	3.20	+1.0
	140	142	5.57		+1.4
	820	819	26.9	42.2	+0.4
	860	866	18.8		+7.0
Water	0.0	0.05	1.17	0.52	0.1
	8.0	7.31	1.5		8.6
	16.0	14	2.20	1.58	−8.6
	20.0	19.8	1.62		−1.0
	100	98.9	4.36	3.31	−1.1
	140	138	8.29		−1.4
	820	796	66.6	92.1	−8.5
	860	844	72.1		−1.9

[a] Each Youden pair was used to calculate one laboratory data point (3.3).

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. 1990. Technical Note No. 36. Dionex, Sunnyvale, Calif.

Davis, K.W., J.A. Cunningham & R.A. Jones. 1994. Determination of dissolved hexavalent chromium in drinking water, ground water and industrial wastewater by ion chromatography. Collaborative study. *J. Assoc. Offic. Anal. Chem.* 77:1094.

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

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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 the 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 ferrous iron, may be sufficiently concentrated to prevent 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 1.8 mg F^-/L. Color development is virtually instantaneous. Color

Table 4500-F^-:I. Concentrations of Some Substances Causing Error Greater than 0.2 mg/L in Fluoride Methods

Substance	Method C (Electrode)		Method D (SPADNS)	
	Conc. mg/L	Type of Error[a]	Conc. mg/L	Type of Error[a]
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 strength; with arsenite Reagent temperature
Color & turbidity				
Iron	200	−	10	−
Hexametaphosphate ($(NaPO_3)_6$)	10 000		1.0	+
Phosphate (PO_4^{3-})	30 000		15	+
Sulfate (SO_4^{2-})	10 000		300	

[a] + denotes positive error.
− denotes negative error.
Blank denotes no measurable error.
[b] One unit of reading. Titration increases 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)

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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)

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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)

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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)

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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

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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 °C)

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 °C)

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 Application:	This method determines non-filterable residue in drinking, 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 °C)

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

36. Appendix AF: EPA Method 2340 B Specific Conductance

National Environmental Methods Index

NEMI Method Summary

Method Information	**Descriptive Name:** Hardness in Water by EDTA Titration **Official Name:** Hardness in Water by EDTA Titration **Method Number:** 2340B **Revision:** Standard Methods Online
Media	**Media:** WATER
Subcategory	Inorganic
Method Source	**Standard Methods**
Citation	**Standard Methods Online - Standard Methods for the Examination of Water and Wastewater**
Brief Method Summary	Select sample volume that will allow color change to occur with the 15 mL addition of 0.01M EDTA titrant to the sample. Complete titration within 5 minutes. Dilute 25 mL of the sample to 50 mL. Add 1-2 mL of NH4Cl-NH4OH to get the pH to 10 or 10.1. Add 1-2 drops of Eriochrome Black T or Calmagite. Slowly add 0.01M EDTA titrant and stir, until end solution is blue. For samples with low hardness, use a 100 to 1000 mL sample for titration, and proportionately larger amounts of reagents.
Scope and Application	This method is applicable to all waters. Polluted waters and wastewater samples must be pretreated. See abstract in Method 3030.
Applicable Conc Range	Method is suitable for all concentration levels.
Method Download	This is a proprietary method. **Please visit the method owner's site for method documentation**
Interferences	(1) A number of metal ions may cause fading or indistinct end points (Standard Methods 2340C provides a listing of interfering metals and their potential effects). (2) Suspended or colloidal organic matter can interfere with the endpoint.

QC Requirements	not included
Sample Handling	Collect 100 mL in glass or plastic container. Acidify samples to a pH < 2 with HNO3. Analyze within 6 months.
Max Holding Time	6 months
Relative Cost/Effort	Less than $50
Sample Prep Method(s)	SM 3030

Method *2340B* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Pct False Positive	Pct False Negative	Spiking Level
Hardness (E-11778)	N/A	100.3 % Rec (ML)	4.4 % RSD (ML)			50 mg/L

export_analytes

Precision Descriptor Notes	Precision and accuracy values were calculated using interlaboratory data from EPA-managed Water Pollution (WP) performance evaluation studies.
Detection Level Note	-

No method download available.

37. Appendix AG: EPA Method 8151A Chlorinated Herbicides and Related Compounds

National Environmental Methods Index

NEMI Method Summary

Method Information	**Descriptive Name:** Chlorinated Herbicides and Related Compounds in Water, Soil, and Waste Samples by Capillary GC-ECD **Official Name:** Chlorinated Herbicides by GC using Methylation or Pentafluorobenzylation Derivatization **Method Number:** 8151A **Revision:** Revision 1, December 1996
Media	**Media:** VARIOUS
Subcategory	Organic
Method Source	**U.S. EPA Office of Solid Waste**
Citation	**SW-846 Online: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods**
Brief Method Summary	Water, soil, and waste samples are extracted and then esterified. The derivatives are determined by gas chromatography with an electron capture detector.
Scope and Application	This method determines the concentration of chlorinated herbicides and related compounds in water, soil, and waste samples by using capillary-column gas chromatography with an electron capture detector.
Applicable Conc Range	Not Available.
Method Download	**Download full method now (PDF file)**
Interferences	(A) Method interferences: Contaminants in solvents, reagents, glassware, and other sample processing hardware may lead to elevated baselines and/or other interferences in the gas chromatograms. Routine analysis of reagent blanks should be performed as a check for method interferences. Glassware must be

	scrupulously cleaned. After use, rinse the glassware with the last solvent that was used in it, wash with detergent and hot water, then with organic-free reagent water, and finish with a rinse of acetone and pesticide-quality hexane. Store in a clean environment and either invert the glassware or cap with aluminum foil. Before use, rinse with the next solvent to be used. Use only high purity reagents and solvents. Purification of solvents by distillation may be required. (B) Matrix interferences: May be contaminants that are coextracted from the sample. Organic acids, especially chlorinated acids, and phenols may interfere with the determination by methylation. The determination using pentafluorobenzylation is more sensitive and more prone to intereferences from organic acids or phenols than by methylation. Alkaline hydrolysis and extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that may interfere with electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids. Herbicides react readily with alkaline substances and may be lost during analysis. Therefore, all glassware must be acid-rinsed and then rinsed to a constant pH with organic-free reagent water. In addition, sodium sulfate may be acidified. Sample extracts must be dry prior to analysis in order to avoid poor recoveries. (C) Refer to Method 8000.
QC Requirements	Refer to Chapter One and Methods 3500 and 8000 for specific QC procedures. Procedures include: validation of sample preparation, introduction, and analysis, initial demonstration of proficiency, calibration verification, evaluation of retention times, analysis of method blanks, matrix spikes, duplicates, surrogates, and laboratory control samples.
Sample Handling	See SW-846 Chapter Four for information. See the introductory material to Chapter Four, "Organic Analytes", Section 4.0. One liter samples should be collected. Extracts must be stored in the dark and refrigerated at 4°C.
Max Holding Time	Aqueous samples - 7 days (sample), 40 days (extract). Solid samples - 14 days (sample), 40 (extract). Also, see SW-846 Chapter Four for information.
Relative Cost/Effort	$201 to $400
Sample Prep Method(s)	Method 3580

Method *8151A* has 22 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Pct False Positive	Pct False Negative	Spiking Level
Bentazon (25057-89-0)	.2 ug/L	120 % Rec	16.8 % RSD			1 ug/L

		(SL)	(SL)	
Organics, semivolatile (E-12884)	N/A	N/A	N/A	
MCPP (93-65-2)	.09 ug/L	N/A	N/A	
Picloram (1918-02-1)	.14 ug/L	91 % Rec (SL)	15.5 % RSD (SL)	.6 ug/L
Acifluorfen (50594-66-6)	.096 ug/L	121 % Rec (SL)	15.7 % RSD (SL)	.2 ug/L
Dicamba (1918-00-9)	.081 ug/L	135 % Rec (SL)	32.4 % RSD (SL)	.4 ug/L
Herbicides (E-12839)	N/A	N/A	N/A	
5-Hydroxydicamba (7600-50-2)	.04 ug/L	103 % Rec (SL)	16.5 % RSD (SL)	.2 ug/L
Chloramben (133-90-4)	.093 ug/L	111 % Rec (SL)	14.4 % RSD (SL)	.4 ug/L
2,4,5-T (93-76-5)	.08 ug/L	117 % Rec (SL)	16.4 % RSD (SL)	.4 ug/L
Pentachlorophenol (87-86-5)	.076 ug/L	130 % Rec (SL)	31.2 % RSD (SL)	.04 ug/L

3,5-Dichlorobenzoic acid (51-36-5)	.061 ug/L	102 % Rec (SL)	16.3 % RSD (SL)	.6 ug/L
Dichlorprop (120-36-5)	.26 ug/L	107 % Rec (SL)	20.3 % RSD (SL)	2 ug/L
2,4-DB (94-82-6)	.8 ug/L	87 % Rec (SL)	13.1 % RSD (SL)	4 ug/L
Dalapon (75-99-0)	1.3 ug/L	100 % Rec (SL)	20 % RSD (SL)	10 ug/L
DCPA Diacid (2136-79-0)	.02 ug/L	74 % Rec (SL)	9.7 % RSD (SL)	.2 ug/L
MCPA (94-74-6)	.056 ug/L	N/A	N/A	
Dinoseb (88-85-7)	.19 ug/L	42 % Rec (SL)	14.3 % RSD (SL)	.4 ug/L
Organochlorine pesticides (E-12851)	N/A	N/A	N/A	
4-Nitrophenol (100-02-7)	.13 ug/L	131 % Rec (SL)	23.6 % RSD (SL)	1 ug/L
2,4-D (94-75-7)	.2 ug/L	131 % Rec	27.5 % RSD (SL)	1 ug/L

		(SL)		
Silvex (93-72-1)	.075 ug/L	134 % Rec (SL)	30.8 % RSD (SL)	.2 ug/L

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Precision Descriptor Notes	Precision and accuracy are based on a single laboratory study using 7 - 8 determinations of spiked organic-free reagent water obtained from diazomethane derivatization.
Detection Level Note	The EDL is defined as either the MDL or a concentration of analyte in a sample yielding a peak in the final extract with signal-to-noise ratio of approximately 5, whichever value is higher.

1.0 SCOPE AND APPLICATION

1.1 Method 8151 is a capillary gas chromatographic (GC) method for determining certain chlorinated acid herbicides and related compounds in aqueous, soil and waste matrices. Specifically, Method 8151 may be used to determine the following compounds:

Compound	CAS No.[a]
2,4-D	94-75-7
2,4-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5

Use a safety screen.
[a]Chemical Abstract Service Registry Number
Use mechanical pipetting aids.

1.2 Because these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151 describes a hydrolysis step that can be used to convert herbicide esters into the acid form prior to analysis. Herbicide esters generally have a half-life of less than one week in soil.

1.3 When Method 8151 is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. Sec. 8.4 provides gas chromatograph/mass spectrometer (GC/MS) criteria appropriate for the qualitative confirmation of compound identifications.

1.4 The estimated detection limits for each of the compounds in aqueous and soil matrices are listed in Table 1. The detection limits for a specific waste sample may differ from those listed, depending upon the nature of the interferences and the sample matrix.

1.5 The following compounds may also be determined using this method:

38. Appendix AH: EPA Method 8015C Nonhalogenated Organics by GC-FID

National Environmental Methods Index

NEMI Method Summary

Method Information	**Descriptive Name:** Nonhalogenated Organics by GC-FID **Official Name:** Nonhalogenated Organics Using GC/FID **Method Number:** 8015C **Revision:** Revision 3, Feb. 2007
Media	**Media:** VARIOUS
Subcategory	Organic
Method Source	**U.S. EPA Office of Solid Waste**
Citation	**SW-846 Update IVB**
Brief Method Summary	Depending on the analytes of interest, samples are introduced into the GC by a variety of techniques, including: purge-and-trap, direct injection of aqueous samples, injection of the concentrate from azeotropic distillation, vacuum distillation, or following solvent extraction. Analytes are detected by a flame ionization detector.
Scope and Application	This method determines the concentration of various nonhalogenated volatile and semivolatile organic compounds in surface water, ground water, and solid matrices. In addition, gasoline range organics and diesel range organics may also be analyzed by this method.
Applicable Conc Range	Not Available.
Method Download	**Download full method now (PDF file)**
Interferences	(A) Contamination: Sample contamination may occur by diffusion of volatile organics through the septum seal during shipment and storage. The analysis of trip blanks will identify this problem.

	(B) Matrix interferences: Since the FID is non-selective, there is a potential for the interference of non-target compounds. (C) Memory Interferences: Carryover may occur whenever high and low concentration samples are analyzed in sequence. Rinse the sample syringe or purging device between samples. Also, unusually concentrated samples should be followed by injection of a solvent blank.
QC Requirements	Refer to Chapter One and Methods 8000, 3500, and 5000 for specific QC procedures. Procedures include: validation of sample preparation, introduction, and analysis, initial demonstration of proficiency, calibration verification, evaluation of retention times, analysis of method blanks, matrix spikes, duplicates, surrogates and laboratory control samples.
Sample Handling	See See the introductory material to SW-846 Chapter Four, "Organic Analytes," and Method 5035 for information. If the headspace technique is used, also see Method 5021.
Max Holding Time	See SW-846 Chapter Four for information
Relative Cost/Effort	$201 to $400
Sample Prep Method(s)	See Method 5000

Method *8015C* has 35 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Pct False Positive	Pct False Negative	Spiking Level
Acetone (67-64-1)	16 ug/L	126 % Rec (SL)	17 % RSD (SL)			25 ug/L
Pyridine (110-86-1)	9 ug/L	92 % Rec (SL)	12 % RSD (SL)			25 ug/L
Paraldehyde (123-63-7)	N/A	N/A	N/A			
Isobutyl alcohol (78-83-1)	8 ug/L	122 % Rec	8 % RSD			25 ug/L

		(SL)	(SL)	
Tert-Butyl Alcohol (75-65-0)	7 ug/L	122 % Rec (SL)	7 % RSD (SL)	25 ug/L
n-Butyl alcohol (71-36-3)	8 ug/L	127 % Rec (SL)	8 % RSD (SL)	25 ug/L
Isopropanol (67-63-0)	17 ug/L	167 % Rec (SL)	13 % RSD (SL)	25 ug/L
n-Propyl alcohol (71-23-8)	7 ug/L	N/A	N/A	25 ug/L
Diethyl ether (60-29-7)	N/A	N/A	N/A	
Allyl alcohol (107-18-6)	N/A	N/A	N/A	
Herbicides (E-12839)	N/A	N/A	N/A	
2-Chloroacrylonitrile (920-37-6)	N/A	N/A	N/A	
Ethylene glycol (107-21-1)	N/A	N/A	N/A	
Ethylene oxide (75-21-8)	9 ug/L	114 % Rec (SL)	10 % RSD (SL)	25 ug/L
Ethanol (64-17-5)	12 ug/L	152 % Rec (SL)	10 % RSD (SL)	25 ug/L

Acrylonitrile (107-13-1)	9 ug/L	179 % Rec (SL)	7 % RSD (SL)	25 ug/L
Ethyl cyanide (107-12-0)	6 ug/L	135 % Rec (SL)	5 % RSD (SL)	25 ug/L
o-Toluidine (95-53-4)	N/A	N/A	N/A	
alpha-Picoline (109-06-8)	N/A	N/A	N/A	
Volatile organic compounds (E-12419)	N/A	N/A	N/A	
Crotonaldehyde (4170-30-3)	N/A	N/A	N/A	
Methyl n-propyl ketone (107-87-9)	2 ug/L	94 % Rec (SL)	3 % RSD (SL)	25 ug/L
Hexafluoro-2-propanol (I.S.) (920-66-1)	N/A	N/A	N/A	
Methyl ethyl ketone (78-93-3)	5 ug/L	105 % Rec (SL)	6 % RSD (SL)	25 ug/L
Acetonitrile (75-05-8)	6 ug/L	147 % Rec (SL)	5 % RSD (SL)	25 ug/L
Methanol (67-56-1)	21 ug/L	166 % Rec (SL)	14 % RSD (SL)	25 ug/L

Ethyl acetate (141-78-6)	8 ug/L	142 % Rec (SL)	7 % RSD (SL)	25 ug/L
p-Dioxane (123-91-1)	15 ug/L	124 % Rec (SL)	16 % RSD (SL)	25 ug/L
4-Methyl-2-pentanone (108-10-1)	2 ug/L	66 % Rec (SL)	4 % RSD (SL)	25 ug/L
Organochlorine pesticides (E-12851)	N/A	N/A	N/A	
N-Nitrosodi-n-butylamine (924-16-3)	N/A	N/A	N/A	
Hexafluoro-2-methyl-2-propanol (I.S.) (515-14-6)	N/A	N/A	N/A	
Acrolein (107-02-8)	15 ug/L	146 % Rec (SL)	13 % RSD (SL)	25 ug/L
p,p'-DDT (50-29-3)	N/A	N/A	N/A	
(E)-Crotonaldehyde (123-73-9)	N/A	N/A	N/A	

export_analytes

Precision Descriptor Notes	Example method performance data for non-purgeable volatiles in groundwater by azeotropic microdistillation (method 5031) are shown as examples and guidance. Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of

	this method.
Detection Level Note	-

1.0 SCOPE AND APPLICATION

1.1 Method 8015 may be used to determine the concentrations of various nonhalogenated volatile organic compounds and semivolatile organic compounds by gas chromatography. The following compounds can be determined quantitatively by this method, using the preparative techniques indicated.

Compound	CAS No.[a]	Appropriate Technique: Purge-and-Trap	Direct Aqueous Injection	Azeo. Dist.[b]	Vacuum Dist.[c]
Acetone	67-64-1	pp	x	x	x
Acetonitrile	75-05-8	pp	x	x	ne
Acrolein	107-02-8	pp	x	x	x
Acrylonitrile	107-13-1	pp	x	x	x
Allyl alcohol	107-18-6	ht	x	x	ne
1-Butanol (*n*-Butyl alcohol)	71-36-3	ht	x	x	ne
t-Butyl alcohol (2-Methyl-2-propanol)	75-65-0	pp	x	x	ne
Crotonaldehyde	123-73-9	pp	x	x	ne
Diethyl ether	60-29-7	x	x	ne	ne
1,4-Dioxane	123-91-1	pp	x	x	ne
Ethanol	64-17-5	l	x	x	x
Ethyl acetate	141-78-6	l	x	x	ne
Ethylene glycol	107-21-1	l	x	ne	ne
Ethylene oxide	75-21-8	l	x	x	ne
Isobutyl alcohol (2-Methyl-1-propanol)	78-83-1	pp	x	x	ne
Isopropyl alcohol (2-Propanol)	67-63-0	pp	x	x	ne
Methanol	67-56-1	l	x	x	ne
Methyl ethyl ketone (MEK, 2-Butanone)	78-93-3	pp	x	x	x
Methyl isobutyl ketone (MIBK, 4-Methyl-2-pentanone)	108-10-1	pp	x	x	x
N-Nitroso-di-*n*-butylamine	924-16-3	pp	x	x	ne
Paraldehyde	123-63-7	pp	x	x	ne
2-Pentanone	107-87-9	pp	x	x	ne
2-Picoline	109-06-8	pp	x	x	ne
1-Propanol (*n*-Propyl alcohol)	71-23-8	pp	x	x	ne

Compound	CAS No.[a]	Appropriate Technique: Purge-and-Trap	Direct Aqueous Injection	Azeo. Dist.[b]	Vacuum Dist.[c]

39. Appendix AI: EPA Method 8260B Volatile Organic Compounds by GC/MS

National Environmental Methods Index

NEMI Method Summary

Method Information	**Descriptive Name:** Volatile Organic Compounds by GC/MS **Official Name:** Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) **Method Number:** 8260B **Revision:** Rev. 2, 1996
Media	**Media:** VARIOUS
Subcategory	Organic
Method Source	**U.S. EPA Office of Solid Waste**
Citation	**SW-846 Online: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods**
Brief Method Summary	Volatile compounds are introduced into a GC with a capillary column by a purge-and-trap method or by other methods including azeotropic distillation, vacuum distillation, head space, direct injection, etc. Identification of target analytes os accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a five-point calibration curve.
Scope and Application	This method determines most volatile organic compounds with boiling points below 200 C.
Applicable Conc Range	Variable per analyte.
Method Download	**Download full method now (PDF file)**

Interferences	Contamination by diffusion of volatile organic compounds may occur through the septum seal of the sample container while in the laboratory or during shipment and/or storage. Special precautions must be taken when analyzing for methylene chloride, a common laboratory solvent. Teh analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explain this in text accompanying the uncorrected data.
QC Requirements	QC procedures include evaluation of retention time windows, calibration verification, acceptable chromatographic analysis of samples, GC/MS tuning to meet specifications including acceptable 4-bromofluorobenzene (BFB) every 12-hour shift, initial demonstration of proficiency by the laboratory, and analysis of appropriate QC samples (including method blanks, matrix spikes, duplicates, and laboratory control samples (LCS) in each analytical batch plus the addition of surrogates to each field sample and QC sample.
Sample Handling	Collect grab samples in 40 mL glass screw cap VOA vials with Teflon-lined silicone septa. If residual chlorine is present collect the sample in a 125 mL container which has been pre-preserved with sodium thiosulfate solution, swirl gently and then transfer to a 40 mL VOA vial. Cool to 4 degrees C and adjust pH to <2 from time of collection until analysis.
Max Holding Time	14 days (after collection)
Relative Cost/Effort	$201 to $400
Sample Prep Method(s)	5030, 5035, 5031, 5032, 5021, 5041

Method *8260B* has 120 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Pct False Positive	Pct False Negative	Spiking Level
1,2,3-Trichlorobenzene (87-61-6)	N/A	102 % Rec	8.7 % RSD (SL)			.5 ug/L

		(SL)		
n-Propylbenzene (103-65-1)	N/A	99 % Rec (SL)	6.7 % RSD (SL)	.5 ug/L
Iodomethane (74-88-4)	N/A	N/A	0 % RSD (SL)	0 ug/L
p-Dioxane (123-91-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
trans-1,2-Dichloroethene (156-60-5)	N/A	98 % Rec (SL)	7.3 % RSD (SL)	.1 ug/L
cis-1,2-Dichloroethene (156-59-2)	N/A	100 % Rec (SL)	3.7 % RSD (SL)	.1 ug/L
2-Chloroethylvinyl ether (110-75-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
Carbon disulfide (75-15-0)	N/A	N/A	0 % RSD (SL)	0 ug/L
Organics, semivolatile (E-12884)	N/A	N/A	N/A	
m-Xylene (108-38-3)	N/A	106 % Rec (SL)	4.3 % RSD (SL)	.5 ug/L
1,1,1-Trichloroethane (71-55-6)	N/A	100 % Rec	4 % RSD (SL)	.5 ug/L

(SL)

Pyridine (110-86-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
n-Propyl Amine (107-10-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
2-Nitropropane (79-46-9)	N/A	N/A	0 % RSD (SL)	0 ug/L
Naphthalene (91-20-3)	N/A	98 % Rec (SL)	7.3 % RSD (SL)	.5 ug/L
Cumene (98-82-8)	N/A	102 % Rec (SL)	6.3 % RSD (SL)	.5 ug/L
2-Hexanone (591-78-6)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,1-Dichloroethene (75-35-4)	N/A	95 % Rec (SL)	9.5 % RSD (SL)	.1 ug/L
p-Chlorotoluene (106-43-4)	N/A	96 % Rec (SL)	7.3 % RSD (SL)	.5 ug/L
n-Butylbenzene (104-51-8)	N/A	94 % Rec (SL)	6.4 % RSD (SL)	.5 ug/L
n-Butyl alcohol	N/A	N/A	0 %	0

(71-36-3)			RSD (SL)	ug/L
Bromodichloromethane (75-27-4)	N/A	100 % Rec (SL)	4.6 % RSD (SL)	.1 ug/L
Bromochloromethane (74-97-5)	N/A	97 % Rec (SL)	6 % RSD (SL)	.5 ug/L
Acetonitrile (75-05-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,2,4-Trichlorobenzene (120-82-1)	N/A	91 % Rec (SL)	17.6 % RSD (SL)	.5 ug/L
Ethyl cyanide (107-12-0)	N/A	N/A	0 % RSD (SL)	0 ug/L
Pentachloroethane (76-01-7)	N/A	N/A	0 % RSD (SL)	0 ug/L
N-Nitrosodi-n-butylamine (924-16-3)	N/A	N/A	0 % RSD (SL)	0 ug/L
Methacrylonitrile (126-98-7)	N/A	N/A	0 % RSD (SL)	0 ug/L
Hexachloroethane (67-72-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
Ethylbenzene	N/A	99 %	5.3 % RSD	.5

(100-41-4)		Rec (SL)	(SL)	ug/L
Ethanol (64-17-5)	N/A	N/A	0 % RSD (SL)	0 ug/L
Erythritol anhydride (1464-53-5)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,4-Dichlorobenzene (106-46-7)	N/A	106 % Rec (SL)	6.1 % RSD (SL)	.1 ug/L
Dibromomethane (74-95-3)	N/A	93 % Rec (SL)	6 % RSD (SL)	.5 ug/L
1,2-Dibromoethane (106-93-4)	N/A	97 % Rec (SL)	5.8 % RSD (SL)	.5 ug/L
Dibromochloromethane (124-48-1)	N/A	99 % Rec (SL)	5.7 % RSD (SL)	.1 ug/L
Chloromethane (74-87-3)	N/A	101 % Rec (SL)	4.7 % RSD (SL)	.5 ug/L
Chloroform (67-66-3)	N/A	105 % Rec (SL)	3 % RSD (SL)	.1 ug/L
Allyl chloride (107-05-1)	N/A	N/A	0 % RSD (SL)	0 ug/L

Herbicides (E-12839)	N/A	N/A	N/A	
Vinyl chloride (75-01-4)	N/A	104 % Rec (SL)	.2 % RSD (SL)	.1 ug/L
Trichlorofluoromethane (75-69-4)	N/A	97 % Rec (SL)	4.7 % RSD (SL)	.1 ug/L
Trichloroethene (79-01-6)	N/A	104 % Rec (SL)	1.9 % RSD (SL)	.1 ug/L
1,1,2-Trichloroethane (79-00-5)	N/A	102 % Rec (SL)	4.8 % RSD (SL)	.5 ug/L
1,1,2,2-Tetrachloroethane (79-34-5)	N/A	100 % Rec (SL)	12 % RSD (SL)	.5 ug/L
Isopropanol (67-63-0)	N/A	N/A	0 % RSD (SL)	0 ug/L
Methylene chloride (75-09-2)	N/A	97 % Rec (SL)	13.4 % RSD (SL)	.5 ug/L
p-Cymene (99-87-6)	N/A	113 % Rec (SL)	11.5 % RSD (SL)	.5 ug/L
Ethyl methacrylate (97-63-2)	N/A	N/A	0 % RSD (SL)	0 ug/L

Ethylene oxide (75-21-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
Ethyl acetate (141-78-6)	N/A	N/A	0 % RSD (SL)	0 ug/L
Diethyl ether (60-29-7)	N/A	N/A	0 % RSD (SL)	0 ug/L
DBCP (96-12-8)	N/A	92 % Rec (SL)	10.9 % RSD (SL)	.5 ug/L
o-Chlorotoluene (95-49-8)	N/A	99 % Rec (SL)	4.6 % RSD (SL)	.5 ug/L
3-Chloropropionitrile (542-76-7)	N/A	N/A	0 % RSD (SL)	0 ug/L
Methyl ethyl ketone (78-93-3)	N/A	N/A	0 % RSD (SL)	0 ug/L
Allyl alcohol (107-18-6)	N/A	N/A	0 % RSD (SL)	0 ug/L
o-Xylene (95-47-6)	N/A	106 % Rec (SL)	7.1 % RSD (SL)	.5 ug/L
Vinyl acetate (108-05-4)	N/A	N/A	0 % RSD (SL)	0 ug/L

Mesitylene (108-67-8)	N/A	101 % Rec (SL)	4.2 % RSD (SL)	.5 ug/L
Propargyl Alcohol (107-19-7)	N/A	N/A	0 % RSD (SL)	0 ug/L
4-Methyl-2-pentanone (108-10-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
Methyl methacrylate (80-62-6)	N/A	N/A	0 % RSD (SL)	0 ug/L
Methanol (67-56-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
Lactonitrile (78-97-7)	N/A	N/A	0 % RSD (SL)	0 ug/L
Epichlorohydrin (106-89-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,3-Dichloro-2-propanol (96-23-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,3-Dichloropropane (142-28-9)	N/A	99 % Rec (SL)	5.9 % RSD (SL)	.5 ug/L
1,1-Dichloroethane (75-34-3)	N/A	98 % Rec (SL)	6.3 % RSD (SL)	.5 ug/L

trans-1,4-Dichloro-2-butene (110-57-6)	N/A	N/A	0 % RSD (SL)	0 ug/L
Crotonaldehyde (4170-30-3)	N/A	N/A	0 % RSD (SL)	0 ug/L
Chlorobenzene (108-90-7)	N/A	91 % Rec (SL)	6.4 % RSD (SL)	.1 ug/L
Carbon tetrachloride (56-23-5)	N/A	108 % Rec (SL)	6.3 % RSD (SL)	.1 ug/L
tert-Butylbenzene (98-06-6)	N/A	110 % Rec (SL)	2.3 % RSD (SL)	.5 ug/L
Bromoform (75-25-2)	N/A	101 % Rec (SL)	5.3 % RSD (SL)	.5 ug/L
Acetone (67-64-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
Volatile organic compounds (E-12419)	N/A	N/A	N/A	
p-Xylene (106-42-3)	N/A	97 % Rec (SL)	6.3 % RSD (SL)	.5 ug/L
1,2,3-Trichloropropane (96-18-4)	N/A	96 % Rec (SL)	6.8 % RSD (SL)	.5 ug/L

o-Toluidine (95-53-4)	N/A	N/A	0 % RSD (SL)	0 ug/L
Styrene (100-42-5)	N/A	96 % Rec (SL)	19.8 % RSD (SL)	.5 ug/L
Propiolactone (57-57-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
Methyl n-propyl ketone (107-87-9)	N/A	N/A	0 % RSD (SL)	0 ug/L
Nitrobenzene (98-95-3)	N/A	N/A	0 % RSD (SL)	0 ug/L
cis-1,3-Dichloropropene (10061-01-5)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,3-Dichlorobenzene (541-73-1)	N/A	101 % Rec (SL)	5.9 % RSD (SL)	.1 ug/L
Bromoacetone (598-31-2)	N/A	N/A	0 % RSD (SL)	0 ug/L
Toluene (108-88-3)	N/A	100 % Rec (SL)	5.9 % RSD (SL)	.5 ug/L
Tetrachloroethene (127-18-4)	N/A	96 % Rec (SL)	5.2 % RSD (SL)	.1 ug/L

alpha-Picoline (109-06-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
Isobutyl alcohol (78-83-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
Hexachlorobutadiene (87-68-3)	N/A	100 % Rec (SL)	6.7 % RSD (SL)	.5 ug/L
trans-1,3-Dichloropropene (10061-02-6)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,1-Dichloropropene (563-58-6)	N/A	102 % Rec (SL)	7.3 % RSD (SL)	.5 ug/L
sec-Dichloropropane (594-20-7)	N/A	99 % Rec (SL)	4.9 % RSD (SL)	.5 ug/L
1,2-Dichloropropane (78-87-5)	N/A	96 % Rec (SL)	6.3 % RSD (SL)	.5 ug/L
Dichlorodifluoromethane (75-71-8)	N/A	99 % Rec (SL)	8.9 % RSD (SL)	.1 ug/L
1,4-Dichloro-cis-2-butene (1476-11-5)	N/A	N/A	0 % RSD (SL)	0 ug/L
Chloral hydrate (302-17-0)	N/A	N/A	0 % RSD (SL)	0 ug/L

sec-Butylbenzene (135-98-8)	N/A	110 % Rec (SL)	6.5 % RSD (SL)	.5 ug/L
Tert-Butyl Alcohol (75-65-0)	N/A	N/A	0 % RSD (SL)	0 ug/L
Benzyl chloride (100-44-7)	N/A	N/A	0 % RSD (SL)	0 ug/L
Benzene (71-43-2)	N/A	99 % Rec (SL)	6.3 % RSD (SL)	.1 ug/L
Acrylonitrile (107-13-1)	N/A	N/A	0 % RSD (SL)	0 ug/L
Acrolein (107-02-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,2,4-Trimethylbenzene (95-63-6)	N/A	96 % Rec (SL)	6.8 % RSD (SL)	.5 ug/L
1,1,1,2-Tetrachloroethane (630-20-6)	N/A	100 % Rec (SL)	4.7 % RSD (SL)	.5 ug/L
n-Propyl alcohol (71-23-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
Paraldehyde (123-63-7)	N/A	N/A	0 % RSD (SL)	0 ug/L

Malononitrile (109-77-3)	N/A	N/A	0 % RSD (SL)	0 ug/L
1,2-Dichloroethane (107-06-2)	N/A	100 % Rec (SL)	6.3 % RSD (SL)	.1 ug/L
1,2-Dichlorobenzene (95-50-1)	N/A	97 % Rec (SL)	3.6 % RSD (SL)	.1 ug/L
Chloroprene (126-99-8)	N/A	N/A	0 % RSD (SL)	0 ug/L
2-Chloroethanol (107-07-3)	N/A	N/A	0 % RSD (SL)	0 ug/L
Chloroethane (75-00-3)	N/A	100 % Rec (SL)	5.8 % RSD (SL)	.1 ug/L
Bromomethane (74-83-9)	N/A	99 % Rec (SL)	7.2 % RSD (SL)	.5 ug/L
Bromobenzene (108-86-1)	N/A	97 % Rec (SL)	7.6 % RSD (SL)	.5 ug/L
Sulfur mustard (505-60-2)	N/A	N/A	0 % RSD (SL)	0 ug/L
(E)-Crotonaldehyde (123-73-9)	N/A	N/A	N/A	

export_analytes

Precision Descriptor Notes	See performance tables in method.
Detection Level Note	Detection limits using a variety of sample preparative methods are included.

1.0 SCOPE AND APPLICATION

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds can be determined by this method:

Compound	CAS No.[b]	Appropriate Preparation Technique[a] 5030/ 5035	5031	5032	5021	5041	Direct Inject.
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein (Propenal)	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d (IS)[c]		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-07-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	l	nd	nd	nd	nd	pc

(continued)

Appropriate Preparation Technique[a]
5030/ Direc

40. Appendix AJ: EPA Method 351.2 Total Kjeldahl nitrogen in water by semiautomated colorimetry

National Environmental Methods Index

NEMI Method Summary

Method Information	**Descriptive Name:** Total Kjeldahl nitrogen in water by semiautomated colorimetry **Official Name:** Method 351.2: Determination of Total Kjeldahl nitrogen by semi-automated colorimetry **Method Number:** 351.2 **Revision:** Revision 2.0, August 1993
Media	**Media:** WATER
Subcategory	Inorganic
Method Source	**U.S. EPA Office of Research and Development and Office of Science and Technology**
Citation	**Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/600/4-79/020)**
Brief Method Summary	The sample is heated in the presence of sulfuric acid, H_2SO_4 for two and one half hours.The residue is cooled, diluted to 25 mL and analyzed for ammonia.This digested sample may also be used for phosphorus determination. Total Kjeldahl nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2SO_4$, under the conditions of digestion described. Organic Kjeldahl nitrogen is the difference obtained by subtracting the free ammonia value from the total Kjeldahl nitrogen value. Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method.
Scope and Application	This method covers the determination of total Kjeldahl nitrogen in drinking, ground, and surface waters, domestic and industrial wastes.The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.

Applicable Conc Range	0.1-20
Method Download	**Download full method now (PDF file)**
Interferences	High nitrate concentrations (10X or more than the TKN level) result in low TKN values.If interference is suspected, samples should be diluted and reanalyzed. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.
QC Requirements	Demonstration of laboratory capability, and periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance.
Sample Handling	Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleansed and rinsed with reagent water. Samples must be preserved with H_2SO_4 to a pH < 2 and cooled to a 4°C at the time of collection. Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples should be maintained at 4°C.
Max Holding Time	28 days
Relative Cost/Effort	Less than $50
Sample Prep Method(s)	

Method *351.2* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Pct False Positive	Pct False Negative	Spiking Level
Total Kjeldahl nitrogen (E-10264)	.1 mg/L	102 % Rec (ML)	19.3 % RSD (ML)			.38 mg/L

export_analytes

Precision Descriptor Notes	Precision and accuracy values were calculated using interlaboratory data from performance evaluation studies.
Detection Level Note	MDL is established using reagent water fortified at a concentration of 2 to 3 times the estimated instrument detection limit and is determined from 7 replicate aliquots of the fortified reagent water and processed through the entire analytical method.

METHOD 351.2

DETERMINATION OF TOTAL KJELDAHL NITROGEN BY SEMI-AUTOMATEDCOLORIMETRY

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

41. Appendix AK: EPA Method 351.2 Turbidity by Nephelometry

National Environmental Methods Index

NEMI Method Summary

Method Information	**Descriptive Name:** Turbidity by Turbidimeter **Official Name:** Determination of Turbidity by Nephelometry **Method Number:** 180.1 **Revision:** Revision 2.0, August 1993
Media	**Media:** WATER
Subcategory	Inorganic
Method Source	**U.S. EPA National Exposure Research Laboratory (NERL) [formerly EMSL]**
Citation	**Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)**
Brief Method Summary	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 the method. A standard suspension (i.e., formazin, AMCO-AEPA-1, or Hach Stablcal) is used to calibrate the instrument.
Scope and Application	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.
Applicable Conc Range	0 - 40 NTU
Method Download	**Download full method now (PDF file)**
Interferences	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. The presence of true color, that is the color of water which is due to dissolved substances which absorb light, will cause turbidities

	to be low, although this effect is generally not significant with finished waters.
QC Requirements	None.
Sample Handling	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.
Max Holding Time	48 hours (MCAWW, Table I)
Relative Cost/Effort	Less than $50
Sample Prep Method(s)	None.

Method *180.1* has 1 analytes associated with it.

Analyte	Detection Level	Bias	Precision	Pct False Positive	Pct False Negative	Spiking Level
Turbidity (E-10617)	0 NTU	N/A	2.3 % RSD (SL)			26 NTU

export_analytes

Precision Descriptor Notes	Precision was determined by analyzing replicate surface water samples in a single laboratory. The method contains data for samples ranging from 26 to 180 NTU.
Detection Level Note	None.

METHOD 180.1

DETERMINATION OF TURBIDITY BY NEPHELOMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
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Water Quality Vital Signs Monitoring Protocol for Park Units in the Northern Colorado Plateau Network

STANDARD OPERATING PROCEDURE (SOP) #**14**

Data Management

Version 2.00 (4/27/2016)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.00	4/2016	H. Thomas	Updates were made throughout to reflect the move from a custom database to NPSTORET and other changes in data management practices.	To reflect current data management practices.	2.00

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. The procedures for data management given in this SOP follow guidelines and standards that are described in the NCPN Data Management Plan (Beer et al. 2005).

Definitions and Acronyms

ARCH	Arches National Park
BLCA	Black Canyon of the Gunnison National Park
BRCA	Bryce Canyon National Park
CANY	Canyonlands National Park

CARE	Capitol Reef National Park
Certified data	Data that have been through all planned QA/QC procedures and meet the quality standards documented in the Quality Assurance Project Plan, and are documented in appropriate metadata formats.
Colorado parks	Parks in which samples are collected by NPS staff and analysis is done by USGS (BLCA and CURE).
CURE	Curecanti National Recreation Area
Data Store	The NPS-wide repository for documents, publications, and data sets related to NPS natural and cultural resources (https://irma.nps.gov/DataStore/).
DINO	Dinosaur National Monument
FGDC	Federal Geographic Data Committee. The interagency committee that promotes the coordinated development, use, sharing, and dissemination of geographic data.
HOVE	Hovenweep National Monument
I&M	Inventory & Monitoring Program of the National Park Service
NABR	Natural Bridges National Monument
NCPN	Northern Colorado Plateau Network
NPS	National Park Service
NPSTORET	Microsoft Access database used to store data locally in a STORET-compatible format.
NWIS	National Water Information System database maintained by USGS.
NWQL	USGS National Water Quality Laboratory.
PDF	Portable Document Format
QA	Quality assurance - procedures implemented to ensure collection of data with minimal errors.
QC	Quality control - procedures implemented to flag or modify data to ensure that data meet established data quality standards.
SOP	Standard Operating Procedure
STORET	STOrage and RETrieval database maintained by EPA, also referred to as EPA-STORET.
TICA	Timpanogos Cave National Monument
USGS-sampled parks	Parks in which USGS handles sample collection and analysis (DINO).
Utah parks	Parks in which samples are collected by NPS staff and analysis is done by the Utah state lab (ARCH, BRCA, CANY, CARE, HOVE, NABR, TICA, and

	ZION)
UTDWQ	Utah Division of Water Quality
WRD	NPS Water Resources Division
WRD-STORET	STORET database maintained by the NPS Water Resources Division.
ZION	Zion National Park

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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 (refer to Figure 1). Data acquired by USGS cooperators reside in the USGS National Water Information System (NWIS) database. Data acquired through the State of Utah reside in EPA-STORET, and field data collected by NCPN staff reside in NPSTORET, a Microsoft Access database that consolidates water quality data collected in and near the 16 NCPN park units.

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, although NCPN water quality monitoring data initially reside in disparate databases, all data will ultimately be stored in WRD-STORET.

The following sections briefly outline the flow of data for the NCPN water quality monitoring program.

1.1. Utah Parks

Samples are collected by NPS staff in eight Utah parks (ARCH, BRCA, CANY, CARE, HOVE, NABR, TICA, and ZION). NCPN does the sampling in BRCA, CARE, and ZION, while park staff collects samples in ARCH, CANY, HOVE, NABR, and TICA. Samples are sent to the UTDWQ laboratory for analysis. The state enters the data into their local Ambient Water Quality Monitoring System (AWQMS), and then transfers it to EPA-STORET and to NCPN. Field data collected by park staff are quality checked and then transferred to NCPN. NCPN uploads all field and lab data to NPSTORET, and performs additional quality checking. On a regular basis (usually annually), NCPN will export the data from NPSTORET and submit it to WRD for upload to WRD-STORET.

1.2. Colorado Parks

Samples are collected by NPS staff in two Colorado parks (BLCA and CURE) and are sent to the USGS National Water Quality Laboratory (NWQL) for analysis. The BLCA/CURE hydrologist quality checks the data and then transfers it to NCPN in a spreadsheet format. NCPN uploads the data to NPSTORET. On a regular basis (usually annually), NCPN will export the data from NPSTORET and submit it to WRD for upload to WRD-STORET.

1.3. USGS-Sampled Parks

Samples are collected by USGS staff at sites in DINO through a cooperative agreement with NCPN. Field and lab data for these sites are sent to NWQL for analysis, entered into the NWIS database, and quality checked by USGS. On a regular basis (usually annually), NCPN will download the data from NWIS and upload it to NPSTORET. NCPN will also export the data from NPSTORET and submit it to WRD for upload to WRD-STORET.

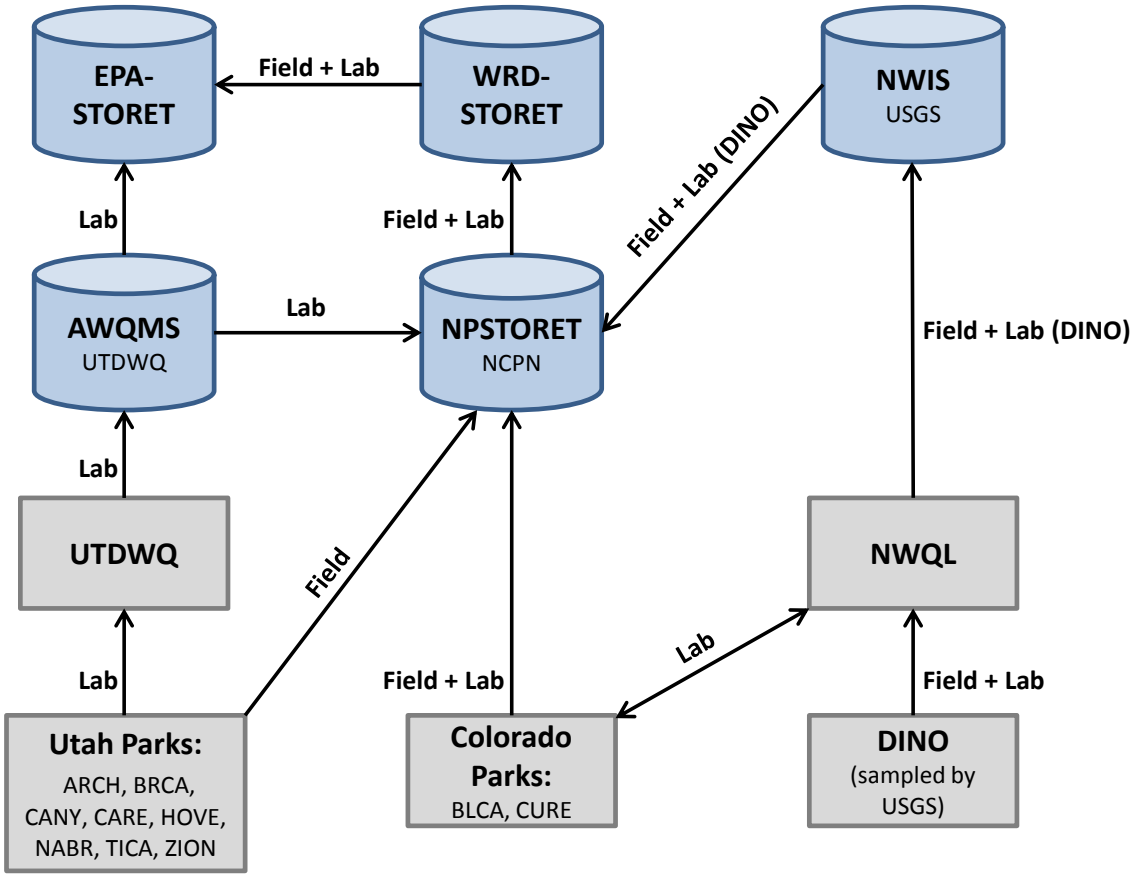


Figure 1. Data flows for the NCPN water quality monitoring program.

2. Database Design

The primary database used to manage NCPN water quality monitoring data is NPSTORET, a Microsoft Access database developed and maintained by the NPS WRD. NPSTORET has a frontend/backend file structure, allowing multiple users to enter data in a network environment. In addition to a data entry interface, tools are available for importing data in a variety of formats, such as EPA-STORET and NWIS data files. NPSTORET also has a number of reporting and export features, as well as built-in water quality standards assessment tools that allow comparisons of historical and current data with applicable state standards.

Figure 2 shows the relationships among the primary tables in NPSTORET (note that only a small subset of the tables is 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 characteristics - 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 data dictionary contained in the NPSTORET installation file (available at http://www.nature.nps.gov/water/vitalsigns/vitalsignsmgt.cfm#downloads).

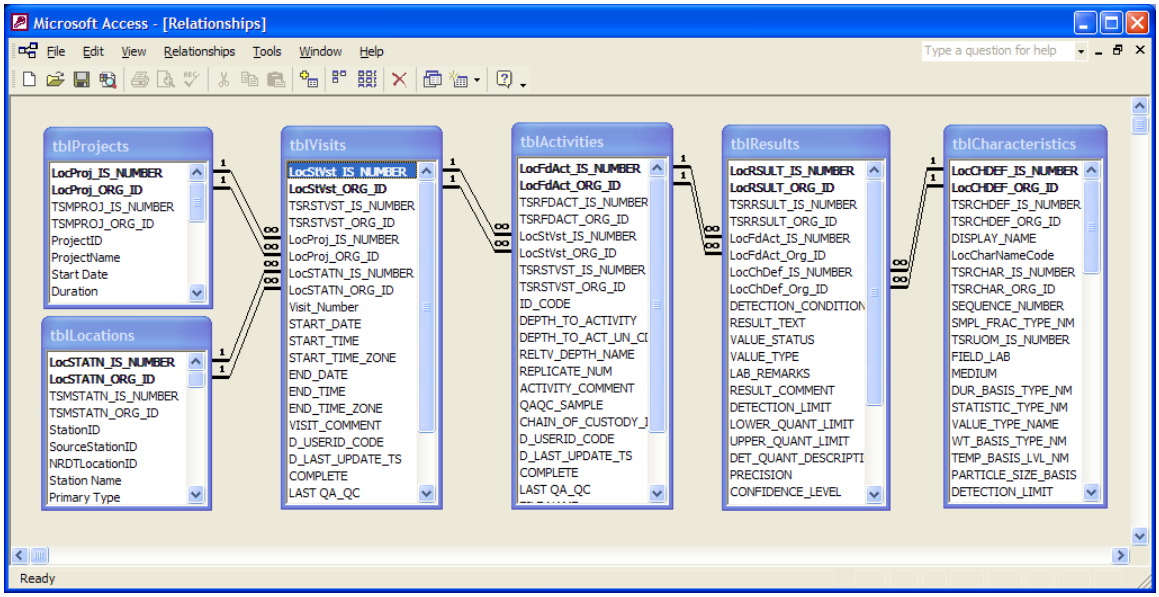


Figure 2. Data model for the NPSTORET database.

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 (refer to 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) to a year (UTDWQ) before it is available. Water quality databases are populated via a combination of data entry and data import.

3.1. Field Data Entry

Certain field measurements are recorded digitally using field instruments, while others are recorded manually on paper field forms. For Utah parks, all field data are recorded in an electronic *Field Form* spreadsheet after a site visit. The Field Form spreadsheet is a custom-developed Excel tool designed for water quality monitoring data. It includes QC checks and accommodates calibration data. Data recorded on paper field forms are entered manually into the Field Form, while data recorded digitally are imported from field instruments. 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 to digital format with 100% accuracy.*

3.2. Data Import into NPSTORET

All field and lab data are imported into NPSTORET. The different data file formats used for the three monitoring groups require different import methods.

3.2.1. Utah Parks

NCPN has developed a tool, FF2NPSTORET, to transform data in the Field Form spreadsheet to a format easily ingested by NPSTORET. FF2NPSTORET is written in Excel Visual Basic for Applications (VBA). It takes one or more Field Form spreadsheets as input and generates an Excel output file that can be imported using the NPSTORET import utility. FF2NPSTORET ensures that field measurements are mapped to the appropriate characteristics in NPSTORET. Field data for Utah parks are imported to NPSTORET using this process on a regular basis, usually at the end of the water year after the data have been quality checked.

NCPN has also developed an Excel VBA tool, UTLAB2NPSTORET, to transform the lab data for Utah parks to a format that is readily imported into NPSTORET. NCPN downloads the lab data from AWQMS in a spreadsheet format. The UTLAB2NPSTORET tool takes a lab data file as input and generates an Excel output file that can be imported using the NPSTORET import utility. The tool ensures that lab results are mapped to the appropriate characteristics in NPSTORET. Lab data for Utah parks are imported to NPSTORET using this approach once UTDWQ makes the data available, usually on an annual basis. Although field and lab data are imported into NPSTORET separately, they are matched during import based on visit date and stored with the same site visit record.

3.2.2. Colorado Parks

NCPN obtains field and lab data for BLCA and CURE from the park hydrologist in a spreadsheet format. NCPN ensures that all results in the data file are mapped to the appropriate characteristics in NPSTORET. The data file is then converted from column-major format to row-major format using the NPSCol2Row utility developed by WRD. NPSCol2Row generates a row-major text file that is then imported using the NPSTORET import utility. Data for Colorado parks is usually imported into NPSTORET on an annual basis.

3.2.3. USGS-Sampled Parks

Field and lab data for DINO sites that are sampled by USGS staff are entered into the NWIS database. NCPN downloads these data from NWIS and then imports them into NPSTORET. The NPSTORET import utility has built-in support for NWIS data files, so no additional tools are needed. Data for USGS-sampled sites are imported into NPSTORET on a regular basis, usually at the end of the water year.

3.3. Data Import into WRD-STORET

On a regular basis (usually annually), NCPN will export newly certified data from NPSTORET and submit it to WRD for upload to WRD-STORET. This can be done using the Export tool in NPSTORET to select the appropriate data by date range and Status Indicator. Results submitted to WRD should have the Status Indicator set to "Final", which indicates that the record is certified, i.e., all QA/QC procedures have been completed and the record meets the quality standards documented in the Quality Assurance Project Plan (refer to SOP 7). WRD staff will upload the data to WRD-STORET.

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 (refer to SOP 7).

4.1. Data 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. All records 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. Data Validation

Data validation involves checking the data for range and logic errors. 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. In addition, each site page of the Field Form spreadsheet contains functions that automatically report completeness for field data collection and the status of quality control procedures.

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.

4.3. 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.

4.4. 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. Adjustments to original data may be made to make data backwards compatible once it is extracted from the database, 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. NCPN uses several techniques to help ensure proper version control: data archiving (refer to the Data Archiving section), directory structure conventions, and backup procedures.

5.1. Directory Structure Conventions

NCPN has adopted directory structure conventions for I&M project folders (NCPN 2007). Based on these conventions, each project has an active folder for storing all current or working project files and an archive folder for storing archived project files. In general, the active project folder stores only the most recent project files (e.g., data and reports from current year, current protocol version). All active project folders are stored on the NCPN shared drive (X:). The active water quality monitoring folder is in the following location:

X:\Active_Monitoring_Projects\Water_Quality

The general structure of this folder is as follows (all subfolders are not shown):

- **Data**: Contains all current year data. Includes the following subfolders to distinguish different types of data (all subfolders are not shown):
    - **Data_lab**: Contains lab results data.
    - **Field_forms**: Contains Field Form spreadsheets.
    - **FlowTracker**: Contains raw flow data.
    - **NPSTORET**: Contains the current NPSTORET database backend file.
    - **Troll**: Contains raw core parameter data.
- **Protocol**: Stores the current version of the protocol, including narrative and SOP files.
- **Reports**: Stores the current annual report and any other reports produced during the current year.

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. More details are provided in the Data Archiving section.

5.2. Backup Procedures

All water quality monitoring files are stored on a secure server with regular backup routines that include an off-site storage rotation. In addition, data files are backed up before any edits are made. For example, before editing data in the NPSTORET database, the user creates a backup of the backend database file and stores it in the designated backup folder. The user appends the date and his or her initials to the backup file. Backup folders are periodically reviewed, and any backup files that are no longer needed are removed.

6. Data Archiving

Archiving is a critical step in ensuring the long-term preservation of data. Water quality monitoring data will generally be archived on an annual basis, after the current year's data have been certified. Archiving will be done for both digital and hard copy data.

6.1. Digital data archiving

All digital water quality monitoring data will be archived on the NCPN read-only shared drive (R:) in the Water Quality archive folder (R:\Monitoring_Archive\Water_Quality). Table 1 lists the water quality digital data files that require archiving, along with their respective archive folder locations. The digital files listed in Table 1 will also be burned onto an archival-standard CD or DVD and sent to the Western Archeological and Conservation Center (WACC) according to the procedures outlined in the NCPN Archiving Guidance Document (NCPN 2010).

Table 1 Water quality monitoring digital data archiving summary

Data Product(s)/File(s)	Archive Location[1]	Comments

[1] Archive location is relative to R:\Monitoring_Archive\Water_Quality unless stated otherwise.

Data Product(s)/File(s)	Archive Location[1]	Comments
Database	Data\<*year*[2]>\Database	Contains the master NPSTORET backend file, cumulative through the year indicated.
Field forms	Data\<*year*>\Field_Forms	Contains archived Field Form spreadsheets for a given year.
Photos	R:\NCPN_Photos_Archive\NCPN Photos\WaterQuality	Archive location is an absolute path. Renamed photo files archived as part of NCPN photo database processing - refer to NCPN Photo Management Guidance Document (NCPN-NPS 2008) for details.
Protocol	Protocol\<*version*[3]>	Contains archived protocol files for a given version.
Raw data	Data\<*year*>\Raw_Data	Contains raw data downloaded from field instruments, with subfolders indicating the type of data (e.g., flow, sonde).
Reports	Reports\<*year*>	Contains archived report files for a given year.

6.2. Hard copy data archiving

All hard copy water quality monitoring data will be archived at WACC according to the procedures outlined in the NCPN Archiving Guidance Document (NCPN 2010). Table 2 lists the water quality hard copy data files that require archiving, along with a summary of archiving procedures.

Table 2 Water quality monitoring hard copy data archiving summary

Data Product(s)/File(s)	Comments
Field forms	Original field forms stored in NCPN archives at WACC.
Reports	Archival copies stored in NCPN archives at WACC. Hard copies retained in NCPN office.
Photos	Selected prints stored in NCPN archives at WACC.
Protocol	Archival copies stored in NCPN archives at WACC. Hard copies retained in NCPN office.
Data/metadata printouts	Archival copies of any data/metadata printouts stored in NCPN archives at WACC. Hard copies retained in NCPN office.

[2] <*year*> indicates year in yyyy format.

[3] <*version*> indicates protocol version key number, e.g., Version_Key_1

7. Metadata

Metadata, which is defined as structured information about the content, quality, condition, and other characteristics of data, is a critical step toward ensuring that data sets are usable for their intended purposes well into the future. Metadata can take many different forms, including formal FGDC-compliant metadata, Data Store References, and protocol version information. This section describes the main types of metadata that NCPN maintains for water quality monitoring.

A metadata record will be created for the NPSTORET database following NCPN metadata procedures (NCPN 2007b) and uploaded to the Data Store. It will describe how the data were collected and quality checked. The metadata record will be updated as new data are certified.

Several types of metadata are maintained within the NPSTORET database, such as characteristic definitions and field/lab analytical procedures. This information describes in detail what was measured and how. These metadata tables also allow changes in instrumentation and/or procedures to be tracked over time.

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 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. References

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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 1.01 (12/13/10)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.00	12/2010	R. Weissinger	Reduce frequency of reports	Accumulate more data for more informative analyses	1.01

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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:

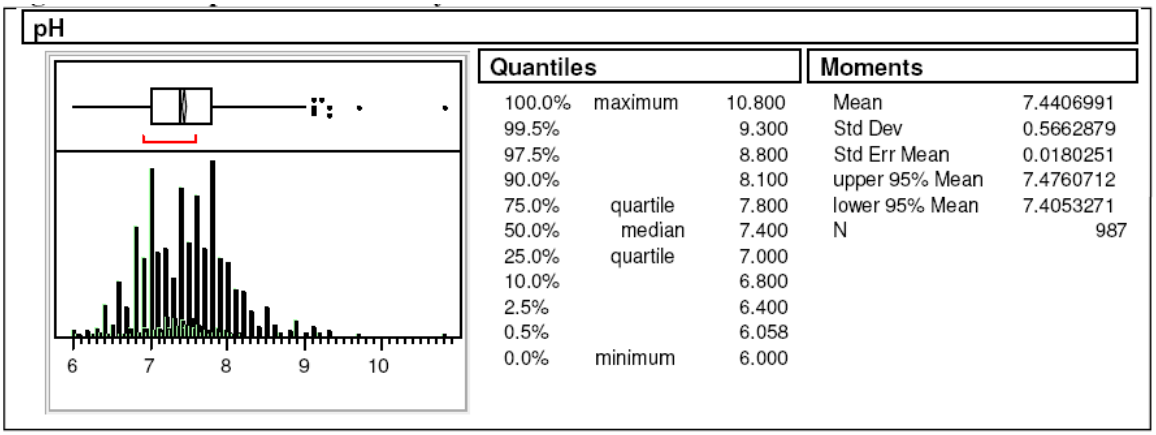
1. histograms (e.g., streamflow vs. number of occurrences): useful for depicting large differences in shape or symmetry. They are better for data that have natural categories or groupings; they are not as good with continuous data since it is difficult to depict this type of data accurately in a discrete 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).

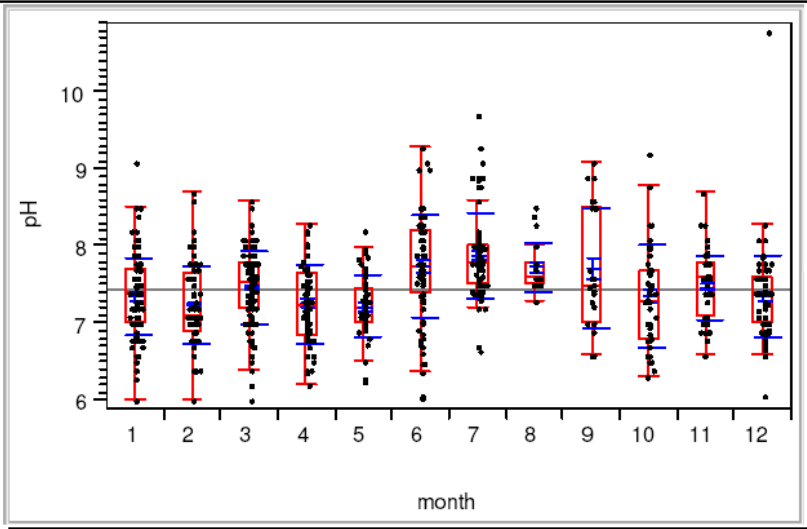
Figure 3.1. Example data summary chart from Stafford and Horne, 2004.



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.

Oneway Analysis of pH By month



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 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. Exceedance and summary reports:

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**

c. Include as a report appendix copies of standards and designated beneficial uses used for comparisons, as water quality standards and beneficial uses can change over time.

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 reports

1. A comprehensive data analysis and synthesis for 5 or more 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 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

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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.

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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:

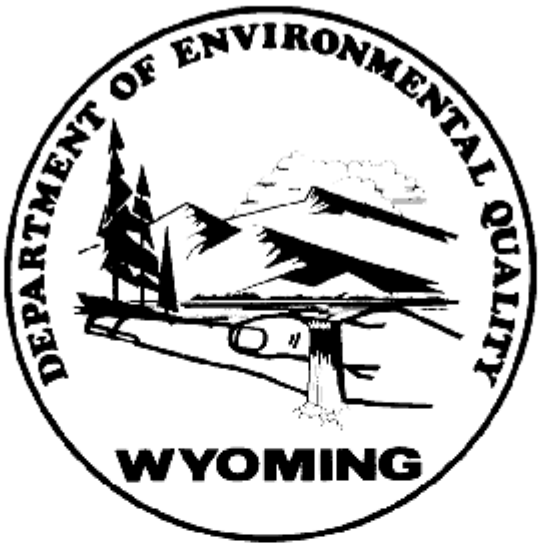
In accordance with the requirements of 40 C.F.R. § 131.6(e), I have reviewed the materials submitted by your office, and hereby certify as legal counsel to the Utah Water Quality Board, that based on that information, the revised water quality standards (R317-2, Utah Administrative Code) which were adopted 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



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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 1.06 (4/29/16)

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	Updated MVT	Changes to SOP #8	1.02
1.02	5/27/09	H. Thomas	Updated MVT	Changes to SOPs #0 and #1	1.03
1.03	8/27/09	H. Thomas	Updated MVT	Changes to SOP #1	1.04
1.04	3/16/10	H. Thomas	Updated MVT	Changes to SOPs 1, 5, 11, and 13	1.05
1.05	4/29/16	H. Thomas	Updated MVT	The narrative, along with SOPs 1, 3, 5, 7, 8, 10, 11, and 13-17, were updated in preparation for publication.	1.06

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://www.sfnps.org/download_product/1163/0.

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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

Effective Date	Narrative	1-Field Safety	2-Staff Training, Colorado Units	3-Staff Training, Utah Units	4-Site Selection	5-Equipment Calibration	6-Cumulative Bias Control	7-Quality Assurance Project Plan	8-Field Methods	9-Sample Processing, Colorado Units	10-Sample Processing, Utah Units	11-Fecal Indicator Bacteria	12-Lab Analysis Methods, Colorado Units	13-Lab Analysis Methods, Utah Units	14-Data Management	15-Data Analysis & Reporting	16-Protocol Revision	17-Annual Project Review
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	1.00	1.00	1.00	1.00	1.00	1.00	1.00
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	1.00	1.00	1.00	1.00	1.00	1.01	1.00
8/12/2008	1.00	1.00	1.00	1.00	1.00	1.01	1.00	1.00	1.01	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.02	1.00
5/27/2009	1.01	1.01	1.00	1.00	1.00	1.01	1.00	1.00	1.01	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.03	1.00
8/27/2009	1.01	1.02	1.00	1.00	1.00	1.01	1.00	1.00	1.01	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.04	1.00
3/16/2010	1.01	1.03	1.00	1.00	1.00	1.02	1.00	1.00	1.01	1.00	1.00	1.01	1.00	1.01	1.00	1.00	1.05	1.00
4/29/2016	1.03	2.01	1.00	1.01	1.00	1.04	1.00	1.01	1.03	1.00	1.02	1.03	1.00	1.02	2.00	1.01	1.06	1.01

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 1.01 (3/24/16)

Revision History Log

Previous version #	Revision date	Author	Change(s) made	Reason for change	New version #
1.0	3/2016	R. Weissinger	Removed old database reference	Only use NPSTORET	1.01

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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 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.

3.2. 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.

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

3.3. 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.

3.4. 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.

3.5. 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.

3.6. Database updates

Evaluate the NPStoret database 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.

3.7. 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)

3.8. 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 summary reports (SOP 15).