

Southeast Coast Network

National Park Service
Inventory and Monitoring Division



Standard Operating Procedure 1.2.07

Estuarine Water and Sediment Laboratory Analysis Method Performance Criteria and Requirements—Version 2.0

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Summary

The following standard operating procedure (SOP) details the requisite laboratory analysis method performance criteria and requirements for implementation of the Southeast Coast Network (SECN) fixed-station and park-wide assessment of water and sediment quality. This SOP will help ensure that data are comparable over time and across parks. The techniques and procedures outlined in this SOP are based on the EPA National Coastal Condition Assessment Laboratory Methods Manual (2009) and the EPA National Coastal Condition Assessment 2015: Laboratory Operations Manual (2016); much of the information herein is modified only slightly from EPA guidance.

Revision Log

Revision Date	Author	Changes Made	Reason for Change	New Version #
1/17/2013		Citation updated	Protocol publication	1.0
4/12/2021	E. N. Starkey	New SOP title is consistent with estuarine water-quality naming convention. Re-organized and added content to clearly show lab requirements for analysis of both water and sediment samples. Added section for grain size. No changes made to MDLs. Updated methods list.	Add clarity and make content consistent with EPA methods and SECN scopes of work. Data deliverable format changed due to new database. IMD issued mandatory use of NELAP accredited labs since version 1.0.	2.0

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Overview

Estuarine water- and sediment-quality monitoring consists of components that are intended to capture both temporal and spatial variability in these systems. Monitoring at permanent fixed stations using continuous data loggers and discrete samples allow for evaluation of temporal patterns in core water-quality parameters (dissolved oxygen, pH, salinity, temperature, and turbidity) and nutrients (nitrogen, phosphorus, and chlorophyll *a*). While fixed station monitoring provides fine resolution data to determine diel, monthly, and seasonal fluctuations in water-quality, it lacks spatial extent. To fill this gap, spatial variability of water and sediment quality is determined with park-wide assessments every five years. These assessments include discrete sampling of nutrients (nitrogen, phosphorus, and chlorophyll *a*), water-quality parameters, and evaluation of sediment contaminants.

Nutrient and sediment sampling for fixed-station and park-wide monitoring components follow methods developed by the Environmental Protection Agency – National Coastal Assessment Program (EPA 2014). This monitoring approach leads to an understanding of the spatiotemporal status and variability of estuarine water-quality. In addition, the use of consistent laboratory analysis methods and performance criteria yield data comparable to that collected by the National Coastal Assessment Program.

The following sections describe the requisite laboratory analysis method performance criteria and requirements for implementation of the fixed-station and park-wide assessment of water and sediment quality. The techniques and procedures outlined below are based on the EPA National Coastal Condition Assessment Laboratory Methods Manual (2009) and the EPA National Coastal Condition Assessment 2015: Laboratory Operations Manual (2016).

Laboratory Accreditation Requirements

All laboratory analyses for fixed-station and park-wide assessments must be performed by an accredited laboratory. The mandatory use of accredited labs applies in all cases where networks use Inventory and Monitoring Division (IMD) or Water Resource Division (WRD) funds to conduct analyses through a contract, cooperative agreement, purchase card, or other for-payment type of arrangement.

Accreditation options in order of decreasing preference:

- National Environmental Laboratory Accreditation Program (NELAP)-accredited laboratories. Labs that have passed round-robin or inter-laboratory comparison proficiency tests are preferred.
- Laboratories that have been approved for use by one or more other federal agencies based on performance on National Environmental Laboratory Accreditation Conference (NELAC) proficiency tests, on-site inspections, and an evaluation of the laboratories' QA/QC programs.

- Laboratories that are on the national NELAP-approved lab lists for specific regulatory programs of interest.

Fixed-Station and Park-wide Assessment: Water Samples

Nutrients and Chlorophyll Method Performance Criteria

Suggested analytical methods for water-quality indicators are described in section 2.2–2.7 of the EPA National Coastal Condition Assessment Laboratory Methods Manual (2009). Laboratories contracted by the Southeast Coast Network may use a different analytical method if the lab is able to achieve the same performance requirements as the standard methods. Performance data must be submitted to the Southeast Coast Network prior to initiating any analyses. Methods performance requirements for this program identify detection limit, precision and accuracy objectives for each indicator. Method performance requirements for water chemistry and chlorophyll *a* sample analyses are shown in Table 1.

Table 1. Laboratory method performance requirements and analysis methods to be used for SECN water chemistry and chlorophyll *a* sample analysis (from EPA 2009).

Analyte	Units	MDL Objective	Transition Value	Precision Objective	Accuracy Objective	Potential Methods conforming to NELAC guidance
Ammonia (NH ₃)	mg N/L	0.01 (0.7 µeq/L)	0.10	± 0.01 or ±10%	± 0.01 or ±10%	- EPA Method 350.2 - Standard Method 4500-NH3 G-1997
Nitrate-Nitrite (NO ₃ -NO ₂)	mg N/L	0.01	0.10	± 0.01 or ±10%	± 0.01 or ±10%	- EPA Method 353.2 - Enzyme Catalyzed Reduction method ASTM D-7781
Total Nitrogen (TN)	mg/L	0.01	0.10	± 0.01 or ±10%	± 0.01 or ±10%	- EPA Method 353.2 - Standard Methods 4500-N C and 4500-NO3 F
Total Phosphorous (TP) and ortho-P	µg P/L	2.0	20.0	± 2 or ±10%	± 2 or ±10%	- EPA Method 365.1 - Standard Methods 4500-P.B.5, and 4500 P.E
Nitrate (NO ₃)	mg N/L	0.01 (10.1 µeq/L)	0.1	± 0.01 or ±5%	± 0.01 or ±5%	- EPA Method 353.2 - Enzyme Catalyzed Reduction method ASTM D-7781 - Calculation [NO ₃]-[NO ₂]
Chlorophyll- <i>a</i>	µg/L in extract	1.5	15	± 1.5 or ±10%	± 1.5 or ±10%	- EPA Method 445.0 - Standard Method 10200H.3

Additional Laboratory Requirements

In addition to water sample analysis services that meet the guidelines above, contracted labs will provide the following sample handling services:

- Provide insulated sample shipping containers, sampling containers, and provide return mailing of containers if needed to remote park locations using NPS-provided shipping accounts.
- Collect sample arrival information or other standard chain of custody information that also notes condition of samples such as arrival time and inside temperature.
- Have in place laboratory QA/QC checks that including running laboratory blanks, check samples and duplicate samples. This information will be provided with sample results by batch. Other QA/QC information including documentation of instrument calibration slopes, limits of detection, statement of instrument comparability, and standard reference material used will be available upon request or posted at designated internet sites.
- Provide confirmation of sample receipt via e-mail to NPS-SECN project manager.
- Sample turnaround within 45 days.

Laboratory Deliverables

Water sample related deliverables provided by contracted labs include:

- Digital data files in Microsoft Excel format that include sample ID, date collected, date analyzed, sample results, and method detection limits. The Excel file format (column order, required fields, etc.) will follow the most recent template provided by the Southeast Coast Network. Examples are provided in Appendix A.
- Digital QA/QC results that may be delivered in separate files or integrated with sample results data files.
- Sample collection containers for the collection of filtered and unfiltered nutrient samples.
- Copies of any relevant sample processing notes upon request.

Park-wide Assessment—Sediment Samples

Sediment Contaminants Method Performance Criteria

Suggested analytical methods for sediment contaminants are described in section 4.0 of the EPA National Coastal Condition Assessment Laboratory Methods Manual (2009). However, some laboratories might employ other analytical methods. Laboratories contracted by the network may use a different analytical method if the lab is able to achieve the same performance requirements as the standard methods. Performance data must be submitted to the network prior to initiating any analyses. Methods performance requirements for this program identify detection limit, precision, and

accuracy objectives for each indicator. Method performance requirements for inorganic and organic contaminants in sediment are shown in Tables 2 and 3.

Table 2. Laboratory method performance requirements for inorganic contaminants in sediment (modified from EPA 2009).

Inorganic Analytes	MDL Objective (dry weight, µg/g (ppm))	Maximum Allowable Accuracy¹	Maximum Allowable Precision²	Completeness Objective³
Aluminum	1,500.00	20%	30%	95%
Antimony	0.20	20%	30%	95%
Arsenic	1.50	20%	30%	95%
Cadmium	0.05	20%	30%	95%
Chromium	5.00	20%	30%	95%
Copper	5.00	20%	30%	95%
Iron	500.00	20%	30%	95%
Lead	1.00	20%	30%	95%
Manganese	1.00	20%	30%	95%
Mercury	0.01	20%	30%	95%
Nickel	1.00	20%	30%	95%
Selenium	0.10	20%	30%	95%
Tin	0.10	20%	30%	95%
Zinc	2.00	20%	30%	95%

¹ Accuracy (bias) goals are expressed either as absolute difference ($\pm$ value) or percent deviation from “true” value.

² Precision goals are expressed as relative percent difference (RPD) or relative standard deviation (RSD) between two or more replicate measurements.

³ Completeness goal is the percentage of expected results that are obtained successfully.

Table 3. Laboratory method performance requirements for organic contaminants in sediment (modified from EPA 2009).

OrganicAnalytes	MDL Objective (dry weight,ng/g (ppb))	Maximum Allowable Accuracy ¹	Maximum Allowable Precision ²	Completeness Objective ³
PAHs	10.0	35%	30%	95%
PCB congeners	1.0	35%	30%	95%
Chlorinated pesticides/DDTs	1.0	35%	30%	95%
TOC	100.0	35%	30%	95%

¹ Accuracy (bias) goals are expressed either as absolute difference ($\pm$ value) or percent deviation from “true” value.

² Precision goals are expressed as relative percent difference (RPD) or relative standard deviation (RSD) between two or more replicate measurements.

³ Completeness goal is the percentage of expected results that are obtained successfully.

Sediment Grain (Particle) Size Method Performance Criteria

Suggested analytical methods for sediment grain size are described in section 5.1 of the EPA National Coastal Condition Assessment Laboratory Methods Manual (2009). However, some laboratories might employ other analytical methods. Laboratories contracted by the Southeast Coast Network may use a different analytical method as long as the lab is able to achieve the same performance requirements as the standard methods. Performance data must be submitted to the Southeast Coast Network prior to initiating any analyses. Methods performance requirements for this program identify detection limit, precision and accuracy objectives for each indicator. Method performance requirements for sediment grain size are shown in Table 4.

Table 4. Laboratory method performance requirements for sediment grain size.

Indicator/Data Type	Maximum Allowable Accuracy (Bias) Goal	Maximum Allowable Precision Goal	CompletenessGoal
Particle Size	NA	10%	95%

Additional Laboratory Requirements

In addition to sediment sample analysis services that meet the guidelines above, contracted labs will provide the following sample handling services:

- Provide pre-labeled sample containers for each sediment sample: 300-milliliter (10.1 ounces) glass jar for Organics/Metals (PAH/Pesticides/PCB/Metals), 300-milliliter glass jar for Grain size (Particle Size), 125-milliliter (4.2 ounces) glass jar for Total Organic Carbon (TOC).
- Provide pre-sorted “sample kits” for each site. Each sample kit consists of a resealable plastic bag with three pre-labeled glass sample jars (listed above) wrapped in bubble wrap.
- Provide laboratory-specific sample tracking forms.
- Provide insulated sample shipping containers (i.e., coolers) and provide return mailing of containers if needed to remote park locations using NPS provided shipping accounts.
- Collect sample arrival information or other standard chain of custody information that also notes condition of samples such as arrival time and inside temperature.
- Provide confirmation of sample receipt via email to SECN project lead.
- Have in place laboratory QA/QC checks that including running laboratory blanks, check samples and duplicate samples. This information will be provided with sample results by batch.
- QA/QC information including documentation of instrument calibration slopes, limits of detection, statement of instrument comparability, and standard reference material used will be provided with the data.
- Sample turnaround within 45 days.

Laboratory Deliverables

Sediment sample deliverables provided by contracted labs include:

- Pre-labeled jars and sample kits described above.
- Digital data files in Microsoft Excel format that include sample ID, date collected, date analyzed, sample results, and method detection limits. The Microsoft Excel file format (column order, required fields, etc.) will follow the most recent template provided by the Southeast Coast Network. Examples are provided in Appendix A.
- Digital QA/QC results in Microsoft Excel format. May be delivered in separate files or integrated with sample results data files.

Copies of any relevant sample processing notes.

Literature Cited

- US Environmental Protection Agency (EPA). 2009. National Coastal Condition Assessment: Laboratory Methods Manual. EPA 841-R-09-002. U.S. Environmental Protection Agency, Office of Water and Office of Research and Development, Washington, DC.
- US Environmental Protection Agency (EPA). 2014. National Coastal Condition Assessment: Field Operations Manual. EPA-841-R-14-007. U.S. Environmental Protection Agency, Washington, DC. 165 p.
- US Environmental Protection Agency (EPA). 2016. National Coastal Condition Assessment 2015: Laboratory Operations Manual. EPA 841-R-14-008. U.S. Environmental Protection Agency, Office of Water and Office of Research and Development, Washington, DC.

Appendix A. Data Deliverable Templates

Note: (1) All metadata will be placed before the header row (see below). The ‘scraping’ of data occurs on a row where ‘SampleID’ occurs in the first column. (2) All column headers (including optional columns) are included in lab data reports. Values within these columns may be optional.

Table A-1. Nutrient discrete fixed-station template columns 1:10.

Sample ID	Sample Date	Received Date	NASL ID	Matrix	Prep Date	Analysis Date	Parameter	<MDL/RL	Result
The fixed-station name	The date that the water sample event occurred on	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	Date	A standards NPS parameter value. (See WQX data dictionary for more information)	(OPTIONAL)	Decimal (10,4)

Table A-2. Nutrient discrete fixed-station template columns 11:15.

Unit	MDL/RL	Method	Analyst	Comments
A standard NPS unit name. (See WQX data dictionary for more information)	Two values separated by a '/' with no space. (Decimal (7,4)/Decimal(7,4))	A standards NPS method name. (See WQX data dictionary for more information)	(OPTIONAL)	(OPTIONAL) with a maximum of 250 characters.

Table A-3. Nutrient synoptic (i.e., park-wide assessment) template columns 1:7.

Sample ID	Sample Date	Received Date	NASL ID	Matrix	Prep Date	Analysis Date
The park-wide-assessment name	The date that the water sample event occurred on	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	Date

Table A-4. Nutrient synoptic (i.e., park-wide assessment) template columns 8:15.

Parameter	<MDL/RL	Result	Unit	MDL/RL	Method	Analyst	Comments
A standard NPS parameter name. (See WQX data dictionary for more information)	(OPTIONAL)	Decimal (10,4)	A standard NPS unit name. (See WQX data dictionary for more information)	Two values separated by a ' ' with no space. (Decimal (7,4)/Decimal(7,4))	A standards NPS method value. (See WQX data dictionary for more information)	(OPTIONAL)	(OPTIONAL) with a maximum of 250 characters.

Table A-5. Synoptic (i.e., park-wide assessment) sediment template columns 1:8.

AEL Project Number	AEL Sample Number	Client Sample ID	Site	Sample_Type	Matrix	Tests_Description	Method
(OPTIONAL)	(OPTIONAL)	Park-wide Assessment name.	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	A standard NPS method name. See WQX dictionary for more information.

Table A-6. Synoptic (i.e., park-wide assessment) sediment template columns 9:16.

CAS	Parameter	Dilution	Adjusted Detection Limit	Adjusted Reporting Limit	Final Result	Units	Qualifier_Only
A standards NPS CAS value. (See WQX dictionary for more information)	A standards NPS parameter value. (See WQX data dictionary for more information)	(OPTIONAL)	Decimal with maximum precision of 4, and total value size of 7	DECIMAL (7,4)	DECIMAL (10,4)	A standard NPS unit value. (See WQX data dictionary for more information)	(OPTIONAL)

Table A-7. Synoptic (i.e., park-wide assessment) sediment template columns 17:24.

Qualifiers	Spike_Recovery	Spike_LCL	Spike_UCL	Analyst	Date of Analysis	Analysis Time	Prep Method
(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	(OPTIONAL)	Date	(OPTIONAL)	(OPTIONAL)

Table A-8. Synoptic (i.e., park-wide assessment) sediment template columns 25:31.

Prep Date	Received Date	Collection Date	Collect Time	Lab ID	% Moisture	Result_Type
(OPTIONAL)	(OPTIONAL)	Date	(OPTIONAL)	A standardized laboratory name. This value should be standardized across all of a particular laboratory's reports.	(OPTIONAL)	(OPTIONAL)