Method 440.0

Determination of Carbon and Nitrogen in Sediments and Particulates of Estuarine/Coastal Waters Using Elemental Analysis

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1.0 Scope and Application

1.1 Elemental analysis is used to determine particulate carbon (PC) and particulate nitrogen (PN) in estuarine and coastal waters and sediment. The method measures the total carbon and nitrogen irrespective of source (inorganic or organic).

Analyte	Chemical Abstracts Service Registry Numbers (CASRN)
Carbon	7440-44-0
Nitrogen	1333-74-0

1.2 The need to qualitatively or quantitatively determine the particulate organic fraction from the total particulate carbon and nitrogen depends on the data-quality objectives of the study. Section 11.4 outlines procedures to ascertain the organic/inorganic particulate ratio. The method performance presented in the method was obtained on particulate samples with greater than 80% organic content. Performance on samples with a greater proportion of particulate inorganic versus organic carbon and nitrogen has not been investigated.

1.3 Method detection limits (MDLs)[1] of 10.5 µg/L and 62.3 µg/L for PN and PC, respectively, were obtained for a 200-mL sample volume. Sediment MDLs of PN and PC are 84 mg/kg and 1300 mg/kg, respectively, for a sediment sample weight of 10.00 mg. The method has been determined to be linear to 4800 µg of C and 700 µg of N in a sample. Multilaboratory study validation data are in Section 13.

1.4 This method should be used by analysts experienced in the theory and application of elemental analysis. A minimum of 6 months experience with an elemental analyzer is recommended.

1.5 Users of the method data should set the data-quality objectives prior to analysis. Users of the method must document and have on file the required initial demonstration of performance data described in Section 9.2 prior to using the method for analysis.

2.0 Summary of Method

2.1 An accurately measured amount of particulate matter from an estuarine water sample or an accurately weighed dried sediment sample is combusted at 975°C using an elemental analyzer. The combustion products are passed over a copper reduction tube to convert the oxides of N into molecular N. Carbon dioxide, water vapor and N are homogeneously mixed at a known volume, temperature and pressure. The mixture is released to a series of thermal conductivity detectors/traps, measuring in turn by difference, hydrogen (as water vapor), C (as carbon dioxide) and N (as N_2). Inorganic and organic C may be determined by two methods which are also presented.

3.0 Definitions

3.1 Sediment Sample -- A fluvial, sand, or humic sample matrix exposed to a marine, brackish or fresh water environment. It is limited to that portion which may be passed through a number 10 sieve or a 2-mm mesh sieve.

3.2 Material Safety Data Sheet (MSDS) -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.3 Instrument Detection Limit (IDL) -- The minimum quantity of analyte or the concentration equivalent which gives an analyte signal equal to three times the standard deviation of the background signal at the selected wavelength, mass, retention time, absorbance line, etc.

3.4 Method Detection Limit (MDL) -- The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

3.5 Linear Dynamic Range (LDR) -- The absolute quantity over which the instrument response to an analyte is linear.

3.6 Calibration Standard (CAL) -- An accurately weighed amount of a certified chemical used to calibrate the instrument response with respect to analyte mass.

3.7 Conditioner -- A standard chemical which is not necessarily accurately weighed that is used to coat the surfaces of the instrument with the analytes (water vapor, carbon dioxide, and nitrogen).

3.8 External Standards (ES) -- A pure analyte(s) that is measured in an experiment separate from the experiment used to measure the analyte(s) in the sample. The signal observed for a known quantity of the pure external standard(s) is used to calibrate the instrument

response for the corresponding analyte(s). The instrument response is used to calculate the concentrations of the analyte(s) in the sample.

3.9 Response Factor (RF) -- The ratio of the response of the instrument to a known amount of analyte.

3.10 Laboratory Reagent Blank (LRB) -- A blank matrix (i.e., a precombusted filter or sediment capsule) that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.11 Field Reagent Blank (FRB) -- An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

3.12 Laboratory Duplicates (LD1 and LD2) -- Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.13 Field Duplicates (FD1 and FD2) -- Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.14 Laboratory Fortified Blank (LFB) -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the method is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.15 Laboratory Fortified Sample Matrix (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.16 Standard Reference Material (SRM) -- Material which has been certified for specific analytes by a variety of analytical techniques and/or by numerous laboratories using similar analytical techniques. These may consist of pure chemicals, buffers or compositional standards. These materials are used as an indication of the accuracy of a specific analytical technique.

3.17 Quality Control Sample (QCS) -- A solution of method analytes of known concentrations which is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

4.0 Interferences

4.1 There are no known interferences for estuarine/coastal water or sediment samples. The presence of C and N compounds on laboratory surfaces, on fingers, in detergents and in dust necessitates the utilization of careful techniques (i.e., the use of forceps and gloves) to avoid contamination in every portion of this procedure.

5.0 Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.[2-5] A reference file of material safety data sheets (MSDS) should also be made available to all personnel involved in the chemical analysis.

5.2 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.

5.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.

5.4 Although most instruments are adequately shielded, it should be remembered that the oven temperatures are extremely high and that care should be taken when working near the instrument to prevent possible burns.

5.5 It is the responsibility of the user of this method to comply with relevant disposal and waste regulations. For guidance see Sections 14.0 and 15.0.

6.0 Apparatus and Equipment

6.1 Elemental Analyzer

6.1.1 An elemental analyzer capable of maintaining a combustion temperature of 975°C and analyzing particulate samples and sediment samples for elemental C and N. The Leeman Labs Model 240 XA Elemental Analyzer was used to produce the data presented in this method.

6.2 A gravity convection drying oven. Capable of maintaining 103-105°C for extended periods of time.

6.3 Muffle furnace. Capable of maintaining 875°C ± 15°C.

6.4 Ultra-micro balance. Capable of accurately weighing to 0.1 µg. Desiccant should be kept in the weighing chamber to prevent hygroscopic effects.

6.5 Vacuum pump or source capable of maintaining up to 10 in. Hg of vacuum.

6.6 Mortar and pestle.

6.7 Desiccator, glass.

6.8 Freezer, capable of maintaining -20°C ± 5°C.

6.9 47-mm or 25-mm vacuum filter apparatus made up of a glass filter tower, fritted glass disk base and 2-L vacuum flask.

6.10 13-mm Swinlok filter holder.

6.11 Teflon-tipped, flat blade forceps.

6.12 Labware -- All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) should be sufficiently clean for the task objectives. Several procedures found to provide clean labware include washing with a detergent solution, rinsing with tap water, soaking for 4 hr or more in 20% (v/v) HCI, rinsing with reagent water and storing clean. All traces of organic material must be removed to prevent C-N contamination.

6.12.1 Glassware -- Volumetric flasks, graduated cylinders, vials and beakers.

6.12.2 Vacuum filter flasks -- 250 mL and 2 L, glass.

6.12.3 Funnel, 6.4 mm i.d., polyethylene.

6.12.4 Syringes, 60-mL, glass.

7.0 Reagents and Standards

7.1 Reagents may contain elemental impurities which affect analytical data. High-purity reagents that conform to the American Chemical Society specifications[6] should be used whenever possible. If the purity of a reagent is in question, analyze for contamination. The acid used for this method must be of reagent grade purity or equivalent. A suitable acid is available from a number of manufacturers.

7.2 Hydrochloric acid, concentrated (sp. gr. 1.19)-HCI.

7.3 Acetanilide, 99.9% + purity, $C_8H_{19}NO$ (CASRN 103-84-4).

7.4 Blanks -- Three blanks are used for the analysis. Two blanks are instrument related. The instrument zero response (ZN) is the background response of the instrument without sample holding devices such as capsules and sleeves. The instrument blank response (BN) is the response of the instrument when the sample capsule, sleeve and ladle are inserted for analysis without standard or sample. The BN is also the laboratory reagent blank (LRB) for sediment samples. The LRB for water samples includes the capsule, sleeve, ladle and a precombusted filter without standard or sample. These blanks are subtracted from the uncorrected instrument response used to calculate concentration in Sections 12.3 and 12.4.

7.4.1 Laboratory fortified blank (LFB) -- The third blank is the laboratory fortified blank. For sediment analysis, add a weighed amount of acetanilide in an aluminum capsule and analyze for PC and PN (Section 9.3.2). For aqueous samples, place a weighed amount of acetanilide on a glass fiber filter the same size as used for the sample filtration. Analyze the fortified filter for PC and PN (Section 9.3.2)

7.5 Quality Control Sample (QCS) -- For this method, the QCS can be any assayed and certified sediment or particulate sample which is obtained from an external source. The Canadian Reference Material, BCSS-1, is just such a material and was used in this capacity for the data presented in this method. The percent PC has been certified in this material but percent PN has not.

8.0 Sample Collection, Preservation and Storage

8.1 Water Sample Collection -- Samples collected for PC and PN analyses from estuarine/coastal waters are normally collected from a ship using one of two methods; hydrocast or submersible pump systems. Follow the recommended sampling protocols associated with the method used. Whenever possible, immediately filter the samples as described in Section 11.1.1. Store the filtered sample pads by freezing at -20°C or storing in a desiccator after drying at 103-105° C for 24 hr. No significant difference has been noted in comparing the two storage procedures for a time period of up to 100 days. If storage of the water sample is necessary, place

the sample into a clean amber bottle and store at 4°C until filtration is done.

8.1.1 The volume of water sample collected will vary with the type of sample being analyzed. Table 1 provides a guide for a number of matrices of interest. If the matrix cannot be classified by this guide, collect 2 x 1L of water from each site. A minimum filtration volume of 200 mL is recommended.

8.2 Sediment Sample Collection -- Estuarine/coastal sediment samples are collected with benthic samplers. The type of sampler used will depend on the type of sample needed by the data-quality objectives.[7] Store the wet sediment in a clean jar and freeze at -20°C until ready for analysis.

8.2.1 The amount of sediment collected will depend on the sample matrix and the elemental analyzer used. A minimum of 10 g is recommended.

9.0 Quality Control

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the continued analysis of laboratory reagent blanks, laboratory duplicates, field duplicates and calibration standards analyzed as samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data thus generated.

9.2 Initial Demonstration of Performance (Mandatory)

9.2.1 The initial demonstration of performance is used to characterize instrument performance (MDLs, linear dynamic range) and laboratory performance (analysis of QC samples) prior to the analyses conducted by this method.

9.2.2 Linear dynamic range (LDR) -- The upper limit of the LDR must be established by determining the signal responses from a minimum of three different concentration standards across the range, one of which is close to the upper limit of the LDR. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. The upper LDR limit should be an observed signal no more than 10% below the level extrapolated from the lower standards. Determined sample analyte concentrations that are 90% and above the upper LDR must be reduced in mass and reanalyzed. New LDRs should be determined whenever there is a significant change in instrument response and for those analytes that periodically approach the upper LDR limit, every 6 months or whenever there is a change in instrument analytical hardware or operating conditions.

9.2.3 Quality control sample (QCS) (Section 7.5) -- When beginning the use of this method, on a quarterly basis or as required to meet data quality needs, verify the calibration standards and acceptable instrument performance with the analyses of a QCS. If the determined concentrations are not within ± 5% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with analyses.

9.2.4 Method detection limits (MDLs) -- MDLs should be established for PC and PN using a low level estuarine water sample, typically three to five times higher than the estimated MDL. The same procedure should be followed for sediments. To determine MDL values, analyze seven replicate aliquots of water or sediment and process through the entire analytical procedure (Section 11). These replicates should be randomly distributed throughout a group of typical analyses. Perform all calculations defined in the method (Section 12) and report the concentration values in the appropriate units. Calculate the MDL as follows:[1]

$$MDL = (t) \times (S)$$

where, S = Standard deviation of the replicate analyses.

t = Student's t value for n-1 degrees of freedom at the 99% confidence limit; t = 3.143 for six degrees of freedom.

MDLs should be determined whenever a significant change in instrumental response, change of operator, or a new matrix is encountered.

9.3 Assessing Laboratory Performance (Mandatory)

9.3.1 Laboratory reagent blank (LRB) -- The laboratory must analyze at least one LRB (Section 3.10) with each batch of 20 or fewer samples of the same matrix. LRB data are used to assess contamination from the laboratory environment. LRB values that exceed the MDL indicate laboratory or reagent contamination. When LRB values constitute 10% or more of the analyte level determined for a sample, fresh samples or field duplicates of the samples must be prepared and analyzed again after the source of contamination has been corrected and acceptable LRB values have been obtained. For aqueous samples the LRB is a precombusted filter of the same type and size used for samples.

9.3.2 Laboratory fortified blank (LFB) -- The laboratory must analyze at least one LFB (Section 7.4.1) with each batch of samples. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required control limits of 85-115%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

9.3.3 The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 85-115% (Section 9.3.2). When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

Upper Control Limit = x + 3S

Lower Control Limit = x - 3S

The optional control limits must be equal to or better than the required control limits of 85-115%. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also the standard deviation (S) data should be used to establish an ongoing precision statement for the level of concentrations included in the LFB. These data must be kept on file and be available for review.

9.4 Assessing Analyte Recovery and Data Quality

9.4.1 Percent recoveries cannot be readily obtained from particulate samples. Consequently, accuracy can only be assessed by analyzing check standards as samples and quality control samples (QCS). The use of laboratory fortified matrix samples has not been assessed.

10.0 Calibration and Standardization

10.1 Calibration -- After following manufacturer's installation and temperature stabilization procedures, daily calibration procedures must be performed and evaluated before sample analysis may begin. Single point or standard curve calibrations are possible, depending on instrumentation.

10.1.1 Establish single response factors (RF) for each element (C,H, and N) by analyzing three weighed portions of calibration standard (acetanilide). The mass of calibration standard should provide a response within 20% of the response expected for the samples being analyzed. Calculate the (RF) for each element using the following formula:

$$\text{Response factor } (\mu v/\mu g) = \frac{RN-ZN-BN}{WTN}$$

where,
- RN = Average instrument response to standard (µv)
- ZN = Instrument zero response (µv)
- BN = Instrument blank response (µv)

and, $WTN = (M)(N_a)(AW/MW)$

where,
- M = The mass of standard material in µg
- N_a = Number of atoms of C, N or H, in a molecule of standard material
- AW = Atomic weight of C (12.01), N (14.01) or H (1.01)
- MW = Molecular weight of standard material (135.2 for acetanilide)

If instrument response is in units other than µv, then change the formula accordingly.

10.1.2 For standard curve preparation, the range of calibration standard masses used should be such that the low concentration approaches but is above the MDL and the high concentration is above the level of the highest sample, but no more than 90% of the linear dynamic range. A minimum of three concentrations should be used in constructing the curve. Measure response versus mass of element in the standard and perform a regression on the data to obtain the calibration curve.

11.0 Procedure

11.1 Aqueous Sample Preparation

11.1.1 Water Sample Filtration -- Precombust GF/F glass fiber filters at 500°C for 1.5 hr. The diameter of filter used will depend on the sample composition and instrument capabilities (Section 8.1.1). Store filters covered if not immediately used. Place a precombusted filter on fritted filter base of the filtration apparatus and attach the filtration tower. Thoroughly shake the sample container to suspend the particulate matter. Measure and record the required sample volume using a graduated cylinder. Pour the measured sample into the filtration tower, no more than 50 mL at a time. Filter the sample using a vacuum no greater than 10 in. of Hg. Vacuum levels greater than 10 in. of Hg can cause filter rupture. If less than the measured volume of sample can be practically filtered due to clogging, measure and record the actual volume filtered. **Do not** rinse the filter following filtration. It has been demonstrated that sample loss occurs when the filter is rinsed with an isotonic solution or the filtrate.[8] Air dry the filter after the sample has passed through by continuing the vacuum for 30 sec. Using Teflon-coated flat-tipped forceps, fold the filters in half while still on the fritted glass base of the filter apparatus. Store filters as described in Section 8.

11.1.2 If the sample has been stored frozen, place the sample in a drying oven at 103-105° C for 24 hr before analysis and dry to a constant weight. Precombust one nickel sleeve at 875° C for 1 hr for each sample.

11.1.3 Remove the filter pads containing the particulate material from the drying oven and insert into a precombusted nickel sleeve using Teflon-coated flat-tipped forceps. Tap the filter pad using a stainless steel rod. The sample is ready for analysis.

11.2 Sediment Samples Preparation

11.2.1 Thaw the frozen sediment sample in a 102-105°C drying oven for at least 24 hr before analysis and dry to a constant weight. After drying, homogenize the dry sediment with a mortar and pestle. Store in a desiccator until analysis. Precombust aluminum capsules at 550°C in a muffle furnace for 1.5 hr for each sediment sample being analyzed. Precombust one nickel sleeve at 875°C for 1 hr for each sediment sample.

11.2.2 Weigh 10 mg of the homogenized sediment to the nearest 0.001 mg with an ultra-micro balance into a precombusted aluminum capsule. Crimp the top of the aluminum capsule with the Teflon-coated flat-tipped forceps and place into a precombusted nickel sleeve. The sample is ready for analysis.

11.3 Sample Analysis

11.3.1 Measure instrument zero response (Section 7.4) and instrument blank response (Section 7.4) and record values. Condition the instrument by analyzing a conditioner. Calibrate the instrument according to Section 10 and analyze all preliminary QC samples as required by Section 9. When satisfactory control has been established, analyze samples according to the instrument manufacturer's recommendations. Record all response data.

11.3.2 Report data as directed in Section 12.

11.4 Determination of Particulate Organic and Inorganic Carbon

11.4.1 Method 1: Thermal Partitioning -- The difference found between replicate samples, one of which has been analyzed for total PC and PN and the other which was muffled at 550°C and analyzed is the particulate organic component of that sample. This method of thermally partitioning organic and inorganic PC may underestimate slightly the carbonate minerals' contribution in the inorganic fraction since some carbonate minerals decompose below 500°C, although $CaCO_3$ does not.[9]

11.4.2 Method 2: Fuming HCI -- Allow samples to dry overnight at 103-105°C and then place in a desiccator containing concentrated HCI, cover and fume for 24 hr in a hood. The fuming HCI converts inorganic carbonate in the samples to water vapor, CO_2 and calcium chloride. Analyze the samples for particulate C. The resultant data are particulate organic carbon.[10]

12.0 Data Analysis and Calculations

12.1 Sample data should be reported in units of µg/L for aqueous samples and mg/kg dry weight for sediment samples.

12.2 Report analyte concentrations up to three significant figures for both aqueous and sediment samples.

12.3 For aqueous samples, calculate the sample concentration using the following formula:

$$\text{Concentration (µg/L)} = \frac{\text{Corrected sample response (µv)}}{\text{Sample volume (L) x RF (µv/µg)}}$$

where, RF = Response Factor (Section 10.1.1)
Corrected Sample Response (Section 7.4)

12.4 For sediment samples, calculate the sample concentration using the following formula:

$$\text{Concentration (mg/kg)} = \frac{\text{Corrected sample response (µv)}}{\text{Sample weight (g) x RF (µv/µg)}}$$

where, RF = Response Factor (Section 10.1.1)
Corrected Sample Response (Section 7.4)

Note: Units of µg/g = mg/kg

12.5 The QC data obtained during the analyses provide an indication of the quality of the sample data and should be provided with the sample results.

13.0 Method Performance

13.1 Single Laboratory Performance

13.1.1 Single laboratory performance data for aqueous samples from the Chesapeake Bay are provided in Table 2.

13.1.2 Single-laboratory precision and accuracy data for the marine sediment reference material, BCSS-1, are listed in Table 3.

13.2 Multilaboratory Performance

13.2.1 In a multilab study, 13 participants analyzed sediment and filtered estuarine water samples for particulate carbon and nitrogen. The data were analyzed

using the statistical procedures recommended in ASTM D2777-86 for replicate designs. See Table 4 for summary statistics.

13.2.2 Accuracy as mean recovery was estimated from the analyses of the NRC of Canada Marine Sediment Reference Material, BCSS-1. Mean recovery was 98.2% of the certified reference carbon value and 100% of the noncertified nitrogen value.

13.2.3 Overall precision for analyses of carbon and nitrogen in sediments was 1-11% RSD, while the analyses of both particulate carbon and nitrogen in estuarine water samples was 9-14% RSD.

13.2.4 Single analyst precision for carbon and nitrogen in sediment samples was 1-8% RSD and 4-9% for water samples.

13.2.5 Pooled method detection limits (p-MDLs) were calculated using the pooled single analyst standard deviations. The p-MDLs for particulate nitrogen and carbon in estuarine waters were 0.014 mg N/L and 0.064 mg C/L , respectively. The p-MDLs for percent carbon and nitrogen in estuarine sediments were not estimated because the lowest concentration sediment used in the study was still 20 times higher than the estimated MDLs. Estimates of p-MDLs from these data would be unrealistically high.

14.0 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction,* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 Waste Management

15.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult *The Waste Management Manual for Laboratory Personnel,* available from the American Chemical Society at the address listed in Section 14.2.

16.0 References

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17.0 Tables, Diagrams, Flowcharts, and Validation Data

Table 1. Filter Diameter Selection Guide

Sample matrix	Filter diameter 47mm	25mm	13mm
	Sample matrix volume		
Open ocean	2000 mL	500 mL	100 mL
Coastal	1000 mL	400-500 mL	100 mL
Estuarine (low particulate)	500-700 mL	250-400 mL	50 mL
Estuarine (high particulate)	100-400 mL	75-200 mL	25 mL

Table 2. Performance Data--Chesapeake Bay Aqueous Samples

Sample	Measured nitrogen concentration (µg/L)	S.D.[A] (µg/L)	Measured carbon concentration (µg/L)	S.D.[A] (µg/L)
1	147	± 4	1210	± 49
2	148	± 11	1240	± 179
3	379	± 51	3950	± 269
4	122	± 9	1010	± 63

[A] Standard deviation based on 7 replicates.

Table 3. Precision and Accuracy Data - Canadian Sediment Reference Material BCSS-1

Element	T.V.[A]	Mean measured value (%)	%RSD[B]	%Recovery[C]
Carbon	2.19%	2.18	± 3.3	99.5
Nitrogen	0.195%	0.194	± 3.9	99.5

[A] True value. Carbon value is certified; nitrogen value is listed but not certified

[B] Percent relative standard deviation based on 10 replicates.

[C] As calculated from T.V.

Table 4. Overall and Single Analyst Precision Estimates from Collaborative Study

Analyte	Sample	N[1]	Mean[2] Conc.	Overall Std. Dev.	Overall %RSD	Analyst Std. Dev.	Analyst %RSD
Particulate Nitrogen (as N) in Estuarine Waters	A	11	0.0655	0.0081	12.4%	0.0050	7.6%
	B	12	0.0730	0.0076	10.3%	0.0057	7.7%
	C	12	0.0849	0.0110	12.9%	0.0060	7.1%
	D	12	0.126	0.0138	11.0%	0.0071	5.6%
	E	11	0.182	0.0245	13.5%	0.0157	8.6%
Nitrogen (as %N) in Estuarine Water	1	10	0.178	0.0190	10.7%	0.0131	7.3%
	2	10	0.295	0.0114	3.9%	0.0046	1.6%
	3	10	0.436	0.0178	4.1%	0.0104	2.4%
	4	10	0.497	0.0183	3.7%	0.0082	1.6%
	5	10	0.580	0.0207	3.6%	0.0150	2.6%
Particulate Carbon (as C) in Estuarine Waters	B	12	0.369	0.0505	13.7%	0.0222	6.0%
	A	12	0.417	0.0490	11.8%	0.0230	5.5%
	D	12	0.619	0..0707	11.4%	0.0226	3.6%
	C	12	0.710	0.0633	8.9%	0.0367	5.2%
	E	12	0.896	0.1192	13.3%	0.0569	6.4%
Carbon (as %C) in Estuarine Sediments	1	13	1.78	0.1517	8.5%	0.1346	7.6%
	2	13	2.55	0.0372	1.5%	0.0204	0.8%
	3	13	3.18	0.0435	1.4%	0.0348	1.1%
	4	13	4.92	0.1201	2.4%	0.0779	1.6%
	5	13	5.92	0.0621	1.1%	0.0547	0.9%

(1) N = Number of participants whose data was used.

(2) Concentration in mg/L or percent, as indicated.