

Chapter 15. Determination of Particulate Organic Carbon and Nitrogen

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1.0 Scope and field of application

This procedure describes a method for the determination of particulate organic carbon and particulate nitrogen in seawater. The assay is appropriate for measuring oceanic levels of particulate organic carbon (5.0 - 500.0 $\mu\text{g C kg}^{-1}$) and particulate nitrogen (0.5 - 100.0 $\mu\text{g N kg}^{-1}$). The principles for this method were first described by Gordon (1969) and Kerambrun and Szekielida (1969). Sharp (1974) describes a number of useful modifications to the existing method applied here. Detailed description of the analytical procedure is given by the manufacturer (Control Equipment Corporation 1988, Exeter Analytical Inc. 1995).

2.0 Definition

2.1 The concentration of particulate organic carbon is given in $\mu\text{g C kg}^{-1}$ seawater.

2.2 The concentration of particulate nitrogen is given in $\mu\text{g N kg}^{-1}$ seawater.

3.0 Principle of Analysis

A dried, acidified sample of particulate matter is combusted at 960°C. The organic carbon is converted to CO_2 and the nitrogen oxides are subsequently reduced to N_2 gas. Both gases are measured by thermal conductivity.

4.0 Apparatus

4.1 *Control Equipment Corporation (CEC) 240-XA Elemental Analyzer* (Exeter Analytical, Inc.)

4.2 CAHN Model 4400 Electrobalance

4.3 IBM-PC Compatible Computer Analytical Software

5.0 Reagents

5.1 *Hydrochloric acid* (concentrated HCl: reagent grade)

5.2 *Acetanilide* (reagent grade): Acetanilide has 0.7109 g C and 0.1036 g N per gram total mass.

6.0 Sampling

Samples are collected at all depths between the surface and 1000 m. The POC/PN samples are taken approximately 30-60 minutes after the CTD/rosette reaches the surface. Samples are collected in 4 liter polypropylene bottles equipped with a 1/4" outlet and tubing at the base. Filtration is "in-line"; each bottle is connected via the tubing to a Delrin filter holder connected to a vacuum system (filtrate-collecting container and pump). Precombusted (450°C, 5 hours) 25 mm Whatman® GF/F filters (nominal pore size 0.7 µm) are mounted in the filter holders. Normally, two liters are filtered at all depths from the surface to 250 m and three liters filtered at depths between 300 and 1000 m, at a vacuum pressure of 100 - 150 mm Hg. This volume may not be adequate for all systems investigated. Filters are placed in acid-washed precombusted (450°C, 5 hours) scintillation vials, capped, and stored in a freezer (-20°C) until processed.

7.0 Procedures

7.1 *Sample Analysis*

7.1.1 Prior to analysis, the sample vials are uncapped and the filters thawed and allowed to dry overnight at 65°C. The vials are then placed overnight in a desiccator saturated with HCl fumes. The air in the desiccator is saturated due to the presence of concentrated HCl in an open container in the lower compartment of the desiccator. Thereafter, the filters are dried at 65°C and packed in precombusted (900°C, 2 hours) nickel sleeves.

7.1.2 The samples are analyzed on a Control Equipment Corporation (CEC) 240-XA Elemental Analyzer, following the guidelines given by the manufacturer. Sixty-four samples are run at a time on the auto-sampler, of which one is a standard (see below) and approximately nine are nickel sleeve blanks. The machine operator checks on the machine regularly to ensure that problems have not developed. Data are collected and stored automatically by a micro-computer.

7.2 *Standardization and blank determination:* Acetanilide standard and blanks (empty Ni sleeves) are measured prior to each batch run of samples (64 samples). For each set of samples a minimum of three empty filters are processed as an ordinary sample and analysed as filter blanks. The acetanilide standard is weighed in acetone washed tin capsules on a CAHN Electrobalance. Standard weights are usually between 0.25 and 2.0 mg. The tin capsule with the standard is put into a nickel sleeve and run on the Elemental Analyzer. The empty filter blanks are treated exactly like sample filters except that no sample water is passed through them.

8.0 Calculation and expression of results

The POC and PN weights of each of the samples are integrated and estimated automatically by the IBM-PC Compatible Computer Analytical Software, supplied with the 240-XA instrument. The program automatically includes the latest Ni sleeve blank into its calculations. The *in-situ* concentrations P_C (POC) and P_N (PN) estimated:

$$P_C, P_N (\mu g \cdot kg^{-1}) = \frac{(S - B)}{\left(\frac{V}{1000}\right)^{\rho}}$$

Where:

S	=	the result for the filtered sample
B	=	the measured filter blank
V	=	volume filtered (l)
r	=	density (a function of T, S and P, where T = temperature at which sample is taken, S = salinity of the sample, and P = 0)

9.0 References

- Control Equipment Corporation. (1988). The automated and advanced Model 240-XA Elemental Analyzer. Lowell, MA.
- Gordon, Jr. D.C. (1969). Examination of methods of particulate organic carbon analysis. *Deep-Sea Research* 16:661-665.
- Kerambrun, P. and K.H. Szekiolda. (1969). Note technique. *Tethys* 1:581-584.
- Sharp, J.H. (1974). Improved analysis for "particulate" organic carbon and nitrogen from seawater. *Limnology and Oceanography* 19:984-989.