

CCE - CalCOFI Methods Manual

CCE - CalCOFI Augmented Cruises *Particulate Organic Carbon and Nitrogen*

Summary

Particulate organic carbon and nitrogen are measured by high-temperature combustion. Samples analyzed within the California Current Ecosystem constrain the mean C:N ratio of small particulates and by difference relative to measured living biomass, the biomass of suspended detritus.

History

The original description of the method was written in 2006 by S. Dovel. Changes to the method are listed below.

Section	Date	Author	Description
1	2006	S. Dovel	Temperature combustion changed from 1020°C to 1000°C. This was to minimize the melting of GF/F glass into quartz and breaking the column. Standards were compared and no difference was observed.

Methods

1. Principle

For the analysis of particulate organic carbon and nitrogen (POC/PON) 4 liters of seawater are filtered on glassfiber filters and stored at -20°C for analysis ashore. Ashore samples are acidified, dried and analyzed by high-temperature combustion (1000°C) on an ECS 4010 CHNSO Analyzer. The sample and tin capsule react with oxygen and combust at 1000°C. The sample is broken down thus converting organic carbon to CO₂ and reducing nitrogen oxides to N₂ gas. Both gases are measured by thermal conductivity.

2. Material Combustion Procedures

To avoid contamination of Carbon, all materials used to process POC/PON samples are cleaned with 91% isopropyl alcohol or combusted in an Isotemp Programmable Muffle Furnace. Combustion process increases 10°C/min to 450°C, maintains high temperature for 6 hours and then slowly cools to room temperature.

3. POC/PON Sampling

3.1. POC/PON samples are collected for 66 CalCOFI stations and 9 SCCOOS stations located in the CCE.

Three to eight depths are sampled. POC/PN samples are collected in 0.5, 1.04 or 2.2 Liter, brown polypropylene bottles. Smaller volumes are collected when high chlorophyll concentrations are measured from the CTD fluorometer. High concentrations are typically measured around the coastal stations.

3.2. Samples are filtered on precombusted Whatman GF/F 25mm under low vacuum pressure (40 mm Hg).

3.3. Filter is removed, folded in half with the particulate part inside the fold and wrapped in precombusted aluminum foil.

3.4. Samples are stored in Liquid Nitrogen and transferred post cruise to a -20°C freezer until processed.

4. POC/PON Sample Analysis

4.1. Prior to analysis, the sample filters and blank are thawed, placed on combusted glass petri plates and acidified in a desiccator overnight with saturated HCl fumes. Saturation is achieved by placing an open container of HCl on the bottom of the desiccator.

4.2. After acidification, the HCl is removed and the samples are dried for 48 hours at 60°C.

4.3. Since half of the 25mm filter is analyzed, the total filter and the portion on the filter analyzed are weighed. The percent filtered analyzed is calculated.

4.4. The portion of the filter analyzed is folded with the sample on the outside and packed into a 9 x 10mm tin capsule.

4.5. Samples are analyzed on an ECS 4010 CHNSO Analyzer following the guidelines of the manufacturer. Thirty-nine samples, one combusted GF/F blank with tin capsule and seven standards are run at a time on an auto-sampler.

5. Standardization Determination

5.1. Three bypass blanks and a tin blank are inserted at the beginning of every run. Five Acetanilide standards are measured prior to each sample batch run. Standard weights are usually between 0 and 2.0mg, packed in a tin capsule and placed throughout the entire run.

5.2. Filter blanks are collected the same way as the samples except the filters are doubled, the top filter is discarded and the bottom filter is kept as a blank. The filter blank is prepared the same way as the samples and placed at the end of the run.

6. Calculations

POC and PON standard and sample peak areas are measured from the CHNSO Analyzer and are manually integrated using the EAS32, Windows™ based software package. Standards are calculated by using a linear reduction from the standard and blank data. Data from the CHNSO Analyzer have units of mg C and mg N per sample. These data need to be corrected for the percent of sample combusted. Percent of the filter combusted is calculated from the total weight of filter and the proportion analyzed.

Concentration of particulate organic carbon is given in $\mu\text{M C}$

$$\mu\text{M C} = (S - B) * 1000 / (V * C)$$

S = Sample corrected for the percent of sample combusted (mg C)

B = Measured GF/F filter blank and tin capsule (mg C)

V = Volume of seawater sample filtered (liters)

C = Standard atomic weight of Carbon (12.0107 $\text{g} * \text{mol}^{-1}$)

1000 is a unit conversion from mmole to μmol

Concentration of particulate organic nitrogen is given in $\mu\text{M N}$

$$\mu\text{M N} = (S - B) * 1000 / (V * N)$$

S = Sample corrected for the percent of sample combusted (mg N)

B = Measured filter blank (mg N)

V = Volume of seawater sample filtered (liters)

N = Standard atomic weight of Nitrogen (14.0067 $\text{g} * \text{mol}^{-1}$)

1000 is a unit conversion from mmole to μmol

Also shown as a C/N Ratio and Molar C/N Ratio:

$$\text{C/N Ratio} = (\text{mg C per L}) / (\text{mg N per L})$$

$$\text{Molar C/N Ratio} = (\text{mg C per L} / C) / (\text{mg N per L} / N)$$

C = Standard atomic weight of Carbon (12.0107 $\text{g} * \text{mol}^{-1}$)

N = Standard atomic weight of Nitrogen (14.0067 $\text{g} * \text{mol}^{-1}$)

7. Equipment/Supplies

Isotemp Programmable Muffle Furnace (Fisher Scientific Cat. No. 10-650)
Whatman GF/F, 25mm (Fisher Scientific Cat. No. 1825 025)
Vacuum filtration apparatus
Forceps, scissors and Aluminum Foil
Pyrex Sleeve Top Desiccator (Fisher Scientific Cat. No. 08-631B)
Pyrex Petri Dishes 60 dia. X 15mm H (Fisher Scientific Cat. No. 08-747A)
Liquid Nitrogen Dewar and -20_C Freezer for storing samples
Isotemp Standard Ovens 600 Series (Fisher Scientific Cat. No. 13-247-625G)
Sartorius microgram electrobalance
Pressed Tin Capsules 9 x 10mm (Costech Analytical Technologies, Inc. Code 041073)
Falcon Tissue Culture Plate (Fisher Scientific Cat. No. 08-772-2B)
ECS 4010 CHNSO Analyzer (Costech Analytical Technologies, INC.)
EAS32, Windows™ based software package

8. Reagents

Hydrochloric acid, Certified ACS Plus (Fisher Chemical Cat. No. A144S-212)
Acetanilide (reagent grade): Acetanilide has 0.7109 g C and 0.1036 g N per g total mass (Perkin Elmer Cat. No. 02401121)
Isopropyl Alcohol (91%)

9. References

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Sharp, J.H. 1974. Improved analysis for "particulate" organic carbon and nitrogen from seawater. *Limnology and Oceanography* **19**: 984-989.



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