

Outline of Standard Protocols for Particulate Organic Carbon (POC) Analyses

**Rich Baldino
Water Chemistry Program
University of Wisconsin-Madison**

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Revision 1

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1.0 Introduction

This method is for the determination of organic carbon on filter borne particles in the presence of inorganic carbon. The method detection limit for this procedure is 5 μg of organic carbon remaining on a GF/F filter. The maximum amount of carbon measurable is approximately 5 mg of carbon.

2.0 Selection and Pre-treatment of POC samples

2.1 Selection of Samples

Particulate organic carbon (POC) analysis begins with the selection of samples for analysis. To select POC samples, consult the complete list of POC samples compiled from the "TMLOGIN" database (DOC Login table) using the report "POC Analysis". This report contains all the POC results up to the date of printing. The next ten samples in order of collection date, which have not yet been analyzed, are selected as the next analysis batch. These ten samples are pulled from the POC freezer for analysis.

2.2 Sample Treatment

2.2.1 Sample treatment begins by placing twelve ashed aluminum planchets in the hood on a plastic tray. Two planchets are designated as blanks and an ashed GF/F glass fiber filter is placed in each. The blank filters are then treated with 200 μL sulfurous acid. The first sample is pulled and the planchet tab is labeled, using a black Sharpie, with the last three characters of the POC sample number (such as "AO3" for TMENAO3) which assures a unique sample number for each POC filter. The GF/F filter is folded in half and 200 μL of sulfurous acid is added directly onto the filter, while being held in the folded position by a pair of stainless steel tweezers. If any visible residue is retained on the aluminum foil used to protect the filter during storage, then the sections coated with the residue are removed and placed in an ashed planchet. Any foil sections are labeled with the same identifier as the filter, with the addition of a second number (e.g. AO31). The section(s) of foil are then also treated with 200 μL of sulfurous acid. The following nine samples are treated in the same manner.

2.2.2 After all filters and foil sections have been treated with 200 μL of acid, the planchets are placed in a 60 °C oven for 20 to 30 minutes. Following drying, all filters and foil sections are treated with an additional 200 μL of sulfurous acid and dried for approximately one hour.

3.0 Instrumental Analysis

3.1 Carbon is quantified on a Perkin-Elmer 2400 CHN elemental analyzer. Details of instrument operation and maintenance can be found in the PE 2400 CHN Elemental Analyzer Instruction

Manual (Part number 0993-7147). While the samples are drying, the CHN analyzer is calibrated and a check standard is run according to the manufacturer's instructions. When the filters are almost dry, clean tin disks are added to the planchets containing filters (do not cover filters) to allow the tin to dry (tin disks are stored in Milli-Q water to minimize contamination). After the filters and disks are completely dry, the planchets are removed from the oven and the filters are rolled inside the tin disks. Just before analysis, the rolled filters/tin are compressed using an aluminum tube and two stainless steel rods so that the samples do not unravel inside the instrument and cause the autosampler to jam.

Samples are analyzed in groups of five, with a treated filter blank, an analysis blank, and a check standard run afterward. Treated filter blanks must have no more than $8.8 \pm 1.5 \mu\text{g}$ carbon. Analysis blanks must agree with the blank runs used to establish the baseline, within ten analysis counts. Check standard carbon results must agree with the true amounts to within two percent.

3.2 Outline of 2400 POC Procedure

3.2.1 Starting up the machine

3.2.1.1 Turn on Oxygen (10-20 psi) and Nitrogen (42.5 psi) tanks.

3.2.1.2 Increase Helium tank to 20 psi.

3.2.1.3 Press Standby to activate purge.

3.2.1.4 Purge.

- a. Press purge gas button
- b. He--yes
- c. Enter time of 300s
- d. O -- no

3.2.1.5 Run Blank.

- a. Press single run button
- b. Press 1 for blank
- c. Press 1 again for one run
- d. Press enter
- e. Press start

3.2.1.6 Read Normals.

- a. Carbon is usually around -20 to -30
- b. Nitrogen is usually around +30 to + 40
- c. If numbers aren't close, purge again for 200s with 1 run
- d. If numbers are still not close, purge again for 100s with 2 runs

- 3.2.2 Making Standards
 - 3.2.2.1 Use acetanilide.
 - 3.2.2.2 Calibrate μ -balance on 20 mg range, using 10.000 mg calibration weight.
 - 3.2.2.3 Use two thimbles to tare balance.
 - 3.2.2.4 Take left thimble and place about 0.6 to 1.2 mg of acetanilide inside with metal spatula.
 - 3.2.2.5 Make three standards and a check standard for the beginning of the run. Then make a standard for every five samples.
 - 3.2.2.6 Place thimble in plastic container after it has been folded.
- 3.2.3 Running Standards
 - 3.2.3.1 Place standards into carousel in order weighed.
 - 3.2.3.2 Press autorun button. Run standards in single runs.
 - 3.2.3.3 Leftmost number should appear as 1. If not, press 4, and then 1 to reset.
 - 3.2.3.4 For standards, press 2 for K factor.
 - 3.2.3.5 Press 1. Punch in weight of first standard. Press Enter. Press Start.
 - 3.2.3.6 For check standard, press 3 for sample.
 - 3.2.3.7 For samples, create an ID#.
 - 3.2.3.8 Press start. Watch and verify carousel alignment.
- 3.2.4 Run Order
 - 3.2.4.1 Three standards, a check standard, five samples, a foil blank, a method blank, a check standard, five samples, a foil blank, a method (empty run) blank, and a check standard.
- 3.2.5 Turning Off the Machine
 - 3.2.5.1 Press auto run.
 - 3.2.5.2 Press standby button.

3.2.5.3 Shut off the Oxygen and Nitrogen tanks.

3.2.5.4 Decrease Helium pressure to 10 psi.