

**Standard Operating Procedure for the
Sampling and Analysis of Total Suspended
Solids in Great Lakes Waters**

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1.0 Scope and Application

This Standard Operating Procedure describes the sampling and analysis of Great Lakes Waters for total suspended solids (TSS). Samples of lake water are collected and filtered through a 0.7 μM pore-size glass fiber filter. Total suspended solids are operationally defined as the mass retained on the filter per unit volume of water.

2.0 Safety and Waste Handling

All applicable safety and waste handling rules are to be followed. These include the proper labeling and disposal of chemical wastes. Over-board discharges of chemical wastes are forbidden. Refer to the GLNPO Safety, Health, and Environmental Compliance Manual for specific rules.

3.0 Summary of Procedure

Great Lakes water samples are collected at pre-determined sampling stations and depths *via* either a submersible pump or Rosette sampler. Sub-samples of water are then filtered under vacuum through a 47 mm diameter glass fiber filter, which has been washed and dried to constant weight. The suspended solids are retained on the filter and frozen at $-10\text{ }^{\circ}\text{C}$ until final weighing on an analytical balance in a land-based laboratory.

4.0 Description of Apparatus

Glass fiber filters are pre-weighed on an analytical balance. Water samples (typically 2-4 liters for open-lake locations) are collected from an over-board pump or Rosette sampler. The filters are supported on a commercially-available, all-glass, 350 mL vacuum filtration apparatus. Two filtration apparatuses are attached, side-by-side, to ring stands. Tygon tubing (3/8" ID) is used to connect the filtration flasks to an oil-less vacuum pump. Final weights of the filters are determined identically to the initial weights. The equipment needed are listed in Table 1.

5.0 Preparation of Filters and Analytical Balance

5.1 Preparation of Filters

- 5.1.1 Filter preparation should take place as close to the start of the survey as possible.
- 5.1.2 Filters are to be handled only with stainless steel forceps. Filters that are mishandled after preparation should be discarded.
- 5.1.3 Label the 47 mm diameter GF/F filters (0.7 μ M pore-size) using a permanent marker on the outer edge of each filter. Label from 1 - "X" with "X" being the total number of filters prepared. Allow the ink to dry for 5 minutes before proceeding to step 5.1.4.
- 5.1.4 Condition the filters using a 350 mL vacuum filtration apparatus. Pass 350 mL of organic-free, distilled, deionized water (from now on referred to as organic-free water) through each filter. Place the filters onto the filtration apparatus with the labeled side facing up.
- 5.1.5 Remove the filters from the filtration apparatus and place them into individual 50 mm aluminum weighing pans. Dry the filters in a 105 °C oven for 2 hours.
- 5.1.6 Remove the filters from the oven and place them into a desiccator. Allow the filters to cool for 5 minutes.
- 5.1.7 Prepare the analytical balance as described in section 5.2.
- 5.1.8 Remove the filters from the desiccator in small groups and weigh them on the analytical balance.
- 5.1.9 Record the initial filter weights on the TSS Sampling Log Sheet and place them individually into identically-numbered 50 mm diameter plastic petri dishes.
- 5.1.10 Every tenth filter must be redried in the 105 °C oven for two hours and re-weighed. Record the second value on the log sheet.
- 5.1.11 If the second weight does not fall within ± 0.1 mg of the initial weight, check if the balance is zeroed correctly. If the balance has deviated, re-zero and re-weigh the filter. If the weight still does not fall within ± 0.1 mg, the previous group of 10 filters must be re-dried and re-weighed.

5.2 Preparation of Analytical Balance

- 5.2.1 A top-loading analytical balance with a capacity of 200 mg and a resolution of 0.01 mg is needed. The balance should be accompanied with a set of calibration weights, preferably NIST traceable. (e.g., Mettler 22 balance with BA monitor.)
- 5.2.2 Allow sufficient time for the analytical balance to warm up to operating temperature. Then zero the balance.

- 5.2.3 Record in the balance logbook the performance over the following range: 10 mg, 30 mg, 50 mg, 100 mg, and 150 mg. This range allows the use of filters weighing 1-150 mg.
- 5.2.4 Record in the balance logbook the temperature of the balance room. Note any fluctuations during use.
- 5.2.5 A new, dry 47 mm GF/F filter weighs approximately 120 mg. Tare the balance to 100.00 mg using the 100 mg weight from the set of calibration weights.
- 5.2.6 During the filter preparation and analysis procedures, the balance is tared to 100.00 mg after every tenth filter. If the balance deviates more than +/- 0.03 mg, the balance is again tared to 100.00 mg, and the previous group of 10 filters is re-weighed.

6.0 Filtration and Analysis Procedures

6.1 Filtration Procedure

- 6.1.1 Using stainless steel forceps, place one 47 mm GF/F filter onto the fitted glass support of the sampling apparatus. Place the glass funnel on top of the filter and secure with the clamp. Label the Great Lake name, station number, sampling depth, and date onto the petri dish.
- 6.1.2 Collect the lake water sub-samples from the submersible pump hose or Rosette sampler. Allow the overboard pump line to flush for 15-30 minutes. Collect the lake water into a 4 liter cubitainer or four, 1 liter bottles. Rinse the container(s) twice with approximately 1 liter of lake water before collecting the sample. If the lake water is to be collected from the Rosette, rinse the container(s) with only 200-300 mL of lake water to insure there is enough remaining to establish a significant particulate load on the filter (see section 6.1.6).
- 6.1.3 Measure the volume of lake water to be filtered in a graduated cylinder, or mark four 1 liter Teflon bottles at the 1 liter level. Prior to filling, rinse the bottles or cylinder twice with approximately 100 mL of lake water.
- 6.1.4 Connect the vacuum pump to the filtration flask. Pour a measured volume of lake water into the glass filtration funnel. Turn on the vacuum pump. Maintain the vacuum between 5-10 inches of Hg during filtration.
- 6.1.5 Continue pouring lake water into the funnel until sufficient suspended solids have been collected.
- 6.1.6 The volume of lake water required to produce a reliable TSS measurement will vary with lake, station location, depth, and time of year. More than 1 mg of suspended solid material is needed. For open-lake, oligotrophic conditions, typically 2-4 liters will provide enough particulate matter. For near-shore locations, or meso-eutrophic and eutrophic conditions, lake water volumes in the range of 200-500 mL are typical. A filter that becomes visibly

covered with solids and a flow of water through the filter that drops significantly are evidence that sufficient suspended solids have been collected.

- 6.1.7 After the lake water has been filtered, rinse the sides of the funnel with approximately 20 mL of organic-free water and filter this rinse. Turn off the vacuum pump.
 - 6.1.8 Remove the funnel and, using stainless steel forceps, fold the filter in half and place it back into the numbered petri dish. Place groups of petri dishes in a labeled Ziplock bag and store at -10 °C. Record the Great Lake name, station number, sampling depth, volume filtered, analyst, date, and time of day on the TSS Sampling Log Sheet.
 - 6.1.9 Empty the filtrate from the filtration flask.
 - 6.1.10 Rinse the filtration funnel, fitted glass support, flask, and the container(s) with organic-free water.
 - 6.1.11 Re-assemble the filtration apparatus.
 - 6.1.12 Place aluminum foil covers over the filtration funnel.
- 6.2 Analysis Procedure
- 6.2.1 Remove the filters from the freezer and allow them to thaw. Using stainless steel forceps, remove the filters from the petri dishes and place each in an individual 50 mm aluminum weighing pan. When handling the filters, grasp only the outer edges with the forceps.
 - 6.2.2 Dry the filters in a 105 °C oven for two hours.
 - 6.2.3 Prepare the analytical balance (section 5.2).
 - 6.2.4 Using the same analytical balance as the initial weighing procedure, follow steps 5.1.6 - 5.1.12 to determine the final weights of the filters.
 - 6.2.5 Store the filters in a freezer after all of them have been weighed and the results recorded.
 - 6.2.6 Calculate the total suspended solids (TSS) as:

$$\text{Total Suspended Solids (mg/L)} = \frac{[\text{Final Weight} - \text{Initial Weight}]}{[\text{sample volume in liters}]}$$

7.0 Quality Control

- 7.1 A duplicate sample will be filtered in parallel at least once during the sampling of each Great Lake.
- 7.2 A TSS matrix blank will be collected, in duplicate, at the beginning of each survey of the Great Lakes and at least once during the sampling of each Great Lake. A TSS matrix blank is collected by filtering 1 liter of organic-free water. The matrix blanks are processed identically to Great

Lakes water samples.

- 7.3 A TSS field blank will be collected, in duplicate, at the beginning of each survey of the Great Lakes and at least once during the sampling of each Great Lake. A TSS field blank is prepared by taking a filter out of the foil envelope, placing it onto the fitted glass support of a clean filtration apparatus, wetting the filter with organic-free water and assembling the filtration apparatus. The apparatus is disassembled, and the filter is removed and processed in the same manner as a sample.
- 7.4 Two TSS trip blanks will be processed after the survey has ended. This is accomplished by placing two filters in their petri dishes into the Ziplock bag and processing these filters like samples.
- 7.5 Because TSS is an ancillary parameter to the determination of hydrophobic organic contaminants (HOCs), the TSS samples during an organics survey are taken simultaneous to the HOC samples. Therefore when a HOC matrix blank, field blank or duplicate sample is collected a TSS matrix blank, field blank or duplicate will also be collected.

Table 1: List of Filtration Equipment

<u>Quantity</u>	<u>Equipment</u>	<u>Source or Equivalent</u>
2	Oil-less Vacuum Pump	Schuco 5711-130
2	Teflon wash bottle	Cole-Parmer N-06052-60
2	350 mL all-glass filtration apparatus	Nucleopore
2	Stainless steel forceps	
2	Support/ring stand for filtration apparatus	
1	Toploading analytical balance 200 mg capacity 0.01 mg resolution calibration weights	
1	Dessicator	
1	Drying oven	

Miscellaneous (some quantities depend on number of samples)

- 47 mm GF/F filters (0.7 µM pore-size) Whatman 1825-47
- Cubitainers
- Tygon tubing (3/8"ID)
- 50 mm diameter aluminum weighing pans
- 50 mm diameter plastic Petri dishes
- permanent markers
- Ziplock freezer bags