

Standard Operating Procedure for:

Total Suspended Solids, Particulate Organic and Inorganic Matter

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1 Identification of the method

- 1.1 Gravimetric measurement of total suspended solids, particulate organic matter, and particulate inorganic matter (APHA Method 2540 D and E).

2 Applicable matrix or matrices

- 2.1 This method is suitable for the analysis of [environmental samples](#).

3 Detection limit

- 3.1 Total Suspended Solids [Method Detection Limit](#): 0.1 mg/L
3.2 Particulate Organic Matter [Method Detection Limit](#): 0.1 mg/L
3.3 Particulate Inorganic Matter [Method Detection Limit](#): 0.1 mg/L
3.4 The above [Method Detection Limits](#) are based on the lowest reliable measurement of the scale used to perform this analysis.

4 Scope of the method

- 4.1 This standard operating procedure is intended to provide MU Limnology [operators](#), [technicians](#), and [analysts](#) with guidance on the gravimetric analysis of total suspended solids (TSS), particulate organic matter (POM), and particulate inorganic matter (PIM). This document is not intended to replace individual training in this method by an experienced MU Limnology [technician](#).

5 Summary of the method

- 5.1 A measured volume of a collected water sample is filtered through a pre-washed (with [UPDI](#)), [pre-combusted](#), and weighed filter. The filter is dried at 105 °C and weighed to determine the total amount of material collected on the filter. The filter is then combusted for 1.5 to 3 hours at 550 °C to combust organic materials. The filter is re-weighed to determine the amount of inorganic material remaining.
- 5.2
- Run Time: 1.5–3 hours
 - Samples per run: 23

6 Interferences

- 6.1 Imprecise filtration of samples will lead to poor replication between duplicates.
6.2 Material collected on the filter are susceptible to loss, filters should be folded in half with collected material to the inside to protect from loss.
6.3 Filters should be stored in paper filter houses that are folded and stapled to reduce the risk of contamination.

7 Health and Safety

- 7.1 This method involves handling freshwater samples that may contain live microorganisms and therefore pose some threat of infection. Laboratory personnel who are routinely exposed to such water samples are encouraged to protect themselves from water borne illnesses by wearing clean disposable gloves and washing hands frequently.
- 7.2 This method involves the use of a high temperature muffle furnace. Heat protective gloves and metal tongs should be used to add and remove materials from the muffle furnace.

8 Personnel qualifications

- 8.1 Samples will be analyzed by MU Limnology staff who have been trained to the [operators](#) or [technician](#) level in this method and who are familiar with all of the MU Limnology sampling handling and labeling procedures and appropriate [SOPs](#).

9 Equipment and supplies

- 9.1 Analytical Balance
- 9.2 Muffle Furnace
- 9.3 Whatman Grade 934-AH, 1.5 μ m pore size, Glass Microfiber Filters, 42.5 mm diameter
- 9.4 47 mm Magnetic Filter Flask, PALL
- 9.5 Internal Vacuum System or Portable Vacuum Pump
- 9.6 250 ml Graduated Cylinder
- 9.7 Strathmore 100% Pure Cotton Wove Paper
- 9.8 Silica Gel Desiccant, Fisher Chemical, S162-212
- 9.9 Long-handled tongs
- 9.10 Heat-proof gloves

10 Quality Control

- 10.1 [Field Blanks](#) and [Field Duplicates](#)
At least once per field season, a [field blank](#) and a [field duplicate](#) should be run.
- 10.2 [Check Weight](#)
A 1 g [check weight](#) will be measured after every 5 filters have been measured. The measured weight will be recorded.
- 10.3 Blanks
For every 22 filters, 3 blanks will be analyzed. These blank filters will be prepared, stored, and analyzed with the same set of 23 filters.

11 Filter Preparation

- 11.1 Prior to use, 934-AH filters require preparation.

- 11.2 Place ~20 filters in a clean beaker with [UPDI](#) and swirl gently. Decant the water and repeat this process until the water comes clear.
- 11.3 Place a single 934-AH filter in a filter flask and rinse three times with >20 ml of [UPDI](#). Make sure the filter is covered completely during each rinse. The filter must dry completely between washes.
- 11.4 Place filters on a tinfoil sheet and place in a drying oven at 105 °C for 20 minutes.
- 11.5 Stack filters in a crucible and pre-combust them at 550 °C for 1 hour.
- 11.6 Once pre-combusted and cooled to room temperature in a desiccator, record the weight of each filter in a logbook and place it into a uniquely numbered filter house.

12 Analysis

12.1 Set-Up and Drying

- Turn on the drying oven and set to 105 °C.
- Turn on the muffle furnace and set to 550 °C.
- Filters should be analyzed 25 at a time (22 samples and 3 blanks).
- Remove the filters from their filter houses using forceps and place them in numbered tin drying dishes, being careful that the filtrate does not make contact with the forceps or the dish. Note any damage to the filter on analytical worksheet.
- Place the filters in the drying oven at 105 °C for 30 minutes.
- Remove the 3 filters from the oven and weigh them, noting their weight on the analytical worksheet.
- Return the filters to the drying oven for 10 minutes and then reweigh them.
- If there is no change in filter weights, the filters are considered dried/stable and you may proceed. If the weight declines between weighing continue to dry the filters until the weight no longer changes.

12.2 First Weighing

- Turn on the analytical balance and tare it after the reading has stabilized.
- Weigh all 25 filters (22 samples, 3 blanks) on the analytical balance to get the “Pre” weight.
- Ensure that the balance reading has stabilized before recording the weight.
- Reweigh the [check weight](#) after every 5 filters measured. If drift has occurred, re-tare the balance and re-weigh the last five filters.

12.3 Muffling

- Using long-handle tongs, place measured filters (still in their tin drying dishes) into the preheated muffle furnace for 20 minutes.

- If the muffle furnace cools significantly below 550 °C while loading, wait for it to reheat before starting the 20-minute timer.

12.4 Second Weighing

- Remove the filters from the muffle furnace after 20 minutes and allow them to cool in a desiccator.
- Turn on the analytical balance and tare it after the reading has stabilized.
- Weigh all 25 filters (22 samples, 3 blanks) on the analytical balance to get the “Post” weight.
- Ensure that the balance reading has stabilized before recording the weight.
- Reweigh the [check weight](#) after every 5 filters measured. If drift has occurred, re-tare the balance and re-weigh the last five filters.

13 Calculations and reporting

13.1 Defined Terms

- TW= Tare Weight (mg)
- PW= Pre-Weight (mg)
- PTW= Post-Weight (mg)
- FV= Filter Volume (ml)

$$TSS \left(\frac{mg}{L} \right) = \frac{PW - TW}{\left(\frac{FV}{1000} \right)}$$

$$POM \left(\frac{mg}{L} \right) = \frac{PW - PTW}{\left(\frac{FV}{1000} \right)}$$

$$PIM \left(\frac{mg}{L} \right) = \frac{PTW - TW}{\left(\frac{FV}{1000} \right)}$$

14 Method performance

- 14.1 Total Suspended Solids [Method Detection Limit](#): 0.1 mg/L
- 14.2 Particulate Organic Matter [Method Detection Limit](#): 0.1 mg/L
- 14.3 Particulate Inorganic Matter [Method Detection Limit](#): 0.1 mg/L
- 14.4 Precision < 10 %

15 Pollution prevention

- 15.1 This method does not produce any pollution/waste of concern.

16 Data assessment, acceptable criteria for quality control measures, and corrective actions for out-of-control or unacceptable data

16.1 [Check Weight](#)

The [check weight](#) should not deviate in recorded weight from 1 g during the course of analysis. If deviation is observed, the recorded filter weight should be adjusted accordingly.

16.2 Blanks

The calculated TSS, POM, and PIM for all blanks should be 0.0 mg/L. If this is not true, a correction factor should be applied to the calculated values for all filters. This correction value should bring the calculated TSS, POM, and PIM for all blanks as close to 0.0 as possible.

16.3 [Field Blank](#) and [Field Duplicates](#)

When run, [field duplicates](#) should not vary in determined concentrations by more than 10 %. Determined concentrations of [field blanks](#) should not exceed the [method detection limit](#).

17 Waste management

17.1 No waste is generated as a part of this method. Spent filters should be disposed of in regular trash.

18 References

18.1 Standard Methods for the Examination of Water and Wastewater, 23rd Edition. 2017. Method 2540 D and E. American Public Health Association. Washington, DC.