

Standard Operating Procedure for the
Determination of Suspended Sediments
CCAL 12A.4

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1.0 Scope and Application

- 1.1 Suspended Sediments are defined as the residue which will pass through a 1 mm² wire mesh screen, and is retained in a glass fiber filter after sample filtration. The typical detection limit for this determination is 2 mg/L nonfilterable solids (see section 13.2 for more information).

2.0 Summary of Method

- 2.1 A homogenous sample aliquot is filtered through a tared, glass fiber filter. The sedimented filter is dried to constant weight at 80°C for five days. The gain in weight of the filter represents the suspended solids per initial volume filtered. CCAL uses Whatman GF/F or GF/C glass microfiber filters; 0.7 and 1.2 um particle retention ratings, respectively.

3.0 Definitions

- 3.1 DI water: Water that has been through a deionization system to produce water similar to ASTM Type I reagent with 16.7 Mohms resistivity (ASTM) (Reference 16.3).

4.0 Interferences

- 4.1 Samples with high mineral content may be hygroscopic and require prolonged drying, followed by proper desiccation and rapid weighing.
- 4.2 Avoid excessive residue, as this may result in formation of a water-trapping crust.
- 4.3 Prolonged filtration times as a result of filter clogging may cause high results due to increased colloidal materials captured on the clogged filter.
- 4.4 Freezing of sample may result in bias of results. Samples should be stored cold at 4°C, but not frozen.

5.0 Safety

- 5.1 The toxicity or carcinogenicity of each reagent has not been precisely determined; however, each chemical should be regarded as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. Cautions are included for known extremely hazardous materials.

6.0 Equipment and Supplies

Note: *Brand names, suppliers and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

- 6.1 Watch glasses
- 6.2 Drying oven equipped with thermostatic control capable of maintaining temperature within 2°C range
- 6.3 Desiccator - with moisture indicating desiccant
- 6.4 Analytical balance - capable of weighing to 0.1 mg
- 6.5 Filtration system - filter funnel, filter stage, filter barrel, clamps, Erlenmeyer filter flask
- 6.6 Graduated cylinder
- 6.7 GF/F or GF/C filter papers
- 6.8 Stainless steel screen with 1 mm² mesh
- 6.9 Large porcelain Buchner funnel
- 6.10 Vacuum system and connecting hoses

7.0 Reagents and Standards

- 7.1 Preparation of Standards

NA

8.0 Sample Handling and Storage

- 8.1 Unfiltered samples are stored at 4°C in the dark. Samples are filtered within 7 days of receipt, unless otherwise specified by investigator or project requirements.

9.0 Quality Control

- 9.1 Immediately after all filters have been weighed, a quality assurance weight check (QA) must be performed on 10% of the filters by someone other than the person performing the initial measurements. The QA weight must be within ± 0.5 mg of the initial weight.
- 9.2 A filter blank is run biyearly, or on a project request basis. A filter blank is 1 L DI water run through the same process as samples for determination of Suspended Sediment.

10.0 Calibration and Standardization

- 10.1 Balances: calibrated yearly by external vendor.

11.0 Procedure

11.1 Filter Preparation

- 11.1.1 Multiple filters are prewashed in a large Buchner Funnel with 500 to 1000 mL DI water to remove residual contaminants from the manufacturing process.
- 11.1.2 Filters are then dried in an 80°C oven overnight.
- 11.1.3 Dried filters are transferred to a desiccator and allowed to cool and stabilize overnight.
- 11.1.4 Filters are numbered with pencil and numbers are copied to the log sheet (see section 17).
- 11.1.5 Filters are returned to the oven for five days.
- 11.1.6 Dried filters are transferred to a desiccator and allowed to cool and stabilize overnight.
- 11.1.7 Weigh clean, dry filters.
- Zero the balance.
 - Weigh a filter.
 - Record weight on the Log Sheet.

d) Recheck balance zero between weighings.

- 11.1.8 Immediately after all filters have been weighed, a quality assurance weight check (QA) must be performed on 10% of the filters by someone other than the person performing the initial measurements. The QA weight must be within ± 0.5 mg of the initial weight, or the filters must be reweighed and QA rechecked. If consistent increase in filter weight is observed, the filters are baked again (section 11.1.2).
- 11.1.9 Tared filters are stored in original box labelled with content numbers.

11.2 Sample Setup

- 11.2.1 Record sample numbers on the Filter Paper Log Sheet by corresponding filter number.
- 11.2.2 Record the filter paper tare weight and the sample number on the Summary Results sheet.
- 11.2.3 Weigh the sample and container and record the gross weight on the Summary Results sheet.
- 11.2.4 Filter the sample; ensure complete transfer of sample contents. Exclude large particulates with filter screen suspended over filter barrel.
- 11.2.5 Weigh empty sample container and record bottle tare weight on the Summary Results sheet.
- 11.2.6 With tweezers, transfer sedimented filter to a clean sectioned and prenumbered watchglass; record section number on the Summary Results sheet.
- 11.2.7 Place samples in the 80°C drying oven and evaporate for at least five days to constant weight.
- 11.2.8 Transfer dried filters to desiccator(s) and allow to stabilize overnight.
- 11.2.9 Weigh sample filters and QA as before (11.1.5 – 11.1.6). Record weights on the Summary Results sheet.
- 11.2.10 The difference between the final filter weight and the filter tare weight, divided by the volume of sample filtered, equals the weight of the suspended solids per volume of sample.

11.3 Procedural Notes

- 11.3.1 Dried filters will gain weight if left in the open air. Minimize time out of the desiccator, close the door of the desiccator after removing/returning a filter.
- 11.3.2 Ensure no residual sediment remains on the filter barrel when disassembling the filter apparatus. Transfer residual sediment with DI water or filtered sample (depending on filtrate use) if necessary.

12.0 Data Analysis and Calculations

- 12.1 Suspended Sediment Results are reported as mg/liter.

$$\text{SSED mg/L} = \frac{(B - A) \times 1000 \text{ mg/g}}{V}$$

Where; *A* = filter tare weight (g), *B* = filter tare weight + dried sediment weight (g), *V* = volume of sample filtered in Liters

13.0 Method Performance

- 13.1 See 9.0 Quality Control
- 13.2 The typical method detection limit in determination of Suspended Sediment is 2 mg/L. The required use of additional filters and/or low volume sample will result in an increase in the detection limit. Similarly, processing of large volume will decrease the detection limit. Accuracy of this determination is highly dependent upon the characteristics of the sample submitted.

14.0 Pollution Prevention

- 14.1 The chemicals used in this method pose little threat to the environment when properly managed.
- 14.2 All standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of disposable waste.
- 14.3 For further information on pollution prevention, consult *Less is Better: Guide to Minimizing Waste in Laboratories*, available from the American Chemical Society at www.acs.org.

15.0 Waste Management

- 15.1 It is the laboratory's responsibility to comply with all federal, state and local regulations governing waste management, and to protect the environment by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is required.

- 15.2 For further information on waste management, consult *Less is Better: Guide to Minimizing Waste in Laboratories*, available from the American Chemical Society at www.acs.org, and *Environmental Management Guide For Small Laboratories* (233B00001) from the US Environmental Protection Agency at <https://nepis.epa.gov>.

16.0 References

- 16.1 Standard Methods For The Examination of Water and Wastewater, Method 2540 – Total Suspended Solids Dried at 103 - 105°C. American Public Health Association. 21st Edition, 2005.
- 16.2 Code of Federal Regulations. Protection of Environment. Section 40, Appendix B to Part 136. Definition and procedure for the determination of the method detection limit. Revision 1.11. Revised July 1, 1990. Office of the Federal Register, National Archives and Records.
- 16.3 ASTM. American Society for Testing and Materials. Standard Specifications for Reagent Water. D1193-77 (Reapproved 1983). Annual Book of ASTM Standards, Vol. 11.01. ASTM: Philadelphia, PA, 1991.

18.0 Document Revision History

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Address update

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Add hold time information; Section 8.0

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Section 11: update procedures for clarification

Section 15: update waste management resources

Update address and general edits throughout