

Standard Operating Procedure for the  
Determination of Suspended Sediments  
CCAL 12A.0

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## 1.0 Scope and Application

- 1.1 Suspended Sediments are defined as the residue which will pass through a 1 mm<sup>2</sup> wire mesh screen, and is retained in a glass fiber filter after sample filtration. The detection limit for this determination is 5 mg/L nonfilterable solids.

## 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.

5.1.1 There are no reagents associated with this method

## 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.*

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- 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 mm<sup>2</sup> 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.

## 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  $\pm 0.5$  mg of the initial weight.

## 10.0 Calibration and Standardization

10.1 Balances: calibrated yearly by external vendor.

## 11.0 Procedure

### 11.1 Filter Preparation

11.1.1 Filters must be prewashed 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 for five days.

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 transferred to the log sheet (see section 17).

11.1.5 Weigh clean, dry filters.

a) Zero the balance.

b) Weigh a filter.

c) Record weight on the Log Sheet.

d) Recheck balance zero every ten filters.

11.1.6 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 is recorded in red on the right side of the log sheet along with the initials of the person performing the QA check. The QA weight must be within  $\pm 0.5$  mg of the initial weight. If the QA weight does not fall to within  $\pm 0.5$  mg of the final initial weight, then the filters must be reweighed in reverse order from the out-of-QA-limit filter until measurements fall to within  $\pm 0.5$  mg of the final initial weight. Reweighed filters must again be QA checked and meet the above criteria or be reweighed

until QA criteria are met. It is especially important that as little time as possible elapse between initial weighing and QA weighing.

- 11.1.7 Tared filters are stored in original box. Record content numbers on the box cover.

## 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 watchglass labeled with the sample name and number.
- 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 tare weight 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, and replace the lid 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 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

### 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: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16<sup>th</sup> Street NW, Washington D.C. 20036, (202) 872-4477.

### 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 "The Waste Management Manual for Laboratory Personnel", and "Less is Better: Laboratory Chemical Management for Waste Reduction", both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16<sup>th</sup> Street NW, Washington DC, 20036.

### 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. 21<sup>st</sup> 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.

## **17.0 Tables, Diagrams, Flowcharts, and Validation Data**

- 17.1 Filter Paper Log Sheet



