Standard Operating Procedure for the Determination of Suspended Sediments CCAL 12A.3

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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^2 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 80ºC 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 4ºC, 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 5^{o}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^2 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 80ºC 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 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).

11.1.7 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. Exception: for standard CCAL issued bottles a previously determined average tare weight is used.

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 80ºC 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, 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, 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

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: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th 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 16th 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 - 105ºC. 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.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Filter Paper Log Sheet

Log Sheet for Filter Paper Tare Weights for Suspended Sediment Determination

Filter Type: ________________

Paper #	Tare Wt.	Sample #	Zero/QA

Paper #	Tare Wt.	Sample #	Zero/QA

Suspended Sediment Summary Results

Project _____ Samples Filtered _____ Final Weighing _____ Analyst _____

Remarks: _____

Sample #	Filter Tare (g)	Filter Tare + Sediment (g)	Bottle Gross Weight (kg)	Bottle Tare Weight (kg)	Net Volume (L)	Notes

18.0 Document Revision History

Original Document: April 2006
Version: 12A.0
Title: Standard Operating Procedure for the Determination of Suspended Sediments

Edit Date: February 2010
New Version: 12A.1
Address update

Edit Date: June 2013
New Version: 12A.2
Add hold time information; Section 8.0
Revise detection limit and add variability statement; Section 1.1 and 13.2
Add exception; Section 11.2.5
Add filter blank; Section 9.2

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General editing
Change acceptance range; Section 6.2