Standard Operating Procedure for the Determination of Total and Total Dissolved Solids CCAL 13A.4

Cooperative Chemical Analytical Laboratory
College of Forestry
Oregon State University
3015 Western Blvd
Corvallis, Oregon

Prepared by Kathryn Motter
And Laura Hartley
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Table of Contents

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1.0 Scope and Application

1.1 Total Dissolved Solids are defined as the material residue left in a vessel, after evaporation of a sample that has passed through a filter. Total Solids are the material residue left after evaporation of an unfiltered sample. The detection limit for this determination is 5 mg/L solid residue.

2.0 Summary of Method

2.1 A thoroughly mixed sample is volumetrically transferred to a tared beaker and evaporated to dryness. The gain in weight represents the total or total dissolved solids per initial volume. CCAL uses Whatman GF/F or GF/C glass microfiber filters; 0.7 and 1.2 um particle retention ratings, respectively. Samples are evaporated at 180ºC for five days.

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 extended drying, followed by desiccation and rapid weighing.

4.2 Samples high in bicarbonate require extended drying to insure complete conversion of bicarbonate to carbonate.

4.3 Avoid excessive residue, as this may result in formation of a water-trapping crust.

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.*

6.1 150 mL Griffin beakers (glass)

6.2 Drying oven equipped with thermostatic control capable of maintaining temperature within 5ºC 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

6.6 100 mL graduated cylinder

6.7 GF/F or GF/C filter papers

6.8 Stainless steel screen with 1 mm^2 mesh.

6.9 Vacuum system and connecting hoses.

7.0 Reagents and Standards

7.1 Preparation of Standards

7.1.1 *1000 ppm NaCl Solution:*
Dissolve 1.000 g NaCl (oven-dried at 80ºC) into 1000 mL of DI water and mix until dissolution is complete.

8.0 Sample Handling and Storage

8.1 Samples are stored at 4°C in the dark.

9.0 Quality Control

9.1 Preparation of standards is recorded on worksheets and documented by weight of standard added to a given flask before dilution to volume with DI water. All records of certification are kept on file at CCAL Laboratory.

9.2 Blank: DI water run twice each analysis set.

9.3 Quality Control Check Standard: 1000 ppm NaCl check standard run once each analysis set.

9.4 Control: empty beaker run through complete process.

10.0 Calibration and Standardization

10.1 Balances: calibrated yearly by external vendor.

10.2 Acceptable recovery of NaCl check standard is ± 2 %.

11.0 Procedure

11.1 Beaker Preparation

11.1.1 Place prenumbered beakers into a 180ºC drying oven and dry for five days to a constant weight.
11.1.2 Transfer dried beakers to desiccator(s) and allow to stabilize overnight or longer.
11.1.3 Record beaker numbers to be used on the Data Summary and Weight Record data sheets.
11.1.4 Zero the balance.
11.1.5 Weigh clean, dry, empty beakers. Record weight on the Weight Record data sheet.
11.1.6 After all beakers have been weighed, a quality assurance weight check (QA) must be performed on 20% of the beakers by someone other than the person performing the initial measurements, or by the same person the following day.

11.1.7 After all beakers have been weighed, transcribe the lowest of consistent weights for each beaker from the Weights Record data sheet to the "Tare" column of the Summary Results data sheet.

11.2 Sample Setup

11.2.1 Assign sample numbers to beaker numbers on the analysis data sheet.
11.2.2 Each analysis batch must have a control beaker to which no sample is added, two DIW blanks, and one 1000 mg/l NaCl standard.
11.2.3 In a graduated cylinder, measure 100 mL of sample, blank or standard into the appropriate dry, tared beaker.
11.2.4 Place samples, control, standard and blanks in the 180ºC drying oven and evaporate for at least five days to constant weight.
11.2.5 Transfer dried beakers to desiccator(s) and allow to stabilize overnight or longer.
11.2.6 Weigh sample beakers as before (11.1.4 – 11.1.6), allowing each beaker to stabilize.
11.2.7 After all beakers have been weighed, transcribe the lowest of consistent loaded weights for each beaker from the Weights Record data sheet to the "Tare + Sediment" column of the Summary Results data sheet.
11.2.8 The difference between the loaded weight and the tare weight equals the weight of the dissolved solids in 100 mL of sample.

11.3 Procedural Notes

11.3.1 Make multiple measurements to assure a constant weight. Repeat weighing procedure until three consecutive measurements match to within 0.2 mg. Hygroscopic matter may result in water retention. If weight continually increases after multiple readings, use the initial weight.
11.3.2 Dried beakers 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 beaker.
11.3.3 Recheck balance zero between weighings.
11.3.4 The QA weight must be within ± 0.5 mg of the final weight. If the QA weight does not meet this criterion, then the beakers must be reweighed. Reweighed beakers must again be QA checked and meet the above standard. If not, the process is repeated, or beakers are returned to the oven for further dry time.
11.3.5 It is especially important that minimal time elapse between final weighing and QA weighing.

11.3.6 If multiple balances are available in the laboratory, use the same balance for the tare and loaded measurements to minimize variability.

11.3.7 If dissolved solids fraction is low (less than l mg) an additional 100 mL of sample can be added to the same beaker and the process repeated.

11.3.8 Beakers with samples should not be dried more than 15 days to prevent "over drying".

11.3.9 Do not touch beakers with bare hands during the weighing and analysis process as skin oils may leave a detectable residue.

12.0 Data Analysis and Calculations

12.1 Filterable residue (total dissolved solids; TDS) or unfiltered residue (total solids; TS)
Results are reported as mg/liter.

$$\text{TS or TDS mg/L} = \frac{(B - A) \times 1000 \text{ mg/g}}{0.100 \text{ L}}$$

where A = beaker tare weight (g), B = beaker tare weight + dried sediment weight (g)

13.0 Method Performance

13.1 This method was validated through inter-laboratory studies. The CCAL participates in the USGS Standard Reference Water QA program.

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 C – Total Dissolved Solids Dried at 180ºC. 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 Total and Total Dissolved Solids Summary Results Data Sheet

Total Dissolved Solids Summary Results

Project: _____ Analyst: _____

	Beakers Initial Weighed	Samples Setup	Final Weighing
Dates:			

Remarks: _____

Sample #	Tare (g)	Tare + Sediment (g)	Volume (L)	Notes	Beaker #
QDSC1				Control	
QDSB1				Blank	
QDSB2				Blank	
QDSL1				1000 ppm Std	

17.2 Total and Total Dissolved Solids Weights Record Data Sheet

Total Dissolved Solids Weights Record

Tare Tare + Sediment (circle one)

Pull Date___________________

Setup Date___________________ Analyst ___________________ Weigh Date ___________________

Project & Sample Number	Beaker #	Weighing # 1 / 5	2 / 6	3 / 7	4 / 8	Zero / QA Check

18.0 Document Revision History

Original Document: March 2006
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Title: Standard Operating Procedure for the Determination of Total Dissolved Solids

Edit Date: February 2010
New Version: 13A.1
Address update
Add Total Solids procedure and change document title to reflect the addition

Edit Date: February 2015
New Version 13A.2
Section 6.2: update temperature acceptance range
Section 11: reorganized for clarity
General editing

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New Version: 13A.3
Address update
General editing

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New Version: 13A.4
General editing
Updated Data Sheets, 17.1 and 17.2