

Standard Operating Procedure for the
Determination of Total and
Total Dissolved Solids
CCAL 13A.3

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Table of Contents

1.0	Scope and Application	3
2.0	Summary of Method	3
3.0	Definitions	3
4.0	Interferences	3
5.0	Safety	4
6.0	Equipment and Supplies.....	4
7.0	Reagents and Standards	4
7.1	Preparation of Standards	4
8.0	Sample Handling and Storage.....	5
9.0	Quality Control	5
10.0	Calibration and Standardization	5
11.0	Procedure	5
11.1	Beaker Preparation.....	5
11.2	Sample Setup.....	6
11.3	Procedural Notes	6
12.0	Data Analysis and Calculations	7
13.0	Method Performance	7
14.0	Pollution Prevention	7
15.0	Waste Management.....	8
16.0	References	8
17.0	Tables, Diagrams, Flowcharts, and Validation Data.....	8
17.1	Total and Total Dissolved Solids Summary Results Data Sheet	8
17.2	Total and Total Dissolved Solids Weights Record Data Sheet.....	9
18.0	Document Revision History.....	10

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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 180°C 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 prolonged drying, followed by proper desiccation and rapid weighing.
- 4.2 Samples high in bicarbonate require prolonged 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 5°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
- 6.6 100 mL graduated cylinder
- 6.7 GF/F or GF/C filter papers
- 6.8 Stainless steel screen with 1 mm² 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 80°C) 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 $\pm 2\%$.

11.0 Procedure

11.1 Beaker Preparation

11.1.1 Place prenumbered beakers into a 180°C 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.

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 Immediately 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.

- 11.1.7 After all beakers have been weighed, transcribe the lowest weight for that beaker from the Weights Record data sheet to the tare column of the Summary Results data sheet for that beaker.

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, blank or standard 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 180°C 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.
- 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 weights for that beaker from the Weights Record data sheet to the tare column of the Summary Results data sheet for that beaker.
- 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 initial weight. If the QA weight does not fall to within ± 0.5 mg of the final initial weight, then the beakers must be reweighed in reverse order from the out-of-QA-limit beaker until measurements fall to within ± 0.5 mg of the final initial weight. Reweighed beakers must again be QA checked and meet the above criteria or be reweighed until QA criteria are met.

- 11.3.5 It is especially important that as little time as possible elapse between initial weighing and QA weighing.
- 11.3.6 If multiple balances are being used use the same balance for the tare and loaded measurements to minimize variability.
- 11.3.7 If dissolved solids fraction is low (less than 1 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 180°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.

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 _____ Log # _____ Analyst _____

Dates: Beakers Weighed _____ Samples Setup _____ Final Weighing _____

Remarks: _____

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

Total Dissolved Solids Weights Record

Tare Tare + Sediment (circle one)

Log # _____

Analyst _____

Date _____

Beaker #	Weighing #					Zero / QA Check
	1	2	3	4	5	
	6	7	8	9	10	

18.0 Document Revision History

Original Document: March 2006

Version: 13A.0

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