

Standard Operating Procedure for the
Determination of Total Dissolved Solids
CCAL 13A.0

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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	3
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	7
12.0	Data Analysis and Calculations	7
13.0	Method Performance	7
14.0	Pollution Prevention	7
15.0	Waste Management	7
16.0	References	8
17.0	Tables, Diagrams, Flowcharts, and Validation Data	8
17.1	Total Dissolved Solids Summary Results Data Sheet.....	8
17.2	Total Dissolved Solids Weights Record Data Sheet.....	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. The detection limit for this determination is 5 mg/L filterable solids.

2.0 Summary of Method

- 2.1 A thoroughly mixed sample is filtered and the filtrate evaporated to dryness in a tared beaker. The gain in weight represents the 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.*

- 16.1 150 mL Griffin beakers (glass)
- 16.2 Drying oven equipped with thermostatic control capable of maintaining temperature within 2°C range
- 16.3 Desiccator - with moisture indicating desiccant
- 16.4 Analytical balance - capable of weighing to 0.1 mg
- 16.5 Filtration system - filter funnel, filter stage, filter barrel, clamps, erlenmeyer filter flask
- 16.6 100 mL graduated cylinder
- 16.7 GF/F or GF/C filter papers
- 16.8 Stainless steel screen with 1 mm² mesh.
- 16.9 Large porcelain buchner funnel.
- 16.10 Vacuum system and connecting hoses.

7.0 Reagents and Standards

- 7.1 Preparation of Standards
 - 7.1.1 *1000 ppm NaCl Solution:*
Dissolve 1 g NaCl (oven-dried at 80°) into 1000 mL of DI water and mix until dissolution is complete.

8.0 Sample Handling and Storage

8.1 Unfiltered 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: 1 ppm NaCl check standards 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.

a) Obtain gross tare weight from the list of beaker weights (located on the wall next to the balance).

b) Set balance adjustments to tare weight values for the current beaker and weigh the beaker.

c) Record weight on the Weight Record data sheet.

d) Make multiple measurements to assure a constant weight.

- e) Switch the balance off but leave the beaker on the balance pan.
 - f) After 90 seconds read the weight again, and record results.
 - g) Repeat weighing procedure until three consecutive measurements match to within 0.2 mg, or until weight increases from previous weighing. Usually the beakers tend to lose weight.
 - h) Recheck balance zero after obtaining a final weight for the first beaker, the fifth beaker and the tenth beaker.
- 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. Using steps above, the QA weight is written in red on the right side of the constant weight data sheet along with the initials of the person performing the QA check. Multiple measurements are not necessary for the QA weight. 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. It is especially important that as little time as possible elapse between initial weighing and QA weighing.
- 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 filtered 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. If multiple balances are being used use the same balance for the tare and loaded measurements to minimize variability.
- 11.2.7 The difference between the loaded weight and the tare weight equals the weight of the dissolved solids in 100 mL of sample.

11.2.8 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 Procedural Notes

11.3.1 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.

12.0 Data Analysis and Calculations

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

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

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: 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 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 Dissolved Solids Summary Results Data Sheet

