Illinois State Water Survey
Health and Environmental Applications Laboratory

Standard Operating Procedure
For
Total Dissolved Solids

(based on SM 2540C 2012, 22th ed.)

SOP Number: AN.HEAL.MN.TDS.3.5

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Prepared by: _____ Date: 12/17/2024
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The following individuals (in addition to those listed on the cover) have reviewed this SOP.

Name (printed)	Signature	Date
Anthony Kilber	Anthony Kilber	12/19/2024
Tatyana Grandt	Tatyana Grandt	01/02/2025

Revision History

Beginning Revision	Ending Revision	Revision Date	Changes
2.3	3.0	1/8/20	Reformatted and updated previous version, incorporated additional details from SM2540C
3.1	3.2	12/21/21	added a note about brine sample volume and keeping residue to ≤0.2g, section 11.5.9. Updated room numbers and wording for clarity.
3.2	3.3	12/6/22	Rearranged section 11 to make analytical flow more accurate to lab process. Added solids preweigh file info and requirement to have 2 consecutive weights before analysis. Clarified dish washing procedure. Added note to leave desiccators closed while weighing.
3.3	3.4	1/16/24	Updated reagent section to include concentrations of TDS for quality control solutions. Updated QC section to include a summary table and updated duplicate acceptance criteria. Fixed numbering. Incorporated lab comments.
3.4	3.5	7/27/24	Updated equipment info, MDL concentration.
3.5	3.6	10/8/24	Added sample storage details in section 11.

Table of Contents

1.0 Scope and Applicability

1.1 Solids refer to matter suspended or dissolved in water or wastewater. Total dissolved solids (TDS) are the portion that pass through a filter of 2 µm or less nominal pore size. This method is applicable to samples with a TDS concentration of 0 to 20,000 mg/L. The Method Detection Limit (MDL) is 8.6 mg/L.

2.1 A well-mixed, 100 mL volume of sample is filtered through a 0.45 µm nitrocellulose filter and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 180°C. The increase in dish weight represents the total dissolved solids.

2.2 The filtrate from the total suspended solids determination (Standard Methods 2540D) may be used for determination of total dissolved solids.

2.3 Waters with high dissolved solids generally are of inferior palatability and may induce an unfavorable physiological reaction. For these reasons, a limit of 500 mg/L is desirable for drinking water.

2.4 Highly mineralized waters are also unsuitable for many industrial applications.

3.0 Definitions

3.1 Consult the HEAL QAP for a list of standard terms and definitions associated with this SOP

HEAL	Health and Environmental Applications Laboratory
ISWS	Illinois State Water Survey
MDL	Method Detection Limit
SOP	Standard Operating Procedure
TDS	Total Dissolved Solids

4.0 Safety Warnings and Waste Management

4.1 Always wear eye protection in the laboratory.

4.2 Food, drinks, and smoking are not allowed in the laboratory.

4.3 Safety Data Sheets (SDS) applicable to this SOP can be found online by using the University of Illinois Division of Research Safety (DRS) website: https://www.drs.illinois.edu/Programs/SafetyDataSheets.

4.4 The Illinois State Water Survey Chemical Hygiene Plan covers the ISWS laboratory safety program, including, but not limited to, personal protective equipment used, control equipment inventory and operations (such as vented hoods), employee training programs, medical programs, and safety. The ISWS Chemical Hygiene Plan is available at https://go.illinois.edu/ISWS-Chemical-Hygiene-Plan.

4.5 The University of Illinois DRS has a laboratory safety guide available at https://www.drs.illinois.edu/site-documents/LaboratorySafetyGuide.pdf. The ISWS has their own laboratory safety manual, available at https://go.illinois.edu/ISWS-Laboratory-Safety-Manual.

4.6 The HEAL practices pollution prevention, which encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. The quantity of chemicals purchased should be based on the expected usage during its shelf life and disposal cost of unused material.

4.7 Laboratory waste management practices must be consistent with all applicable rules and regulations. Excess reagents and samples and method process wastes should be characterized and disposed of by DRS. It is the responsibility of the user of this method to comply with relevant disposal and waste regulations.

4.8 The HEAL has listed known health and safety warnings for this SOP, but this list should not be assumed to comprise all health and safety issues.

5.0 Cautions

None.

6.0 Interferences

6.1 Highly mineralized waters with considerable calcium, magnesium, chloride, and/ or sulfate content may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.

6.2 Samples high in bicarbonate require careful and possibly prolonged drying at 180°C to insure complete conversion of bicarbonate to carbonate.

6.3 Brines that exceed the range tend to “creep” out of the dish while drying, so the sample volume should be limited to 1 mL or less in these instances.

6.4 Excessive solids in the dish can form a crust while drying, trapping water and prolonging drying time.

6.5 The desiccant inside the desiccators (Drierite) can become exhausted and will no longer hold water. The indicating Drierite is blue when active but becomes pink when exhausted. When the stones start to show a pink color, they can be regenerated by heating them at 210°C for 1 hour in a drying oven. Be sure to use an oven safe pan and keep the Drierite depth to a 1 granule layer, to allow all the stones to dry completely. If the stones do not change color, then discard them.

7.0 Personnel Qualifications

7.1 Analysts will be trained for a minimum of one week by an experienced analyst before performing routine analysis. An initial demonstration of capability is also required before routine analysis (HEAL QAP).

8.0 Equipment and Supplies

8.1 Equipment

8.1.1 Evaporating dishes, 100 mL capacity, glass (Pyrex), 80 mm diameter

8.1.2 Boekel Water bath

8.1.3 Forceps with flat tips, so they don't poke holes in filters

8.1.4 Desiccator with a desiccant containing a color indicator of moisture concentration.

8.1.5 Drying oven for operation at 180°C.

8.1.6 Electronic analytical balance: Mettler-Toledo AX304 or equivalent, capable of weighing to 0.1 mg (Max 310 g), connected to a computer with LabX software installed.

8.1.7 Graduated cylinder, 100 mL capacity, TC/TD.

8.1.8 Calibrated check weights for the analytical balance

8.1.9 Filtration apparatus, with reservoir and filter support.

8.1.10 Adjustable pipettes

8.1.11 Nitrocellulose fiber filter disks, Millipore Type HA, or equivalent, 0.45 µm pore size, 47 mm diameter.

8.2 Reagents and Solutions

8.2.1 Only high-purity reagents that conform to the American Chemical Society specifications (AR grade or better) should be used. NIST traceable stock standards may be purchased from reputable commercial suppliers. As an alternative, they may be prepared from ACS reagent grade materials (dried at 105°C for 30 min) and verified with NIST materials.

8.2.2 TDS stock: Sodium Chloride (NaCl): 30,300 mg/L (50 mS/cm), for spiking the LFB and LFM. Weigh 30.30 g of NaCl, previously dried overnight in an oven at 180°C and dilute to 1 L mark in a volumetric flask with deionized water.

8.2.3 MDL verification sample: prepare an MDL verification sample using the same concentration as the MDL spike that was used to determine the initial MDL. Adjust spiking concentration if MDL changes.

8.2.4 LRB (0 mg/L): use fresh deionized water with resistivity of at least 18.2 Mohms

8.2.5 LFB (200 mg/L): add 660 µL of TDS stock to 100 mL deionized water.

8.2.6 LFM (+1500 mg/L): add 4950 µL of TDS stock to 100 mL sample.

8.2.7 MDL solution (70 mg/L): Add 230 µL of TDS stock to 100 mL deionized water.

9.0 Instrument Setup and Calibration

9.1 Balance calibration should be tested daily by the analyst, using at least 3 weights traceable to NIST calibration/certification. If the scale is going to be calibrated, perform this before the sample analysis.

10.0 Sample Collection, Preservation, and Storage

10.1 All sample collection bottles must be thoroughly cleaned and rinsed with reagent water. Unfiltered samples should be collected in 500 mL HDPE bottles (or larger) supplied by the laboratory. Previously filtered samples may be collected in 250 mL bottles. This volume is sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

10.2 Ship and store samples at 4°C until the time of analysis to minimize microbiological decomposition of solids.

10.3 Begin analysis as soon as possible after sample receipt because of the impracticality of preserving the sample. The sample holding time is 7 days after collection. If the holding time is exceeded, it must be documented with the data.

10.4 Allow sample to reach room temperature before analysis. It is best to take the samples out of the refrigerator early in the morning to allow sufficient time for them to warm up.

11.0 Sample Preparation and Analysis

11.1 Use the Lab-X software to check the calibration of the scale.

11.1.1 Open the Lab-X software. If the quick start wizard opens, click next twice to proceed to step 3 of 4. Do not change any default settings on step 2.

11.1.2 Use the dropdown menu titled “Layout Name” to select the proper format for the task at hand. Usually this is either “TDS or TSS”, or “Balance Calibration Checks”. Don’t change any other settings and then click next to proceed to step 4.

11.1.2 Use the dropdown menu to select the file or application you are transferring data into (usually this is Excel or a specific spreadsheet). There is also the option to use the cursor location to enter the data, which works the same way.

11.1.3 Place 50 g weight on the balance. Right-click on ‘50’ in the weight column, then left click to select balance. When the circle disappears from balance read out, click out of the ‘50’ box to save the reading.

11.1.4 If the scale needs to be re-calibrated, it should be done before analysis, not during.

11.2 Sample Preparation: Filtering

11.2.1 Preparation of membrane filter disk

Insert filter disk into filtration apparatus with forceps, wrinkle side up. (Discard paper divider packaged with filters). Apply vacuum and wash disk with 50 to 100 mL of reagent-grade water. Continue suction to remove all traces of water. Discard washings.

11.2.2 Pour the raw sample through a washed filter with applied vacuum. Collect filtrate in a clean container.

11.2.3 If analysis is not going to be performed immediately, store the filtrate in a sealed container at 4°C for up to 7 days.

11.3 Preparation of evaporating dish

11.3.1 Wash dishes with laboratory cleaner and rinse thoroughly with reagent-grade water. Dishes can be dried on the countertop or placed into the oven while still wet.

11.3.2 Heat clean dishes at 180 ± 2°C for at least 1 h in an oven. Usually, they are heated for 4 hours or overnight. It is important to keep the heating times consistent throughout the analytical process.

11.3.3 Allow dishes to cool completely (at least two hours) in a desiccator until needed. It is best to leave the desiccator(s) in the same room as the analytical balance to reduce the chances of temperature changes affecting the weights. Similar to heating, it is best to keep the desiccation time consistent.

11.3.4 Open the “SolidsPreWeigh” excel spreadsheet the file in “\\pri-fs1\HEAL\Data\Lab Templates”. Enter dish IDs in the spreadsheet, making sure to include enough dishes for all samples and duplicates/QCs. The dish ID numbers will be matched up with sample IDs later.

11.3.5 Weigh each dish to four decimal places and record weights in the SolidsPreWeigh file. Keep a running list of dishes prepared for analysis in this file (no need to make a new file for each analysis).

NOTE: When using glass desiccators, it is best to keep the lids on between weighing each dish. This will prevent humid air from affecting the weights of the dishes in the desiccator and will prolong the life of the desiccant.

11.3.6 Repeat drying, desiccating and weighing the dishes, until obtaining two consecutive weights that are consistent (difference is less than 0.0005 g or 4%, whichever is less). The SolidsPreWeigh excel sheet includes logic to check the weights and will display a flag in the “passed” column as either ‘pass’ or ‘fail’. When two weights are consistent, then the dish is ready for analysis.

11.3.7 Store dishes in a desiccator until ready to use.

11.4 Sample Analysis

11.4.1 Turn on the steam bath, turn on the water, and let it drip. This takes a while to heat up,

so it's best to get this started early.

11.4.2 Retrieve samples from the cooler and let them warm up to room temperature before starting analysis.

11.4.3 Create an excel spreadsheet by opening the TDS template file in "\\pri-fs1\HEAL\Data\Lab Templates" and saving a new copy with a filename that includes the analysis date.

11.4.4 Use the SolidsPreWeigh spreadsheet to record the final passing pre-analysis weight for each dish. Then, copy passing weights and dish numbers into the analytical template named "TDS_Template". These weights are the "dish date weight" (cells colored in dark green). Be sure to use enough dishes for all samples and QCs. Rename template with the analysis date.

11.4.5 Create a Prep Batch and Analytical Batch in LIMs. Copy an Excel file from LIMs to the TDS working spreadsheet. Each sample should have a corresponding 'passing' dish number assigned to it at this point.

11.4.6 Transfer the dishes to the water bath, if not already done.

11.4.7 Selection of sample size: Use a sample volume of 100 mL. If the amount of sample available is limited, use a volume of 50 mL (or smaller). For brines, use a sample volume of 0.5 to 1.0 mL.

NOTE: If sample volume is changed, be sure to update the TDS template file for the analysis to ensure the proper value is calculated.

11.4.8 Begin each run with the following quality control samples: MDL ongoing verification, LRB, and LFB. Prepare and analyze one LFM in 10% of the samples. Analyze a duplicate every 10 samples.

11.4.9 Verify calibration on the scale by weighing the check weights (50g, 20g, 10g, 1g, 0.02g) and recording the results on the excel worksheet using the LabX software.

11.4.10 Shake sample and pour 50 mL into a 100 mL graduated cylinder, then shake again and pour another 50 mL into the same cylinder for 100 mL total volume. Check sample ID number and dish number, then pour the sample into the dish matching the sample number. Repeat for each sample.

NOTE: Brine samples typically yield a residue of greater than 0.2g when using 100mL volume, which can cause inconsistent weights during the drying process. For this reason, brine samples should use a maximum of **1mL of sample**. Use a pipette to measure 0.5 to 1.0 mL of sample. Be sure to rinse the pipette tip three times by aspirating deionized water and dispensing this rinsate into the sample vessel.

11.4.11 If sample quantity exceeds open slots in the steam bath, then rotate samples every hour until the liquid is completely evaporated.

11.4.12 Once samples are completely evaporated, turn off the water bath and transfer the dishes into the 180°C oven. Let them dry overnight.

11.4.13 The next morning, take the samples out of the oven and place in the desiccator for two hours before weighing.

11.4.14 Verify the calibration on the scale by weighing the check weights (50g, 20g, 10g, 1g, 0.02g) and recording the results with LabX.

11.4.15 Use the excel spreadsheet that was created earlier for TDS samples to record sample weights.

11.4.16 Repeat drying, desiccating and weighing following the steps above (11.4.12 to 11.4.16) until obtaining two consecutive weights (within 0.0005g or 4%, whichever is less). The spreadsheet will check this for you and indicate if the weights are passing. If they are, then analysis is complete. If the weight difference exceeds 0.0005g or 4%, then another cycle of drying and weighing is needed.

11.4.17 Transfer all the completed samples into a tray labeled with the sample ID and store covered until QA is approved.

12.0 Troubleshooting

12.1 If sample weights are inconsistent, the dish should be re-dried and re-weighed. Desiccant and ambient conditions should be considered when weights fluctuate over time. Use a dehumidifier when the laboratory is humid, particularly in the summer. Re-dry desiccator stones when necessary.

12.2 Use good weighing practices to ensure that interferences are reduced: do not lean on the counter while weighing, do not leave windows open, ensure there are no excessive air currents in the room, weigh at a time when temperature is consistent, and check the level bubble on the balance to make sure it is level.

12.3 Sample residue should be between 0.01 to 0.2g. This is particularly important for brine samples, as they can produce residue in excess of 1g, which causes water to be trapped under a crust and will cause inconsistent weights.

13.0 Data and Record Management

13.1 Calculate the concentration of TDS at each drying temperature as follows, and report in whole numbers (Note: this is automatically calculated by the TDS excel spreadsheet):

$$\text{total dissolved solids, mg/L} = \frac{(A - B) \times 1000}{\text{sample volume, mL}}$$

where: A = weight of dried residue + dish, mg
B = weight of dish, mg

13.2 Upload analytical file in LIMs and attach a copy of it to the analytical file for the QA Officer for review. Complete Prep Batch in LIMs.

14.0 Quality Control and Quality Assurance

14.1 Laboratory Reagent Blank

14.1.1 One laboratory reagent blank (LRB) shall be analyzed with each preparation batch. The LRB must be less than the MDL (8. mg/L).

14.2 Laboratory Fortified Blank

14.2.1 One laboratory fortified blank (LFB) is analyzed with each preparation batch. The acceptance criteria for LFB's are 85 to 115%.

14.3 Laboratory Fortified Sample Matrix

14.3.1 In 10% of the samples, a laboratory fortified sample matrix (LFM) must be analyzed. The original (not fortified) sample must be run in addition to the fortified sample. The analyte recovery of the LFM must be within 75 to 125%.

14.4 Duplicates

14.4.1 A duplicate must be analyzed at least once per 10 samples in a preparation batch (minimum of one duplicate per preparation batch). Duplicates should agree within 10% relative percentage difference (RPD) when the concentration is >10*MDL. For concentrations <10*MDL, the acceptance limit is ±MDL.

$$RPD = \left(\frac{|C_2 - C_1|}{\frac{C1 + C2}{2}} \right) * 100$$

14.5 MDL ongoing verification sample -- Analyze one MDL verification sample per month, made from separate batches. The MDL ongoing verification sample should be at the same concentration that was used to establish the MDL. There are no acceptance criteria for this sample.

14.6 Corrective Action

14.6.1 Further corrective actions for nonconforming quality control samples are given in the Corrective Action Policy (AD.HEAL.0.CorrectiveAction).

Table 14-1. Summary of quality control samples and acceptance criteria.

QC Type	Frequency	Acceptance Criteria
LRB	1 every batch	<8.6 mg/L
LFB	1 every batch	200 mg/L ± 85-115%
LFM	10% of samples in prep batch	± 75-125%
Duplicate	Every 10 samples in prep batch	±10% when >10*MDL

15.0 References

15.1 American Public Health Association, American Water Works Association, Water Environment Federation. (2012). Total Dissolved Solids Dried at 180°C, Method 2540 C. In A. Eaton, L. Clesceri, and A. Greenberg (Eds.), Standard Methods for the Examination of Water and Wastewater (22th ed.). Washington, D.C.: American Public Health Association.

15.2 Definition and Procedure for the Determination of the Method Detection Limit – Revision 1.11, In *Code of Federal Regulations 40*, Chapter 1, Part 136, Appendix B.

15.3 Rea, E. 2023. Health and Environmental Applications Laboratory Quality Assurance Plan. Illinois State Water Survey. Champaign, IL.

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