TOTAL DISSOLVED SOLIDS

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1. SCOPE AND APPLICATION

Total dissolved solids (TDS) is defined as the portion of solids that passes through a filter of 2.0 microns nominal pore size. This method is applicable to drinking, surface, and saline waters. The practical range of determination is from the method detection limit (MDL) 15 mg/L to 20,000 mg/L. For very high salinity samples the range can be extended by adjusting the sample volume according to Table 1 in section 6.10. The practical quantitation limit (PQL) is determined by the concentration at which 2.5 mg of residue remain in the crucible as required by the method. For a sample volume of 100 mL, this is 25 mg/L. The test covered by this SOP is W-TDS.

2. SUMMARY OF METHOD

2.1. This method is based on Standard Methods 2540 C-1997.

2.2. A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is then poured into a pre-weighed evaporating dish and evaporated to constant weight at 180 °C. The evaporating dish is then cooled in a desiccator, weighed and the results used to compute the TDS of the sample.

Note: The suspended matter left behind on the filter may be used for a total suspended solids (TSS) determination. See DEP SOP NU-051.

2.3. Interferences - highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing. Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180 °C to insure that all the bicarbonate is converted to carbonate. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

3. EQUIPMENT AND SUPPLIES

3.1. Glass Fiber Filters (Whatman G6: 1.5 micron pore size, 5.5 cm in diameter. If TSS is also being performed, Environmental Express ProWeigh™ 47 mm glass fiber filters may be used for the filtration)

3.2. Filter holder with a coarse fritted disc as a filter support and membrane filter funnel

3.3. Desiccators

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3.4. Evaporating dishes, porcelain, 100 mL and 130 mL volume, numbered individually in the lab for identification purposes.

3.5. Laboratory ovens that can be set at 95 °C and 180 °C.

3.6. Dish tongs

3.7. Analytical balance (capable of weighing to 0.1 mg)

3.8. Class A 100 mL, 50 mL and 25 mL graduated cylinders (or 250 mL graduated cylinder if TSS is also determined for the same sample)

3.9. Eppendorf pipettes

3.10. Vacuum pump

3.11. Digital thermometer, calibrated at points bracketing the range of use, with min/max function

4. REAGENTS AND STANDARDS (SEE DEP SOPS NU-041 and NU-042)

Note: Before preparing reagents or standards, or running samples, please read the safety notes in the Safety/Hazardous Waste Management section of this SOP and the MSDS for the chemicals used in this analysis.

Note: Standard preparations must be documented in the Laboratory Information Management System (LIMS) 'Standard Preparation Tracker' module. Unique ID numbers for each standard are generated in the module and documented in the TDS excel worksheet submitted with each analysis folder. A Certificate of Analysis for each purchased standard is maintained in a binder in C307. All containers holding standards must be labeled with their unique ID and expiration date.

4.1. Deionized (DI) water: for blanks (MDL and MBLKs), and washing/rinsing.

4.2. Desiccant - a mixture of blue (97 % $CaSO_4$ and 3 % $CoCl_2$ -size 8 mesh) and white (100 % $CaSO_4$ -size 10-20 mesh) desiccant.

4.2.1. A mixture of approximately 10 % blue and 90 % white desiccant should be placed in the desiccator used to store filters and dishes. The desiccator is used to store and transport filters and dishes before weighing.

4.2.2. Once the desiccant has turned from blue to pink (indicating water absorption), heat the desiccant in a 180 °C oven for one hour, let cool and

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reuse. Desiccant may be recharged 3 times then replace with new desiccant and discard the old desiccant in the trash.

4.3. Nitric Acid Solution - fill up a designated container with DI water and add 100 mL of nitric acid for acid bath. Allow the crucible to soak at least overnight then scrub with cleaning pad and wash 4-5 times with DI water.

4.4. A certified quality control sample (AltQC) prepared following the supplier's instructions. See the certificate of analysis for the concentration and expiration date of the purchased standard. The purchased and prepared standards must be recorded in the standard preparation tracker in the Laboratory Information Management System (LIMS). Holding time of the prepared standard is180 days.

5. SAMPLE HANDLING AND PRESERVATION

5.1. Samples are stored on ice or in a refrigerator at ≤ 6 °C (not frozen) in order to minimize microbiological decomposition of solids.

5.2. Non-representative particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.

5.3. Samples are stored in the Nutrient refrigerator in the receiving room of the DEP laboratory. Allow samples to come to room temperature before processing.

5.4. Samples should be analyzed within seven days of collection. Samples analyzed after this seven day holding time must be reported with a test comment describing the nature of the delay as well as the appropriate qualifying code, Q.

6. SAMPLE ANALYSIS/QUANTITATION (SEE DEP SOP NU-033)

Note: Be sure to record all of the necessary information neatly and correctly on the TDS Excel worksheet. See Appendices B and C for example cover page and worksheet. The worksheet is located at \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\Solids\. The TDS results are automatically calculated on the worksheet.
Important: The worksheet contains functional formatting to indicate unacceptable results – do not copy/paste or "click and drag" any information in the worksheet.

Note: Do not handle the dishes with your hands. Always use tongs when handling the dishes.

Note: All ovens are kept at a constant temperature. The minimum and maximum (MIN/MAX) oven temperatures are recorded for each drying cycle of the analysis on the Oven

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Temperature Log Sheet (see Appendix A). The log sheet is located at \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\Solids\.
To use the MIN/MAX function on the digital thermometer, see the manufacturer directions located on the door of each oven. Record the START time of the drying cycle as the time when the oven has reached its set temperature, and the MIN/MAX function on the thermometer has been activated. After the MIN/MAX settings have been checked and recorded to verify the oven was in compliance, record the STOP time for the drying cycle as the time when samples are removed from the oven. If an oven temperature is found to be out of compliance, an explanation and corrective action is recorded on the log sheet, and another drying cycle is monitored. The oven temperature log sheet is submitted with each analytical folder. Make sure that whenever samples are in the oven at least one temperature reading is recorded every day or the Min/Max temperature is recorded over an extended period of time. If samples are in the oven over the weekend the MIN/MAX temperature is recorded as well.

6.1. If a programmable oven is available, then set the oven at its correct temperature to initiate its temperature ramp (initial temperature for 95 °C and a ramp to 180 °C). If the programmable oven is not available, then set one laboratory oven at a temperature of 95 °C and a second at 180 °C. It will take about 1 hour for the ovens to reach their set temperatures.

6.2. To clean the evaporating dishes for use, wash the evaporating dishes 5-6 times with hot tap water and rinse 2 times with DI water and then follow section 4.3.

6.3. If the dish number is fading, re-write the number using a permanent marker. If the dish number is not clear, assign the dish the next number using the information contained in the crucible logbook.

6.4. Place the dishes in a 180 °C oven for at least 4 hours to dry and remove any trapped water in the porcelain. You can dry overnight if it is convenient.

6.5. Remove the dishes from the oven with dish tongs and place the dishes in desiccators to cool. Clean dishes are stored in the desiccators until they are needed. If clean dishes are needed immediately, then allow them to cool for at least 2 hours prior to weighing. More time will be needed if there are a large number of hot dishes in the desiccators. Do not weigh any dish that is still hot.

6.6. Check the calibration of the analytical balance using the certified weights in the laboratory and document the work in the balance maintenance logbook. If the balance passes the calibration check, continue with the operation. If not, recalibrate the balance with the certified weights following the manufacturer's instructions and recheck the calibration. Record your actions in the balance maintenance logbook. If after the first calibration attempt the balance does not seem to function properly, inform your supervisor immediately and record your

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observation in the balance maintenance logbook. Do not use this balance until the calibration problem has been corrected.

6.7. After verifying the calibration of the balance, open desiccators, remove the first clean dish to be weighed (using tongs) and close the desiccator door immediately. Place the dish on the analytical balance and close the balance door. Allow the balance to stabilize before taking a reading. This is the initial or TARE weight of the first dish. Repeat this process for each dish that is needed.

6.8. Check to see if your samples require TSS. If the TSS test is requested, then the filtrate from the TSS test can be used for TDS providing you follow the modification stated here. Please familiarize yourself with DEP SOP NU-051 prior to combining these tests.

An example run sequence includes:

- MDL (DI water, 4.1.)
- ALTQC (4.4.)
- Batched samples, including an MBLK (DI water, 4.1.) and one sample run in duplicate for each batch
- Blank (DI water, 4.1.)

6.9. Assemble the vacuum filtration flask by attaching the filtration flask to the filter holder. Connect the flask to the vacuum.

6.9.1. TDS ONLY - Place a glass fiber filter onto the filter holder. Turn on vacuum and rinse the filter with three aliquots of DI water (about 20 mL each). This is to remove any water-soluble impurities that may reside on the glass filters and bias the TDS residue high. Turn off vacuum, disconnect flask and discard the rinse water. Dry the flask by shaking out as much of the water as possible and reattach to filter assembly.

6.9.2. FOR TSS/TDS - For samples above 15000 umhos/cm, refer to section 6.3.2. of DEP SOP NU-051. For samples below 15000 umhos/cm, place a ProWeigh brand filter (see 3.1.) onto the filter holder and attach the membrane filter funnel over the filter. Thoroughly wet the filter with DI water and apply vacuum. When all water has passed through the filter, turn off the vacuum, remove the filter flask, pour out the rinse water, and re-attach the flask. Make sure the interior of the flask is as dry as possible.

6.9.3. FOR TSS/TDS - Turn on the vacuum. Vigorously shake the sample and rapidly transfer 250 mL of sample to a graduated cylinder. Pour the contents of the cylinder onto the filter and collect the filtrate in the flask. <u>DO NOT rinse the graduated cylinder at this stage</u>.

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Note: If it is not possible to filter the amount of sample required for TDS (see Table below) or 250 ml for TSS due to particulate matter, determine the sample volume you are able to filter and start over at step 6.9.1 through 6.9.3.

6.10. If the field conductivity of the sample is greater than about 3000 umhos/cm, there may be too much residue in the dish for the TDS measurement. The method recommends that the total residue should not be more than 200 mg. If there is more than 200 mg, it may be difficult to dry to a constant weight (especially if it is a saltwater). Using Table 1, determine how much sample volume you will need to perform TDS. For volumes of 25 mL and above, use a measuring cylinder. For volumes of 10 mL and below, use an Eppendorf pipette.

Table 1

If the field conductivity (umhos/cm) is:	Measure out this amount for TDS:
0 – 3000	100 mL
3001 – 5000	50 mL
5001 – 10,000	25 mL
10,001 – 18,000	10 mL
18,001 – 25,000	5 mL
25,001 – 30,000	3 mL
> 30,000	2 mL

6.11. FOR TDS ONLY - Turn on the vacuum. Vigorously shake the sample and rapidly but ACCURATELY transfer a volume of sample according to Table 1 onto the filter and collect the filtrate in the flask. You may rinse the cylinder (if used) with a few squirts of DI water and pour those onto the filter. Do not add too much water.

6.12. FOR TDS ONLY - After the sample has passed through the filter, continue to apply the vacuum for approximately 30 seconds to remove as much water as possible from the glass filter. Pour the contents of the filter flask into a drying dish. You may rinse the flask with a few squirts of DI water.

6.13. FOR TSS/TDS - Turn off the vacuum and remove the flask. Transfer the recommended amount of filtrate into a drying dish (see Table 1).

6.14. FOR TSS/TDS - Re-attach the flask and turn on the vacuum. Rinse the filter with DI water (approx. 3 x 30 mL) to remove any sample that remains on the filter and on the sides of the filtering apparatus. (See DEP SOP NU-051 for details on rinse procedure for high conductivity samples).

6.15. FOR TSS/TDS - Continue to apply the vacuum for 2-3 minutes after the sample has completely passed through and the filter has been rinsed, to assure that all

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traces of water have been removed. Discard the filtrate and remove the filter for TSS analysis (See DEP SOP NU-051 for details).

6.16. Carefully place the dishes in a 95 °C oven to allow the sample to evaporate to dryness. This takes approximately 18 hours. DO NOT spill ANY sample. If any sample is lost, you will need to discard the sample and filter a new aliquot.

6.17. After 18 hours, transfer the dishes from the 95 °C oven to the 180 °C. Dry the dish for at least 1 hour at 180 ± 2 ºC. See the **Note** above regarding documentation of drying cycles on the Oven Temperature Log Sheet.

6.18. Check the desiccant in the desiccators for blue color. Change the desiccant if it is pink. See Section 4.2.

6.19. Remove the dishes from the oven and place in the desiccators to cool for at least 2 hours. DO NOT weigh any dishes that are still hot. You may leave dishes in the desiccators overnight.

6.20. Weigh the cooled dishes using the same analytical balance that was used to tare the dishes. Do not recalibrate the balance between weighings. Record the weight on the TDS worksheet under Crucible and Residue Weight (g): 1st.

6.21. Return the dishes to the 180 ± 2 °C oven for 1-2 hours to repeat drying cycle. Return the dishes to the desiccators and cool.

6.22. Weigh the cooled dishes again and record this second weight on the TDS worksheet under Crucible and Residue Weight (g): 2nd. If the two weights have a difference of less than 0.5 mg no more drying/weighing cycles are required. If not, return to the drying oven for a third time and repeat the weighing process. Record third weight on the TDS worksheet under Crucible and Residue Weight (g): 3rd. Continue the drying/weighing cycles recording the weight in the appropriate cell in the TDS worksheet until the last two weights differ by less than 0.5 mg. Do not perform more than six weighings. Samples with large amounts of calcium sulfate or salts are very difficult to dry and may require data qualifiers.

6.23. The TDS is calculated using the following equation:

$$\text{TDS (mg/L)} = \frac{\{[\text{final wt. of dried residue + dish (g)}] - [\text{wt. of dish (g)}]\} \times 1000000}{\text{Volume of sample (mL)}}$$

6.24. Save the TDS Excel file as a text file (.txt) and use the GenChem: TSS-TDS Balance–M310 driver or the GenChem: TSS-TDS Balance-XP204 driver to convert the text file into QC Manager. Check that the number of significant figures has been set correctly.

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6.25. Place the completed TDS worksheets along with the standard sheet in a folder labeled YYMMDD**M**D (for the Denver Analytical M310 balance) or YYMMDD**X**D (for the Mettler Toledo XP204 balance), where YY is the year, MM is the month, DD is the day of the analyses, M or X identifies the balance, and D indicates a TDS analysis. Initial the folder and hand to a supervisor for review.

7. DATA ARCHIVAL

7.1. The raw data is archived in paper format. A report folder is created for each analytical run. Occasionally more than one run may be in one folder. The folder is labeled with the test (W-TDS), filename/s, and initials of the analyst.

7.2. Each report folder should contain the following items - the TDS excel worksheet, bench sheet(s), the oven temperature log sheet, the backlog containing the batching documentation, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

7.3. The report folders are on file with the nutrients and general chemistry supervisory personnel. Periodically, the files are moved to the State Records Center for long time storage according to DEP SOP LB-006.

7.4. Excel transfer files for upload to QC manager are stored in electronic format in the current year's subfolder located at \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\Solids\.

8. QUALITY CONTROL/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES (SEE DEP SOPS NU-038 AND NU-043)

8.1. An MDL (4.1., analyzed at the beginning of every run), MBLK (4.1., analyzed at the beginning of every batch of ten samples or less), and the final blank on the run must be < 15 and > -15 mg/L in order to report the results for that batch without qualification. If the result is outside of this range, the blank and all associated samples may be rerun until the blank passes or the results may be qualified with an appropriate code and comment (refer to DEP SOP NU-043, Section 4.2.1.).

8.2. One set of sample duplicates per batch of ten samples or less. If the sample result is above the PQL (25 mg/L), the relative percent difference (RPD) must be less than 10 % to report the result without qualification. If the RPD is equal to or greater than 10 % the samples and duplicates may be rerun until a passing RPD is achieved. If the replicate sample result is below the PQL, no precision data is uploaded and an appropriate test comment is added to all samples in the batch.

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8.3. The AltQC (4.4.) is analyzed at the beginning of every run. Acceptance limits are provided by the manufacturer.

8.4. Sample results obtained based on a residue exceeding 200 mg must be confirmed using a smaller sample volume or qualified appropriately. It is recommended to pour an aliquot for confirmation as soon as the first weight indicates that the residue may exceed the limit.

8.5. For general method performance and acceptance criteria for QC measures and corrective action for out-of-control data and contingencies for handling out-of-control or unacceptable data, refer to DEP SOP NU-038 and DEP SOP NU-043.

8.6. For general calculations, including RPD, see Section 5.9 of the FDEP Quality Manual.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT

9.1. Safety glasses, gloves and lab coats should be worn at all times whilst handling samples. Excess samples may be flushed down the sink.

9.2. Review the Laboratory Health and Safety Plan (accessible on the intranet at http://depnet/burlabs/safety.htm).

10. REFERENCES/DEFINITIONS/VALIDATION DATA

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use.

10.1. Standard Methods Online, 2540C-1997.

10.2. DEP SOP NU-033, Summary of Sample Analysis Procedures for the General Chemistry and Nutrients Workgroups.

10.3. DEP SOP NU-038, Nutrients and General Chemistry: Quality Assurance and Quality Control.

10.4. DEP SOP NU-041, Reagent Preparation Procedure.

10.5. DEP SOP NU-042, Standard Preparation Procedure.

10.6. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines.

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10.7. DEP SOP NU-051, Total Suspended Solids.

10.8. Oven Manuals.

10.9. Quality Manual, Bureau of Laboratories, FDEP.

10.10. Laboratory Health and Safety Plan.

10.11. DEP SOP LB-006, Standard operating procedure for records maintenance and storage.

10.12. DEP SOP CM-011, Gravimetric Analysis: Analytical Weight and Balance Calibration.

Appendix of Significant Changes

03/07/2008	Batches changed from 20 samples to ten. Acceptance limits for duplicates changed to 20% from 10%.
06/03/2008	Reference method changed from 160.1 to SM 2540 C.
05/19/2011	The Practical Quantitation Limit (PQL) was changed from 60 mg/L to 25 mg/L. See Section 1 for details.
05/09/2012	Section 6 was edited to include modifications to the TSS analysis procedure put into practice on 05/21/2012.
05/07/2013	Section 4.4 was edited to correct the holding time of the ALTQC. Section 6.9.3 was further clarified. Section 7.2 was edited to correct the RPD control limit.
04/16/2015	Data archival information was clarified in section 7. Oven temperature recording procedure was modified in section 6.

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Appendix A:
Oven Temperature Log Sheet

FDEP Chemistry Laboratory – Temperature Log for Solids

DEP SOPs NU-047(TDS), NU-051 (TSS), and NU-099 (TS)

Oven ID #:

Filename: **Thermometer Calibration Date:**

Thermometer S/N: **Thermometer Expiration Date:**

For Total Dissolved Solids (TDS) analysis, the oven must be 180 ± 2 °C.
For Total Solids (TS)/Total Suspended Solids (TSS) analysis, the oven must be 103 - 105 °C.

Date	Analyst Initials	START Time	STOP Time	MIN temp	MAX temp	Oven in compliance? (Y/N)	Corrective action? (Y/N)	Comments

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Appendix B:
TDS Excel Worksheet Cover Page

TDS (TOTAL DISSOLVED SOLIDS) SM 2540 C (NU-047)

Residue, Filterable (Dried at 180 °C)

Instrument: Denver Instruments Balance M-310 Serial # P119505

OVENS: 180 +/- 2 °C:

95 °C: Oven # 10 S/N 1580070237078

Primary Analyst:	**Secondary Analyst:**
Date:	**File Name:**
Start Time:	
End Time:	

ALTQC##	**Conc:**	**mg/L**	**ALTQC## S/N:**	**Exp Date:**
	Upper Limit:	**mg/L**		
	Lower Limit:	**mg/L**		

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Appendix C:
TDS Excel Worksheet

File Name: 0

TDS (NU-047)

Analyst: 0 **Date:** 01/00/00 **Start Time:** 0:00 **End Time:** 0:00

Sample ID	Crucible ID	Sample Volume (mL)	Crucible Weight (g)	Crucible and Residue Weight (g)					TDS (mg/L)	Conductivity (umhos/cm)
				1st	2nd	3rd	4th	5th		
MDL		100								
ALTQC##_2		100								
MBLK		100								
		100								
		100								
		100								
		100								
		100								
		100								
		100								
		100								
		100								
		100								
		100								

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