TOTAL SOLIDS

1. SCOPE AND APPLICATION

"Total Solids" is defined as the sum of the homogenous suspended and dissolved materials in a sample. This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The practical range of determination is from the method detection limit (MDL), 15 mg/L, to 20,000 mg/L. 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. This method is based on SM2540 B-97. The test covered by this method is W-TS.

2. SUMMARY OF METHOD

2.1. A well-mixed aliquot of sample is quantitatively transferred to a pre-weighed evaporating dish and evaporated to dryness at 105 °C. The dish is then cooled and reweighed and the Total Solids is determined by difference.

2.2. 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. 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. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting. 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. APPARATUS AND EQUIPMENT

3.1. Evaporating Dishes, porcelain, 100 mL capacity

3.2. 1 Desiccator

3.3. Analytical Balance (capable of weighing to 0.1 mg)

3.4. 100 mL Graduated Cylinder

3.5. Dish tongs

3.6. Laboratory ovens that can be set at 95 °C and 105 °C.

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.

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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 TS 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 desiccators used to store filters and dishes and in the desiccators used to cool 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 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 crucibles 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 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 is 180 days.

5. SAMPLE HANDLING AND PRESERVATION

5.1. Samples are preserved by refrigeration at ≤ 6 °C (not frozen) in order to minimize microbiological decomposition of solids. Analysis should begin as soon as possible after collection. Samples must be analyzed within seven days of sampling. Samples analyzed after this seven day holding time must be reported with an attached comment describing the nature of the delay as well as the appropriate qualifying code, Q.

5.2. Samples are stored in the Nutrient refrigerator in the Receiving Room of the DEP laboratory. Allow samples to come to room temperature before processing.

5.3. Evaporating dishes are cleaned by washing 5-6 times with hot water. For dishes with stains, use diluted Nitric Acid solution (4.3.) to remove stains. Rinse with deionized water 5-6 times. Allow the dishes to air-dry for at least a couple of hours.

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5.4. All samples should be treated as potentially hazardous; gloves should be worn whilst handling samples and acids.

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

Note: Be sure to record all of the necessary information neatly and correctly on the TS Excel worksheet. The worksheet is located at \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\Solids\. The TS results are automatically calculated on the worksheet.
Important -The worksheet contains conditional 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 Temperature Log Sheet (NU-OVENTEMPLOG). The log sheet is located at \\FLDEP1\DEAR\Labs\lab3\Chem_Data\Inorganic\Nutrients\Solids\In Progress_[analyst's initials]\Templates.

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. If an oven temperature has to be changed, an explanation is recorded on the log sheet and the new temperature is monitored.

6.1. Set one laboratory oven at a temperature of 95 °C and a second at 105 °C. It will take about an 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.

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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 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. Record the weight on the TS worksheet under Crucible Weight (g). Repeat this process for each dish that is needed.

6.8. If the field conductivity of the sample is greater than about 3000 µmhos/cm, there may be too much residue in the dish for the TS 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 TS. 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 (µmhos/cm) is:	Measure out this amount for TS:
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

6.9. After the samples have come to room temperature shake the sample vigorously and using a graduated cylinder rapidly transfer 100 mL of sample to a numbered, weighed, evaporating dish. Wash the graduated cylinder several times with deionized water to prevent sample carryover.

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6.10. 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. Note the temperature should be kept below the boiling point to prevent splattering and loss of the sample.

6.11. After the initial 18 hours, place the evaporated sample in an oven at 103-105 °C to further dry for at least 4 additional hours to assure that there is no moisture remaining in the dish.

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

6.13. Remove the dishes from the oven and place in the desiccators to cool for at least an hour. DO NOT weigh any dishes that are still hot. You may leave dishes in the desiccators overnight. Do not leave the desiccator door open too long. If the samples are exposed to the air for a long period of time, the sample results may be affected.

6.14. 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 TS worksheet under Crucible and Residue Weight (g): 1st.

6.15. Return the dishes to the 103-105 °C oven for 1-2 hours to repeat drying cycle. Return the dishes to the desiccators and cool.

6.16. Weigh the cooled dishes again and record this second weight on the TS worksheet under Crucible and Residue Weight (g): 2nd. The two weights should have a difference of less than 0.5 mg. If not, return to the drying oven for a third time and repeat the weighing process. Record third and final weight on the TS worksheet under Crucible and Residue Weight (g): 3rd. Continue the drying/weighing cycles recording the weight in the appropriate cell in the TS 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.

6.17. Determination of Total Solids:

6.17.1. Determine the amount of total solids. Record the information in the TS Excel Worksheet.

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

6.17.2. Determine the precision for the batch. One sample is run in duplicate giving results A and B.

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$$\textbf{Relative Percent Difference (RPD)} = \frac{|A - B|}{(A + B)} \times 200$$

6.18. Save the TS Excel file as a text only 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.

6.19. Place the completed TS worksheets along with the standard sheet in a folder labeled YYMMDD**M**T (for the Denver Analytical M310 balance) or YYMMDD**X**T (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 T indicates a TS 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-TS), filename/s, and initials of the analyst.

7.2. Each report folder should contain the following items - the TS 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

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

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

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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.

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 https://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents).

10. REFERENCES

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, 2540 B-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-047, Total Dissolved Solids.

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

10.9. Oven Manuals.

10.10. Quality Manual, Bureau of Laboratories, FDEP.

10.11. Laboratory Health and Safety Plan.

10.12. DEP SOP LB-006, Standard Operating Procedure for Records Maintenance and Storage.

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

Appendix of Significant Changes

06/19/2014 This SOP is an updated version of SOP NU-050-2.9, which was retired in 2005. It has since been updated to conform to standard method SM 2540 B-97, and was renamed NU-099-1.0.

06/05/2015 Data archival information was clarified in section 7. Oven temperature recording procedure was modified in section 6.

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