TOTAL SUSPENDED SOLIDS

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1. SCOPE AND APPLICATION

Total suspended solids (TSS) is defined as the portion of solids that is retained on 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) 2 mg/L (3.0 mg/L for samples with conductivity ≥ 15,000 µmhos/cm) to 20,000 mg/L The practical quantitation limit (PQL) is determined by the concentration at which 2.5 mg of residue as required by the method remains on the filter paper. For a sample volume of 250 mL, this is 10 mg/L. The test covered by this SOP is W-TSS.

2. SUMMARY OF METHOD

2.1. This method is based on Standard Methods 2540 D-2011.

2.2. A well-mixed sample is filtered through a pre-weighed glass fiber filter. The filter and any residue are then dried to a constant weight at 103-105 °C. The filter is cooled in a desiccator, weighed and the result used to compute the TSS of the sample.

Note: The filtrate from this analysis may be used for a total dissolved solids (TDS) determination if the sample requires both tests. See DEP SOP NU-047.

2.3. Interferences - samples high in filterable residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. See section 6.3. for the procedure to analyze such samples in order to minimize this interference.

3. EQUIPMENT AND SUPPLIES

3.1. ProWeigh™ Filters (preweighed 47 mm glass fiber filters for TSS)

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

3.3. 1 L Filter flask

3.4. Vacuum pump

3.5. Drying ovens with adjustable temperature capable of heating up to 105 °C

3.6. Desiccators

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

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3.8. Forceps

3.9. Class A, 250, 100, and 25 mL graduated cylinders

3.10. Calibrated digital thermometer with a min/max function

3.11. Needle-nose water bottle

3.12. 1 L Class A volumetric flask

4. REAGENTS AND STANDARDS (SEE 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. If available, a Certificate of Analysis for each purchased reagent, standard or chemical is also maintained in the REAGENT_PREP folder on the Nutrients drive at \\FLDEP1\DEAR\labs\Lab3\Chem_Data\Inorganic\Nutrients\. **All containers holding standards must be labeled with their unique ID and expiration date.**

4.1. Deionized (DI) water: for blanks (MDL and MBLK), 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 reuse. Desiccant may be regenerated 3 times before replacing with new desiccant. The old desiccant can be discarded in the trash.

4.3. A certified quality control sample (AltQC) is 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 is 180 days. For high conductivity samples, prepare in the salt blank (4.4.).

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4.4. A salt blank (MDL_SLT and MBLK_SLT) - Ricca Synthetic Sea Water (Fisher catalog no. 83635), or 30 g of sodium chloride (NaCl) is dissolved in 2 L of DI water. See manufacturer's information for expiration date.

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. Samples must be brought to room temperature before processing.

5.4. Samples should be analyzed within seven days of collection. Samples analyzed after this 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 SOP NU-033)

Note: Be sure to record all of the necessary information neatly and correctly on the TSS Excel worksheet (NU-TSS). Make sure to always use the latest version of the NU-TSS form 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 filters with your hands. Always use forceps when handling the filters.

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\.
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

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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. ProWeigh filters are precleaned, preweighed and certified by the manufacturer. One ProWeigh filter for each run (one per day) must be placed in the oven with the samples, then desiccated and weighed and the result compared with the manufacturer's certified weight. Record these weights on page 1 of the TSS worksheet.

An example run sequence includes:

- MDL (DI water, 4.1.)
- ALTQC (4.3.)
- MDL_SLT (synthetic sea water, 4.4.)
- Batched samples, including an MBLK (DI water, 4.1.) or MBLK_SLT (synthetic sea water, 4.4.) and one sample run in duplicate for each batch
- Blank (DI water, 4.1.)

6.2. Procedure for samples with conductivity < 15,000 µmhos/cm

Note: If the field conductivity for the sample is not available, the lab conductivity must be determined (hand-held conductivity meter or auto-titrator instrumentation can be used) and recorded on the TSS worksheet.

6.2.1. When ready to filter, assemble the filtering apparatus. Connect the vacuum hose to the filter flask.

6.2.2. Remove the ProWeigh filter from the aluminum dish, place it onto the filter apparatus 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, and pour out the rinse water, re-attach the flask, and re-apply vacuum.

6.2.3. While the vacuum is still on, shake the sample and transfer 250 mL to the filtering device using a clean 250 mL graduated cylinder. Rinse the cylinder with enough DI water to remove any remains of the sample.

Note: If TDS is also being determined, be sure to remove the filtrate and obtain the TDS aliquot before rinsing the cylinder. This will ensure the filtrate used for TDS is not diluted.

Note: If it is not possible to filter 250 ml of sample due to particulate matter, determine the sample volume you are able to filter and start over at step 6.2.1 through 6.2.3.

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6.2.4. Once all the liquid has passed through the filter, 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.

6.2.5. Continue to apply the vacuum for 2-3 minutes after the sample has completely passed through and the filter has been rinsed, to ensure that all traces of water have been removed.

6.3. Procedure for samples with conductivity > 15,000 µmhos/cm (batched separately)

6.3.1. When ready to filter, assemble the filtering apparatus. Connect the vacuum hose to the filter flask.

6.3.2. Remove the ProWeigh filter from the aluminum dish and place it onto the filter apparatus. Thoroughly wet the filter with DI water, apply vacuum, and attach the membrane filter funnel over the filter. When all water has passed through the filter, turn off the vacuum, remove the filter flask, pour out the rinse water, re-attach the flask, and re-apply vacuum.

6.3.3. While the vacuum is still on, shake the sample and transfer 250 mL to the filtering device using a clean 250 mL graduated cylinder. Rinse the cylinder with enough DI water to remove any remains of the sample.

6.3.4. When all the water has been drawn through the filter, turn off vacuum and rinse with 30 mL DI water. Reapply vacuum, drawing DI water through the filter.

6.3.5. Repeat step 6.3.4.

6.3.6. Rinse the filtration tower once more with minimal volume and flow of DI water to remove any sample that remains on the sides of the tower.

6.3.7. While the vacuum is still on, remove the filter tower and gently rinse the portion of the filter that was under the filter tower with DI water one time. Use minimal flow and direct the water toward the center of the filter to avoid washing any particulate material from the filter.

6. 4. Carefully remove the filter with forceps, return to the aluminum dish, and place in an oven set at 103-105 °C. Wash the graduated cylinder, the filter holder, membrane filter funnel, and the filter flask with DI water before filtering the next sample to avoid sample carryover. The sample filters should remain in the oven for at least 1 hour, but usually remains overnight to ensure thorough drying. See the **Note** above regarding documentation of drying cycles on the Oven Temperature Log Sheet.

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6.5. Remove the dried filters and place in a desiccator to cool for at least 30 minutes. Check the calibration of the balance using certified weights and document the results in the balance maintenance logbook. Weigh the filters and record their weights accurately and legibly on the TSS worksheet under Filter and Residue Weight (g): 1st. 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.

6.6. Repeat drying cycle in the 103-105 °C oven for one more hour, then cool in a desiccator and weigh again following the same procedures as described in step 6.5. Make sure to record the weights on the TSS worksheet under Filter and Residue Weight (g): 2nd.

6.7. If the weight change is less than 0.5 mg then the analysis is complete. If this is not the case, repeat step 6.6. Record the weights on the TSS worksheet under Filter and Residue Weight (g): 3rd. If after three weighing cycles the weight change between the last two readings is still greater than 0.5 mg, consult a supervisor.

6.8. The TSS is calculated using the following equation,

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

6.9. Save the TSS Excel file as a text file (.txt) and use the GenChem: TSS-TDS Balance-XPE2042 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.

6.10. Place the completed TSS worksheets along with the standard sheet in a folder labeled YYMMDD**XE**S (for the Mettler Toledo XPE204-2 balance) or YYMMDD**X**S (for the Mettler Toledo XP204 balance), where YY is the year, MM is the month, DD is the day of the analyses, XE or X identifies the balance, and S indicates a TSS 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-TSS), filename/s, and initials of the analyst.

7.2. Each report folder should contain the following items - the TSS excel worksheet, bench sheet(s), the oven temperature log sheet, the backlog containing the batching

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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\.

7.5. Method validation data is on file with the supervisor. Digital copy is also stored on the network, under an appropriate sub-folder in \\fldep1\dear\Labs\Lab5\Chemistry\Inorganic\Nutrient\Validations.

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 are analyzed with samples having a conductivity below 15000 µmhos/cm and must be < 2.0 and > -2.0 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 is analyzed per batch of ten samples or less. If the sample result is above the PQL (10 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.

8.3. The AltQC (4.3.) is analyzed at the beginning of every run. Acceptance limits are provided by the manufacturer.

8.4. An MDL_SLT (4.4.) (analyzed at the beginning of every run) and MBLK_SLT (4.4.) (analyzed at the beginning of every salt batch) are run with samples having a conductivity above 15000 µmhos/cm and must be < 3.0 and > -3.0 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 salt 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.).

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8.5. One ProWeigh filter for every run must be desiccated and weighed. Record this weight and the certified weight of the filter on the TSS worksheet. The determined weight should be +/- 0.4 mg of the certified weight. If the weight is outside this range, put the filter back in the desiccator and leave it for at least 30 mins. Reweigh. If the weight is still outside the acceptable range, inform a supervisor.

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

8.7. 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.

8.8. 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/DEFINITIONS

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 D-2011.

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.

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

10.7. DEP SOP NU-047, Total Dissolved 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 from 20% to 10%.
06/03/2008	Reference method changed from 160.2 to SM 2540 D.
05/19/2011	The Method Detection Limit (MDL) and Practical Quantitation Limit (PQL) were changed to 2 and 10, respectively. See Section 1 for details.
05/08/2012	Section 6 was edited to include the new analysis procedures for samples below and above 15000 umhos/cm put into practice on 05/21/2012. The MDL for samples above 15000 µmhos/cm was also added.
08/23/2012	MDL for samples with conductivity ≥ 15,000 µmhos/cm was set to 3.0 mg/L.
05/08/2013	Section 4.3 was edited to correct the holding time of the ALTQC. Section 6.2.3 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.
11/29/2018	Appendix A and B were removed. The relevant forms are referenced in section 6. Reference method version was updated to the 2011 revision.

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