

Standard Operating Procedure for the Determination of Filterable and Non-Filterable Residue

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Revisions

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1.0 Scope and Application

The SOP is applicable to drinking, surface, saline, and industrial wastewater. This SOP should be used in conjunction with methods 2540C and 2540D, Standard Methods for Examination of Water and Wastes, U.S. EPA, Edition 22, March 2012.

2.0 Summary

This Standard Operating Procedure (SOP) describes analyzing environmental samples for filterable, and non-filterable residue in the sample. The procedure includes the following:

- Determination of non-filterable residue in the sample
- Determination of total filterable residue in the sample
- Quality Assurance and Reporting

3.0 Acronym/Definitions

- QA – Quality Assurance
- QC – Quality Control
- TDS – Total Dissolved Solids
- TSS – Total Suspended Solids

4.0 Health and Safety Warnings

The analyst should read and be thoroughly familiar with the USEPA Region I "General Laboratory Safety Procedures" SOP, current revision prior to running this method.

All applicable procedures in that SOP should be followed.

5.0 Interferences

- Samples high in filterable residue, such as saline waters, brines and some wastes, may be subject to a positive interference for non-filterable residue.
- Non-representative particulates should be excluded from the sample if determined that their inclusion is not desired in the final result.
- Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and sulfate may be hygroscopic and will require prolonged drying, proper desiccation and rapid weighing for filterable residue.

- Samples containing high concentration of bicarbonate will require prolonged drying to ensure that all 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.

6.0 Personnel Qualifications

The analyst should have at least a 4-year degree in a physical science. The analyst must have a satisfactory IDC in place before analyzing samples. All personnel shall be responsible for complying with all QA/QC requirements that pertain to their organizational/technical function.

7.0 Equipment and Supplies

- Filtering apparatus with reservoir and coarse (40–60 microns) fritted disk as filter support
- Glass fiber ProWeigh filters, 47 mm diameter. Filters come already pre-washed, pre-dried and pre-weighed
- Suction flask
- Drying oven, 103–105°C; 180°C ± 2°C
- Desiccator
- Analytical balance capable of weighting to 0.1 mg
- Crucibles

8.0 Procedures

8.1 Sample handling and preservation

- Preservation of the sample is not practical; samples should be analyzed as soon as possible and be refrigerated to 4°C prior to analysis.
- Holding times for TSS, and TDS are 7 days.

8.2 Non-Filterable residue (TSS)

- Place the ProWeigh filter (wrinkled side up) on the membrane filter apparatus and apply vacuum. Wet the filter with small volume of distilled water to seat filter against the fritted disc.

- Shake the sample well and quantitatively transfer up to 500 mL of sample, depending on the matrix, to filter using graduated cylinder. Transfer in a steady stream and do not allow the filter to dry.
- If the filter becomes plugged, discard that filter and begin the filtering process for that sample with a new filter using smaller volume of sample.
- After the sample has been filtered, rinse the graduated cylinder three times with distilled water and pour each rinse into the funnel, allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after rinse has passed through. The total volume of rinse water should equal approximately 30 mL for 47 mm filter.
- Carefully remove filter from the filter support. Dry at least one hour at 103– 105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).
- Prepare one duplicate, one blank and one QC sample per twenty samples or per batch (whichever is more frequent).
- To prepare the blank, filter a volume of distilled water equivalent to the volume of the largest sample volume used.
- The QC standard is usually purchased from a vendor.
- Follow the vendor's instruction for preparation.

8.3 Filterable Residue (TDS)

- Prepare evaporating dish by drying it in the oven at 180 ± 2 °C overnight and cool in desiccator. Weigh immediately before use.
- Shake the sample vigorously, rapidly transfer 100 mL to a cylinder and filter the sample. Rinse the graduated cylinder three times with distilled water and pour each rinse into the funnel.
- Transfer filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.
- Transfer to the oven and dry the evaporated sample for at least 1 hour at 180 ± 2 °C.
- Cool in desiccator and weigh.
- Repeat the drying cycle until constant weight is obtained or until weight difference is less than 0.5 mg.

8.4 Calculations

Record Sample number, volume, filter ID, weight of filter (without residue) on TSS/TDS form located in K:\LOCKED CALCULATION SPREADSHEETS.

For total non-filterable residue:

$$TSS, \frac{mg}{L} = \frac{(A - B) \times 1000}{C}$$

Where:

A = weight of the filter + residue in mg
B = weight of filter in mg
C = volume of sample filtered, mL

For total filterable residue:

$$TDS, \frac{mg}{L} = \frac{(A - B) \times 1000}{C}$$

Where:

A = weight of the evaporating dish + dry residue in mg
B = weight of evaporating dish in mg
C = volume of sample filtered, mL

The reporting limits for these methods: 10 mg/Liters of sample filtered for these methods, where 1.0 mg the practical low limit of measurement.

9.0 Data Records and Management

All raw data must be recorded on the TDS/TSS Log sheets. Log sheet can be found K:\LOCKED CALCULATION SPREADSHEETS

10.0 Quality Control and Quality Assurance

Upon completion of a project, a project review form should be filled out and accompany the final report in the report folder. The first section (requested analysis and data folder completeness checks) should be completed by the analyst. The last two sections (data evaluation and final report) should be completed by two different chemists that have knowledge of the method. Project review form for the TDS/TSS will be stored in K Drive.

Documentation of quality control standard information should include all pertinent information available on the bottle (vendor name, lot number, expiration date, date opened, date received, concentration of the standard, etc.). This information must be included with the raw data in the logbook. Copies of information sheets provided by the vendor can also be included with the raw data package.

Acceptance criteria are given in the chart in Appendix I. If acceptance criteria cannot be met, corrective actions must be taken, and the samples must be re-analyzed within holding time (see also Appendix I).

11.0 Waste Management and Pollution Prevention

NERL encourages all chemist and biologists to investigate micro analytical techniques, innovative technologies, and chemical substitution in laboratory processes to reduce waste and prevent pollution. As analytical SOPs are reviewed, on an annual basis, the responsible chemist or biologist will incorporate waste minimization practices where practicable and where these practices have been demonstrated to return data of equivalent quality. Chemists and biologists must refer to the Waste Management Program SOP, current revision for proper disposal of laboratory waste. Personnel should contact the Environmental, Safety and Health Department if changes in the analytical SOP will generate new waste streams. Questions regarding the proper disposal of laboratory waste and purchase of new reagents should be directed to the Environmental, Safety and Health Department in advance of actually initiating a change in the analytical method.

12.0 Preventative Maintenance

Balance calibration must be checked prior to weighing out samples or equipment.

13.0 References

Method SM2540C; Total Dissolved Solids Dried at 180°C (TDS) 20th Ed, Revised in 1998.

Method SM2540D; Total Suspended Solids, 22nd edition (1997)

Appendix I: Acceptance Criteria

QA/QC Sample	Parameter	Frequency	Acceptance Criteria	Corrective Action
Laboratory Method Blank	TSS/TDS	1 per batch	Less than the reporting limit	All samples associated with a contaminated blank must be reanalyzed. Otherwise, flag in the report (B) all data associated with this Blank
Quality Control Sample	TSS/TDS	1 per batch	Recoveries according to manufacturer's specifications	Explain in narrative on report.
Laboratory Duplicate	TSS/TDS	1 per batch	20 % RPD ¹	Estimate results for that sample (J) and explain in narrative on report.
Holding Times	TSS/TDS	—	Samples must be analyzed within hold time- 7 days from sample collection.	If re-sampling is not available, estimate result (J).
IDC	TSS/TDS	One a year per analyst	4 replicates of QCS	--

¹ = Acceptance criteria calculated from in-house historical data (control charts)