

SECTION D.11 TOTAL SUSPENDED SOLIDS

CEDR Method Codes: TSS L01 (Gravimetric; 103-105°C; *Subsampled*)
TSS L02 (Gravimetric; 103-105°C; *Entire Sample*)

a) Scope and Application

This method is applicable to the determination of non-filterable matter in drinking, surface, and saline waters. The reporting level is 5 mg/L, based on the requirement for 2.5 mg of residue on the filter and a typical sample volume of 500 mL.

b) Summary of Method

- i) A well-mixed aliquot of sample is vacuum-filtered through a glass-fiber filter, and the residue retained on the filter is dried to constant weight at 103–105°C. The increase in weight of the filter represents the total suspended solids.
- ii) The method may be modified to filter the entire contents of a water sample. Report these results with method code TSS L02.

c) Interferences

- i) Samples high in filterable residue (dissolved solids), such as saline waters, may be subject to a positive interference. Take care in selecting filtration equipment and sample volumes to ensure that rinsing the filter and residue minimizes this potential interference.
- ii) Certain biological materials, such as algae, slimes, insects or other small crustaceans may be considered to be positive interferences for non-filterable matter. Modifications or adjustments may be needed to generate a representative subsample.
- iii) The filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

d) Apparatus and Materials

- i) Glass fiber filter discs, without organic binder: Whatman® 47 mm diameter, 0.70 - 1.5µm pore size or equivalent must be used. Document the filter type and pore size in the field and/or laboratory SOP.
- ii) Vacuum filtering apparatus with reservoir and a coarse (40-60 microns) fritted disc as a filter support. Vacuum flasks with Gooch crucibles may be used for laboratory filtration.
- iii) Vacuum pump.
- iv) Drying oven capable of maintaining a temperature of 103-105°C.

- v) Desiccator with color indicator.
 - vi) Analytical balance capable of weighing to 0.1 mg.
 - vii) Graduated cylinders, Class A, TC (to contain). Plastic graduated cylinders are permitted for field filtration.
 - vii) Wide-bore pipets, Class A.
 - viii) Wash bottle with deionized water.
- e) Sample Handling
- i) Non-representative particulates such as leaves, sticks, stones, and detritus may be excluded from the sample if it is determined that their inclusion is not desired in the final result.
 - ii) Store aqueous samples in plastic or resistant-glass containers and refrigerate them at $\leq 6^{\circ}\text{C}$ to minimize microbiological decomposition of solids. Begin analysis as soon as possible, preferably within 24 hours of sample collection. The maximum holding time for lab-filtered TSS samples is 7 days.
 - iii) Field-filtered filter pads must be frozen at $\leq -20^{\circ}\text{C}$ ($\leq -4^{\circ}$ Fahrenheit) by the end of the sampling day. The maximum holding time for frozen field-filtered TSS filters is 28 days from the day of collection.
- f) Procedure
- i) Preparation of filters (this step is omitted if using pre-prepared, pre-weighed filters)
 - (1) Place the glass fiber filter or Gooch on the filter apparatus.
 - (2) While vacuum is applied, wash each filter with three successive 20 mL volumes of deionized, distilled water. Allow the vacuum pump to run until the filters are "dry".
 - (3) Remove the filter (and Gooch if used) from membrane filter apparatus, place on tray and dry in an oven at $103\text{--}105^{\circ}\text{C}$ for at least one hour.

If fixed suspended solids will also be measured, ignite washed filters at 550°C for 15 minutes in a muffle furnace instead of the drying oven.
 - (4) Place dried filters in a desiccator and cool to room temperature. Remove filters one-by-one and weigh each on a calibrated analytical balance and record the weights.
 - (5) Return the filters to the oven for one hour. Repeat the drying cycle until a constant weight is obtained, i.e., the second weight of each filter is within 10% of initial weight.
 - (6) Record the second filter weight and store the filters in a desiccator until needed.

ii) Sample analysis

- (1) Assemble the filtering apparatus and place a pre-weighed filter (and Gooch if used) wrinkle side up on the filtering apparatus. Wet the filter and begin suction.
- (2) For lab-filtered samples:
 - a. Choose a sample volume to yield between 2.5 and 200 mg of residue. If the volume filtered fails to meet the minimum yield, increase sample volume up to 1 L. If the filtration time exceeds 10 minutes discard the measured sample volume and filter and use another filter with a smaller volume size.
 - b. If samples contain sand or a large amount of suspended matter, use a magnetic stirrer and a wide-bore pipet to obtain a representative subsample.
- (3) For field-filtered samples, filter 500mL of sample. Reduce the volume as necessary to keep the filtration time under 10 minutes.
- (4) Shake the sample vigorously and quickly fill a graduated cylinder to the desired volume to prevent the solids from settling. Quantitatively transfer the subsample to the filtration apparatus and remove all traces of water by continuing to apply vacuum after sample has passed through. Record the volume of sample filtered.
- (5) With suction on, wash the filter, residue and wall of filter-funnel or crucible with three portions of deionized water, allowing complete drainage between washing. Remove all traces of water from the filter by continuing to apply vacuum pressure after water has passed through.
- (6) Field-filtration only: Place filter into sample container and place on ice or in freezer for storage and transport. Carefully remove the filter (or Gooch) from the filter support.
- (7) Place filters in oven and dry for at least one hour at 103-105°C. Cool in a desiccator and weigh using an analytical balance. On 10% or more of filters¹, repeat the drying cycle until a constant weight is obtained (no more than 10% difference). Record final weight.
- (8) If conducting subsequent analyses for fixed suspended solids or particulate phosphorus, return the filters to the desiccator or other means to prevent contamination.
- (9) Determine the concentration of Total Suspended Solids (TSS) in mg/L by calculating the amount of non-filterable residue as follows:

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

¹ Standard Methods requires that all of the filters be re-weighed to a constant weight. The CBP requirement is less stringent.

Commented [MEL1]: Standard Methods says 4% difference

Where: A = weight of filter and dried residue (mg).
 B = weight of filter (mg).

(10) Report TSS concentrations as follows:

- a. Concentrations less than 1,000 mg TSS/L - report 3 or more significant figures
- b. Concentrations of 1,000 mg TSS/L and above - report only whole numbers with 3 or more significant figures.

g) Quality Control

- i) Reporting Limit: The reporting limit is dependent on the maximum volume of sample filtered. For 500 mL of sample and ≥ 2.5 mg of residue on the filter pad, the reporting limit will be 5 mg/L. If the maximum sample volume is 1000 mL, the reporting limit will be 2.5 mg/L.
- ii) Method blank: See Chapter 6, Section 6.
- iii) Laboratory duplicate: See Chapter 6, Section 6.
- iv) Reference materials: The laboratory must analyze a TSS standard reference material at least quarterly.
- v) Balance calibration: Check the calibration of the analytical balance each day of use with NIST-traceable weights that bracket the working range, e.g., a high and low weight.

h) References

Standard Methods for the Examination of Water and Wastewater, Method 2540 D-2011, "Total Suspended Solids Dried at 103 – 105 ° C, 22nd Edition, pp 2-66, 2012.

U.S. Geological Survey, Techniques of Water-Resources Investigations of the United States Geological Survey. Chapter A1, Methods for the Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5, Laboratory Analysis, 3rd Ed.; Method I-3765-85, p. 443, (1989).