SECTION D.12
FIXED SUSPENDED SOLIDS

CEDR Method Codes: FSS L01 (Gravimetric; 550°C; *Subsampled*)
FSS L02 (Gravimetric; 550°C; *Entire Sample*)

a) Scope and Application

This procedure is used to obtain the amount of fixed matter present in the solid fraction of total suspended solids. This procedure is applicable to the determination of fixed matter in drinking, ground, surface, and saline waters, domestic and industrial wastes.

b) Summary of Method

The residue obtained from the determination of total suspended solids is ignited at 550 ± 50°C in a muffle furnace. The weight of the residue after ignition is reported as mg fixed suspended solids/L.

c) Interferences

i) The principal source of error in the determination is failure to obtain a representative sample.

ii) The test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion.

d) Apparatus and Materials

i) Filter pad from completed TSS analysis, with final weight of dried suspended residue recorded. See Section 6.D.11, Total Suspended Solids.

ii) Muffle Furnace capable of maintaining a temperature of 550 ± 50°C.

iii) Desiccator with calcium chloride desiccant and color-indicator.

iv) Analytical balance capable of weighing to 0.1 mg.

e) Sample Handling

i) Use only TSS filters whose preparation included muffling at 550°C for 15-20 minutes. Follow the sample handling and preparation procedures in Section 6.D.11, Total Suspended Solids.

ii) Analyses must be completed within the holding times listed in Table 6.1 (7 days for whole-water chilled samples or 28 days for field-filtered, frozen filters).

iii) Store weighed TSS filters with residue in desiccator if muffling is delayed.

f) Procedure

i) Ignite the filter with residue from the suspended solids procedure at 550 ± 50°C for approximately 15 to 20 minutes in a muffle furnace to a constant weight.

ii) Let the filter and nonvolatile residue partially cool in air until most of the heat has dissipated. Transfer to a desiccator, cool to room temperature and record the filter weight using an analytical balance. On 10% or more of filters[1], repeat the cycle of ignition, cooling and desiccating, until a constant weight is obtained (i.e., within 10% of initial weight). Record final weight of the filter.

Commented [MEL1]: Standard Methods is 4%

g) Calculations

i) Calculate the concentration of **fixed** suspended solids in mg/L using the following equation.

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

Where: B = weight of filter and residue after ignition (mg)
C = weight of filter without residue (mg)

ii) Calculate the concentration of **volatile** suspended solids in mg/L using the following equation.

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

Where: A = weight of filter and dried residue before ignition (mg)
B = weight of filter and residue after ignition (mg)

h) 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. For a maximum sample volume of 1000 mL, the reporting limit will be 2.5 mg/L.

ii) Filter blank: Ignite a blank glass fiber filter along with samples. Or, do a method blank as in Chapter 6, Section 6?

Commented [MEL2]: From Standard Methods

iii) Laboratory duplicate: see Chapter 6, Section 6.

iv) 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.

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

i) References

Standard Methods for the Examination of Water and Wastewater, Method 2540 E-2011 , "Fixed and Volatile Solids Ignited at 550°C", 22nd Edition, pp 2-67, 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.; Solids, nonvolatile on ignition, suspended (parameter code 00540) Method I-3766-85, p. 457, (1989).

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).