



Applied Microbiology
&
Biotechnology Laboratory

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Standard Operating Procedure

AMBL-105-E

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Fixed (Inorganic Ash) and Volatile Solids by Gravimetric Determination

METHOD SUMMARY

This SOP describes the procedure for measuring the fixed solids or inorganic ash and volatile solids portions of the total solids, total dissolved solids and total suspended solids in water and wastewater. This method is based on Method 2540 E of *Standard Methods for the Examination of Water and Wastewater*, 23rd Edition.

ENVIRONMENTAL HEALTH AND SAFETY

Hazards Assessment: This method involves the use of a muffle furnace. The specific hazards associated with this method are as follows.

Burns: Burns to the hands or arm are possible if the sides of the muffle furnace are touched when placing the sample into or removing it from the oven or furnace. Burns will also occur if the hot porcelain evaporating dish or filter pan is touched.

Safety Equipment and Engineering Controls: This method requires that the muffle furnace is operated beneath a capture hood to control smoke, fumes and odors. A set of long metal tongs to handle the filter pans and evaporating dishes when loading and unloading the muffle furnace are to be used.

Personal Protective Equipment (PPE): This method requires the use of the following PPE.

Gloves (nitrile, PVC or neoprene)

Heat resistant gloves

Safety goggles or glasses

Laboratory coat

Analysis-derived Wastes and Disposal:

Waste Generated	Hazardous (Y / N)	Disposal
This procedure generates an inorganic ash residue on the surface of a glass-fiber filter or in an evaporating dish.	N	The ash residue is to be absent of any organic materials, including bacteria. The filter with ash residue may be disposed in the laboratory trash. The ash residue in the evaporating dish may be either disposed in the laboratory trash or washed down the laboratory sink.

METHOD DESCRIPTION**1.0 Introduction and Applicability**

Fixed solids or inorganic ash is the measure of solid matter in a water that remains after the total solids, total dissolved solids or total suspended solids have been evaporated and then ignited to burn off the combustible portion. Volatile solids are the combustible portion. Volatile solids are a rough estimate for the organic portion of the solids, which has been used to evaluate the impact of solids loading from wastewaters entering treatment processes or natural waters, the character of the activated sludge process, and the performance of a sedimentation process. A sample having been used to determine total solids, total dissolved solids or total suspended solids is ignited to constant weight at a temperature of $550 \pm 50^{\circ}\text{C}$. The mass of the inorganic ash remaining is measured and used to determine both the fixed and volatile solids of the sample. Fixed and volatile solids may be expressed as a concentration, in mg/L, or as a percent of the solids type being reported.

This method is applicable for measurement of fixed and volatile solids in all natural waters, in raw, process and treated agricultural, municipal and industrial wastewaters. This method is not considered applicable to wastewater slurries behaving as a Newtonian fluid, non-Newtonian fluids or treated drinking water.

2.0 Apparatus

- Muffle furnace operated at $550 \pm 50^{\circ}\text{C}$.
- Desiccator containing a desiccant that responds (color change) to moisture or a hygrometer that measures moisture.
- Analytical balance capable of weighing to the nearest 0.1 mg or less.

3.0 Procedure

- a. Read Method 2540E Fixed and Volatile Solids Ignited at 550°C (*Standard Methods*).
- b. Make sure that the evaporating dish used for the total solids or dissolved solids analysis, or the filter used for the total suspended solids analysis has been pre-conditioned at $550 \pm 50^\circ\text{C}$ as described in SOPs 105A, 105C and 105D, respectively. If this has not been done, the values resulting from this method must be reported as “estimates” and if possible, the sample must be reanalyzed.
- c. Insert the evaporating dish from SOP 105A or 105C, or the filter pan from SOP 105D into the muffle furnace. Close the door and once the temperature has returned to $550 \pm 50^\circ\text{C}$, allow the sample to ignite for no less than 15 minutes.
- d. Remove the evaporating dish or filter pan from the muffle furnace. Place the evaporating dish onto a heat resistant surface, allowing it to cool to nearly room temperature, and then transfer to evaporating dish to a desiccator to finish cooling to room temperature. Place the filter pan onto a heat resistant surface for 2 or 3 minutes before transferring to the desiccator to finish cooling to room temperature.
- e. Weight the sample as previously weighed in SOPs 105A, 105C and 105D, and record this weight as the first 550°C weight.
- f. Repeat the ignition again for no less than 15 minutes, cooling to room temperature as before, weigh and record this as the second 550°C weight.
- g. Calculate the weight change between the first and second weights, and if the change is >0.5 mg, repeat the ignition cycle until the change in weight between the final weight and the previous weight is ≤ 0.5 mg. Record and use this final 550°C weight.

4.0 Calculation and Reporting

- a. Calculate the concentration of total fixed solids

$$\text{Total Fixed Solids, as mg FS/L} = \frac{(B - D) \times 1,000}{S}$$

where B = final 550°C weight of the inorganic ash + the tared dish, mg,

D = tared dish weight, mg (from SOP 105A), and

S = mL of sample volume (from SOP 105A).

- b. Report as “Total Fixed Solids (FS) = ____ mg/L”
or as “____ mg/L FS”

- c. Calculate the percent of total fixed solids

$$\text{Total Fixed Solids, as \% of TS} = \frac{B - D}{A - D} \times 100$$

where A = final 103°C weight of the dried residue + the tared dish,
mg (from SOP 105A),

B = final 550°C weight of the inorganic ash + the tared dish,
mg, and

D = tared dish weight, mg (from SOP 105A)

- d. Report as “Percent Total Fixed Solids (%FS) = ____”
or as “____ %FS”

- e. Calculate the concentration of total volatile solids

$$\text{Total Volatile Solids, as mg VS/L} = \frac{(A - B) \times 1,000}{S}$$

where A = final 103°C weight of the dried residue + the tared dish,
mg (from SOP 105A),

B = final 550°C weight of the inorganic ash + the tared dish,
mg,

S = mL of sample volume (from SOP 105A).

- f. Report as “Total Volatile Solids (VS) = ____ mg/L”
or as “____ mg/L VS”

- g. Calculate the percent of total volatile solids

$$\text{Total Volatile Solids, as \% of TS} = \frac{A - B}{A - D} \times 100$$

where A = final 103°C weight of the dried residue + the tared dish,
mg (from SOP 105A),

B = final 550°C weight of the inorganic ash + the tared dish,
mg,

D = tared dish weight, mg (from SOP 105A), and

- h. Report as “Percent Total Volatile Solids (%VS) = ____”
or as “____ %VS”

- i. Calculate the concentration of fixed dissolved solids

$$\text{Fixed Dissolved Solids, as mg FDS/L} = \frac{(B - D) \times 1,000}{S}$$

where B = final 550°C weight of the inorganic ash + the tared dish, mg,

D = tared dish weight, mg (from SOP 105B), and

S = mL of sample volume (from SOP 105B).

- j Report as “Fixed Dissolved Solids (FDS) = ____ mg/L”
or as “____ mg/L FDS”

- k. Calculate the percent of fixed dissolved solids

$$\text{Fixed Dissolved Solids, as \% of TDS} = \frac{B - D}{A - D} \times 100$$

where A = final 180°C weight of the dried residue + the tared dish, mg (from SOP 105B),

B = final 550°C weight of the inorganic ash + the tared dish, mg,

D = tared dish weight, mg (from SOP 105B), and

- l. Report as “Percent Fixed Dissolved Solids (%FDS) = ____”
or as “____ %FDS”

- m. Calculate the concentration of volatile dissolved solids

$$\text{Volatile Dissolved Solids, as mg VDS/L} = \frac{(A - B) \times 1,000}{S}$$

where A = final 180°C weight of the dried residue + the tared dish, mg (from SOP 105B),

B = final 550°C weight of the inorganic ash + the tared dish, mg,

S = mL of sample volume (from SOP 105B).

- n Report as “Volatile Dissolved Solids (VDS) = ____ mg/L”
or as “____ mg/L VDS”

- o. Calculate the percent of volatile dissolved solids

$$\text{Volatile Dissolved Solids, as \% of TDS} = \frac{A - B}{A - D} \times 100$$

where A = final 180°C weight of the dried residue + the tared dish, mg (from SOP 105B),

B = final 550°C weight of the inorganic ash + the tared dish, mg,

D = tared dish weight, mg (from SOP 105B), and

- p. Report as “Percent Volatile Dissolved Solids (%VDS) = _____”
or as “_____ %VDS”

- q. Calculate the concentration of fixed suspended solids

$$\text{Fixed Suspended Solids, as mg FSS/L} = \frac{(B - F) \times 1,000}{S}$$

where B = final 550°C weight of inorganic ash + the tared filter, mg,

F = tared filter weight, mg (from SOP 105D), and.

S = mL of sample volume (from SOP 105D).

- r. Report as “Fixed Suspended Solids (FSS) = _____ mg/L”
or as “_____ mg/L FSS”

- s. Calculate the percent of fixed suspended solids

$$\text{Fixed Suspended Solids, as \% of TSS} = \frac{B - F}{A - F} \times 100$$

where A = final 103°C weight of the dried residue + the tared filter, mg (from SOP 105D),

B = final 550°C weight of the inorganic ash + the tared filter, mg, and

F = tared filter weight, mg (from SOP 105D).

- t. Report as “Percent Fixed Suspended Solids (%FSS) = _____”
or as “_____ %FSS”

- u. Calculate the concentration of volatile suspended solids

$$\text{Volatile Suspended Solids, as mg VSS/L} = \frac{(A - B) \times 1,000}{S}$$

where A = final 103°C weight of the dried residue + the tared filter, mg (from SOP 105D),

B = final 550°C weight of inorganic ash + the tared filter, mg, and

S = mL of sample volume (from SOP 105D).

- v. Report as “Volatile Suspended Solids (VSS) = ____ mg/L”
or as “____ mg/L VSS”

- w. Calculate the percent of volatile suspended solids

$$\text{Volatile Suspended Solids, as \% of TSS} = \frac{A - B}{A - F} \times 100$$

where A = final 103°C weight of the dried residue + the tared filter, mg (from SOP 105D),

B = final 550°C weight of the inorganic ash + the tared filter, mg, and

F = tared filter weight, mg (from SOP 105D).

- x. Report as “Percent Volatile Suspended Solids (%VSS) = ____”
or as “____ %VSS”

5.0 Quality Control

Determining the fixed and volatile portions of a samples solid material can lead to variable results and thus quality control is considered to be an important part of this method.

- Analyze a method blank (a clean, dried, and tared filter) with each batch of 20 or fewer samples. If a single sample is being analyzed, a method blank must also be analyzed. This is accomplished by continuing to analyze the method blank in SOP 105A, 105B or 105D with this method.
- Analyze at least one sample in duplicate with each batch of 20 or fewer samples. If a single sample is being analyzed, this sample must be analyzed in duplicate.
- A laboratory-fortified blank and laboratory-fortified blank duplicate sample set (LFB/LFBD) to demonstrate initial and ongoing capability has not been established for this method.
- Evaluate the results obtained from QC data as follows: The method blank results must demonstrate that the initial tared dish weight does not differ by more than ± 0.5 mg. The relative percent difference (RPD) of duplicate samples should not exceed an absolute value of 10%.

6.0 Bibliography

- Rodger B. Baird, Andrew D. Eaton, and Eugene W. Rice (2017)
Standard Methods for the Examination of Water and Wastewater.
APHA, Washington, DC, 23rd Edition.