
	<p style="text-align: center;"><i>Standard Operating Procedure</i></p> 	Effective Date:	Version:
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Standard Operation Procedure – Solids

Introduction

Solids refer to matter suspended or dissolved in water, wastewater and faecal sludge. Solids may affect water or effluent quality adversely in a number of ways. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory wastewater effluent limitations.

Total Solids are a term applied to material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids include **Total Suspended Solids**, the portion of solids retained by a filter and **Total Dissolved Solids**, the portion that passes through the filter of 2.0µm or smaller. **Fixed Solids** is the term applied to residue of total, suspended or dissolved solids after heating to dryness for a specified time at a specified temperature. The weight loss on ignition is called **Volatile Solids**.

Total Solids Dried at 103°C - 105°C

1. Scope and Field of Application

- Total Solids are determined in a wide variety of liquid and semi-liquid materials.
- These include portable waters, domestic and industrial waters, polluted waters and faecal sludge produced from treatment processes. It is of particular importance for the efficient operation of a treatment plant.
- A known volume of well-mixed sample is evaporated to dryness in a porcelain crucible in a hot air oven at 105°C; the solids remaining are cooled and weighed.
- The residual material in the crucible is classified as total solids, and may consist of organic, inorganic, dissolved, suspended or volatile matter.

1. Apparatus

- 50ml capacity evaporating porcelain crucibles
- Desiccator
- Drying oven
- Analytical Balance

2. Reagents

- None

3. Interferences

- Highly mineralised water with a significant concentration of calcium, magnesium, chloride and sulphate may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing.
- Exclude large, floating particles from the sample if it is determined that their inclusion is not desired in the final result.
- Disperse visible floating oil and grease with a blender before withdrawing sample portion for analysis because excessive residue in the dish may form a water-trapping crust.

4. Sampling

- Mix the sample well to suspend solids uniformly.
- Remove the test portion rapidly before any settling of solid matter occurs.
- Use a measuring cylinder and not a pipette for sludge and wastewater samples.
- Use a crucible for faeces.

- Use a volume or mass of sample to ensure a measurable residue- limit sample to no more than 200mg residue.
- Suitable aliquots: Liquid samples – 100ml, Sludge -30ml, Faeces 10-20g

5. Safety Precautions

- Always use safety goggles, gloves and laboratory coat while working in laboratory.
- Wear gloves suitable for withstanding high temperatures when removing crucibles from the oven.
- After the analysis, clean bottles and beakers with clear water keep it for drying.
- Dispose the used gloves after completion of analysis.
- Clean your hands using antiseptic soap.
- Disinfect hands after washing with soap.
- Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

6. Calibration

- Check the temperature throughout the oven area by placing a calibrated thermometer on each shelf, after 30 min, check temperature at each level against oven setting.
- Adjust oven setting if necessary.
- If temperatures are uneven on the shelves, check insulation.

7. Procedure

Prepare Crucible

- If volatile solids are to be measured, ignite clean crucible at 550°C for 1 hour in the furnace. If only total solids are to be measured, heat clean crucible to 103-105°C for 1 hour. Store and cool dish in a desiccator until needed. Weigh immediately before use.....W1g.

Sample Analysis

- Measure out appropriate volume (30ml) /minimum mass (10-20g) that will yield a residue between 2.5 and 200mg of a mixed sample using correct volume measuring cylinder or analytical balance.....Vml...Wg. Transfer quantitatively to the weighed crucible, rinsing the cylinder with small volumes of distilled water to dislodge heavy particles. Add washings to the crucible.
- Place in hot oven at 103-105°C for 24 hours.
- ***Dry sample for at least 1hr in an oven 103-105°C, to desiccator to balance temperature and weigh. Repeat cycle of drying, cooling and weighing until a constant weight is obtained, or until weight change is less than 4% of previous weight or 0.5mg, whichever is less.***
- Remove the next day and cool for 15 minutes and weigh.....W2g

8. Calculation

$$\text{Total Solids in Sample (mg/l)} = \frac{(W_2 - W_1)g \times 100\,000}{V_{\text{sample}} \text{ (ml)}}$$

$$\text{Total Solids in Wet Sample (g/g)} = \frac{(W_2 - W_1)g}{W_{\text{sample}} \text{ (g)}}$$

$$\text{Moisture Content (g)} = W_{\text{sample}}(g) - [(W_2 - W_1)]g$$

Total Suspended Solids Dried at 103-105°C

1. Scope and Field of Application

Suspended solids are useful determinants in the analysis of polluted, re-use and wastewaters. It is used to evaluate the strength of domestic/industrial wastewaters and to determine the efficiency of treatment units, such as settling tanks, biological filters, and the activated sludge. Use of glass fibre filter pads is preferred to crucibles because of the saving in filtration time and the only prior preparation necessary is drying in an oven for 30mins at 105°C.

A measured volume of well shaken is vacuum filtered through a dried pre-weighed 110mm diameter glass fibre filter. The filters and residue is dried to a constant weight at 103-105°C.

The increase in weight of the filter represents the total suspended solids.

2. Apparatus

- Four- place Analytical balance
- 110mm diameter funnel and flask
- Vacuum pump

3. Reagents

- Nil

4. Interferences

- Exclude isolated large floating particles.
- Samples high in dissolved solids must be washed adequately.
- Loss in mass of the rinsed glass fiber filters must be taken into the final calculation.
- The larger the sample, the smaller the factor applied in the calculation, but avoid prolonged filtrations.

5. Sampling

- Take the sample at a point of turbulence to ensure that it is truly representative.
- Mix sample thoroughly and remove test portion rapidly before segregation occurs.
- Use appropriate volume measuring cylinder and not pipettes.

6. Safety Precautions

- Exercise care when using glassware, vacuum pumps and ovens.
- Good housekeeping and cleanliness are essential for obtaining accurate results.

7. Sample Preparation – Faecal Sludge

- Weigh out between 1.8g and 2g of well-mixed faecal sludge sample.
- Place the weighed out sample into a blender with 250ml of distilled water.
- Blend for 30 seconds.
- Transfer the blended mixture into a volumetric flask and top up to 1L with distilled water.
- Transfer the 1L solution to a plastic bottle and store in the cold room.

8. Calibration

- The analytical balance and ovens are checked and serviced weekly.

9. Procedure

Dry Filter Paper

- Use 110mm glass fiber filter paper Whatman No 4(20-25 μ m) for sludge.
- Use 20ml sample on a 40mm, 0.45 μ m glass fibre filter for wastewater and urine (change to a 1 μ m pore size if the dried residue is more than 200mg).
- Use a smaller pore size if the dried residue is lower than 2.5mg.
- Mark the filter paper with a pen.
- Place papers on the stainless steel mess of appropriate size.
- Position on top shelf in oven at 105°C for 30 min.
- If volatile solids are to be measured, ignite at 550°C for 15 min in a furnace.
- Transfer to desiccator.
- Cool for 20 mins before weighing.

Weigh Filter Paper

- Transfer filter paper rapidly to balance.
- Note mass (W1) grams, to fourth decimal place.

Prepare for Analysis

- Place filter paper into a 110mm diameter funnel, with the marking on the lower side.
- Measure out appropriate volume to yield between 2.5 and 200mg dried residue of well-mixed sample.
- Place funnel into flask with side arm attached to a vacuum pump.
- Apply pump.
- Wet paper with distilled water to seal edges of the paper to surface of the funnel.
- Pour sample onto the filter paper, keeping sample in the middle of the paper.
- When filtration is complete. Remove paper by placing the end of a small thin spatula along the edge of the filter paper and lift slowly.
- Remove the paper with a pair of tweezers, taking care not to tear the paper.
- Fold paper twice to form a triangle enclosing sample residue. This seals the residue in the filter paper and protects it from contact with air.

Dry and Weigh

- Place triangles on a stainless steel mesh.
- Place in oven at 105°C for 2 hours.
- Remove from oven and place in desiccator.
- Cool to room temperature.
- Weigh after 20 mins, as rapidly as possible.
- Note mass (W_2) grams.

10. Calculation

$$\text{Total Suspended Solids (g/ml)} = \frac{(W_2 - W_1)}{V_{\text{sample}}(\text{ml})}$$

$$\text{Total Suspended Solids in Wet Sample (g/g)} = \text{TSS (g/ml)} \times \text{DF}$$

$$\text{Total Suspended Solids in Dry Sample (g/g)} = \frac{\text{TSS}_{\text{wet sample}}}{\text{Total Solids (g/g)}}$$

W_1 = weight of filter paper before oven (105°C) (g)

W_2 = weight of residue + filter paper after oven (105°C) (g)

DF = Dilution Factor

Fixed and Volatile Solids Ignited at 550°C

1. Introduction

The residue from the above methods is ignited to constant weight at 550°C. The remaining solids represent the fixed total, dissolved or suspended solids while the weight lost on ignition is the volatile solids. The determination is useful in control of wastewater treatment plant operation because it offers a rough estimate of the amount of organic matter present in the solid fraction of wastewater, activated sludge and industrial wastes.

2. Apparatus

- Muffle Furnace
- As above

3. Interferences

- Negative errors in the volatile solids may be produced by loss of volatile matter during the drying.

4. Procedure

- Ignite residue from the total solids to constant weight in a muffle furnace at a temperature of 550°C.
- Have furnace up to temperature before inserting sample.
- Usually 2 hours for VIP and sludge samples, 15-20 min for wastewater (200mg residue).
- Let the crucible cool partially in air until most of the heat has dissipated.
- Transfer to a desiccator for final cooling. Do no overload the desiccator.
- Weigh dish as soon as it has cooled to balance temperature.

5. Calculation

$$\text{Volatile Solids in Wet Sample (g/g)} = \frac{(B - C)}{W_{\text{sample}}(g)}$$

$$\text{Volatile Solids in Dry Sample (g/g)} = \frac{VS_{\text{wet sample}}}{\text{Total Solids(g/g)}}$$

$$\text{Fixed Solids in Wet Sample(g/g)} = \frac{(C - D)}{W_{\text{sample}}(g)}$$

$$\text{Fixed Solids in Dry Sample(g/g)} = \frac{FS_{\text{wet sample}}}{\text{Total Solids(g/g)}}$$

B = weight of residue + crucible before ignition – 550°C (g)

C = weight of residue + crucible after ignition -550°C (g)

D = weight of crucible (g)

6. References

Standards Methods for the Examination of Water and Wastewater, 18th Edition, p. 2-62, Methods 2540 Solids, A,B,C,D,E(1992).

APPROVAL OF STANDARD OPERATING PROCEDURE

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