

DETERMINATION OF SOLIDS

Solids refer to matter suspended or dissolved in water or wastewater. Solids may affect water or effluent quality adversely in a number of ways. Waters with high dissolved solids generally are of inferior palatability and may induce an unfavorable physiological reaction in the transient consumer. For these reasons, a limit of 500 mg dissolved solids/L is desirable for drinking waters. Highly mineralized waters also are unsuitable for many industrial applications. Waters high in suspended solids may be esthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations.

Definitions:

"Total solids" is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids includes **"total suspended solids"** the portion of total solids retained by a filter, and **"total dissolved solids"** the portion that passes through the filter. The type of filter holder, the pore size, porosity, area, and thickness of the filter and the physical nature, particle size, and amount of material deposited on the filter are the principal factors affecting separation of suspended from dissolved solids. **"Dissolved solids"** is the portion of solids that passes through a filter of 2.0 μm (or smaller) nominal pore size under specified conditions. **"Suspended solids"** is the portion retained on the filter. **"Fixed solids"** is the term applied to the 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"**. Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because the loss on ignition is not confined to organic matter. It includes losses due to decomposition or volatilization of some mineral salts. Better characterization of organic matter can be made by such tests as total organic carbon.

"Settleable solids" is the term applied to the material settling out of suspension within a defined period. It may include floating material, depending on the technique.

DETERMINATION OF TOTAL SOLIDS

1. Apparatus

- a) Evaporating porcelain dishes: Dishes of 100-mL capacity
- c) Muffle furnace for operation at 550°C.
- d) Steam bath
- e) Desiccator
- f) Drying oven, for operation at 103 to 105°C
- g) Analytical balance, capable of weighing to 0.1 mg.
- h) Magnetic stirrer, pipets, graduated cylinder etc.

2. Procedure

2.1. Preparation of evaporating dish:

- Heat clean dish to 103 to 105°C for 1 h.
- Store and cool dish in desiccator until needed.
- Weigh immediately before use.

2.2. Sample analysis:

- a) Choose a sample volume that will yield a residue between 2.5 and 200 mg.
- b) Pipet a measured volume of well-mixed sample, during mixing, to a
- c) Take a preweight of dish.
- d) Add successive sample portions to the same dish.
- e) When evaporating in a drying oven, lower temperature to approximately 2°C below boiling to prevent splattering.
- f) Dry evaporated sample for at least 1 h in an oven at 103 to 105°C, cool dish in desiccator to balance temperature, and weigh.

3. Calculation

$$\text{mg total solids/L} = ((A - B) \times 1000) / (\text{sample volume, mL})$$

where:

A = weight of dried residue + dish, mg

B = weight of dish, mg

DETERMINATION OF FIXED AND VOLATILE SOLIDS

a) Ignite residue produced by Total Solids analysis to constant weight in a muffle furnace at a temperature of 550°C.

b) Have furnace up to temperature before inserting sample.

c) Usually, 15 to 20 min ignition are required for 200 mg residue.

d) Let dish or filter disk cool partially in air until most of the heat has been dissipated.

e) Transfer to a desiccator for final cooling in a dry atmosphere.

f) Weigh dish

Calculation

mg volatile solids/L = $((A - B) \times 1000) / \text{sample volume,}$

mL mg fixed solids/L = $((B - C) \times 1000) / (\text{sample volume,}$

mL) where:

A = weight of residue + dish before ignition, mg

B = weight of residue + dish or filter after ignition, mg

C = weight of dish or filter, mg

DETERMINATION OF TOTAL SUSPENDED SOLIDS AND VOLATILE SUSPENDED SOLIDS

Apparatus

- a) Glass- fiber filter paper (0.7-1.5 μm)
- b) Aluminum weighing dishes
- c) Muffle furnace for operation at 550°C.
- d) Steam bath
- e) Desiccator
- f) Drying oven, for operation at 103 to 105°C
- g) Analytical balance, capable of weighing to 0.1 mg.
- h) Magnetic stirrer, pipets, graduated cylinder etc.

Procedure

- a) Dry filter paper in an oven at 103 to 105°C for 1 h.
- b) Cool in desiccator to balance temperature and weigh.
- c) Store in desiccator until needed.
- d) Choose sample volume to yield between 2.5 and 200 mg dried residue.
- e) Assemble filtering apparatus and filter and begin suction
- f) Wet filter with a small volume of reagent-grade water to seat it.
- g) Stir sample with a magnetic stirrer
- h) While stirring, pipet a measured volume onto the seated glass-fiber filter.
- i) Wash filter with three successive 10-mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete.
- i) Carefully remove filter from filtration apparatus and transfer to an aluminum weighing dish as a support.
- j) Dry for at least 1 h at 103 to 105°C in an oven, cool in a desiccator to balance temperature, and weigh.

k) If volatile solids are to be measured, ignite at 550°C for 15 min in a muffle furnace and dry it for 20 min to 550 °C.

Calculation

mg total suspended solids/L = $((A - B) \times 1000) / (\text{sample volume, mL})$

where:

A = weight of filter + dried residue, mg

B = weight of filter, mg.

mg volatile suspended solids/L = $((B - C) \times 1000) / (\text{sample volume, mL})$

C: weight of filter +dried residue after ignition, mg

SETTLEABLE SOLIDS

Apparatus

A glass vessel with a minimum diameter of 9 cm.

Procedure

- a. Fill an Imhoff cone to the 1-L mark with a well mixed sample.
- b. Settle for 45 min, gently agitate sample near the sides of the cone with a rod or by spinning.
- c. settle 15 min longer, and record volume of settleable solids in the cone as milliliters per liter.

Calculation

mg settleable solids/L = mg total suspended solids/L — mg nonsettleable solids/L

CONDUCTIVITY

Conductivity of a substance is defined as 'the ability or power to conduct or transmit heat, electricity or sound'. When an electrical potential difference is placed across a conductor, its movable charges flow, giving rise to an electric current. This property is called conductivity. Since the charge on ions in solution facilitates the conductance of electrical current, the conductivity of a solution is proportional to its ion concentration.

The electrical conductivity can be expressed as mhos (Reciprocal of ohms) or as siemens. The conductivity of water is a measure of the ability of water to carry an electric current. In most water, the conductivity is very low, so millisiemens or microsiemens are used as units for water conductivity. The conductivity of water is directly linked to the concentration of the ions and their mobility. The ions in water act as electrolytes and conduct the electricity.

The conductivity depends on the value of the pH, on the temperature of measurement and on the amount of CO₂ which has been dissolved in the water to form ions. The conductivity is also affected by the concentration of ions already present in the water such as chloride, sodium and ammonium.

Chemical composition of water determines its conductivity. Hence this becomes the most widely used measure of the purity of water.

- Electrical conductivity measurements are often employed to monitor desalination plants.
- It is useful to assess the source of pollution.
- In coastal regions, conductivity data can be used to decide the extent of intrusion of sea water into ground water.
- Conductivity data is useful in determining the suitability of water and wastewater for disposal on land. Irrigation waters up to 2 millisiemens / cm conductance have been found to be suitable for irrigation depending on soils and climatic characteristics.
- It is also used indirectly to fine out inorganic dissolved solids.

1. Experimental Procedure

Conductivity is measured with a probe and a meter. A voltage is applied between the two electrodes in the probe immersed in the sample water. The drop in voltage caused by the resistance of the water is used to calculate the conductivity per centimeter.

Conductivity (G), the inverse of resistivity (R) is determined from the voltage and current values according to Ohm's law. i.e. $R = V/I$ then, $G = 1/R = I/V$.

The meter converts the probe measurement to micro mhos per centimeter and displays the result for the user.

1.1. Materials and Equipment

- Conductivity Meter with Electrode /ATC probe
- Magnetic Stirrer with stirring bead
- Standard flask
- Measuring jar
- Beaker 250 mL
- Funnel

2. Steps of the Experiment

2.1. Sample Handling and Preservation

Water samples should be collected in plastic cans or glass bottles. All bottles must be cleaned thoroughly in phosphate-free detergent and rinsed thoroughly with both tap and distilled water.

Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal. Analysis should begin as soon as possible after the collection.

If the analysis is not completed within 12 hours of sample collection, sample should be filtered through a 0.45 μ filter paper and stored at 4°C. High quality distilled water must be used for washing the filter and apparatus and needs to be rinsed with sample before use. No chemical preservation is required.

Keep the samples at 4°C. Do not allow samples to freeze.

2.2. Precautions

The following precautions should be observed while performing the experiment:

- Switch on the conductivity meter for at least 30 minutes before starting the experiment so that the instrument gets stabilizes.
- As it involves instruments for analyzing do not forget to calibrate the instrument.
- Always prepare the calibration solution freshly before the start of the experiment.
- As conductance is influenced by temperature, always use a conductivity meter with temperature control.
- Always dip the electrode in distilled water and do not expose it to air.

2.3. Calibration

- Switch on the Electronic balance, keep the weighing pan, and set the reading to zero.
- Measure 50 mL of distilled water and transfer it to the beaker.
- Weigh 0.7456g of Potassium chloride.
- Transfer the 0.7456g of potassium chloride to the beaker contains distilled water and mix it by the glass rod until it dissolves thoroughly.
- Transfer the contents to the 100 mL standard flask.
- Make up the volume to 100 mL, by adding distilled water and shake the contents well. This solution is used to calibrate the conductivity meter.

2.4. Testing Of Water Sample

- Rinse the electrode thoroughly with deionised water and carefully wipe with a tissue paper.
- Measure 200 mL of water sample and transfer it to a beaker and place it on the magnetic stirrer.

- Dip the electrode into the sample solution taken in a beaker and wait for a steady reading.
- Make sure that the instrument is giving stable reading.
- Note down the reading in the display directly, which is expressed in millisiemens.

