EXPERIMENT 3

SOLIDS DETERMINATION

A. PURPOSE

To familiarize the students with various analytical operations such as weighing, filtration, evaporation and combustion which are commonly encountered in gravimetric analysis.

To demonstrate the separation and categorization of different kinds of solids.

B. THEORY

The term “solids” is generally used when referring to any material suspended or dissolved in water or wastewater that can be physically isolated either through filtration or through evaporation. Solids can be classified as either filterable or non filterable. Filterable solids may either be settleable or non settleable. Solids can be classified as organic or inorganic.

Total solids are dissolved solids plus suspended and settleable solids in water. In stream water, dissolved solids consist of calcium, chlorides, nitrate, phosphorus, iron, sulfur, and other ions particles that will pass through a filter with pores of around 2 microns (0.002 cm) in size. Suspended solids include silt and clay particles, plankton, algae, fine organic debris, and other particulate matter. These are particles that will not pass through a 2-micron filter. Sources of total solids include industrial discharges, sewage, fertilizers, road runoff, and soil erosion. Total solids are measured in milligrams per liter (mg/L).

Wastewater contains a variety of solid materials varying from rags to colloidal material. In the characterization of wastewater, coarse materials are usually removed before the sample is analyzed for solids. The interrelationship between the various solids fractions found in wastewater is illustrated graphically on Fig.1. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations. Total solids consist of suspended and filterabe solids. Typically, about 60 percent of the suspended solids in a municipial wastewater are settleable. Total solids (TS) are obtained by evaporating a sample of wastewater to dryness and measuring the mass of the residue.

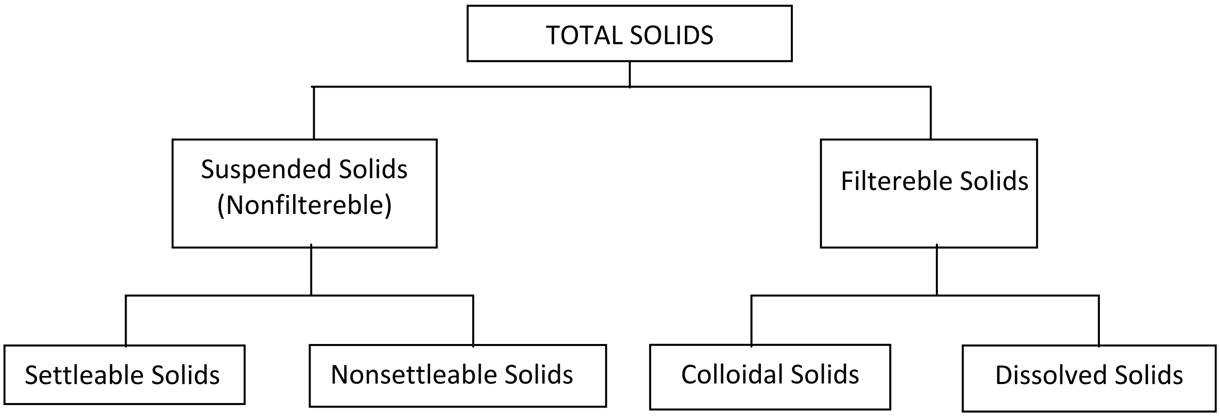


Fig.1. Solids Classification

C. WHY ARE TOTAL SOLIDS IMPORTANT?

The concentration of total dissolved solids affects the water balance in the cells of aquatic organisms. An organism placed in water with a very low level of solids, such as distilled water, will swell up because water will tend to move into its cells, which have a higher concentration of solids. An organism placed in water with a high concentration of solids will shrink somewhat because the water in its cells will tend to move out. This will in turn affect the organism's ability to maintain the proper cell density, making it difficult to keep its position in the water column. It might float up or sink down to a depth to which it is not adapted, and it might not survive.

Higher concentrations of suspended solids can serve as carriers of toxics, which readily cling to suspended particles. This is particularly a concern where pesticides are being used on irrigated crops. Where solids are high, pesticide concentrations may increase well beyond those of the original application as the irrigation water travels down irrigation ditches. Higher levels of solids can also clog irrigation devices and might become so high that irrigated plant roots will lose water rather than gain it.

A high concentration of total solids will make drinking water unpalatable and might have an adverse effect on people who are not used to drinking such water. Levels of total solids that are too high or too low can also reduce the efficiency of wastewater treatment plants, as well as the operation of industrial processes that use raw water.

Total solids also affect water clarity. Higher solids decrease the passage of light through water, thereby slowing photosynthesis by aquatic plants. Water will heat up more rapidly and hold more heat; this, in turn, might adversely affect aquatic life that has adapted to a lower temperature regime.

D. APPARATUS AND MATERIALS

1- Analytical balance
2- Drying oven, 103ºC
3- Muffle furnace, 550±50ºC
4- 0.45 µm diameter filter
5- Filter holder, membrane filter funnel, filteration flask, vacuum pumb
6- Forceps and Tongs
7- Evaporating dishes, porcelain, 100 mL volume.
8- Heating water bath
9- Hot-plate
10- Desiccator
11- Imhoff Cones
12- Crucibles, 10 mL volume.

E. TOTAL SOLIDS ANALYSIS PROCEDURE

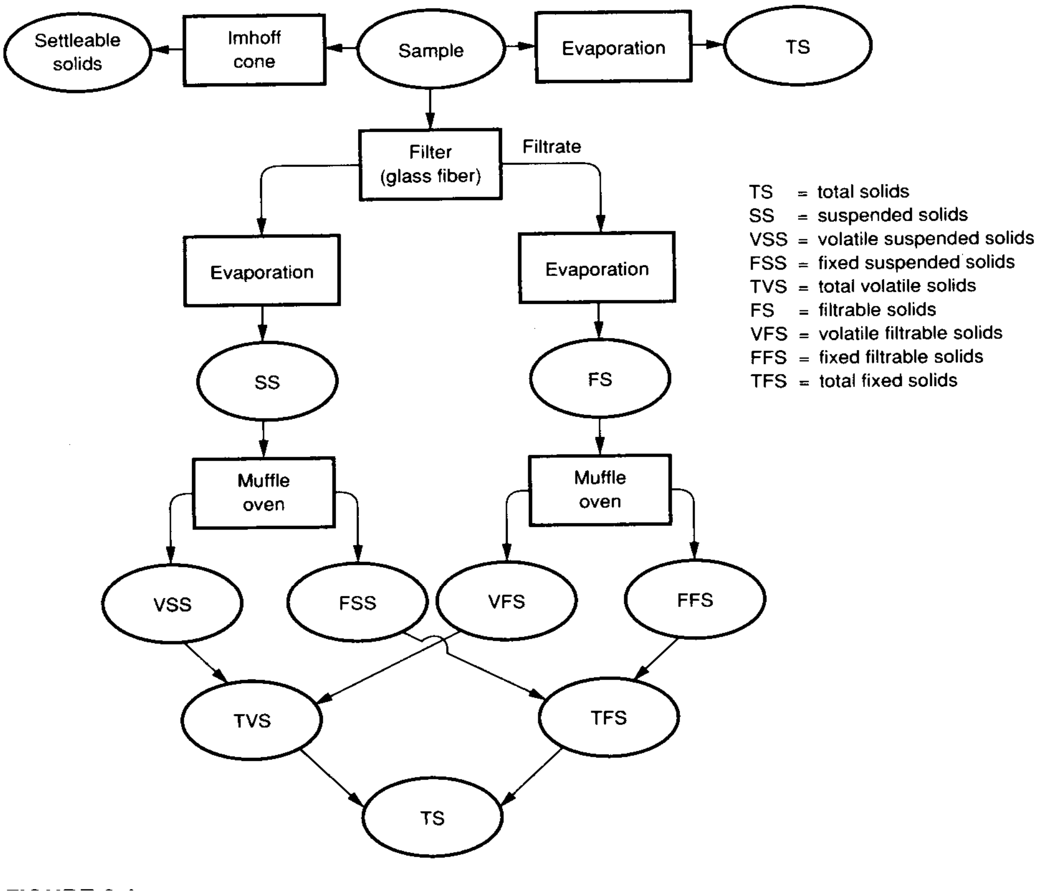


Fig. 2. Different types of solids

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 (Fig.2). 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 2.0 μm (or smaller) nominal pore size filter. Suspended solids is the portion retained on the filter. Fixed solids is the term applied to the residue of total, suspended, or dissolved solids remaining after combustion at 500 °C. The weight lost during combustion is referred to as volatile solids. Fixed and volatile may not be the best measure of inorganic or organic material. For example, the loss of mass during combustion is not confined to organic material, and may include the decomposition or volatilization of some mineral salts. More appropriate methods of characterizing organic material include total organic carbon (Standard Method 5310), BOD (Standard Method 5210), and COD (Standard Method 5220). More appropriate methods of characterizing inorganic material include alkalinity, hardness, and chromotography techniques for the analysis of specific constituents.

Gravimetric analysis is based on the determination of constituents or categories of materials by measurement of their weight. In this experiment, filtration, evaporation and combustion operations are illustrated in addition to weighing.

Filtration is used to separate the suspended solids from the dissolved portion of polluted water. Glass-fiber filters are used for this purpose.

Evaporation is used to separate water from dissolved or suspended solids. Evaporation of water and wastewaters is usually done at 103-105 ºC at which decomposition and volatilization of carbonates and chlorides are not seen.

Combustion is used to determine the organic portion of dissolved or suspended solids. The standard procedure is to conduct ignitions at 550±50 ºC. This is about the lowest temperature at which organic matter is converted to carbon dioxide and water.

1- Total solids is the term applied to the material residue left in a vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature (either 103 or 180 °C). Firstly, weigh the empty crucible in analytical balance. Using pipette transfer of unfiltered sample in the crucible. Switch on the oven and allowed to reach 105 ºC. Check and regulate oven or heating water bath furnace temprature frequently to maintain the desired temprature range. Transfer a measured aliquot of sample to the pre-weighed dish and evaporate to dryness on a steam bath or in a drying oven during evaporation or boiling. Dry the evaporated sample in the oven for at least one hour at 103-105 ºC. Cool in a desiccator and weigh until a constant weight is obtained. Keep desicator cover greased with the appropriate type of lubriant in order to seal the desiccator and prevent moisture from entering the desiccator as the test glassware cools (Fig.3).

If volatile residue is to be measured, heat at 550±50 ºC for one hour in a muffle furnace. If only total solids are to be measured, heat the clean crucible to 103-105 ºC for one hour. Cool in a desiccator and weigh until a constant weight is obtained.

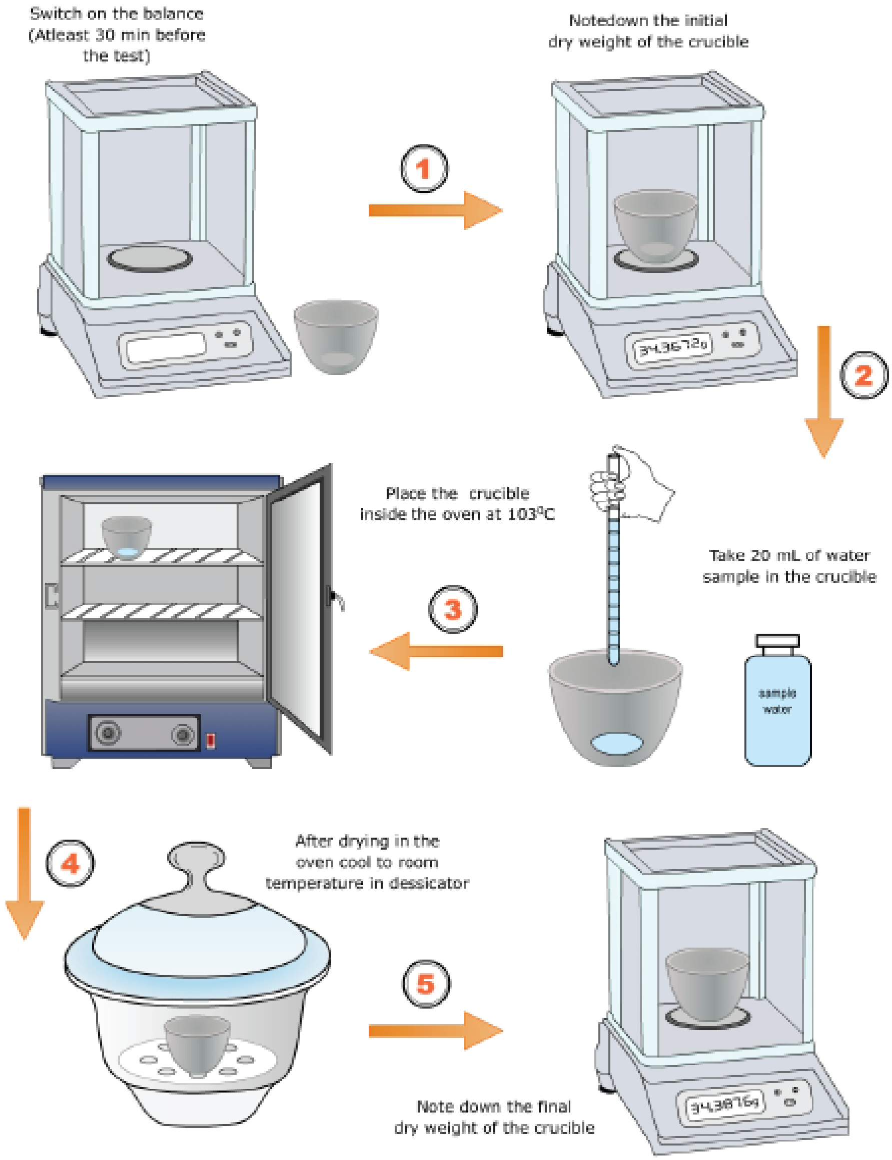


Fig. 3. Analysis of total solid

Use the following calculation to determine TS:

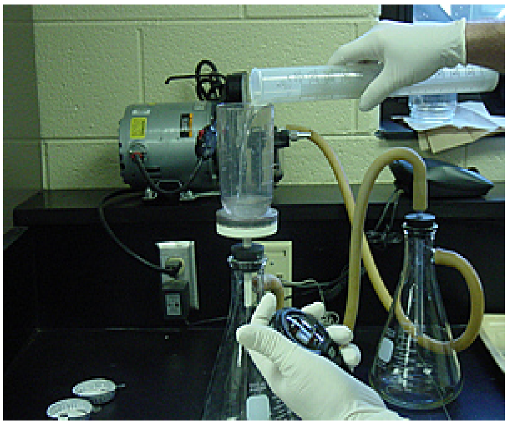
$$\text{TS (mg/L)} = \frac{(Residue + crucible)(mg) - Crucible(mg)}{Samplevolume(mL)} \times 1000(\frac{mL}{L})$$

2- Total Suspended Solids

Firstly, you should weight 0,45 µm membrane filter. Place the glass fiber filter on the membrane filter apparatus with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus and dry in an oven at 103-105 ºC for one hour. Cool in a desiccator and weigh until a constant weight is obtained (Fig. 4).

Weigh 0,45 µm membran filter

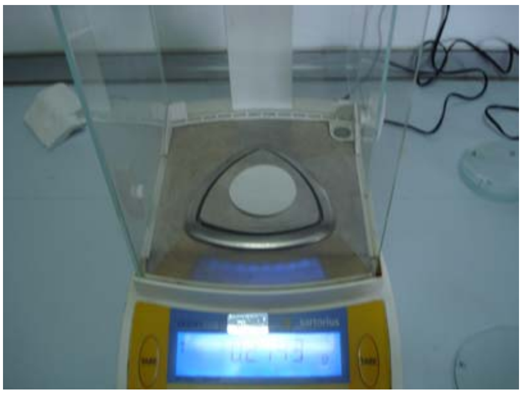
Place filter on the membrane filter apparatus



Membrane filter apparatus

Dry in oven

Cool in desiccator



Dry weight of the filter

Fig. 4. Analysis of suspended solid

Use the following calculation to determine TSS:

$$\text{TSS (mg/L)} = \frac{(\text{Residue} + \textit{filter})(mg) - \textit{Filter}(mg)}{\textit{Samplevolume}(mL)} \times 1000\left(\frac{mL}{L}\right)$$

3- Total Dissolved Solids

Preparation of evaporating dishes: If volatile residue is also to be measured, heat the clean dish to 550±50ºC for one hour in a muffle furnace. If only total dissolved solids are to be measured, heat the clean evaporating dish to 103-105 ºC for one hour. Cool, desiccate, weigh and store in desiccator.

Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 50 mL to the funnel by means of a 50 mL graduated cylinder. Filter the sample through glass fiber filter, rinse with three 10 mL portions of distilled water and continue to apply vacuum for about 3 min. after filtration is complete to remove as much water as possible. Transfer 50 mL of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath. Dry the evaporated sample for at least one hour at 103-105ºC. Cool in a desiccator and weigh until a constant weight is obtained.

Use the following calculation to determine TDS:

TDS (mg/L) = $\frac{(\mathrm{Re}\,sidue + evaporatingdish)(mg) - Evaporatingdish(mg)}{Samplevolume(mL)} \times 1000(\frac{mL}{L})$

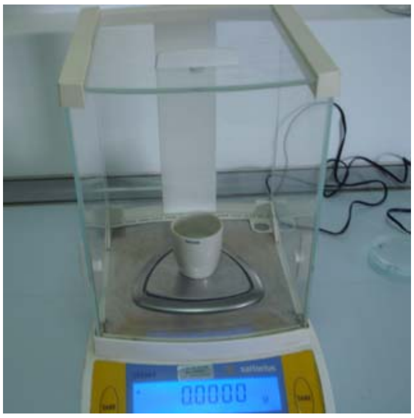
4- Volatile and Fixed Solids

Material that can be volatilized and burned off when ignited at 550 °C is classified as volatile. In general, volatile solids (VS) are presumed to be organic matter, although some organic matter will not burn and some inorganic solids break down high temperatures. Fixed solids (FS) comprise the residue that remains after a sample has been ignited. Thus, TS, TSS, and TDS are comprised of both fixed solids and volatile solids. The ratio of the VS to FS is often used to characterize the wastewater with respect to amount of organic matter present.

Ignite residues produced from Total Solids determination to constant weight in a muffle furnace at 550±50ºC (Usually 15-20 min. ignition is required). Allow the dish to cool partially in air until most of the heat has been dissipated and transfer to a desiccator for final cooling in a dry atmosphere. Weight the dish as soon as it has cooled completely. Repeat weighing until a constant weight is obtained (Fig. 5).

Use the following calculation to determine volatile solids (VS):

VS (mg/L) = $\frac{(\mathrm{Re}\,sidue + crucible)(mg) - Crucible\,\&\,solid(mg)}{Volumeofsample(mL)} \times 1000(\frac{mL}{L})$



Weigh crucible&solids   Ignite in muffle furnace 550 °C for 15-20 minutes

Cool in drying oven

Cool in desiccator

Weigh crucible plus ash

Fig. 5. Analysis of volatile solid

5- Volatile Suspended Solids

Ignite residue on filter produced from Total Suspended Solids determination at 550±50ºC to a constant weight. (Usually 15-20 min. ignition are required). Cool in desiccator and reweigh until a constant weight is obtained.

Use the following calculation to determine volatile solids (VSS):

$$\text{VSS (mg/L)} = \frac{(Residue + crucible\,\&\,ash)(mg) - Crucible(mg)}{Sample - volume(mL)} \times 1000\left(\frac{mL}{L}\right)$$

6- Settleable Solids

Settleable solids in surface and saline waters as well as domestic and industrial wastes may be determined and reported on either a volume (ml/L) or a weight (mg/L) basis. The standard test for settleable solids consists of placing a wastewater sample in a 1-liter Imhoff cone and nothing the volume of solids in millimeters that settle after a specified time period (1 h) (Fig.6). Read the graduated scale on the Imhoff cone at the top of the solids layer as mL/L settleable matter at 10 min intervals for 1 hour.

Fig. 6. Analysis of settleable solid (Imhoff cones)

CALCULATION

Suspended Solids and Volatile Suspended Solids

Sample Volume = mL

Tare mass of filter paper	
Weight of filter paper and residue remained (after 103ºC)	
Tare mass of crucible	
Weight of filter paper + crucible + filter residue (after 550 ºC)	

Total Dissolved Solids and Total Volatile Suspended Solids

Sample Volume = mL

Tare mass of evaporating dish	
Weight of dish + sample (after 103ºC)	
Weight of dish + sample (after 550 ºC)	

Total Solids and Total Volatile Solids

Sample Volume = mL

Tare mass of crucible	
Weight of crucible + sample (after 103 ºC)	
Weight of crucible + sample (after 550 ºC)	

Settling Test Data (in an Imhoff Cone for 1 hour)

Sample Volume = 1 L

t (min)	10	20	30	40	50	60
mL/L						