SOLIDS

SOLIDS

Background

The term "solids" is generally used in referring to the material suspended or dissolved in wastewater. These solids fall into two general categories, organic and inorganic. Organic solids are those materials which originally come from living plants and animals. These will include body wastes, food scraps, cooking grease, potato peelings, and sometimes even, old leather shoes. Inorganic solids are made of substances which were never living, such as gravel, salt, metal nuts and bolts, etc..

The amount of solids in a wastewater is frequently used in describing the strength of the waste. The more solids present in a particular wastewater, the stronger that wastewater will be.

Normal domestic wastewater contains a very small amount of solids when compared to the amount of water that carries it, usually less than 0.1%. This can be misleading, however, because it takes only a very small amount of solids to create large pollution problems. The number and severity of pollution problems will depend on the type of solids that are involved.

As a general rule, large quantities of organic solids will create more pollution problems than will the same quantity of inorganic solids. Therefore, not only is it important to know how many solids are present in the system, but, also, the type of solids that are present. The test procedures for solids provide essential information about the types of solids coming into the treatment plant, the amount of (solids or strength of the influent), and whether the solids are actually being removed in the plant processes.

We perform "solids" analyses to determine the amount of solids material in wastewater. The solids test provides essential information about the types of solids coming into the treatment plant, the amount of solids or strength of the influent and how well the plant or a given process is performing.

Definitions

Fixed Solids - those solids (total, suspended or dissolved) which remain after ignition for 15 to 20 minutes time at 550° ± 50°C. Those are also commonly referred to as ash. In general, fixed solids are made up of inorganic material.

Settleable Solids - the term applied to the material settling out of a sample within a one hour period. Settleable solids may include floating material depending on the technique used in the test.

Total Dissolved Solids - this term refers to those solids which will pass through a standard glass fiber filter.

Total Solids - the term applied to the material left in a dish after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids include "Total Suspended Solids" and "Total Dissolved Solids".

Total Suspended Solids - those solids which will not pass through a standard glass fiber filter. This will include both those solids that will settle or float in the clarifier and the lighter non-settleable solids (also referred to as "non-filterable residue").

Volatile Solids - those solids which are lost during ignition (in a sense, by burning) for 15-20 minutes at 550° ± 50°C. In general volatile solids are made up of organic material.

We will also discuss in this section:

Sampling - Samples must be taken from sample points that provide well-mixed, representatives samples. For composite samples, individual sample volumes (should be) proportional to the flow rate at the time the sample is taken. When pouring a sample into a graduated cylinder, it should be mixed or stirred well and poured in such a manner that the solids will not settle out before pouring is completed. Large solids, such as pieces of wood, should be removed from the sample. It is highly recommended that samples with large solids, such as the influent, be well blended before performing the analysis. Samples collected for the analysis of settled sludge volume must be fresh grab samples.

Settled sludge volume - a test that imitates but does not duplicate the action of the clarifier. Activated sludge from the aeration tank of a secondary plant is allowed to settle out in a graduated vessel. At specified time intervals, the level of the sludge is read.

Spin Testing - Using a centrifuge and a conversion factor, quick, process control-type measurements can be made on aeration tanks, return sludges and waste sludges.

TOTAL SOLIDS METHODS

Equipment

porcelain, platinum, or Vycor evaporating dishes (9 cm)
drying oven
desiccator
analytical (4-place) balance
25 ml graduated cylinder
muffle furnace
wide-bore pipets
heat resistant gloves/tongs
hot pad

Temperature

103°C to 105°C for water low in organic material such as streams and secondary effluents.

180°C for water high in organic matter such as influents and primary effluents.

Procedure

1) Prepare dishes--wash with soap and hot water, rinse with tap water, 10% HCL acid soak, tap water rinse, distilled water rinse two times.

2) Dry in oven a temperature to be used, overnight.

3) If doing fixed total solids (fired in furnace), the dishes must be fired for one hour then put in the oven for one hour.

4) Cool in desiccator.

5) Weigh.

6) Return to drying oven at 103° to 105° for at least one hour.

7) Return to desiccator to cool to room temperature.

8) Re-weigh. Weight should be within 0.5 mg of initial weight. Re-dry and re-weigh if not within 0.5 mg.

9) Pour measured sample into the dish (enough to yield a residue between 10 and 200 mg).

10) Place in oven and dry at approximately 98°C until all liquid has evaporated (or use steam bath). Bring oven to proper temperature of 103 to 105°C and dry overnight.

11) Cool dishes in desiccator to room temperature.

12) Weigh.

13) Return to drying oven at 103° to 105°C.

14) Return to desiccator for one hour.

15) Re-weigh. Again, the weights must be within 0.5 mg or the dish must be re-dried and re-weight until a constant weight is achieved.

STEPS 16-21 are for Fixed Solids

If **"fixed"** total solids are desired, at this point:

16) Place the dishes in muffle furnace at 550°C for 15 minutes to one hour.

17) Remove and cool in air until most of the heat has dissipated.

18) Transfer to desiccator, cool to room temperature.

19) Weigh.

20) Return to oven for at least one hour.

21) Redesiccate for one hour and re-weigh to be sure of constant weight.

Calculations

$$\text{Total Solids mg/l} = \frac{(A-B) \times 1000}{\text{sample volume, ml}}$$

Where: A = weight of filter + residue (in mg.)
B = weight of filter (in mg.)

$$\text{Total Volatile Solids mg/l} = \frac{(A-B) \times 1000}{\text{sample volume, ml}}$$

Calculations (continued)

$$\text{Total Fixed Solids mg/l} = \frac{(B-C) \times 1000}{\text{sample volume, ml}}$$

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

The potential sources of errors are from temperature variations. It is important to monitor the drying oven and muffle furnace temperatures closely, and from non-homogenous samples. Be sure the samples are well-mixed before pouring out for analysis. A blank (distilled water only) should be run with each analysis. The blank should be treated in exactly the same manner as the actual sample.

Quality Control for
TOTAL SOLIDS
STANDARD METHOD #2540-B

Document

Sampling (Grab or Composite)
- Grab - Exact Time and Date Sampled
- Composite - Flow Proportioning Date
- Grab or Composite -
  - Volume
  - Location

Glassware and Equipment
- Evaporating dish
  - Type:
  - Method of Cleaning:
    - 1 hour at 500° ± 50°C for volatile solids
    - 1 hour at 103 - 105°C for only total solids

Oven Temperature
- Verify oven temperature at least twice per drying cycle

Duplication Schedule
- Analysis should be run in replicate (100%) - Duplicates should be within 5% of their average.

Blank Result -
- If not zero check for error.

The troubleshooting guide and references can be found at the end of the Solids Section.

REPORTING TOTAL SOLIDS RESULTS

TOTAL SOLIDS BENCH SHEET
ANALYST:
EXACT TIME AND DATE SAMPLE WAS COLLECTED:
EXACT TIME AND DATE SAMPLE WAS ANALYZED:
SAMPLE LOCATION:
SAMPLE VOLUME (mls):
WEIGHT OF EVAPORATED DISH and DRIED RESIDUE IN GRAMS (after evaporation):
WEIGHT OF EMPTY DISH ONLY (after evaporation):
WEIGHT OF DISH and DRIED RESIDUE minus (-) WEIGHT OF DISH ONLY - RESULT IN mg/L:
**** SHOW ALL CALCULATIONS AND RAW DATA:** Blank Data All weighings to establish constant weight Duplicate or Replicate Data

TOTAL SUSPENDED SOLIDS
Method 2540 D

Equipment

gooch crucibles - used for influents, primary effluents and effluent
buchner funnel (or equivalent) - used for sludges, mixed liquors & final effluent
drying oven (103°C to 105°C)
desiccator
analytical (4-place balance)
assorted graduated cylinders
muffle furnace (if interested in "Fixed" Solids)
glass fiber filters to fit gooch crucibles or buchner funnels - Whatman 934AH; Gelman type A/E; Millipore type AP4O or equivalent filters
vacuum pump with water traps
wash bottle with distilled water

Temperature

103°C to 105°C

Procedure (The procedure for Buchner funnels is identical).

1) Prepare crucibles and/or funnels--wash with hot soapy water, rinse with tap water, 10% HCL acid soak, tap water rinse, distilled water rinse 2 or 3 times.

2) Seat the filters in the filtering apparatus, wrinkled side up.

3) Rinse the crucibles down three times with about 25 mls of distilled water.

4) Dry for at least 1 hour at 103°C to 105°C.

5) If crucibles are to be fired for "fixed" solids, place crucibles in muffle furnace for one hour at 550°C.

6) Transfer to oven for at least one hour.

7) Cool in desiccator for 15 to 30 minutes.

8) Weigh.

8a) Transfer to oven for at least one hour.

9) Re-desiccate for 15 to 30 minutes.

10) Re-weigh to ensure a constant weight; the second weighing should be within 0.5 mg of the first.

11) Select sample volume, pour sample through filter.

12) Rinse graduated cylinder into the crucible and rinse the sides of the crucible with distilled water.

13) Dry in oven at 103°C to 105°C for at least one hour, longer time may be required for sludges.

14) Cool to room temperature in desiccator.

15) Weigh.

16) Place back into drying oven for at least one hour.

17) Place in desiccator until cooled to room temperature.

18) Re-weigh; if more than 0.5 mg difference from the previous weighing, put back in oven for 1 hour and repeat desiccation and weighing steps until constant weight is reached.

19) If "fixed" suspended solids are desired, the crucibles may be treated as the dishes for fixed total solids.

Calculations

$$\text{TSS in mg/l} = \frac{\text{final weighing - initial weighing}}{\text{sample size in mls}} \times 1{,}000{,}000$$

"Fixed Suspended Solids"

$$\text{"Fixed Suspended Solids mg/l} = \frac{(A - B) \times 1{,}000{,}000}{\text{sample volume (mls)}}$$

where: A = dry weight (in grams) of the suspended solids and filter after ignition at 550°C for 15 minutes.

B = dry weight (in grams) of the suspended solids and filter before ignition at 550°C for 15 minutes.

Volatile Suspended Solids

VSS mg/l = TSS - "Fixed" Suspended Solids

It is very important that constant weights are achieved. If errors are allowed to accumulate in weighings, the large multiplication factors involved make for large errors in the final results. Temperatures are also very important for the accurate analysis of total suspended solids. Be sure to monitor the oven and furnace temperatures closely. Trying to filter too much sample through the filter will also lead to problems. Successive layers of solids on the filter will lead to smaller effective pore sizes because of the "mat" formed by the solids themselves. Avoid this problem by using smaller sample volumes. If the sample filters quickly, more can be measured out and added.

TOTAL SUSPENDED SOLIDS
TROUBLESHOOTING GUIDE

PROBLEM	MOST LIKELY CAUSE	SOLUTION
Can't get constant weight (readings consistently vary by more than .5 mg)	Insufficient drying of filter and/or crucible.	Allow more time in drying oven before transferring to desiccator.
	Filter and/or crucible not at ambient (room) temperature when weighed.	Allow more time in desiccator before weighing.
	Desiccant is bad.	Replace or rejuvenate (by drying in oven) the desiccant. It is smart to use an indicator drying agent in the desiccator that changes color when the desiccant is going bad.
	Balance not properly zeroed before each weighing.	Always level and zero the balance before weighing anything.
Filter clogs before entire sample volume is poured through.	Too much sample used.	Reduce sample volume.
Filter develops holes when vacuum is applied.	Too much vacuum pressure.	Reduce vacuum pressure/add an air trap.
Replicate results are not within 10% of each other.	Sample not thoroughly shaken before being poured off.	Always shake the sample just before pouring off to assure a good homogeneous mix. Also rinse all measuring devices into filter apparatus and rinse funnel crucible thoroughly.
Filter weighs less after sample is filtered than it did before sample had been filtered through it.	Insufficient drying time initially. Constant weight not achieved.	Do not proceed with analysis until constant weight has been achieved.

Quality Control for
TOTAL SUSPENDED SOLIDS

Standard Method #2540-D

Document

Sample Collection

Duration of Composite
Flow Proportioning
Include proportioning factor (i.e. 100 mls/1000 gallons)
Flow measuring device calibration
Collection device
Refrigeration of Sample During and After Compositing Period
Location
Representative Nature
Volume - sufficient to produce > 2.5 mg residue
Hold Time - max 7 days at 4°C

Equipment

Size and Type of Filtering Apparatus
Size and Type Filter
Calibration of 4 Place Analytical Balance
Constant Temperature of 103 to105°C in Drying Oven

Glassware

Class A Graduated
Clean and Rinsed with distilled H_2O

Procedure

Establish constant weight of filter (& crucible) before and after sample filtration
Thorough rinse of measuring glassware and funnel onto filter
Desiccant conditions

Blank and Duplicate Analysis Schedule

100 % replicate required
5% duplicate (minimum)

100 % Blank suggested
Report Value to appropriate significant figure.

REPORTING TOTAL SUSPENDED SOLIDS RESULTS

TOTAL SUSPENDED SOLIDS BENCH SHEET
ANALYST:
EXACT TIME AND DATE SAMPLE WAS COLLECTED:
EXACT TIME AND DATE SAMPLE WAS ANALYZED:
SAMPLE LOCATION:
SAMPLE VOLUME (*mls*):
AVERAGE INITIAL WEIGHT OF CRUCIBLE AND/OR FILTER (before filtering sample):
AVERAGE FINAL WEIGHT OF CRUCIBLE AND/OR FILTER (after filtering sample):
AVERAGE FINAL WEIGHT MINUS (-) AVERAGE INITIAL WEIGHT:
RESULT IN *mg/L*:
**** SHOW ALL CALCULATIONS AND RAW DATA:** Blank Data All weighings to establish constant weight Duplicate or Replicate Data NOTE: For Volatile or "Fixed" Suspended Solids record the weight of the container and dried residue prior to ignitions, the weight of the container and dried residue after ignition in addition to all the TSS data. Show all calculations.

SETTLED SLUDGE VOLUME

Equipment

Mallory settleometer, a glass or plastic cylinder approximately 7 inches high x 5 inches diameter, a graduated cylinder will not suffice because of the great friction of the cylinder walls.
Paddle (a section of license plate will do)
Timer

Procedure

1) Take at least a two liter sample with as little agitation as possible. Keep out of direct sunlight and run immediately.

2) Pour well-mixed sample into the settleometer.

3) Using the paddle, mix well by turning back and forth then still the solution by holding the paddle motionless and remove the paddle.

4) Immediately set the timer for five minutes and observe the way in which the sludge floc settles. It is important to note the quality of the supernate, is it turbid or clear?, the way the sludge particles floc together, etc.

5) At five minute intervals, read the level of the top of the floc blanket and record. After 30 minutes, the levels can be read every 10 minutes until 60 minutes.

6) Plot these readings on a graph of Settled Sludge Volume (SSV) versus time.

Calculation

From the SSV test, much can be learned. The SVI, or sludge volume index, can be calculated from the settled sludge volume at 30 minutes and the aeration tank suspended solids (MLSS):

$$SVI = \frac{SSV_{30}}{MLSS, mg/l} \times 1{,}000$$

An SVI of less than 80 is indicative of an old, fast settling sludge, of between 80 and 150 is a normal sludge, and above 150 is a young, slower settling sludge.

More useful than the SVI is the information that can be gained by keeping daily graphs of SSV vs. time plotted on tracing paper which is laid over the master graph. By laying a week's graphs over one another, trends can be spotted in the settling characteristics of the sludges. If the lines move steadily lower, the sludge is settling faster, steadily higher indicates the sludge is settling more slowly.

If your sludge is settling very poorly, that is, if the 30 minute reading is greater than 800 or so, there are two possible causes. There may be excessive filamentous bacteria present or the concentration of solids is in the mixed liquor may be too high. A microscopic examination of the sludge will reveal whether or not excessive filaments is the problem. If this is not the case, a variation on the settled sludge volume test will indicate if a high solids concentration is the problem.

Unchlorinated plant effluent should be added to the settleometer to the 500 ml mark. Mixed liquor is then added to the 1,000 ml mark and the test performed on this 50% dilution is described above. If the sludge settles at a rate greater than twice that of the undiluted sample, excessive solids concentration in the mixed liquor is responsible for the poor settling.

For example, if the undiluted 30 minutes SSV is 900 and the 50% dilution 30 minute SSV is 375, a high solids concentration in the mixed liquor is indicated.

Sources of Error

Samples for SSV testing must be very fresh, any delay in testing will affect the health of the micro-organisms and greatly affect the way the sludge flocs and settles. Samples must be stilled before starting the test. Even very slow circular currents can greatly accelerate the settling rate. Finally, the settleometer must be kept out of direct sunlight because of the effect on settling of convection currents set up by the sun's heat.

QC - Record % solids at 5 minute intervals. Use proper volume.

SETTLEABLE SOLIDS

Equipment

Imhoff cones, 1 liter
2 foot glass stirring rod
Timer

Procedure

1) Take at least a 1 liter sample, this can come from composites or grab samples. (consult your permit)

2) Mix the sample well.

3) Pour into the Imhoff cone to the 1 liter mark.

4) Set timer for 45 minutes.

5) At the end of this period, run the glass rod gently around the edge of the cone to dislodge solids adhering there, or turn cone slowly, one revolution.

6) Set timer for 15 minutes.

7) Read volume after these 15 minutes (total settling time = 1 hour), allowing for any voids in the solids layer and subtracting them from the total reading.

Error may be introduced into this procedure as with the SSV test, by the action of currents due to sunlight or very cold samples in warm rooms. Failure to allow for the voids in the samples leads to inaccurate readings.

Quality Control for
SETTLEABLE SOLIDS
Standard Method #2540-F

Document

Sampling

Grab - Time and Date Collected
Time and Date Analyzed

HOLD TIME - 48 hours (max)

Location

Glassware

Rinse after each use

Shake sample thoroughly before pouring off.

Procedure

Settle 45 minutes
Gently dislodge solids adhering to glass
Settle 15 minutes longer
Account for voids
Record results ml/l

SPIN TESTING

The centrifuge can be a helpful tool in solids testing. The solids content of any sample thick in solids such as mixed liquors, return sludges, and waste sludges can be approximated by centrifugation. It is necessary that the centrifuge be calibrated against the balance procedure weekly to use this method.

Equipment

Centrifuge
15 ml graduated centrifuge tube
Timer

Procedure

1) Pour off duplicate tubes and load in centrifuge on opposite sides (the centrifuge must be loaded evenly).

2) Turn on and set timer for 15 minutes.

3) Shut down and read level of solids in test tubes.

Calculation

The tube readings are multiplied by the factor established from the weighing calibration procedure which should be done on a weekly basis. The same sample should be run both by the spin and the suspended solids methods. The result obtained from the suspended solids test should be divided by the spin test result for the factor:

$$\text{Factor} = \frac{\text{TSS, mg/l}}{\text{spin test, mls or \%}}$$

This factor should be used for all testing during that week. Because the sludge's composition varies so much, it is recommended that separate factors be established for each different type of sludge and be established every week or two weeks.

References

Total Solids:	Standard Methods 18th Edition 2540-B Page 2-54
Fixed and Volatile Solids:	Standard Methods 18th Edition 2540-E Page 2-57
Total Suspended Solids:	Standard Methods for Examination of Water and Wastewater 18th Edition, Method 2540-D, Page 2-56
Settled Sludge Volume:	Operation of Wastewater Treatment Plants, A Field Study Training Program, Volume 2, Pages 377-379
Settleable Solids:	Standard Methods 18th Edition 2540-F Page 2-57
Spin Testing:	Operations of Wastewater Treatment Plants, A Field Study Training Program, Volume 2, Page 367