

Solids

**VT WSMD Wastewater Program Lab Manual Section
#10**

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Preface to 2021 Update

With some notable exceptions, the methods for “solids” analyses have remained somewhat the same since the 1990 version of the manual was prepared. Some significant differences include the permit limits imposed and the very low results being reported, especially regarding the Total Suspended Solids (TSS) analysis. Whereas it is possible to achieve accurate results down to 2.5 mg/L TSS using graduated glassware as described in Standard Methods For the Examination of Water and Wastewater, results less than 2.5 mg/L are not considered valid unless volumetric glassware/measuring device is used. Also, increased sample volume may be necessary to generate accurate results less than 2.5 mg/L. This will be described in detail at the end of the Total Suspended Solids section of this manual. One other notable change in the TSS section in the 23rd edition of Standard Methods is the formula used for calculation of TSS. NOTE that in the 23rd edition the formula is:

$$TSS \left(\frac{mg}{L} \right) = \frac{((\text{Weight of Filter and residue in mg}) - (\text{weight of filter in mg})) * 1000}{\text{Volume of Sample (mL)}}$$

Notice the difference between this and the formula from the original manual:

$$TSS \left(\frac{mg}{L} \right) = \frac{((\text{weight of filter and residue in g}) - (\text{weight of filter in g})) * 1,000,000}{\text{Volume of Sample (mL)}}$$

In order to use the “new” formula you must first convert the weights from grams to milligrams

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 concentration 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 concentration 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 concentration of solids to create large pollution problems. The number and severity of pollution problems will depend on the type of solids that are involved.

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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 concentration 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 degrees C +/- 50 degrees C. Those are also commonly referred to as ash. In general, fixed solids are thought to be made up of inorganic material, although some inorganic material can be lost during ignition.

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 with a pore size of 2 microns or less.

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 with a pore size of 1.5- 2 microns. 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°C +/- 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, representative 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

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that samples with large solids, such as the influent, be well-mixed 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 high-temperature glass 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 at 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°C to 105°C 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).

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- 10) Place in oven and dry at approximately 98°C until all liquid has evaporated.
- 11) Bring oven to proper temperature of 103°C to 105°C and dry overnight.
- 12) Cool dishes in desiccator to room temperature.
- 13) Weigh.
- 14) Return to drying oven at 103°C to 105°C for approximately one hour.
- 15) Return to desiccator for one hour.
- 16) Re-weigh. The weights must be within 0.5 mg or the dish must be re-dried and re-weigh until a constant weight is achieved.

- **STEPS 16-21 for Fixed Solids.** If fixed total solids are desired:

- 17) Place the dishes in muffle furnace at 550°C for 15 minutes to one hour.
- 18) Remove and cool in air until most of the heat has dissipated.
- 19) Transfer to desiccator, cool to room temperature.
- 20) Weigh.
- 21) Return to oven for at least one hour.
- 22) Re-desiccate for one hour and re-weigh to be sure of constant weight.

Calculations

$$\text{Total solids } \left(\frac{\text{mg}}{\text{L}} \right) = \frac{(A - B) * 1000}{\text{Sample Volume (mL)}}$$

Where:

- A = weight of filter/dish and residue in mg
- B = weight of filter/dish in mg

$$\text{Total Volatile Solids } \left(\frac{\text{mg}}{\text{L}} \right) = \frac{(C - D) * 1000}{\text{Sample Volume (mL)}}$$

$$\text{Total Fixed Solids } \left(\frac{\text{mg}}{\text{L}} \right) = \frac{(D - E) * 1000}{\text{Sample Volume (mL)}}$$

Where:

- C = weight of residue and dish before ignition in mg
- D = weight of residue and dish or filter after ignition in mg
- E = weight of dish or filter in mg

The potential sources of errors are from temperature variations and from non-homogenous samples. It is important to monitor the drying oven and muffle furnace temperatures closely and 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 the same manner as the actual sample.

Quality Control for Total Solids Standard Method #2540-B

Document the Following (Including but not limited to):

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°C +/- 50°C for volatile solids
 - 1 hour at 103°C - 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%) - Replicates 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 (mL):	
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: Standard Method #2540-D

The method for Total Suspended Solids has not changed significantly since the last version of this manual was written in 1990. However, many operators are apparently interested in determining just how "low they can go" regarding the reporting of Total Suspended Solids results. Results are often reported as <2 mg/L TSS. Results below 2.5 mg/L are obtainable but in order to achieve these results the use of volumetric glassware/equipment for measurement of sample volumes is required. AND since the method requires selection of a sample volume to produce between 2.5 and 200 mg dried residue the volume of sample may have to be increased substantially. The bottom line is this: Reporting TSS results of <2 mg/L is technically invalid because in order to obtain such results using one liter of sample the required minimum 2.5 mg of dried residue cannot have been satisfied. HOWEVER: By using volumes in excess of 1000 milliliters lower results can be reported. This will be explained in detail at the end of this section.

Equipment

- Gooch crucibles – sometimes used for influents, primary effluents
- Buchner funnel (or equivalent) - used for sludges, mixed liquors, influent and 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 AP40 or equivalent filters
- vacuum pump with water traps
- wash bottle with distilled water

Temperature

- 103°C to 105°C

Procedure

- 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/funnels down three times with about 25 mL 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.
- 9) Transfer to oven for at least one hour.
- 10) Re-desiccate for 15 to 30 minutes (the same time in desiccator should be used as for the first weighing).

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- 11) Re-weigh to ensure a constant weight; the second weighing should be within 0.5 mg of the first.
- 12) Select sample volume, pour sample through filter.
- 13) Rinse graduated cylinder into the crucible and rinse the sides of the crucible/funnel with distilled water.
- 14) Dry in oven at 103°C to 105°C for at least one hour (longer time may be required for sludges).
- 15) Cool to room temperature in desiccator. (15-30 minutes, same each time)
- 16) Weigh.
- 17) Place back into drying oven for at least one hour.
- 18) Place in desiccator until cooled to room temperature. (15-30 minutes, same each time)
- 19) 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.
- 20) If "fixed" suspended solids are desired, the crucibles may be treated as the dishes for fixed total solids.

Calculations

$$TSS \left(\frac{mg}{L} \right) = \frac{(Initial\ Weighing\ (mg) - Final\ Weighing\ (mg)) * 1000}{Sample\ Volume\ (mL)}$$

$$Fixed\ Suspended\ Solids \left(\frac{mg}{L} \right) = \frac{(A - B) * 1000}{Sample\ Volume\ (mL)}$$

Where:

- A = Dry Weight in mg of the suspended solids and filter **after** ignition at 550°C for 15 minutes
- B = Dry Weight in mg of the suspended solids and filter **before** ignition at 550°C for 15 minutes

$$Volatile\ Suspended\ Solids\ (VSS) \left(\frac{mg}{L} \right) = TSS \left(\frac{mg}{L} \right) - Fixed\ Suspended\ Solids \left(\frac{mg}{L} \right)$$

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)	<p>Insufficient drying of filter and/or crucible.</p> <p>Filter and/or crucible not at ambient (room) temperature when weighed.</p> <p>Desiccant is bad.</p> <p>Balance not properly zeroed before <u>each</u> weighing.</p>	<p>Allow more time in drying oven before transferring to desiccator.</p> <p>Allow more time in desiccator before weighing.</p> <p>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.</p> <p>Always level and zero the balance before weighing anything.</p>
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 the Following (Including but not limited to):

Sample Collection

- Duration of Composite
- Flow Proportioning
- Include proportioning factor (i.e., 100 mL/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 to 105°C in Drying Oven

Glassware

- Class A measuring device or auto pipettor
- Clean and Rinse with distilled H₂O

Procedure

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

Blank and Duplicate Analysis Schedule

- 10 % replicate required
- 5% duplicate (minimum)
- 100 % Blank (distilled water) 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 (mL):
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:
<p>** SHOW ALL CALCULATIONS AND RAW DATA:</p> <p>Blank Data</p> <p>All weighings to establish constant weight</p> <p>Duplicate or Replicate Data</p> <p>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.</p>

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
- 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, turbid or clear, the manner in which 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):

$$SVI = \frac{SSV_{30}}{MLSS \left(\frac{mg}{L} \right)} * 1000$$

An SVI of less than 80 is indicative of an old, fast settling sludge, 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 mL or so, there are two possible causes. There may be excessive filamentous bacteria present or the concentration of solids in the mixed liquor may be too high. A microscopic examination of the sludge will reveal whether or not excessive filaments are 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

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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 mL and the 50% dilution 30-minute SSV is 375 mL, 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 still 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 volumes.

Settleable Solids

Equipment

- Imhoff cones, 1 liter
- 2-foot glass or plastic 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 the Following (Including by not limited to):

Sampling

- Grab –
 - Time and Date Collected
 - Time and Date Analyzed
 - **HOLD TIME - 48 hours (max)**
- Location

Glassware

- Wash and Rinse after each use

Procedure

- Settle 45 minutes
- Gently dislodge solids adhering to sides of cone
- 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 tubes
- Timer

Procedure

- 1) Shake sample thoroughly before pouring off
- 2) Pour off duplicate tubes and load in centrifuge on opposite sides (the centrifuge must be loaded evenly).
- 3) Turn on and set timer for 15 minutes.
- 4) 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

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suspended solids methods. The result obtained from the suspended solids test should be divided by the spin test result for the factor:

$$Factor = \frac{TSS \left(\frac{mg}{L} \right)}{Spin Test (mL)}$$

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 for the Examination of Water and Wastewater 23 rd Edition 2540-B
Fixed and Volatile Solids:	Standard Methods for the Examination of Water and Wastewater 23 rd Edition, Method 2540-D
Total Suspended Solids:	Standard Methods for Examination of Water and Wastewater 23 rd Edition, Method 2540-D
Settled Sludge Volume:	Operation of Wastewater Treatment Plants, A Field Study Training Program, Volume 2, Pages 377-379
Settleable Solids:	Standard Methods for the Examination of Water and Wastewater 23 rd Edition 2540-F
Spin Testing:	Operations of Wastewater Treatment Plants, A Field Study Training Training Program, Volume 2, Page 367