Chapter 14  Solids

In wastewater analysis, if the water is evaporated, solids are left. These solids can be defined in numerous ways such as total solids, suspended solids, dissolved solids, settleable solids, volatile solids, and fixed solids. Total solids are defined as the material left in a container after all the water has been evaporated, usually at 103-105°C.

**Total Solids Procedure**

1. Wash an evaporating dish with DI water and dry in the drying oven at 103-105°C for a minimum of 1 hour. Cool the evaporating dish in the desiccator to balance temperature. The time in the desiccator will vary depending upon the size and number of dishes being cooled. The more dishes to be cooled, the longer the dishes must be in the desiccator prior to the initial weight. Usually a minimum of 45 minutes is needed to cool the dish to room/balance temperature. Weigh the dish on the analytical balance.

2. Transfer the evaporating dish to the analytical balance using forceps and record the initial tare weight.

3. Since this is wastewater, a graduated cylinder is usually a better choice than a pipet to measure 50 ml of sample and transfer to the evaporating dish. Shake the sample well and quickly pour into the graduated cylinder. The sample may be homogenized in a blender if necessary. Measure the volume of sample. Select a volume of sample that will have between 0.0025 - 0.2000 gm. of dry solids. The volume of sample needs to be measured accurately but does not need to be exactly 50 ml. Transfer speed is important, since solids will begin to settle as soon as mixing stops. For instance, shake and pour an influent sample into a 50 ml graduated cylinder, the volume measures 43 ml. Do not try to add more sample to reach the 50 ml mark, since solids in the sample have now begun to settle.

4. Pour the sample in the graduated cylinder into the evaporating dish. Rinse the graduated cylinder with small volumes of DI water and transfer to the evaporating dish.

5. Using tongs, place the dish in the drying oven. If spattering is a concern, lower the drying oven temperature to below boiling (98 °C) until the water has evaporated. After the water has evaporated, raise the temperature to 103-105°C for a minimum of 1 hour. Remove from the oven and cool to room temperature in the desiccator.

[Figure: Notice the solids that remain after evaporation.]

6. Record the 1st dry weight.

7. Return the dish to the oven, re-dry for an additional 1 hour at 103-105°C. Re-cool in the desiccator and re-weigh.

8. Record the 2nd dry weight. The difference between the first and second weighing should be <0.0005 gm. or less than 4% of the previous weighing to prove the sample was dried completely.

**Total Solids Calculations**

First dry weight + dish  =  46.9088 gm  
Second dry weight + dish  =  46.9086 gm  
Difference  =  0.0002 gm
The difference of 0.0002 gm is < 0.0005gm indicating the sample has been completely dried.

Second dry weight + dish = 46.9086 gm
Dish weight (tare) = 46.8193 gm
Difference = 0.0893 gm

The difference of 0.0893 gm is the weight of the dry solids from the 43 ml poured into the dish. The weight of 0.0893 is acceptable since it is between 0.0025 - 0.2000 gm. Weight below 0.0025 gm is statistically invalid, while a weight above 0.2000 gm will likely cause solids to form a crust which interferes with water evaporation.

Determine the total solids by using the formula:

\[
\text{Weight of dry solids} \times \frac{1000000}{\text{Volume of sample}} = \text{Weight of dry solids} \times \frac{1000000}{43 \text{ ml}} = 2080 \text{ mg/L}
\]

As discussed earlier, the total solids can be reclassified in a number of ways by changing the test procedure. Total solids can be subdivided into Total Suspended Solids (TSS) and Total Dissolved Solids (TDS). Total suspended solids are defined as the portion of the solids that are retained on a 2 um (or smaller) glass fiber filter. Total dissolved solids are defined as the portion of solids that pass through a 2 um (or smaller) glass fiber filter.

**Total Suspended Solids (TSS)**

Suspended solids are of interest in a WWTP because they indicate the effectiveness of physical and biological treatment. Solids entering a WWTP are initially separated by reducing the flow so that heavy inorganic suspended solids (grit) can settle in the grit chamber. Measuring solids before and after the grit chamber can help the operator determine the operating condition of the grit chamber. Likewise, total suspended solids can be run on wastewater entering and leaving the primary clarifier. Changes in efficiency may indicate problems with the sludge collection mechanism, changes in flow, changes in water temperature, short circuiting, etc.

Suspended solids leaving the primary clarifier are converted into settleable solids in secondary treatment processes such as trickling filters, SBRs or activated sludge. The suspended solids are digested and entrained with microorganisms as floc, which is heavy enough to settle in the secondary clarifier. Significant reduction of total suspended solids should take place between the primary and secondary clarifier effluents. Changes in the secondary clarifier TSS effluent can be an indication of problems with the secondary treatment processes.

**TSS Theory**

A well mixed sample is filtered through a pre-washed, pre-weighed, glass fiber filter. The suspended solids are retained on the filter. The filter is then rinsed, dried, and weighed. The increase in weight represents the suspended solids.

**Filter Preparation**

There are many manufacturers of satisfactory glass fiber filters. Be sure to purchase filters without organic binders (glue). The filters usually come in packages of 100 or 1000. These filters are not ready to use and must be pre-treated using the same procedure as the sample. All filters must be pre-washed and those that will be used for volatile solids must be pre-volatilized. Pre-washing and pre-volatilizing removes loose materials from the manufacturing process that will give a significantly higher answer.
1. Place the filter onto the filter holder, rough side up, screen side down. Look closely to see the screen.

![Figure: Place filter on holder rough side up.]

2. Turn on the vacuum and quickly pour three successive 20 ml portions of deionized water. Allow the water to be completely removed between each rinse, usually 1-2 minutes is sufficient.

3. Remove the filter from the filter holder and place in an aluminum weighing dish.

![Figure: Always use forceps to handle the filter.]

4. Place the filter in the drying oven at 103-105°C for a minimum of 1 hour. If volatile suspended solids are to be measured, place the filter in the muffle furnace at 550°C for 15 minutes after first drying in the drying oven. Cool in the desiccator until at room temperature.

5. Record the first dry weight.

6. Repeat the cycle of drying, volatilizing, cooling, and weighing

7. Record the second dry weight to prove the filters are dry. Dry filters should show a difference in weight of <0.0005gm.

Repeat for all filters. Store the filters in the desiccator until needed.

Note 1: This is the procedure recommended by Standard Methods and makes the assumption that the washing step is effective. The best practices procedure would be to go all the way back and prewash the filters a second time to document the fact that the washing procedure is effectively rinsing out all the particles and then redry the filters to show that the weight has not changed.

Sample Collection
TSS samples can be either composite or grab samples and can be collected in either glass or plastic. Samples should be analyzed as soon as possible or preserved by ice or refrigeration to reduce microorganism activity. Holding time is no longer than 7 days.

TSS Procedure
1. **Set-up.** Obtain a clean, dry filter funnel, flask, and vacuum hose. Warm the sample to room temperature. If necessary, the sample can be warmed quickly using a waterbath or hot plate with stirring.

2. **Tare Weight Determination.** Remove an aluminum dish containing a pre-washed filter from the desiccator. Zero the analytical balance, place the aluminum dish containing the filter on the balance using forceps. **Record the tare weight on the bench sheet.**

![Figure: Weigh the dish and filter not just the filter.]

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2. **Tare Weight Determination.** Remove an aluminum dish containing a pre-washed filter from the desiccator. Zero the analytical balance, place the aluminum dish containing the filter on the balance using forceps. **Record the tare weight on the bench sheet.**
3. Remove the filter from the aluminum dish using forceps and place on the filter funnel. Wet the filter with a small volume of reagent water to seat the filter.

4. If warming the sample on a hot plate, stir the sample with sufficient speed to provide a homogenous solution. Stirring too fast may cause particles to separate by size and weight. Pipet the desired volume from a point middepth and midway between the wall and the vortex. Do not pipet from the vortex.

Pipet the sample onto the seated glass fiber filter. Proceed to step 5.

Alternate Mixing Procedure
Shake the sample vigorously. Pour the sample quickly into a graduated cylinder to prevent solids from settling. Choose a volume of sample that will yield between 0.0025 - 0.2000 gm. Influent samples generally require less than 100 ml while clean effluent samples can use up to 1000 ml. Do not filter more than 1000 ml. Pour quickly and do not try to get a specific volume. Trying to get a specific volume will allow heavy particles to settle and have the same precision problems as stirring.

Measure the volume in the graduated cylinder. Record the volume on the bench sheet.

Pour the entire volume into the filter funnel, turn on the vacuum and filter the sample. **TIP:** If the sample does not filter within 1-2 minutes, the sample volume is too high. It will be faster to repeat the test with a smaller volume than wait for the sample to completely filter. Filtration should not take longer than 10 minutes. **TIP:** If the sample filters quickly, a second volume can be added. It is important to add enough sample to achieve a dry solids weight of at least 0.0025 gm.

5. Rinse the with 3 successive 10 ml volumes of deionized water and pour into the funnel.

Allow complete drainage between washings and continue suction for about 3 minutes.

6. Rinse the the filter funnel with deionized water to recover any solids remaining on the sides of the funnel. Turn off the vacuum.

Note 2: The mixing procedure above is from Standard Methods and by its own admission is prone to large errors. If the point of sample removal is important, the sample is by definition not homogeneous and according to Standard Methods will produce imprecise data. It is important for the technician to obtain a well mixed sample that will provide good precision.

Note 3: This procedure in Standard Methods may work well for influent samples or poor effluent samples but does not utilize best practices for medium or high quality effluent samples. The procedure requires pipetting the sample onto the filter but also indicates up to 1 liter can be filtered. To pipet 1 liter of effluent using a 25 ml serological pipet would require 40 pipettings which is poor practice. For sample volumes larger than 25 ml, a graduated cylinder should be used.

Alternate Mixing Procedure
Shake the sample vigorously. Pour the sample quickly into a graduated cylinder to prevent solids from settling. Choose a volume of sample that will yield between
**TIP:** If the vacuum is left "on" for a long period of time, the filter may stick to the funnel base causing a loss of weight when the filter is removed. This can usually be avoided by re-wetting the filter with deionized water then remove as usual.

Figure: Rinse with DI water from top to bottom.

7. Remove the filter from the filter funnel using forceps and return to the aluminum dish.

Figure: Notice that the solids have been distributed evenly throughout the filter.

**TIP:** If the filter sticks to the bottom of the funnel, replace the funnel and turn the vacuum back on. With the vacuum on, remove the funnel. Now turn off the vacuum and remove the filter with forceps. Be careful not to tear the filter. If the filter tears, repeat the analysis.

8. Place the aluminum dish in the drying oven at 104°C for a minimum of 1 hour.

**TIP:** Place the filter on the top shelf to prevent debris from dropping onto the filter.

9. Remove the dry aluminum dish and filter from the drying oven and place in the desiccator for a minimum of 15 minutes to cool to balance temperature.

10. Remove the dish and filter from the desiccator, place the dish and filter on the analytical balance. Remember to zero the balance. **Record the 1st dry weight on the bench sheet.**

11. Return the aluminum dish to the drying oven at 104°C for a minimum of 1 hour. Remove the dry aluminum dish and filter from the drying oven and place in the desiccator for a minimum of 15 minutes.

12. Remove the dish and filter from the desiccator, place the dish and filter on the analytical balance. **Record the 2nd dry weight on the bench sheet.**

If the difference between the first and second weighings is <0.0005 gm, the filter is dry. If the difference between the first and second weighing is >0.0005 gm, the filter is still wet and must be returned to the drying oven until the weight difference is <0.0005 gm.

**TSS Calculations**

\[
\text{TSS mg/L} = \frac{2^{nd} \text{ dry weight of solids (gm)} \times 1,000,000}{\text{Volume of sample (ml)}}
\]

Report results to 3 significant figures.

Example 1: The technician quickly pours 730 ml of well mixed effluent into the filter funnel. The tare weight of the filter is 1.5546 gm. After rinsing, drying, cooling, and weighing, the first dry weight is 1.5622 gm. The filter is returned to the oven and dried, cooled, and weighed. The second dry weight is 1.5619 gm. Calculate the TSS

First dry weight = 1.5622 gm
Second dry weight = 1.5619 gm
Difference = 0.0003 gm
Is the filter dry? Yes, the difference is <0.0005 gm

Second dry weight = 1.5619 gm
Tare weight = 1.5546 gm
Weight of dry solids = 0.0073 gm

Is the weight of the dry solids acceptable? Yes, the weight of solids is between 0.0025-0.2000 gm.

TSS = \( \frac{2^{nd} \text{ dry weight of solids (gm)} \times 1,000,000}{\text{Volume of sample (ml)}} \)

TSS = \( \frac{0.0073 \text{ gm} \times 1000000}{730 \text{ ml}} \) = 10 mg/L

Example 2: The technician quickly pours 41 ml of well mixed influent into the filter funnel. The tare weight of the filter is 1.4603 gm. After rinsing, drying, cooling, and weighing, the first dry weight is 1.4722 gm. The filter is returned to the oven and dried, cooled, and weighed. The second dry weight is 1.4700 gm. Calculate the TSS

First dry weight = 1.4722 gm
Second dry weight = 1.4700 gm
Difference = 0.0022 gm

Is the filter dry? No, the difference is >0.0005 gm. The filter must be returned to the drying oven and be redried until the difference is <0.0005 gm. The redried sample now weighs 1.4699

Second dry weight = 1.4699 gm
Tare weight = 1.4603 gm
Weight of dry solids = 0.0096 gm

Is the weight of the dry solids acceptable? Yes, the weight of solids is between 0.0025-0.2000 gm.

TSS = \( \frac{2^{nd} \text{ dry weight of solids (gm)} \times 1,000,000}{\text{Volume of sample (ml)}} \)

TSS = \( \frac{0.0096 \text{ gm} \times 1000000}{41 \text{ ml}} \) = 234 mg/L

### TSS Troubleshooting

TSS is generally a fairly simple test with few trouble spots. The most common mistakes involve filter preparation and sample mixing. Running duplicates on a continuous basis provides helpful information and builds confidence in the technician's procedure.

### Blanks are inconsistent

Methods blanks should be performed on a routine basis, a least once per batch of filters prepared. The method blank is the same as re-rinsing the filters with 100 ml of deionized water. If the filters have been washed properly and consistently, the filters should not lose particles and should have an initial and final dry weight difference close to zero. If there is a difference of >0.0002 gm or duplicate filters are not consistent, the filters may not have been rinsed completely. For instance, if two method blanks are run and the first drops by 0.0001 gm and the second drops by 0.0012 gm, the difference may indicate something is wrong with the way the filters were rinsed. Method blanks can be run with each set of samples to show that rinsing is effective and that sample is not being carried over.

### Not Enough Sample

The selection of the "right" sample volume is often a guess. The sample volume should ideally add a significant amount of solids to the filter to minimize any balance errors. That is why the minimum weight is supposed to be >0.0025 gm. Analytical balances have an electronic error of \( \pm 0.0001 \text{ gm} \). This uncontrollable instrumental error would be a 10% error if the amount of solids was 0.0010 gm. The error caused by the electronic noise goes down as more sample is added to the filter. When at least 0.0025 gm of solid have been added, the error is now a reasonable 4% and will get better as more weight is added. Always add as much sample as possible. If
the sample is still filtering without clogging, and the filter looks clean, add more well mixed sample.

![Figure](image1.png)

**Figure**: This cracking indicates too much sample was filtered. Reduce the volume next time.

![Figure](image2.png)

**Figure**: This filter may not have enough weight. Increase the sample volume to reduce error.

**Too Much Sample**

This is a problem just the opposite of the last one. In this case, too much sample has clogged the filter and the remaining sample is just slowly dripping. Several problems result. First, the filtration time becomes excessive. If the sample is still filtering after 10 minutes, discard the filter and repeat using a smaller volume. Second, a clogged filter will interfere with the rinsing step. The rinsing step should recover any straggler solids from the sides of the graduated cylinder and funnel and rinse out any trapped TDS. Trapped TDS will give a positive bias to the answer. Third, the excessive solids trapped on the filter will form a hard crust on the filter and may prevent water from evaporating completely. This is visible when the filter has a lot of crazing or cracking on it. This is similar to clay. Clay looks dry but when broken into pieces, the inside is still wet. This water weight will also give the sample a positive bias.

![Figure](image3.png)

**Figure**: This sample was poured slowly and results in poor distribution of the solids. High solids in one area may not dry completely.

**Duplicates are inconsistent**

TSS samples should always be run in duplicate. It takes very little time to run a second sample. If the duplicates are consistent, the technician's technique, sample handling, mixing, rinsing, etc. is validated. If the duplicates are inconsistent, sample handling problems may be present. Each sample measured should be mixed and poured completely. For instance, if 67 ml of sample has been measured, all 67 ml should be passed through the filter and rinsed. The sample should not be split, say 25 and 42 ml. While the average of the sample may be correct, the duplicate answers may be far apart. The 25 ml sample may have a TSS answer of 50 mg/L while the 42 ml sample may have a TSS of 84 mg/L. This large difference indicates the solids in the sample settled between the 25 and 42 ml pouring. It
would be better to run a 30 ml sample all at once then mix and pour a second sample of 35 ml. The answers for these two separate samples will be more consistent. Homogenizing the sample may also help improve the precision.

**ODD Stuff**
In spite of the best effort to mix the sample, some samples contain odd stuff such as bugs, twigs, grease balls, etc. These odd particles can give very positively biased samples (high answers). Standard Methods allows you the technician to decide if these materials of truly representative of the sample. If the sample contains a lot of swimming critters, then perhaps they should be included in the TSS filter. If there is only 1 or 2 mosquitos and the technician pours one out onto the filter, perhaps this mosquito should be removed. The same argument applies to grease and oil. Grease and oil stick together and cling to the top of the sample bottle. It is often difficult to adequately mix these materials, so they get pipetted or poured off into the graduated cylinder because they are normally floating at the top of the sample. Is a large grease chunk on one filter representative? Is it a grease chunk? The technician may wish to run the sample and make a note of the abnormal particle.

**Quality Control**
Decisions on the accuracy of the reported data will be based on the quality control information.

**Sample QC**
- Sample holding time cannot exceed 7 days.
  Corrective Action: Reject samples and request a resample.
- Samples must be preserved on ice or refrigeration until time of analysis. *Record the temperature of the refrigerator.*
  Corrective Action: Adjust refrigerator to below 6°C. Service the refrigerator if the temperature does not adjust properly
- Samples must be warmed to room temperature prior to TSS analysis.
  Corrective Action: Run samples in duplicate 100% of the time if possible. Use approximately the same volume of sample for both. Remix sample between tests.
- *Record sample date, time, type, sampler, date and time of analysis, analyst and method used.*
- Samples with large chunks of non-homogeneous materials should be homogenized for 1-2 minutes for better precision and accuracy. Avoid excessive homogenization which might cause volatilization of some solids.
- Samples must be mixed well and poured quickly.

**Equipment QC**
- Drying Oven must be 104°C ± 1.0°C
  Record the temperature of the drying oven.
  Corrective Action: Incubator outside the control limits must be adjusted. An oven temperature below 103 °C may not dry the sample completely. An oven temperature above 105 °C may cause some organics to volatilize.
- Immerse the oven thermometer in sand to prevent inaccurate temperature readings when the oven door is opened frequently.
- Calibrate the oven thermometer at least annually against a NIST certified thermometer. The calibration must include date, thermometer correction factor, serial number, and initials of the person performing the calibration.
Record the calibration data
- Use an analytical balance capable of weighing 0.0001 gm.
- Calibrate the analytical balance annually using a certified balance technician.
- Document date, balance condition, and name of technician and company.
- Calibrate the analytical balance at least monthly using Class 1 weights. Select a series of weights which covers the range of balance operation. Usually 1, 2, 5, 20, 50, and 150 gm weights are used. Record weight values on Balance Log
  Corrective Action: If the weights deviate more than 0.0002 grams, the balance needs service. Use another calibrated balance until the next service cycle if possible

TSS Test QC
- Pre-wash filters with deionized water and perform at least 2 method blanks on each lot washed.
  Corrective Action: If the weight difference is >0.0002 gm, the filter has not been washed completely. All the filters in this lot must be rewashed and the process repeated until the difference is within 0.0002 gm
  Document all method blanks.
- Store pre-washed filters in the desiccator to avoid water absorption.
- Zero the analytical balance prior to each weighing series
  Document the balance was zeroed.
- Use large bore pipets for small sample volumes and graduated cylinders for large volumes.
- Pipets and graduated cylinders are rinsed with deionized water and the rinse is added to the filter after the sample has been filtered.
- Use sufficient sample to obtain a minimum of 0.0025 gm of TSS on the filter.
  Corrective action: If the 2nd dry sample weights <0.0025 gm, invalidate the results and repeat the analysis using more sample volume. Exception: If the weight is <0.0025 gm as a result of filtering 1000 ml, the results are valid and the data reported.
- Filters are checked for dryness. The difference between the 1st and 2nd dry weight is <0.0005 gm. Document the weights.
  Corrective Action: If the 2nd dry weight has changed by more than 0.0005 gm, the filter is not dry and must be re-dried in the drying oven until the change in weight is <0.0005 gm.
- Filters are placed in the desiccator upon removal from the drying oven.
  Document the condition of desiccator.
- Performance evaluation samples should be run at least annually.
- Split samples can be run with other nearby facilities
- Duplicate sample TSS results should be within 10% of their average.
  Corrective Action: Homogenize samples with large amounts of chunky suspended solids to obtain a more uniform sample. Remix samples between duplicates and measure quickly.
- Perform monthly in-house performance evaluation samples. Weigh 0.1000 gram of dried Infusorial Earth and place in a 1 liter volumetric flask. The answer should be 100 mg/L. Percent recovery should be 100 ± 5%.
  Corrective Action: Review all the above QC parameters and repeat analysis until the results are acceptable.

Volatile Suspended Solids (VSS)
Volatile suspended solids are defined as the suspended solids that can be ignited at 550°C. Volatile suspended solids gives a rough approximation of the amount of organics present in the sample. Volatile suspended solids may be run on grit chamber solids to determine if heavy organics are settling in the grit chamber. Volatile suspended solids are also run to determine the amount of organics in the mixed liquor of an activated sludge system.
If volatile suspended solids are to be run, the glass fiber filters must be pre-ignited in the muffle furnace at 550 °C to remove any organic contaminants on the filter. If the filters have been pre-volatilized, the filter used for the TSS sample can be volatilized to give the VSS portion. The difference between TSS and VSS is called ash or fixed solids. The ash represents primarily the inorganic component of the suspended solids.

VSS Procedure
1. Pre-heat the muffle furnace to 550°C.
2. Place the dry TSS filter in the muffle furnace for 15 minutes.
3. Remove from the muffle furnace and allow to partially cool in the air before placing in the desiccator. Cool to room temperature in the desiccator.
4. Record the weight of the ash on the filter.
5. Calculate the VSS.

VSS Calculations
Volume filtered = 45 ml
Weight of TSS filter + dish = 1.4983 gm
Weight of ash + dish after 550°C = 1.4956 gm
Weight of VSS = 0.0027 gm

\[
\text{VSS} = \frac{\text{2nd dry weight of solids (gm)} \times 1,000,000}{\text{Volume of sample (ml)}}
\]

\[
\text{VSS} = \frac{0.0027 \text{ gm}}{45 \text{ ml}} = 60 \text{ mg/L}
\]

Fixed Solids or Ash
From the same data above, the ash can be determined by subtracting the VSS from the TSS. For example, if the TSS was 100 mg/L and the VSS was 60 mg/L, the ash would be 40 mg/L.

Total Dissolved Solids (TDS)
Total Dissolved Solids are defined as the portion of solids that pass through a 2 um (or smaller) glass fiber filter. The TDS sample can be prepared at the same time the TSS sample is run. The sample which passes through the TSS filter into the receiving flask is the TDS sample. This is called filtrate.

The filtrate is placed in a weighed evaporating dish, evaporated, and dried to a constant weight at 180°C. TDS is typically unaffected by the wastewater treatment process. However, excess TDS may interfere with treatment processes. In New Mexico, TDS is required for WWTP’s that have groundwater discharge permits. Drinking water has a TDS target of <500 mg/L. If the effluent is percolated through the soil, the TDS can be used as a tracer, to determine the effect of the effluent on the groundwater. TDS samples are often collected from monitoring wells surrounding the effluent disposal area. Will an effluent TDS of 1200 mg/L effect the groundwater having a TDS of 400 mg/L? Yes. The TDS downstream from the WWTP will begin to increase.

Total Dissolved Solids Procedure
1. Wash a 100 ml evaporating dish with DI water and dry in the drying oven at 180 ± 2°C for a minimum of 1 hour. Cool the evaporating dish in the desiccator, usually a minimum of 45 minutes, then weigh the dish on the analytical balance. The time in the desiccator will vary depending upon the size and number of dishes being cooled. The more dishes to be cooled, the longer the dishes must be in the desiccator prior to the initial weight.
2. Transfer the evaporating dish to the analytical balance using forceps and record the initial tare weight.

3. The volume of sample can be measured using a graduated cylinder or volumetric pipet. The sample is passed through a glass fiber filter and collected in a clean dry filtration flask. The filter is then rinsed with three successive 10 ml volumes of DI water. The washings are collected in the filtration flask. The contents of the filtration flask are then poured into the weighed evaporating dish. Rinse the filtration flask with small volumes of DI water to recover all the TDS.

4. Using tongs, place the dish in the drying oven at 180 \( \pm 2^\circ\)C. If spattering is a concern, lower the drying oven temperature to below boiling until the water has evaporated. After the water has evaporated, raise the temperature to 180 \( \pm 2^\circ\)C for a minimum of 1 hour. Remove from the oven and cool to balance temperature in the desiccator.

5. Record the 1st dry weight.

6. Return the dish to the oven, re-dry for an additional 1 hour at 180 \( \pm 2^\circ\)C. Re-cool, and re-weigh.

7. Record the 2nd dry weight. The difference between the first and second weighing should be <0.0005 gm. or less than 4% of the previous weighing to prove the sample was dried completely.

**Total Dissolved Solids Calculations**

<table>
<thead>
<tr>
<th>Calculation</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>First dry weight + dish</td>
<td>49.9778 gm</td>
</tr>
<tr>
<td>Second dry weight + dish</td>
<td>49.9774 gm</td>
</tr>
<tr>
<td>Difference</td>
<td>0.0004 gm</td>
</tr>
</tbody>
</table>

The difference of 0.0004 gm is < 0.0005gm indicating the sample has been completely dried.

<table>
<thead>
<tr>
<th>Calculation</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Second dry weight + dish</td>
<td>49.9776 gm</td>
</tr>
<tr>
<td>Dish weight (tare)</td>
<td>49.8193 gm</td>
</tr>
<tr>
<td>Difference</td>
<td>0.1583 gm</td>
</tr>
</tbody>
</table>

The difference of 0.1583 gm is the weight of the dry dissolved solids from the 50 ml poured into the dish. The weight of 0.1583 gm is acceptable since it is between 0.0025 - 0.2000 gm.

Determine the total dissolved solids by using the formula:

\[
\text{Weight of dry solids} \times 1000000 = \frac{\text{Volume of sample}}{50 \text{ ml}}
\]

\[
0.1583 \text{ gm} \times 1000000 = 3170 \text{ mg/L}
\]

Notice the volumes of DI rinse water are not added to the sample volume since pure water should not have any effect and is evaporated away.

**Problems**

This procedure works fine for most industrial, influent and monitoring well samples. A small volume of sample (50 ml) can yield reasonable TSS weights yet not generate a large volume of filtrate. The method becomes a problem when clean effluent is used since as much as 1 liter of filtrate may be produced. According to the procedure all the filtrate must be transferred to the evaporating dish which means the evaporating dish must be continually "refilled" with effluent until the effluent is gone, which could take many hours. This
may create a second problem of excessive weight (>0.2000 gm) in the evaporating dish. A low TSS effluent sample could have a high TDS value, i.e. TSS of 4 mg/L and TDS of 2700 mg/L.

**Settleable Solids**
Settleable solids is defined as the solids that settle to the bottom of an imhoff cone in 1 hour. The test is helpful in monitoring the effectiveness of the clarifiers. The test is usually measured in volume settled rather than weight.

**Settleable Solids Procedure**
1. Shake a representative sample and quickly pour 1 liter into a 1 liter imhoff cone.
2. Start a timer and allow the solids to settle for 45 minutes.
3. At the end of 45 minutes, take a stirring rod or pipet and slowly agitate any solids adhering to the sides of the imhoff cone.
4. Allow the solids to settle for an additional 15 minutes, then measure the volume of settled solids.

Do not include floating solids in the answer. If the solids do not settle uniformly, estimate the volume of water and subtract from the imhoff cone reading.

**Settleometer**
This test is often confused with the settleable solids test but is usually used as a tool to monitor the condition of the activated sludge process.