# 16.0 EXPERIMENT ON DETERMINATION OF TOTAL ORGANIC AND INORGANIC SOLIDS IN WATER

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PREAMBLE:

“How to determine total organic and inorganic solids in Water and Wastewater”.

Test procedure is in accordance to IS: 3025 (Part 18) - Reaffirmed 2002. In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in


16.1 AIM

To determine total organic and inorganic solids in the given water sample with the stipulations as per IS: 3025 (Part 18) - Reaffirmed 2002.

16.2 INTRODUCTION

The term total volatile solids refer to materials that are completely volatilised from water at higher temperature (550ºC). These solids are often referred to the organic content of the water. The term total fixed solids can be referred to materials which are not volatilised from water at higher temperature (550ºC). These solids are often referred to the inorganic content of the water.

16.2.1 ENVIRONMENTAL SIGNIFICANCE

- The water which consists of high volatile solids is not suitable for drinking purpose and indicates that the water may have been polluted by domestic wastes or other organic wastes.

- Volatile solids test is normally applied to sludges. It is indispensable in the design and operation of sludge digest, vacuum filter and incineration plants.

- Before the development of the COD test, it is used to find out the strength of industrial and domestic wastewater. It is helpful in assessing the amount biologically inert organic matter, such as lignin in case of wood pulping waste liquors.

- The determination of volatile and fixed components in the residue is useful in the control of waste water plant operation because it offers an approximate amount of organic matter present in the solid fraction of wastewater.
16.3 PRINCIPLE
The sample is evaporated in a weighed dish on a steam bath and is dried to a constant mass in an oven at 103-105°C. The residue obtained is ignited to constant weight at 550°C. The remaining solids represent the total fixed solids and the weight lost during the ignition represents the total volatile solids.

16.4 MATERIALS REQUIRED

16.4.1 APPARATUS REQUIRED

1. Evaporating Dish
2. Water Bath (Steam Bath)
3. Oven
4. Desiccators
5. Weighing balance
6. Dish Tongs
7. Magnetic Stirrer
8. Wash Bottle
1. Switch on the balance (Atleast 30 min before the test)
2. Notedown the initial dry weight of the crucible
3. Take 20 mL of water sample in the crucible
4. After drying in the oven cool to room temperature in dessicator, Note down the dry weight of the crucible
5. Place the crucible inside the muffle furnace at 550°C
6. After drying in the oven cool to room temperature in dessicator, Note down the final dry weight of the crucible
16.5 SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

Both the characteristics and the amount of solids may change.

To reduce this change in samples taken for solids determinations, keep all samples at 4°C. Do not allow samples to freeze.

Analysis should begin as soon as possible.

16.5.1 PRECAUTIONS

The following precautions should be observed while performing the experiment:

- Negative errors in volatile solids may be produced by loss of volatile matter during drying in the oven.
- In the presence of high concentration fixed solids, the determination of low concentration of volatile solids may be subject to considerable error. In those cases, the measure of volatile components by some other method like total organic carbon is advisable.
- Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before sub-sampling.
- Volume of sample should be adjusted to have residue left after drying as 100 to 200mg. It is mainly to prevent large amount of residue in entrapping water during evaporation.

16.6 PROCEDURE

16.6.1 TESTING OF SAMPLE

- To measure total volatile solids and fixed solids, take a clean silica crucible which has been washed and dried in a hot air oven at 105°C for one hour and ignited at 550°C to remove all organic materials present in it.
- Now weigh the empty silica crucible in analytical balance. Let’s denote the weight measured as \( W_1 = 52.8701 \)g
- Using pipette transfer 75mL of unfiltered sample in the porcelain dish.
- Switch on the oven and allowed to reach 105°C. Check and regulate oven and furnace temperatures frequently to maintain the desired temperature range.
- Place the silica crucible in the hot air oven and care should be taken to prevent splattering of sample during evaporation or boiling.
- Dry the sample to get constant mass. Drying for long duration is done to eliminate necessity of checking for constant mass.
• Cool the container in a desiccator. Desiccators are designed to provide an environment of standard dryness. This is maintained by the desiccant found inside. Don't leave the lid off for prolonged periods or the desiccant will soon be exhausted.
• We should weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature.
• Samples need to be measured accurately, weighed carefully, and dried and cooled completely.
• Note the weight with residue as \( W_2 = 52.8833 \text{ g} \)
• Switch on the furnace and allow it to reach 550°C. Check and regulate the furnace temperatures frequently to maintain the desired temperature range.
• Place the silica crucible in the furnace and care should be taken while keep the crucible inside the furnace since it will be too hot.
• Allow it to ignite for 20 minutes to get constant mass.
• As above, cool the silica crucible in a desiccator to room temperature.
• Weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature.
• Note the weight with residue as \( W_3 = 52.8715 \text{ g} \)

16.7 CALCULATION

**Total Volatile Solids**

Initial weight of the evaporating dish + sample \((W_1)\) = \( \ldots \ldots \) g
Final weight of the evaporating dish + sample after drying at 105°C \((W_2)\) = \( \ldots \ldots \) g
Final weight of the evaporating dish + sample after drying at 550°C \((W_3)\) = \( \ldots \ldots \) g

Weight of volatile substance \((W)\) = \( W_2 - W_3 \) g

Amount of total solids present in the sample = \( \frac{1000 \times 1000 \times W}{V} \)

\( W = \) weight of total residue in (mg). (Therefore multiply \( W \) with 1000)
\( V = \) Volume of the sample (mL) (To convert mL to L)

\( = \ldots \ldots \) mg/L
Total Fixed Solids

Initial weight of the evaporating dish \((W_1)\) = ............ g
Final weight of the evaporating dish + sample after drying at 105\(^\circ\)C \((W_2)\) = ............ g
Final weight of the evaporating dish + sample after drying at 550\(^\circ\)C \((W_3)\) = ............ g

Weight of non volatile substance \((W)\) = \(W_3 - W_1\) g

Amount of total fixed solids present in the sample \[\frac{1000\times (W)}{V}\]
\(W = \) weight of total residue in (mg). (Therefore multiply \(W\) with 1000)

\(V = \) Volume of the sample (mL) (To convert mL to L)

\(=\) ............mg/L

16.7.1 TABLE

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of the clean silica crucible (g)</td>
<td>(W_1)</td>
</tr>
<tr>
<td>Weight of the silica crucible and the residue (g)</td>
<td>(W_2)</td>
</tr>
<tr>
<td>Weight of residue (g)</td>
<td>(W)</td>
</tr>
<tr>
<td>Weight of the silica crucible and the ash (g)</td>
<td>(W_3)</td>
</tr>
<tr>
<td>Weight of ash (g)</td>
<td>(W)</td>
</tr>
<tr>
<td>Volume of the Sample (mL)</td>
<td>(V)</td>
</tr>
<tr>
<td><strong>Total Volatile Solids (mg/L)</strong></td>
<td><strong>TVS</strong></td>
</tr>
</tbody>
</table>

The Weight of the clean silica crucible (g) \(W_1 = 52.8701\) g
The Weight of the clean silica crucible and the residue (g) \(W_2 = 52.8833\) g
The Weight of the residue (g) \(W = 0.0132\) g
The Weight of the silica crucible and the ash (g) $W_3 = 52.8715 \, g$

Weight of the ash (g) $W = 0.0118$

The volume of the sample (mL) $V = 100 \, mL$

**Total Fixed Solids**

<table>
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<th>Description</th>
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</thead>
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<tr>
<td>Weight of the clean silica crucible (g)</td>
<td>$W_1$</td>
</tr>
<tr>
<td>Weight of the silica crucible and the residue (g)</td>
<td>$W_2$</td>
</tr>
<tr>
<td>Weight of residue (g)</td>
<td>$W$</td>
</tr>
<tr>
<td>Weight of the silica crucible and the ash (g)</td>
<td>$W_3$</td>
</tr>
<tr>
<td>Weight of ash (g)</td>
<td>$W$</td>
</tr>
<tr>
<td>Volume of the Sample (mL)</td>
<td>$V$</td>
</tr>
<tr>
<td><strong>Total Fixed Solids (mg/L)</strong></td>
<td><strong>TFS</strong></td>
</tr>
</tbody>
</table>

The Weight of the clean silica crucible (g) $W_1 = 52.8701 \, g$

The Weight of the silica crucible and the residue (g) $W_2 = 52.8833 \, g$

Weight of the residue (g) $W = 0.0132 \, g$

Weight of the silica crucible and the ash (g) $W_3 = 52.8715 \, g$

Weight of the ash (g) $W_3 = 0.0118$

Volume of the sample (mL) $V = 100 \, mL$
16.7.2 DATA SHEET
DETERMINATION OF TOTAL VOLATILE SOLIDS
DATA SHEET

Date Tested : August 30, 2010
Tested By : CEM Class, Group A
Project Name : CEM, NITTTR Lab
Sample Number : BH1
Sample Location : Perungudi (Lat 12’ 57’’ 31.74 & Long 80’14’’ 8.82)
Sample Description : Surface water

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of the clean silica crucible (g)</td>
<td>(W_1)</td>
</tr>
<tr>
<td>Weight of the silica crucible and the residue (g)</td>
<td>(W_2)</td>
</tr>
<tr>
<td>Weight of residue (g)</td>
<td>(W)</td>
</tr>
<tr>
<td>Weight of the silica crucible and the ash (g)</td>
<td>(W_3)</td>
</tr>
<tr>
<td>Weight of ash (g)</td>
<td>(W)</td>
</tr>
<tr>
<td>Volume of the Sample (mL)</td>
<td>(V)</td>
</tr>
<tr>
<td><strong>Total Volatile Solids (mg/L)</strong></td>
<td><strong>TVS</strong></td>
</tr>
</tbody>
</table>

**Sample Calculation:**
\[
\begin{align*}
W_2 &= 52.8833 \text{ g} \\
W_3 &= 52.8715 \text{ g} \\
V &= 100.0 \text{ mL} \\
W &= W_2 - W_3 = 52.8833 - 52.8715 = 0.0118 \text{ g} \\
W (mg) &= 0.0118 \times 1000 \\
&= 11.8 \text{ mg} \\
\end{align*}
\]

Weight of residue in mg (To convert \(W\) (g) to \(W\) (mg), multiply \(W\) (g) with 1000)

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

**Total Volatile Solids (mg/L)**
\[
\begin{align*}
V &= \text{Volume of the sample (mL)} (\text{To convert mL to L, multiply by 1000}) \\
&= 11.8 \text{ mg/100 mL} = 0.118 \text{ mg/mL} \\
&= 0.118 \text{ mg/mL} \times 1,000 \text{ mL/L} = 118 \text{ mg/L} \\
\end{align*}
\]
### DETERMINATION OF TOTAL FIXED SOLIDS

**DATA SHEET**

**Date Tested**: August 30, 2010  
**Tested By**: CEM Class, Group A  
**Project Name**: CEM, NITTTR Lab  
**Sample Number**: BH1  
**Sample Location**: Perungudi (Lat 12° 57’ 31.74 & Long 80° 14’ 8.82)  
**Sample Description**: Surface water

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of the clean silica crucible (g)</td>
<td>W₁</td>
</tr>
<tr>
<td>Weight of the silica crucible and the residue (g)</td>
<td>W₂</td>
</tr>
<tr>
<td>Weight of residue (g)</td>
<td>W</td>
</tr>
<tr>
<td>Weight of the silica crucible and the ash (g)</td>
<td>W₃</td>
</tr>
<tr>
<td>Weight of ash (g)</td>
<td>W</td>
</tr>
<tr>
<td>Volume of the Sample (mL)</td>
<td>V</td>
</tr>
</tbody>
</table>

| Total Fixed Solids (mg/L)                        | TFS        | 14.0     |

**Specimen Calculation:**

\[
\begin{align*}
W₁ &= 52.8701 \text{ g} \\
W₂ &= 52.8715 \text{ g} \\
V &= 100.0 \text{ mL} \\
W &= W₂ - W₁ \\
&= 52.8715 - 52.8701 \\
&= 0.0014 \text{ g} \\
W \text{ (mg)} &= 0.0014 \times 1000 \\
&= 1.4 \text{ mg}
\end{align*}
\]

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

**Total Fixed Solids (mg/L)**

\[
\begin{align*}
V &= \text{Volume of the sample (mL)} \ (\text{To convert mL to L, multiply by 1000}) \\
&= 1.4 \text{ mg/100 mL} = 0.014 \text{ mg/mL} \\
&= 0.014 \text{ mg/mL} \times 1,000 \text{ mL/L} = 14 \text{ mg/L}
\end{align*}
\]
16.8 INTERPRETATION OF RESULTS
In the given sample, total volatile solids is equivalent to **118.0 mg/L** and total fixed solids is **14 mg/L**.

16.9 INFERENCE
In domestic wastewater, solids are about 50 percent organic, which in turn contaminates the ground and fresh water. These solids are generally from vegetable, dead animal matter, and also include synthetic organic compounds. They can be ignited or burned. Since the organic fraction can be driven off at high temperatures, they are called volatile solids. Inorganic solids are frequently called mineral substances and include sand, gravel and silt as well as the mineral salts in the water supply which produce the hardness and mineral content of the water. Mostly, they are non-combustible. They are called non volatile solids.

16.10 EVALUATION
1. The Total Volatile Solids determination is very important in the control of
   a) Water treatment plant
   b) Sewage treatment plant
   c) Desalination plant
   d) Effluent treatment plant

2. The crucible with sample, should be placed in the muffle furnace for atleast _______.
   a) one hour
   b) two hours
   c) 20 minutes
   d) 10 minutes

3. The Total Fixed Solids is the measure of
   a) all the solids present
   b) inorganic solids present
   c) the salt content
   d) organic solids
4. The method used for the determination of solids is _____.
   a) volumetric method
   b) gravimetric method
   c) instrumentation method
   d) visual method

5. The crucible after ignition should be cooled in a desiccator
   a) because it is hot
   b) to avoid moisture absorption
   c) to cool
   d) to incubate

6. Putrescible solid means
   a) pure solids
   b) dissolved solids
   c) solids with high BOD
   d) suspended solids

   a) True
   b) False

8. The determination of total volatile solids is interfered by
   a) Loss of volatile solids during the drying process
   b) Large volatile solids water sample
   c) Dissolved salts
   d) Suspended salts

9. While placing the crucible in muffle furnace it is advisable to wear gloves made of
   a) Leather
   b) Rubber
   c) Resin
   d) Polythene
10. The Total Volatile Solids is the measure of
   a) all the solids present
   b) organic solids present
   c) the salt content
   d) inorganic salts present

KEY TO ITEMS:

 1) b
 2) c
 3) b
 4) b
 5) b
 6) c
 7) False
 8) a
 9) a
10) b