

*Indian Standard***METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTE WATER****PART 19 SETTLEABLE MATTER***(First Revision)*

1. Scope — Prescribes two methods, one gravimetric and the other volumetric, for the determination of settleable matter. These methods are applicable to all types of water and waste water.

2. Gravimetric Method

2.1 Principle — Settleable matter is determined from the difference in the values of non-filterable residue of the sample, and of suspended matter of the supernatant liquid portion of the sample after it has been allowed to stand for 1 hour. This technique determines matter which will not stay in suspension during settling period and either settles at the bottom or floats to the top.

2.2 Apparatus

2.2.1 Filters — One of the following may be used:

- a) *Gooch crucible* — 30 ml capacity with 2.1, 2.4 or 5.5 cm diameter glass fibre filter disc of pore size 1.2 μm (Whatman GF/C or equivalent); or
- b) *Crucible* — Porous-bottom silica, sintered glass, porcelain, stainless steel or alundum crucible with a maximum pore size of 5 μm .

2.2.2 Filtering apparatus — Depending on type of filter used.

2.2.3 Drying oven — With a thermostatic control for maintaining temperature up to $180 \pm 2^\circ\text{C}$.

2.2.4 Desiccator — Provided with a colour indicating desiccant.

2.2.5 Analytical balance — 200 g capacity, capable of weighing to nearest 0.1 mg.

2.3 Procedure

2.3.1 Preparation of glass fibre filter disc — Place the glass fibre filter on the membrane filter apparatus or insert into bottom of a suitable gooch crucible with wrinkled surface up. While vacuum is applied, wash the dish with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus, or both crucible and filter if gooch crucible is used, and dry in an oven at $103-105^\circ\text{C}$ for 1 hour. Transfer to desiccator and weigh after half an hour. Repeat the drying cycle until a constant mass is obtained (mass loss is less than 0.5 mg in successive weighings). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only. If determinations are to be carried out at 180°C then the filter or crucible/filter should be dried at 180°C .

2.3.2 Sample volume — As in potable waters non-filterable residue is usually small, relatively large volume of water is passed through filter so as to obtain at least 2.5 mg residue. For deciding volume to be taken, turbidity values may be taken into consideration. If turbidity value of a sample is less than 50, filter 1 litre sample and if turbidity value exceeds 50 units, filter sufficient sample so that non-filterable residue is 50 to 100 mg.

2.3.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fitted support.

2.3.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected according to 2.3.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

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2.3.5 With suction on, wash the graduated cylinder, filter and non-filterable residue with portions of distilled water, allowing complete drainage between washings. Remove all traces of water by continuing to apply vacuum after the wash water has passed through.

2.3.6 After filtration, transfer the filter along with contents to an oven maintained at either 103 - 105°C or 179 - 181°C for at least 1 hour. Cool in a desiccator and weigh. Repeat the drying cycle till constant mass is obtained. Alternatively, remove crucible and filter from crucible adapter, wipe dry from outside with filter paper and dry at 103 - 105°C or 179 - 181°C in an oven. Cool in a desiccator and weigh. Repeat the drying cycle till constant mass is obtained.

2.3.7 Take a glass vessel of not less than 9 cm diameter and pour well mixed sample in this vessel in quantity not less than 1 litre and sufficient to have a depth of 20 cm. Glass vessel of greater diameter and a larger volume of sample may also be used. Allow the sample to stand for 1 hour and without disturbing floating or settled material, siphon 250 ml of sample from centre of glass vessel at a point half way between the surface of settled sludge and liquid surface. Determine suspended matter (in mg/l) in an aliquot portion or all of this supernatant liquid as given in 2.3.1 to 2.3.6. This is non-settleable matter.

2.4 Calculation — Calculate the settleable matter from the following equation:

$$\text{Settleable matter, mg/l} = \frac{1\,000\,M_1}{V_1} - \frac{1\,000\,M_2}{V_2}$$

where

M_1 = mass of non-filterable residue in mg,

V_1 = volume of sample in ml,

M_2 = mass of non-settleable matter in mg, and

V_2 = volume of supernatant liquid used for determining non-settleable matter in ml.

2.5 Report — Report in whole numbers for less than 100 mg/l and to three significant figures for higher values.

3. Volumetric Method

3.1 Principle — Volume of settleable matter is determined by allowing the sample to stand for 1 hour in an Imhoff cone.

3.2 Apparatus — Standard Imhoff cone, suitably calibrated bottom to top and having 1-litre mark.

3.3 Procedure — Mix the sample thoroughly and fill the Imhoff cone up to the 1-litre mark. Let it settle for 45 minutes and gently stir the sides of the cone with a rod or by spinning and let it settle for further 15 minutes. Record the volume of settleable matter in the cone as ml/l.

3.4 Report — Report the volume of settleable matter in ml/l.