I

## Indian Standard

# METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTE WATER

## PART 16 FILTERABLE RESIDUE (TOTAL DISSOLVED SOLIDS)

## (First Revision)

(Incorporating Amendment No. 1)

**1. Scope** — Prescribes a gravimetric method for the determination of filterable residue. This method is applicable to all types of water and waste water.

**2. Principle** — The sample is filtered and the filtrate evaporated in a tared dish on steam-bath. The residue after evaporation is dried to constant mass at 103-105°C or 179-181°C.

Note — The value of filterable residue obtained by drying at 179-181°C conforms more closely to those obtained by summation of various constituents. (Since bicarbonates decompose to carbonates, only half of bicarbonate should be taken while summing up of the various constituents.)

**3.** Interferences — Interferences are the same as those for total residue.

### 4. Apparatus

**4.1** *filter* — Any one of the following may be used.

**4.1.1** Glass fibre filter disc — (Whatman GF/C or equivalent) 2.1 to 5.5 cm in diameter, pore size  $1.2 \,\mu\text{m}$ .

**4.1.2** Paper — Acid washed, ashless hard filter finish; filter paper sufficiently retentive for fine particles (Pore size 2-2.5  $\mu$ m equivalent to Whatman filter No. 542.)

**4.1.3** Gooch crucible — 30 ml capacity with 2.1 or 2.4 cm diameter glass fibre filter disc. (Whatman or equivalent.)

**4.1.4** Sintered disc — G-5 or its equivalent with pore size 1 to  $2 \mu m$ .

**4.1.5** *Membrane filter* — 0.45 µm membrane.

**4.2** *Filtering Assembly* — Depending upon the type of filter selected.

**4.3** Drying Oven — With thermostatic control for maintaining temperature up to  $180 \pm 2^{\circ}$ C.

4.4 Desiccator — Provided with a colour indicating desiccant.

**4.5** Analytical Balance — 200 g capacity and capable of weighing to nearest 0.1 mg.

**4.6** Magnetic stirrer — With teflon coating stirring bars.

**5. Sample Handling and Preservation** — Preservation of the samples is not practical. Analysis should begin as soon as possible. Refrigeration or chilling to 4°C to minimise microbiological decomposition of solids is recommended.

### 6. Procedure

**6.1** Heat the clean evaporating dish to  $180^{\circ}$ C for 1 hour. Cool in the desiccator, weigh and store in the desiccator until ready for use.

**6.2** Filter a portion of the sample through any of the filters mentioned in **4.1**. Select volume of the sample which has residue between 25 and 250 mg, preferably between 100 to 200 mg. This volume may be estimated from values of specific conductance. To obtain a measurable residue, successive aliquots of filtered sample may be added to the sample dish.

**6.3** Stir volume of sample with a magnetic stirrer or shake it vigorously. Pipette this volume to a weighed evaporating dish placed on a steam-bath. Evaporation may also be performed in a drying oven. The temperature shall be lowered to approximately 98°C to prevent boiling and splattering of the sample. After complete evaporation of water from the residue, transfer

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the dish to an oven at 103-105°C or 179-181°C and dry to constant mass, that is, till the difference in the successive weighings is less than 0.5 mg. Drying for a long duration (usually 1 to 2 hours) is done to eliminate necessity of checking for constant mass. The time for drying to constant mass with a given type of sample when a number of samples of nearly same type are to be analysed has to be determined by trial.

**6.4** Weigh the dish as soon as it has cooled avoiding residue to stay for long time as some residues are hygroscopic and may absorb water from desiccant that is not absolutely dry.

7. Calculation — Calculate the filterable residue from the following equation:

Filterable residue, mg/l =  $\frac{1\ 000\ M}{V}$ 

where

M = mass in mg of filterable residue, and

V = volume in ml of the sample.

**8. Report** — Report in whole numbers for less than 100 mg/l and to three significant figures for values above 100 mg/l. Report the temperature of determination.

**9. Precision and Accuracy** — The precision of the method is about 5 percent. Accuracy cannot be estimated because filterable residue as determined by this method is a quantity defined by the procedure followed.

#### EXPLANATORY NOTE

Filterable residue is the term applied to the residue remaining in a weighed dish after the sample has been passed through a standard fibreglass filter and dried to constant mass at 103-105°C or 179-181°C.

This method supersedes clause **12** of IS : 3025-1964 'Methods of sampling and test (physical and Chemical) for water used in industry'.

This edition 2.1 incorporates Amendment No. 1 (December 1999). Side bar indicates modification of the text as the result of incorporation of the amendment.