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Indian Standard

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

PART 18 VOLATILE AND FIXED RESIDUE (TOTAL, FILTERABLE AND NON-FILTERABLE)

(First Revision)

(Incorporating Amendment No. 1)

Water Sectional Committee, CDC 26 and Panel for Methods of Test for Water and Effluents, CDC 26 : P1 [Ref : Doc : CDC 26 : (8850)]

1. Scope — Prescribes a gravimetric method for the determination of volatile and fixed portions of total, filterable and non-filterable residues. The method is applicable to all types of water and waste water.

2. Terminology

2.1 *Total Fixed Residue* — The dish with residue after completion of test for total residue is heated in a muffle furnace at 550°C for 1 hour. Total fixed and volatile residue are calculated from loss in mass, on ignition.

2.2 *Filterable Fixed Residue* — The dish with residue after completion of test for filterable residue is heated in a muffle furnace at 550°C for 1 hour. Filterable fixed and volatile residue are calculated from loss of mass, on ignition.

2.3 *Non-filterable Fixed Residue* — The filter with residue after completion of test for non-filterable residue is heated in a muffle furnace at 550°C for 1 hour. Non-filterable fixed and volatile residue are calculated from loss in mass after ignition.

3. Limitations

3.1 The test is subject to many errors due to loss of water of crystallization, loss of volatile matter prior to combustion, incomplete oxidation of certain complex organics and decomposition of mineral salts during combustion.

3.2 The results should not be considered as accurate measure of organic carbon in the sample.

3.3 An important source of error in the determination is failure to obtain a representative sample.

4. Apparatus

4.1 *Evaporating Dish* — 90 mm, 100 ml capacity made of platinum, porcelain, silica or borosilicate glass. Platinum is suitable for all tests; porcelain, silica and glass may be used for samples with a *p*H value less than 9.0.

4.2 *Steam-Bath*

4.3 *Drying Oven* — With thermostatic control for maintaining temperature up to 180 ± 2°C.

4.4 *Desiccator* — Provided with a colour indicating desiccant.

4.5 *Muffle Furnace* — Capable of operation at 550°C.

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

5. Sample Preservation and Handling — Preservation is not practical. Refrigeration or chilling to 4°C is recommended.

6. Procedure

6.1 Heat the clean evaporating dish to 550°C for 1 hour. Cool, desiccate, weigh and store in desiccator until ready for use.

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6.2 Select volume of the sample which has residue between 25 and 250 mg, preferably between 100 and 200 mg. This volume may be estimated from values of specific conductance. To obtain a measurable residue, successive aliquots of sample may be added to the sample dish.

6.3 Pipette this volume in a weighed evaporating dish on 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 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 elliminate 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 absolutely dry.

6.5 After weighing, ignite the dish in a muffle furnace at 550°C for 1 hour. After ignition, allow the vessel to partially cool in air and transfer to desiccator, cool and weigh.

Note — Residues from determination of total, filterable or non-filterable solids as determined In IS : 3025 (Part 15)-1984 'Methods of sampling and test (physical and chemical) for water and waste water: Part 15 Total residue (total solids — dissolved and suspended)', IS : 3025 (Part 16)-1984 'Method of sampling and test (physical and chemical) for water and waste water: Part 16 Filterable residue (total dissolved solids)' and IS : 3025 (Part 17)-1984 'Methods of sampling and test (physical and chemical) for water and waste water: Part 17 Non-filterable residue (total suspended solids)' may also be used for the determination of volatile and fixed residue (as the case may be).

7. Calculations — Calculate the fixed residue and volatile residue as follows (total, filterable or non-filterable):

$$\text{Volatile residue, mg/l} = \frac{(A - B)\ 1\ 000}{V}$$

$$\text{Fixed residue, mg/l} = \frac{(B - C)\ 1\ 000}{V}$$

where

A = mass in mg of residue and dish/filter before ignition,

B = mass in mg of residue and dish/filter after ignition,

C = mass in mg of dish/filter, and

V = volume in ml of the sample.

8. Report — Report to the nearest whole number for values up to 100 mg/l and to three significant figures for higher values. Report the temperature of determination.

EXPLANATORY NOTE

The volatile and fixed components in the residue may be determined igniting the sample at 550°C. The determination is useful in the control of waste water plant operation because it offers a rough approximation of the amount of organic matter present in the solid fraction of waste water, activated sludge, industrial wastes, or bottom sediments. Since the results may also reflect loss of water of crystallization, loss of volatile organic matter before combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion, it may not yield an accurate measure of organic carbon.

This method supersedes **9** of IS : 2488 (Part 5)-1976 'Methods of sampling and test for industrial effluents, Part 5' and **11** 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.