IS : 3025 (Part 15) - 1984
(Reaffirmed 1998)
Edition 2.1
(2000-01)

UDC 628.1/.3 : 543.3 : 543.814

Indian Standard

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

PART 15 TOTAL RESIDUE (TOTAL SOLIDS — DISSOLVED AND SUSPENDED)

(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(8847)]

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

2. Principle — The sample is evaporated in a weighed dish on a steam-bath and is dried to a constant mass in an oven either at 103-105°C or 179-181°C. Total residue is calculated from increase in mass.

Note — In general, by evaporating and drying water samples at 179-181°C values are obtained which conform more closely to those obtained by summation of individually determined mineral salts.

3. Interferences

3.1 Highly mineralized waters containing significant concentration of calcium, magnesium, chloride and/or sulphate may be hygroscopic. These may require prolonged drying, desiccation and rapid weighing. However, prolonged drying may also cause loss of constituents, particularly nitrates and chlorides.

3.2 A large amount of residue in the evaporating basin may crust over and entrap water preventing its evaporation during drying. For this reason, the volume of the sample should be adjusted so that the residue left after drying should be about 100 to 200 mg.

4. Apparatus

4.1 *Evaporating Dish* — of 90 mm diameter, 100 ml capacity made of platinum, nickel, porcelain, silica or borosilicate glass. Platinum is suitable for all tests. Nickel is satisfactory if residue is not to be ignited. 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* — drying oven with thermostatic control for maintaining temperature up to 180 ± 2°C.

4.4 D*esiccator* — 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 coated 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 minimize microbiological decomposition of solids is recommended.

6. Procedure

6.1 Heat the clean evaporating dish to 180°C for 1 hour. Cool, desiccate, weigh and store in desiccator until ready for use.

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; if necessary, add successive sample portion to the same dish after evaporation.

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 should 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

Adopted 25 January 1984 | | **Price Group 1**

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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 should 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 which may not be absolutely dry.

7. Calculation — Calculate the total residue using the following equation:

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

where

M = mass in mg of total residue, and

V = volume in ml of the sample.

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

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

EXPLANATORY NOTE

Total residue is the term applied to the material left in the vessel after evaporation of a sample of water and its subsequent drying in an oven at a definite temperature. Total residue includes non-filterable residue (the portion of the total residue retained by a filter), and filterable residue (the portion of the total residue which passes through the filter).

This method supersedes **10** 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 (January 2000). Side bar indicates modification of the text as the result of incorporation of the amendment.