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IS : 3025 (Part 33) - 1988

Indian Standard

(Reaffirmed 1999)

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

PART 33 IODIDE

(First Revision)

1. Scope — This standard prescribes a method for determination of iodide in water and wastewaters. The spectrophotometric method shall be followed as a referee method in case of difference of opinion.

2. Principle — Iodide can be determined in water by using its ability to catalyze the reduction of cerium (IV) by *arsenious* acid. For given reaction conditions and *p*H, the catalytic effect is proportional to concentration of iodide. At the end of the fixed reaction time, the reaction is stopped by cooling. The concentration of ceric ion remaining is determined by measuring the absorbance of the solution at 520 nm and comparing with a calibrated graph based on the absorbance of iodide standard solution treated similarly. The detection limit is 5 µg/l.

3. Interference — The formulation of non-catalytic forms of iodine and inhibitory effects of silver and mercury are reduced by adding excess of sodium chloride that sensitized the reaction. The interference of organic materials that can be oxidized by cerium (IV) is removed by digesting sample in an alkaline medium.

4. Apparatus

4.1 *Spectrophotometer* — For use at wavelength of 520 nm and provided with a light path of 1 cm.

4.2 *Stop Watch*

4.3 *Test Tubes* — 2 × 15 cm.

5. Reagents

5.1 *Sodium Chloride Solution* — Dissolve 200·0 g of sodium chloride in water and dilute to 1 litre. Recrystallize the sodium chloride if an interfering amount of iodine is present, using water ethanol mixture.

5.2 *Arsenious Acid* — 0·1 N. Dissolve 4·946 g of arsenic trioxide in water containing 0·20 ml of concentrated sulphuric acid. Dilute to 100 ml.

5.3 *Potassium Hydroxide* — 2 N.

5.4 *Zinc Sulphate* — 100 g/l.

5.5 *Ceric Ammonium Sulphate* — 0·02 N. Dissolve 12·64 g of ceric ammonium sulphate $[Ce (NH_4)_4 (SO_4)_4 . 2H_2O]$ in water, add 44 ml of concentrated sulphuric acid and make up to 1 litre.

5.6 *Potassium Chlorate Solution Saturated*

5.7 *Stock Iodide Solution* — Dissolve 261·6 mg of anhydrous potassium iodide in water and dilute to 1 000 ml; 1·00 ml = 200 µg of iodide.

5.8 *Intermediate Iodide Solution* — Dilute 50 ml of stock iodide solution to 1000 ml with water; 1·00 ml = 10·00 µg of iodide.

5.9 *Standard Iodide Solution* — Dilute 10 ml of intermediate solution to 1 litre; 1 ml = 0·1 µg of iodide.

Water Sectional Committee, CDC 26; Panel for Methods of Test for Water, Wastewaters and Effluents, CDC 26 : P1 [Ref : Doc : CDC 26 (9301)]

Adopted 1 January 1988

Gr 1

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6. Procedure

6.1 *Sample Pre-treatment* — To eliminate interference from organic materials capable of being oxidized the samples are digested in an alkaline medium. To a 6 ml sample placed in a test tube, add 1 ml of zinc sulphate, 2 ml of 2 N potassium hydroxide and 2 drops of a saturated solution of potassium chlorate solution, and mix. Evaporate to 105°C until dry. Heat for 30 minutes at 500°C, then for 1 hour at 600°C. After cooling, add 6 ml of distilled water and centrifuge for 15 minutes. If the solution is not turbid, it can be used for analysis. It may be necessary to clarify further by filtration.

6.2 A 2 ml volume of sample, standard or blank having a potassium hydroxide concentration of 0·66 N is placed in a test tube. The iodide content should be 0·005 to 0·100 μg. The test tube is placed in ice water bath and the sample permitted to equilibrate after which 2·0 ml of arsenious acid solution, ceric ammonium sulphate solution are added and solution is carefully mixed. The sample is then placed in a water bath at 40°C for exactly 20 minutes. The reaction is then stopped by placing the test tube in ice water bath where it is allowed to stand for exactly 10 minutes. After this period, measure the absorbance at 520 nm using distilled water as a reference. The absorbance of the blank is subtracted from that of the sample to obtain the net absorbance of the sample.

6.3 *Calibration Graph Preparation* — Add 33 ml portions of 2 N potassium hydroxide solution to each of a series of 100 ml volumetric flasks. Prepare standards and blank by adding 0 to 50·0 ml portions of the iodide working solutions and diluting to 100 ml. Aliquots of these are treated in the same manner as are samples except that they need not be subjected to the digestion procedure. Plot a graph absorbance *versus* concentration.

7. Calculation

$$\text{Iodide (as I), mg/l} = \frac{m}{v}$$

where

m = μg of iodide equivalent from the calibration standard curve, and
v = volume in ml of the sample.

EXPLANATORY NOTE

Iodide is generally present in microgram per litre quantity in most natural waters. Higher concentrations may be found in brine, certain industrial wastes, etc. This standard supersedes **25** of IS : 3025-1964 'Methods of sampling and test (physical and chemical) for water used in industry'.

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