UDC 628·1/·3:543·3 [546·214]

(Reaffirmed 1999)

Indian Standard

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

PART 36 OZONE, RESIDUAL

(First Revision)

Water Sectional Committee, CDC 26; Panel for Methods of Test for Water, Wastewaters and Effluents, CDC 26 : P1 [Ref : Doc : CDC 26 (9307)]

1. Scope — Prescribes a method for determination of ozone, residual in water and wastewaters.

2. Principle — Ozone liberates iodine from potassium iodide solution. After immediate acidification, the liberated iodine is titrated with standard sodium thiosulphate with starch as indicator.

3. Interference — Ozonated water may contain manganese dioxide, ferric ion, chlorine, nitrite, peroxide and other oxidation products. Avoid their interference by passing ozone through the gaseous phase into potassium iodide solution for titration. The stability of ozone solution decreases progressively at increment in temperature above freezing and with each increment in *p*H above 3·0. The minimum detectable concentration by this method is 30 μg/litre.

4. Apparatus

4.1 *Gas-washing Bottles and Absorbers* — 1 000 and 500 ml capacities.

4.2 *Pure Air or Nitrogen Supply* — 0·2 to 1·0 litre/min capacity.

4.3 *Glass, Stainless Steel or Aluminium, Piping* — for carrying ozonized air (good quality PVC tubes may be used for short runs but not rubber).

5. Reagents

5.1 *Potassium Iodide Solution* — Dissolve 20 g of potassium iodide, free from iodate and reducing agents in 1 litre of freshly boiled and cooled water. Store in brown bottle in a refrigerator.

5.2 *Sulphuric Acid* — 1 N.

5.3 *Standard Sodium Thiosulphate* — 0·1 M. Dissolve 25 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in 1 litre freshly boiled distilled water. Standardize against potassium hydrogen iodate or potassium dichromate.

5.3.1 *Standard sodium thiosulphate* — 0·005 M. Dilute proper volume (about 50 ml) of 0·1 M solution (**5.3**) to 1 000 ml. For accurate work, standardize this also against potassium hydrogen iodate or potassium dichromate.

5.4 *Starch Indicator Solution* — Prepare as given in IS : 2263-1979 'Methods of preparation of indicator solutions (*first revision*)'.

5.5 *Standard Iodine Solution* — 0·1 M. Dissolve 40 g of potassium iodide in 25 ml of distilled water. Add 13 g of resublimed iodine and stir until dissolved. Dilute to one litre and standardize against arsenite solution.

5.5.1 *Standard iodine solution* — 0·005 M. Dissolve 16 g of potassium iodide in little distilled water in a 1 litre volumetric flask, add proper volume of 0·1 M iodine solution and dilute to mark. For accurate work, standardize daily. Store in a brown bottle or in the dark. Protect from direct sunlight and keep from contact with rubber.

6. Procedure

6.1 *Sample Collection* — Collect an 800 ml sample in a 1 litre gas washing bottle. Pass stream of pure air or nitrogen through the sample and through an absorber containing 400 ml of potassium iodide solution. Continue for 5 to 10 minutes at a rate of 0·2 to 1·0 litre/min to insure that all ozone is swept from the sample and absorbed in potassium iodide solution.

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6.2 *Titration* — Transfer potassium iodide solution to a 1 litre beaker, rinse absorber and add 20 ml of 1 N sulphuric acid to reduce *p*H below 2·0. Titrate with 0·005 M sodium thiosulphate titrant until yellow colour of liberated iodine is almost is discharged. Add 4 ml of starch indicator solution and continue titrating carefully but rapidly to the end point, at which the blue colour just disappears. Long contact of iodine and starch develops a blue compound that is difficult to decolourize.

6.3 *Blank Test* — Correct sample titration result by determining black contributed by such reagent impurities as free iodine or iodate in potassium iodide or traces of reducing agents that might reduce liberated iodine.

6.3.1 Take 400 ml of potassium iodide solution, 20 ml of 1 N sulphuric acid and 4 ml of starch indicator solution.

Perform whichever of the following blank titration is applicable :

a) If a blue colour appears, titrate with 0·005 M sodium thiosulphate to disappearance of blue and record result.

b) If no blue colour appears, titrate with 0·005 M iodine solution until a blue colour appears. Back titrate with 0·005 M sodium thiosulphate to disappearance and record the difference.

Before calculating ozone concentration, subtract blank titration from (a) above from sample titration, or add result of (b) above.

7. Calculation

7.1 Ozone (residual) (as O_3), mg/litre $= \dfrac{(A \pm B) \times M \times 24\,000}{V}$

where

A = volume in ml of titrant for sample,

B = volume in ml of titrant for blank,

M = molarity of thiosulphate, and

V = volume in ml of sample used in the test.

EXPLANATORY NOTE

Ozone is a potent germicide. It is also used as an oxidizing agent for destruction of organic compounds producing taste and odour in water, for destruction of organic colouring matter and for the oxidation of reduced iron or manganese salts to insoluble oxides which can be precipitated or filtered from the water.

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