

*Indian Standard***METHODS OF SAMPLING AND TEST  
( PHYSICAL AND CHEMICAL ) FOR WATER  
AND WASTEWATER****PART 28 SULPHITES***( First Revision )*

**1. Scope** — Prescribes iodide-iodate titrimetric method for determination of sulphite in water and wastewater. This method is applicable to a minimum detectable concentration of 2 mg/l sulphite.

**2. Theory and Principle** — Acidified water sample containing sulphite reduces iodate to iodine. Any excess of iodate added after all the sulphite is used up, oxidizes iodide to free iodine under acidic pH condition. Iodine oxidizes sulphite to sulphate. Any excess iodine results in the formation of blue colour in presence of starch indicator.

**3. Interferences**

**3.1** Interferences due to the presence of sulphides may be removed by the addition of zinc acetate.

**3.2** Copper ions rapidly accelerate the oxidation of sulphite ions. Certain heavy metals may also react in a similar manner as copper. Proper sampling and immediate fixing by acid addition should minimise such difficulties.

**3.3** Addition of sulphamic acid helps to eliminate interference due to nitrate.

**4. Sampling and Storage**

**4.1** Highly polluted or contaminated samples should be stored at low temperature, allowing as little contact with air as possible.

**4.2** Since at pH 8.0 or above sulphite may be oxidised to sulphate, pH should, therefore be adjusted below 8.0.

**4.3** Fix the sample ( at about 50°C ) immediately by adding 1 ml EDTA solution for each 100 ml of sample.

**5. Reagents****5.1 Sulphuric Acid**

**5.2 Starch Indicator Solution** — Add 5.0 g starch to 800 ml boiling distilled water and stir. Dilute to one litre and boil for a few minutes and let settle overnight. Use the clear supernatant liquid. Add a few drops of chloroform or salicylic acid ( 1.3 g/l ) to preserve the indicator.

**5.3 Standard Iodide-Iodate Solution ( 0.0125 M )** — Dissolve 0.4458 g primary grade potassium iodate ( dried for 4 h at 120°C ), 4.35 g of potassium iodide and 310 mg sodium bicarbonate in distilled water and dilute to 1000 ml. One millilitre of this solution is equivalent to 500 mg of sulphite.

**5.4 Sulphamic Acid** — Crystalline.

**5.5 EDTA Reagent** — Dissolve 2.5 g of disodium EDTA in 100 ml of distilled water.

**6. Procedure**

**6.1** Add 1 ml sulphuric acid and about 0.1 g of sulphamic acid crystals into a 250-ml long necked flask.

**6.2** Measure a suitable volume of EDTA stabilized sample ( 50 ml to 100 ml ) ( see 4.3 ) and transfer to the flask keeping the tip of the pipette below the surface of liquid. Add 1 ml of starch indicator solution ( see 5.2 ).

**6.3** Titrate immediately with potassium iodide-iodate standard solution until a faint permanent blue colour develops (view the colour change against a white background). Carry out a blank, using distilled water instead of sample.

**7. Calculation**

$$SO_2 = \frac{(A-B) \times N \times 40\,000}{V} \text{ (mg/l)}$$

where

**A** = volume in ml of standard iodide-iodate solution used for sample,

**B** = volume in ml of standard iodide-iodate solution used in blank,

**N** = normality of potassium iodide-iodate solution, and

**V** = volume in ml of sample taken for the test.

**EXPLANATORY NOTE**

Sulphites may occur in natural waters or wastewaters as a result of industrial pollution and in treatment plant effluents dechlorinated with sulphur dioxide. Control of sulphite ion in wastewater treatment and discharge may be important environmentally, mainly because of its toxicity to fish and other aquatic life and its rapid oxygen demand. The presence of other oxidizable materials, such as sulphide, thiosulphate,  $Fe^{2+}$  ions can apparently give higher readings of sulphites. Some metal ions like  $Cu^{2+}$  may catalyze the oxidation of sulphite.

This method supersedes clause 21 of IS : 3025-1964 'Methods of sampling and test (physical and chemical) for water used in industry'.