

Indian Standard
METHODS FOR
MEASUREMENT OF AIR POLLUTION

PART VII HYDROGEN SULPHIDE

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART VII HYDROGEN SULPHIDE

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Indian Standard
**METHODS FOR
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PART VII HYDROGEN SULPHIDE**

0. FOREWORD

0.1 This Indian Standard (Part VII) was adopted by the Indian Standards Institution on 3 July 1973, after the draft finalized by the Chemical Hazards Sectional Committee had been approved by the Chemical Division Council.

0.2 Hydrogen sulphide is a highly toxic gas. Repeated exposure to low concentration has an irritating effect on mucous membranes, eyes and respiratory tract.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part VII) prescribes a method for the measurement of concentration of hydrogen sulphide in community air.

1.1.1 The range of concentration covered in this method is $6 \mu\text{g}/\text{m}^3$ to $600 \mu\text{g}/\text{m}^3$ of hydrogen sulphide in air.

2. PRINCIPLE

2.1 The determination of hydrogen sulphide by colorimetric method is based on the reaction which takes place under suitable conditions between N, N-dimethyl-*p*-phenylenediamine sulphate, ferric sulphate and sulphide ions, resulting in the formation of methylene blue.

*Rules for rounding off numerical values (revised).

3. SENSITIVITY

3.1 Minimum detectable concentration of hydrogen sulphide is 0.03 μg per millilitre of the absorbing solution with an overall accuracy of ± 20 percent. Beer's law is obeyed up to 2 μg of hydrogen sulphide per millilitre of absorbing solution.

4. INTERFERENCES

4.1 Some strong reducing agents like sulphites and thiosulphates prevent the formation of the colour or diminish its intensity. These may be eliminated by adding sufficient amount of ferric sulphate solution.

4.2 Atmospheric oxides of nitrogen cause interference. They may be avoided by adding sulphamic acid after the sampling is over.

4.3 Other possible atmospheric interferences like sulphur dioxide, ozone, and ammonia are not serious at their normal existing levels of concentrations.

5. REAGENTS

5.1 Absorbing Solution — Dissolve 4.3 g of cadmium sulphate ($\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$) in water. Add 0.3 g of sodium hydroxide dissolved in a small amount of water and dilute to one litre. Mix well before use. Store the solution in a refrigerator.

5.2 N, N-Dimethyl-*p*-Phenylene-Diamine-Sulphate Solution — Add 50 ml of concentrated sulphuric acid (conforming to IS : 266-1961*) to 30 ml of distilled water and cool. Add 12 g of N, N-dimethyl-*p*-phenylene diamine or 27.2 g of N, N-dimethyl-*p*-phenylene-diamine sulphate. Stir the bulk solution till it is completely dissolved. Store it as stock solution in refrigerator.

5.2.1 For the purpose of preparing standards, dilute 25 ml of the stock solution to one litre with dilute sulphuric acid (1 : 1).

5.3 Ferric Sulphate Solution — Add excess of ferric sulphate in 100 ml of water to obtain a saturated solution.

5.4 Sulphamic Acid Solution — 1 percent (m/v). Dissolve one gram of sulphamic acid in 100 ml of water.

5.5 Sodium Sulphide Solution — Dissolve 1.2 g of sodium sulphide of analytical grade ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$), in one litre of pre-refrigerated distilled water and stopper it. Since the solution is very unstable, it shall be prepared just before use. It shall be stored in a refrigerator. Standardize this solution against standard sodium thiosulphate solution (0.025 N) as follows.

*Specification for sulphuric acid (*revised*).

5.5.1 Take 100 ml of distilled water in 250-ml conical flask and add 20 ml of standard iodine solution (0.025 N). Add 25 ml of hydrochloric acid (0.1 N) and titrate with the thiosulphate solution using starch solution as indicator. Note the titrant reading as *A*.

5.5.2 Take 100 ml of distilled water in 250-ml conical flask and add 20 ml of standard iodine solution (0.025 N). Add 25 ml of hydrochloric acid (0.1 N) and 20 ml of sodium sulphide solution (5.5) and repeat the titration with the thiosulphate solution. Note the titrant reading as *B*.

5.5.3 Calculate the strength of sodium sulphide solution in terms of hydrogen sulphide as follows:

$$\text{Hydrogen sulphide, } \mu\text{g/ml} = \frac{(A-B) \times \text{normality of thiosulphate solution} \times 17 \times 10^3}{20}$$

5.5.4 For the purpose of preparing standards for comparison, dilute a portion of the sodium sulphide solution so that it contains equivalent of 10 $\mu\text{g/ml}$ of hydrogen sulphide.

6. PREPARATION OF STANDARDS

6.1 Prepare a set of different concentrations of hydrogen sulphide solution as follows.

6.1.1 Arrange 11 tubes (50-ml capacity) in line and put serial numbers on them.

6.1.2 Add 15 ml of absorbing solution to each tube.

6.1.3 As indicated below, add to each of the 10 test-tubes an amount of dilute sodium sulphide solution necessary to give a concentration series of 1 μg to 25 μg in increasing order:

Tube No.	Blank	1	2	3	4	5	6	7	8	9	10
Amount in ml of standard sulphide solution (see 5.5.4)	0	0.1	0.2	0.3	0.5	0.7	1.0	1.3	1.7	2.0	2.5

6.1.4 Add 1 ml of sulphamic acid solution, 0.6 ml of N,N-dimethyl-*p*-phenylene-diamine sulphate solution (see 5.2.1), and 0.05 ml of ferric sulphate solution to each of the test-tubes in that order, shaking well after each addition.

6.1.5 Make up the volumes to 20 ml in each of the tubes with distilled water and mix thoroughly.

6.2 Preparation of Calibration Curve

6.2.1 Allow 30 minutes for the development of full colour in the tubes.

6.2.2 Measure the colour intensity of the solutions in a spectrophotometer at 670 nm on transmission scale. Use the reagent blank as control.

6.2.3 Draw the calibration curve of percent transmission *versus* micrograms of hydrogen sulphide.

7. SAMPLING APPARATUS

7.1 The equipment to be used for sampling consists of a standard impinger of 35 ml capacity, a trap, a flowmeter or critical orifice device, and a suction pump. The arrangement of the sampling train is illustrated in Fig. 1.

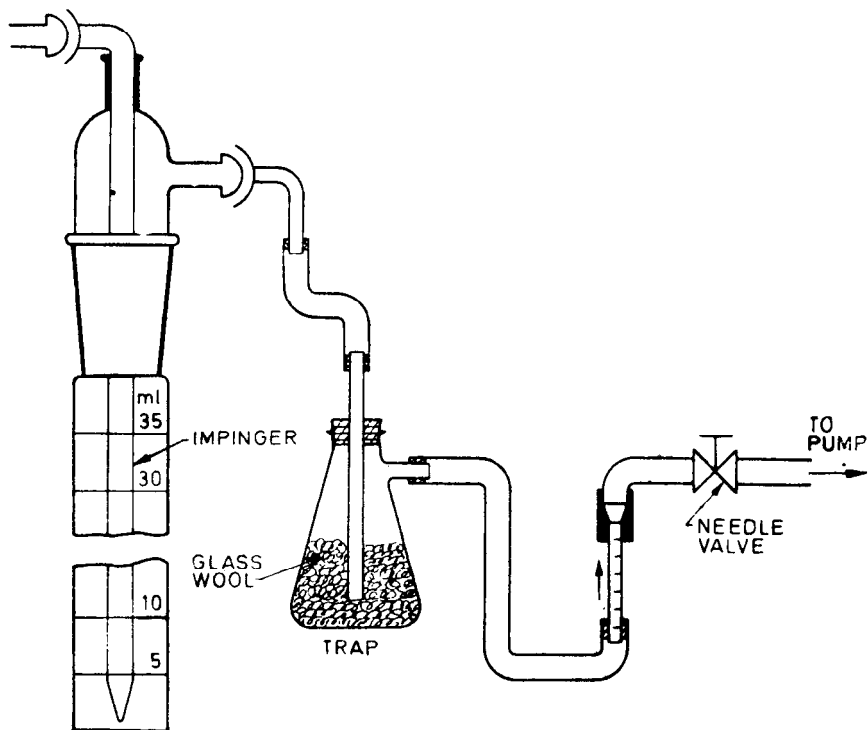
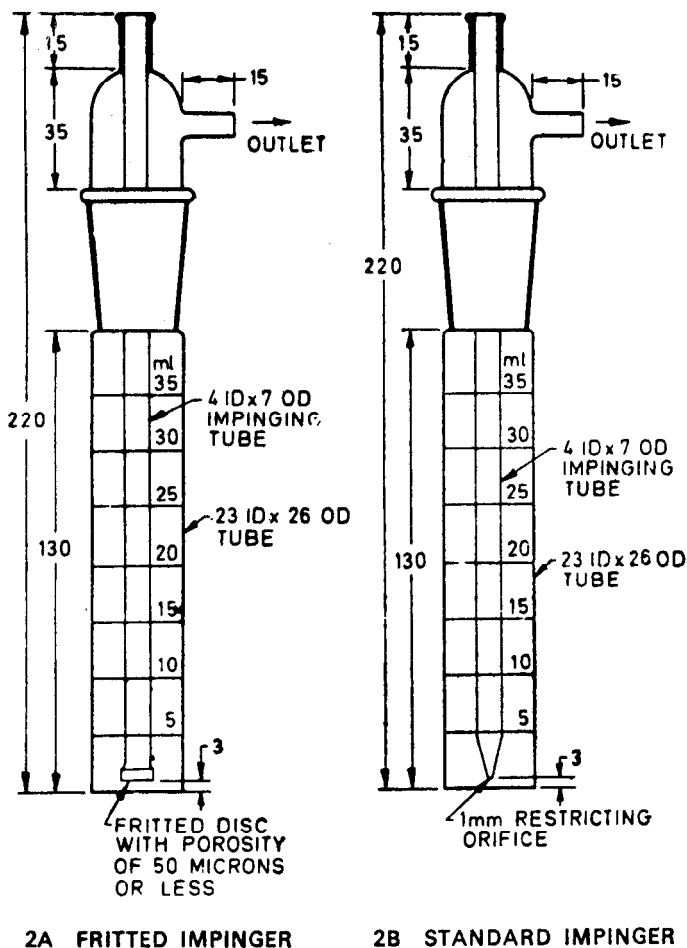


FIG. 1 SAMPLING TRAIN

7.1.1 Impingers — All-glass impinger, 35 ml capacity, 220 mm in length and 26 mm wide. Impinging end of the tube shall be of 1 mm bore size or preferably with fritted disc with porosity of 50 microns or less (see Fig. 2A and 2B). Impingers shall be made from borosilicate glass.



All dimensions in millimetres.

FIG. 2 IMPINGER FOR AIR SAMPLING

7.1.2 Flowmeter — A rotameter calibrated accurately for the flow range from 0 to 3 l/min.

7.1.3 Critical Orifice Device — This device is used to give a flow of about 1 l/min. Normally hypodermic needles are used as critical orifice. The needles shall be protected by membrane filters. The arrangement is illustrated in Fig. 3.

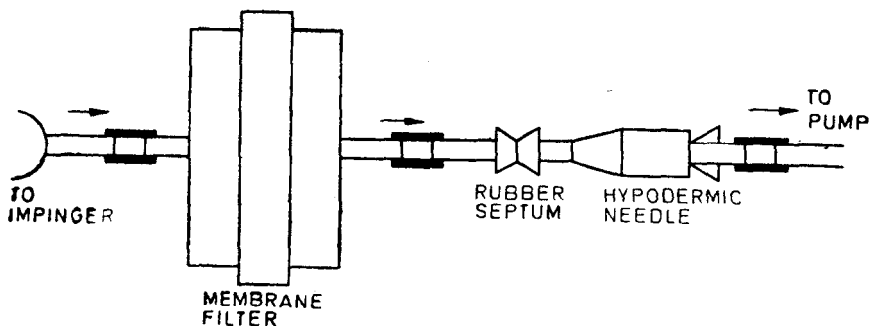


FIG. 3 CRITICAL ORIFICE FLOW DEVICE

7.1.4 Trap — This may be a membrane filter (see Fig. 3) or a glass wool trap (see Fig. 2).

7.1.5 Pump — It shall be a suction device, capable of drawing air into the sampling train at the rate of 10 to 20 l/min, provided with vacuum regulator and a moisture trap. It shall be of light mass, suitable for field work (portable), with continuous rating, self-lubricating and capable of operating on 230 V ac, 50 Hz.

8. PROCEDURE

8.1 Sampling

8.1.1 Procedures are described for short term (30 minutes and 1 hour) and for long term (24 hours) sampling. One can select different combinations of sampling rate and time to meet special requirements. Sample volumes shall be adjusted, so that linearity is maintained between transmission and concentration over the dynamic range.

8.1.2 Thirty-Minute and One-Hour Samplings — Place 20 ml of the absorbing solution in an impinger. Collect the sample at the rate of 1 l/min for 30 minutes, or at 0.5 l/min for 1 hour using either the rotameter, or a critical orifice device to control the flow. Shield the

absorbing reagent from direct sunlight during and after the sampling by covering the impinger with aluminium foil to prevent deterioration. Determine the volume of air sampled by multiplying the flow rate by the time in minutes and record the atmospheric pressure and temperature. Remove and stopper the impinger. If the sample is to be stored for more than a day before analysis, keep it at 5°C in a refrigerator.

8.1.3 Twenty Four-Hour Sampling — Place 20 ml of the absorbing solution in an impinger and collect the sample at the rate of 0.2 l/min for 24 hours. Make sure that no entrainment of solution results with the impinger. During collection and storage, protect from direct sunlight. Determine the total air volume by multiplying the air flow rate by the time in minutes. If storage is necessary, refrigerate the sample at 5°C.

8.2 Analysis

8.2.0 General — Both the samples and each of the reagents to be used for analysis shall be deoxygenated by passing a current of pure nitrogen through them for at least 5 minutes just before use.

8.2.1 Sample Preparation — If a precipitate is observed in the sample, remove it by centrifugation.

8.2.2 Add 1 ml of sulphamic acid solution. Add 0.6 ml of dilute N, N-dimethyl-*p*-phenylene diamine sulphate solution and 0.05 ml of ferric sulphate solution to the sample. Mix well by shaking the impinger after the addition of each reagent. Make up the evaporation losses by adding the absorbing solution to 20 ml mark of the impinger. Allow the mixture to develop full colour by letting it stand for 30 minutes. Also prepare a reagent blank following the above procedure.

8.2.3 Measure the colour intensity (percent transmission) of the sample in a spectrophotometer at 670 nm. Compute from calibration curve the concentration in micrograms of hydrogen sulphide in the sample.

9. CALCULATION

9.1 Hydrogen sulphide

$$\text{concentration, } \mu\text{g/m}^3 = \frac{\text{micrograms of hydrogen sulphide in the sample}}{\text{litres of air sampled}} \times 10^3$$

NOTE — 1 $\mu\text{g/litre}$ hydrogen sulphide = 0.72 ppm hydrogen sulphide at 25°C and 760 mmHg pressure.

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