

भारतीय मानक
वायु प्रदूषण मापन पद्धति
भाग 2 सल्फर डाईआक्साईड
(पहला पुनरीक्षण)

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 2 SULPHUR DIOXIDE

(First Revision)

ICS 13.040.20; 71.040.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 2001

Price Group 5

FOREWORD

This Indian Standard (Part 2) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Air Environment Protection Sectional Committee had been approved by the Chemical Division Council.

Practically all fuels in common use contain variable amounts of sulphur, most of which is discharged to the atmosphere as sulphur dioxide during combustion. In addition, specific industrial process produce large quantities of sulphur dioxide, some of which may escape into the air. The concentration to be found in the atmosphere is governed not only by the number and size of local sources but by factors, such as the height of chimneys and meteorological conditions.

Pollution of the air by sulphur dioxide is widespread since it exists wherever fossil fuels are burned. Concentrations in the atmosphere are normally far below the level which can be detected by odour but, being present to some extent at all times, the gas does produce detectable effects. It is harmful to stonework and metal work, it can damage plant tissues, and may aggravate such illnesses as bronchitis.

Because it is known to be potentially harmful both from the health and the economic point of view, a knowledge of the level at which sulphur dioxide is present in the air is important. No method of measuring sulphur dioxide using a physical property appears to have been developed and all the volumetric methods recommended are based on chemical reaction involving an absorbent.

There are several volumetric methods which are in use and applicable to a wide range of concentrations of sulphur dioxide. Of these, two methods have been thoroughly investigated and standardized. The first is the hydrogen peroxide method which is the simplest one to use and is normally operated over periods of 12 to 72 h. The second method is based on tetrachloromercurate. It is specific for sulphur dioxide and can be operated over sampling periods of 5 min to 24 h.

In the preparation of this standard considerable assistance has been derived from the following publications:

BS 1747-3 : 1969 Method for the measurement of air pollution, Part 3, Sulphur dioxide. British Standards Institution.

Report of the working party on methods of measuring air pollution and survey techniques. 1964, a publication of Organization for Economic Co-operation and Development, Paris.

Selected methods for the measurement of sulphur dioxide, a publication of US Department of Health, Education and Welfare, Public Health Service, USA.

This standard was published in 1969. In this the sampling clause has been modified to suit specific needs. Sampling efficiency has been defined, in addition to defining the precision and accuracy of the method. Applicability, range and sensitivity and the possible interferences of the method have also been identified.

The composition of the committee responsible for preparation of this standard is given in Annex A.

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 'Rules for rounding off numerical values (revised)'.

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 2 SULPHUR DIOXIDE

(First Revision)

1 SCOPE

This standard (Part 2) prescribes the methods for the measurement of concentration of sulphur dioxide present in the atmosphere.

2 REFERENCE

The following Indian Standard contains provisions which through reference in this text, constitutes provision of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	Title
IS 4167 : 1980	Glossary of terms relating to air pollution (<i>first revision</i>)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4167 shall apply.

4 FIELD OF APPLICATION

4.1 Applicability

The method is applicable to the measurement of sulphur dioxide in ambient air.

4.2 Range and Sensitivity

Concentrations of sulphur dioxide in the range of 25 to 1 050 $\mu\text{g}/\text{m}^3$ can be measured under the given conditions. Concentrations below 25 $\mu\text{g}/\text{m}^3$ can be measured by sampling larger volumes of air, but only if the absorption efficiency of the particular system is first determined and found to be satisfactory. Higher concentrations can be analyzed by using smaller gas samples, or a suitable aliquot of the collected sample. Beer's Law is followed through the working range from 0.03 to 1.0 absorbance unit. This corresponds to 0.8 to 27 μg of sulphite ion in 25 ml of final solution calculated as sulphur dioxide. The lower limit of detection of sulphur dioxide in 10 ml absorbing reagent is 0.75 μg based on twice the standard deviation which represents a concentration of 25 $\mu\text{g}/\text{m}^3$ in an air sample of 30 l.

5 INTERFERENCES

The effects of the principal known interferences have

been minimized or eliminated. Interferences by oxides of nitrogen are eliminated by 1 ml of 0.06 percent sulphamic acid. Ozone is made to decompose by allowing the solution to stand for some time prior to analysis. The interference of trace metals may be eliminated by the addition of 1 drop of 0.01 percent ethylene diamine tetra acetic acid (EDTA) to the absorbing solution prior to sampling. At least 60 μg iron (III), 10 μg manganese (II) and 10 μg chromium (III) in 10 ml absorbing reagent may be tolerated in the procedure. No significant interference was found from 10 μg copper (II) and 22 μg vanadium (V). Ammonia, sulphides, and aldehydes do not interfere.

6 PRINCIPLE

Sulphur dioxide is absorbed from air in a solution of potassium tetrachloromercurate (TCM). A dichlorosulphitomercurate complex which resists oxidation by the oxygen in the air, is formed. This complex is stable to strong oxidants such as ozone and oxides of nitrogen and therefore the absorber solution may be stored for some time prior to analysis. The complex is made to react with para rosaniline and methylsulphonic acid. The absorbance of the solution is measured by means of a suitable spectrophotometer.

7 REAGENTS

7.1 Distilled Water

Distilled water used shall be free from oxidants, particularly chlorine, which may not be removed by distillation. The criterion shall be observed whether water is prepared by distilling or deionizing or by using a combination of both techniques.

7.2 Potassium Tetrachloromercurate (TCM)-0.04 M

Dissolve 10.86 g mercuric chloride, 0.066 g EDTA, and 6.0 g potassium chloride in distilled water and bring to the mark in a 1 litre volumetric flask. The pH of this reagent shall be approximately 4.0 but it has been shown that there is no appreciable difference in collection efficiency over the range of pH 5 to 3. The absorbing reagent is normally stable for six months. If a precipitate forms, discard the reagent after recovering the mercury.

CAUTION — HIGHLY POISONOUS IF SPILLED ON SKIN. FLUSH OFF WITH WATER IMMEDIATELY.

7.3 Sulphamic Acid — 0.6 percent.

Dissolve 0.6 g of sulphamic acid in 100 ml of distilled water. Prepare fresh, when needed.

7.4 Formaldehyde — 0.2 percent approx.

Dilute 5 ml of formaldehyde solution (36 to 38 percent) to 1 litre with distilled water. Prepare fresh, when needed.

7.5 Stock Iodine Solution — 0.1 N

Take 12.7 g of iodine in a 250-ml beaker; add 40 g of potassium iodide and 25 ml of water. Stir to dissolve completely, then dilute to 1 litre with distilled water.

7.6 Iodine Solution — 0.01 N

Prepare approximately 0.01 N iodine solution by diluting 50 ml of stock solution (see 7.5) to 500 ml with distilled water.

7.7 Starch Indicator Solution

Triturate 0.4 g of soluble starch and 0.002 g of mercuric iodide preservative with a little water and add the paste slowly to 200 ml boiling water. Continue boiling until the solution is clear; cool, and transfer to a glass-stoppered bottle.

7.8 Stock Sodium Thiosulphate Solution — 0.1 N.

7.8.1 Prepare a stock solution by taking 25 g of sodium thiosulphate pentahydrate in a beaker, add 0.1 g of sodium carbonate, and dissolve using boiled and cooled distilled water making the solution up to a final volume of 1 litre. Allow the solution to stand one day before standardizing.

7.8.2 To standardize, accurately weigh, to the nearest 0.1 mg, 1.5 g of primary standard potassium iodate dried at 180°C, dissolve, and dilute to 500 ml in a volumetric flask. Take 50 ml of iodate solution by pipette into a 500 ml iodine flask, add 2 g of potassium iodide and 10 ml (1:10) hydrochloric acid and stopper the flask. After 5 min titrate with stock thiosulphate solution to a pale yellow colour. Add 5 ml starch indicator solution and continue the titration until the blue colour disappears. Calculate the normality of the stock solution.

Normality of Thiosulphate Solution

The normality of this solution, N , is calculated as follows:

$$N = \frac{M \times 2.80}{V}$$

where

V = volume in ml of thiosulphate used, and

M = mass in g of potassium iodate.

7.9 Sodium Thiosulphate Titrant — 0.01 N.

Dilute 100 ml of the stock thiosulphate solution to

1 litre with freshly boiled distilled water.

7.10 Stanadarized Sulphite Solution for Preparation of Working Sulphite — TCM Solution

Dissolve 0.30 g of sodium metabisulphite (NaHSO_3) or 0.40 g of sodium sulphite (Na_2SO_3) in 500 ml of freshly boiled and cooled distilled water. Sulphite solution is unstable; it is therefore important to use water of the highest purity to minimize this instability. This solution contains the equivalent of 320–400 $\mu\text{g/ml}$ of SO_2 . The actual concentration of the solution is determined by adding excess iodine and back-titrating with standard sodium thiosulphate solution. To back-titrate, pipette out 50 ml of the 0.01 N iodine solution into each of two 500-ml iodine flasks A (blank) and B (sample). To flask A (blank) add 25 ml of distilled water and into flask B (sample) measure 25 ml of sulphite solution. Stopper the flasks and allow to react for 5 min. By means of a burette containing standardized 0.01 N thiosulphate, titrate each flask in turn, to a pale yellow colour. Then add 5 ml of starch solution and continue the titration until the blue colour disappears.

7.11 Working Sulphite — TCM Solution

Measure 2 ml of the standard sulphite solution (7.10) into 100-ml volumetric flask by pipette and bring to mark with 0.04 M TCM (see 7.2). Calculate the concentration of sulphur dioxide in the working solution in μg of sulphur dioxide per millilitre. This solution is stable for 30 days if kept in the refrigerator at 5°C. Otherwise, prepare fresh, when needed.

7.11.1 Calculation

$$C = \frac{(V_1 - V_2) \times N \times 32\,000 \times 0.02}{25}$$

where

C = concentration of SO_2 solution in $\mu\text{g/ml}$ in ml;

V_1 = volume in ml of thiosulphate used for blank;

V_2 = volume in ml of thiosulphate used for sample;

N = normality of thiosulphate;

32000 = milli equivalent weight SO_2 , μg ;

25 = volume of standard sulphite solution, ml; and

0.02 = dilution factor.

7.12 Purified Para Rosaniline Stock Solution — 0.2 percent concentration.

7.12.1 The para rosaniline dye shall have a wavelength of maximum absorbance at 540 nm when assayed in a buffered solution of 0.1 M sodium acetate — acetic

acid; the absorbance of the reagent blank which is temperature-sensitive to the extent of 0.015 absorbance unit/ $^{\circ}\text{C}$, shall not exceed 0.170 absorbance unit at 22°C with a 1 cm optical path length, when the blank is prepared according to the prescribed analytical procedure and to the specified concentration of the dye; the calibration curve, shall have a slope of 0.030 ± 0.002 absorbance unit/ $\mu\text{g SO}_2$ at this path length when the dye is pure and the sulphite solution is properly standardized.

7.12.2 Preparation of Stock Solution

Dissolve 0.5 g of para rosaniline chloride in 100 ml distilled water. Keep it for 2 days and filter the solution. The filtrate solution is stable for 3 months, if stored in the refrigerator.

7.12.3 Working Para Rosaniline Solution

Add 15 ml concentrated hydrochloric acid to 10 ml stock para rosaniline solution and dilute to 250 ml with distilled water in a 250 ml volumetric flask. It may be stored at room temperature in an amber coloured bottle for one to two weeks, if stored in a refrigerator.

8 APPARATUS

8.1 Absorbers normally used in air pollution sampling are acceptable for concentrations above $3 \mu\text{g}/\text{m}^3$

(or 0.000 1 ppm). An all-glass midget impinger, as shown in Fig. 1, is suitable for 30-min and 1-h samples. For 24-h sampling, an absorber train could be assembled.

8.2 Polypropylene Two-Port Tube Closures

8.3 Glass Impingers

Having a tube of 60 mm outer diameter and 15 mm length. One end is drawn to small diameter so that a No. 79 jewellers drill bit passes through, while a No. 78 jeweller's drill bit does not pass through. The other end is fire polished.

8.4 Polypropylene Tubes

Having a dimension of $164 \text{ mm} \times 32 \text{ mm}$.

8.5 Pump

Capable of maintaining an air pressure differential greater than 0.7 atm at the desired flow rate.

8.6 Air Flowmeter of Critical Orifice

A calibrated rotameter or critical orifice (see Fig. 2) capable of measuring air flow within 2 percent. For 30 min sampling, a 22-gauge hypodermic needle of 25 mm long may be used as a critical orifice to give a flow of about 1 litre/min. For 1-h sampling, a 22-gauge needle

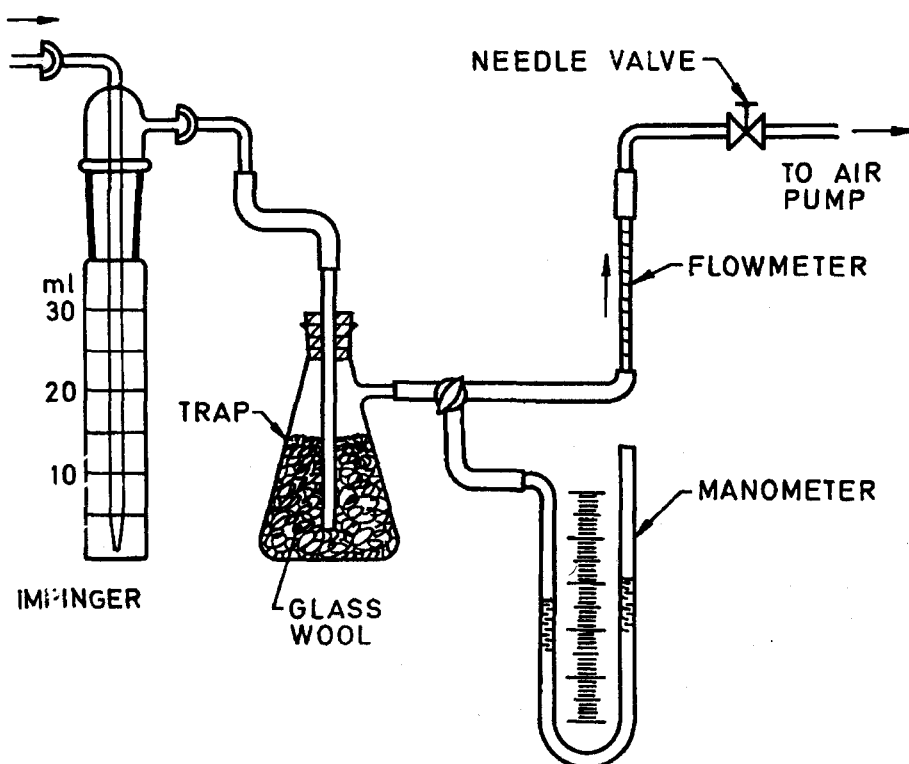


FIG. 1 SO₂ SAMPLING TRAIN

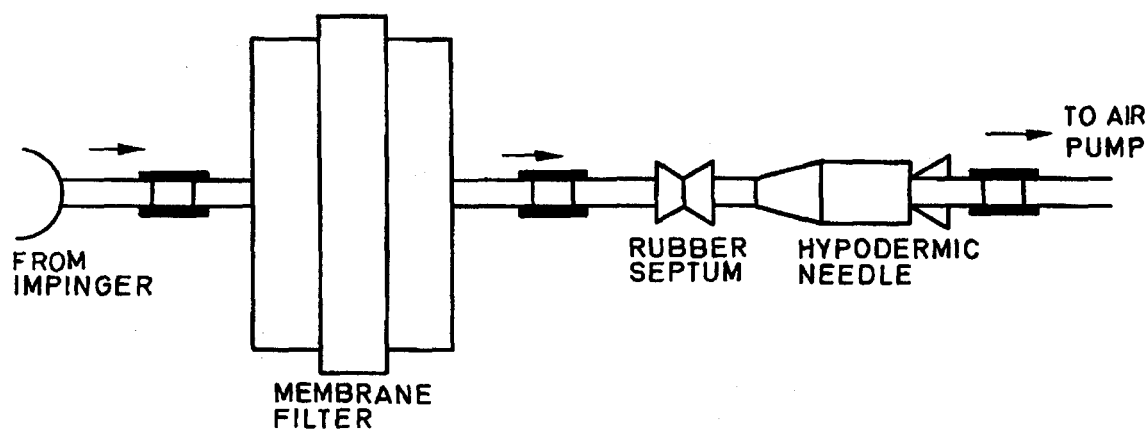


FIG. 2 CRITICAL ORIFICE CONTROL

(of 0.318 mm internal diameter) and of 16 mm length gives a flow of about 0.5 litre/min and a 27 gauge needle (of 0.191 mm internal diameter) and of 9 mm length gives a flow of about 0.2 l/min.

8.7 A spectrophotometer suitable for measurement of absorbance at 560 nm with an effective spectral band width of not more than 8 nm is required. Problems may occur with spectrophotometer having greater spectral band widths. The wavelength calibration of the instrument shall be verified. If transmittance (T) is measured, this can be converted to absorbance (A) by the formula:

$$A = \log_{10} (1/T)$$

9 SAMPLING

9.1 Procedures are described for short-term (30 min and 1 h) and for long-term (24 h) sampling. One can select different combinations of sampling rate and time to meet special needs. Sample volumes shall be adjusted, so that linearity is maintained between absorbance and concentration over the range in question.

9.2 30-min and 1-h Sampling

Insert a midjet impinger into the sampling system. Add 10 ml of TCM solution to the impinger. Collect sample at the rate of 1 l/min for 30 min, or 0.5 l/min for 1 h using either a rotameter, or a critical orifice, to control flow. Shield the absorbing reagent from direct sunlight during and after sampling by covering the impinger with aluminium foil to prevent deterioration. Determine the volume of air sampled by multiplying the flow rate by time (in minutes). Record the atmospheric pressure and temperature. Remove and stopper the impinger. If the sample has to be stored for more than a day before analysis, keep it at 5°C in a refrigerator; during hot weather, sampling is not recommended unless it is possible to refrigerate the samples.

NOTE — After sample collection, the solutions are

relatively stable. At 22°C, losses of sulphur dioxide occur at the rate of 1 percent per day. When samples are stored at 5°C for 30 days, no detectable losses of sulphur dioxide occur. The presence of EDTA enhances the stability of sulphur dioxide in solution, and the rate of decay is independent of the concentration of sulphur dioxide.

9.3 24-Hours Sampling

Place 50 ml of TCM solution in a larger absorber and collect the sample at 0.2 l/min for 24 h from midnight to midnight. Make sure no entrainment of solution results with the impinger. Protect from direct sunlight during collection and storage. Determine the total air volume by multiplying the air flow rate with time (in minutes). The correction of 24 h measurements for temperature and pressure may be difficult and is not normally done. However, the accuracy of the measurement will be improved if meaningful corrections can be applied. If the sample has to be stored, keep it at 5°C in a refrigerator. During hot weather, sampling is not recommended unless it is possible to refrigerate the samples (see also Note given under 9.2).

10 PROCEDURE

10.1 Sample Preparation

After collection of sample if a precipitate is observed in the sample, remove it through centrifuging.

10.1.1 30-Minutes and 1-Hour Sample

Transfer the sample quantitatively to a 25-ml volumetric flask using about 5 ml of distilled water for rinsing. Delay the analysis for 20 min to allow any ozone to decompose.

10.1.2 24-Hour Samples

Dilute the entire sample to 50 ml with absorbing solution. Measure 5 ml of the sample into a 25-ml volumetric flask by pipette for chemical analysis. Bring volume to 10 ml with absorbing reagent. Delay analysis for 20 min to allow any ozone to decompose.

10.2 Determination

10.2.1 For each set of determinations prepare a reagent blank by adding 10 ml of unexposed TCM solution to a 25-ml volumetric flask. Prepare a control solution by measuring 2 ml of working sulphite-TCM solution into a 25-ml volumetric flask by pipette. To each flask containing sample or control solution or reagent blank, add 1 ml of 0.6 percent sulphamic acid and allow to react for 10 min to destroy the nitrite resulting from oxides of nitrogen. Add 2 ml of 0.2 percent formaldehyde solution and 5 ml of para rosaniline solution. Start a laboratory timer that has been set for 30 min. Bring all flasks to volume with freshly boiled and cooled distilled water and mix thoroughly. Within 30 to 60 min, determine the absorbances of the sample, reagent blank, and the control solution at 560 nm using cells with a 1 cm path length.

10.2.2 Use distilled water (not the reagent blank) as the optical reference. This is important because of the colour sensitivity of the reagent blank to temperature changes which may be induced in the cell compartment of a spectrophotometer. Do not allow the coloured solution to stand in the absorbance cells, because a film of dye may be deposited. Clean cells with the help of alcohol and pipe-cleaner after use. If the temperature of the determinations does not differ by more than 2°C from the calibration temperature, the reagent blank should be within 0.03 absorbance unit of the y-intercept of the calibration curve (*see* 10.4.2). If the reagent blank differs by more than 0.03 absorbance unit from that found in the calibration curve, prepare a new curve.

10.3 Absorbance Range

If the absorbance of the sample solution lies between 1.0 and 2.0 the sample solution can be diluted to 1:1 with a portion of the reagent blank and read within a few minutes. Solutions with higher absorbance can be diluted up to six fold with the reagent blank in order to obtain on-scale readings within 10 percent of the true absorbance value.

10.4 Calibration and Efficiencies

10.4.1 Flowmeter and Hypodermic Needle

Calibrate flowmeter and hypodermic needle against a calibrated wet test meter.

10.4.2 Calibration Curve-Procedure with Sulphite Solution

Measure by graduated pipette amounts of the working sodium sulphite tetrachloro mercurate (TCM) solution (such as 0, 0.5, 1, 2, 3 and 4 ml) into a series of 25-ml volumetric flasks. Add sufficient TCM solution to each flask to bring the volume to approximately 10 ml. Then add the remaining reagents as described in 10.2. For maximum precision use a constant temperature bath.

The temperature of calibration shall be maintained within $\pm 1^\circ\text{C}$ and within the range of 20 to 30°C. The temperature of calibration and that of analysis shall be maintained within two degrees. Plot the absorbance against the total concentration in micrograms sulphur dioxide for the corresponding solution. The total micrograms sulphur dioxide in solution equals the concentration of the standard in micrograms sulphur dioxide per millilitre times the millilitre of sulphite solution added ($\mu\text{g SO}_2 = \mu\text{g ml SO}_3 \text{ per ml} \times \text{ml added}$). A linear relationship should be obtained, and the y-intercept should be within 0.03 absorbance unit of the zero standard absorbance. For maximum precision determine the line of best fit using regression analysis by the method of least squares. Determine the slope of the line of best fit, calculate its reciprocal, and denote as B, the calibration factor. This calibration factor can be used for calculating results provided there are no radical changes in temperature or pH. At least one control sample containing a known concentrations is recommended to ensure the reliability of this factor.

10.4.3 Calibration Curve

Procedure with sulphur dioxide permeation tubes.

10.4.3.1 General considerations

Atmospheres containing accurately known amounts of sulphur dioxide at levels of interest can be prepared using permeation tubes. In the systems for generating these atmospheres, the permeation tube emits sulphur dioxide gas at a known, low, constant rate, provided the temperature of the tube is held constant ($\pm 0.1^\circ\text{C}$) and the tube has been accurately calibrated at the temperature of use. The sulphur dioxide gas diffusing from the tube is carried by a low flow of inert gas to a mixing chamber where it is accurately diluted with air free of sulphur dioxide to the level of interest and the sample taken. These systems are shown schematically in Fig. 3 and 4.

10.4.3.2 Preparation of standard SO_2 atmospheres

Permeation tubes may be prepared or purchased. (Tubes with a certified permeation rate are available from the National Bureau of Standards, Washington, D.C.) TUBE permeation rates from 0.2 to 0.4 $\mu\text{g/min}$, inert gas flows of about 50 ml/min, and dilution air flow rates from 1.1 to 15 l/min conveniently give standard atmospheres containing desired levels of sulphur dioxide, that is 25 to 390 $\mu\text{g/m}^3$. The concentration of sulphur dioxide in any standard atmosphere can be calculated as given below.

Concentration of Standard Sulphur Dioxide Atmosphere

The concentration of sulphur dioxide (C) in such atmospheres, in micrograms of sulphur dioxide per

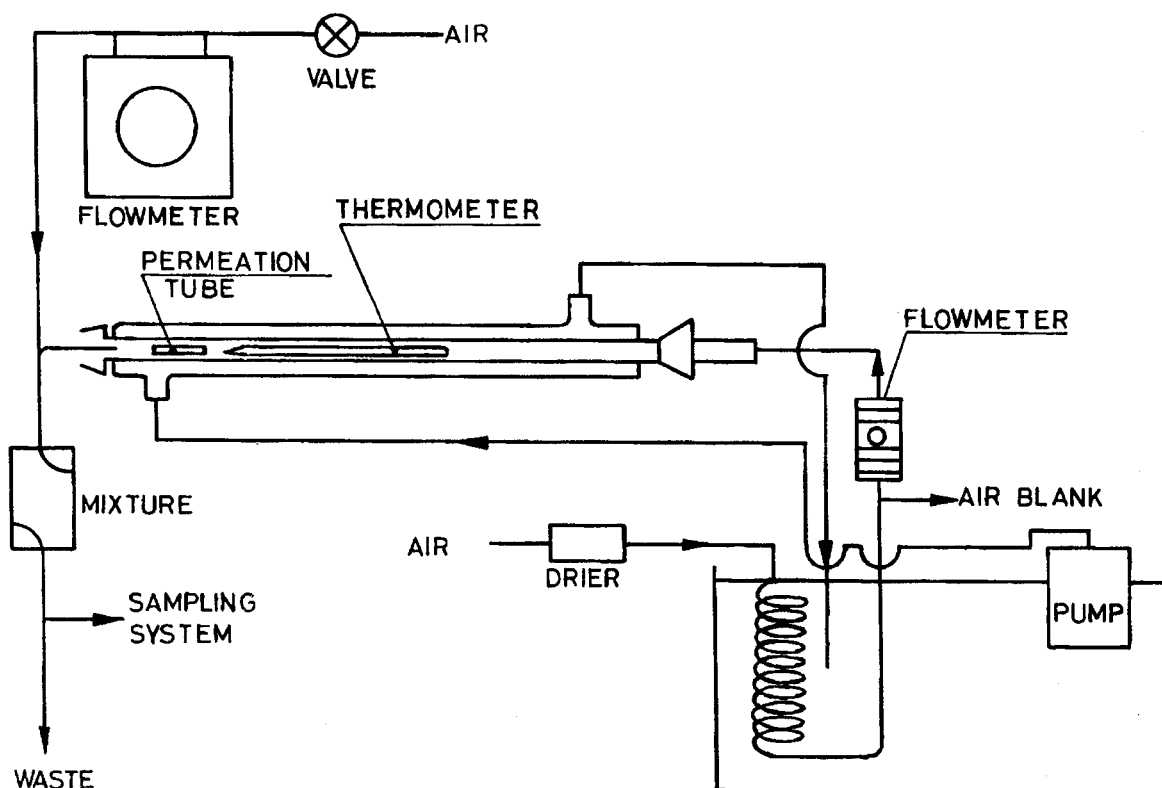


FIG. 3 LABORATORY APPARATUS

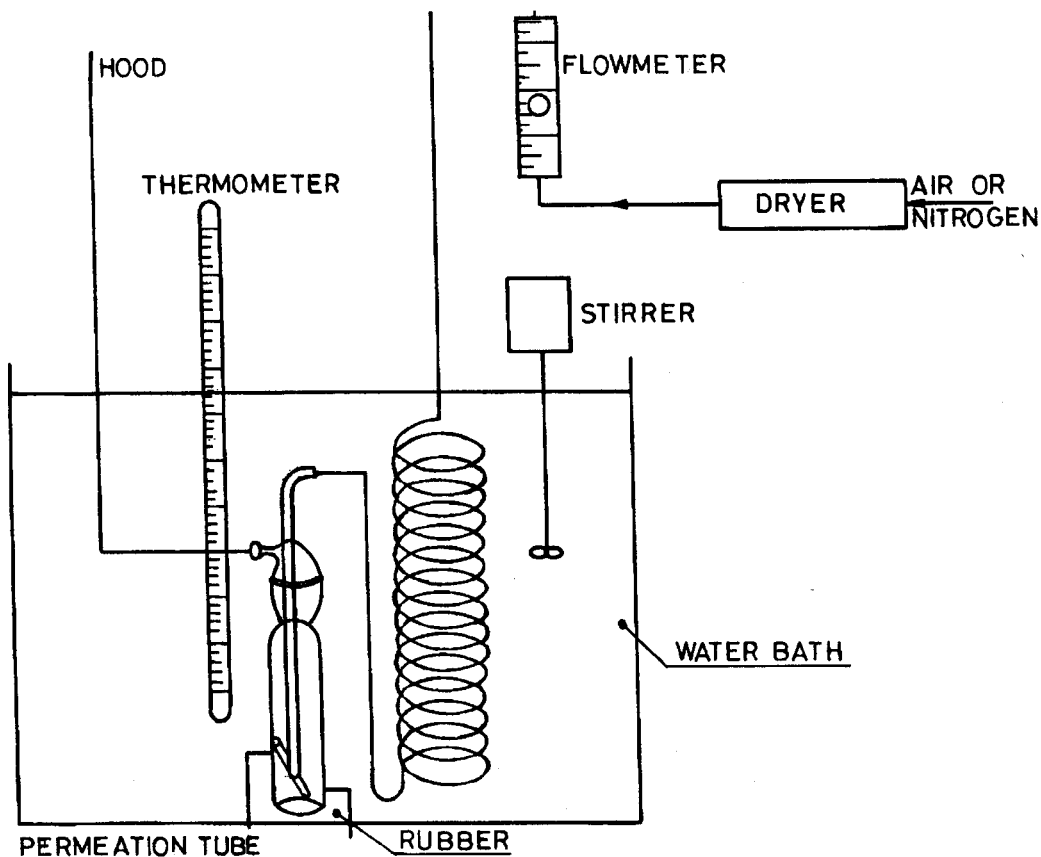


FIG. 4 PERMEATION TUBE CALIBRATION APPARATUS

cubic metre is

$$C = \frac{P \times 10^3}{R_d + R_i}$$

where

P = permeation rate at standard temperature, $\mu\text{g}/\text{min}$;

R_d = flow rate dilution air at standard temperature and pressure, l/min ; and

R_i = flow rate of inert gas at standard temperature and pressure, l/min .

Conversion of Volume

Convert the volume of air sampled to the volume in litre at the reference conditions of 25°C and $760\text{ mm}/\mu\text{g}$

$$V_n = V \times \frac{P}{760} \times \frac{298}{t + 273}$$

where

V_n = volume of air at 25°C and 760 mm Hg , l ;

V = volume of air sampled, l ;

P = barometric pressure, mm Hg ; and

t = temperature of air sampled, $^\circ\text{C}$.

Sulphur Dioxide Concentration using Sulphite Solution

When sulphite solutions are used to prepare calibration curves, compute the concentration of sulphur dioxide, (C) in micrograms per cubic metre, in the sample as follows:

$$C = \frac{(A - A_0) \times 10^3 \times B}{V_r} \times D$$

where

A = sample absorbance;

A_0 = reagent blank absorbance;

10^3 = conversion of litres to cubic metres;

V_r = the sample corrected to 25°C and 760 mm Hg ;

B = calibration factor, $\mu\text{g}/\text{absorbance unit}$; and

D = dilution factor: for 30-min and 1-h samples, $D = 1$; for 24-h samples, $D = 10$.

Sulphur Dioxide Concentration using Sulphur Dioxide Gas Standard Atmosphere

When sulphur dioxide gas standard atmospheres are used to prepare calibration curves, compute the sulphur dioxide concentration (C) in micrograms per cubic metre, in the sample by the following formula:

$$C = (A - A_0) \times B$$

where

A = sample absorbance;

A_0 = reagent blank absorbance; and

B = reciprocal of slope (see 6.6.2).

Conversion of Micrograms per Cubic Metre to Parts per Million

If desired, the concentration of sulphur dioxide may be calculated as parts per million of sulphur dioxide at reference conditions as follows:

$$\text{SO}_2, \text{ppm} = \mu\text{g SO}_2/\text{m}^3 \times 3.82 \times 10^{-4}$$

10.4.3.3 Sampling and preparation of calibration curve

Prepare a series (usually six) of standard atmospheres containing sulphur dioxide levels from 25 to $390\text{ }\mu\text{g SO}_2/\text{m}^3$. Sample each atmosphere using similar apparatus and taking exactly the same air volume as will be done in atmospheric sampling. Determine absorbance values (Y -axis), draw the straight line of best fit, and determine the slope. Alternatively, regression analysis by the method of least squares may be used to calculate the slope. Calculate the reciprocal of the slope and denote as B .

10.4.3.4 Sampling efficiency

Collection efficiency is generally above 98 percent; efficiency may fall off, however, at concentrations below $25\text{ }\mu\text{g}/\text{m}^3$.

11 PRECISION AND ACCURACY

Relative standard deviation at the 95 percent level is 4.6 percent for the analytical procedure using standard sample.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Air Environmental Protection Sectional Committee, CHD 32

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Review of Indian Standards

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This Indian Standard has been developed from Doc : No. CHD 32 (143).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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