

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART VI NITROGEN OXIDES

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INDIAN STANDARDS INSTITUTION
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Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART VI NITROGEN OXIDES

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0. FOREWORD

0.1 This Indian Standard (Part VI) was adopted by the Indian Standards Institution on 14 July 1975, after the draft finalized by the Air Pollution Sectional Committee had been approved by the Chemical Division Council.

0.2 The method covers the procedure for the determination of oxides of nitrogen based on the procedure of Jacobs and Hochheiser. This involves diazotization of sulphanilic acid by nitrous acid derived from nitrogen dioxide followed by a coupling reaction with N (1-naphthyl) ethylenediamine dihydrochloride to form the dye. The method is generally specific for nitrogen dioxide. In high temperature combustion processes, some oxides of nitrogen are yielded which further oxidize to nitrogen dioxide in air at ordinary temperatures. So the method is employed to determine the nitrogen oxides content in the atmosphere as nitrogen dioxide.

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 prescribes a method for measurement of nitrogen oxides (as NO_2) in air.

2. PRINCIPLE AND APPLICABILITY

2.1 Nitrogen oxides as nitrogen dioxide are collected by bubbling air through a sodium hydroxide solution to form a stable solution of sodium nitrite. The nitrite ion produced during sampling is determined colorimetrically by reacting the exposed absorbing reagent with phosphoric acid, sulphanilamide, and N (1-naphthyl) ethylenediamine dihydrochloride.

*Rules for rounding off numerical values (revised).

IS : 5182 (Part VI) - 1975

2.2 The method is applicable to collection of 24 hour samples in the field and subsequent analysis in the laboratory.

3. RANGE AND SENSITIVITY

3.1 The range of the analysis is 0.01 to 1.5 $\mu\text{g NO}_2/\text{ml}$. With 50 ml absorbing reagent and a sampling rate of 200 ml/min for 24 hours, the range of the method is 20 to 740 $\mu\text{g}/\text{m}^3$ (0.01 to 0.4 ppm) nitrogen dioxide.

3.2 A concentration of 0.01 μg oxides of nitrogen (as NO_2/ml) will produce an absorbance of 0.005 using 1-cm cells.

4. INTERFERENCE

4.1 The interference of sulphur dioxide is eliminated by converting it to sulphuric acid with hydrogen peroxide before analysis.

5. APPARATUS

5.1 Absorber — Polypropylene tubes 164×32 mm, equipped with polypropylene two port closures. Rubber stoppers cause high and varying blank values and should not be used. A gas dispersion tube with a fritted end of porosity B (70 to 100 μm maximum pore diameter) is used.

5.2 Spectrophotometer — capable of measuring absorbance at 540 nm. Band width is not critical.

6. REAGENTS

6.1 Absorbing Reagent — Dissolve 4.0 g of sodium hydroxide in distilled water and dilute to 1 000 ml.

6.2 Sulphanilamide — Dissolve 20 g of sulphanilamide in 700 ml of distilled water. Add, with mixing, 50 ml of concentrated phosphoric acid (85 percent) and dilute to 1 000 ml. This solution is stable for a month if refrigerated.

6.3 NEDA Solution — Dissolve 0.5 g of N (1-naphthyl) ethylenediamine dihydrochloride in distilled water. This solution is stable for a month if refrigerated and protected from light.

6.4 Hydrogen Peroxide — Dilute 0.2 ml of 30 percent hydrogen peroxide to 250 ml with distilled water. The solution may be used for a month if protected from light.

6.5 Standard Nitrite Solution — Dissolve sufficient desiccated sodium nitrite [NaNO_2 (assay of 97 percent or greater)] and dilute with distilled water to 1 000 ml so that a solution containing 1 000 $\mu\text{g NO}_2/\text{ml}$ is obtained. The amount of sodium nitrite to use is calculated as follows:

$$G = \frac{1.500}{A} \times 100$$

where

G = amount in g of sodium nitrite;

1.500 = gravimetric factor in converting NO_2 into sodium nitrite;
and

A = assay, percent.

7. PROCEDURE

7.1 Sampling — Assemble the sampling train including the absorber, critical flow control device and pump [see IS: 5182 (Part V)-1974*]. Add 50 ml absorbing reagent to the absorber. Disconnect funnel, insert calibrated flowmeter, and measure flow before sampling. If flow rate before sampling is less than 85 percent of needle calibration, check for leak or change filters as necessary. Remove flowmeter and replace funnel. Sample for 24 hours from midnight to midnight and measure flow at the end of sampling period.

7.2 Analysis — Replace any water lost by evaporation during sampling. Pipette 10 ml of the collected sample into a test-tube. Add 1.0 ml of hydrogen peroxide solution, 10.0 ml of sulphanilamide solution, and 1.4 ml of NEDA solution with thorough mixing after the addition of each reagent. Prepare a blank in the same manner using 10 ml of absorbing reagent. After a 10 minute colour-development interval, measure the absorbance at 540 nm against the blank. Read $\mu\text{g NO}_2/\text{ml}$ from the standard curve (see 8.2).

8. CALIBRATION

8.1 Sampling

8.1.1 Calibration of Flowmeter — Using a wet test meter and a stopwatch, determine the rates of airflow (ml/min) through the flowmeter at several ball positions. Plot ball positions *versus* flow rates.

8.1.2 Calibration of Hypodermic Needle — Connect the calibrated flowmeter, the needle to be calibrated, and the source of vacuum in such a way that the direction of airflow through the needle is the same as in the sampling

*Methods for measurement of air pollution: Part V Sampling of gaseous pollutants.

train. Read the position of the ball and determine flow rate in ml/min from the calibration chart prepared in 8.1.1. Reject all needles not having flow rate of 190 to 210 ml/min before sampling.

8.2 Calibration Curve—Dilute 5.0 ml of the 1000 $\mu\text{g NO}_2/\text{ml}$ solution to 200 ml with absorbing reagent. This solution contains 25 $\mu\text{g NO}_2/\text{ml}$. Pipette 1, 2, 5 and 15 ml of the 25 $\mu\text{g NO}_2/\text{ml}$ solution into 50, 50, 100 and 250 volumetric flasks and dilute to the mark with absorbing reagent. The solutions contain 0.50, 1.00, 1.25, and 1.50 $\mu\text{g NO}_2/\text{ml}$ respectively. Run standards as instructed in 7.2. Plot absorbance *versus* $\mu\text{g NO}_2/\text{ml}$.

9. CALCULATION

9.1 Calculate the volume of air sampled as follows:

$$= \frac{F_1 + F_2}{2} \times T \times 10^{-6}$$

where

V = volume of air sampled, m^3 ;

F_1 = measured flow rate before sampling, ml/min;

F_2 = measured flow rate after sampling, ml/min; and

T = time of sampling, min.

9.2 Calculate the concentration of nitrogen dioxide as follows:

$$\text{Mass of nitrogen dioxide in } \mu\text{g per m}^3 = \frac{(\mu\text{g NO}_2/\text{ml}) \times 50}{V \times 0.35}$$

where

50 = volume of absorbing reagent used in sampling, ml;

V = volume of air sampled, m^3 ; and

0.35 = overall average efficiency.

9.2.1 If desired, concentration of nitrogen dioxide may be calculated as ppm NO_2 .

$$\text{NO}_2, \text{ppm} = (\mu\text{g NO}_2/\text{m}^3) \times 5.32 \times 10^{-4}$$

**AMENDMENT NO. 1 FEBRUARY 2004
TO
IS 5182 (PART 6) : 1975 METHODS FOR
MEASUREMENT OF AIR POLLUTION
PART 6 NITROGEN OXIDES**

(First Revision)

(Page 4, clause 6.3, line 1) — Substitute the following for the existing:

6.3 NEDA Solution — Dissolve 0.5 g of N (1-Naphthyl) ethylenediamine dihydrochloride in 500 ml distilled water.'

(CHD 32)

Reprography Unit, BIS, New Delhi, India