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(पहला पुनरीक्षण)

Indian Standard
METHODS FOR MEASUREMENT OF
AIR POLLUTION
PART 11 BENZENE
(*First Revision*)

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Environmental Protection Sectional Committee had been approved by the Chemical Division Council.

This Indian Standard was first published in 1982. Based on the experience gained and the development on the analytical procedures the technical committee responsible for the formulation of this standard decided to revise it. In this revision a newer method having a different type of collection, desorption media and N, N-Bis-cyanoethylformamide (BCEF) has been introduced.

Benzene is used as raw material in the manufacture of synthetic chemicals, dyes, paints, medicines, explosives, etc, and as solvent. Benzene is liable to cause various types of hazards due to its toxicity, flammability and explosiveness. Acute poisoning results from inhalation of high concentration of benzene vapours (above 3 000 ppm by volume in air) usually for short periods of time, often a matter of minutes and chronic poisoning results from daily exposure to unsafe concentrations of benzene vapours in air over a prolonged period of time. Regular and systematic procedures for inspection are, therefore, necessary to ensure safety against the hazards involved.

In reporting the result of a test 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*)'.

The compositions of the Sectional Committee and Subcommittee responsible for the formulation of this standard are given at Annex A.

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 11 BENZENE

(First Revision)

1 SCOPE

This standard prescribes two gas chromatographic methods for measurement of benzene in air.

2 METHOD 1

2.1 Principle

A measured volume of air is drawn through silica gel by means of a personal sampler. Any benzene vapour present in the sampled air is absorbed. The silica gel is shaken with *iso*-propyl alcohol (IPA) which absorbs benzene quantitatively. The components of alcohol layer are separated by gas-liquid chromatography over polypropylene glycol — 400 (PPG) using flame ionization detection.

2.2 Apparatus

2.2.1 Gas Chromatograph

Any suitable gas chromatograph with flame ionization detector with the following set of column and conditions:

Length × ID	: 3.6 m × 3 mm
Stationary phase	: PPG-400
Solid support	: Chromosorb W acid washed (180 μm-250 μm)
Stationary phase/ support	: 0.25
Detector	: Flame ionization detector (FID)
Column temperature	: 75°C
Injection part temperature	: 75°C
Carrier gas	: Nitrogen (Hydrogen may also be used) 3 l/h
Sample injection	: 10 μl
Lower detection limit	: 0.1 ml (vapour)/m ³ (0.3 mg/m ³)

2.2.2 Sampling Device

Consisting of portable (intrinsically safe if high benzene concentration is involved) pump,

battery powered, with absorption tube and a flow meter in the range of 0.5 to 5 l/min. Any other suitable portable flame proof suction device with absorption tube and a flow meter capable of measuring at a sampling rate in the range of 0.5 to 1.5 l/min may also be used.

2.3 Reagents

2.3.1 Silica Gel

300 to 600 μm size, chromatography grade. Activate for at least 16 hours at 190 ± 5°C, preferably in a vacuum oven. Allow to cool in a desiccator without desiccant. Do not open the desiccator until the gel is to be used.

2.3.2 Benzene — AR grade.

2.3.3 *Iso*-propyl Alcohol (IPA) — Free from benzene, AR grade.

2.4 Sample

It is recommended that such quantity of air is used that approximately 50 μg of benzene vapour is absorbed on the silica gel. In the chromatogram obtained according to conditions described in 2.2.1 and 2.6, the benzene peak will show a height of about 30 mm (see Fig. 1).

For a benzene concentration of 1 ml (vapour)/m³ the volume of air to be passed through is approximately 20 litres. Normally, a sample volume of 20 to 30 litres is sufficient. However, while determining extremely low concentrations, up to 70 litres of air may be sampled if the ambient temperature is below 26°C and relative humidity is less than 70 percent. Beyond these limits the humidity of air may interfere as water absorbed on the silica gel may displace trapped benzene. This displacement starts interfering when the silica gel has absorbed 0.15 g of water per gram of silica gel. Therefore, 0.12 g of water per gram of silica gel may be taken as an allowable maximum and the maximum sample size may be calculated as follows:

$$V = \frac{12 \times 10^3 M}{HC}$$

where

V = maximum volume of air sample, l;

M = mass of silica gel used, g;

H = relative humidity of the atmosphere, percent; and

C = concentration of water vapour in air saturated with water vapour at the temperature of test and 101.3 kPa, mg/l.

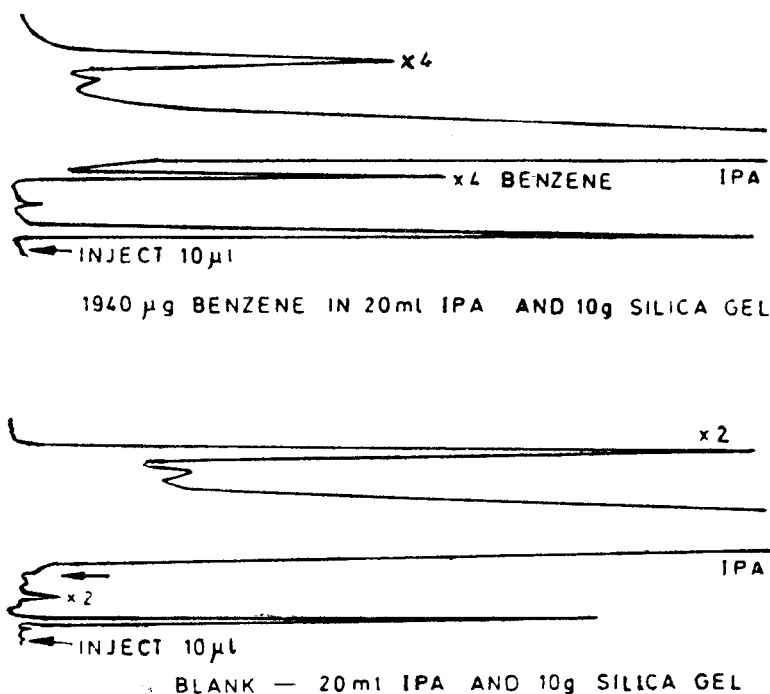
2.5 Calibration

Prepare five calibrating solutions of benzene in *iso*-propyl alcohol containing 15, 25, 125 and 500 mg/l of benzene. Pipette 20 ml of each solution into separate stoppered bottles containing 10 g of silica gel. Shake for two minutes and allow to settle for at least five minutes. Analyse

alcohol layer on gas chromatograph and plot the benzene peak height/area against concentration of benzene in $\mu\text{g}/20\text{ ml}$ (see Fig. 2).

2.6 Procedure

Fill the absorption tube with 5 to 15 g of silica gel. Mount the tube in the holder, read the timer, and start the pump. Pass requisite volume of air sample through the tube at a rate not exceeding 1.5 l/min. Transfer the silica gel into a 50 ml bottle. Add 20 ml *iso*-propyl alcohol, stopper, shake for two minutes, and allow to settle for at least five minutes. Analyse the alcohol layer for benzene using conditions identical to those for calibration. Measure the height/area of benzene peak. Read from calibration graph the concentration of benzene in *iso*-propyl alcohol in micrograms per 20 ml.



Column conditions:

Length \times ID	— 180 cm \times 4/6 mm, copper
Column	— 20 percent PPG 400 on chromosorb P (180 μm — 250 μm)
Carrier gas	— Hydrogen, 5 l/h
Flow rate of air	— 50 l/h
Temperature	— 75°C
Sample size	— 10 μl
Recorder range	— 2 mV
Chart speed	— 5 mm/minute
Retention time of benzene	— 18 mm from point of injection

FIG. 1 ILLUSTRATIVE CHROMATOGRAM

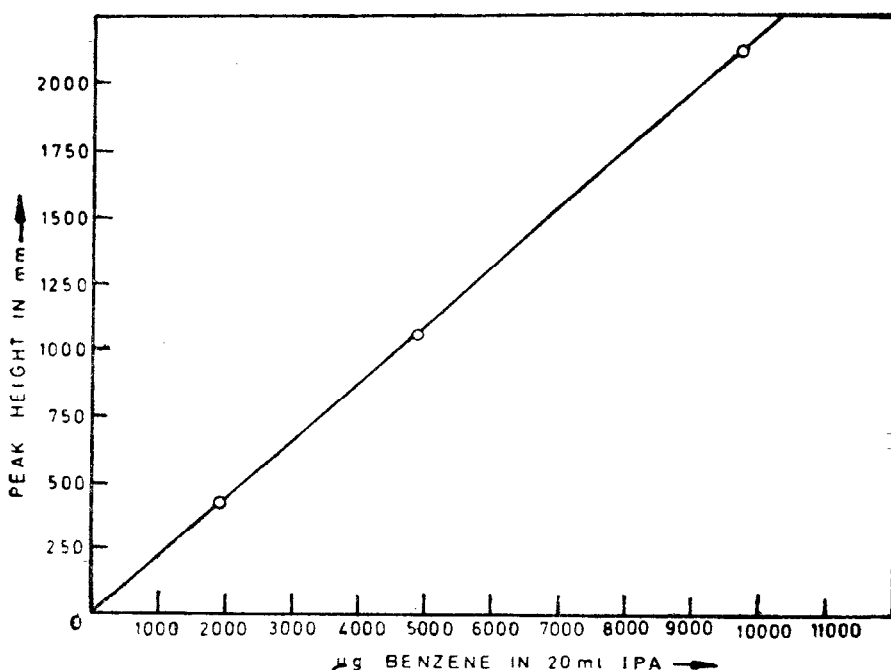


FIG. 2 ILLUSTRATIVE CALIBRATION CURVE

2.7 Calculation

Calculate the amount of benzene vapour content in the air at 0°C and 101.3 kPa (1 013 mbar, 760 mmHg) by any of the following equations:

$$\text{Benzene, mg/m}^3 = \frac{101.3 A (273 + T)}{273 P F t}$$

$$\text{Benzene, ml (vapour)/m}^3 = \frac{101.3 A (273 + T)}{3.48 \times 273 P F t}$$

where

A = concentration of benzene in *iso*-propyl alcohol (IPA), read from the calibration curve, µg/20 ml;

T = temperature in 0°C of the air;

P = atmospheric pressure, kPa;

F = flow rate of sampling, l/min; and

t = time in minutes of sampling.

NOTE — In case barometers calibrated in Pascals are not available, express pressure in millibar or millimetres of mercury and replace the factor 101.3 by 1 013 or 760, respectively.

3 METHOD 2

3.1 Principle

A measured volume of air is drawn through a tube containing two sections of activated charcoal to trap the benzene. The charcoal in the two sections are then transferred to two vials containing carbon disulphide to desorb the

benzene. The carbon disulphide is then injected into a gas chromatograph using a N, N-Bis-Cyanoethylformamide (BCEF) column and flame ionization detector.

3.2 Apparatus

3.2.1 Gas Chromatograph

Any suitable gas chromatograph with a flame ionization detector having the following column and conditions:

Length × I. D.	1.8 m × 4.5 mm
Stationary phase	BCEF
Solid support	Chromosorb P NAW (180 µm — 250 µm)
Stationary phase/ support	3-7
Detector	Flame ionization detector (FID)
Column temperature	70°C
Injection port temperature	150°C
Detector temperature	150°C
Carrier gas	Nitrogen 20 ml/min
Sample size	5 microlitre

3.2.2 Personal Sampler

Intrinsically safe, portable, battery operated pump with NIOSH type charcoal tubes and a

flow meter for measuring the rate of air being sampled.

3.3 Reagents

3.3.1 Benzene — AR grade.

3.3.2 Hexane

Analytical reagent grade, free from impurities coeluting with benzene.

NOTE — If required, purification can be carried out by passing through column containing activated molecular sieves 13 X in a fume cupboard.

3.3.3 Carbon Disulphide

Analytical reagent grade, free from impurities coeluting with benzene.

NOTE — Carbon disulphide should be used in a fume cupboard. The vapours may ignite on contact with a hot surface. Prevent contact with the skin. Handle benzene with the same precautions.

3.4 Sample

3.4.1 The quantity of air sampled will depend on the levels of benzene expected. A volume of 15 litres sampled at 100 ml/min would be sufficient to detect 0.3 mg/m³ of benzene. The volume may be reduced if the amount of benzene in the second charcoal layer is more than 25 percent of that found in the first layer.

3.5 Calibration

3.5.1 Prepare a solution of 50 µg/µl benzene in hexane. Prepare further dilute solutions of 10, 1.0 and 0.10 µg/µl from this. In a clean vial introduce 1.00 ml of carbon disulphide and immediately add 10 µl of the standard 50 µg/µl benzene in hexane solution. Cap it and mix. Inject 5 µl into the gas chromatograph and let the chromatogram develop. Repeat with the 10, 1.0 and 0.10 µg/µl standard solutions. Prepare a blank by introducing 10 µl hexane instead of the standard solutions and inject 5 µl of the blank also. Plot the areas of the benzene peaks after correction for blank against the amounts of benzene present.

3.5.2 Determine the desorption efficiency by injecting 10 µl of the standard 50 µg/µl benzene in hexane into the front (first) charcoal layer of a charcoal tube connected to the sampler. Allow the pump to run for 10 minutes at 100 ml/min. Stop the pump and analyse the two layers separately as 3.6.1. Repeat for standards and a blank in the same way. Read the amount of benzene from the calibration curve prepared in 3.5.1.

Calculate the desorption efficiency for each standard solution, as follows:

Desorption efficiency

$$(DE) = \frac{(A_1 + A_2 - B_1) \times 200}{C}$$

where

A_1 = amount of benzene read from the calibration curve for the first section, µg;

A_2 = amount of benzene read from the calibration curve for the second section, µg;

B_1 = amount of benzene read from the calibration curve for the blank µg; and

C = amount of benzene injected into the charcoal tube (500 µg, 100 µg, 10 µg and 1 µg respectively), µg.

Plot the DE values against the net amounts read from the calibration curve ($A_1 + A_2 - B_1$) $\times 200$ for the standard solutions (DE curve).

3.6 Procedure

3.6.1 The smaller section of the charcoal tube is inserted into the pump after carefully breaking the two ends. Avoid the use of rubber tubing. Start the pump and sample at 100 ml/min. The sample volume will depend on the amount of benzene expected. Note the flow rate and the sampling time. After sampling is over, cap the tube with plastic cap and store it in a refrigerator as soon as possible, if the analysis is to be done later.

After sampling transfer the charcoal from the two sections of the charcoal tube into two vials, immediately add 1.0 ml of carbon disulphide. Cap it and mix. Allow the benzene to be desorbed for 30 minutes with occasional swirling. Inject 5 µl of carbon disulphide from each vial into the gas chromatograph and let the chromatogram develop (see Fig. 3).

Repeat for a blank using a charcoal tube from the same batch and following the same procedure except that no air is passed through the tube.

Read the amount of benzene from the area of the benzene peak from the calibration curve. If the amount of benzene in the rear smaller section is more than 25 percent of that found in the larger front section, repeat the test taking a smaller volume.

3.7 Calculate the amount of benzene in the air at 0 °C and 101.3 kPa by using the following equation:

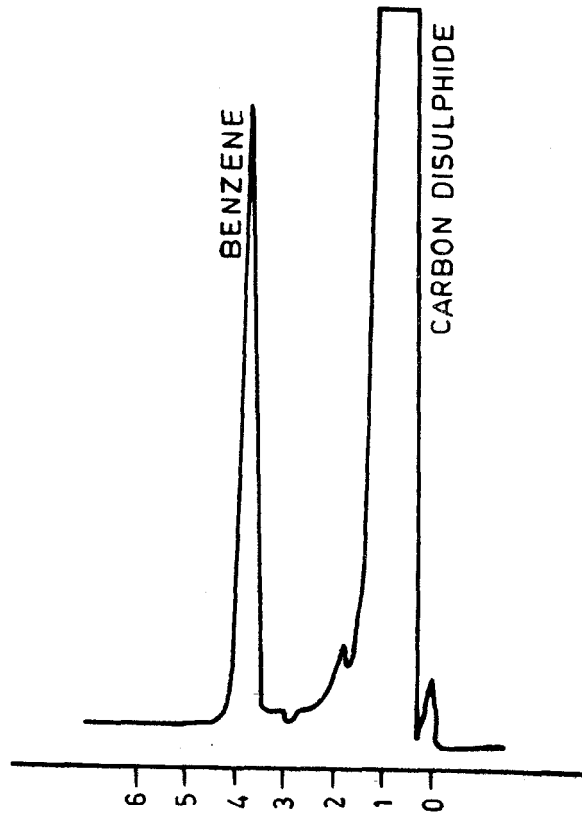


FIG. 3 ILLUSTRATIVE CHROMATOGRAM

Benzene, $\text{mg}/\text{m}^3 =$

$$\frac{(F_1 + F_2 - B_2) \times 200 \times (273 + t) \times 101.3}{DE \times T \times G \times 273 \times P}$$

Benzene, $\text{ml (vapour)}/\text{m}^3 =$

$$\frac{(F_1 + F_2 - B_2) \times 200 \times (273 + t) \times 101.3}{3.48 \times DE \times T \times G \times 273 \times P}$$

where

F_1 = amount of benzene read from the calibration curve for the first section (front section), μg ;

F_2 = amount of benzene read from the calibration curve for the second section (rear section), μg ;

B_2 = amount of benzene read from the calibration curve for the blank, μg ;

DE = desorption efficiency as read from the DE curve taking $(F_1 + F_2 - B_2) \times 200$ as the amount of benzene found;

T = sampling time, min;

G = flow rate of the sampler, l/min ;

t = temperature of the air, $^{\circ}\text{C}$; and

P = atmospheric pressure in kPa .

NOTE — In case pressure is to be expressed in mm of mercury, replace the factor 101.3 by 760.

ANNEX A

(Foreword)

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