Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“पुराने को छोड नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 11 BENZENE, TOLUENE AND XYLENE (BTX)

(Second Revision)

ICS 13.040.30; 71.080.15
FOREWORD

This Indian Standard (Part 11) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

The aromatic hydrocarbons, namely, benzene, toluene and xylene have many industrial uses, most notably as a solvent for numerous materials and as a fuel additive. They are also used in the manufacture of various chemicals, rubber, insecticides, pharmaceuticals, explosives, etc. Though they are very useful chemicals, they are also extremely hazardous. They are highly flammable. While benzene is well known to be carcinogenic, there is recent evidence of carcinogenicity of toluene and xylene at high concentrations in experimental animals. It should also be noted that any future epidemiological observations of cancer risks associated with toluene or xylene would have to take account of the suspected effects of benzene impurities. Regular and systematic procedures for inspection are, therefore, necessary to ensure safety against the hazards involved.

This standard was first published in 1982 and revised in 1993 based on the development of the analytical procedures to introduce a newer method having a different type of collection, desorption media and use of N-N-Bis-cyanoethylformamide (BCEF) for determination of benzene only. The Committee responsible for the formulation of this standard further decided to revise it based on the experience gained during the last decade as well as technological development in the field. During the revision method for determination of toluene and xylene are incorporated. The revised methods include both active and passive sampling using low flow pump. Apart from the conventional CS$_2$ desorption, the modern techniques of automated thermal desorption without use of organic solvents is also incorporated in this revision.

There is no ISO Standard on the subject. The standard is prepared based on the measuring techniques available and use in India.

The Committee composition responsible for the formulation of this standard is given at Annex A.

In reporting the results 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 11  BENZENE, TOLUENE AND XYLENE (BTX)

(Second Revision)

1 SCOPE
This standard (Part 11) prescribes active and passive sampling techniques with three gas chromatography based analytical methods for measurement of benzene, toluene and xylene in air.

2 REFERENCES
The following standard contains provisions, which through reference in this text constitute provisions 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 edition of the standard indicated below:

<table>
<thead>
<tr>
<th>IS No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>4167:1980</td>
<td>Glossary of terms relating to air pollution</td>
</tr>
</tbody>
</table>

(First Revision)

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

4 METHOD 1 (ACTIVE SAMPLING USING ACTIVATED CHARCOAL TUBES, DESORBED BY CARBON DISULPHIDE)

4.1 Principle
The charcoal tubes are available in different sizes and contain varying amount of activated charcoal. The ambient air is sucked through the tube using a low flow sampler used for collection of BTX sample in a way that results in an enrichment of the relevant substances in the activated charcoal. Desorption of the adsorbed benzene is done using carbon disulphide (CS₂). The substances desorbed in the CS₂ are analyzed by capillary gas chromatography. A flame ionization detector (FID) is used for analysis while quantification is performed using the internal/external standard.

4.2 Apparatus

4.2.1 Low Volume Pump — Portable, battery powered pump with a low flow controller with operating range between 5 to 500 ml/min (+0.2 ml/min) to suck the air sample.

NOTE — Wherever necessary intrinsically safe pumps may be used.

4.2.2 Sampling Sorbent (Sample) Tubes — Glass lined (or fused silica lined) stainless steel tube or stainless steel sorbent tubes of 6 mm O.D., 8.9 cm long tubes with a 6 cm of sorbent bed of 200 mg of activated charcoal (coconut shell) or other suitable adsorbent. A typical sorbent/sample tube is shown in Fig. 1 and Fig. 2.

Modular glass or stainless tube (OD 6-8 mm length 10-15 cm) packed with chromatography grade coconut shell activated charcoal, chromatography grade. Tube must have provision for fitting of backup section with provision to measure pressure drop across the tube during sampling. The minimum quantity of charcoal required in front section is 200 mg and in backup section 50 mg. Glass beads or any other porous inert material must be packed in inlet part of front section for uniform distribution of sucked air through tube at the time of sampling.

4.2.3 Gas Chromatograph — Any suitable gas chromatograph with flame ionization detector (FID) with fused silica capillary columns having a length of 25 m or more, an internal diameter of 320 μm or below and with a stationary phase film thickness less than 1.5 μm as follows or equivalent may be recommended:

Capillary 624 Column : Coating: cyanopropyl phenyl polysiloxane, Length × ID : 30 m × 0.25 mm, Film thickness \( (d_f) = 1.4 \mu m \)

Capillary Column : Coating : 5 percent phenyl, 95 percent dimethyl polysiloxane Length × ID : 25 m × 0.20 mm, Film thickness \( (d_f) = 0.33 \mu m \)

Wall Coated Column : Coating: Fused Silica PONA CB, Length × ID : 50 m × 0.21 mm, Film thickness \( (d_f) = 0.5 \mu m \)
Capillary Column : Coating : Fused silica 100 percent dimethyl polysiloxane, Length x ID: 30 m x 0.32 ID, Film thickness ($d_f$): 1.0 μm

4.3 Reagents

4.3.1 Suitable Adsorbent — Chromatographic grade activated charcoal (coconut shell) or other suitable adsorbent, that is, Chromosorb 106 or other suitable adsorbent having particle size in the range 60 to 80 mesh.

4.3.2 Carbon Disulphide (CS₂) — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0005 percent, Benzene 0.001 percent, $H_2O$ < 0.02 percent.

4.3.3 Benzene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0003 percent, $H_2O$ < 0.02 percent.

4.3.4 Toluene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0003 percent, $H_2O$ < 0.02 percent.

4.3.5 Xylene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0003 percent, $H_2O$ < 0.02 percent.

4.3.6 Carrier Gas — Helium or Nitrogen of purity > 99.9 percent, $H_2O$ < 0.02 percent, Residues < 0.0003 percent.

4.4 Sampling

4.4.1 Selection of Sorbent Tube — Samples are collected in glass sampling tube filled with a activated charcoal (coconut shell), Chromosorb 106 or other suitable adsorbent.

4.4.2 Sample Tubes Labelling

Sample tubes are labelled with a unique identification number and the direction of sampling flow. If empty sample tubes are obtained without labels, it is important to label and condition them before and after they are packed with adsorbent prior to use them for sampling.

---

**FIG. 1** SORBENT/SAMPLE TUBE FOR ACTIVE SAMPLING FILLED WITH ACTIVATED COCONUT SHELL CHARCOAL (CSC)

**FIG. 2** INLET AND OUTLET OF THE SORBENT/SAMPLE TUBE
4.4.3 Sampling Procedure and Sampling Rate

A sample is collected by opening a tube at two ends, connecting it to a sample pump, and pulling air through the tube with the pump. Airborne chemicals are trapped onto the surface of the sorbent:

a) Two tubes are used in series to take care of breakthrough (if any) compatible to the thermal desorber. The sampling is carried out using low flow sampler. The schematic diagram of sampling train is given in Fig. 3.

b) Keep the tube in a vertical position during sampling to prevent the possibility of channelling that can lead to under sampling.

c) The arrow on the tube indicates air flow direction and should point to the tube holder and pump. If no arrow is present, the smallest section should be near the tube holder.

d) Sampling flow rate in the range of 20-100 ml/min is required (+2 percent) for ambient air.

e) A sample component may breakthrough from the back end of tube, if excessive flow rates are used. Sample is to be discarded, if the breakthrough is observed more than 10 percent. If analyzed concentration in backup section is more than 10 percent of front section, sample needs to be discarded.

The tube is then sealed with push-on caps, and sent to a laboratory for analysis.

4.4.4 Storage of Blank and Sampled Tubes

Seal clean, blank sorbent tubes and sampled tubes using inert fittings and PTFE ferrules. Wrap capped tubes individually in uncoated aluminum foil. Use clean, sealable metal cans containing a small packet of activated charcoal or activated charcoal:silica gel for storage and transportation of multiple tubes. Store the multi-tube storage container in a clean environment at 4 ± 1°C.

4.5 Procedure

4.5.1 Calibration

Prepare a mix stock standard solution of 50 μg/μl of benzene, toluene and xylene each gravimetrically using a micro syringe in the eluting solvent that is CS₂. Prepare further diluted solutions of concentration range of 10, 1.0, 0.10 μg/μl with CS₂ from stock standard in a clean vial. Make up to 1 ml solution. Introduce immediately 1 μl standard solution into the injector of GC directly and plot the curve between the concentration and response (peak area). Prepare fresh standard solutions with each batch of samples. A typical chromatogram of standard mixture is given in Fig. 4.

4.5.2 Analytical Procedure

Samples collected through active sampling (sorbent tubes) are extracted or desorbed by conventional solvent (generally 1-5 ml of carbon disulphide) using ultrasonication for 15 min to remove analyte from the sorbent material. Desorbed samples are analyzed using gas chromatograph (GC) fitted with capillary column and flame ionization detector (FID). A single tube may provide enough samples to permit several analyses.

The following set of conditions is generally used:

a) Gas flow:
   - Nitrogen : 30 ml/min (FID make up + Column),
   - Hydrogen : 30 ml/min
   - Air : 300 ml/min
   - (Column flow 1 ml/min approximately)
NOTE — Instead of nitrogen, helium may also be used as carrier gas for flow setting and corresponding retention time of analytes may vary.

Capillary column 624, Coating: cyanopropyl phenyl polysiloxane, Length x ID: 30 m x 0.25 mm, Film thickness (d.) : 1.4 μm

Temperature programming
Injection port: 250°C
FID: 300°C

Column/Oven: 50°C (hold for 3 min), ramp 1 @ 10°C/min to 140°C (1 min) ramp 2 @ 20°C/min to 240°C (1 min)

Injection volume: 5 μl, Total run time: 19.5 min, Split: 10

- Benzene RT 6.80 min, Search window: 1.00 s, 3.00 percent
- Toluene RT 9.18 min, Search window: 1.00 s, 3.00 percent
- Xylene RT 11.37 min, Search window: 1.00 s, 3.00 percent

NOTE — Temperature programming and retention time (RT) of analyte may vary column to column to get appropriate resolution of analyte peaks. Injection volume and split may also vary according to nature and probable concentration of analyte present in the extract.

4.6 Calculation

Amount of analyte compound found on tube can be converted into mg/m³, by using the formula:

\[
\text{Concentration (μg/m³) at (STP)} = \frac{C \times 101.3 \times (273 + T)}{273 \times P}
\]

where

- \( C \) = amount of compound found injection sample volume from standard curve, in μg/μl;
- \( V_1 \) = total volume of the sample extracted in ml;
- \( V_2 \) = volume of sample extract injected into GC, in μl; and
- \( V_3 \) = volume of air sucked through the tube, in m³.

Blank value is to be subtracted from the amount of compound found in the sample.
where

\[ C = \text{concentration at ambient condition, in } \mu\text{g/m}^3; \]
\[ T = \text{temperature of the ambient air, in } ^\circ\text{C}; \]
\[ P = \text{atmospheric pressure, in kPa}. \]

5 Method 2 (Active Sampling Using Tenax/Chromosorb 106 Sorption Tubes, Desorbed Thermally)

5.1 Principle

Thermal desorption tubes filled with Tenax TA or other suitable adsorbent as Chromosorb-106, etc., are used for adsorption of benzene, toluene, and xylene in place of charcoal tube. The ambient air is sucked through the tube using a low flow personal sampler in a way that results in an enrichment of the relevant substances on the adsorbent. These tubes are directly connected to the automated thermal desorbers coupled with the gas chromatograph equipped with capillary column and flame ionization detector. The thermal desorption technique offer the advantage of a greatly improved analytical sensitivity, as solvent is not used in this process and the collected sample is not diluted. In most cases analytical recovery is close to 100 percent and desorption efficiency corrections are not required.

5.2 Apparatus

5.2.1 Sampling Device: Low Volume Pump — Intrinsically safe, portable, battery powered pump (SKC, PA, USA or equivalent make) (see Fig. 3) with a low flow capable of accurate and adjustable flow controller with operating range between 5 to 500 ml/min to suck the air sample with great accuracy in the range of 20-100 ml/min is required (+2 percent). The time programmable, built in flow indicator, rechargeable battery operated low flow pump with adjustable run time up to 8 h should be preferred for sampling of BTX.

5.2.2 Sampling Sorbent (Sample) Tube — Automated Thermal Desorption (ATD) tubes of stainless steel filled with absorbing material are required. Stainless steel or glass sorbent tubes (see Fig. 5) of 8.9 cm long, 6 mm O.D. with a 6 cm sorbent bed in the central portion packed with greater than 200 mg of solid adsorbent material (that is Tenax TA, Chromosorb106 or any other suitable adsorbent).

NOTE — To be suitable for thermal desorption, sorbent must meet exact specifications that include low contaminant background, high thermal stability and sufficient adsorptive strength to retain components of interest and should also release them quickly when heat is applied.

5.2.3 Automated Thermal Desorption Apparatus (Two-Stage Thermal Desorption)

Two-stage automated thermal desorption is recommended to use heat and a flow of inert (carrier) gas to extract volatiles from a solid adsorbent matrix directly into the carrier gas and transfer them to downstream system elements such as the analytical column of a GC.

Two-stage automated thermal desorption is used for the best high resolution capillary chromatography (that is, analytes desorbed from the sorbent tube must be refocused before being rapidly transferred to the GC analytical column).

Typical key components and operational stages of a two-stage desorption system are presented in Fig. 6 and Fig. 7.

5.2.4 Focusing Tube

The narrow (typically < 3 mm ID) tube containing a small bed of sorbent, which is maintained near or below ambient temperature and used to refocus analytes thermally desorbed from the sorbent tube. The focusing trap is typically packed with 20 mg of Carbopack™ B (60/80 mesh) and 50 mg of a Carboxen™ 1000-type sorbent (60/80 mesh).
Once all the BTX have been transferred from the sorbent tube to the focusing tube, the focusing tube is heated rapidly to transfer the analytes into the capillary column of GC in the form of a band of vapor.

5.2.5 Electronic Cryogen Systems
Automated thermal desorber have electronic systems to cool the focusing tube or cold trap. Other non-automated desorber require typically cryogens, that is, liquid nitrogen, liquid argon, or liquid carbon dioxide to cool the focusing tube.

The cryogen-free trap cooling option with a multistage Peltier electrical closed cycle coolers is used. At its low temperature, the trap must provide quantitative analyte retention for target compounds as well as quantitative and rapid desorption of target analytes.

5.2.6 Thermal Desorber — GC Interface
The interface line is leak-tight and lined with an inert
material such as deactivated fused silica. Alternatively, thread the capillary column itself through the heated transfer line/interface and connected directly into the thermal desorber.

Place the sealed tubes on the thermal desorber (Perkin Elmer Model ATD 400 Automated System or equivalent). Heat the interface between the thermal desorber and the GC uniformly.

Other thermal desorbers may have different arrangements for automation. Alternatively, use equivalent manual desorption.

NOTE — Use of a metal syringe-type needle or unheated length of fused silica pushed through the septum of a conventional GC injector is not recommended as a means of interfacing the thermal desorber to the chromatograph. Such connections result in cold spots, cause band broadening and are prone to leaks.

5.2.7 High Resolution Capillary Column Chromatography
Any suitable gas chromatograph equipped with flame ionization detector (FID) with fused silica capillary columns having a length of 25 metres or more, an internal diameter of 320 μm or below and with a stationary phase film thickness less than 1.5 μm as follows or equivalent may be recommended:

Capillary 624 Column : Coating: cyanopropyl phenyl polysiloxane, Length × ID : 30 m × 0.25 mm, Film thickness \((d_f) : 1.4 \mu m\)

Capillary Column : Coating: 5 percent phenyl 95 percent dimethyl polysiloxane, Length × ID : 25 m × 0.20 mm, Film thickness \((d_f) : 0.33 \mu m\)

Wall Coated Column : Coating: Fused Silica PONA CB, Length × ID : 50 m × 0.21 mm, Film thickness \((d_f) : 0.5 \mu m\)

Capillary Column : Coating: Fused silica 100 percent dimethyl polysiloxane, Length × ID : 30 m × 0.32 mm, 1.0 μm film thickness

5.3 Reagent

5.3.1 Carbon Disulphide — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.000 5 percent, Benzene < 0.001 percent, H₂O < 0.02 percent.

5.3.2 Benzene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.000 3 percent, H₂O < 0.02 percent.

5.3.3 Toluene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.000 3 percent, H₂O < 0.02 percent.

5.3.4 Xyylene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.000 3 percent, H₂O < 0.02 percent.

5.3.5 Carrier Gas — Helium or Nitrogen of purity > 99.9 percent, H₂O < 0.02 percent, Residues < 0.000 3 percent.

5.4 Sampling

5.4.1 Sampling Location — Site should be free from any obstacle to free flow of the air in the vicinity.

5.4.2 Selection of Sorbent Tube and Sorbent Mesh Size — Samples are collected in SS or glass sampling tube filled with Tenax TA, Chromosorb 106 or other suitable adsorbent (two in series to take care of breakthrough, if any) and compatible to the thermal desorber. The sorbents of particle size in the range 60 to 80 mesh should be used for tube packing.

5.4.3 Conditioning the Tube
Condition newly packed tubes for at least 2 h (30 min for preconditioned, purchased tubes) at 320°C while passing at least 30 ml/min of pure Nitrogen or Helium carrier gas through them.

Tube conditioning before reuse of sample tube is also must. Once conditioned, seal the tube with brass, 1/4 inch fittings and PTFE ferrules. Wrap the sealed tubes in uncoated aluminium foil and place the tubes in a clean, air-tight, opaque container.

A package of clean sorbent material, for example, activated charcoal or activated charcoal/silica gel mixture, may be added to the container to ensure clean storage conditions.

Store in a refrigerator (organic solvent-free) at 4±1°C, if not to be used within a day. On second and subsequent uses, the tubes will generally not require further conditioning as above. However, tubes with an immediate prior use indicating high levels of pollutant trace gases should be reconditioned prior to continued usage.

NOTE — Other sorbents may require different conditioning temperatures.

5.4.4 Sample Tubes Labelling
Sample tubes are labelled with a unique identification number and the direction of sampling flow. Stainless steel tubes are most conveniently labelled by engraving. Glass tubes are best labelled using a temperature resistant paint. If empty sample tubes are obtained without labels, it is important to label them before packing and condition them after packing with adsorbent prior to use them for sampling.
5.4.5 Sampling Procedure and Sampling Rate

A sample is collected by opening a tube at two ends, connecting it to a sample pump, and pulling air through the tube with the pump. Airborne chemicals are trapped onto the surface of the sorbent:

a) Two tubes are used in series to take care of breakthrough (if any) compatible to the thermal desorber. The sampling is carried out using low flow sampler. The schematic diagram of sampling train is given in the Fig. 3.

b) Keep the tube in a vertical position during sampling to prevent the possibility of channelling that can lead to under-sampling.

c) The arrow on the tube indicates air flow direction and should point to the tube holder and pump. If no arrow is present, the smallest section should be near the tube holder.

d) Sampling flow rate in the range of 20 - 30 ml/min is required (+0.2 ml/min) for ambient air.

e) A sample component may breakthrough from the back end of tube if excessive flow rates are used.

Sample is to be discarded if the breakthrough is observed more than 10 percent.

NOTE — A sample component may breakthrough from the back end of tube if excessive flow rates are used. The sample is to be discarded, if the breakthrough is observed more than 10 percent.

5.4.6 Sampling Period

The sorbent tubes are exposed in field for previously determined period (generally between 1 - 4 h or so). Before and after sampling the samples are stored and transported to field/laboratory in sealed containers.

NOTE — Exposure period may be shortened for highly polluted area that is near gasoline dispensing station, garage, refinery or other direct emission source.

5.4.7 Blank and Sampled Tube Storage

Seal clean, blank sorbent tubes and sampled tubes using inert, fittings and PTFE ferrules. Wrap capped tubes individually in uncoated aluminium foil. Use clean, sealable metal cans containing a small packet of activated charcoal or activated charcoal/silica gel for storage and transportation of multiple tubes. Store the multi-tube storage container in a clean environment at 4 ± 1°C.

5.5 Procedure

5.5.1 Calibration

A standard solution of the compounds of interest in the elution solvent is prepared gravimetrically, using a micro syringe, by adding pure compounds or pre-weighted blends to flasks partially filled with the elution solvent (CS₂). Prepare Benzene standard solution and a blank 0.043 5 µg/ml, 0.087 µg/µl, 0.174 µg/µl, 0.261 µg/µl and 0.348 µg/µl.

1 µl each of standard solution was injected into the sorption/sample tube, which is desorbed thermally, and analyte is transferred to capillary GC directly. Plot the curve between the concentration and response (peak area).

Multi-point external calibration is used on ATD-GC taking 5 levels of BTX standard solution using CS₂ as a diluting solvent or introduction of a fixed volume gas phase standard (optional).

Typical chromatogram for benzene, toluene and xylene is given in Fig. 8 and typical calibration graphs for benzene, toluene and xylene is given in Fig. 9.

5.5.2 Analytical Procedure

Remove the sorbent and extract the trapped chemical from sample tubes using heat. Samples collected through this technique (sorbent tubes) may be desorbed by Automated Thermal Desorber generally by 2-stage desorption technique on ATD-GC. The desorbed samples are transferred to gas chromatograph (GC) directly and analysed using capillary column and flame ionization detector (FID). No solvent is required in this process.

a) Desorption of the sorbent tube onto a focusing trap — Place the sealed tubes on the thermal desorber (Automated system or equivalent). Heat the interface between the thermal desorber and the GC uniformly.

Desorption of the sorbent tube (typically 200-300°C for 5-15 min with a carrier gas flow of 30-100 ml/min and refocusing of the target analytes on a focusing trap held at near-ambient or sub-ambient temperatures.

Reverse the flow direction of N₂ or He gas, set the flow rate to at least 30 ml/min, and heat the tube to 325°C (in this case) to achieve a transfer of BTX onto a focusing tube at a temperature of 27°C or so.

NOTE — Analytes should be desorbed from the tube in backflush mode, that is, with the gas flow in the reverse direction to that of the air flow during sampling.

b) Rapid desorption of the focusing trap — Rapid desorption of the focusing trap (typically 40°C/s to a top temperature of 250-350°C, with a hold time of 10-15 min at the top temperature and an inert/carrier gas flow of 30-100 ml/min) take place to transfer the analytes into the analytical column.

Analytes are transferred to the column in the form of narrow band of vapor. Desorption in the focusing
trap initiates the analytes to run through GC column. Different thermal desorbers may have different arrangements for automation. Alternatively, use equivalent manual desorption.

NOTE — Components should normally be desorbed from the focusing trap in backflush mode, that is, with the gas flow through the cold trap in the reverse direction to that used during analyte focusing.

c) Sample splitting — If the sample loading is high, it is usually possible to eliminate sufficient water to prevent analytical interference by using sample splitting.

Sample may be split either: (a) between the focusing trap and the capillary column (single splitting) during trap (secondary) desorption, or (b) between both the

8A Typical View

8B Overlay View on ATD-GC-FID System

**FIG. 8 CHROMATOGRAMS OF BENZENE, TOLUENE AND XYLENE**

(Using Column PE5, 25 m x 0.20 mm, d, = 0.33 μm 5 percent phenyl 95 percent dimethyl polysiloxane, at concentration of analytes, 0.0174 μg/μl)
tube and the focusing trap during primary (tube) desorption, that is, double splitting. It may, in fact, be necessary to split the sample in some cases to prevent overloading the analytical column or detector due to excess water accumulation or during the analysis of high concentration/large volume air samples.

NOTE — Sample splitting is one of the key approaches to water management. Moisture management by sample splitting is applicable to relatively high concentrations (10 ppb) or large volume air samples. The mass of water retained by the sorbent tube during sample collection may be sufficiently reduced by the split alone to eliminate the need for further water management steps.
d) Trap desorption and GC/MS analysis — After each tube is desorbed, rapidly heat the focusing trap and apply pure Nitrogen or helium carrier gas. Sample splitting is necessary to accommodate the capillary column. Analytes are transferred to the column in a narrow band of vapor.

The GC run is initiated based on a time delay after the start of thermal desorption. The analytical cycle and ATD and GC conditions are described as follows:

1) ATD Conditions

   i) Purge time: 1 min (After leak test air is purged to reduce analyte oxidation).

   ii) Tube oven temperature: 300°C, Desorb time is 12 min.

   iii) Cold trap low temperature: –30°C.

   iv) Heat rate of cold trap: 40°C/s up to 225°C for 20 min.

   v) Heated valve temperature: 6 Port rotary valve: 200°C.

   vi) Transfer line temperature: 225°C.

   vii) Inlet and Outlet split: 50 and 20 ml/min before and after cold trap respectively.

   (These vary depending on nature and probable concentration of analyte in the sample).

2) GC Conditions

   i) The ambient laboratory temperature should be between 10°C and 35°C with a relative humidity 20 percent to 75 percent with no condensation. The GC-ATD will operate safely between 15°C and 32°C.

   ii) Capillary Column, coating: 5 percent phenyl 95 percent dimethyl polysiloxane, Length x ID : 25 m x 0.20 mm, \( d_p = 0.33 \mu m \).

   iii) Detector: Flame ionization detector (FID) at 260°C.

   iv) Air and H₂ Gas: 400 ml/min and 40 ml/min (10 : 1).

   v) Carrier Gas: Nitrogen.


   vii) Injector: Off.

   viii) Oven initial temperature: 50°C hold for 2 min. Ramp 1 – 8.0°C/min to 140°C hold for 3 min. Ramp 2 – 10.0°C/min to 250°C hold for 3 min.

   ix) Run Time : 30.25 min.

   x) Benzene RT 4.57 min, Search window : 1.00 s, 3.00 percent.

   xi) Toluene RT 6.04 min, Search window : 1.00 s, 3.00 percent.

   xii) Xylene RT 8.00 min, Search window : 1.00 s, 3.00 percent.
NOTE — Temperature programming and retention time (RT) of analyte may vary column to column to get appropriate resolution of analyte peaks. Injection volume and split may also vary according to nature and probable concentration of analyte present in the extract.

e) Conditioning of sorbent tubes reuse — All volatiles should be stripped from the sorbent tubes during the thermal desorption process leaving them clean and ready for reuse. The tubes should be resealed to ensure they are kept clean and ready for immediate reuse.

5.6 Calculation

Amount of analyte compound found on tube can be converted into mg/m³ by using the formula:

\[
\text{Volume of air, in m}^3 \text{ (sucked through the adsorption tube)} = \frac{s \times t}{10^4}
\]

where

\( s = \) sampling rate, in ml/min; and

\( t = \) sampling time, in min.

Concentration, in \( \mu g/m³ \) (at ambient condition) = \( \frac{C_2}{V_3} \)

where

\( C_2 = \) amount of analyte compound found on sample tube in \( \mu g \); and

\( V_3 = \) volume of air sucked through the tube, in m³.

Blank value is to be subtracted from the amount of compound found in the sample.

Concentration, in \( mg/m³ \) at (STP) = \( \frac{C \times 101.3 \times (273 + T)}{273 \times P} \)

where

\( C = \) concentration at ambient condition, in \( mg/m³ \);

\( T = \) temperature of the ambient air, in °C; and

\( P = \) atmospheric pressure, in kPa.

6 METHOD 3 (PASSIVE SAMPLING USING COCONUT SHELL ACTIVATED CHARCOAL PASSIVE DIFFUSION SAMPLER TUBES)

6.1 Principle

Controlled diffusion with an activated charcoal tube is used to enrich the substances targeted for analysis. A diffusion sampling system comprises a sampling layer and a diffusion path in front of this layer. The diffusion path is filled with porous cellulose acetate, to prevent convection currents. The sample is taken by exposing the tube to ambient air (protected from rain). During this exposure time, the analytes stream into the activated charcoal due to the concentration gradient between the air and the desorption layer hand are adsorbed by the charcoal. Once the sample has been collected, the tubes are taken to the laboratory where desorption is done and the substances dissolved in the CS₂ are analyzed using capillary gas chromatography (GC) equipped with flame ionization detector (FID).

6.2 Apparatus

6.2.1 Sampling Device — Passive diffusion sampler or Sorption diffusion tube (Fig. 10) of known dimensions (length, internal diameter etc), or standard make [Orsa-5, Drager, Lubeck, Germany; Radiello diffusive sampler, Fondazione Salvatore Maugeri (FSM), Italy; SKC diffusive sampler series 5, PA, USA or other equivalent make] filled with known amount (generally 400 mg or so but less than 600 mg) of coconut shell activated charcoal (crystalline form, mesh size between 30 and 80 mesh) and of known diffusion constant, uptake rate and desorption efficiency (for benzene toluene and xylene) provided with protecting hood and passive diffuser tube
holder to protect the tube from rain and direct sunlight. Suitable diffusion barrier like acetate cellulose is provided at ends of diffusive sampler tubes. All the supporting parts that is diffusive tube body, tube holder, clip etc should be made of stainless steel or polycarbonate or polyethylene. The glass bottles (see Fig. 10) are used for storing and transporting the sample tubes before and after sampling to/from field and laboratory.

6.2.2 Gas Chromatograph — Any suitable gas chromatograph equipped with flame ionization detector (FID) with fused silica capillary columns having a length of 25 m or more, an internal diameter of 320 μm or below and with a stationary phase film thickness less than 1.5 μm as follows or equivalent may be recommended.

Capillary 624 Column: Coating: cyanopropyl phenyl polysiloxane, Length × ID: 30 m × 0.25 mm, Film thickness \(d_i\): 1.4 μm

Capillary Column: Coating: 5 percent phenyl, 95 percent dimethyl polysiloxane, Length × ID: 25 m × 0.20 mm, Film thickness \(d_i\): 0.33 μm

Wall Coated Column: Coating: Fused Silica PONA CB, Length × ID: 50 m × 0.21 mm, Film thickness \(d_i\): 0.5 μm

Capillary Column: Coating: Fused silica 100 percent dimethyl polysiloxane, Length × ID: 30 m × 0.32 ID, Film thickness \(d_i\): 1.0 μm

6.3 Reagents

6.3.1 Carbon Disulphide (CS₂) — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0005 percent, Benzene 0.0001 percent, H₂O < 0.02 percent.

6.3.2 Benzene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0003 percent, H₂O < 0.02 percent.

6.3.3 Toluene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0003 percent, H₂O < 0.02 percent.

6.3.4 Xylene — Chromatographic grade, Purity > 99.9 percent (GLC), Residue < 0.0003 percent, H₂O < 0.02 percent.

6.3.5 Carrier Gas — Helium or Nitrogen of purity > 99.9 percent, H₂O < 0.02 percent, Residues < 0.0003 percent.

6.4 Sampling

6.4.1 Sampling Location

The sorption diffusion tube with tube hood is placed with the pillar at the height of 1.8-2.1 m at desired location. Site should be free from any obstacle to free flow of the air in the vicinity.

6.4.2 Sampling Rate

The sampling is performed through natural diffusion (sampling rate generally range between 5 and 10 ml/min). The analyte is adsorbed on to activated charcoal.

6.4.3 Sampling Period

The diffusive samplers are exposed in field for previously determined period [generally for a fortnight (15 days) or so].

NOTE — Exposure period may be shortened to a week or few days only for highly polluted area that is near gasoline emissions or dispensing station, garage or so.

6.4.4 Sample Diffuser Tubes Labelling — Sample tubes are labelled with a unique identification number.

6.4.5 Blank and Sampled Tube Storage

Before and after sampling the samples are stored and transported to field/laboratory in sealed glass bottle. Store these tubes in storage container having clean environment maintained at 4 - 5°C.

6.5 Procedure

6.5.1 Calibration

Prepare a mix stock standard solution of 50 μg/μl of benzene, toluene and xylene each gravimetrically, using a micro syringe in the elution solvent that is CS₂. Prepare further diluted solutions of concentration range of 10, 1.0, 0.10 μg/μl with CS₂ from stock standard in a clean vial. Make up to one millilitre solution. Introduce immediately 1 ml standard solution into the injector of GC directly and plot the curve between the concentration and response (peak area). A typical chromatogram of standard mixture is given in Fig. 11.

6.5.2 Analytical Procedure

Samples collected through passive technique (sorbent diffusion tubes) may be desorbed by conventional solvent (generally carbon disulphide). The samples extracted in carbon disulphide are analysed on Capillary GC equipped with flame ionization detector (FID). 1 μl of each standard solution is injected into the column. GC conditions are given as follows:

**GC-FID conditions**

Capillary column: Wall coated fused silica, PONA, \(d_i\): 0.5 μm
FIG. 11 STANDARD CHROMATOGRAM OF BENZENE, TOLUENE AND XYLENE
(Using CP_Silica PONA capillary column 50 m x 0.2 mm ID, film thickness $d_f$ = 0.5 μm)

Length × ID : 50 m × 0.21 mm

Gas flow:
   a) Nitrogen : 30 ml/min (Make up + column),
       (Column flow: 1 ml/min)
   b) Hydrogen : 30 ml/min
   c) Air : 300 ml/min

Temperature programming:
Injection port : 250°C   FID : 300°C
Oven : 60°C - 230°C @ 10°C/min

Typical Injection volume: 2 μl, total run time: 20 min.
- Benzene RT 8.06 min, Search window : 1.00 s,
  3.00 percent
- Toluene RT 9.86 min, Search window : 1.00 s,
  3.00 percent
- Xylene RT 11.78 min, Search window : 1.00 s,
  3.00 percent

NOTE — Temperature programming and retention time (RT) of analyte may vary column to column to get appropriate resolution of analyte peaks. Injection Volume and split may also vary according to nature and probable concentration of analyte present in the extract.

6.6 Calculation

Calculations are given as follows:

$$C = (M - M_{\text{blank}}) \times K_{\text{ORSA}} \times 1000 / DE \times D \times t$$

where

- $C$ = concentration of the measured compound, in μg/m³;
- $M$ = determined mass of the measured compound, in ng;
- $M_{\text{blank}}$ = weight of analyte organic vapour on blank tube, in ng;
- $K_{\text{ORSA}}$ = equipment constant of the diffusive sampler (that is 0.8 cm⁻¹ for Drager’s ORSA 5 diffusive sampler);
- $1000$ = conversion factor to get μg/m³ from, in mg/m³;
- $DE$ = desorption efficiency (that is 0.98 for Drager’s ORSA 5 diffusive sampler);
- $D$ = diffusion coefficient in cm²/s at 25°C and 1013 kPa (Benzene 0.0859 cm²/s, Toluene 0.0764 cm²/s, Xylene 0.0727 cm²/s for Drager’s ORSA 5 diffusive sampler); and
- $t$ = sampling duration, in seconds.

Alternatively following formulae may be applied for calculations:

$$C = (M - M_{\text{blank}}) / DE \times U \times t$$

where

- $C$ = concentration of the measured compound, in μg/m³;
- $M$ = determined mass of the measured compound, in ng;
- $M_{\text{blank}}$ = weight of analyte organic vapour on blank tube, in ng;
DE = desorption efficiency (0.98);

\( U = \) uptake rate in l/h at 25°C (benzene 0.387 l/h, toluene 0.343 l/h, xylene); and

\( t = \) sampling duration, in hours.

Concentration (mg/m³) at (STP)

\[
C = \frac{C_1 \times 101.3 (273 + T)}{298 \times P}
\]

where

- \( C_1 = \) concentration at ambient condition, in µg/m³;
- \( T = \) temperature of the ambient air, in kelvin; and
- \( P = \) atmospheric pressure, in kPa.

7 CONVERSION OF CONCENTRATION IN PPB

\[ C \text{ [ppb]} = C \text{ [µg/m³]} \times \frac{24.1}{M} \]

where

- 24.1 = molar volume at 20°C in litres; and
- \( M = \) molar mass.

8 INTERFERENCES AND LIMITATIONS

8.1 Interference from Sorbent Artifact and Minimizing Artifact Interference

Stringent tube conditioning and careful tube capping and storage procedures are essential for minimizing artifacts. System and sorbent tube conditioning must be carried out using more stringent conditions of temperature, gas flow and time than those required for sample analysis.

NOTE — A reasonable objective is to reduce artifacts to 10 percent or less of individual analyte masses retained during sampling.

8.2 Artifacts from Long-Term Storage of Blank Tubes

Literature reports of the levels of artifacts on (a) Carbotrap/pack™ C, Carbotrap/pack™ B; and Carbosieve™ SIII multi-bed tubes; and (b) Tenax® GR tubes by workers when sealed the tubes using metal Swagelok®-type caps and PTFE ferrules with multi-tube, glass storage jars are reported to be between 0.01 ng after 1-2 months and 0.1 ng after six months respectively. Artifact levels reported for other porous polymers are higher, for example, 5 ng for Chromosorb 106 after one week.

Some varieties of charcoal contain metals which will catalyze the degradation of some organic analytes during thermal desorption at elevated temperatures thus producing artifacts and resulting in low analyte recoveries.

8.3 Artifacts Generated During Sampling and Sample Storage

8.3.1 Active Sampling

Benzaldehyde, phenol and acetophenone artifacts are reported to be formed via oxidation of the polymer Tenax when sampling high concentration (100 - 500 ppb) ozone atmospheres.

Tenax should thus be used with an ozone scrubber when sampling low levels (< 10 ppb) of these analytes in areas with appreciable ozone concentrations.

Carbotrap pack type sorbents have not been reported to produce this level of artifact formation. Once retained on a sorbent tube, chemically stable VOCs, loaded in laboratory conditions, have been shown to give good recoveries, even under high ozone concentrations for storage of a year or more.

8.3.2 Passive Sampling

The uptake rate of diffusive samplers is not significantly affected by air movement, provided the air velocity exceeds a threshold value which depends on design. Generally, air velocities greater than 0.1 m/s¹ and below 10 m/s¹ are sufficient for the passive sampling.

8.3.3 Temperature correction for sampled air volume is to be made, if sampling is performed below 20°C or above 30°C.

9 DETECTION LIMITS AND MAXIMUM QUANTIFIABLE CONCENTRATIONS OF AIR POLLUTANTS

The method of detection limit is defined for each system by making seven replicate measurements of a concentration of the compound of interest near the expected detection limit (within a factor of five), computing the standard deviation for the seven replicate concentrations, and multiplying this value by 3.5 (the Student's \( t \) value for 99 percent confidence for seven values).

Detection limits for atmospheric monitoring vary depending on several key factors. They are:

a) Sample storage condition,

b) Injection volume,

c) Minimum artifact levels,

d) GC detector selection, and

e) Volume of air sampled. The volume of air sampled is in turn dependent upon a series of variables including SSVs, pump flow rate limitations and time-weighted-average monitoring time constraints.

Generally detection limits range about sub-ppb for BTX in one litre air samples using the GC-FID. Detection limits are greatly dependent upon the proper management of water for GC capillary analysis of volatile organics in air using sorbent technology.
9.1 Safe Sampling Volume (SSV)
Usually calculated by halving the retention volume (indirect method) or taking two-thirds of the breakthrough volume (direct method), although these two approaches do not necessarily give identical results. The latter definition is generally used.

9.2 Breakthrough Volume (BV)
The volume sampled when the amount of analyte collected in a backup sorbent tube reaches a certain percentage (typically 5 percent) of the total amount collected by both sorbent tubes.

10 QUALITY ASSURANCE (VALIDATING THE SAMPLE COLLECTION PROCEDURE)

10.1 Blanks
Artifact levels on laboratory and field blanks should be at the low or sub-nanogram level for carbonaceous sorbents and Tenax® and at the double digit ng level for Porapaks®, Chromosorb®. If artifact levels are considerably above this, careful attention must be paid to the tube conditioning and storage procedures.

10.2 Performance Criteria for the Monitoring Pump
Records of the pump flow rate delivered against the pump flow rate or pressure selected on a pump should be reviewed at least once per three months. If the performance of any pump has been found to have changed significantly over that time, for example if completely different pump settings are required to deliver the saline pump flow rate, the pump should be serviced by the manufacturer or their approved agent.

10.3 Performance Criteria for the Solid Adsorbent Sampling of Ambient Air
There are four performance criteria, which must be met for a system. These criteria are:

- a) A method detection limit of 0.5 ppb,
- b) Duplicate (analytical) precision within 20 percent on synthetic samples of a given target analyte or vapor in a typical vapor mix in humidified zero air,
- c) Agreement within 25 percent for distributed volume pairs of tubes taken in each sampling set, and
- d) Audit accuracy within 30 percent for concentrations normally expected in contaminated ambient air (0.5 to 25 ppb).

10.4 Calibration of Response
The multi-level calibration procedures and calibration frequencies should be followed for this. It is also advisable to analyze a single level calibrant (i.e. tubes loaded with analyte masses in the mid-range of those expected to be collected during sampling) approximately every tenth sample during an analytical sequence, as a check on system performance.

10.5 Analytical Precision of Duplicate Pairs
The measure of analytical precision used for this method is the absolute value of the relative difference between two identical samples (same flow rate over the same time period from with a common inlet to the sample volume). The analytical precision is expressed as a percentage as follows:

\[
\text{Analytical precision} = \left[ \frac{(X_1 - X_2)}{X} \right] \times 100
\]

where

- \(X_1\) = a measurement value taken from one of the two tubes used in sampling,
- \(X_2\) = a measurement value taken from the second of two tubes used in sampling, and
- \(X\) = average of \(X_1\) and \(X_2\).

The analytical precision is a measure of the precision achievable for the entire sampling and analysis procedure including the sampling and thermal desorption process mentioned above and the analytical procedure.

10.6 Accuracy
A measure of accuracy is the degree of agreement with audit standards. Audit accuracy is defined as the relative difference between the measurement result and the nominal concentration of the compound:

\[
\text{Audit accuracy, percent} = \frac{(\text{Spiked value} - \text{Observed value})}{\text{Spiked value}} \times 100
\]
ANNEX A
(Foreword)

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This Indian Standard has been developed from Doc: No. CHD 32 (1336).

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