

# Evaluation of Tenax TA for the determination of chlorobenzene and chloronitrobenzenes in air using capillary gas chromatography and thermal desorption

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## Abstract

A method for the determination of chloronitrobenzenes (CNBs) and chlorobenzene (CB) in air was developed. CNBs and CB were sampled on Tenax TA sampling tubes, thermally desorbed and analysed by capillary gas chromatography (cGC) with flame ionization detection. The sampling efficiency was found to be independent of flow-rate (25 and 50 ml/min) and relative humidity (30–90%) generated in the laboratory. Recovery studies on Tenax TA (20–35, 35–60 and 60–80 mesh) in the concentration range 0.041–8.212 mg/ml were conducted using cGC coupled with an automatic thermal desorption system (ATD-50). Nearly quantitative recoveries were obtained at all levels of concentrations. Further, it was observed that flow-rates and relative humidity had no effect on the adsorption and thermal desorption efficiency of CNBs and CB when present as a mixture in workplace air. No loss of sample was observed up to 30 days of storage at room temperature. With this method, a time-weighted average concentration of less than 0.04 mg/m<sup>3</sup> could be determined in an 8-h sampling period with a precision of 1.4–12% for the whole method.

## 1. Introduction

Chloronitrobenzenes (CNBs, *o*-, *m*- and *p*-) and chlorobenzene (CB) are used extensively for manufacturing dyes, pesticides, agrochemicals, pharmaceuticals and rubber chemicals. It has been known for a long time that CNBs and CB are irritants to the eyes and mucous membranes in the respiratory tract and have acute and chronic effects. The threshold limit values (TLVs) for *o*-, *m*- and *p*-CNB and CB are 1, 1, 3

and 350 mg/m<sup>3</sup>, respectively, for an 8-h exposure [1].

For the last two decades, preconcentration of toxic organic vapours used in most NIOSH procedures [2] utilizes charcoal or silica gel as the adsorbent followed by solvent desorption [3] and gas chromatographic (GC) analysis [4–6]. However, charcoal and silica gel have certain disadvantages. Air humidity may reduce their sorption efficiency and the attendant contaminants may displace the target substances on the sorbent.

Another approach uses a porous polymer for

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contaminant adsorption and thermal desorption into a gas chromatograph [7], which offers better sensitivity with respect to solvent extraction as the whole sample is injected [8]. Porous polymer sorbents (e.g., Tenax, Chromosorbs, Polysorbs, Porapak and Amberlite XAD series) are relatively inert and hydrophobic and normally have large surface areas. Porous polymers are most successfully used to trap toxic agents of high molecular mass and low- or non-volatile substances [8–11]. Among different sorbents, Tenax GC/TA, a polymer of 2,6-diphenyl-*p*-phenylene oxide, has been used extensively in air pollution monitoring applications because of its excellent properties of trapping the pollutants and high thermal (350–400°C) stability, which in turn facilitates thermal desorption [12,13]. The suitability of Tenax and its ability to concentrate diverse organic compounds with a wide range of boiling temperatures have made it indispensable in the analysis of the complex composition of toxic agents in GC and thermal desorption techniques [8,14].

The determination of chlorobenzene in air using a solid sorption technique and subsequent gas chromatographic analysis has been described [8,15,16]. However, to our knowledge, no method for monitoring chloronitrobenzenes in the presence of chlorobenzene has been described. The serious effects of CNBs and CB on human health necessitate the development of a reliable method of monitoring the workplace environment. This study was aimed at determining traces of these pollutants in humid workplace air where the temperature varied from 20 to 40°C, in a chemical plant involving the process of nitration of CB to CNB. The process chemicals were adsorbed on Tenax TA solid sorbent and analysed using GC coupled with an automatic thermal desorption system.

## Experimental

### 2.1. Chemicals

Chlorobenzene and *o*-, *m*- and *p*-chloronitrobenzenes were of analytical-reagent grade

from Fluka (Buchs, Switzerland). Methanol used as a solvent was of analytical-reagent grade from S.D. Fine Chemicals (Bombay, India).

### 2.2. Apparatus

For the analysis of standard test atmosphere samples, spiked samples and samples of workplace air, a Perkin-Elmer (Beaconsfield, UK) Model 8500 gas chromatograph with a flame ionization detector and coupled with an ATD-50 automatic thermal desorption system was used. The chromatograms obtained were recorded on a Perkin-Elmer GP 100 printer-plotter and the areas under the curve were evaluated with the help of a built-in data processor.

Sampling rates were maintained by a water-siphoning system for field samples, the details of which were described in a previous paper [8]. Portable sampling pumps (SKC, USA) supplied by Spantech Products (UK) were used during the sampling from standard test atmospheres with different relative humidities.

### 2.3. Sample tubes

For sampling in the solid sorbent method, stainless-steel sampling tubes (Perkin-Elmer) of 89 mm × 5 mm I.D. with stainless-steel wire gauges on both ends to hold the adsorbent were used. Metal sealing caps were used during the storage of samples. Outer and inner analytical end-caps were used during the thermal desorption and the analysis of samples.

### 2.4. Columns

A DB 225 fused-silica wide-bore capillary column of 15 m × 0.53 mm I.D. (J and W Scientific, Folsom, CA, USA) with a film thickness of 1 μm was used for the analysis of samples obtained under various experimental conditions.

Retention volumes and safe sampling volumes were determined on laboratory-packed glass columns (1 m × 2 mm I.D.) of Tenax TA of 60–80, 35–60 and 20–35 mesh (180–250, 250–500 and 500–850 μm).

## 2.5. Solid adsorbents

Tenax TA of three different mesh sizes was obtained from Ohio Valley Speciality Chemicals (Marietta, OH, USA) and was used throughout the experiments.

## 2.6. Retention volume

The retention volumes and safe sampling volumes of CNBs and CB on Tenax TA porous polymer of three different mesh sizes were evaluated for concentrating all the compounds in the course of sampling without breakthrough.

Three glass chromatographic columns (1 m × 2 mm I.D.) were filled with a known mass of Tenax TA of 20–35, 35–60 and 60–80 mesh. Each column was conditioned at 25°C for 45 min. Thereafter, the temperature was increased by 2°C/min to the final temperature of 300°C, which was maintained for 24 h with a nitrogen flow-rate of 20 ml/min. After conditioning of the columns, standard solutions of *o*-, *m*- and *p*-CNB and CB were injected separately at different column temperatures with a nitrogen flow-rate of 20 ml/min. The retention volumes were recorded along with the absolute column temperatures.

## 2.7. Preparations of standard solutions

Stock standard solutions were prepared by

dissolving 1.030, 1.024, 1.027 and 1.026 g of *o*-, *m*- and *p*-CNB and CB, respectively, in 100 ml of methanol. Working standard solutions in the concentration range of interest (0.041–8.212 mg/ml) were prepared by serial dilution of the stock standard solutions with methanol in a 25-ml volumetric flask. To these working standard solutions, 2 ml of a standard solution of aniline (20.37 mg/ml) was added as an internal standard.

## 2.8. Calibration of method by GC

A 0.2- $\mu$ l volume of each standard solution was injected into the GC column (DB 225). The initial temperature of the column was 40°C, held for 3 min, then the temperature was increased at 30°C/min to 115°C, which was maintained for 8 min. The temperatures of the injector and the detector were maintained at 280 and 300°C, respectively. The flow-rate of nitrogen through the column was 11 ml/min.

## 2.9. Generation of test atmosphere

Chloronitrobenzenes and chlorobenzene in air were generated by the dynamic generation equipment shown in Fig. 1. A tube containing each analyte was inserted into a 200-ml glass vessel, placed in a water-bath. A stream of the analyte of interest was generated by blowing dried air into the vessel. The outlet was con-

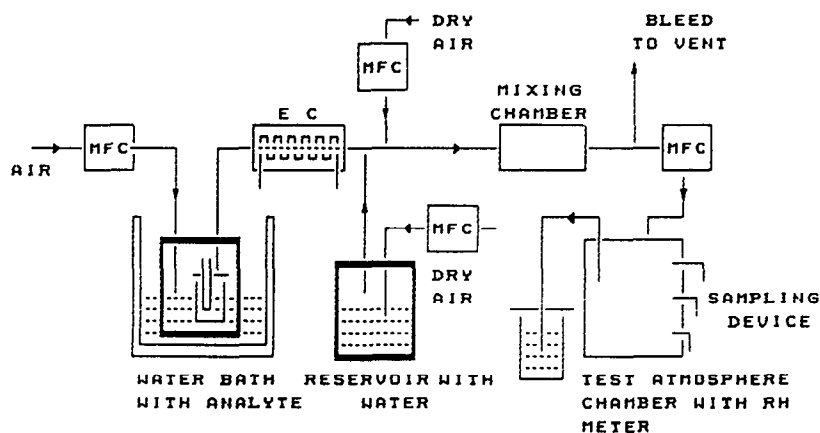


Fig. 1. Scheme of the dynamic dilution system. MFC = Electronic mass flow controller; EC = evaporation chamber.

nected to a mixing chamber where the analyte atmosphere was further diluted with air using a mass flow controller (Porter Instrument Co., USA). Further dilution with a controlled relative humidity (RH) were made by using another mass flow controller in the system. The air stream, after appropriate dilution and thorough mixing, was fed into a glass exposure chamber of 5 l capacity with an air-tight removable lid with the help of the mass flow controller. The flow-rate was kept at 40–60 ml/min.

Samples were drawn at each concentration of CNBs and CB through the two sampling tubes of Tenax TA. The sampling flow-rates were maintained at 25 and 50 ml/min with the help of portable sampling pumps for different periods of time. During the sampling the RH was maintained at 30, 60 and 90%, which were achieved by mixing dry air with air bubbled through deionized water. The exposure chamber was provided with two outlets for sampling and a third outlet was left in water to maintain atmospheric pressure in the exposure chamber during sampling.

The concentration in the exposure chamber was monitored at 30-min intervals using the Tenax sampling tubes with GC determination for control purpose. Different concentrations were obtained by varying the temperature in the water-bath between 50 and 100°C and by changing the flow-rate of the air blowing over the vessel and in the mixing chamber. Relative humidities between 30 and 90% could be generated.

#### 2.10. Thermal desorption recovery

To determine the thermal desorption recovery of Tenax TA of three different mesh sizes in the concentration range of interest, a set of 35 sampling tubes were filled with 200 mg of Tenax TA of a chosen particle size. All the tubes were conditioned in a specially made tube conditioner (SkyLab, India) at 300°C for 24 h with a nitrogen flow-rate of 20 ml/min and were sealed before use with storage caps supplied by Perkin-Elmer. Each conditioned sample tube was fitted into the GC injection port maintained at 280°C with a nitrogen flow-rate of 15 ml/min and a set of five

tubes were spiked with 0.2  $\mu$ l of each standard solution at room temperature. The spiked tube was disconnected after 2 min and exposed to different RH for 4–8 h in order to check the potential losses during field sampling.

These tubes were then immediately desorbed under the following optimum desorption conditions: desorption temperature, 250°C; desorption time, 10 min; transfer line temperature, 110°C; cold trap low temperature, -30°C; cold trap high temperature, 300°C; cold trap adsorbent, Tenax TA (60–80 mesh). After desorption, the cold trap was heated spontaneously and desorbed material was fed to the GC capillary column for analysis. The analysis were performed using the optimized GC conditions used for the calibration of the method.

#### 2.11. Storage of samples

Storage tests were performed by loading a set of five tubes with standard solutions as mentioned above. The tubes were sealed with storage caps and stored at room temperature (25–27°C) for 5, 15 and 30 days. Each set of tubes was analysed after a specified time using the optimized conditions of thermal desorption and GC.

#### 2.12. Workplace environment atmosphere

Air samples were collected in a chemical plant manufacturing CNBs using CB as raw material. To achieve more or less the same pressure drop and constant flow-rates during sampling, each tube was filled with an equal amount (200 mg) of Tenax TA of 20–35, 35–60 and 60–80 mesh. Before starting the field sampling, all sampling tubes were preconditioned for 12 h at 300°C with a nitrogen flow-rate of 20 ml/min. After conditioning, the tubes were sealed immediately with storage caps to avoid cross-contamination due to the diffusion process. The RH and temperature of the workplace during the period of investigation were found to vary in the ranges 50–90% and 20–40°C, respectively.

Sample volumes in the range 4–20 l with flow-rates of 25 and 50 ml/min were collected. Samples were also collected for short periods with

higher flow-rates using portable pumps, to establish the suitability of Tenax TA for short-term monitoring, often required prior to shut-down jobs in a plant.

The tubes were fitted with analytical end-caps and placed on the turntable of thermal desorption system for analysis. The tubes were thermally desorbed and analysed using the optimized conditions of thermal desorption and GC.

### 3. Results and discussion

#### 3.1. Standards

The identities of the CNBs and CB were confirmed by GC-MS. The purity was also checked using GC with flame ionization detection and found to be more than 99%.

#### 3.2. Retention volumes

The retention volumes of each analyte at different temperatures were recorded. The logarithm of the specific retention volume was plotted against the reciprocal of the absolute column temperature, which gave a linear relationship. The retention volumes at 25 and 30°C were obtained by extrapolation (Table 1). Half of the retention volume was considered as a safe sampling volume for sampling purposes in the field. It was observed that with this retention volume, the determination of CNBs and CB is feasible without any breakthrough in 10–20-l air samples.

Table 1  
Adsorptive properties of Tenax TA of different mesh sizes

Compound	Retention volume (l/g) <sup>a</sup>		
	60–80 mesh	35–60 mesh	20–35 mesh
CB	281 (184)	280 (181)	232 (153)
<i>m</i> -CNB	18 836 (11 290)	16 463 (9767)	6994 (4363)
<i>p</i> -CNB	25 119 (14 944)	24 126 (14 150)	10 455 (6445)
<i>o</i> -CNB	25 699 (15 313)	21 739 (12 841)	8142 (5083)

<sup>a</sup> Values at 25°C; values in parentheses are retention volumes at 30°C.

#### 2.3. Gas chromatography and quantitative analysis

The calibration graph for each analyte was obtained by plotting the average peak area ratio against the amount injected. A typical chromatogram of a mixture is shown in Fig. 2. A linear graph passing through the origin was obtained in the investigated concentration range of 0.04–8.212 mg/ml for each analyte. The pooled ac-

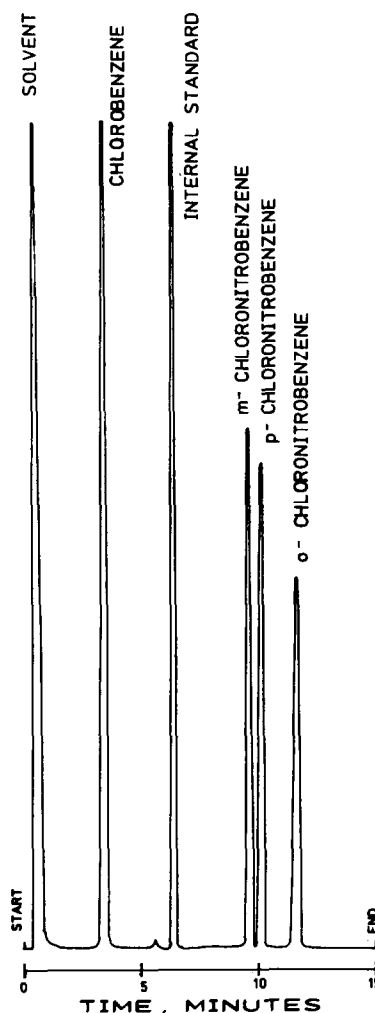


Fig. 2. Chromatogram of calibration mixture containing 4.1 mg/ml of CB and *m*-, *p*- and *o*-CNB. Injection volume, 0.2  $\mu$ l; column (polar), DB 225 (15 M  $\times$  0.53 mm I.D.) with film thickness 1  $\mu$ m; oven programme, 45°C for 5 min, increased at 30°C/min to 115°C, held for 8 min. Aniline was added as an internal standard.

curacy of the method is better than 99.95%. The regression coefficients for CB and *o*-, *m*- and *p*-CNB are 0.9999, 0.9305, 0.9999 and 0.9999 respectively.

For establishing the suitability of the column with respect to the identification and determination of analytes, a comparison between the results obtained using non-polar (DB 1701 and DB 210) and polar (DB 225) columns was made. It was found that a fused-silica capillary column (DB 225) gave a better performance than the others under identical conditions of length, internal diameter and film thickness. When the DB 225 column was used, no signs of adsorption or decomposition were observed. The overall analytical precision of the method was found to be in the range 0.30–7%.

### 3.4. Sampling efficiency

The sampling efficiency for different concentrations of *o*-, *m*- and *p*-CNB and CB was determined by analysing sampling tubes exposed to known concentrations of test atmosphere. The results are summarized in Table 2. Neither the concentration nor the RH seems to affect the sampling efficiency seriously for sampling rates

of 25 and 50 ml/min. Three parallel samplings with flow-rates of 25 and 50 ml/min were performed at 30, 60 and 90% RH. The overall sampling efficiency at different flow-rates and RH was found to be more than 99%, showing complete adsorption and thermal desorption of CNBs and CB from Tenax TA.

### 3.5. Thermal desorption recovery

The recovery of *o*-, *m*- and *p*-CNB and CB on solid sorbent tubes packed with Tenax TA of different mesh sizes was investigated at various concentrations. The tubes were spiked with different concentrations of standard solutions. After the spiking, each tube was connected to the pump and exposed to different RH for 4–8 h in order to check for potential losses during the field sampling. As the recoveries of the analytes were found to be independent of the mesh size of Tenax TA, only the data for 35–60-mesh Tenax TA are presented in Table 3.

The results in Table 3 indicate nearly quantitative recoveries (>95%) of all the analytes at all levels of spiking within relative standard deviations of 1–10% for the whole method for a sampling volume of 10 l. A typical chromato-

Table 2  
Sampling efficiency of Tenax TA sorbent for CNBs and CB at different concentrations and relative humidity in air

Compound	Concentration in air (mg/m <sup>3</sup> )	Relative humidity (%)	Sampling volume (l)	Sampling efficiency (%)	No. of determinations	Precision (R.S.D., %)
CB	4.75	30	8	105	5	2.80
	9.35	90	7.5	103	5	2.69
	13.50	60	8	108	6	2.64
<i>m</i> -CNB	0.56	30	10	105	5	7.15
	2.15	90	9	103	5	6.05
	3.24	60	8	99	4	5.51
<i>p</i> -CNB	1.0	30	8	103	5	6.87
	2.15	90	9	99	5	3.10
	6.73	60	12	102	4	2.50
<i>o</i> -CNB	0.45	30	8	102	5	7.38
	1.85	90	10	103	5	5.78
	3.57	60	8	101	5	4.67

The sampling rate was 25 and 50 ml/min.

Table 3  
Thermal desorption recovery on Tenax TA (35–60 mesh) at 250°C

Standard No.	Recovery ( $\mu\text{g}$ ) <sup>a</sup>			
	CB	<i>m</i> -CNB	<i>p</i> -CNB	<i>o</i> -CNB
1	0.049 (121) [6.03]	0.045 (110) [7.53]	0.042 (102) [10.28]	0.046 (111) [7.03]
2	0.088 (108) [7.27]	0.085 (104) [11.8]	0.076 (94) [12.17]	0.093 (114) [10.97]
3	0.174 (106) [2.78]	0.166 (101) [6.105]	0.162 (99) [5.378]	0.173 (105) [5.80]
4	0.428 (104) [2.47]	0.428 (104) [5.466]	0.415 (101) [3.501]	0.431 (105) [4.587]
5	0.852 (104) [2.388]	0.795 (97) [3.566]	0.786 (96) [3.459]	0.820 (99) [2.438]
6	1.322 (107) [2.44]	1.265 (103) [1.196]	1.256 (102) [1.824]	1.268 (102) [1.445]
7	1.720 (105) [1.695]	1.674 (102) [1.638]	1.647 (100) [1.322]	1.669 (101) [1.479]

<sup>a</sup> Each value is the average of five independent measurements with the percentage recoveries in parentheses and the R.S.D. (%) in square brackets.

gram of thermal desorption is shown in Fig. 3. It was observed that the adsorption of substances from the calibration mixtures and subsequent thermal desorption were complete under the conditions specified. Further, it was also found that RH and flow-rate had no effect on the adsorption and thermal desorption using Tenax TA.

### 3.6. Storage

The stability of *o*-, *m*- and *p*-CNB and CB on Tenax TA was studied. A set of five tubes at three concentrations were immediately analysed while another set of tubes were kept at room temperature (25–27°C) for 5, 15 and 30 days until analysis. The recoveries during storage were not influenced by the RH in the sampled air. At lower concentration, the recovery of CB was found to be 141%, which is relatively high compared with the results obtained at higher concentrations. This is attributed to the blank values contributed by Tenax TA arising from

storage. A typical chromatogram depicting the higher background levels is shown in Fig. 4. The results presented in Table 4 indicate nearly quantitative recoveries of 96–115, 97–117 and 100–119% for 5, 15 and 30 days, respectively. Almost no change in recovery was observed for samples stored in a refrigerator (5°C) for 8 weeks.

### 3.7. Workplace air sample analyses

Air samples taken by Tenax TA adsorption tubes were analysed using the standard conditions for GC and thermal desorption mentioned previously. The results obtained at each location for three individual samples are in good agreement with each other. A typical representative example of air sample analyses obtained by using 35–60-mesh Tenax TA is presented in Table 5. The concentrations of CNBs and CB were found to be well within the threshold limit values prescribed for the individual compounds. It should also be noted that no breakthrough was

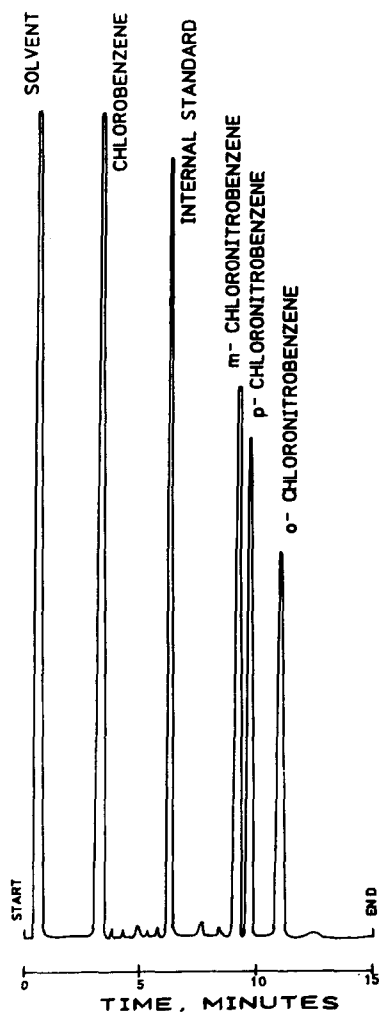


Fig. 3. Chromatogram of thermal desorption recovery of CB and *m*-, *p*- and *o*-CNB on 35–60-mesh size Tenax TA. Thermal desorption conditions: desorption temperature, 250°C; desorption time, 10 min; transfer line temperature, 110°C; cold trap low temperature, -30°C; cold trap high temperature, 300°C; cold trap adsorbent, Tenax TA (60–80 mesh); injection volume, 0.2  $\mu$ l.

observed under the different prevailing conditions existing in the workplace air during sampling.

### 3.8. Hygienic effect

The hygienic effect [5] of the contaminants in an air sample is defined as the sum of the ratios

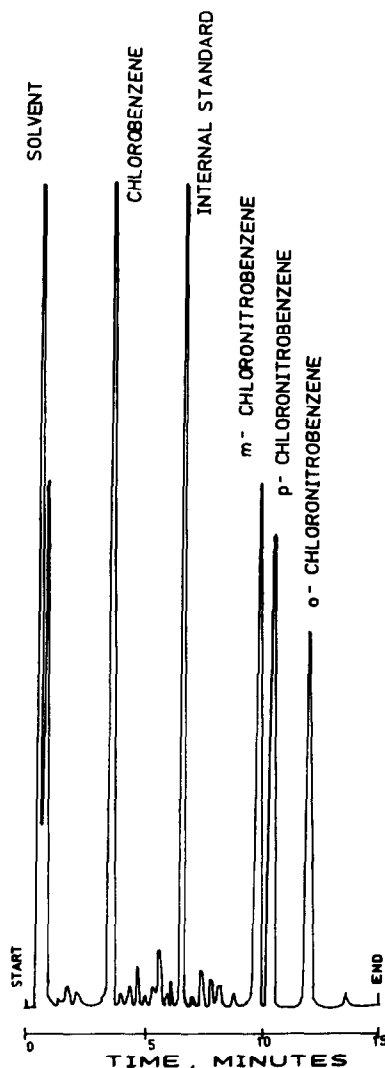


Fig. 4. Chromatogram of thermal desorption recovery after storage for 30 days. Thermal desorption conditions as in Fig. 3.

between the concentrations and threshold limit values (TLV) of each component. This is valid assuming that each component has a similar effect on man. When the sum of the ratios exceeds unity, the TLV of the mixture is exceeded. The hygienic effect (Table 5) was found to be less than unity for the compounds studied in the workplace air, confirming that the analyte concentrations are well below the prescribed limits.



Table 4  
Recovery and precision for sample storage for 5, 15 and 30 days at room temperature (25–27°C)

Compound	Amount added ( $\mu\text{g}$ )	5 days		15 days		30 days	
		Found ( $\mu\text{g}$ ) <sup>a</sup>	R.S.D. (%)	Found ( $\mu\text{g}$ ) <sup>a</sup>	R.S.D. (%)	Found ( $\mu\text{g}$ ) <sup>a</sup>	R.S.D. (%)
CB	0.082	0.098 (120)	11.7	0.099 (121)	4.46	0.116 (141)	4.1
	0.410	0.440 (107)	2.7	0.480 (117)	5.61	0.489 (119)	8.46
	1.231	1.332 (108)	0.7	1.383 (112)	0.41	1.382 (112)	1.11
<i>m</i> -CNB	0.082	0.080 (98)	4.1	0.090 (110)	8.24	0.093 (113)	1.33
	0.409	0.406 (99)	5.3	0.410 (100)	2.31	0.427 (104)	1.99
	1.228	1.210 (99)	1.7	1.315 (107)	1.11	1.270 (103)	0.23
<i>p</i> -CNB	0.082	0.079 (96)	6.2	0.082 (100)	7.65	0.085 (104)	0.2
	0.411	0.400 (97)	6.9	0.397 (97)	3.22	0.411 (100)	1.46
	1.233	1.199 (97)	2.4	1.300 (105)	1.18	1.247 (101)	0.57
<i>o</i> -CNB	0.082	0.094 (115)	26.4	0.081 (106)	5.26	0.095 (116)	4.27
	0.412	0.434 (105)	7.5	0.407 (99)	3.05	0.435 (106)	0.74
	1.236	1.215 (98)	1.9	1.324 (107)	1.73	1.266 (102)	0.49

<sup>a</sup> Each value is the average of five independent measurements with percentage recoveries in parentheses.

#### 4. Conclusions

The proposed method for monitoring CNBs and CB in air was found to be reliable with a detection limit much below the expected average

concentration found in the workplace environment. The sampling efficiency of Tenax TA is not affected by the RH and temperature prevailing in the air. Further, this method can also be used for monitoring the analytes using high flow-rates as the adsorption and thermal desorption processes were found to be independent of flow-rate. No significant loss of CNBs and CB was observed during storage for up to 1 month on Tenax TA.

For long-term monitoring sampling rates of 25 and 50 ml/min are preferable. This method gave the same results (within experimental errors) for monitoring CNBs and CB in both a standard test atmosphere and a workplace environment. This method could be used routinely for monitoring CNBs and CB in the workplace environment.

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Table 5  
Results of workplace air sample analysis on Tenax TA (35–60 mesh)

Sample No.	Concentration ( $\text{mg}/\text{m}^3$ ) <sup>a</sup>				Hygienic effect <sup>b</sup>
	CB	<i>m</i> -CNB	<i>p</i> -CNB	<i>o</i> -CNB	
1	0.070	0.165	0.178	0.242	0.47
2	0.128	—	0.156	0.265	0.32
3	0.030	0.033	0.564	0.030	0.25
4	0.115	0.062	0.364	0.222	0.40
5	0.264	—	0.958	0.044	0.36
6	0.069	0.028	0.223	0.147	0.25
7	0.174	0.037	0.427	0.269	0.45
8	0.864	0.027	0.562	0.524	0.74
9	0.112	—	0.242	0.424	0.50
10	0.056	0.027	0.348	0.276	0.42

<sup>a</sup> Average of three independent measurements at the same location.

<sup>b</sup> See text for definition.

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