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**PROPOSAL OF METHODS FOR THE
DETERMINATION OF VINYL
CHLORIDE AND BENZENE IN
WORKPLACE ATMOSPHERES**

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PROPOSAL OF METHODS FOR THE DETERMINATION OF VINYL CHLORIDE AND BENZENE IN WORKPLACE ATMOSPHERES

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Abstract - This report gives basic practical information on the determination of (i) vinyl chloride and (ii) benzene in workplace atmospheres. Consideration has been given to sampling by charcoal tubes and by means of a glass syringe. The respective literature of the analytical procedure - the gas chromatographic method - is referred to. Determination by direct reading detector tubes has also been included in this paper.

INTRODUCTION

Many treatises have been published on determining the vinyl chloride and benzene concentration in workplace atmospheres, therefore, this paper shall not give a detailed description of analytical methods. Following an appraisal of already existing methods, the Commission decided to publish this paper to help the analyst in selecting the optimum conditions (especially) for sampling vinyl chloride and benzene at workplaces. The main purpose of this paper is to remind the analyst that a reliable solution of the measuring task is only possible by careful planning of the measuring programme, from sample taking through analysis to final evaluation.

Some information is given on the toxicological effects of vinyl chloride and benzene; the data have been taken from "Patty's Industrial Hygiene and Toxicology" (Ref. 1).

Consideration has been given to sampling of vinyl chloride and benzene by charcoal tubes and in addition to sampling by means of a glass syringe. The analytical procedure, using the gas chromatograph, is not described in detail, but the respective literature is referred to. The basic work of compiling literature is being done within the ISO Technical Committee 146 (Ref. 2).

Detector tubes have also been included in this IUPAC paper. An extensive evaluation by direct reading detector tubes in the case of vinyl chloride has been conducted especially in Great Britain by the Vinyl Chloride Committee of the Chemical Industrial Association (Ref. 3), which has shown that detector tubes are suitable for air testing at workplaces. General aspects of measuring air contaminants and collection of samples were described in an earlier report of the Commission (Ref. 4).

I. VINYL CHLORIDE

I.1 PHYSIOLOGICAL RESPONSE

Vinyl chloride is a cancer suspect agent (Ref. 1). It is significantly more toxic (Table 1) than was generally accepted prior to 1974, the year when it was reported that three cases of angiosarcoma of the liver had occurred in workmen.

TABLE 1. Toxicity levels of vinyl chloride

Dose	No. of Animals	Results
25 ppm	120	5 liver angiosarcomas 4 zymbal gland carcinomas 1 nephroblastoma
10 ppm	120	1 liver angiosarcoma 2 extrahepatic angiosarcomas 2 zymbal gland carcinomas

Industrial Experience. There appears to be no question that angiosarcoma of the liver has been caused by exposure to vinyl chloride primarily while cleaning reactor vessels. As of May 1980, approximately 85 cases have been found worldwide.

Hygienic standards. The American Conference of Governmental Industrial Hygienists in 1981 recommended as threshold limit value (TLV) 5 ppm (13 mg/m³). The U.S. Department of Labor (OSHA) standard is 1 ppm. In Germany F.R. a technical recommended concentration for the limit value of 2 ppm has been laid down (for existing plants a value of 3 ppm is being tolerated).

I.2 SAMPLING BY MEANS OF CHARCOAL TUBES AND SUBSEQUENT GAS CHROMATOGRAPHY

I.2.1 Field of application

The method is applicable to short-term and long-term sampling of vinyl chloride contents from 0.1 ppm (0.25 mg/m³) to 100 ppm (250 mg/m³). The maximum sampling volume is 30 litres of air. The sampling volume can be reduced down to 0.1 litre. The total operative time is from 1 minute up to 8 hours. The method gives the average concentration during the sampling time.

Organic components which have similar retention time as vinyl chloride during gas chromatographic analysis will interfere. Interferences can be minimized by proper selection of gas chromatographic columns and conditions. Water mists and high humidity reduce the adsorptive capacity of the charcoal for vinyl chloride.

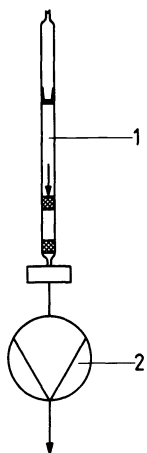


Fig. 1. Sampling of vinyl chloride in workplace atmospheres (principle).

- 1 sorbent tube
- 2 pump for drawing the air sample

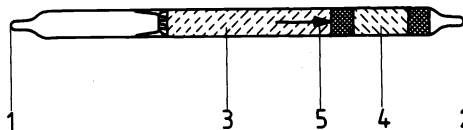


Fig. 2. Description of sorbent tube (the vinyl chloride is adsorbed in this tube).

- 1 and 2 sealed tips
- 3 adsorption section (charcoal)
- 4 back-up section (charcoal)
- 5 arrow (points towards pump during sampling)

I.2.2 Principle (see Figure 1)

A known volume of air is drawn (by means of a pump) through a sorbent tube containing two layers of activated charcoal (made from coconut shells), which adsorbs the vinyl chloride present in the air sample. The collected vinyl chloride is then desorbed with carbon disulfide and the resulting solution is analysed by gas chromatography with a flame ionization detector. The areas under the resulting peaks are compared with areas from the injection of standards.

Safety precautions. Carbon disulfide is highly flammable and toxic. Any contact must be avoided and usage should be restricted to a well ventilated hood.

I.2.3 Equipment and Materials

Sorbent tubes filled with activated charcoal (see Figure 2). The sorbent tube (glass tube) has both ends flame-sealed; inside diameter in the range from 5 to 6 mm, total length at least 100 mm. The sorbent tube contains two sections of activated charcoal; the first section contains at least 750 mg and the back-up section at most 250 mg charcoal. The sections are held in place with glass-wool plugs or other suitable fixing elements.

Plastic caps (polyethylene) for capping the sorbent tubes.

Sampling pump. The pump should conform to the local safety regulations, e.g. explosion-proof. The flow rate or volume of the pump should be properly calibrated with a representative sorbent tube in the sampling line. The following pumps are suitable:

- a) Diaphragm pump c) Automatic bellows pump
b) Peristaltic pump d) Hand-operated bellows pump

For long-term sampling one usually selects a pump having a lower flow rate (e.g. 10 to 20 mL/min). For shorter sampling periods a pump having a higher flow rate can be used (e.g. up to 1000 mL/min).

Extension hose. The material of the extension hose should be rubber or plastic, about 1 m long and equipped with a spring clip to hold the hose and the sorbent tube in place (e.g. on workers lapel area).

Gas chromatograph equipped with a flame ionization detector. It must be emphasized that any compound which has the same retention time as vinyl chloride at the operating conditions described in this method is an interferent. Hence, retention time data on a single column, or even on different columns, cannot be considered as proof of chemical identity. If the possibility of interference exists, separating conditions (column packing, temperature) must be changed.

A number of separating columns that have been found suitable are specified in Table 2. This table is not comprehensive.

TABLE 2. Examples of suitable columns for the separation of vinyl chloride from other components

Column	Footnote No.
10 % DC 200 on Chromosorb W/HP	1
10 % SE 30 on Chromosorb W/AW/DMCS	2
Porapak Q	3
Chromosorb 102	4
0.19 % Picric acid on Carboapak C	5
Chromosorb 101	6
Porapak QS	7
Apiezon L on Chromosorb and Carboapak B/HT-100	8

Footnotes:

- 1: P.W. Bankovich, et al., Am.Ind.Hyg.Assoc.J. 37, 640 (1976).
2: P & CAM 178, NIOSH Manual of Analytical methods (1977).
3: J.E. Cuddeback, et al., Environ.Sci.Techn. 9, 1168 (1975)
4: R.C. Lao, et al., Am.Ind.Hyg.Assoc.J. 37, 1 (1976).
5: E. Burghardt, et al., Atmospheric Environment 13, 1057 (1979).
6: S.A. Myers, et al., Am.Ind.Hyg.Assoc.J. 36, 332 (1975).
7: L.W. Severs, et al., Am.Ind.Hyg.Assoc.J. 36, 669 (1975).
8: B. Miller, et al., Analyst 103, 1165 (1978).

1.2.4 Sample collection

Immediately before sampling, the ends of the sorbent tube are broken to provide an opening of at least 1 mm.

Air being sampled is not to be passed through any hose or tubing before entering the sorbent tube. The sorbent tube is either connected to the extension hose or directly to the pump.

The inside of the pump or of the extension hose may liberate fugitive contaminants. To prevent any back-diffusion of these substances (before, during, or after sampling) through the outlet of the sorbent tube into the back-up section, one should insert a filter tube (charcoal) between sorbent tube and connection of hose or pump (see Figure 3a and 3b).

The flow rate and time, or volume, must be measured accurately. The sample is taken at any flow rate in the range from 10 to 1000 mL/min (the flow selected at the beginning of the test has to be maintained during the whole sampling time). The tube has to be placed on the sampling side and operated up to the wanted operative time (one minute up to eight hours).

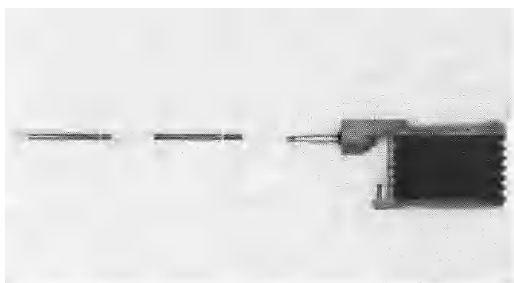


Fig. 3a

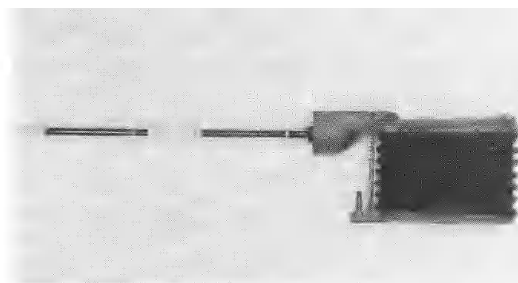


Fig. 3b

Fig. 3 To prevent back-diffusion of fugitive contaminants into back-up section of sorbent tube one can insert a filter tube between sorbent tube and connection of hose (3 a) or pump (3 b)

Temperature and pressure of the atmosphere being sampled, should be measured.

Immediately after sampling, the sorbent tube is capped with plastic caps. (Under no circumstances are rubber caps to be used).

One tube is handled in the same manner as the sample tube (breaking, sealing and transporting), except that no air is sampled through this tube. This tube is labelled as a blank.

Before shipping, the capped tubes are packed tightly to minimize breakage during transport to the laboratory. The period between sampling and analysis should not exceed one week.

Analysis of samples according to already existing laboratory methods.

Evaluation of the results according to the given task.

I.3 GRAB SAMPLING USING A GLASS SYRINGE

I.3.1 Field of application

The method is applicable to vinyl chloride concentrations from 0.1 ppm (0.25 mg/m³) to 100 ppm (250 mg/m³). The sampling volume is 100 mL. The total operative time is usually in the range of seconds.

I.3.2 Principle

The air is sampled by means of a syringe. Afterwards - for analysis - the air sample is blown directly into a gas chromatograph equipped with a flame ionization detector. The areas under the resulting peaks are compared with areas from standards.

I.3.3 Equipment and Materials

Glass syringe (piston-type sampler) with stop cock (Figure 4).

Fig. 4 Grab sampling of vinyl chloride by means of a glass syringe (piston-type sampler with stop cock)



Do not lubricate the syringe. The inside of the syringe and the surface of the piston must be clean. The polished surfaces of the piston and the syringe chamber must seal perfectly with respect to one another, but still move smoothly (precision polishing).

Gas chromatograph equipped with a flame ionization detector - as described under Section I.2.3.

I.3.4 Sample collection

Immediately before sampling, the stop cock must be opened.

Air being sampled is not to be passed through any hose or tubing before entering the syringe.

If necessary the syringe can be flushed by moving the piston to and fro several times.

Usually it will take a few seconds to draw the sample into the syringe.

After sampling, the stop cock must be closed.

Temperature and pressure of the atmosphere being sampled, should be measured.

Before transportation, the syringe is packed tightly to prevent breakage. The period between sampling and analysis should not exceed 24 hours.

Analysis of samples according to already existing laboratory methods.

Evaluation of the results according to the given task.

I.4 DETERMINATION BY SHORT-TERM DETECTOR TUBES

I.4.1 Field of application

The properties of the detector tubes must be in accordance with the IUPAC Performance standard for detector tube units as described in Pure Appl. Chem., Vol. 54, No. 9 (September 1982), pp. 1763-1767.

The total measuring range should at least cover concentrations from 1 to 50 ppm vinyl chloride. The test with this detector tube should not take more than 10 minutes.

I.4.2 Principle (see Figure 5)

A known volume of air is drawn (by means of a pump) through the detector tube containing the necessary filling layers. The vinyl chloride, present in the air sample reacts in the filling layer yielding a colour indication. The length of the discoloration is a measure of the vinyl chloride concentration.

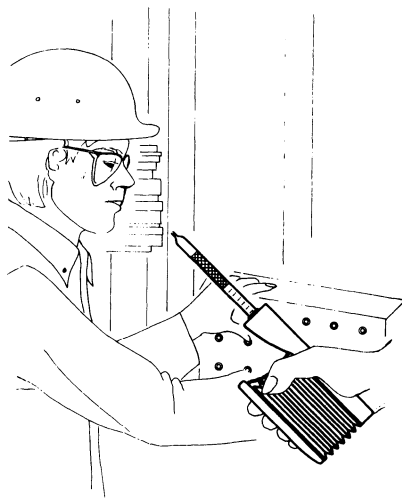


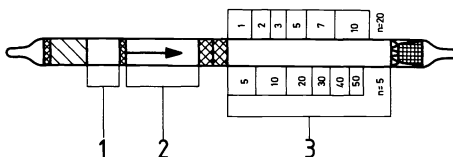
Fig. 5 Determination of vinyl chloride with short-term detector tubes

I.4.3 Equipment and MaterialsDetector tube

- (a) Standard range of measurement 1 to 10 ppm vinyl chloride
(20 °C, 1013 mbar) 5 to 50 ppm vinyl chloride
- (b) Number of strokes of the gas detector pump n = 20
n = 5
- (c) Relative standard deviation 15 to 10 %
- (d) Description (see Fig. 6)
Scale tube * greenish-grey precleanse layer (1), reagent: alkali hydroxide * brownish-grey oxidation layer (2), reagent: permanganate * white indicating layer (3), reagent: o-tolidine * colour change to weak yellowish-orange.

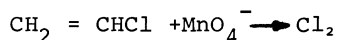
Fig. 6 Construction of short-term detector tube for vinyl chloride determinations

- 1 greenish-grey precleanse layer
2 brownish-grey oxidation layer
3 white indicating layer

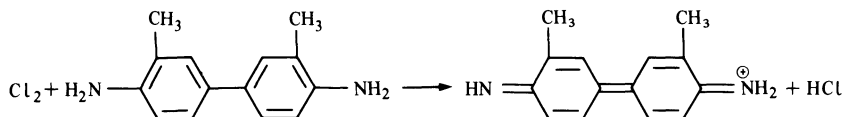


(e) Reaction principle

(in the oxidation layer):



(in the indicating layer):



(f) Cross-sensitivity

Chlorine and hydrogen chloride are absorbed in the precleanse layer and do not, therefore, interfere with the vinyl chloride indication.

Other chlorinated hydrocarbons (e.g. trichloroethylene, benzyl chloride) may give similar reactions as vinyl chloride.

In the presence of hydrocarbons (e.g. butadiene, ethylene, heptane, toluene), part of the permanganate is consumed. Less vinyl chloride than is actually present may be indicated.

At the request of the tube user, the manufacturer should supply information about the influence of interferences on the course of the reaction and the reliability of the indication.

Sampling pump. Pump and detector tube together form a complete unit.

The pump assigned for use with the vinyl chloride detector tube must have the same flow characteristics as those of the pump used by the manufacturer in calibrating the tubes. Because pumps made by different manufacturers may not operate at the same rate even when they draw the same volume, they cannot be used interchangeably.

I.5 DETERMINATION BY LONG-TERM DETECTOR TUBES

I.5.1 Field of application

The properties of the detector tubes must be in accordance with the IUPAC Performance standard for detector tube units as described in Pure Appl. Chem., Vol. 54, No. 9 (September 1982), pp. 1763-1767.

The detector tubes should be designed to determine the time-weighted average.

I.5.2 Principle

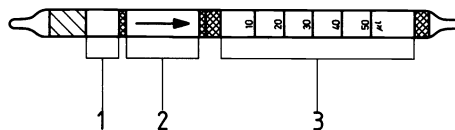
A known volume of air is drawn (by means of a pump at a flow rate of approx. 1 liter/h) through the detector tube containing the necessary filling layers. The vinyl chloride, present in the air sample, reacts in the filling layer yielding a colour indication. The length of the discoloration is a measure of the vinyl chloride concentration.

I.5.3 Equipment and MaterialsDetector tube

- (a) Range of measurement (20 °C, 1013 mbar)
- | | |
|-----------------------------------|----------------------------------|
| - in absolute units | 10 to 50 µL vinyl chloride (gas) |
| - for maximum period of use (8 h) | 1.3 to 6.3 ppm vinyl chloride |
| - for shorter period of use (2 h) | 5 to 25 ppm |
| - for shorter period of use (1 h) | 10 to 50 ppm |
- (b) Sampling volume
approximately 1 liter per hour.
- (c) Relative standard deviation 15 to 10 %
- (d) Description (see Figure 7)
Scale tube * greenish-grey precleanse layer (1), (drying agent) * yellowish-orange oxidation layer (2), reagent: chromate * light grey indicating layer (3), reagent: o-tolidine * colour change to orange

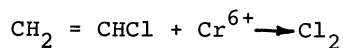
Fig. 7 Construction of long-term detector tube for vinyl chloride determinations

- 1 greenish-grey precleanse layer
2 yellowish-orange oxidation layer
3 light grey indicating layer

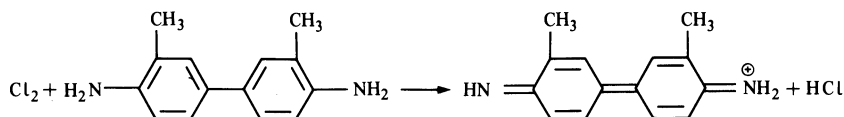


(e) Reaction principle

(in the oxidation layer):



(in the indicating layer):



(f) Cross-sensitivity

Easy to split halogenated hydrocarbons are also indicated; for example: Vinylidene chloride (1,1-dichloroethylene) same sensitivity as vinyl chloride (systematic deviation up to $\pm 30\%$).

1,2-Dibromoethane double sensitivity as vinyl chloride; for evaluation multiply the dibromoethane reading by factor 0.5 (systematic deviation up to $\pm 30\%$).

Cl_2 and HCl are trapped in the precleanse layer and therefore do not interfere.

In presence of easy to oxidize organic compounds, part of the oxidation layer (Cr^{6+}) may be consumed; in such a case the vinyl chloride reading will be too low: e.g.:

10 ppm vinyl chloride + 10 ppm ethylene give reading of 7 ppm
10 ppm vinyl chloride + 100 ppm ethylene give reading of 1 ppm

Sampling pump. The aspirating pump assigned for use with the long-term detector tube must have the same flow characteristics as those of the pump used by the manufacturer in calibrating the tubes. Several pumps are available on the market, fulfilling these requirements in the range of low flow rates.

II. BENZENE

II.1 PHYSIOLOGICAL RESPONSE

Benzene has been recognized as a toxic substance capable of causing acute or chronic effects. Inhalation is the primary route of entry into the body (Ref. 5). Experiments on laboratory animals have demonstrated that benzene can be absorbed through the skin to some extent (Ref. 1). The following data have been taken from literature (Ref. 5), in which an overview is provided about publications on physiological and toxicologic effects of benzene.

Acute toxicity. The primary acute toxic effect of benzene is on the central nervous system and is seen initially at exposures above 250 ppm. Exposure to concentrations of the order of 2.5% by volume in air (about 25000 ppm) is fatal within minutes. The prominent signs are central nervous system depression and convulsions with death usually following as a consequence of cardiovascular collapse. Milder exposures (4000 ppm) produce euphoria followed by giddiness, headache, nausea, staggering gait, and finally unconsciousness if exposure continues. Inhalation of lower concentrations (250-500 ppm) produces vertigo, drowsiness, headache and nausea, symptoms which clear rapidly once exposure ceases.

Chronic toxicity. Exposures above about 50 ppm for extended periods may induce changes in the bone marrow which result in decreased levels of platelets, red blood cells or white blood cells. The effects of benzene on the bone marrow may become irreversible unless benzene exposure is stopped. The signs of chronic benzene poisoning can appear any time following a few weeks to several years of exposure. Typical symptoms are rather non-specific; severe fatigue, headache, dizziness, nausea, vertigo, stomach pain, loss of appetite or feeling cold. Prolonged or repeated skin contact may produce a dry, scaly dermatitis, erythema, and blistering.

Leukemia. The evidence for benzene-induced leukemia rests largely on epidemiological grounds. The clearest relationship between benzene exposure and leukemia was demonstrated in studies of benzene exposure prior to 1960 when high benzene concentrations (> 200 ppm) in the air were likely. The most common form is acute myelogenous leukemia. On the other hand, no increased incidence of leukemia was observed among workers employed in the petrochemical industries (outdoor, enclosed systems) or in coke-oven byproduct workers, despite the fact that the latter group of workers may have been exposed to high levels of benzene from fugitive emissions.

In all well documented cases of benzene-related leukemia, victims had developed a previously detectable decrease in blood levels of one or more of the cell lines.

Reproductive effects. Such risks could involve male and female reproductive organs and functions as well as the developing fetus. A number of studies in animals bear on this issue. A publication made mention of testicular changes in guinea pigs and rabbits exposed repeatedly to benzene vapor at about 80 ppm. Four teratology studies using rats and conducted in different laboratories demonstrated fetal toxicity at 40, 50, 500 and 2200 ppm; maternal toxicity at 500 ppm. Some malformations were seen at 500 ppm and other changes at 100 and 300 ppm. Studies at 10 ppm produced conflicting results. A fourth series of experiments at 500 ppm showed fetal toxicity in mice and some slight developmental changes in mice and rabbits.

The overall conclusion from this series of animal studies is that benzene has been observed to have a weak toxic effect on pregnant females and on fetuses.

Workplace limits. The present occupational exposure limit in USA is 10 ppm (32 mg/m³) TWA for an 8-hour workday/40-hour work week. A ceiling value (15-minute) of 25 ppm (80 mg/m³) is also included. Other countries have set standards for maximum allowable levels of benzene in air. In West Europe, present recommendations are generally set at 10 ppm (8-hour) and 20-25 ppm ceiling (15-minute) except for Germany where a TRK value (tolerable risk value) of 8 ppm was established. Individual companies have adopted a 10 ppm ceiling (15-minute) limit to reduce excursions or because of uncertainty regarding potential reproductive risks. An alternative approach made by Exxon Corporation (1982) uses 250 ppm x minutes for exposures between 5 and 30 minutes; this provides a maximum level of 50 ppm for 5 minute exposure. Higher levels require the use of respiratory protection or other control means. They also recommend 5 ppm (8-hours) and 3 ppm (12-hours).

II.2 SAMPLING BY MEANS OF CHARCOAL TUBES AND SUBSEQUENT GAS CHROMATOGRAPHY

II.2.1 Field of Application

This method is applicable to benzene concentrations from 0.1 ppm (0.33 mg/m³) to 100 ppm (326 mg/m³). The maximum sampling volume is 50 L of air. The sampling volume can be reduced down to 0.1 L. The upper total amount for benzene is 6 mg per sample. The total operative time is from 1 minute up to 8 hours. The method gives the average concentration during the sampling time.

Interferences from organic components can be minimized by proper selection of gas chromatographic columns and conditions. Water mists and high humidity reduce the adsorptive capacity of the charcoal for benzene.

II.2.2 Principle (see Figure 1)

As described under vinyl chloride, Section I.2.2.

II.2.3 Equipment and Materials

Sorbent tubes (as described under vinyl chloride, Section I.2.3) filled with activated charcoal (Figure 2) of particle size 0.35 to 0.85 mm have the inside diameter 4 mm and total length 70 mm. The first section contains 100 mg and the back-up section 50 mg of activated charcoal.

Plastic caps, Sampling pump, Extension hose, and Gas chromatograph used are as described under vinyl chloride (Section I.2.3).

The separating columns found suitable are listed in Table 3. This table is not comprehensive.

TABLE 3. Examples of suitable GC columns for the separation of benzene from other components

Column	Footnote No.
Porapac Q	1
Chromosorb W/AW/DMCS	2
Chromosorb P/AW/DMCS	3

Footnotes:

- 1: Method S 311 in NIOSH Manual of Analytical Methods (1977).
- 2: Criteria for a Recommended Standard. Occupational Exposure to Benzene. US Department of Health Education and Welfare (1974).
- 3: G.G. Esposito et al. "Chromatic Determination of Aromatic Hydrocarbons in Ambient Air" Am. Ind. Hyg. Assoc. J., 38, 401 (1977).

II.2.4 Sample collection

As described under vinyl chloride, Section I.2.4.

II.3 GRAB SAMPLING USING A GLASS SYRINGE

II.3.1 Field of Application

The method is applicable to benzene concentrations from 0.1 ppm (0.33 mg/m³) to 100 ppm (326 mg/m³). The sampling volume is 100 mL. The total operative time is usually in the range of seconds.

II.3.2 Principle

As described under vinyl chloride, Section I.3.2.

II.3.3 Equipment and Materials (see Figure 4)

As described under vinyl chloride, Section I.3.3.

II.4 DETERMINATION BY SHORT-TERM DETECTOR TUBES

II.4.1 Field of Application

The total measuring range should at least cover concentrations from 5 to 50 ppm benzene.

II.4.2 Principle (see Figure 5)

As described under vinyl chloride, Section I.4.2.

II.4.3 Equipment and Materials

Detector tube

- | | |
|---|--|
| (a) Standard range of measurement
(20 °C, 1013 mbar) | 5 to 50 ppm benzene |
| (b) Number of strokes of the gas detector pump | n = 20 to 2 (depending on concentration) |
| (c) Relative standard deviation | Not more than ± 35% at 10 ppm benzene
(related to 95% confidence level) |
| (d) Description (See Fig. 8) | |

Colour comparison tube * pale grey precleanse layer (1), reagent: acid and aldehyde
 * white indicating layer (2), reagent: formaldehyde and sulfuric acid * colour
 change to pale reddish brown * pale reddish brown colour comparison layer (3).

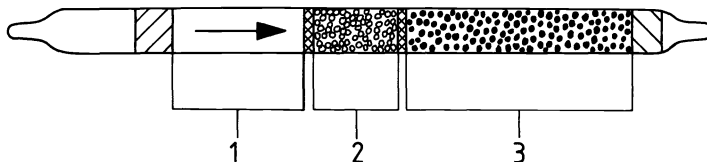
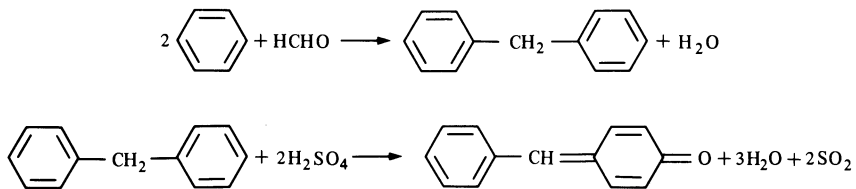


Fig. 8 Construction of short-term detector tube for benzene determinations

- 1 pale grey precleanse layer
- 2 white indicating layer
- 3 pale reddish-brown colour comparison layer

(e) Reaction principle



(f) Cross-sensitivity

The precleanse layer absorbs interfering components (e.g. toluene, xylene) yielding a reddish-brown reaction in the precleanse layer.

With high concentrations of interfering substances, the entire precleanse layer changes colour and the discoloration can then extend into the indicating layer (interfering substances react in the indicating layer). In this case, determination of benzene is no longer possible.

Examples for absorption capacity of pre-layer (no break-through of substance) after 10 strokes:

Xylene	100 ppm	Butadiene	500 ppm
Styrene	100 ppm	Toluene	20 ppm

Sampling pump. As described under vinyl chloride, Section I.4.3.

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5. R.S. Brief et al., Benzene in the Workplace. Am. Ind. Hyg. Assoc. J. 41 (Sept. 1980) 616-623.