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Determination of Benzene in Wastewater by Gas Chromatography

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Benzene is encountered in a wide variety of chemical processes. Because of its high toxicity, its determination in air has been reported by several research groups (Takeuchi et al.1961, Wathne 1983, Asai et al. 1989), but few reports concerning its analysis in wastewater have appeared. In order to establish a simple and precise method for the determination of benzene in wastewater, we have studied a gas chromatographic method.

MATERIALS AND METHODS

One hundred mL of a water sample was placed in a separatory funnel, and 5 mL of m-xylene was added to the water. The mixture was shaken for 1 min. The separated m-xylene layer was subjected to gas chromatography (GLC) to assay the concentration of benzene. The assay was performed on a Shimadzu Model GC-6A gas chromatograph equipped with a flame ionization detector and a glass column (3 m x 3 mm I.D.) packed with 20 % polyethyleneglycol 1500 on 60-80 mesh Celite 545. An initial column temperature at 30°C was gradually raised to 125°C at a rate of 3°C/min. Both the detector and the injection port were operated at 150°C. A flow rate of carrier gas was kept at 40 mL/min. Under these conditions, benzene eluted at a retention time of 10 min. The peak heights were estimated on the gas chromatograms obtained from a sample and benzene standard solutions. Concentrations of benzene in 90 samples of wastewater from the wastewater storage chamber collected from chemical laboratories of Kobe Women's College of Pharmacy were determined throughout a one-year period (from March 1990 to February 1991).

RESULTS AND DISCUSSION

A GLC method for the determination of benzene in

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wastewater has been investigated. Takeuchi et al. (1961) reported a GLC method using DOP (diiso-octyl phthalate) as a stationary phase for the determination of benzene, toluene and xylene in air. Wathne (1983) used a glass capillary column packed with Ucon LB 550X 33 for assaying benzene, toluene and xylene in air. We have found that a column of 20 % polyethyleneglycol 1500 on Celite 545 (60-80 mesh) gives better separation of organic solvents than the other stationary phases previously reported. Figure 1 shows a gas chromatogram on a mixed sample of 13 organic solvents usually used in chemical laboratories. A peak corresponding to benzene eluted as the 5th peak in the chromatogram; it was satisfactorily separated from the other solvents. Figure 2 shows a calibration curve obtained for benzene. A straight line passing through the origin was obtained in the plot of concentrations of benzene against peak heights or peak areas. In order to extract organic solvents from aqueous samples, solvent extraction with m-xylene, headspace analysis and closed-loop stripping techniques were applied. Samples of wastewater from chemical laboratories were subjected to the proposed procedure with or without addition of 1.0 mg/L of the standard compound to perform recovery tests. As shown in Table 1, the recoveries of benzene were within the range of 96.3 and 102.0 % and the coefficients of variation (CV) were within 7.5 %. The detection limit was 0.05 mg/L. Since it takes only approximately 1 hr to perform the whole procedure, the proposed method is very simple as a routine one. When this method was applied to 90 samples of wastewater periodically collected from chemical laboratories (from March 1990

Table 1. Recovery of benzene added to wastewater from chemical laboratories

Wastewater Sample (1990)	Benzene Added (mg/L)	Recovery $\bar{M} \pm SD$ (%)	CV (%)
March 5	1.0	102.0 ± 4.0	3.9
March 7	1.0	96.3 ± 4.5	4.7
March 16	1.0	99.0 ± 2.3	2.3
March 29	1.0	101.0 ± 2.5	2.5
April 10	1.0	99.3 ± 6.2	6.2
April 18	1.0	99.0 ± 7.4	7.5

All values are the mean of triplicate determinations.

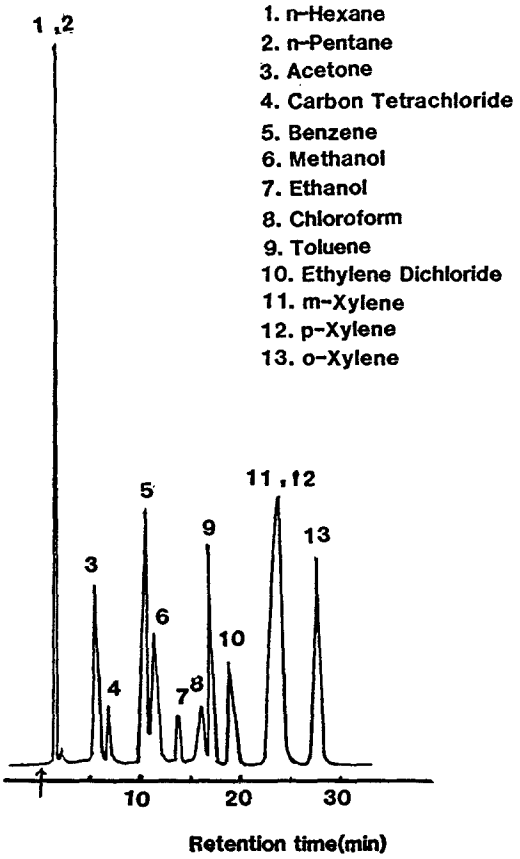

Figure 1. Gas chromatogram of various organic solvents

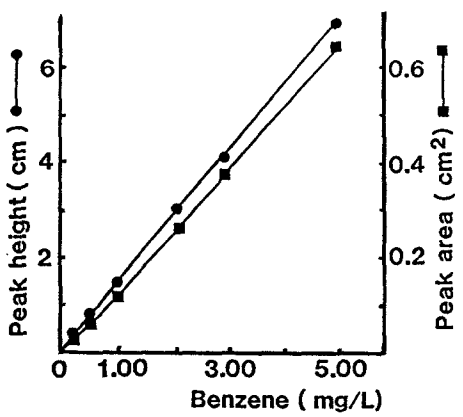

Figure 2. Calibration curve of benzene by GLC method

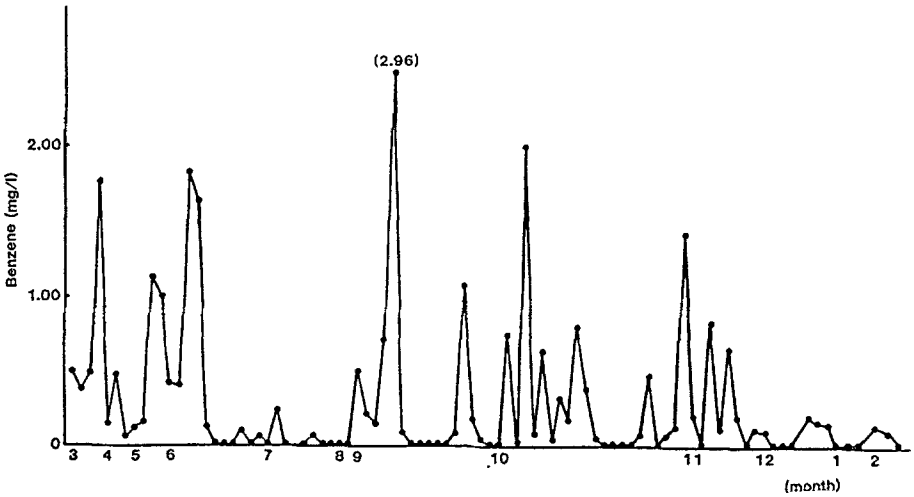

Figure 3. Variation of benzene in wastewater from chemical laboratories

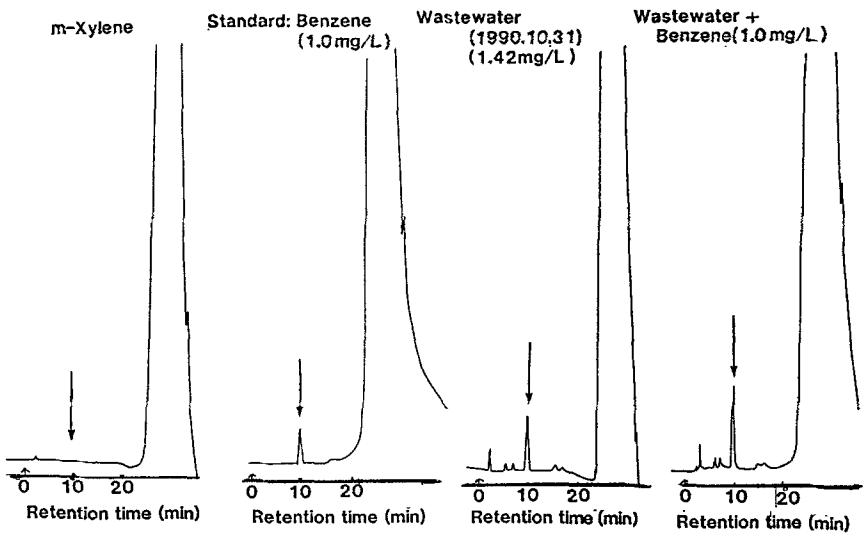

Figure 4. Gas chromatograms of wastewater from chemical laboratories

through February 1991), the assayed values were distributed between 0.05 and 2.96 mg/L (Figure 3). Figure 4 shows the gas chromatograms of standard compounds of m-xylene and benzene and a sample of wastewater from a chemical laboratory. A peak corresponding to benzene was observed in the chromatograms of the wastewater sample, and it was not disturbed by the

other peaks (Figure 4). The peak was confirmed as benzene by a co-elution test and gas chromatography-mass spectrometry. In conclusions, the presently proposed method is useful for the routine determination of benzene in wastewater.

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