Notes

Adsorption of polar substances on the solid support of column packings in the gas–liquid chromatographic analysis of air contaminants*

Many air contaminants can be sampled efficiently with the sixfold absorber developed by BERGSHOEFF[1]. If for instance 50 l of air containing 80 mg benzene per m^3 are drawn through 100 ml of liquid the resulting solution contains 40 p.p.m. of benzene.

On analyzing such test solutions gas chromatographically we found that polar compounds practically disappeared due to adsorption on the silanised support coated with a nonpolar liquid phase. We believe this phenomenon may be useful in analyzing air contamination.

Experimental

Test solutions, with propanol-2 (I), hexane (II) and decane (III) as absorption liquids, each containing 600 p.p.m. of benzene, *m*-xylene, isobutyl acetate and 1400 p.p.m. of butanol-2, were made up. These "concentrated" solutions, designated I. 1, II. 1 and III. 1, were also diluted twenty times. The "concentrated" and "diluted" designated I. 2, II. 2 and III. 2 solutions were analyzed gas chromatographically.

For the GLC analysis we used an Aerograph model 1520 with a flame ionization detector and a set of copper columns (length 5 m, I.D. 0.065 in.) packed with 20 % Apiezon L on commercial silanised Chromosorb W, 80–100 mesh.

GLC conditions. Test solutions I. 1 and I. 2: Sample 0.3 μl; R. 1; Att. 16, 8, 4, 2 and 1 (dependent on the tailing of propanol-2); column temp. 101°; injector and detector 180°; carrier gas (N_2) and hydrogen 25 ml/min.

Test solutions II. 1 and II. 2: sample 2.1 μl; R. 1; Att. 8 and 2; column temp. 102°; injector 155° and detector 175°; carrier gas (N_2) and hydrogen 25 ml/min.

Test solutions III.1 and III.2; sample 1.7 μl; R.1; Att. 1; column temp. 15 min at 100°, 15 min programming at a rate of 6°/min, 15 min at 190°; injector and detector 190°; carrier gas (N_2), analytical column 20, reference column 25 ml/min; hydrogen 25 ml/min.

Results

The results of the GLC analysis of the various test solutions are given in Table I.

Discussion

From the results it can been seen that very small quantities of polar components are not detected with the nonpolar columns used. At first we supposed this was caused by decomposition in the injector. However, no peaks on the chromatograms originating from decomposition products (*e.g.* butene) were seen. We now believe that small

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TABLE I

RESULTS OF THE GLC ANALYSIS OF THE TEST RESOLUTIONS ON THE NON-POLAR COLUMN

Test solution		*Response**		
		Aromatics	*Ester*	*Alcohol*
Propanol-2 solutions	I.1	+ +	+	+
	I.2	+ +	—	—
Hexane solutions	II.1	+ +	+	x
	II.2	+ +	—	x
Decane solutions	III.1	+ +	+	—
	III.2	+ +	—	—

* + + = Positive response; + = positive response, much tailing; — = no response; x = retention time equal to that of hexane.

quantities of polar compounds are adsorbed on the solid support, which is insufficiently deactivated for these traces.

KUSÝ[2] discusses the adsorption of polar substances on the solid support in a study concerning the accuracy of quantitative GLC. We believe that the detection of butanol-2 present in test solution (I.1) is caused by a deactivation of the solid support by the absorption liquid propanol-2. The detection of the ester and not the alcohol in the "concentrated" solutions (*e.g.* III.1) will be caused by a stronger adsorption of the alcohol through hydrogen bonding.

To see if there is any adsorption of small quantities of polar substances on the solid support when using a polar liquid phase, a test solution containing 60 p.p.m. butanol-2 in cyclohexanol was chromatographed on the Aerograph 1520 with a flame ionization detector. The appearance of a butanol-2 peak without tailing indicates that there is no adsorption in this case. (A set of copper columns was used, length 3 m, I.D. 0.065 in., packed with 10 % Carbowax 1500 on commercial silanised Chromosorb W, 60–80 mesh. GLC conditions: sample 1 μl; R.1; Att. 4; temperature 10 min at 100°, 20 min programming at a rate of 6°/min; injector and detector 190°; carrier gas (N_2), analytical column 20, reference column 25 ml/min, hydrogen 25 ml/min).

It is concluded that the phenomenon of the adsorption of polar substances on the solid support of nonpolar columns may be used in the qualitative analysis of air contaminants. When this analysis is performed on both polar and nonpolar packed columns, then, very probably, polar substances will not be detected on nonpolar packed columns.

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1 G. BERGSHOEFF, *Chem. Weekblad*, 57 (1961) 637 and 645.
2 V. KUSÝ, *Anal. Chem.*, 37 (1965) 1748.

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