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## Note

### JADO carbon as an adsorbent in sampling columns\*

V. PATZELOVÁ

*J. Heyrovský Institute of Physical Chemistry and Electrochemistry, Czechoslovak Academy of Sciences, Máchova 7, 121 38 Prague 2 (Czechoslovakia)*

and

M. WURST and V. ČATSKÁ

*Institute of Microbiology, Czechoslovak Academy of Sciences, Budějovická 1083, 140 00 Prague 4 (Czechoslovakia)*

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Recently, adsorbents employed in gas chromatography (GC) have been used to concentrate organic compounds in the air. Different methods involving these GC column packings have been developed for headspace and ambient air analysis in medical and environmental research<sup>1-7</sup>. In some cases, the adsorption capacity is increased by maintaining the trapping column at the temperature of liquid nitrogen or of solid carbon dioxide. After the sample has been collected, it is desorbed by heating at an elevated temperature and eluted with carrier gas from the collecting column to an analytical column. Alternatively, the sample may be further condensed in a cold trap<sup>4</sup> and aliquots of the condensate then removed for analysis by GC. A desiccator may also be used to eliminate the water from the sample before the GC analysis<sup>7</sup>.

Direct transfer of the preconcentrated components into the analytical column yields results which are not influenced by non-quantitative condensation of highly volatile compounds, e.g., low-molecular-weight hydrocarbons, or by interactions of low-molecular-weight alcohols or fatty acids with desiccants. Thus, this method seems to be the most suitable. Nonetheless, errors may result from non-quantitative adsorption or desorption of all the components.

Generally, an ideal adsorbent should have a large adsorption capacity but the energy of adsorption for the adsorbates should be low. Butler and Burke<sup>8</sup> have studied different types of commercially available porous polymeric adsorbents in terms of these general requirements. We have examined the application of a new type of active carbon, JADO<sup>9</sup>, as an adsorbent for preconcentration of organic compounds from air.

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## EXPERIMENTAL

JADO active carbon was prepared by electrochemical reduction of polytetrafluoroethylene (PTFE) by lithium amalgam at a low temperature<sup>9,10</sup>. Here, 0.3 mm PTFE particles were used, covered with a carbon layer. Before the measurements the material was first conditioned at 563°K for 3 h in a flow of helium. After the conditioning no measureable decrease in weight was observed.

Reagent grade methanol, ethanol, formic acid, tetrachloromethane, ethylene, *n*-hexane, *n*-heptane and benzene were used as probes without further purification. All measurements were carried out on a CHROM-41 gas chromatograph (Laboratorní Přístroje, Prague, Czechoslovakia) with a thermal conductivity detector. U-shaped glass columns (600 × 3 mm I.D.) were used.

Frontal and elution chromatographic techniques were employed. The helium carrier gas was presaturated in a two step saturator consisting of saturating, diluting and mixing devices. This equipment was connected to the inlet of the chromatograph. The concentration of the adsorbate vapour was proportional to the saturator temperature and to the carrier gas velocity. The actual value of the adsorbate partial pressure in the carrier gas was calculated before the measurements from the weight increment of a cold trap connected to the saturator outlet.

Sample collection efficiencies equal to the dynamic adsorption capacities for each adsorbate were calculated from the frontal analysis experiments with the aid of the following expression.

$$a = \frac{(t - t_M)F}{W} \cdot c$$

where  $a$  is the adsorption capacity of the given adsorbate in grams per gram of adsorbent,  $t$  is the time of saturation of the adsorbent in sec,  $t_M$  is the gas retention time in sec,  $W$  is the weight of adsorbent in grams,  $F$  is the carrier gas flow-rate in  $\text{cm}^3 \text{ sec}^{-1}$ ,  $c$  is the concentration of adsorbate in the carrier gas in  $\text{g cm}^{-3}$  and  $(t - t_M)F/W$  is the volume of carrier gas containing the adsorbate of concentration  $c$  which is necessary to saturate 1 g of adsorbent under the given working conditions at  $T = 293^\circ\text{K}$ .

The calculated values were compared with the data obtained from the direct weight of the column before and after the saturation. The temperature of the saturator and the carrier gas velocities were optimized in order to yield an adsorbate partial pressure of ca. 1.3 Pa.

For ethylene, a simple two-step dilutor was used. In order to test the rate of desorption at  $T = 558^\circ\text{K}$ , the elution GC technique was employed. Samples containing 0.05–5  $\text{mm}^3$  of liquid and 0.5–30  $\text{mm}^3$  of gaseous adsorbate phase were injected using Hamilton gas-tight syringes.

## RESULTS AND DISCUSSION

The physical characteristics of JADO carbon are given in Table I. For comparison, data are given for Porapak R and Tenax-GC, published elsewhere<sup>8</sup>.

The dynamic adsorption capacities of selected adsorbates are listed in Table II. It follows from these data that the amounts trapped on 1 g of JADO are detectable

TABLE I  
PHYSICAL CHARACTERISTICS OF JADO CARBON

Adsorbent	Surface area ( $\text{m}^2 \text{g}^{-1}$ )	Packing density ( $\text{g cm}^{-3}$ )	Temperature limit ( $^{\circ}\text{K}$ )
JADO	800	0.87	563
Porapak R*	550-750		523
Tenax-GC*	19		648

\* Ref. 8.

TABLE II  
ADSORPTION CAPACITIES OF JADO CARBON

$T = 293^{\circ}\text{K}$ ;  $p_{\text{ads}} = 1.33 \text{ Pa}$ .

Adsorbate	Adsorption capacity	
	$\text{mmole g}^{-1}$	$\text{mg g}^{-1}$
Methanol	0.037	1.184
Ethanol	0.022	1.012
Formic acid	0.024	1.104
Benzene	0.014	1.092
Tetrachloromethane	0.009	1.386
Ethylene	0.001	0.280
<i>n</i> -Hexane	0.007	0.602
<i>n</i> -Heptane	0.007	0.686

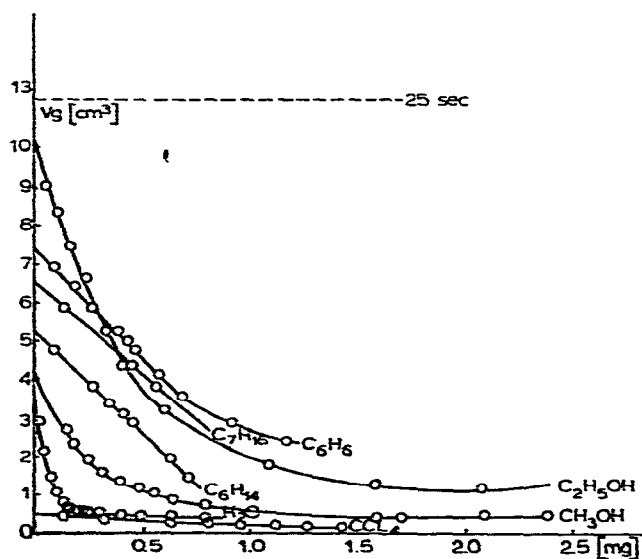


Fig. 1. Dependences of specific retention volumes,  $V_g$ , on the sampled amount of adsorbate.  $T = 558^{\circ}\text{K}$ ,  $F = 45 \text{ cm}^3 \text{ min}^{-1}$ .

by a thermal conductivity detector. On the other hand, it was necessary to determine the rate of desorption at an elevated temperature,  $T = 558^\circ\text{K}$ .

In our system the metallic column containing the adsorbent was closed after the adsorption and then heated. A temperature of  $558^\circ\text{K}$  was reached after *ca.* 5 min. The carrier gas was introduced into the column and the desorbed components were transferred into the chromatographic column. Under these conditions it can be assumed that the time necessary for the quantitative desorption of a component at an elevated temperature will be approximately the same as the retention time of the component injected into the column.

The dependence of the specific retention volumes on the injected amounts is given in Fig. 1. These graphs permit a determination of the time necessary for quantitative desorption of the adsorbates under the given working conditions ( $T = 550^\circ\text{K}$ ,  $F = 45 \text{ cm}^3 \text{ min}^{-1}$ ). The time does not exceed 25 sec.

The values of  $V_g$  for hydrocarbons in the region of coverage studied are in directly proportion to the injected amount. This fact indicates that the rate of desorption is limited by the slope of the linear adsorption isotherm. For oxygen-containing components, the dependence was linear only in the region of very low coverages.

In order to demonstrate the usefulness of JADO carbon for practical applications, an example is given. The scheme in Fig. 2 illustrates the equipment for determination of trace amounts of organic compounds in the atmosphere of an experimental biological container. In the first step adsorption takes place. The air from the container is then pumped through the trapping column in a closed circuit. In the second step both ends of the trapping column are closed and it is placed in a small quartz furnace and heated. In the third step the adsorbates are swept by the carrier gas into the chromatographic column. During the first and second steps the chromatograph works separately.

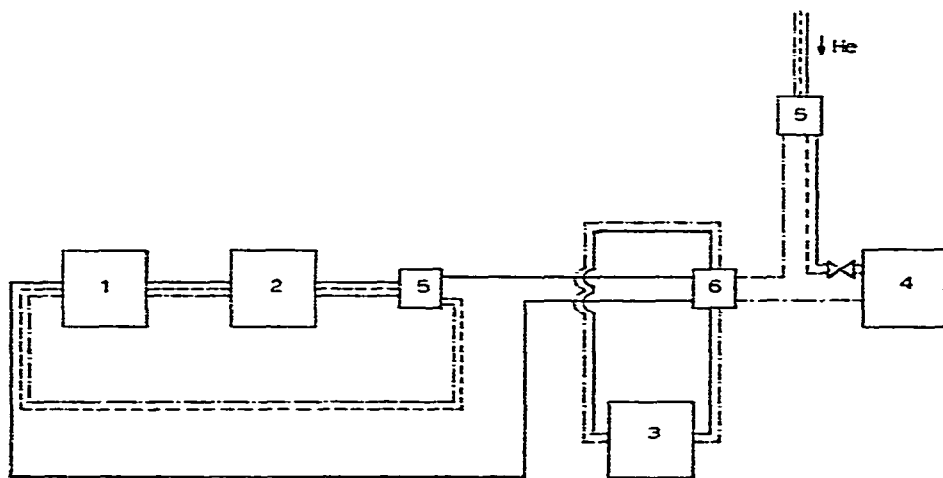


Fig. 2. Scheme of the trapping equipment. 1 = Membrane pump; 2 = biological container; 3 = trapping column with JADO carbon; 4 = chromatograph; 5 = two-way valve; 6 = six-way valve. —, Adsorption step; - - -, heating step; - · -, desorption step.

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