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Note

Determination of the amount of stationary phase in packings for gas–liquid chromatography

Stationary phases for use at high temperatures

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In a previous paper[1], it was shown that the amount of stationary phase in columns for gas–liquid chromatography could be found by measuring the evaporation of the phase from the packing. For this purpose were used packings of squalane, dinonylphthalate and β,β'-oxydipropionitrile on Sterchamol, Chromosorb P and Chromosorb W.

Bearing in mind the principle of the method, evaporation of the stationary phase from the packing, it can be assumed that stationary phases used at high temperatures would be the most difficult to study by this method. The purpose of this work, therefore, was to show the applicability of the evaporation method to the analysis of such packings, in which the stationary phases have low volatility.

EXPERIMENTAL

The method was evaluated by the analysis of packings with known amounts of the stationary phases Apiezon M (grease), polyethylene glycol adipate and polyethylene glycol 6000. Sterchamol with particle diameter of 0.2–0.3 mm, dried for 1 h at 110°, was used as the inert support.

The required amounts of support and stationary phase were weighed to an accuracy of $\pm 2 \cdot 10^{-4}$ g. The coating of the stationary liquid on the support was carried out in the crucible in which the determination was later made.

The analyses of the packings, prepared as described in the previous work[1], were carried out by heating them in a pot furnace. The amount of stationary phase present was calculated from the difference between the weights of the sample of the packing and the support that remained in the crucible.

RESULTS AND DISCUSSION

Evaporation of the stationary phase occurs even during the preparation of the

TABLE I

WEIGHT LOSSES OF THE STATIONARY PHASES AS A PERCENTAGE OF THE INITIAL WEIGHTS OF THE PHASES

Stationary phase	*In bulk state*	*Coated on support*
SE-30 silicone elastomer	0.18	0.37
Squalane	0.44	2.32
Apiezon M (grease)	—	0.02
Polyethylene glycol adipate	0.67	2.25
Polyethylene glycol 6000	0.92	8.85

packing. It was found that the heating of the solution of the stationary phase plus the support during the coating, in order to remove the solvent, also leads to some losses.

Table I shows the results for the weight losses of some stationary phases heated at 100° for 2 h as bulk liquids and coated on Sterchamol (about 20% of the weight of the support). The losses were calculated as a percentage of the initial weight of the stationary phases in the sample.

The two main factors that influence the evaporation of the phase are the time and temperature of heating, and these factors are related. However, it is not acceptable to decrease the time of analysis by using an excessively high temperature.

Table II indicates the extent of evaporation from the packing liquid phase, depending on the time of analysis. For this purpose, packings with 5% and 10% Apiezon M and 5% polyethylene glycol adipate on Sterchamol were used. The samples were heated first at 300° and, when their weights remained constant, the temperature was increased to 400°. The results are presented as a percentage from the starting amount of the phase in the packing.

From the results, it is evident that after a certain time at a given temperature, the percentage of the liquid phase evaporated reaches a constant value and subsequent heating does not lead to further evaporation. The amount of liquid phase retained in the packing could be evaporated by increasing the temperature.

The retention of a small proportion of the stationary phase on the support

TABLE II

AMOUNT OF STATIONARY PHASE EVAPORATED FROM PACKINGS DEPENDING ON THE TIME AND TEMPERATURE OF HEATING

Temperature (°C)	*Time (h)*	*Packing*		
		5% Apiezon M	*10% Apiezon M*	*5% Polyethylene glycol adipate*
300	0.5	70.50	66.14	93.37
	1.0	72.69	71.55	95.23
	1.5	74.51	73.61	95.43
	2.0	76.33	74.49	95.43
	2.5	77.06	75.08	95.43
	3.0	77.06	75.08	95.43
400	3.5	99.91	99.98	99.99
	4.0	99.91	99.98	99.99

TABLE III

TIME (HOURS) NECESSARY FOR QUANTITATIVE EVAPORATION OF THE STATIONARY PHASES

Loading of phase (%)	*Temperature (°C)*	*Stationary phases in the packings*		
		Apiezon M	*Polyethylene glycol adipate*	*Polyethylene glycol 6000*
10	250	—	—	2
	300	—	—	1
	350	—	1	0.5
	400	1.5	1	0.5
5	250	—	—	1.5
	300	—	—	1
	350	2	1.5	0.5
	400	1.5	1	0.5
1	250	—	—	1
	300	—	—	0.5
	350	1.5	1	0.5
	400	1	0.5	0.5

can be explained by its location in the narrowest pores or at the most active adsorption centres, from where its removal is very difficult.

Table III gives the temperatures and times required for the analysis of packings with about 1, 5 and 10% Apiezon M, polyethylene glycol adipate and polyethylene glycol 6000. The values given represent the time (hours) for quantitative evaporation of the phase, and the dashes indicate that quantitative evaporation is impossible at the particular temperature.

Regardless of some differences in the experimental conditions used for the quantitative evaporation of the phases from the three packings, it is evident that heating at 400° for 2 h gives good results in all instances.

In order to show the accuracy of the method for the determination of stationary phases with low volatilities, six analyses of packings with 1, 5 and 10% Apiezon M, polyethylene glycol adipate and polyethylene glycol 6000 were carried out. It was found that the absolute error does not depend on the percentage of the stationary phase in the packing and its value is about $\pm$ 0.1% absolute.

The relative error, $(\Delta X/X) \cdot 100$, as would be expected, is highest when the content of the stationary phase is lowest. For packings with 10% of stationary phase the relative error is about 0.9–1.0%, for packings with 5% of stationary phase it is 2–3% and for those with 1% of stationary phase it is 9–10%.

In conclusion, it seems that this method can be used successfully for the determination of stationary phases with low volatilities.

REFERENCE

1 N. D. Petsev, R. N. Nikolov and A. Kostova, *J. Chromatogr.*, 93 (1974) 369.