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Note

A comparison of gas–liquid chromatographic retention indices on support-coated open tubular columns and on packed columns for a series of central nervous stimulant drugs

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Packed columns have long been used for the analysis of basic drugs and in recent years several workers have developed screening methods using such columns for drugs present in biological materials[1–4]. However, with the advent of support-coated open tubular columns (SCOT columns), the resolution of gas–liquid chromatographic (GLC) systems has been increased, without the loss of sensitivity and the increase of analysis time associated with the use of wall-coated open tubular columns (WCOT columns). In consequence, it is likely that the use of SCOT columns will increase in future years and some use has already been made of them in drug analysis[5]. We have therefore compared the retention properties of conventional packed columns with those of SCOT columns using a non-polar (Apiezon L) and a polar (Carbowax 20M) stationary phase to determine if the retention characteristics of the two types of columns are similar for stimulant drugs.

EXPERIMENTAL

The packed columns were: 2 m, 2% Apiezon L/5% KOH; and 1 m, 1% Carbowax 20M/5% KOH; both using Chromosorb G (acid washed, DMCS treated, 80–100 mesh) as the support and nitrogen as the carrier gas. Either Pye 104 or Perkin-Elmer F11 gas chromatographs, using flame ionization detectors were used isothermally in the range 110–160°.

The capillary columns have been fully described elsewhere[5]. Four columns were used each using the same support, *viz.* Universal B (120–150 mesh) with 10% lithium chloride as a binder. Two columns used 10% Carbowax 20M/10% KOH, one used 10% Apiezon L/10% KOH and the last used 10% Apiezon L/20% KOH as the stationary phase. Nitrogen was used as the carrier gas and a Varian Aerograph 1840-3 instrument, fitted with flame ionization detectors, was used at 150° and 190°.

Retention indices[6] were determined for eleven basic drugs on each column using 1-μg samples of the base or alkane in 1-μl solvent for the packed columns, and

submicrogram samples in 0.1 μl solvent for the SCOT columns. The mean retention index of at least two determinations was calculated for the packed columns, and for the SCOT columns the mean of values obtained at two temperatures on two columns was determined where possible. With some compounds separation was not achieved on SCOT columns at one of the quoted temperatures, and in such cases, the retention indices were thus calculated at the other temperature only.

RESULTS AND DISCUSSION

The retention indices of the eleven drugs examined are given in Table I. Day-to-day variations of the retention indices for each drug on the SCOT or packed column were not more than $\pm$ 10 from the mean value for the Apiezon L / KOH columns and not more than $\pm$ 25 for the Carbowax 20M / KOH columns, although the great majority of results were within $\pm$ 3. This is in good agreement with other workers[7–10] who also found that, using packed columns and standard conditions, retention indices were reproducible to within an error of a few units. Neither SCOT nor packed columns showed any advantage as far as reproducibility of results was concerned.

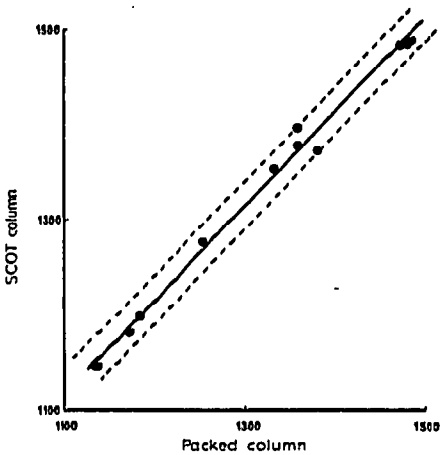
TABLE I

RETENTION INDICES FOR 11 CENTRAL NERVOUS SYSTEM STIMULANT DRUGS ON SCOT AND PACKED COLUMNS USING TWO STATIONARY PHASES

Drug	*Apiezon L / KOH*		*Carbowax 20M / KOH*	
	SCOT	*Packed*	*SCOT*	*Packed*
Amphetamine	1148	1136	1656	1475
Phentermine	1183	1170	1638	1467
Methylamphetamine	1200	1182	1623	1461
Mephentermine	1277	1255	1677	1502
Phenylpropanolamine	1354	1332	2254	2046
Nicotine	1373	1382	1901	1658
Ephedrine	1379	1360	2126	1965
Chlorphentermine	1401	1360	1933	1710
Nikethamide	1484	1474	2386	2142
Diethylpropion	1489	1487	2007	1845
Phenmetrazine	1485	1482	2158	1974

Good agreement was found when retention indices obtained on the packed or SCOT columns using Apiezon L / KOH as the stationary phase were compared (Table I). The packed column gave slightly lower indices, but in no case was this difference greater than 41. Fig. 1 shows the highly significant correlation between the retention indices obtained on the different types of column ($r = 0.995$) and no value was more than $\pm$ 26 from the regression line (standard error of estimate, $S_x = 12.8$). Although $\pm$ 26 is a greater error than would be expected using a single column with standard conditions, it is surprisingly low considering that different temperatures and different solid supports with very different stationary phase/support ratios were used in the two types of column.

The polar Carbowax 20M / KOH columns showed much greater differences in retention behaviour —the packed columns giving retention indices approx. 200 below



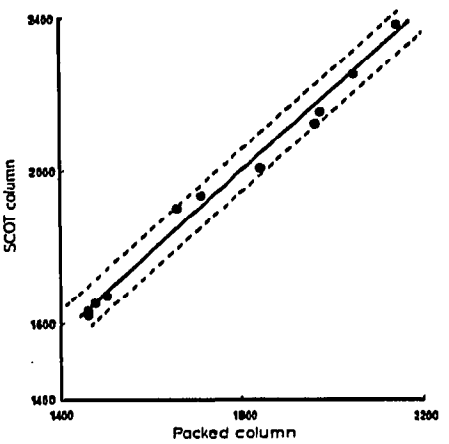


Fig. 1. The correlation of the retention indices of some stimulant drugs on a packed and a SCOT column using Apiezon L / KOH as the stationary phase. The solid line represents the regression equation and the dotted lines each represent two standard errors from the line (S_x = 12.8).

Fig. 2. The correlation of the retention indices of some stimulant drugs on a packed and a SCOT column using Carbowax 20M / KOH as the stationary phase. The solid line represents the regression equation and the dotted lines each represent two standard errors from the line (S_x = 28).

those obtained on the SCOT columns (Table I). However, in common with the Apiezon L / KOH columns, the results were highly correlated (r = 0.994) and no value was greater than ± 56 from the regression line (Fig. 2) (standard error of estimate, S_x = 28). Thus, although the individual differences in retention indices were as high as 244 (nikethamide), the data on one column could be derived from the other set of data using the regression equation to within an error of 56 (Fig. 2). This type of approach has also been successfully used in paper[11] and thin-layer chromatography[12], where a regression equation of the form $R_Fc = aR_F + b$ was used to increase the reproducibility of a single system and to convert data from one system to another.

Retention indices for basic drugs are thus in good agreement not only between like columns, but also between SCOT and packed columns and this applies to both polar and non-polar stationary phases. Consequently, once a new column (SCOT or packed) has been standardised by chromatographing two or more compounds, and the appropriate regression equation calculated, identifications in routine analysis can be assisted by the use of an appropriate retention index data bank without any of the limitations imposed by strict experimental conditions.

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