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CALCULATION OF INVARIANT RETENTION INDICES FOR A SERIES OF ALIPHATIC ALCOHOLS AND ACETATES BY CONSIDERATION OF ADSORPTION AT THE INTERFACE

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SUMMARY

A study of the gas-liquid chromatographic behaviour of series of aliphatic alcohols and acetates on the stationary phases OV-1, OV-17, OV-225 and diethylene glycol succinate has shown that the retention parameters of these compounds are greatly dependent on the temperature, type of solid support and concentration of the stationary phase used. On the basis of equations derived from the additive theory of retention, invariant logarithmic indices for 34 compounds using different sets of standards, *n*-alkanes, *n*-alkan-1-ols and corresponding acetates, were determined.

INTRODUCTION

Many long-chain saturated and unsaturated aliphatic alcohols and acetates have been shown to function as sex pheromones for the majority of lepidopterous insects^{1,2}. However, in spite of the relative simplicity of their chemical structures, the identification of natural pheromones remains difficult since only minute amounts of active compounds (usually 1-5 ng) are present in the complex mixtures produced by insects. Thus, the application of various chromatographic methods is essential for the structural elucidation of these compounds. In many cases, identification has been accomplished by comparing the retention time of the analyzed compound with those of available synthetic standards on columns with stationary phases of different polarities^{3,4}, or using tables of relative retention times⁵.

It is more appropriate to employ logarithmic retention indices as the chromatographic parameters for identification⁶. Tables of Kovats indices of series of saturated and monoenic alcohols and acetates on two stationary phases have been used for the component identification of sex pheromones of several Noctuidae species⁷. However, the extent of the application of this method is limited by its low resolving power and poor inter-laboratory reproducibility of Kovats indices, especially on polar stationary phases. One reason for this might be the adsorption at

the stationary phase-solid and stationary phase-gas interfaces which is known to influence the retention volume and, consequently, the retention indices*.

The purpose of the present work is to determine logarithmic retention indices for series of saturated, monoenic and dienic alcohols and corresponding acetates on four stationary phases of different polarities using several sets of standards: *n*-alkanes, *n*-alkan-1-01s and *n*-alkan-1-01 acetates. This paper also presents the calculation of retention indices which are independent of the type of solid support and of the concentration of stationary phase used.

EXPERIMENTAL

The saturated alcohols and acetates were obtained from commercial sources. Monoenic and dienic alcohols and acetates were prepared by E. R. Möttus and co-workers (Tartu State University) and by B. G. Kovalev and co-workers (Institute of Biological Control, Kishinev).

A Tsvet Model 5 gas chromatograph equipped with a flame ionization detector and standard 1 m × 3 mm I.D. glass columns was used to determine retention data. The solid supports were Chromaton N-AW (80-100 mesh) (Chemapol) and Chromosorb W AW DMCS (X0-100 mesh) (Applied Science Labs., State College, PA, U.S.A.). All stationary phases [silicone OV-1, OV-17, OV-225 and diethylene glycol succinate (DEGS)] (Applied Science Labs.) were coated in chloroform onto the solid support at 3, 6, 9, 12 and 15% (w/w). The gas hold-up time was determined according to the method of Peterson and Hirsch⁹.

RESULTS AND DISCUSSION

Selection of standards

The appropriate selection of standards is one of the basic conditions for the reliable determination of relative chromatographic parameters for investigated compounds. It has previously been shown by many scientists that *n*-alkanes are not always the best standards for the calculation of Kováts indices¹⁰⁻¹⁵.

On the other hand, to consider "automatically" the specific interactions between the sorbate, stationary phase and solid support and to minimize the temperature effect it is desirable to choose for the retention indices calculation standards whose chemical structures are as close to those of the analyzed sorbates as is possible. However, as shown by Berezkin¹⁶ and Soják *et al.*¹⁷, for the determination of the adsorption contribution to retention it is necessary to use standards whose retention is mainly determined by their solubility in the stationary phase. For non-polar and medium polarity stationary phases, *n*-alkanes meet this requirement; for polar ones, *n*-alkan-1-01s and/or the corresponding acetates.

Taking into consideration these facts as well as that most published retention data are presented in the form of Kováts (alkanic) indices, we investigated the possibility of using all sets of standards on stationary phases of different polarities. Table I lists the indices of 8,10-dodecadien-1-ol selected as a model compound relative to alkanes and alcohols, on four stationary phases on columns with different loadings on the solid support. As is seen, the use of *n*-alkan-1-ols for the retention indices calculation on OV-1 and OV-17 is reasonable because the parameters obtained (al-

TABLE I

KOVÁTS INDICES AND ALCOHOLIC INDICES (IN PARENTHESES) FOR **8,10-DODECADIEN-1-OL** ON COLUMNS WITH DIFFERENT CONCENTRATIONS OF STATIONARY PHASES ON THE SOLID SUPPORT

Experimental conditions: solid support, Chromaton N-AW (80–100 mesh); column temperature, 130°C.

Stationary phase	Stationary phase concentration on solid support (%)				
	3	6	9	12	15
DEGS	2043 (1434)	2098 (1448)	2166 (1474)	2300 (1510)	2310 (1515)
ov-225	1970 (1362)	1969 (1360)	1968 (1357)	1967 (1355)	1967 (1355)
ov-17	1751 (1331)	1716 (1322)	1683 (1310)	1678 (1309)	1676 (1308)
OV-1	1515 (1244)	1503 (1242)	1488 (1238)	1482 (1234)	1480 (1232)

coholic indices) are much less dependent on the stationary phase concentration on the solid support than alkanic (**Kováts**) indices. We suggest that on the non-polar and medium polarity stationary phases the main contribution to **sorbate** adsorption at the interphase boundaries is that from alcoholic functional groups, and the use of **n-alkan-1-ols** as standards for the calculation of retention indices of unsaturated alcohols permits a large measure of compensation for adsorption effects.

On OV-225, both alcoholic and alkanic indices showed a weak dependency on the stationary phase concentration, and consequently both sets of standards can be used.

The use of n-alkanes as standards for the retention indices calculation on the highly polar stationary phase DEGS seems not to be appropriate for the following reasons:

- (1) significant peak asymmetry of n-alkanes due to their poor solubility complicates the precise measurement of their retention times;
- (2) the use of long-chain n-alkanes (C_{20} – C_{27}) with high boiling points requires a significant increase of the evaporator temperature that may cause undesirable chemical transformations of the investigated compounds;
- (3) the violation of the linear relationship between the logarithm of the retention time and the carbon chain length as well as the significant spread of alkanic indices as a function of the DEGS concentration on the solid support (cu. 270 index units for **8,10-dodecadien-1-ol**) greatly increase the error in the determination of invariant retention parameters.

Taking into consideration these facts, we determined only alcoholic and **acetic** indices for DEGS, but on the other stationary phases all types of logarithmic retention indices were calculated.

Selection of the equation for the calculation of invariant retention indices

Earlier, several equations were proposed for the determination of the adsorption contribution to the retention parameters and for the calculation of invariant retention indices^{8,18}

$$I = I_0 + \lambda/V_L \quad (1)$$

$$I = I_0 + a/P_L \quad (2)$$

$$Z = I_0 + b/k_s \quad (3)$$

where I = logarithmic retention index of the sorbate, I_0 = invariant retention index, V_L = stationary phase volume in the column, P_L = percentage of stationary phase on the solid support, k_s = capacity factor for the reference compound the retention of which is mainly determined by its solubility in the stationary phase, I , a and b = constants. Eqns. 2 and 3 are more convenient for practical purposes because of experimental problems in the measurement of V_L .

Table II shows the results of comparative calculations of invariant retention indices for two model compounds and two types of solid support on the basis of eqns. 2 and 3. It follows that it is more preferable to use eqn. 3 rather than eqn. 2 for such calculations since the latter results in higher errors in the determination of the invariant parameters and, moreover, in some cases the violation of the linear relationship was observed (eqn. 2 was not valid). This appears to be due to the low precision of the P_L determination because of stationary phase losses during the coating procedure. On the contrary, the k_s value can be determined experimentally with a high degree of accuracy. Furthermore, it should be noted that the use of k_s permits the calculation of invariant retention indices with rather small errors also in capillary chromatography^{19,20}. The comparison of the obtained I_0 values (see Table II) showed that they were equal within the experimental error and independent of the type of solid support used.

Calculation of invariant retention indices for series of aliphatic alcohols and acetates

Tables III-VI show the invariant values of alkanic, alcoholic and acetatic indices for 34 aliphatic alcohols and acetates with different degrees of unsaturation.

TABLE II

STATISTICAL PARAMETERS FOR THE RELATIONSHIPS $I = f(1/P_L)$ AND $I = f(1/k_s)$ AND INVARIANT ALCOHOLIC INDICES, I_0 , FOR 8-DODECEN-1-OL AND 7,9-DODECADIEN-1-OL

Experimental conditions: stationary phase, DEGS, solid supports, Chromaton N-AW (80-100 mesh) and Chromosorb W AW DMCS (80-100 mesh). Boundary value of linear correlation coefficient, $r_{\text{theor}} = 0.878$ ($n = 5$). $\varepsilon_{0.95}$ is 95% confidence interval.

Compound	Solid support	Argument!	$I_0 \pm \varepsilon_{0.95}$	Linear correlation coefficient, r_{exp}
(1) 7,9-Dodecadien-1-ol	Chromaton	$1/P_L$	1516 ± 46	-0.880
	Chromaton	$1/k_s$	1537 ± 8	-0.997
	Chromosorb	$1/P_L$	1528 ± 26	-0.920
	Chromosorb	$1/k_s$	1533 ± 6	-0.996
(2) 8-Dodecen-1-ol	Chromaton	$1/P_L$	1282 ± 28	-0.859
	Chromaton	$1/k_s$	1290 ± 4	-0.998
	Chromosorb	$1/P_L$	1287 ± 36	-0.820
	Chromosorb	$1/k_s$	1292 ± 8	~ 0.946

TABLE III

INVARIANT KOVÁTS INDICES AND ALCOHOLIC INDICES (IN PARENTHESES) OF SERIES OF SATURATED AND UNSATURATED ALCOHOLS ON STATIONARY PHASES OV-1 AND ov-17

Experimental conditions: temperature 130°C; solid support, Chromaton N-AW (80-100 mesh).

Compound	OV-1			OV-17		
	$I_0 \pm \varepsilon_{0.95}$	$b \cdot 10^3$	r_{exp}	$I_0 \pm \varepsilon_{0.95}$	$b \cdot 10^3$	r_{exp}
(1) Dodecan-1-ol	1432 ± 12 (1200)	1.06	0.998	1535 ± 14 (1200)	1.98	0.998
(2) 7-Dodecen-1-ol	1420 ± 8 (1187 ± 6)	1.09	0.996	1547 ± 16 (1210 ± 6)	2.0	0.995
(3) 8-Dodecen-1-ol	1424 ± 12 (1189 ± 7)	1.10	0.997	1552 ± 11 (1214 ± 6)	2.03	0.997
(4) 9-Dodecen-1-ol	1428 ± 7 (1192 ± 7)	1.08	0.996	1556 ± 14 (1217 ± 6)	2.03	0.996
(5) 7,10-Dodecadien-1-ol	1429 ± 8 (1194 ± 6)	1.12	0.996	1580 ± 10 (1240 ± 8)	3.14	0.995
(6) 5,7-Dodecadien-1-ol	1465 ± 20 (1221 ± 12)	1.96	0.996	1608 ± 34 (1285 ± 16)	5.71	0.990
(7) 7,9-Dodecadien-1-ol	1470 ± 16 (1226 ± 14)	1.85	0.995	1612 ± 31 (1288 ± 15)	5.68	0.989
(8) 8,10-Dodecadien-1-ol	1475 ± 18 (1231 ± 23)	1.90	0.994	1616 ± 44 (1291 ± 23)	5.76	0.989
(9) Tetradecan-1-ol	1630 ± 16 (1400)	1.28	0.996	1733 ± 8 (1400)	3.12	0.996
(10) 9-Tetradecen-1-ol	1618 ± 12 (1382 ± 6)	1.25	0.994	1756 ± 8 (1415 ± 6)	3.18	0.995
(11) 11-Tetradecen-1-ol	1624 ± 14 (1386 ± 8)	1.24	0.992	1760 ± 12 (1418 ± 6)	3.12	0.995
(12) 9,12-Tetradecadien-1-ol	1626 ± 18 (1390 ± 8)	1.38	0.992	1784 ± 14 (1446 ± 12)	3.85	0.990
(13) 9,11-Tetradecadien-1-ol	1672 ± 24 (1426 ± 12)	1.98	0.990	1814 ± 22 (1492 ± 18)	5.98	0.986
(14) Hexadecan-1-ol	1824 ± 12 (1600)	2.51	0.989	1930 ± 20 (1600)	3.82	0.992
(15) 7-Hexadecen-1-ol	1804 ± 14 (1581 ± 8)	2.40	0.990	1939 ± 16 (1612 ± 8)	4.06	0.994
(16) 11-Hexadecen-1-ol	1814 ± 12 (1592 ± 8)	2.60	0.989	1944 ± 18 (1618 ± 8)	4.0	0.992
(17) 10,12-Hexadecadien-1-ol	1880 ± 20 (1635 ± 8)	3.10	0.985	2020 ± 28 (1695 ± 12)	5.72	0.990

These values were calculated on the basis of eqn. 3 by the least squares method. The adsorption contribution to the retention of a given compound can be evaluated by the slope b of eqn. 3. As seen from the obtained data, the b values are similar for members of the same homologous series and, consequently, can also be used for identification purposes.

The obtained invariant retention indices can be used for group identification of organic compounds by construction of characteristic zones similar to those proposed earlier²¹ and/or individual identification by examination of the tables. In the

TABLE IV

INVARIANT KOVÁTS INDICES AND ACETATIC INDICES (IN PARENTHESES) OF SERIES OF SATURATED AND UNSATURATED ACETATES ON STATIONARY PHASES OV-1 AND ov-17

Experimental conditions as in Table III.

Compound (acetate of)	OV-1		ov-17	
	$I_0 \pm \varepsilon_{0,95}$	$b \cdot 10^3 r_{exp}$	$I_0 \pm \varepsilon_{0,95}$	$b \cdot 10^3 r_{exp}$
(1) Dodecan-1-ol	1576 ± 15 (1200)	0.56 0.997	1635 ± 7 (1200)	0.77 0.971
(2) 7-Dodecen-1-ol	1560 ± 12 (1185 ± 6)	0.62 0	1695 ± 10 (1212 ± 8)	0.96 0
(3) 8-Dodecen-1-ol	1564 ± 10 (1187 ± 6)	0.60 0	1696 ± 12 (1214 ± 6)	0.99 0
(4) 9-Dodecen-1-ol	1568 ± 14 (1192 ± 6)	0.62 0	1699 ± 10 (1216 ± 6)	0.98 0
(5) 7,10-Dodecadien-1-ol	1570 ± 12 (1195 ± 8)	0.68 0	1725 ± 12 (1236 ± 12)	0.99 0.42
(6) 5,7-Dodecadien-1-ol	1598 ± 16 (1216 ± 8)	1.38 0.60	1786 ± 24 (1298 ± 12)	3.36 1.18
(7) 7,9-Dodecadien-1-ol	1602 ± 14 (1218 ± 10)	1.30 0.55	1790 ± 18 (1302 ± 8)	2.26 1.16
(8) 8,10-Dodecadien-1-ol	1608 ± 16 (1220 ± 12)	1.32 0.58	1795 ± 18 (1306 ± 8)	3.25 1.14
(9) Tetradecan-1-ol	1780 ± 14 (1400)	0.75 0.993	1832 ± 10 (1400)	0.92 0.988
(10) 9-Tetradecen-1-ol	1765 ± 21 (1385 ± 8)	0.84 0	1890 ± 14 (1412 ± 10)	1.34 0
(11) 11-Tetradecen-1-ol	1770 ± 15 (1392 ± 6)	0.87 0	1895 ± 12 (1418 ± 12)	1.32 0
(12) 9,12-Tetradecadien-1-ol	1772 ± 14 (1395 ± 8)	0.89 0	1916 ± 14 (1438 ± 10)	1.89 0.52
(13) 9,11-Tetradecadien-1-ol	1810 ± 18 (1425 ± 9)	1.48 0.68	1990 ± 18 (1504 ± 12)	3.16 1.28
(14) Hexadecan-1-ol	1976 ± 16 (1600)	0.95 0.992	2030 ± 15 (1600)	1.48 0.992
(15) 7-Hexadecen-1-ol	1960 ± 14 (1584 ± 8)	0.98 0	2082 ± 16 (1610 ± 8)	1.92 0
(16) 11-Hexadecen-1-ol	1973 ± 12 (1590 ± 8)	1.02 0	2089 ± 16 (1620 ± 12)	1.90 0
(17) 10,12-Hexadecadien-1-ol	2012 ± 18 (1620 ± 12)	1.95 0.65	2200 ± 19 (1710 ± 10)	3.20 1.28

latter case it is appropriate to use alcoholic and acetatic retention indices since the accuracy of their determination is significantly higher than for alkanic indices.

CONCLUSIONS

The most suitable equation for the calculation of invariant retention indices was shown to be that which linearly relates the retention index and the reciprocal of the capacity factor of the reference compound. On the basis of this equation the

TABLE V

INVARIANT KOVÁTS INDICES AND ALCOHOLIC INDICES (IN PARENTHESES) OF SERIES OF SATURATED AND UNSATURATED ALIPHATIC ALCOHOLS ON STATIONARY PHASES OV-225 AND DEGS

Experimental conditions as in Table III.

Compound	ov-225		DEGS		
	$I_0 \pm \epsilon_{0.95}$	$b \cdot 10^3 r_{exp}$	$I_0 \pm \epsilon_{0.95}$	$b \cdot 10^3 r_{exp}$	
(1) Dodecan-1-ol	1799 ± 10 (1200)	0.42	0.978	(1200)	
(2) 7-Dodecen-1-ol	1823 ± 12 (1222 ± 6)	0.44	0.977	(1286 ± 6)	-0.60 -0.997
(3) 8-Dodecen-1-ol	1829 ± 10 (1226 ± 5)	0.44	0.978	(1290 ± 4)	-0.64 -0.998
(4) 9-Dodecen-1-ol	1834 ± 10 (1230 ± 5)	0.44	0.978	(1294 ± 4)	-0.65 ±0.997
(5) 7,10-Dodecadien-1-ol	1910 ± 6 (1292 ± 4)	0.52	0.980	(1395 ± 8)	-0.89 -0.996
(6) 5,7-Dodecadien-1-ol	1954 ± 8 (1340 ± 8)	0.10	0.996	(1531 ± 10)	-1.90 ± 0.996
(7) 7,9-Dodecadien-1-ol	1960 ± 6 (1346 ± 8)	0.12	0.993	(1537 ± 8)	-1.87 ±0.997
(8) 8,10-Dodecadien-1-ol	1965 ± 4 (1350 ± 8)	0.10	0.986	(1541 ± 10)	± 1.88 -0.997
(9) Tetradecan-1-ol	2000 ± 12 (1400)	0.48	0.984	(1400)	
(10) 9-Tetradecen-1-ol	2028 ± 10 (1431 ± 8)	0.64	0.986	(1492 ± 10)	-1.03 -0.996
(11) 11-Tetradecen-1-ol	2034 ± 12 (1437 ± 6)	0.64	0.986	(1498 ± 12)	-1.05 -0.993
(12) 9,12-Tetradecadien-1-ol	2114 ± 9 (1495 ± 6)	0.85	0.988	(1598 ± 8)	-1.25 -0.990
(13) 9,11-Tetradecadien-1-ol	2157 ± 12 (1536 ± 10)	0.26	0.994	(1742 ± 10)	-2.31 -0.989
(14) Hexadecan-1-ol	2204 ± 10 (1600)	0.47	0.990	(1600)	
(15) 7-Hexadecen-1-ol	2225 ± 12 (1620 ± 8)	0.56	0.989	(1681 ± 10)	-0.79 -0.985
(16) 11-Hexadecen-1-ol	2231 ± 10 (1625 ± 7)	0.61	0.987	(1687 ± 12)	-0.79 -0.984
(17) 10,12-Hexadecadien-1-ol	2365 ± 12 (1758 ± 8)	0.24	0.996	(1948 ± 8)	-2.32 -0.985

invariant alkanic, alcoholic and acetatic indices for insect sex pheromones and their structural analogue belonging to series of saturated and unsaturated alcohols and corresponding acetates were calculated. The obtained parameters were independent of the stationary phase concentration and type of solid support used.

The homologous series of *n*-alkanic-1-ols and corresponding acetates were found to be the most suitable standards for these calculations with the minimum errors.

TABLE VI

INVARIANT KOVÁTS INDICES AND ACETATIC INDICES (IN PARENTHESES) OF SERIES OF SATURATED AND UNSATURATED ACETATES ON STATIONARY PHASES OV-225 AND DEGS

Experimental conditions as in Table III.

Acetates of	O v-225			DEGS		
	$I_0 \pm \varepsilon_{0.95}$	b	$10^3 r_{exp}$	$I_0 \pm \varepsilon_{0.95}$	b	$10^3 r_{exp}$
(1) Dodecan-1-ol	1841 ± 1 (1200)	0	0.999	(1200)		
(2) 7-Dodecen-1-ol	1860 ± 5 (1220 ± 2)	0	0.998	(1285 ± 8)	-0.85	-0.984
(3) 8-Dodecen-1-ol	1864 ± 4 (1222 ± 2)	0	0.998	(1289 ± 10)	-0.83	-0.984
(4) 9-Dodecen-1-ol	1871 ± 6 (1227 ± 3)	0	0.997			
(5) 7,10-Dodecadien-1-ol	1938 ± 12 (1290 ± 4)	0	0.996	(1295 ± 8)	-0.83	-0.985
(6) 5,7-Dodecadien-1-ol	1982 ± 4 (1340 ± 1)	0	0.999	(1390 ± 10)	-0.98	-0.986
(7) 7,9-Dodecadien-1-ol	1987 ± 1 (1342 ± 1)	0	0.999	(1510 ± 14)	-1.50	-0.982
(8) 8,10-Dodecadien-1-ol	1991 ± 2 (1346 ± 1)	0	0.999	(1516 ± 20)	-1.48	-0.981
(9) Tetradecan-1-ol	2036 ± 4 (1400)	0	0.996	(1520 ± 14)	-1.48	-0.986
(10) 9-Tetradecen-1-ol	2065 ± 2 (1424 ± 1)	0	0.998	(1400)		
(11) 11-Tetradecen-1-ol	2071 ± 2 (1429 ± 2)	0	0.999	(1489 ± 10)	-0.95	-0.989
(12) 9,12-Tetradecadien-1-ol	2130 ± 6 (1487 ± 2)	0	0.999	(1496 ± 12)	-0.96	-0.989
(13) 9,11-Tetradecadien-1-ol	2187 ± 4 (1544 ± 2)	0	0.996	(1600 ± 10)	-1.15	-0.990
(14) Hexadecan-1-ol	2238 ± 2 (1600)	0	0.999	(1715 ± 12)	-2.21	-0.980
(15) 7-Hexadecen-1-ol	2258 ± 2 (1620 ± 1)	0	0.999	(1600)		
(16) 11-Hexadecen-1-ol	2266 ± 2 (1626 ± 2)	0	0.999	(1682 ± 10)	-0.96	-0.990
(17) 10,12-Hexadecadien-1-ol	2398 ± 5 (1746 ± 2)	0	0.997	(1690 ± 12)	-0.98	-0.990
		0	0.998	(1940 ± 10)	-2.56	-0.987

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REFERENCES

- 1 M. Jacobson, *Insect Sex Attractants*, Academic Press, New York, 1972, p. 382.
- 2 T. Ando, S. Yoshida and S. Tatsuki, *Agr. Biol. Chem.*, 41 (1977) 1485.
- 3 M. Beroza, *J. Chromatogr. Sci.*, 13 (1975) 314.
- 4 W. L. Roelofs, A. Comeau and G. Milicevic, *Science*, 174 (1971) 297.
- 5 L. M. McDonald and J. Weatherston, *J. Chromatogr.*, 118 (1976) 195.
- 6 E. Kováts, *Helv. Chim. Acta*, 41 (1958) 1915.
- 7 S. F. Nedopekina, B. G. Kovalev and A. N. Kost, *Khim. Pri. Soedin.*, 4 (1981) 501.
- 8 V. G. Berezkin, *J. Chromatogr.*, 91 (1974) 559.
- 9 M. L. Peterson and J. Hirsch, *J. Lipid Res.*, 1 (1959) 132.
- 10 J. K. Haken, *J. Chromatogr.*, 99 (1974) 329.
- 11 W. A. Aue and V. Paramasigamani, *J. Chromatogr.*, 166 (1978) 253.
- 12 A. Grobler, *J. Chromatogr. Sci.*, 10 (1972) 128.
- 13 R. G. Ackman, *J. Chromatogr. Sci.*, 10 (1972) 535.
- 14 S. J. Hawkes, *J. Chromatogr. Sci.*, 10 (1972) 536.
- 15 L. J. Lorenz and L. B. Rogers, *Anal. Chem.*, 44 (1971) 1593.
- 16 V. G. Berezkin, *J. Chromatogr.*, 159 (1978) 359.
- 17 L. Soják, V. Berezkin and J. Janák, *J. Chromatogr.*, 209 (1981) 15.
- 18 J. Rijks, K. Leuten, C. Cramers and V. G. Berezkin, *Zh. Anal. Khim.*, 29 (1974) 858.
- 19 J. Krupčík, E. Matisová, J. Garaj, L. Soják and V. G. Berezkin, *Chromatographia*, 16 (1982) 166.
- 20 E. Matisová, J. Krupčík and J. Garaj, *Chromatographia*, 16 (1982) 169.
- 21 V. G. Berezkin and J. J. Walraven, *Zh. Anal. Khim.*, 26 (1971) 1442.