*Journal of Chromatography*, 271 (1983) 213–307
*Chromatographic Reviews*
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHREV 170

# TWENTY-FIFTH ANNIVERSARY OF THE RETENTION INDEX SYSTEM IN GAS-LIQUID CHROMATOGRAPHY

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(Received March 16th, 1983)

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## 1. HISTORY

> *"If somebody wants to break a new path,*
> *he has to know the old ones, to get there,*
> *where he wants to begin the new."*
> L. ERDEY*

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* L. Erdey (1910–70) was Professor of the Institute for General and Analytical Chemistry (Technical University, Budapest, Hungary) for 20 years.

In the early 1950s, at the advent of the revolution in science and technology, one of the greatest successes in the field of analytical chemistry was the discovery of gas–liquid chromatography (GLC) by James and Martin[1381].

During the past 30 years, the number of papers published on GLC and its applications has exceeded 70,000 However, the style of publication of retention data, in spite of much effort, is still not uniform. Published retention data are given in many forms, and therefore the usefulness of these data in different laboratories is limited. However, the elaboration of the retention index concept by Kováts[1] in 1958 certainly aroused considerable interest.

The Kováts retention index[1] solves the problem of the uniform expression of retention data The retention index is the only retention value in GLC in which the two fundamental quantities, *viz* , the relative retention and the specific retention volume, are united. Moreover, a series of explicit relationships between the retention indices and the physico-chemical quantities related to GLC have been developed Similarly, numerous linear relationships between the retention index values determined under different conditions and other fundamental quantities such as boiling point, molecular weight, carbon number, etc have been derived

Fortunately, many of the retention data previously published in other forms can be converted into retention indices, so they can be used in different laboratories.

In spite of the elaboration of the retention index concept, the method was not extensively applied at first. This lack of success can be explained by the fact that Kováts' paper appeared in German, whereas owing to the technical revolution in the 1950s most scientific papers were published in English. The spread of GLC began after 1958 and from that time on numerous papers appeared dealing with gas chromatography, but in this stream of information only a few workers, such as Stadler and Oberhànsli[3] and Wehrli and Kováts[4] paid attention to Kováts results[1,4,6,25].

Meanwhile, an increasing number of researchers began to work in Switzerland on the determination of retention indices[8, 17, 23, 24, 26, 33, 34], and besides Kováts and his co-workers, a second group of scientists was formed, headed by Gàumann[24,33,34]. In 1959, Peterson and Hirsch published new results[2] on gas holdup time, these were aided by Gold's publication[12], which gave the data needed for the calculation of retention indices.

Scott's recapitulatory paper[7] in 1961 aroused general interest in the retention index concept. At the same time, work began on various theoretical and practical questions of the retention index concept by Chovin[5], Baron and Maume[9], Ferrand[11], Huguet[13], Landault and Guiochon[15], Pourchez *et al.*[16] and Zulaica and co-workers[18,19], who established the basis of the French school

In Britain, Clayton[10], Evans[22,41] and Preston *et al*[31] did pioneering work, as was done in Germany by Klouven and Ter Heide[14], Kuhn and Egge[27] and Schomburg[32] and in Austria by Cremer and Nonn[39].

Among Japanese workers, Matukuma[28] was the first to discover the possibilities of retention indices and to begin extensive research work on the retention index of alkanes.

Van den Dool and Kratz[21] in 1963 described the determination and calculation of retention indices in temperature-programmed gas chromatography, and thus an important and broad field for the application of retention indices was discovered.

Regarding the prevalence of the retention index concept, 1964 was an important year. At the Second International Symposium on Gas Chromatography (Houston, TX, U.S.A ), Kováts summarized the most important questions and knowledge about retention indices. Subsequently, Keulemans discussed the various error sources in retention index determinations These lectures aroused such interest that Ettre published a paper40 recapitulating the various statements about the retention index system and the most important theoretical and practical considerations involved Ettre was thus among the first scientists to recognize the importance and perspectives of retention indices; incidentically, it was he who coined the expression "retention index system" (RIS) Ettre worked hard to ensure progress of the RIS concept in the U S.A One of his research programmes started with the detailed elucidation of theoretical and practical questions and the numerical determination of retention indices Scientific centres were formed in the U.S A [38, 44-48, 62, 68, 69, 71-75], in The Netherlands around Keulemans[63] and in Czechoslovakia[60,61] around Janák More new results were collected in Switzerland[47, 50, 79, 80], in France[30, 35-37, 42, 43, 46, 53, 57, 61, 67], in Austria[54,55], in Britain[58] and in Germany[48, 52, 89, 70, 77, 78] More life was put into the research work by Kováts' further publications[65,66], which recapitulated the latest results. Around this time papers appeared in Australia from Haken and co-workers[95] and in the Soviet Union from Golovnya *et al* [92] and Kudrayavtseva *et al.*[101]

Numerous research groups worked in the second half of the 1960s on the various questions of RIS and their results were published in more than 200 papers[81-284]

1969 was again an important year in the course of development and spreading of RIS, the Fifth Symposium on Separation Methods: Column Chromatography being held in Lausanne (Switzerland) At that time Kováts was a Professor at the École Polytechnique in Lausanne It is not surprising that numerous lectures at the above Symposium dealt with the theoretical and practical questions of RIS[285, 288, 330, 332-335, 349, 352, 363-365, 367, 370]. These lectures and the subsequent discussions promoted considerably the further development of RIS.

Although important discoveries and results were scarce in the 1970s, the fields of application of RIS widened[285-1242] Owing to the spread of desktop computers and programmable calculators, the number of researchers who applied RIS grew steadily. At the same time, research work and calculations were carried out on retention indices in connection with molecular structure This progress, although its rate was slower, than in the past, was characteristic of the early 1980s Nowadays, the basic program of the better reporting integrators is equipped with a calculation program for retention indices and work has been carried out on programs to examine the relationship between retention index and molecular structure.

To summarize the 25-year-old history of RIS, it can be stated that the earlier researchers threw light on almost all the main theoretical and practical questions

Theoretically, RIS fulfils the task of a universal data providing system. In practice this can be realized only if a data bank is available in which reliable retention indices are stored that can be easily retrieved by researchers in any gas chromatographic laboratory in the world by telephone link or satellite.

During this research work carried out in the last quarter of a century, striking results were obtained by individual researchers that had a major influence on the progress of RIS Without trying to be exhaustive, the work of Rohrschneider[77, 111, [214–216, 272, 273, 351, 352, 358, 655, 656], McReynolds[103, 344] and Loewenguth and Tourres[160, 161, 204 206, 225, 236] should be mentioned, together with a paper by Hively and Hinton[198].

The groups of scientists interested in this topic grew over the years and important research centres were formed, where the various problems of RIS were examined. Within the space available here, the following names of researchers working in this field can be mentioned D'Amato[236, 581, 582, 1087, 1172, 1354], Andersen[231, 289, 290, 980, 981], Ashes[376, 377, 574, 575, 683, 684, 879, 982, 983, 1073, 1244], Avots[378, 468, 856, 1053, 1054, 1144], Bardou[473, 692–694, 881, 1028], Berezkin[81, 296, 382, 598, 688–691, 1078, 1346], Berthou[473, 692–694, 881 1028, 1182 1314], Bonastre[125, 169–172, 407, 1015, 1016, 1188], Brooks[232, 292, 386–388 469, 579, 697, 713, 807, 899, 900, 963, 1002], Buryan[737, 989, 990, 1085 1086, 1125, 1365], Castello[236, 390, 479 581–584, 991, 1087, 1172, 1354], Chastrette[480, 585–588, 698–700, 884, 1088, 1089], Chovin[5, 37, 85, 180, 373], Chrétien[704, 794, 795, 885, 994, 1090, 1204, 1249, 1264, 1320, 1331], Dabrio[854, 855, 960, 961, 1048], Dimov[239, 302, 799, 887, 998, 1037, 1091, 1170, 1175, 1176, 1279, 1339, 1356], Druilhe[416, 417, 698, 752, 753, 818, 913, 1022], Dubois[704, 885, 994, 1090, 1249, 1320], Eisen[101, 181, 304, 395, 419, 433, 437, 490, 531, 533, 534, 650, 846, 847, 949–951, 1042, 1043], Engewald[708, 709, 820, 863, 890–892, 1003, 1093, 1151, 1162, 1179 1342], Erdey[305, 306, 308, 363, 396], Ettre[40, 87, 88 130, 131, 182, 595, 710, 711, 802, 803, 1252], Evans[22, 41 58 89, 132, 133, 183, 240, 397, 491, 552, 596, 893, 1094, 1180, 1253, 1357], Garzó[134 398, 399, 681, 682, 695, 1034], Gäumann[24, 33, 34, 578, 599, 651], Golovnya[92, 136–139, 187–189, 245–247, 312–315, 402–405, 494 497, 600, 601, 714, 809–811, 901–907, 976, 977, 1012–1014, 1101–1104, 1184–1187, 1257, 1258], Grenier[125, 169–172, 407, 792, 1015, 1016, 1188], Gröbler[501, 715, 1189, 1190, 1330], Guiochon[15, 18, 19, 35, 36, 42, 43, 46 51, 57, 94, 120, 149 311, 373, 410, 759, 1204, 1335, 1344, 1345, 1364, 1372], Haken[95, 243, 244, 286, 287, 318 376, 377, 411, 502, 503, 574, 575, 607, 683, 684, 717, 813–815, 879, 982, 983, 1019, 1020, 1073, 1146, 1191–1193, 1241, 1260, 1305, 1306, 1331, 1332, 1355], Hedin[68, 104, 140, 210, 282, 321, 414, 415, 448, 449, 505, 608, 609, 742, 743, 781, 817, 835, 836, 938], Heintz[416, 417, 698, 752, 753, 818, 913 1022], Hrivňák[142, 275, 507, 545–547, 663, 737, 770, 823, 862, 922, 923], Howery[567–569, 721, 722], Janák[60, 61, 98, 99, 112, 141, 200, 340, 371, 462, 507, 546–550, 566, 615, 642, 663, 770, 825, 922, 1057–1059, 1149, 1235, 1296, 1297, 1346], Kaiser[253, 325–328, 420–422, 705, 726, 1025, 1108, 1200], Karasek[329, 423, 511, 512, 1080, 1110, 1111], Kiselev[299, 709, 1100, 1201, 1333], Kováts[1, 6, 17, 23, 25, 26, 47, 64–66, 107, 159, 203, 257, 330, 597, 614, 620, 953 1183, 1226], Krupčík[256, 331, 515, 547, 549, 771, 823, 824, 922–924, 927, 1205, 1215, 1234, 1267, 1294, 1297, 1335, 1364], Ladon[227, 333, 334, 426, 624, 732], Leary[628, 629, 736, 816, 831], Lefort[16, 29, 416, 417, 698, 752, 753, 818, 913, 1022], Macák[737, 989, 990, 1085, 1086, 1125, 1365], Mázor[209, 278, 294, 341, 342, 348, 360], Minyard[68, 104, 153, 210, 282], Mitooka[430, 519–526], Mitra[211, 217, 264, 346, 347, 354, 527, 538, 634, 635, 657, 658, 740, 741, 1216 1337, 1369], Mody[742, 743, 835, 836, 938], Moffat[636, 744, 837, 1287, 1312, 1377], Novák[106, 154, 371, 642, 748, 825, 990, 1370], Onuska[644, 645, 750, 839, 943, 1222], Orav[650, 846, 949–951, 1042, 1043], Pacáková[435, 646, 751 819, 937, 1035, 1127, 1128 1207, 1237, 1324 1347], Palm[70, 108, 267–269, 367], Papazova[998, 1037, 1091, 1175, 1176, 1279, 1339], Parcher[570, 679, 816, 841, 945, 1038, 1281], Peetre[647, 706, 707, 754–756], Preston[31 72, 73, 109, 436, 632], Rang[101, 181, 437, 490, 533, 534, 650, 846, 847, 949–951, 1042, 1043], Rijks[300, 301, 653, 761, 762, 771, 964, 1057–1059, 1249, 1295, 1302], Saha[186, 211, 217, 264, 346, 347, 354, 527, 538, 634, 635, 657, 658, 1045, 1134], Sakamoto[765, 851–853, 959, 1046, 1047, 1371], Sanz[854, 855, 960, 961, 1048], Schomburg[32, 49, 113–115, 157, 193, 220, 221, 356, 439, 440, 659, 660, 701, 766, 1050, 1051, 1230], Shats[1053, 1054, 1144, 1231, 1232, 1292, 1373], Singliar[256, 274, 544, 767, 824], Soják[275, 358, 507, 545–550, 592, 615, 663, 688, 770, 771, 862, 964, 1057–1059 1078, 1149, 1234, 1235, 1249, 1294–1297, 1346, 1375], Strickler[17, 50, 65, 79, 117, 159], Tesařik[462, 548, 780, 823], Thompson[68, 104, 140, 210, 282, 448, 449, 781, 817], Tóth[281, 450–452, 517, 783–785], Vergnaud[162, 309, 310, 317, 408, 723], Vernon[454, 455, 518, 673, 674, 871–873, 1069, 1155–1157, 1193, 1261, 1303], Vigdergauz[284, 339, 456, 457, 592, 643, 671, 676, 677, 857, 894, 934, 970, 992, 1113, 1114, 1159, 1160], Walraven[63, 226, 227, 370, 382], Weiner[567–570, 679, 722], Welsch[708, 709, 890, 1003, 1151, 1162] and Zulaica[18, 19, 35, 36, 120]

In the following, the main theoretical and practical questions of RIS will be summarized First, the nomenclature and the most important definitions and relationships will be recapitulated

## 2 NOMENCLATURE, SYMBOLS AND DEFINITIONS

During the last 25 years, about 1500 papers have been published in this field of GLC. In these papers numerous symbols were used and it is therefore advisable to define them.

The retention index system (RIS) includes the basic equation elaborated by Kováts[1], the various equations derived from the Kováts' equation using mathematical transformations, the entirety of retention index data determined with the help of the previously mentioned equations and the theoretical and practical relationships achieved with the help of retention indices.

The RIS elaborated by Kováts[1] uses the homologous series of *n*-alkanes as a standard; the retention index expresses the retention of a compound relative to *n*-alkanes analysed under identical, isothermal conditions. The retention index of a particular compound can be defined as the carbon number of a hypothetical *n*-alkane multiplied by 100 which would have exactly the same retention characteristics (adjusted retention time, adjusted, specific or net retention volume) as the compound of interest

For example, if the retention index of benzene on squalane at 75°C is 644 index units (i.u.), a hypothetical *n*-alkane with 6.44 carbon atoms in its molecule would have the same retention characteristics as benzene. It should be noted that the retention index is a dimensionless quantity[956].

The retention index, of a compound is given by, for example,

$$I^{\text{squalane}}_{\text{benzene}} \ (75^\circ\text{C}) = 644 \text{ i.u.} \tag{1}$$

*i.e* , the sample, the stationary phase and the column temperature are specified. The fundamental equation for the retention index is

$$I^{\text{st ph}}_{\text{s}} \ (T) = 100 \left[ \frac{\log X_s - \log X_z}{\log X_{(z+1)} - \log X_z} + z \right] \tag{2}$$

where $I$ = isothermal retention index (i.u ); s = the compound of interest, st ph. = stationary phase; $z$ and $z+1$ = *n*-alkanes with $z$ and $z+1$ carbon numbers, respectively, and $X$ = retention value used in the calculation

In the most rigorous treatment, values of the specific ($V_g$)[1382] or net retention volumes ($V_N$) should be substituted for $X$.

In classical GLC, $T$, $p_i$ and $F$ are constants, where $T$ = column temperature (°C or °K), $p_i$ = inlet pressure of carrier gas and $F$ = flow-rate of carrier gas. Hence the net retention volume (and therefore also the specific retention volume) of every component is constant; if the column temperature is constant, then the adjusted retention volumes ($V'_R$) or adjusted retention times ($t'_R$) can serve equally well.

It is assumed that

$$X_z \leqslant X_s \leqslant X_{(z+1)} \tag{3}$$

Eqn. 3 indicates that in some instances, one of the reference *n*-alkanes may elute together with the compound of interest.

The calculation of the retention index value, from the experimental data can be illustrated by the following example. Retention time of cyclopentane (cyc$C_5$) at 50°C on Apolane-87 (AP-87) stationary phase = 333.3 sec; gas holdup time = 221.3 sec; retention time of *n*-pentane = 275.4 sec; retention time of *n*-hexane = 364.7 sec. Then,

$$I^{\mathrm{AP\text{-}87}}_{\mathrm{cycC_5}}\,(50\,^{\circ}\mathrm{C}) = 100\left[\frac{\log(333.3-221.3)-\log(275.4-221.3)}{\log(364.7-221.3)-\log(275.4-221.3)}+5\right] = 574.7\ \mathrm{i.u.} \tag{4}$$

Eqn. 2 can further be generalized for the case where two non-consecutive *n*-alkanes are used for the calculation

$$I^{\mathrm{st\,ph}}_{s}\,(T) = 100n\left[\frac{\log X_s-\log X_z}{\log X_{(z+n)}-\log X_z}\right]+100\,z \tag{5}$$

where $n$ = the difference in the carbon numbers of the two *n*-alkanes, for example, if the two *n*-alkanes are *n*-hexane and *n*-octane, then $n = 8 - 6 = 2$. Thus, if the retention time of *n*-octane is 1228.8 sec, then

$$I^{\mathrm{AP\text{-}87}}_{\mathrm{cycC_5}}\,(50\,^{\circ}\mathrm{C}) = 100\cdot 2\left[\frac{\log(333.3-221.3)-\log(364.7-221.3)}{\log(1228.8-221.3)-\log(364.7-221.3)}\right]+100\cdot 6$$
$$= 574.7\ \mathrm{i.u.} \tag{6}$$

If possible, one should avoid using *n*-alkanes more distant from one another, *i.e.* with $n > 2$, because of the possibility of non-linearity of the relationship

$$\log t'_R\,(z) = bz + a \tag{7}$$

where $b$ is the slope, $z$ is the carbon number of the members of the *n*-alkane homologous series and $a$ is intercept on the ordinate. Although linearity does not always apply over the whole range, it is accepted if $z \geqslant 7$

The retention indices of the *n*-alkanes used as reference compounds are bound defined as follows for any stationary phase and at any column temperature:

$$I(n\text{-P}_z) = 100\,z \qquad (8)$$

where $n$-$P_z$ represents an $n$-alkane with $z$ carbon atoms. Thus, for example, the retention index of $n$-pentane is $100 \cdot 5 = 500$ and that of $n$-decane is $100 \quad 10 = 1000$ on any stationary phase at any column temperature Hydrogen is taken as the zero point of the $n$-alkane scale. This is justified if we substitute $z = 0$ into the general formula of the $n$-alkanes, $C_zH_{2z+2}$.

In classical GLC, where the column temperature and the carrier gas inlet pressure and flow-rate are kept constant, numerous simplifications can be made to the fundamental equation For example, according to Kováts and Weiss[66], eqn. 2 can be written in terms of chemical potential, $\mu$

$$I_s^{st\,ph}(T) = 100\left[-\frac{\Delta\mu^0(s) - \Delta\mu^0(z)}{\Delta\mu^0(z+1) - \Delta\mu^0(z)}\right] + 100z \qquad (9)$$

where $\Delta\mu^0 = \mu^{st\,ph} - \mu^{gas\ phase}$ ($\mu$ = chemical potential)

During the last 25 years, authors used many different designations for the various terms, and we shall summarize the most important ones here to help formulate a uniform symbol system.

In addition to the individual retention index values, the differently formed retention index differences are of great significance because of their physico-chemical meaning Some of these are given below.

$$\Delta I(T) = I_s^p(T) - I_s^{Np}(T) \qquad (10)$$

where $\Delta I$ = difference between the retention indices of a single substance (s) measured on two different stationary phases (p and Np) at an identical, isothermal column temperature ($T$); p = polar stationary phase, Np = non-polar stationary phase, *e.g.*, squalane.

$$\delta I_s^{st\,ph} = I_s^{st.ph}(T_2) - I_s^{st\,ph}(T_1) \qquad (11)$$

where $\delta I$ = difference between the retention indices of a single substance (s) measured at two different, isothermal column temperatures ($T_1$ and $T_2$) on the same stationary phase (st ph.)

$$dI_{s(2)-s(1)}^{st\,ph}(T) = I_{s(2)}^{st\,ph}(T) - I_{s(1)}^{st\,ph}(T) \qquad (12)$$

where $dI$ = difference between the retention indices of two substances, s(1) and s(2), on the same stationary phase (st ph.) at an identical, isothermal column temperature ($T$)

These three types of retention index difference have significant practical importance The $\Delta I$ values can be approximately used for the calculation of individual relationships, to follow their variations, for the characterization of the polarity of stationary phases, to predict the values of retention data, etc. With the help of $\partial I$ values, the effect of column temperature on the retention index can be examined. If this relationship is linear, as it is in practice with an apolar stationary phase and an apolar substance, then the temperature coefficient of the retention index can be calculated. The $dI$ values are applied in molecular structure investigations.

If, for example, on squalane as the stationary phase the retention indices of benzene and toluene are determined at 100°C, then the difference between them gives approximately the variation of the retention index due to a methyl group. In the following the calculation of retention indices is discussed, the basic data needed for the calculations were obtained using temperature-programmed GLC.

## 3 RETENTION INDEX IN TEMPERATURE-PROGRAMMED GLC

In order to facilitate the analytical application of GLC for the investigation of samples with wide boiling point ranges (most natural samples belong to this group), temperature-programmed GLC was developed[96]. This raised numerous theoretical and practical problems in connection with peak identification. Consider the use of a linear temperature programme with a constant heating rate during the whole analysis, starting at the instant of sample introduction, so that all components elute during this programme. In such a case, the retention index ($I_{\mathrm{TPGC}}$) can be calculated from the equation of Van den Dool and Kratz[21]:

$$I^{\mathrm{st.ph.}}_{\mathrm{TPGC},s} = 100n\left[\frac{T_R(s) - T_R(z)}{T_R(z+n) - T_R(z)}\right] + 100z \tag{13}$$

where s refers to the substance of interest, $n$ is the difference in the carbon numbers of the two $n$-alkanes, $T_R$ is the retention temperature (°C or °K) and $z$ is the carbon number of the $n$-alkane. For example, if retention temperature of T2-mycotoxin = 286.5°C, retention temperature of $C_{26}$ $n$-alkane = 273.0°C and retention temperature of $C_{28}$ $n$-alkane = 287.3°C on OV-101 at an initial temperature of 200°C, then

$$I(\mathrm{TPGC})^{\mathrm{OV\text{-}101}}_{\mathrm{T2}} = 100 \cdot 2\left(\frac{286.5 - 273.0}{287.3 - 273.0}\right) + 100 \cdot 26 = 2788.8\ \mathrm{i.u.} \tag{14}$$

Assuming a linear heating rate of $\beta$ °C/min and using the retention time, eqn. 13 takes the form

$$I^{\mathrm{st.ph.}}_{\mathrm{TPGC},s} = 100\,n\left[\frac{t_R(s) - t_R(z)}{t_R(z+n) - t_R(z)}\right] + 100z \tag{15}$$

Most of the integrators used in practice indicate and also register the retention time values, so eqn. 15 can easily be applied to the calculation of the appropriate retention indices. The basic programme of numerous reporting integrators includes this relationship and among the results they print the value of the retention index.

TABLE 1

COMPARISON OF $I_{TPGC}$ (i.u.) MEASURED ON PEGA AND CALCULATED BY GOLOVNYA AND URALETZ[187]

Inlet pressure of carrier gas, 131.72 kPa; $\beta = 3°C/min$

| *Compound* | *Initial temperature (°C)* | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | *75.0* | | | *100.0* | | | *125.0* | | |
| | *Measured* | *Calculated* | *Difference* | *Measured* | *Calculated* | *Difference* | *Measured* | *Calculated* | *Difference* |
| *n*-Pentyl methyl ketone | 1026 | 1025 | 1 | 1039 | 1044 | −5 | 1055 | 1062 | −7 |
| *n*-Hexyl methyl ketone | 1130 | 1129 | 1 | 1142 | 1146 | −4 | 1159 | 1164 | −5 |
| *n*-Heptyl methyl ketone | 1233 | 1233 | 0 | 1244 | 1248 | −4 | 1261 | 1266 | −5 |
| *n*-Octyl methyl ketone | 1336 | 1338 | −2 | 1348 | 1353 | −5 | 1363 | 1368 | −5 |
| *n*-Nonyl methyl ketone | 1441 | 1443 | −2 | 1452 | 1457 | −5 | 1466 | 1472 | −6 |
| *n*-Undecyl methyl ketone | 1654 | 1654 | 0 | 1664 | 1667 | −3 | 1674 | 1679 | −5 |

For example, using OV-101 as the stationary phase the retention data were as follows: retention time of T2-mycotoxin = 1153.0 sec, retention time of $C_{26}$ $n$-alkane = 973.7 sec, retention time of $C_{28}$ $n$-alkane = 1163.6 sec at an initial temperature of 200°C. Hence

$$I(\mathrm{TPGC})^{\mathrm{OV\text{-}101}}_{\mathrm{T2}} = 100 \cdot 2 \left[ \frac{1153.0 - 973.7}{1163.6 - 973.7} \right] + 100 \cdot 26 = 2788.8 \text{ i.u.} \tag{16}$$

The calculation of the retention index for cases where temperature programming is combined with isothermal periods or where the programming rate is changed during the analysis is more complicated[738]. Most retention index values are determined under isotherm conditions, and numerous workers therefore dealt with the conversion of the indices into temperature-programmed conditions.

Giddings[1383] derived a simple solution based on theoretical considerations

$$T = 0.92\ T_R \tag{17}$$

where $T$ is the isothermal column temperature (°C or °K). A simpler solution was derived by Guiochon[42], which seemed to be valid in several cases

$$T = T_R - 20 \tag{18}$$

A broad research programme was carried out by Golovnya and Uraletz[187], who used various homologous series as model compounds. Some of their results for the effect of initial temperature and heating rate on the $I_{\mathrm{TPGC}}$ value are presented in Table 1. It can be seen that an increase in the initial temperature increases the difference between the calculated and measured retention indices.

We compared data in the literature[405] with those calculated by using eqn. 13. The results are given in Table 2.

TABLE 2

COMPARISON OF RETENTION INDICES (PTGC) MEASURED BY GOLOVNYA AND URALETZ[405] AND BY VAN DEN DOOL AND KRATZ[21] ON PEGA

| *Compound* | *Retention index (i.u.)* | | *Difference (i.u.)* |
|---|---|---|---|
| | *Golovnya and Uraletz* | *Van den Dool and Kratz* | |
| 2-Hexanone | 1121 | 1118 | +3.0 |
| 2-Heptanone | 1225 | 1223 | +2.0 |
| 2-Octanone | 1329 | 1329 | 0.0 |
| 2-Nonanone | 1434 | 1434 | 0.0 |
| 2,4-Hexadienal | 1457 | 1454 | +3.0 |
| 2,4-Octadienal | 1656 | 1658 | −2.0 |
| 2-Dodecanone | 1747 | 1749 | −2.0 |
| 2,4-Nonadienal | 1768 | 1770 | −2.0 |

Based on the relationship between the retention index and column temperature, Erdey *et al.*[308] showed that the value of $I_{\text{TPGC}}$ can be calculated by the following equation

$$I^{\text{st ph}}_{\text{TPGC,s}} = A + \frac{B \ln\left(\dfrac{T_{\text{R}} + C}{T_0 + C}\right)}{T_{\text{R}} - T_0} \tag{19}$$

where $A$, $B$ and $C$ are constants[528], $T$ is the initial temperature (°K) and $T_{\text{R}}$ is the retention temperature (°K). The application of eqn. 19 can be demonstrated by the following example. The dependence of the retention index on the column temperature, using PEG-1500 as the stationary phase, can be given by an Antoine-type equation:

$$I^{\text{PEG-1500}}_{\text{benzene}}(T) = 1718.0 - \frac{1032240.0}{T + 967.0} \tag{20}$$

Using 84.0°C (357.16°K) as the initial temperature and a heating rate of 4.5°C/min the value of retention temperature was 99.0°C (372.16°K). Substituting into eqn. 19, we obtain

$$I^{\text{PEG-1500}}_{\text{TPGC,benzene}} = 1718.0 - \frac{1032240.0 \;\ln\left[\dfrac{372.16 + 967.0}{357.16 + 967.0}\right]}{372.16 - 357.16} = 942.8 \text{ i.u.} \tag{21}$$

The measured value was 943.0 i.u.

We compared the calculated data obtained by using different equations. The results are given in Table 3.

Similarly to the example presented above, if reliable values for the constants $A$, $B$ and $C$ are used in eqn. 19, it gives fairly good approximations because it is not affected by the TPGC parameters. However, the disadvantage of its application is that regression analysis and computer calculations have to be carried out for the

TABLE 3

COMPARISON OF RETENTION INDICES CALCULATED USING DIFFERENT EQUATIONS FOR BENZENE ON PEG-1500 AS STATIONARY PHASE IN TPGC

| *Retention index (i.u.)* | | | | |
|---|---|---|---|---|
| *Measured* | *Calculated* | | | |
| | *By eqn. 13* | *By eqn. 17* | *By eqn. of Golovnya and Uraletz*[187] | *By eqn. 21* |
| 943 | 944 | 943 | 943 | 942.8 |

determination of correct values of $A$, $B$ and $C$ For further discussion, see refs. 42, 44, 57, 75, 76, 123, 127, 162, 187, 212, 222, 223, 306, 315, 342, 346, 347, 384, 386, 405, 428, 447, 465, 481, 482, 591, 594, 738, 742, 782, 1083, 1105 and 1340

## 4 RETENTION INDEX IN PRESSURE- OR FLOW-PROGRAMMED GLC

Using these conditions the retention index will remain constant and identical with the value measured without programming, because programming of the carrier gas influences only the calculation of the net retention volumes and not the retention index values[209,294]

## 5 INDEPENDENCE OF THE RETENTION INDEX OF SOME GAS CHROMATOGRAPHIC PARAMETERS

The retention index is independent of the following gas chromatographic parameters

(a) the quality and chemical nature of the *inert* support and/or the quality of the open-tubular (capillary) column, assuming that this column does not show any wall effects after appropriate pre-treatment,

(b) the chemical nature and physical parameters of the carrier gas (inlet and outlet pressure, flow-rate, etc ); if these parameters are constants

However, the inert support and the inner wall of an open-tubular column create numerous problems. Theoretically, in GLC

$$V_N = KV_L \tag{22}$$

where $V_N$ is the net retention volume (cm$^3$ carrier gas), $K$ is the partition coefficient and $V_L$ is the volume of the stationary phase present in the column (cm$^3$). If the support is not inert, or the open-tubular column shows wall effects, then adsorption processes are taking place at the same time as absorption. If absorption and adsorption act together in establishing equilibrium, the net retention value of the solute can be written as[1384]

$$V_N = KV_L + K_aA_L \tag{23}$$

where $K_a$ is the adsorption coefficient (cm$^3$/m$^2$) and $A_L$ is the surface area of the stationary phase (m$^2$).

In most instances the circumstances of adsorption differ in various columns, in contrast with GLC, where under analytical conditions the specific retention volumes of substances are constants. Therefore, the specific retention volume is a characteristic value of a given column Naturally, this results in different retention index values This is the reason why retention indices may differ markedly from each other although they have been determined under identical conditions of gas chromatography, in packed or open-tubular columns As an example, Table 4 gives retention index data taken from Chien *et al.*'s paper[1319].

Consequently, correct retention index values can be obtained with packed columns only if inert supports are used.

TABLE 4

COMPARISON OF RETENTION INDICES MEASURED ON DIFFERENT COLUMN TYPES ON OV-1 AT 80°C BY CHIEN *et al.*[1319]

| *Solute* | *Retention index (i.u.)* | | |
|---|---|---|---|
| | *On packed column* | *On open-tubular column* | *Difference (i.u.)* |
| *cis*-2-Pentene | 510.5 | 514.9 | −4.4 |
| Cyclohexane | 669.4 | 670.3 | −0.9 |
| Benzene | 659.3 | 659.7 | −0.4 |
| 2-Methyl-2-pentene | 605.3 | 608.5 | −3.2 |
| 2,3-Dimethylbutane | 569.0 | 570.7 | −1.7 |

TABLE 5

EFFECT OF THE INERT SUPPORT ON RETENTION INDICES ON SQUALANE AT 100°C (AFTER EVANS AND SMITH[133])

| *Support* | *Retention index (i.u.)* | | | |
|---|---|---|---|---|
| | *Chlorobenzene* | *1-Pentanol* | *2-Hexanone* | *Benzonitrile* |
| Chromosorb W | 825.7 | 800.0 | 774.0 | 918.6 |
| Mikrosorb W | 827.1 | 781.9 | 755.3 | 910.4 |
| Celite | 826.0 | 763.5 | 747.2 | 904.2 |
| Anakrom | 826.8 | 714.9 | 733.3 | 896.5 |
| Chromosorb G | 826.9 | 714.4 | 732.6 | 896.6 |
| Gas-Chrom Q | 826.8 | 703.9 | 730.9 | 893.1 |
| Silanized Chromosorb W | 826.5 | 694.9 | 728.7 | 893.9 |

The effect of the inert support on the retention indices, as reported by Evans and Smith[133], is shown in Table 5.

With open-tubular columns, adequate pre-treatment of the column is needed to prevent wall effects.

## 6 DEPENDENCE OF THE RETENTION INDEX ON SOME GAS CHROMATOGRAPHIC PARAMETERS

The retention index value depends on the following gas chromatographic parameters: (a) the chemical nature of the substance examined, (b) the chemical nature of the stationary phase and (c) the column temperature.

### 6.1 *Chemical nature of the substance examined*

Only one retention index value can correspond to a particular chemical structure under given gas chromatographic conditions. However, the inverse of this statement is not true: several chemical structures can correspond to a single retention index value under given gas chromatographic conditions. Therefore, in peak identification and/or elimination with help of retention indices, a single determination (on one stationary phase at one column temperature) is not sufficient. For example, using squalane as the stationary phase, at 90°C the retention index of 2,4-dimethylpentane is 731.0 i.u. Assuming that the standard deviation is ±0.5 i.u., under the same gas chromatographic conditions the same retention index is valid for 1,1,3-trimethylcyclopentane, 1-chloropentane, etc. Evidently, this single retention index is inadequate to identify and/or to eliminate peaks (see Table 42).

### *6.2 Chemical nature of the stationary phase*

By this concept we mean the dependence of the retention index values on the actual stationary phase: its chemical structure, its impurities and its physico-chemical properties.

In practice, stationary phases have different characteristics may be marketed under the same product name. For example, the influence of the difference in origin on McReynolds constants determined on DEGS at 120°C, according to Ettre[595], is shown in Table 6.

TABLE 6

INFLUENCE OF THE ORIGIN OF THE STATIONARY PHASE ON THE McREYNOLDS CONSTANTS DETERMINED ON DEGS AT 120°C (AFTER ETTRE[595])

| *Supplier* | $x'$ | $y'$ | $z'$ | $u'$ | $s'$ |
|---|---|---|---|---|---|
| Supelco (No. 1045) | 470 | 705 | 558 | 788 | 779 |
| Chemical Research Services | 492 | 733 | 581 | 833 | 791 |
| Supelco Inc. (No. 1303) | 496 | 746 | 590 | 837 | 835 |
| PolyScience Corp. | 499 | 751 | 593 | 840 | 860 |

Early investigations showed that, even with chemically identical stationary phases, the retention index values are influenced by the amount of the stationary phase present in the column under otherwise identical gas chromatographic conditions, if there is a significant difference in this amount, such 1 and 15% (w/w).

For example, the influence on some retention indices measured by Bonastre *et al.*[170] on PEG-20M stationary phase at 120°C of the amount of stationary phase present in the column is shown in Table 7.

TABLE 7

INFLUENCE OF THE AMOUNT OF STATIONARY PHASE PRESENT IN THE COLUMN ON RETENTION INDICES (i.u.) MEASURED BY BONASTRE *et al.*[170] ON PEG-20M STATIONARY PHASE AT 120°C

| *Compound* | *Amount of stationary phase (%, w/w)* | | | |
|---|---|---|---|---|
| | *2.5* | *5.0* | *10.0* | *15.0* |
| Benzene | 832 | 890 | 925 | 943 |
| Toluene | 922 | 978 | 1018 | 1037 |
| Methanol | 778 | 807 | 818 | 841 |
| 1-Butanol | 987 | 1040 | 1070 | 1092 |
| 2-Pentanone | 868 | 916 | 939 | 964 |

This effect can be explained in the following way The value of the retention index is only independent of the amount of stationary phase if merely absorption and desorption processes take place in the column (GLC) Based on experience, this phenomenon occurs on most supports/stationary phases if the wetting is about 10% (w/w) If the wetting is less, say 1–3% (w/w), then, as mentioned previously, adsorption processes occur and influence the value of determined retention index. It is assumed that the amount of stationary phase has an effect on retention indices only through this secondary process

In experiments carried out with a mechanically treated sodium chloride support that had previously been treated with ethanol, adsorption did not occur even with 0.25% (w/w) wetting and the retention indices seemed to be reliable (see Table 43).

### *6.3. Column temperature*

Since the introduction of the RIS, the most discussed question has been the temperature dependence of retention indices. Although this question had been examined by many investiators, it has not yielded unambiguous results.

For many years the cause of this lack of success was the assumption that a linear relationship exists between retention index and column temperature. In the early years the determination of retention indices was carried out mostly on apolar stationary phases, and alkanes were used as model substances (apolar stationary phase/apolar substance), and the relationship appeared to be linear. Examining the phenomenon in detail and increasing the number of stationary phases and model substances, *i.e.*, their polarity, a lack of conformity of the results became evident and the validity of linear relationship became questionable This led researchers to derive non-linear approximate equations. In 1969 an Antoine-type hyperbolic approximation was developed that was generally accepted about 7 years later[279] Kováts and co-workers made the following remarks in a paper[953] dealing with the elaboration of Apolane-87, when they examined the dependence of the retention indices of neopentane and tetramethylsilane on column temperature "It can be seen that, in fact, the temperature dependence of the retention index is hyperbolic . . ." Now it can be stated that the dependence of retention indices on column temperature can be correctly described by an Antoine-type hyperbolic curve:

$$I_s^{\mathrm{st\,ph}}\,(T) = A + \frac{B}{T + C} \qquad (24)$$

where $T$ is the column temperature (°K) and $A$, $B$ and $C$ are constants[279] The curve according to such an Antoine-type equation can nevertheless have a significant linear portion, the length of which depends mainly on the polarity of the substance examined, on the stationary phase applied and on their interactions. It is well known that the retention indices of non-polar substances on non-polar stationary phases show an almost completely linear dependence on column temperature In fact, in this instance a linear regression gives more accurate results than the hyperbolic approximation.

In practice, three retention indices ($I_1$, $I_2$ and $I_3$) determined at temperatures $T_1$, $T_2$ and $T_3$ are used for the calculation of the constants in eqn. 24·

$$C = \frac{(T_2 - T_1)(I_3T_3 - I_1T_1) + (T_3 - T_1)(I_1T_1 - I_2T_2)}{(T_3 - T_1)(I_2 - I_1) - (T_2 - T_1)(I_3 - I_1)} \tag{25}$$

$$A = \frac{I_2T_2 - I_1T_1 + C(I_2 - I_1)}{T_2 - T_1} \tag{26}$$

$$B = (I_2 - A)(T_2 + C) \tag{27}$$

When calculating $C$, owing to the random trend of the data it often occurs that the denominator of eqn. 24 is zero. In these instances it is advisable to increase the value of $I_2$ by 0.1 i.u. and to repeat the calculations. If programmes are prepared for desktop calculators or minicomputers, this step has to be put in advance in the programme using an "if" command if $(T_3-T_1)(I_2-I_1) = (T_2-T_1)(I_3-I_1)$, then

$$I_2 = I_2 + 0.1 \tag{28}$$

and the calculation of $C$ has to be started again.

The spread of minicomputers facilitated the calculation of hyperbolic regression based on more than three basic data ($n$ = 5, 7 etc.) and the accuracy of calculation of the constants in eqn. 24 increased.

A very interesting, but for the present only theoretically significant, retention index value is obtained if in the Antoine-type equation describing the column temperature dependence of the retention index (eqn. 24) we substitute $T$ = 0°K

$$I_0^{\mathrm{st\ ph}} = A + \frac{B}{C} \tag{29}$$

where $I_0$ is the zero-point retention index[363] at 0°K

In order to illustrate the simplified calculation, we can utilize the retention indices of aniline on SE-30 at three different column temperatures

$$I_1^{\mathrm{SE\text{-}30}}\ (383\ ^\circ\mathrm{K}) = 968.9\ \mathrm{i.u.} \tag{30}$$

$$I_2^{\mathrm{SE\text{-}30}}\ (403\ ^\circ\mathrm{K}) = 974.7\ \mathrm{i.u.} \tag{31}$$

$$I_3^{\mathrm{SE\text{-}30}}\ (433\ ^\circ\mathrm{K}) = 984.7\ \mathrm{i.u} \tag{32}$$

$A$, $B$ and $C$ are calculated as follows

$$C = \frac{20\,(426375.1 - 371088.7) + 50\,(371088 - 392804.1)}{290.0 - 316.0} = -767.6\ \mathrm{K} \tag{33}$$

$$A = \frac{21715.4 - 767.6 \cdot 5.8}{20} = 863.2\ \mathrm{i.u} \tag{34}$$

$$B = (-364.6) \cdot 111.5 = -40652.9\ \mathrm{i.u\ \ K} \tag{35}$$

TABLE 8

COMPARISON OF RETENTION INDICES MEASURED BY RIEDO *et al*[953] AND CALCULATED FOR NEOPENTANE ON APOLANE-87 STATIONARY PHASE AT DIFFERENT COLUMN TEMPERATURES

| *Column temperature (°C)* | *Retention index (i.u.)* | | |
|---|---|---|---|
| | *Measured* | *Calculated by eqn. 24** | *Difference* |
| 50 | 405.1 | 405.5 | −0.4 |
| 70 | 406.3 | 406.9 | −0.6 |
| 90 | 407.6 | 408.1 | −0.5 |
| 110 | 408.8 | 409.0 | −0.2 |
| 150 | 410.6 | 410.5 | 0.1 |
| 170 | 411.3 | 411.0 | 0.3 |
| 190 | 411.8 | 411.5 | 0.3 |
| 210 | 412.1 | 411.9 | 0.2 |

* The constants of eqn. 24 were calculated by eqns. 25–27 at 30, 130 and 230°C

Thus, the retention index of aniline on SE-30 at various column temperatures can be calculated from the following equation:

$$I^{\text{SE-30}}_{\text{aniline}}(T) = 863.2 - \frac{40652.9}{T - 767.6} \qquad (36)$$

Retention indices calculated with eqn. 24 were compared with those determined experimentally or with data from the literature. In almost all instances the agreement was good (Table 8). Hence eqn. 24 produces reliable results although it is based on only three retention indices.

In the last 25 years, many researchers have dealt with the determination of

TABLE 9

RETENTION INDICES OF OCTANES AND OCTENES AT 30.0 AND 50.0°C ON SQUALANE, MEASURED BY TOURRES[160]

| *Compound* | *Retention index (i.u.)* | |
|---|---|---|
| | *30°C* | *50°C* |
| 2,2,4-Trimethylpentane | 688.0 | 690.0 |
| 2,2-Dimethylhexane | 718.5 | 719.5 |
| 2,5-Dimethylhexane | 728.0 | 729.0 |
| 2,4-Dimethylhexane | 731.5 | 732.5 |
| 2,2,3-Trimethylpentane | 734.0 | 737.0 |
| 3,3-Dimethylhexane | 741.0 | 743.5 |
| 2,4-Dimethyl-1-hexene | 737.5 | 738.5 |
| 2,5-Dimethyl-1-hexene | 740.0 | 741.0 |
| 3,4-Dimethyl-1-hexene | 755.5 | 756.0 |
| 2-Methyl-2-heptene | 789.5 | 789.5 |

TABLE 10

RETENTION INDICES OF 2-PHENYL-4-ALKYLTETRAHYDRO-1,4-OXAZINES MEASURED BY BONDIVENNE AND BUSCH[173] ON XF-1150 AT DIFFERENT COLUMN TEMPERATURES

| *Alkyl group in oxazine* | *Retention index (i.u.)* | | |
|---|---|---|---|
| | *175.0°C* | *200.0°C* | *225.0°C* |
| *n*-Propyl | 2200.0 | 2259.0 | 2293.5 |
| *n*-Butyl | 2294.3 | 2361.9 | 2381.9 |
| *n*-Pentyl | 2377.7 | 2459.5 | 2484.3 |
| *n*-Hexyl | 2478.3 | 2660.6 | 2580.9 |

TABLE 11

RETENTION INDICES OF ADAMANTANES AT DIFFERENT TEMPERATURES ON DIFFERENT STATIONARY PHASES, MEASURED BY BURKHARD *et al.*[235]

| *Compound* | *Retention index (i.u.)* | | | | | |
|---|---|---|---|---|---|---|
| | *SE-30* | | | *PEG-20M* | | |
| | *145°C* | *160°C* | *175°C* | *145°C* | *160°C* | *175°C* |
| Adamantane | 1118 | 1132 | 1143 | 1320 | 1339 | 1363 |
| 1-Methyladamantane | 1137 | 1151 | 1162 | 1313 | 1325 | 1348 |
| 1,3-Dimethyladamantane | 1151 | 1163 | 1174 | 1296 | 1310 | 1331 |
| 1,3,5-Trimethyladamantane | 1163 | 1173 | 1185 | 1274 | 1292 | 1310 |
| Adamantanone | 1320 | 1344 | 1357 | 1867 | 1895 | 1918 |
| 1-Adamantanol | 1268 | 1292 | 1301 | 1844 | 1862 | 1882 |
| 1-Fluoroadamantane | 1159 | 1174 | 1184 | 1512 | 1534 | 1557 |
| 1-Choloroadamantane | 1298 | 1315 | 1331 | 1689 | 1713 | 1741 |
| 3,5-Dimethylbromoadamantane | 1401 | 1417 | 1432 | 1762 | 1788 | 1815 |

TABLE 12

RETENTION INDICES OF ALIPHATIC AND ALICYCLIC SULPHIDES AT DIFFERENT COLUMN TEMPERATURES AND ON DIFFERENT STATIONARY PHASES, MEASURED BY MARTINU AND JANÁK[340]

| *Compound** | *Retention index (i.u.)* | | | | | |
|---|---|---|---|---|---|---|
| | *Isodecyl phthalate* | | | *Apiezon L* | | |
| | *110°C* | *130°C* | *150°C* | *110°C* | *130°C* | *150°C* |
| $CH_3$-S-$CH_3$ | 579 | 585 | 592 | 516 | 523 | 528 |
| $CH_3$-S-nPr | 783 | 787 | 792 | 718 | 723 | 727 |
| $CH_3$-S-secBu | 843 | 849 | 853 | 784 | 790 | 796 |
| Et-S-Et | 759 | 765 | 772 | 694 | 698 | 704 |
| Et-S-nBu | 957 | 962 | 965 | 896 | 901 | 906 |
| nPr-S-secBu | 996 | 999 | 1005 | 940 | 946 | 951 |
| iPr-S-secBu | 941 | 946 | 951 | 886 | 890 | 897 |

* nPr = *n*-propyl, iPr = isopropyl, secBu = *sec*-butyl, nBu = *n*-butyl, Et = ethyl

TABLE 13

RETENTION INDICES OF *n*-ALKENES ON DI-*n*-BUTYL TETRACHLOROPHTHALATE AT DIFFERENT TEMPERATURES, MEASURED BY RYBA[537]

| *Compound* | *Retention index (i.u.)* | | | |
|---|---|---|---|---|
| | *60°C* | *80°C* | *100°C* | *120°C* |
| 1-Heptene | 710.0 | 710.4 | 710.7 | 711.1 |
| 1-Octene | 810.3 | 810.7 | 811.0 | 811.4 |
| 1-Nonene | 910.7 | 911.1 | 911.5 | 911.9 |
| 1-Decene | 1011.0 | 1011.4 | 1011.8 | 1012.2 |
| *trans*-3-Heptene | 703.5 | 703.3 | 703.1 | 702.9 |
| *trans*-3-Octene | 805.2 | 805.0 | 804.7 | 804.5 |
| *trans*-3-Nonene | 904.0 | 903.7 | 903.5 | 903.3 |
| *trans*-3-Decene | 1003.5 | 1003.2 | 1003.0 | 1002.7 |
| *cis*-4-Octene | 809.7 | 810.7 | 811.6 | 812.5 |
| *cis*-4-Nonene | 906.8 | 907.8 | 908.8 | 909.7 |
| *cis*-4-Decene | 1003.9 | 1004.9 | 1005.9 | 1006.8 |

TABLE 14

RETENTION INDICES OF ISOMERIC MONOBROMO-1,2-DICARBA-*CLOSO*-DODECABORANES ON QF-1 AT DIFFERENT COLUMN TEMPERATURES, MEASURED BY STUCHLÍK *et al*[666]

| *Compound* | *Retention index (i.u.)* | | | | |
|---|---|---|---|---|---|
| | *140°C* | *150°C* | *160°C* | *170°C* | *180°C* |
| 1-Bromo-*o*-carborane | 1564 | 1573 | 1582 | 1591 | 1600 |
| *o*-Carborane | 1650 | 1659 | 1668 | 1677 | 1686 |
| 3-Bromo-*o*-carborane | 1753 | 1765 | 1776 | 1788 | 1800 |
| 4-Bromo-*o*-carborane | 1918 | 1937 | 1957 | 1976 | 1996 |
| 8-Bromo-*o*-carborane | 2100 | 2122 | 2145 | 2167 | 2190 |
| 9-Bromo-*o*-carborane | 2145 | 2169 | 2192 | 2216 | 2240 |

retention indices on different stationary phases at different column temperatures. Without claiming to be comprehensive, some of the results are given in Tables 9–14.

For further details, see refs. 130, 198, 205, 206, 207, 217, 231, 235, 236, 281, 310, 373, 408, 424, 490, 530, 581, 583, 634, 638, 701, 752, 785, 939, 949, 951, 953, 1033, 1042, 1043, 1071, 1317 and 1352

## 7 SOURCES OF ERROR AND ERRORS IN RETENTION INDEX DETERMINATION

The important sources of error are as follows:

(a) errors in the determination of retention times (measurement errors, influence of the amount of sample, operator errors);

(b) errors in the measurement or calculation of the gas holdup time (*e.g.*, calculation is carried out in the non-linear range of *n*-alkanes);

(c) the support is not perfectly coated, or is active; there are wall effects in the open-tubular (capillary) column;

(d) inhomogeneous stationary phase,

(e) fluctuations of instrumental and/or gas chromatographic parameters

Experience in the 1960s showed that the determination of the retention indices of highly polar substance on non-polar stationary phases, and the determination of the retention indices of non-polar substances, including *n*-alkanes, on highly polar stationary phases, are problematic, especially with respect to accuracy and reproducibility. In our opinion the solution to the problem lies in the exact knowledge of the range where gas chromatographic retention is independent of the concentration of the substance examined. Because in the above mentioned instances this range is very narrow, it is difficult to keep it within the boundaries of this range and this error appears as an error in the retention index.

The discussion of the problems related to the analysis of highly polar substances on non-polar stationary phases and *vice versa* is an oversimplification. The problem here really is the solubility of a particular sample in a particular stationary phase.

In the last 25 years, many workers have dealt with the accuracy and reproducibility of the retention index determinations and the following can be mentioned: Van Kemenade and Groenendijk[254, 255], Oberholtzer and Rogers[266], Cramers and co-workers[300, 301], Kaiser[326, 327, 420–422], Pacáková *et al.*[646], Gavrichev and Berezkin[598], Loewenguth[335], Uhdeová[366], Wicarowá *et al.*[371], Dondi and co-workers[392, 593], Rijks and co-workers[761, 762], Hoch and Pacáková[819], Ezrets and Vigdergauz[894], Guardino *et al.*[909], Ryba[955], Van Lenten *et al.*[1031], Schomburg *et al.*[1051], Mathiasson *et al.*[1123] and Pacáková and Kozlik[1127].

The more important questions have been studied by Rijks[653], who studied the role of all of the relevant parameters that contribute to the random error. The more important factors proved to be fluctuations of column temperature, carrier gas velocity, time measurement and amount of sample. It was shown that a precision corresponding to a standard deviation of 0.05 i.u. is attained and that it is possible to reproduce retention indices to within about 0.2 i.u. in different laboratories.

The equation giving the greatest error in the determination of retention indices[307] has been named after Erdey

$$E_1 \leqslant \left\{ \frac{1}{[\bar{t}_R(S) - \bar{t}_M] \log\left[\dfrac{\bar{t}_R(z+1) - \bar{t}_M}{\bar{t}_R(z) - \bar{t}_M}\right] 2.30258} \cdot \Delta t_R(S) + \right.$$

$$+ \frac{\log\left[\dfrac{\bar{t}_R(z+1) - \bar{t}_M}{\bar{t}_R(S) - \bar{t}_M}\right]}{[\bar{t}_M - \bar{t}_R(z)] \log^2\left[\dfrac{\bar{t}_R(z+1) - \bar{t}_M}{\bar{t}_R(z) - \bar{t}_M}\right] 2.30258} \Delta t_R(z) +$$

$$+ \frac{\log\left[\dfrac{\bar{t}_R(S) - \bar{t}_M}{\bar{t}_R(z) - \bar{t}_M}\right]}{[\bar{t}_M - \bar{t}_R(z+1)] \log^2\left[\dfrac{\bar{t}_R(z+1) - \bar{t}_M}{\bar{t}_R(z) - \bar{t}_M}\right] 2.30258} \Delta t_R(z+1) +$$

$$\left.\frac{\dfrac{\bar{t}_R(S) - \bar{t}_R(z)}{[\bar{t}_R(S) - \bar{t}_M]\,[\bar{t}_R(z) - \bar{t}_M]} \cdot \log\left[\dfrac{\bar{t}_R(z+1) - \bar{t}_M}{\bar{t}_R(z) - \bar{t}_M}\right] - \dfrac{\bar{t}_R(z+1) - \bar{t}_R(z)\cdot\log\left[\dfrac{\bar{t}_R(S) - \bar{t}_M}{\bar{t}_R(z) - \bar{t}_M}\right]}{[\bar{t}_R(z) - \bar{t}_M]\,[\bar{t}_R(z+1) - \bar{t}_M]}}{\log^2\left[\dfrac{\bar{t}_R(z+1) - \bar{t}_M}{\bar{t}_R(z) - \bar{t}_M}\right] 2.30258}\right| \cdot \Delta t_M \Bigg\} \cdot 1$$

where:

$E_1$ = total error of the retention index determination (index units) under the conditions of classical gas chromatography ($T$, $F$, $p_i$ and $j$ are all constants)

$$\Delta \bar{t}_R(X) = \frac{|t_{R,1}(X) - \bar{t}_R(X)| + |t_{R,2}(X) - \bar{t}_R(X)| + \ldots + |t_{R,g}(X) - \bar{t}_R(X)|}{m} \qquad (38)$$

$$\bar{t}_R(X) = \frac{t_{R,1}(X) + t_{R,2}(X) + \ldots + t_{R,g}(X)}{m} \qquad (39)$$

$\bar{t}_R(X)$ = average retention time (min),
X = $s$, $z$ and $z + 1$;
$g$ = serial number;
$m$ = number of parallel measurements; in this instance $m = 11$,
$T$ = temperature of column (°K);
$F$ = flow-rate of carrier gas (ml/min);
$p_i$ = inlet pressure of carrier gas (kPa);
$j$ = compressibility factor according to James and Martin[38];
$t_R$ = retention time (min);
$t_M$ = time of passage of the inert substance through the column (min);
s = substance;
$z$ and $z + 1$ = $n$-alkanes with carbon numbers $z$ and $z + 1$, respectively;
$z$ = carbon number.

As an example, the calculation for styrene on squalane at 70°C is as follows [$\bar{t}_M = 26.4$ sec; $\Delta t_M = 0.25$ sec; $\bar{t}_R(n\text{-}C_8) = 637.8$ sec, $\bar{t}_R(\text{styrene}) = 1070.0$ sec, $\bar{t}_R(n\text{-}C_9) = 1520.7$ sec; $\Delta\bar{t}_R(n\text{-}C_8) = 3.20$ sec, $\Delta\bar{t}_R(\text{styrene}) = 5.40$ sec; $\bar{t}_R(n\text{-}C_9) = 7.60$ sec]

$$E_I \leqslant \left\{ \left| \frac{1}{(1070.0 - 26.4)\log\left(\dfrac{1520.7 - 26.4}{637.8 - 26.4}\right) 2.30258} \right| \cdot 5.4 + \right.$$

$$+ \left| \frac{\log\left(\dfrac{1520.7 - 26.4}{1070.0 - 26.4}\right)}{(26.4 - 637.8)\log^2\left(\dfrac{1520.7 - 26.4}{637.8 - 26.4}\right) 2.30258} \right| 3.2 +$$

$$+ \Bigg| \frac{\log\left(\dfrac{1070.0 - 26.4}{637.8 - 26.4}\right)}{(26.4 - 1520.7)\log^2\left(\dfrac{1520.7 - 26.4}{637.8 - 26.4}\right) 2.30258} \Bigg| 7.6 +$$

$$\Bigg| \frac{\dfrac{1070.0 - 637.8}{(1070.0 - 26.4)(637.8 - 26.4)} \log\left(\dfrac{1520.7 - 26.4}{637.8 - 26.4}\right) - \dfrac{(1520.7 - 637.8)\log\left(\dfrac{1070.0 - 26.4}{637.8 - 26.4}\right)}{(637.8 - 26.4)(1520.7 - 26.4)}}{\log^2\left(\dfrac{1520.7 - 26.4}{637.8 - 26.4}\right) 2.30258} \Bigg| 0.25 \Bigg\}$$

$$2.4 \cdot 10^{-1} + 3.4 \cdot 10^{-1} + 5.8 \cdot 10^{-1} + 3 \cdot 10^{-5}) 100 = 1.2 \text{ i.u.} \tag{40}$$

where 2.30258 is the log–ln conversion factor

Some interesting results are given in Tables 15–18. For further information, see refs. 194, 253, 267, 269, 270, 369, 452, 476, 508, 559, 604, 705, 715, 726, 760, 999, 1020, 1052, 1142, 1146, 1194, 1252, 1281, 1305 and 1350b.

## 8 DETERMINATION AND/OR CALCULATION OF THE GAS HOLDUP TIME

The gas holdup time ($t_M$) is a particularly widely disputed problem in GLC. Experiments have been carried out to try to eliminate it, but these could not succeed because its existence originates from the basic principles of GLC. The gas holdup time plays a very important role in solving theoretical and practical problems in GLC.

The gas holdup time has special significance in the retention index system because it occurs indirectly in the equation expressing the retention index and it also influences the accuracy of retention index determinations, although this also depends on the magnitude of the retention index[361].

TABLE 15

COMPARISON OF RETENTION INDICES OF DIMETHYLUNDECANES DETERMINED BY HOSHIKAWA *et al*[506] AT 100°C ON SQUALANE AND BY SCHOMBURG AND HENNEBERG[220]

| *Undecane* | *Retention index (i u.)* | | |
|---|---|---|---|
| | *Schomburg and Henneberg* | *Hoshikawa et al.* | *Difference* |
| 3,7-Dimethyl | 1217.9 | 1219.4 | −1.5 |
| 3,8-Dimethyl | 1226.8 | 1227.2 | −0.4 |
| 3,9-Dimethyl | 1238.1 | 1238.5 | −0.4 |
| 6,6-Dimethyl | 1200.0 | 1201.0 | −1.0 |

TABLE 16

REPRODUCIBILITY OF RETENTION INDICES (i u ) ON SQUALANE AT 70°C, MEASURED BY RIJKS[653] USING DIFFERENT INSTRUMENTS, COLUMNS AND OPERATORS

| *Compound* | *System* | | *Difference* |
|---|---|---|---|
| | *I* | *II* | |
| 2-Methylhexane | 666 96 | 666 98 | −0.02 |
| 2,2-Dimethylhexane | 720 47 | 720 47 | 0 0 |
| 2,2,3,3-Tetramethylpentane | 857 49 | 857 48 | +0 01 |
| Cyclohexane | 667 12 | 667 14 | −0 02 |
| 1-Heptene | 682 33 | 682 32 | +0 01 |
| Benzene | 641 81 | 641.90 | −0 09 |

TABLE 17

RETENTION INDICES OF TOLUENE ON SQUALANE AT 65, 78 and 100°C MEASURED BY MEMBERS OF THE DATA SUB-COMMITTEE OF THE GC DISCUSSION GROUP[56]

| *Operator* | *Retention index (i.u )* | | | *Deviation from the mean (i u )* | | |
|---|---|---|---|---|---|---|
| | *65°C* | *78°C* | *100°C* | *65°C* | *78°C* | *100°C* |
| A | 750 | 753 | 759 | −1 | −1 | −1 |
| C | 747 | 751 | 756 | +2 | +1 | +2 |
| D | 748 | 752 | 757 | +1 | 0 | +1 |
| E | 748 | 751 | 756 | +1 | +1 | +2 |
| F | 751 | 755 | 761 | −2 | −3 | −3 |
| H | 748 | 752 | 756 | +1 | 0 | +2 |
| I | 751 | 755 | 761 | −2 | −3 | −3 |
| Mean | 749 | 752 | 758 | | | |

TABLE 18

INFLUENCE OF SAMPLE SIZE ON THE RETENTION INDICES (i u ) OF FATTY ACID METHYL ESTERS AT 200°C ON DEGS, MEASURED BY GOLOVNYA *et al*[714]

| *Compound* | *Sample size (mg)* | | | | | | |
|---|---|---|---|---|---|---|---|
| | $1 \; 10^{-5}$ | $1 \; 10^{-4}$ | $2 \; 10^{-3}$ | $1 \; 10^{-3}$ | $2 \; 10^{-3}$ | $1 \; 10^{-2}$ | $2 \; 10^{-2}$ |
| 18 0 | 2313 | 2315 | 2314 | 2315 | 2314 | 2317 | 2323 |
| 19 0 | 2407 | 2408 | 2408 | 2408 | 2407 | 2416 | 2420 |
| 20 0 | 2501 | 2500 | 2501 | 2501 | 2500 | 2512 | 2517 |
| 21 0 | 2594 | 2594 | 2595 | 2596 | 2596 | 2608 | 2615 |
| 22 0 | 2688 | 2688 | 2689 | 2690 | 2705 | 2705 | 2714 |
| 18 2 | 2314 | 2314 | 2316 | 2313 | 2317 | 2314 | 2316 |
| 18 3 | 2314 | 2314 | 2313 | 2312 | 2316 | 2315 | 2316 |
| 20 4 | – | 2501 | 2501 | 2500 | 2505 | 2504 | 2524 |

For the experimental determination of the gas holdup time, suitable substances are those which, under given gas chromatographic conditions, are not retarded by the stationary phase, in other words, which can be regarded as inert, travelling through the column at the same speed as the carrier gas

In contrast to certain substances (hydrogen, argon, neon, helium, methane, etc ) used as carrier gases, others do not give a signal on FID or show a certain retention, which posed unexpected difficulties. Although research work on gas holdup time began 30 years ago, its significance still stimulates researchers to study these problems Studies have been made of the problems concerning the relationships valid for various homologous series (first for $n$-alkanes) Dealing with the homologous series of $n$-alkanes, the log $t_R'$ *versus* $z$ relationship is linear from $n$-heptane. The value of $t_M$ can be calculated using any three consecutive members in this linear section according to Peterson and Hirsch[2]:

$$t_M = \frac{t_R^2(z+1) - t_R(z)\, t_R(z+2)}{2t_R(z+1) - t_R(z) - t_R(z+2)} \tag{41}$$

Thus, the gas holdup time can be calculated from the following equation, *e.g.*, on Apolane-87 as stationary phase at 50°C, if the retention time of $n$-heptane = 601.4 sec, that of $n$-octane = 1228.8 sec and that of $n$-nonane = 2891.8 sec

$$t_M = \frac{1228.8^2 - 601.4 \cdot 2891.8}{2 \cdot 1228.8 - (601.4 + 2891.8)} = 221.3 \text{ sec} \tag{42}$$

The measured gas holdup time using methane was 223.0 sec

Difficulties arise because the retention times of a maximum of four consecutive $n$-alkanes can be accurately determined under isothermal conditions so that the capacity ratio of the first eluting $n$-alkane is equal to or greater than unity. If these conditions can be fulfilled, then the $t_M$ value given by eqn 41 allows the calculation of correct retention indices

The results obtained by eqn 41 are compared with values of $t_M$ measured by

TABLE 19

RETENTION INDICES OF HYDROCARBONS USING $t_M$ VALUES, DETERMINED WITH DIFFERENT METHODS, MEASURED BY CRAMERS *et al*[301] AT 70°C ON SQUALANE

| *Compound* | *Retention index (i.u.)* | | | |
|---|---|---|---|---|
| | *$t_M$ (measured)* | | *$t_M$ (calculated)* | |
| | *With $H_2$* | *With $CH_4$* | *From $n$-$C_4$–$C_6$* | *From $n$-$C_6$–$C_8$* |
| Ethylcyclopropane | 511.51 | 511.53 | 511.38 | 511.31 |
| 2,2,3-Trimethylbutane | 642.58 | 642.59 | 642.46 | 642.40 |
| Cyclohexane | 667.09 | 667.10 | 666.99 | 666.94 |
| 2,2-Dimethylhexane | 720.47 | 720.47 | 720.43 | 720.41 |
| 2,2,3-Trimethylpentane | 740.10 | 740.11 | 740.06 | 740.03 |
| 3-Methylheptane | 772.91 | 772.92 | 772.88 | 772.86 |

TABLE 20

EFFECT OF GAS HOLDUP TIME ON THE CALCULATION OF RETENTION INDICES (i.u.) ON SQUALANE AT 70°C (AFTER RIJKS[653])

| *Compound* | *Origin of gas holdup time* | | | | |
|---|---|---|---|---|---|
| | *$CH_4$* | *Inert gas* | *Extrapolated from* | | |
| | | | $C_4$–$C_6$ | $C_5$–$C_7$ | $C_6$–$C_8$ |
| 2-Methylbutane | 475.47 | 475.37 | 474.82 | 474.80 | 474.66 |
| 2-Methylpentane | 570.01 | 569.97 | 569.71 | 569.70 | 569.64 |
| 2,2-Dimethylpentane | 626.95 | 626.93 | 626.82 | 626.82 | 626.79 |
| Gas holdup time (sec) | 965.2 | 963.6 | 955.8 | 953.8 | 950.7 |

using methane, hydrogen and inert gases. Some interesting results according to Cramers *et al.*[301] and Rijks[653] are given in Tables 19 and 20.

It is evident from Tables 19 and 20 that the value of $t_M$ determined with methane is slightly greater than that calculated from retention data for *n*-alkanes. The reason is that methane is partitioned in some extent between the carrier gas and the squalane stationary phase under the given conditions. We measured this partition experimentally at a column temperature of 70°C.

In the last 25 years many researchers have dealt with the determination and or pre-calculation of the gas holdup time. Without claim to be comprehensive we discuss below some interesting papers in this field.

Palm[267–269], Pauschmann[270] and Versino[369] proposed different methods for the determination of gas holdup time, but these methods are not widely used. Groenendijk and Van Kemenade[194] made careful studies of the applicability of eqn. 41 and their paper produced considerable discussion. Papers by Kaiser[253] and Rohrschneider[272] on this theme also roused great interest. The problems concerning the value of $t_M$ and its effect on the accuracy of retention index determinations were first discussed in detail by Kaiser[326, 327].

Caesar[476] used the relationship between adjusted retention times and retention indices to solve the problem. The results of his efforts, obtained by complicated calculations, were similar to those reported in other papers and consequently his method has not spread widely. The general use of programmable calculators has influenced the determination of $t_M$ values as developed in the considerable work by Kaiser[726].

Riedmann[760] determined $t_M$ with the help of the air peak, using an FID. Haferkamp[94a] described a method for the calculation of $t_M$ in a chapter in Kaiser's book.

Ezrets and Vigdergauz[894] discussed some theoretical and practical problems of $t_M$ determination. Guardino *et al.*[909] dealt in detail with the different questions of gas holdup time and confirmed earlier experience. Dominguez *et al.*[999] studied the method for $t_M$ determination using methane. They presented some possible computer calculation methods on the basis of the retention data of *n*-alkanes.

Haken *et al.*[1020] compared various methods, all based on the retention data of *n*-alkanes. They established that the more complicated methods did not give better results than the simple method based on the linearity of the *n*-alkane curve

Sevčik[1052,1142] published important papers in this field He suggested that it is possible to carry out calculations using the differences in the retention times instead of the absolute retention times of *n*-alkanes Smith *et al* [1146] presented the results of very important, comprehensive work and pointed out the advantages of the method of Gróbler and Bálizs[715]

Heeg *et al.*[1194] developed a cubic calculation method to overcome the difficulties in determining $t_M$ This method, which allows the calculation of retention indices without a knowledge of the $t_M$ value, is based on a third-order relationship between the logarithm of differences of unadjusted retention times of homologues and the carbon number. The determination of $t_M$ value is not necessary, but the linearity of the *n*-alkane curve is very important

Ettre[1252] was the first researcher to distinguish the direct determination of adjusted retention time (first approximation) and the calculation of this value following the determination of gas holdup time (second approximation). His conclusions were supported by numerous examples and tabulated data

Gróbler and Bálizs[715] worked out a remarkable computer method for statistical calculation of retention indices. The slope of the *n*-alkane curve and the gas holdup time are calculated estimated by a regression technique Guberska[604, 716] investigated the differences between the $t_M$ values measured with methane and the values calculated from the retention times of three *n*-alkanes and described an empirical equation to correct the data measured with methane. Sharples and Vernon[1143a] compared the $t_M$ values calculated from data for three *n*-alkanes with experimental data

Al-Thamir *et al.*[1164a] made a valuable contribution to the problem of $t_M$ values. They applied natural logarithms instead of the common decimal logarithms to the *n*-alkane curve, *i.e.*, ln $t'_R(z)$ *versus* $z$ They obtained some simple relationships by using the retention time differences of *n*-alkanes.

A detailed review on this subject was published by Wainwright and Haken[1305] They compared calculated gas holdup times obtained by different methods with the values determined experimentally with methane or with data calculated by various equations.

Summarizing the results on gas holdup times, it can be stated that if correct retention time data are available, the simple method applying three *n*-alkanes is equivalent to the more complicated computer methods. With incorrect retention time data, even a skillful computer operator is unable to produce correct retention index values Therefore, it is essential to select correct, calculated or determined gas holdup times for the calculations of exact retention indices For further details, see refs. 508, 705, 953, 1281, 1305 and 1350b

## 9 THE SLOPE OF THE *n*-ALKANE PLOT THE *b*-VALUE

By the *n*-alkane curve we mean the plot of the log $t'_R(z)$ *versus* carbon number ($z$) relationship for *n*-alkanes:

$$\log t'_R(z) = bz + a \qquad (43)$$

where $t'_R$ = adjusted retention time:

$$t'_R = t_R - t_M \tag{44}$$

$t_R$ = retention time; $t_M$ = gas holdup time; $z$ = carbon number of the *n*-alkane; $b$ = slope of the linear section of the *n*-alkane curve:

$$b = \log t'_R\,(z+1) - \log t'_R\,(z) \tag{45}$$

$a$ = intercept on the ordinate.

This plot is generally linear except for the first few members of the homologous series

The linearity of the *n*-alkane curve can be illustrated by the example of Apiezon-L at 100°C. The measured retention times were as follows: methane 15, ethane 24, propane 45, *n*-butane 96, *n*-pentane 192, *n*-hexane 398, *n*-heptane 830, *n*-octane 1753 and *n*-nonane 3717 sec. The $b$ values calculated with eqn. 45 were as follows: from $C_3$ and *n*-$C_4$, 0.523; from *n*-$C_4$ and *n*-$C_5$, 0.431; from *n*-$C_5$ and *n*-$C_6$, 0.340, from *n*-$C_6$ and *n*-$C_7$, 0.335; from *n*-$C_7$ and *n*-$C_8$, 0.329; and from *n*-$C_8$ and *n*-$C_9$, 0.338 (the standard deviation of this calculation is 0.003).

In order to eliminate the error in the gas holdup time determination or calculation the following relationship has been proposed for the determination of the $b$ value[452], if $z \geqslant 7$:

$$b = \log\left[\frac{t_R(z+2) - t_R(z+1)}{t_R(z+1) - t_R(z)}\right] \tag{46}$$

For the calculation of the $b$ value by eqn. 46, squalane as the stationary phase at 65°C can be used as an example. Measured retention times were *n*-heptane 2.937, *n*-octane 5.000 and *n*-nonane 10.079 min. Thus,

$$b = \log\left(\frac{10.079 - 5.000}{5.000 - 2.937}\right) = 0.391 \tag{47}$$

The temperature dependence of $b$ can be approximately described with the following equation:

$$b_T^{\text{st.ph.}} = \frac{D}{T} + E \tag{48}$$

where $D$ and $E$ are constants and $T$ is the column temperature (°K). Eqn 48 is a practical possibility for the check of the effective column temperature as follows:

$$T = \frac{D}{b_T^{\text{st ph}} - E} \tag{49}$$

The equations used for temperature control of columns with various stationary phases are as follows:

on squalane:

$$T = \frac{208.47}{b_T^{\mathrm{SQ}} + 0.231} \tag{50}$$

on Apiezon-L:

$$T = \frac{227.39}{b_T^{\mathrm{APL}} + 0.294} \tag{51}$$

on Apolane-87:

$$T = \frac{198.54}{b_T^{\mathrm{AP\text{-}87}} + 0.225} \tag{52}$$

on SE-30

$$T = \frac{216.65}{b_T^{\mathrm{SE\text{-}30}} + 0.302} \tag{53}$$

on PEG-20M

$$T = \frac{192.70}{b_T^{\mathrm{PEG\text{-}20M}} + 0.287} \tag{54}$$

on Dexsil-410

$$T = \frac{163.22}{b_T^{\mathrm{Dexs\text{-}410}} + 0.188} \tag{55}$$

on OV-17:

$$T = \frac{209.41}{b_T^{\mathrm{OV\text{-}17}} + 0.280} \tag{56}$$

on QF-1

$$T = \frac{206.62}{b_T^{\mathrm{QF\text{-}1}} + 0.308} \tag{57}$$

on SE-52:

$$T = \frac{223.27}{b_T^{\mathrm{SE\text{-}52}} + 0.319} \tag{57a}$$

on DC-550

$$T = \frac{215.59}{b_T^{\mathrm{DC\text{-}550}} + 0.291} \tag{57b}$$

on FFAP

$$T = \frac{184.82}{b_T^{\mathrm{FFAP}} + 0.248} \tag{57c}$$

TABLE 21

SLOPES (*b*) OF THE *n*-ALKANE CURVE (LOG $V_g$ *vs* $z$) ON SQUALANE AT 65, 78 AND 100°C, MEASURED BY MEMBERS OF THE DATA SUB-COMMITTEE OF THE GC DISCUSSION GROUP[56]

| *Operator* | *b* | | | *Deviation from the mean* | | |
|---|---|---|---|---|---|---|
| | *65°C* | *78°C* | *100°C* | *65°C* | *78°C* | *100°C* |
| C | 0.397 | 0.361 | 0.330 | −0.007 | +0.002 | −0.004 |
| D | 0.390 | 0.370 | 0.327 | 0 | −0.007 | −0.001 |
| E | 0.390 | 0.363 | 0.324 | 0 | 0 | +0.002 |
| F | 0.394 | 0.367 | 0.328 | −0.004 | −0.004 | −0.002 |
| G | 0.388 | 0.360 | 0.325 | +0.002 | +0.003 | +0.001 |
| H | 0.391 | 0.365 | 0.325 | −0.001 | −0.002 | +0.001 |
| I | 0.385 | 0.359 | 0.323 | +0.005 | +0.004 | +0.003 |
| Mean | 0.390 | 0.363 | 0.326 | | | |

The transformed equations are applicable for the calculations of *b* values at different column temperatures (eqn. 48).

In the following an example is presented for Dexsil-410 as the stationary phase. The determined value of *b* was 0.1566 at 200°C. This value was substituted into eqn. 54 for the calculation of the effective column temperature.

$$T = \frac{163.22}{0.1566 + 0.188} = 473.66°\mathrm{K} = 200.50°\mathrm{C} \qquad (58)$$

The difference between the calculated effective temperature and that measured with a thermometer was 0.50°C. For a standard chromatograph, this agreement is acceptable.

As mentioned previously in connection with $t_M$ values, the curve for *n*-alkanes is linear only from *n*-heptane. The data of *n*-alkanes have to fullfil two conditions ($z \geqslant 7$ is one of them) if they are applied in the calculation of *b* values. Regarding the second condition, it is essential for the capacity ratio of the first-eluting *n*-alkane to be equal to or greater than unity.

Several researchers have investigated the linearity and the slope of the *n*-alkane

TABLE 22

TEMPERATURE DEPENDENCE OF THE SLOPE (*b*) OF THE *n*-ALKANE CURVE ON DIFFERENT STATIONARY PHASE (AFTER GARZÓ *et al.*[134])

| *Stationary phase* | *Column temperature (°C)* | | | | | | |
|---|---|---|---|---|---|---|---|
| | *40* | *60* | *80* | *100* | *120* | *140* | *160* |
| Apiezon L | 0.468 | 0.411 | 0.365 | 0.326 | 0.293 | 0.262 | 0.238 |
| SE-30 | 0.409 | 0.362 | 0.320 | 0.286 | 0.255 | 0.227 | 0.202 |
| QF-1 | 0.354 | 0.312 | 0.272 | 0.238 | 0.218 | 0.187 | 0.170 |

TABLE 23

COMPARISON OF SLOPES (*b*) ON SQUALANE AT DIFFERENT COLUMN TEMPERATURES MEASURED BY ETTRE AND BILLEB[131] AND CALCULATED BY EQN 50

| *Column temperature (°C)* | *b* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Calculated* | |
| 60 | 0 402 | 0.395 | 0 007 |
| 74 | 0 375 | 0.370 | 0 005 |
| 80 | 0 362 | 0.360 | 0 002 |
| 90 | 0 345 | 0.343 | 0 002 |

TABLE 24

*b* VALUES ON APIEZON N STATIONARY PHASE AT 150°C (AFTER GUERIN AND BANKS[93])

| *Stationary phase (%, w/w)* | *b** |
|---|---|
| 6 | 0.313 |
| 12 | 0 316 |
| 18 | 0 318 |
| 27 | 0 319 |

* Standard deviation = 0 003

curve as a function of stationary phases and column temperature. Some examples are presented in Tables 21–24.

The data in Table 21 show excellent conformity of the *b* values determined in various laboratories. Table 22 presents useful data for the calculation of the relationship between *b* and column temperature. The data in Table 23 illustrate the good agreement between the values calculated by eqn. 50 and those determined by Ettre and Billeb[131]. As shown in Table 24, the *b* value in GLC is independent of the amount of stationary phase if the support is inert[93].

For further details, see refs. 216, 280, 365, 452, 711, 1281 and 1306.

## 10. PEAK IDENTIFICATION AND/OR ELIMINATION WITH THE HELP OF RETENTION INDICES

Although other methods (GC–MS, etc.) are combined with retention index determinations for proving the presence or the absence of a particular material (the latter can be as important as the former), the problem nevertheless can be solved solely with retention indices. We have to measure retention indices on different stationary phases (at least three). The probability of peak coincidence in all three instances is minimal. It should be noted that the smaller the error of the determined retention index, the more reliable is the application for this purpose.

The following is an example of peak identification. In course of a gas chromatographic analysis of a hydrocarbon mixture on squalane as the stationary phase at 70°C, we have 612.9 i.u for the retention index of an unknown peak with ± 0.5

i.u. accuracy. We have found in our data bank that the following substances can be considered on the basis of the measured retention index under the given conditions 3-methyl-*cis*-2-pentene, $I^{sq}$(70°C) = 613.7 i.u.; 3-methyl-*trans*-2-pentene, $I^{sq}$(70°C) = 612.8 i.u.[206]; 1,3-hexadiene (*cis–trans*), $I^{sq}$(70°C) = 612.5 i.u.[206]; and 2,3-dimethyl-1,3-butadiene, $I^{sq}$(70°C) = 613.1 i.u. The retention indices of these compounds were listed by computer from the data bank. We obtained the following results. 3-methyl-*cis*-2-pentene, $I^{SE\text{-}30}$(70°C) = 615 i.u.[163], $I^{PEG\text{-}20M}$(70°C) = 660 i.u.[163], $I^{TCEP}$(70°C) = 766 i.u.[163]; 3-methyl-*trans*-2-pentene, $I^{SE\text{-}30}$(70°C) = 612 i.u.[163], $I^{PEG\text{-}20M}$(70°C) = 650 i.u.[163], $I^{TCEP}$(70°C) = 756 i.u.[163]; 1,3-hexadiene (*cis–trans*), $I^{SE\text{-}30}$(70°C) = 613 i.u., $I^{PEG\text{-}20M}$(70°C) = 648 i.u., $I^{TCEP}$(70°C) = 753 i.u.; and 2,3-dimethyl-1,3-butadiene, $I^{SE\text{-}30}$(70°C) = 614 i.u., $I^{PEG\text{-}20M}$(70°C) = 658 i.u., $I^{TCEP}$(70°C) = 763 i.u. Retention indices measured on squalane are omitted. We repeated the analysis of the mixture on PEG-20M and on TCEP at 70°C and obtained the following retention indices for the unknown peak with ±0.5 i.u. accuracy $I_s^{PEG\text{-}20M}$ (70°C) = 661.2 i.u.; $I_s^{TCEP}$ (70°C) = 765.7 i.u. Comparing the measured retention indices and those listed by computer, we can conclude that the unknown peak is 3-methyl-*cis*-2-pentene.

It should be noted that for most analyses of unknown mixtures we need a GC–MS system with a computer for the identification of peaks.

## 11. CONNECTION BETWEEN THE PHYSICO-CHEMICAL QUANTITIES AND RETENTION INDICES

In practice, the following relationship exists between the specific retention volume ($V_g$, cm$^3$ of carrier gas per gram of stationary phase) and the retention index ($I$):

$$\log V_g(\mathrm{s})_T = \left[ \frac{I_{\mathrm{s}}^{\mathrm{st\ ph}}\ (T) - 100z}{100} \right] \cdot b_T^{\mathrm{st.ph.}} + \log V_g(z) \qquad (59)$$

A useful relationship is available if the following data of the gas chromatographic system are known.

$$\log V_g(\mathrm{s}_2) = \left[ \frac{I_{\mathrm{s}_2}^{\mathrm{st\ ph}}\ (T) - I_{\mathrm{s}_1}^{\mathrm{st\ ph}}\ (T)}{100} \right] \cdot b_T^{\mathrm{st\ ph}} + \log V_g(\mathrm{s}_1) \qquad (60)$$

where $\mathrm{s}_1$ and $\mathrm{s}_2$ are two examined substances.

A well known relationship exists between the partition coefficient and the specific retention volume[1384]:

$$K = V_{g\rho}^{\mathrm{st\ ph}}\ T/273.16 \qquad (61)$$

where $K$ is the partition coefficient and $\rho$ is the density of the stationary phase. Similar simple relationships exist between the heat of dissolution and $V_g$ and between the partial molar free energy of solution and $V_g$. The equations are valid for a relatively narrow temperature range in GLC[1385]

$$\log V_g(s) = -\frac{\Delta H_{(s)}}{2.3RT} + c_H \tag{62}$$

where $\Delta H$ is the heat of solution, $R$ is the universal gas constant and $c_H$ is a constant, and

$$\log V_g(s) = -\frac{\Delta G(s)}{2.3RT} + c_G \tag{63}$$

where $\Delta G$ is the partial molar free energy of solution and $c_G$ is a constant if $T$ = constant.

Yufit *et al*[252] examined the relationships between various thermodynamic parameters and retention indices, also applying a sodium chloride support, and developed numerous interesting connections, *e g*,

$$I = 100\left(\frac{\Delta F_x^0 - \Delta F_z^0}{\Delta\Delta F_{\mathrm{CH}_2}^0} + z\right) \tag{64}$$

where $\Delta F^0$ is the free energy of solution and $\Delta\Delta F_{\mathrm{CH}_2}^0$ is the free energy of solution for a methyl group $= \Delta F_{z+1}^0 - \Delta F_z^0$.

Golovnya and Arsenyev[402–404] showed that

$$\Delta G(s)_T = -2.3RT\left[\left(\frac{I_s^{\mathrm{st\ ph}} - 100z}{100}\right)b + \log\left(\frac{V_g(z)T\rho}{273.16}\right)\right] \tag{65}$$

and

$$\Delta G^{\mathrm{CH}_2} = -2.3RTb \tag{66}$$

Since

$$\left(\frac{I(s_2) - I(s_1)}{100}\right)b = \log \alpha_{s_2 s_1} \tag{67}$$

where $\alpha$ is the relative volatility, we can write

$$\Delta G(s_2) - \Delta G(s_1) = 2.3RT \log \alpha_{s_2 s_1} \tag{68}$$

In addition to the paper by Hammers and De Ligny[412], one of their papers[403] is one of the best reviews of the field

The second thoroughly investigated field is the relationship between retention index and boiling point. This question was first examined by Kováts[1], who pointed out, ". if the difference in boiling points of two isomers is $\partial t_b$ then the difference in their retention indices (d$I$) on a non-polar stationary phase is given by

$$\mathrm{d}I \approx 5\partial t_b \tag{69}$$

where $\partial t_b$ is the difference in the boiling points of the two isomers". Several researchers dealt with these problems using Kováts' relationship between retention index and boiling point. We must emphasize the paper by Matukuma[208], who also investigated the connection between retention index and boiling point index:

$$I_B = 10^{(0.001340527T_b + 2.558916)} - 440.5 \tag{70}$$

where $I_B$ is the boiling point index (i u.) and $T_b$ is the boiling point (°K at 101.325 kPa). Many of his results have given an excellent basis for investigating relationships between molecular structure and boiling point, between retention index and boiling point index, etc

Mitra and Saha[211] plotted the logarithm of retention indices *versus* the reciprocal values of boiling points and obtained linear relationships on SE-30 as the stationary phase using saturated hydrocarbons as test substances.

Soják and co-workers[546–548,663,771] carried out the most detailed research work in this field We present one of their results, for the relationship between the boiling points of alkenes and their retention indices

$$T_b(s) = T_{b,1} + \frac{\mathrm{d}I\,(T_{b,2} - T_{b,1})}{100} \tag{71}$$

where $T_b(s)$ = boiling point of the alkene s; $T_{b,1}$ = boiling point of the *n*-alkene $z$; $T_{b,2}$ = boiling point of the *n*-alkane $z+1$; d$I$ = difference between the retention indices of the alkene s and the *n*-alkane $z$.

Some results are given in Tables 25 and 26.

The relationship worked out by Putman and Pu[74] between retention indices and molar refraction is also of interest. Some results are given in Table 27

Calixto and Raso[1318,1353] studied the correlations between Van der Waals

TABLE 25

COMPARISON OF BOILING POINTS MEASURED AND CALCULATED BY MATUKUMA[208]

| *Compound* | *Boiling point (°C)* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Calculated* | |
| 2,2-Dimethylpentane | 79.197 | 76.650 | 2.547 |
| 2-Ethylpentane | 94.000 | 93.900 | 0.100 |
| 4,4-Dimethylpentane | 72.518 | 70.240 | 2.278 |
| *n*-Octane | 125.665 | 125.740 | −0.075 |

TABLE 26

COMPARISON OF BOILING POINTS MEASURED AND CALCULATED BY SOJÁK *et al*[549]

| *Compound* | *Boiling point (°C)* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Calculated* | |
| 1-Ethyl-2-*n*-propylbenzene | 203.0 | 202.1 | +0.9 |
| *n*-Pentylbenzene | 205.5 | 205.6 | −0.1 |
| 1-Methyl-2-*n*-butylbenzene | 208.0 | 207.9 | +0.1 |

TABLE 27

RELATIONSHIP BETWEEN MOLAR REFRACTIONS AND RETENTION INDICES, MEASURED BY PUTNAM AND PU[74] ON APIEZON L AT 190°C

| *Compound* | *Retention index (i.u.)* | *Molar refraction* | |
|---|---|---|---|
| | | *Measured* | *Calculated* |
| Tetramethyltin | 630 | 36.73 | 36.75 |
| Trimethylvinyltin | 703 | 40.69 | 40.92 |
| Trimethylethyltin | 728 | 41.32 | 41.40 |
| Trimethylisopropyltin | 794 | 46.47 | 46.05 |
| Tetravinyltin | 911 | 52.99 | 53.43 |
| Tetraethyltin | 1049 | 55.38 | 55.34 |
| Tetra-*n*-propyltin | 1327 | 74.59 | 73.94 |

volume and the retention index on PEG-1440 and squalane as stationary phases. The influence of functional groups was quantified and a method for obtaining one general equation, valid for an indeterminate number of homologous series, was proposed.

We now discuss another important group of problems closely related to the relationship between retention index and physico-chemical parameters, which is the connection between molecular structure and retention index. For detailed information, see refs. 1, 74, 75, 208, 211, 218, 380, 459, 548, 600, 601, 748, 766, 786, 841, 864, 985, 1091, 1257, 1258, 1353a, 1358 and 1362

## 12 RETENTION INDEX AND MOLECULAR STRUCTURE

Kováts' paper[1] is a source also in this field; in this and later papers[4,6,25] all the important questions or their first germs are present. One of the most important fields of RIS is the relationship between retention index and molecular structure, and numerous investigators had dealt with theoretical and practical questions and achieved significant results.

When examining the relationship between molecular structure and retention index, this problem must clearly be distinguished from the study of molecular interactions These two concepts are often confused in the literature. If the relationship between molecular structure and retention index is studied, then all the investigations should be performed on a particular stationary phase at an isothermal column temperature On the other hand, for the study of molecular interactions, retention index data are needed that have been measured simultaneously on several (at least two) stationary phases at an isothermal column temperature or on a particular stationary phase at different column temperatures. The reason for the confusion is that the molecular structure also plays an important role in the formation of molecular interactions.

As mentioned above, the bases of the connection between molecular structure and retention index were established by Kováts[1,64] For example, if we subtract the values of the retention indices of the reference *n*-alkanes from those of 1-chloroalkanes measured on Apiezon L at 130°C by Kováts[1], we obtain the following results chloromethane 227, 1-chloroethane 235, 1-chloropropane 245, 1-chlorobutane 254, 1-chloropentane 254 and 1-chlorohexane 255 i.u. It is obvious from these results that the insertion of a chlorine atom in the molecule caused about a 250 i.u. change.

Another interesting occurrence was found by Kováts[64] on the basis of results measured by Evans and Smith[1386]: if the retention indices of two symmetrically substituted substances $s_1$ and $s_2$ are $I(R^x,R^x)$ and $I(R^0,R^0)$, then the retention index of the asymmetrically substituted compound $s_3$ is given by

$$I(R^x,R^0) = \frac{I(R^x,R^x) + I(R^0,R^0)}{2} \tag{72}$$

where the molecular structures are as follows:

$$\underset{s_1}{R^x\text{–}Q\text{–}R^x} \qquad \underset{s_2}{R^0\text{–}Q\text{–}R^0} \qquad \underset{s_3}{R^x\text{–}Q\text{–}R^0}$$

The exceptions are substances whose substituents can conjugate with the part Q of the molecule. In the demonstrated case, $Q = \text{–CO–O–}$, $R^x = CH_3\text{–}CH_2\text{–}$ and $R^0 = (CH_3)_2CH\text{–}$.

Kováts[1,6] first studied the relationship between molecular structure and retention index with retention index increments, and obtained good results. For example, the difference between the measured and calculated retention indices of nerol was 3 i.u. Similarly good results were obtained by Chovin[5], Baron and Maume[9] and Zulaica *et al.*[19].

Research into the relationship between molecular structure and retention index was aided by the determination of new retention index increments by Schomburg[49] and Janák *et al.*[60]. Rohrschneider[111] opened up new perspectives, and his paper will be discussed later.

Schomburg[113–115] continued his extensive investigations of molecular structure and retention index and introduced the so-called homomorph factor (H).

$$H^{\text{st.ph.}}(T) = I^{\text{st.ph.}}_{\text{substance}}(T) - I_{n\text{-alkane}} \tag{73}$$

Loewenguth[204] presented a series of retention index increments, and considered some further important problems connected with RIS (retention index data bank, etc.). Altenburg[229,230,288] considered the connection between molecular structure and retention index on the basis of physico-chemical parameters.

The results of Burkhard *et al*[235] on adamantane derivatives, Castello *et al.*[236] on alkyl iodides and Germaine and Haken[243,244] on esters were published at almost the same time and gave an important basis for studies of the relationship between molecular structure and retention index. The retention data publishing by Zarazir *et al.*[373] are also important for studies on the retention indices of alcohols, silyl ethers and acetals on different stationary phases at different column temperatures

Brooks and Harvey[386] studied the retention indices of corticosteroid boronates and Brooks *et al.*[388] extended this to steroidal drugs and derivatives.

Using published results, we put retention index increment studies on a new basis As discussed[446], the retention index was divided into three components, namely,

$$I^{\text{st ph}}_{\text{substance}}(T) = I_a + I_b + I_i^{\text{st ph}}(T) \qquad (74)$$

where $I$ = retention index (i u.) under isothermal conditions; $T$ = column temperature (°C or °K); $I_a$ = atomic index contribution (i.u.), $I_b$ = bond index contribution (i.u.), $I_i$ = interaction index contribution (i.u.).

Tóth[451] published his first results in the field of molecular structure and retention. Although he gave many suitable retention indices on different stationary phases at different column temperatures, his paper did not attract a great attention because his results were published in Hungarian only Unfortunately his later papers are also in Hungarian, so the results of his extensive research work [281,450–452,517,783–785] have become known to only a limited number of workers.

Berthou *et al.*[473], Cook and Raushel[486], Eisen *et al.*[490], Golovnya and co-workers[494–497], Haken and Khemangkorn[503], Karasek and co-workers[511,512], Mitooka[519–526], Paris and Alexandre[532], Ryba[537] and Singliar[544] have published hundreds of retention indices on different stationary phases at different column temperatures. The possibilities hidden in this enormous set of retention indices have not been completely utilized in studies on the connection between molecular structure and the retention index.

Weiner and co-workers[567–570] brought some new perspectives to this field by introducing factor analysis. Besson and Gäumann[578], Castello and co-workers[581–584], Hanny *et al*[608], Ladon[624], Rappoport and Gäumann[651], Schomburg and Dielmann[659,660], Soják *et al.*[663], Stuchlik *et al.*[665,666], Tomori[672] and Vilceanu and Schulz[678] increased the data basis and the set of retention index increments.

Chastrette and co-workers[585–588] presented a method involving topology information and gave examples of the utilization of their method in practice. Dubois and Chrétien[704] summarized the results of their research work on their topological system, which may be considered to be an important method. Peetre and co-workers[706,707,754–756], Engewald and co-workers[708,709], Pascal *et al*[752,753], Sakamoto *et al.*[765], Soják and co-workers[770,771] and Svob and co-workers[776,777] contributed to the examination of this important part of RIS with new results.

Our research group received significant help from Souter[773,774] (apart from his some small misunderstandings[779]), who pointed out the problems with our retention index increment concept Accepting his criticism, we gave up the horizontal expansion* of our retention index increment method[561,668] and began its critical revision and continued its vertical elaboration**.

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* Horizontal direction the application of vapor pressure index ($I^0$), the definition of the average interaction index contribution ($I_{ig}$), etc

** Vertical direction the dependence of the index increments on the column temperature, the pre-calculation of the index increments on different stationary phases at different column temperatures, etc.

Donike[800], Gaskell *et al.*[807], Gassiot *et al.*[808], Heintz *et al.*[818], Kecskés and Juricskay[821], Lafosse and Durand[828], Lindgren and Olsson[829], Moffat[837], Nau *et al.*[838], Pias and Gasco[843–845], Rang and co-workers[846,847], Sakamoto and co-workers[852,853], Santz *et al.*[854,855], Shlyakhov *et al.*[860], Vernon and Edwards[871,872] and West and Hall[875] presented many further retention indices measured on different stationary phases at different column temperatures Vanheertum[869] drew attention to the inaccuracy of some of our index increment values. Berthou *et al.*[881], Firpo *et al.*[896], Gal'Pern *et al.*[897], Ter Heide[912], Krupcik and co-workers[922–924], Menez *et al.*[935], Onuska and Comba[943], Popl *et al.*[947], Rang *et al.*[949–951], Riedo *et al.*[953], Ryba[958] and Soják and Rijks[964] have also contributed to the investigation of the connection between molecular structure and retention index with new retention indices.

Sanz *et al.*[960,961] described some new aspects of retention index increment calculations Albro *et al.*[979], Andersen and co-workers[980,981], Berninger and Möller[986], Castello and D'Amato[991], Dimov and Papazova[998], Grenier-Loustalot and co-workers[1015,1016,1188], Kerebel *et al.*[1028], Petersson[1039], Rang *et al.*[1042,1043], Sakamato and co-workers[1046,1047], Shats *et al.*[1053,1054], Soják *et al.*[1057–1059], Spivakovskii *et al.*[1060], Yabumoto *et al.*[1071], De Beer *et al.*[1077], Buryan and Macák[1086], Castello *et al.*[1087], Chastrette and Tagand[1088,1089], Chrétien and Dubois[1090], Dimov and Papazova[1091], Engewald *et al.*[1093], Stopp *et al.*[1151], Welsch *et al.*[1162], Grzybowski *et al.*[1259], Nabivach and Kirilenko[1275,1276], Ardrey and Moffat[1312], Czerwiec *et al.*[1321], Sellier *et al.*[1344,1345] and Shats *et al.*[1373] further investigated the connection between molecular structure and retention index, and published many retention indices and retention index increments. Kaliszan and Lamparczyk[1026,1109] investigated the relationship between retention index and connectivity index.

The different graphical methods ($I$ *versus* $z$, $I$ *versus* $I$, etc.) represent the other major area of investigation of the connection between molecular structure and retention index and some examples are given here.

The first group of papers are characterized by plots of retention indices *versus* physico-chemical parameters. The members of different homologous series generally represent model substances. Typical representatives of the group are papers of those by Iwata *et al.*[724] and Randíc[1132]

The second group of these works are represented by plots of retention indices *versus* carbon number, *e.g.*, the paper by Hoshikawa *et al.*[506].

The third group is formed by papers with plots of retention indices obtained in different way *versus* each other ($I$–$I$ plots) The so-called "roofing tile effect" was worked out by Walraven and co-workers[226,227,370] for alkanes which were given special code number, according to the number of the primary, secondary, tertiary and quaternary carbon atoms. For example, the 2-methylhexane has the code number 3310 because there are 3 primary, 3 secondary and 1 tertiary carbon atoms in its molecule Walraven[226] demonstrated that compounds with the same code numbers lie on the same line in an $I$ *versus* $I$ coordinate system It is well known that if two different points lie on the same line they satisfy the equation of the line

$$y = bx + a \tag{75}$$

where $x$ and $y$ are coordinates of the $I$ *versus* $I$ coordinate system, $b$ is the slope of the line and $a$ is the intercept on the ordinate

Substituting the coordinates of two points into eqn. 75, we can write

$$y_1 = bx_1 + a \tag{76}$$

and

$$y_2 = bx_2 + a \tag{77}$$

Subtracting eqn. 76 from eqn. 77, for the slope of line we obtain

$$b = \frac{y_2 - y_1}{x_2 - x_1} \tag{78}$$

and in an *I*–*I* coordinate system:

$$b = \frac{I(s_2)_2 - I(s_1)_2}{I(s_2)_1 - I(s_1)_1} \tag{79}$$

We present a numerical example using the retention indices of 2- and 3-methylhexane and 3-ethylpentane measured by Hively and Hinton[198] on squalane at 27 and 86°C (Table 28). Substituting retention index values into eqn. 79, we obtain

$$b_1 = \frac{685.6 - 675.9}{668.3 - 677.7} = 0.915 \tag{80}$$

$$b_2 = \frac{675.9 - 666.3}{677.7 - 667.2} = 0.914 \tag{81}$$

This means that the three compounds having the same code number really lie on same line. This method has importance in the field of qualitative gas chromatographic analysis and pre-calculation

The best summary of this field was given by Tóth[450] (unfortunately only in Hungarian).

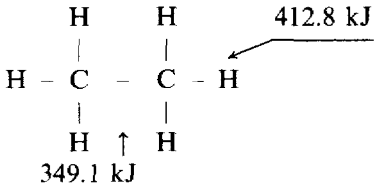
The connection between molecular structure and retention index has been very widely studied; an enormously valuable mass of knowledge and retention data has been collected that has not been critically evaluated.

Of the various methods available, it is suggested that the increment method is one of the most favourable. At present, with an insufficient number of results determined by numerous researchers, a universal increment method fails for the RIS, and its elaboration is a task for the near future.

Our research group has collected valuable experience on increments, and the results are summarized below. The basic research work on index increments is a simple reversible, computerized code system. This code system[931] is based on the chemical bond, including its primary and secondary environment[1321]. The bond index increments are calculated in retention index units (i.u.) from the bond energy by the following factor.

$$f_c = 0.018 \text{ i.u./kJ} \tag{82}$$

The energy of the bonds in the ethane molecule is as follows:



$$H_3C-CH_3$$

Thus, the bond index increment values of ethane can be calculated from the following equations:

$$i_b(\text{C–H}) = 0.018 \cdot 412.8 = 7.43 \text{ i.u.} \tag{83}$$

$$i_b(\text{C–C}) = 0.018 \cdot 349.1 = 6.28 \text{ i.u.} \tag{84}$$

and

$$I_b(\text{ethane}) = 6.28 + 6 \cdot 7.43 = 50.86 \text{ i.u} \tag{85}$$

The atomic and molecular index contributions of ethane can be calculated as follows:

$$I_a(\text{ethane}) = \frac{6 \cdot 1 + 2 \cdot 12}{10} = 3.00 \text{ i.u.} \tag{86}$$

and

$$I_M = I_a + I_b = 3.00 + 50.86 = 53.86 \text{ i.u.} \tag{87}$$

If the energy of the examined bond is unknown, then it can be calculated empirically using, *e.g.*, the bond distance. The following example shows the calculation of bond energy from the atomic distance of the $C_1$–$C_2$ bond in oestradiol:

$$E_{C_1-C_2} = \frac{71.463}{0.1390} = 514.1 \text{ kJ} \tag{88}$$

where $E_{C_1-C_2}$ is the bond energy of the $C_1$–$C_2$ bond, 71.463 (kJ · nm) is the quantum chemical conversion empirical factor for C–C aromatic bonds and 0.1390 nm is the $C_1$–$C_2$ bond length.

In addition to the bond index increment, the interaction index increment can be used in practical calculations. These increments allow the numerical examination of all the questions relating to retention of molecular structure, interrelationship between molecular structure and the stationary phase, effect of temperature, etc

Studies of retention index increments are based on the following equations:

$$I_s^{st.ph}\ (T) = I_a + I_b + I_i^{st\ ph}\ (T) = I_M + I_i^{st\ ph}\ (T) \tag{89}$$

where $I_a$ is the atomic index contribution,

$$I_a = \sum_{q=1}^{h} i_a(g) = \frac{\text{molecular weight}}{10} \tag{90}$$

$I_a$ is the atomic index increment:

$$i_a = \frac{\text{atomic weight}}{10} \tag{91}$$

and

$$I_M = I_a + I_b \tag{92}$$

where $g$ is a serial number, $h$ is the number of atoms in the molecule,

$$I_b = \sum_{g=1}^{q} i_b(g) \tag{93}$$

$i_b$ is the bond index increment and $q$ is the number of increments in the molecule.

Since

$$I_i^{st\ ph}\ (T) = I_{ig}^{st\ ph}\ (T) + I_{ii.}^{st\ ph}\ (T) \tag{94}$$

and

$$I_{ig}(T) = I_s^0(T) - I_a - I_b = I_s^0(T) - I_M \tag{95}$$

where $I_{ig}$ = general interaction index contribution, $I^0$ = vapour pressure index[125]; $T$ = column temperature (°C or °K); $I_i$ = interaction index contribution; $I_{ii}$ = individual interaction index contribution,

$$I_s^0(T) = 100\left[\frac{\log p^0(s) - \log p^0(z)}{\log p^0(z+1) - p^0(z)} + z\right] \tag{96}$$

$$I_{ig}(T) = \sum_{q=1}^{q} i_{ig}(g)_T \tag{97}$$

$i_{ig}$ = general interaction index increment, i.u.

$$I_{ii,s}^{st\,ph}(T) = \sum_{g=1}^{q} i_{ii}(g)_{T}^{st\,ph} \tag{98}$$

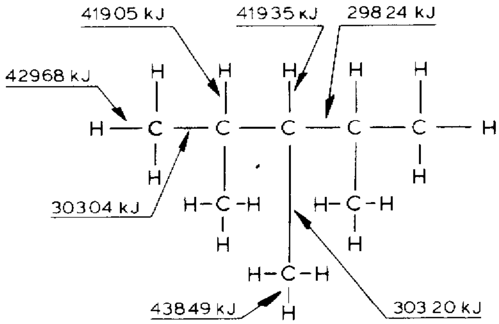
$$I_{ii,s}^{st\,ph.}(T) = I_{s}^{st.ph}(T) - I_{s}^{0}(\mathrm{T}) \tag{99}$$

For the elucidation of the complete calculation series, the determination of the data for 2,3,4-trimethylpentane is shown below

The molecular weight of 2,3,4-trimethylpentane (2,3,4-$M_3C_5$) is 114.0, so from eqn. 91 we have

$$I_a\,(2,3,4\text{-}M_3C_5) = \frac{114.0}{10} = 11.40 \text{ i.u.} \tag{100}$$

The bond energies of 2,3,4-trimethylpentane are as follows:



Thus, the bond index increment values can be calculated from the following equations:

$$i_b(\text{C1–H1}) = 0.018 \cdot 429.68 = 7.73 \text{ i.u.} \tag{101}$$

$$i_b(\text{C1–C2}) = 0.018 \cdot 303.04 = 5.45 \text{ i.u.} \tag{102}$$

$$i_b(\text{C2–H2}) = 0.018 \cdot 419.05 = 7.54 \text{ i.u.} \tag{103}$$

$$i_b(\text{C3–H31}) = 0.018 \cdot 419.35 = 7.55 \text{ i.u.} \tag{104}$$

$$i_b(\text{C3–C31}) = 0.018 \cdot 303.20 = 5.46 \text{ i.u.} \tag{105}$$

$$i_b(\text{C31–H331}) = 0.018 \cdot 438.49 = 7.89 \text{ i.u.} \tag{106}$$

$$i_b(\text{C3–C4}) = 0.018 \cdot 298.24 = 5.37 \text{ i.u.} \tag{107}$$

The bond index contribution ($I_b$) of 2,3,4-trimethylpentane can be calculated from eqn. 93 as follows:

$$I_b(2{,}3{,}4\text{-}M_3C_5) = 12 \cdot 7.73 + 4 \cdot 5.45 + 2 \cdot 7.54 + \\ + 2 \cdot 5.37 + 7.55 + 5.46 + 3 \cdot 7.89 = 177.06 \text{ i.u.} \tag{108}$$

Hence

$$I_M(2{,}3{,}4\text{-}M_3C_5) = I_a + I_b = 11.40 + 177.06 = 188.46 \text{ i.u.} \tag{109}$$

The calculation of the interaction index contribution using Apolane-87 as the stationary phase at 70°C, where the retention index of 2,3,4-trimethylpentane is 755.5 i.u.[953], can be effected with eqn. 89:

$$I^{\text{Apolane-87}}_{1,2,3,4\text{-}M_3C_5}\,(70°C) = I^{\text{Apolane-87}}_{2,3,4\text{-}M_3C_5}\,(70°C) - I_M(2{,}3{,}4\text{-}M_3C_5) \tag{110}$$
$$= 755.5 - 188.46 = 567.04 \text{ i.u.}$$

The general interaction index contribution ($I_{ig}$) can be calculated from eqns. 95 and 96 as follows:

$$I^0_{2,3,4\text{-}M_3C_5}\,(70°C) = 749.0 \text{ i.u.} \tag{111}$$

and

$$I_{ig,2,3,4\text{-}M_3C_5}\,(70°C) = I^0\,(70°C) - I_M \\ = 749.0 - 188.46 = 560.54 \text{ i.u.} \tag{112}$$

Hence from eqn. 94 we obtain

$$I^{\text{Apolane-87}}_{ii,2,3,4\text{-}M_3C_5}\,(70°C) = 567.04 - 560.54 = 6.50 \text{ i.u.} \tag{113}$$

Assuming a practically homogeneous distribution of the interactions in the molecule, which can of course be realized only approximately in reality because of orientation effects and other reasons (charge transfer, hydrogen bonding, etc.), we can write

$$I_b/I_{ig} = i_b(g)/i_{ig}(g) \tag{114}$$

and

$$I_b/I_{ii} = i_b(g)/i_{ii}(g) \tag{115}$$

TABLE 28

RETENTION INDICES MEASURED ON SQUALANE AT DIFFERENT COLUMN TEMPERATURES BY HIVELY AND HINTON[198]

| *Compound* | *Retention index (i.u)* | | *Walraven's code* |
|---|---|---|---|
| | *27°C* | *86°C* | |
| 2-Methylhexane | 666 3 | 667 2 | 3310 |
| 3-Methylhexane | 675 9 | 677.7 | 3310 |
| 3-Ethylpentane | 685 6* | 688 3 | 3310 |

* Corrected value

For example, let us calculate the interaction index increment values for the C3–H31 bond in 2,3,4-trimethylpentane using Apolane-87 as the stationary phase at 70°C.

$$177.06/560.54 = 7.55/i_{ig}\,(\text{C3–H31}) \qquad (116)$$

Therefore

$$i_{ig}\,(\text{C3–H31}) = \frac{560.54 \cdot 7.55}{177.06} = 23\ 90\ \text{i.u} \qquad (117)$$

$$177.06/6.50 = 7.55/i_{il}\,(\text{C3–H31}) \qquad (118)$$

Therefore

$$i_{il}\,(\text{C3–H31}) = \frac{6.50 \cdot 7.55}{177.06} = 0.28\ \text{i.u.} \qquad (119)$$

As the bond index contributions and/or the bond increment values do not depend on the stationary phase used or on the column temperature, it is possible to study the dependence of the interaction index increment on column temperature. Váradi and Tóth's experimental data[785] (Table 29) show the temperature dependence

TABLE 29

CHANGES IN THE INTERACTION INDEX INCREMENT ($i_i$) ON PEG-20M STATIONARY PHASE WITH COLUMN TEMPERATURE (AFTER VÁRADI AND TÓTH[785])

| *Column temperature (°C)* | *Interaction index increment (C–N) (i u )* |
|---|---|
| 100 | 76 79 |
| 120 | 77 66 |
| 140 | 78 61 |
| 160 | 79 44 |
| 180 | 80 83 |
| 200 | 82 13 |

of the interaction index increment ($i_i$) between the C–N bond of aniline using PEG-20M as the stationary phase.

For further information see refs. 61, 197, 224, 300, 301, 368, 370, 376, 377, 390, 416, 417, 429, 435, 440, 443, 480, 558, 578, 607, 638, 673, 674, 698–701, 859, 882, 890, 892, 913, 929, 955, 966, 967, 969, 990, 994, 1003–1007, 1010, 1022, 1026, 1033, 1037, 1053, 1054, 1067, 1068, 1093, 1175, 1176, 1200, 1224, 1246, 1315b, 1316, 1319, 1342, 1352 and 1365

## 13 ROHRSCHNEIDER'S CONCEPT AND CALCULATION METHOD THE ROHRSCHNEIDER EQUATION

Rohrschneider[111] gave a simple interpretation of the interactions between a sample substance and the stationary phase. The essence of the Rohrschneider's concept is that under the conditions of classical GLC, where the column temperature, carrier gas inlet and outlet pressures and flow-rate are constants during the whole analysis, the individual interaction of any substance can be approximately described by means of the interactions of a few selected reference substances, *e.g.*, benzene, ethanol, methyl ethyl ketone, nitromethane and pyridine, and the specific constants of the examined substance (Rohrschneider's constants), using the corresponding retention indices.

$$\Delta I_s = a^R x^R + b^R y^R + c^R z^R + d^R u^R + e^R s^R \tag{120}$$

(Rohrschneider's equation), where

$$\Delta I_s = I_s^p - I_s^{SQ} \tag{121}$$

p = polar stationary phase; SQ = squalane stationary phase; s = examined substance; $a^R$,$b^R$,$c^R$,$d^R$ and $e^R$ = substance-specific factors according to Rohrschneider[111].

$$x^R = \frac{\Delta I\ (\text{benzene})}{100} \tag{122}$$

$$y^R = \frac{\Delta I\ (\text{ethanol})}{100} \tag{123}$$

$$z^R = \frac{\Delta I\ (\text{methyl ethyl ketone})}{100} \tag{124}$$

$$u^R = \frac{\Delta I\ (\text{nitromethane})}{100} \tag{125}$$

$$s^R = \frac{\Delta I\ (\text{pyridine})}{100} \tag{126}$$

Table 30 gives the Rohrschneider constants of some stationary phases.

For example, let us calculate the $\Delta I$ value of toluene on SP-2401 at 100°C by eqn. 120 using Rohrschneider's constants (Table 30) and the substance specific factors of toluene[111] as follows: $a^R = 108.33$, $b^R = 3.77$; $c^R = 8.75$; $d^R = -7.01$ and $e^R = -7.61$. Hence,

$$\Delta I_{\text{toluene}}^{\text{SP-2401-SQ}}(100^\circ\text{C}) = 108.33 \cdot 1.41 + 3.77 \cdot 2.13 + 8.74 \cdot 3.55 + + (-7.11) \cdot 4.73 + (-7.61) \cdot 3.04 = 135.5 \text{ i.u.} \tag{127}$$

The measured retention index difference was 136.0 i.u.

The first task with the derived equation was the characterization of the polarity of stationary phases, and this is discussed in Section 14. Although the Rohrschneider equation is suitable for the characterization of stationary phase polarity, it is even more useful for predicting calculations (*cf.* ref. 111 and Table 41). The Rohrschneider equation is the only relationship in GLC that is applicable for the predicting calculation of individual relationships.

Rohrschneider himself emphasized the applicability of his equation for the examination of stationary phase polarity[214–216]. The best known work in this field was carried out by McReynolds[344], who was supported by Rohrschneider's work[111] and formed his system for the investigation of stationary phase polarity (McReynolds' system is discussed in detail in Section 14).

Supina and Rose[359] discovered the potential hidden in Rohrschneider's idea and determined the Rohrschneider constants of numerous stationary phases. Moreover, they demonstrated the possibilities of predicting calculations hidden in Rohrschneider equation. These results are included in Table 30.

Ashes and Haken[376] used Rohrschneider constants successfully to characterize the polarity of polysiloxane stationary phases. Table 31 gives Rohrschneider constants determined by Ashes and Haken[376] on different stationary phases.

Lorenz and Rogers[427] investigated the polarity of tri-, tetra-, penta-, hexa- and octaethylene glycol stationary phases using Rohrschneider constants. Weiner *et al.*[567] successfully used factor analysis when dealing with Rohrschneider's concept. Leary *et al.*[629] recognized the possibilities of the Rohrschneider concept and proposed to revise the Rohrschneider constants.

An important review of the Rohrschneider concept was made by Haken[813], who recognized that the column temperatures used by Rohrschneider (100°C) and McReynolds (120°C) were different to the commonly used temperatures. He has determined Rohrschneider constants at seven column temperatures on different stationary phases. These seven values can serve as an initial basis of a hyperbolic regression analysis by computer[528].

Hartkopf[718] and Hartkopf *et al.*[719] summarized the theoretical and practical questions of the Rohrschneider concept. Souter[772] studied the calculation of Rohrschneider constants and the geometrical interpretation of Rohrschneider's procedure.

The most successful research work on Rohrschneider's concept was carried out by Ashes and co-workers[683,879,982,983]. They compared their method[772] with other calculation methods and critically evaluated the results. The value of their results is not decreased by the fact that they did not recognize that the substance-

## TABLE 30

## ROHRSCHNEIDER CONSTANTS OF SOME STATIONARY PHASES USED IN GLC (AFTER ROHRSCHNEIDER[111] AND SUPINA AND ROSE[359])

Retention indices of Rohrschneider's constants on squalane at 100.0°C benzene = 649, ethanol = 384, methyl ethyl ketone = 531, nitromethane = 457 and pyridine = 695 i.u.

| *Stationary phase* | *x* | *y* | *z* | *u* | *s* |
|---|---|---|---|---|---|
| Acetyl tributylcitrate | 1.36 | 2.66 | 2.11 | 3.70 | 2.33 |
| Amine-220 | 1.07 | 3.88 | 1.63 | 2.80 | 2.14 |
| Apiezon L | 0.32 | 0.39 | 0.25 | 0.48 | 0.55 |
| Armeen 2HT | 0.24 | 0.96 | 0.29 | 0.51 | 0.36 |
| Armeen 2S | 0.35 | 0.45 | 0.37 | 0.57 | 1.03 |
| Armeen SD | 0.44 | 1.06 | 0.79 | 1.36 | 0.78 |
| Aroclor-1254 | 1.27 | 1.59 | 1.83 | 2.19 | 2.04 |
| $\beta,\beta'$-oxydipropionitrile | 5.88 | 8.48 | 8.14 | 12.58 | 9.19 |
| Bis-2-methoxyethyl adipate | 2.21 | 4.07 | 3.16 | 5.41 | 3.67 |
| Celanese ester No. 91 | 0.83 | 1.76 | 1.28 | 2.36 | 1.46 |
| Cyanoethylsucrose | 5.40 | 8.71 | 7.34 | 10.78 | 8.69 |
| DC-200 | 0.15 | 0.56 | 0.47 | 0.78 | 0.44 |
| DC-560 | 0.31 | 0.49 | 0.82 | 1.08 | 0.83 |
| DC-710 | 1.05 | 1.50 | 1.61 | 2.51 | 1.90 |
| DEGS | 4.93 | 7.58 | 6.14 | 9.50 | 8.37 |
| Dibutyl phthalate | 1.30 | 2.53 | 2.18 | 3.57 | 2.27 |
| Diethyl sebacate | 0.73 | 1.65 | 1.15 | 2.20 | 1.24 |
| Diisodecyl phthalate | 0.83 | 1.65 | 1.43 | 2.53 | 1.54 |
| Dinonyl phthalate | 0.84 | 1.76 | 1.48 | 2.70 | 1.53 |
| Emulphor ON-870 | 2.04 | 4.08 | 2.72 | 5.12 | 3.51 |
| Ethophat 60/26 | 1.78 | 3.79 | 2.48 | 4.74 | 3.20 |
| Ethylene glycol adipate | 3.43 | 5.46 | 4.52 | 7.11 | 6.00 |
| Ethylene glycol bis-cyanoethyl ether | 5.19 | 7.60 | 7.00 | 10.41 | 8.12 |
| Ethylene glycol isophthalate | 3.00 | 4.76 | 4.10 | 6.51 | 5.17 |
| Ethylene glycol succinate | 4.51 | 7.06 | 5.67 | 9.04 | 7.69 |
| FFAP | 3.21 | 5.79 | 4.11 | 7.17 | 6.45 |
| Fluoroluble GR-362 | 0.51 | 3.16 | 2.48 | 2.51 | 4.00 |
| Gastorwax | 1.05 | 2.72 | 1.80 | 2.66 | 2.56 |
| Hallcomid M-18 | 0.78 | 2.71 | 1.36 | 2.69 | 1.47 |
| Hallcomid M-18-01 | 0.96 | 2.93 | 1.59 | 2.99 | 1.77 |
| Halocarbon K-352 | 0.51 | 4.17 | 3.44 | 2.61 | 0.85 |
| 1,2,3,4,5,6-Hexakis(2-cyano-ethoxy)cyclohexane | 5.89 | 8.65 | 7.65 | 11.09 | 8.74 |
| Igepal CO-990 | 2.94 | 5.12 | 3.59 | 6.64 | 4.67 |
| JXR | 0.16 | 0.20 | 0.50 | 0.85 | 0.48 |
| LAC-2-R-446 (polyester) | 4.93 | 7.58 | 6.14 | 9.50 | 8.37 |
| LAC-4-R-886 (polyester) | 4.51 | 7.06 | 5.67 | 8.24 | 7.69 |
| Marlophene-87 | 1.78 | 3.72 | 2.53 | 4.49 | 3.34 |
| Marlophene-814 | 2.22 | 4.23 | 2.92 | 5.35 | 3.78 |
| MER-2 | 3.67 | 5.27 | 4.54 | 7.10 | 5.88 |
| MER-21 | 3.16 | 5.28 | 3.78 | 7.04 | 5.07 |
| MER-35 | 1.56 | 2.64 | 2.26 | 3.22 | 3.10 |
| Neopentyl glycol isophthalate | 2.07 | 3.56 | 3.15 | 4.99 | 3.77 |
| Neopentyl glycol succinate | 2.68 | 4.88 | 3.87 | 6.13 | 5.21 |
| OS-124 | 1.75 | 2.27 | 2.34 | 3.26 | 2.84 |
| OV-1 | 0.16 | 0.20 | 0.50 | 0.85 | 0.48 |
| OV-3 | 0.42 | 0.81 | 0.85 | 1.52 | 0.89 |

TABLE 30 (*continued*)

| *Stationary phase* | *x* | *y* | *z* | *u* | *s* |
|---|---|---|---|---|---|
| OV-7 | 0.70 | 1.12 | 1.19 | 1.98 | 1.34 |
| OV-11 | 1.13 | 1.57 | 1.69 | 2.70 | 1.95 |
| OV-17 | 1.30 | 1.66 | 1.79 | 2.83 | 2.47 |
| OV-22 | 1.58 | 1.80 | 2.04 | 3.27 | 2.59 |
| OV-25 | 1.76 | 2.00 | 2.15 | 3.34 | 2.81 |
| OV-101 | 0.16 | 0.20 | 0.50 | 0.85 | 0.48 |
| OV-210 | 1.41 | 2.13 | 3.55 | 4.73 | 3.04 |
| OV-225 | 2.17 | 3.20 | 3.33 | 5.16 | 3.69 |
| PEG-20M | 3.18 | 5.33 | 3.81 | 7.02 | 5.04 |
| PEG-4000 | 3.22 | 5.46 | 3.86 | 7.15 | 5.17 |
| Phenyldiethanolamine succinate | 3.61 | 6.24 | 4.70 | 7.24 | 6.32 |
| Polyphenyl ether (5-ring) | 1.75 | 2.27 | 2.34 | 3.26 | 2.84 |
| Polypropylene sebacate | 1.93 | 3.38 | 2.58 | 4.36 | 3.27 |
| QF-1 | 1.09 | 1.86 | 3.00 | 3.94 | 2.41 |
| Quadrol | 2.00 | 5.62 | 3.47 | 5.24 | 4.65 |
| Reoplex-400 | 3.56 | 5.75 | 4.44 | 7.43 | 5.95 |
| SAIB | 1.73 | 2.36 | 2.65 | 4.39 | 2.96 |
| SE-30 | 0.16 | 0.20 | 0.50 | 0.85 | 0.48 |
| SP-392 | 1.37 | 1.73 | 1.87 | 2.85 | 2.23 |
| SP-400 | 0.33 | 0.49 | 0.82 | 1.08 | 0.83 |
| SP-2401 | 1.41 | 2.13 | 3.55 | 4.73 | 3.04 |
| Squalane | 0 | 0 | 0 | 0 | 0 |
| STAP | 0.89 | 2.56 | 1.64 | 2.44 | 2.35 |
| Tergitol NPX | 1.94 | 3.96 | 2.68 | 4.82 | 3.49 |
| Tetracyanoethylated pentaerythritol | 5.11 | 7.65 | 6.79 | 9.93 | 8.17 |
| Tricresyl phosphate | 1.74 | 3.22 | 2.58 | 4.14 | 2.95 |
| Trimer acid | 0.89 | 2.73 | 1.64 | 2.06 | 3.68 |
| 1,2,3-Tris-2-cyanoethoxypropane | 6.00 | 8.71 | 7.94 | 11.53 | 9.40 |
| Triton X-100 | 2.01 | 4.09 | 2.80 | 4.99 | 3.61 |
| Tween-80 | 2.14 | 4.20 | 2.78 | 5.20 | 3.65 |
| XE-60 | 2.08 | 3.85 | 3.62 | 5.33 | 3.45 |
| XE-61 | 0.98 | 1.30 | 1.57 | 2.38 | 1.85 |
| XF-1150 | 2.86 | 4.80 | 4.49 | 6.82 | 4.89 |
| Ucon 550X (PPG) | 1.14 | 2.76 | 1.68 | 3.12 | 2.08 |
| Zonyl E-91 | 1.34 | 2.59 | 3.38 | 4.03 | 2.98 |

specific factors were independent of the stationary phases with respect to the standard deviation of the experimental data (Table 31)

Vigdergauz and Bankovskaya[970] discovered an interesting connection between the Rohrschneider polarity factors and the specific selectivity values.

Csizmadia[995] investigated the Rohrschneider constants of potassium-treated PEG-2000 stationary phase and found $x^R = 2.69$; $y^R = 5.11$; $z^R = 3.38$; $u^R = 6.53$; $s^R = 4.40$. Evans and Osborn[1253] studied the effect of oxidation on the polarity of some stationary phases with help of Rohrschneider constants.

Tekler and Takács[1299] demonstrated new possibilities of the Rohrschneider concept and found some new fields of application. They demonstrated theoretically and experimentally that the Rohrschneider concept could apply to retention indices also, not only to the differences between them:

TABLE 31

EFFECT OF EXPERIMENTAL PARAMETERS ON THE SUBSTANCE-SPECIFIC FACTORS DETERMINED BY ASHES *et al*[683]

| *Stationary phase* | *Substance-specific factor* | | | | |
|---|---|---|---|---|---|
| | $a^R$ | $b^R$ | $c^R$ | $d^R$ | $e^R$ |
| Squalane | −19.63* | 0.74* | 12.97* | −1.94* | 0.68* |
| | −20.285 | 0.389 | 12.331 | −1.151 | 0.753 |
| DC-200 | −21.034 | 0.331 | 12.180 | −0.714 | 0.938 |
| Apiezon L | −21.028 | 0.316 | 12.163 | −0.700 | 0.945 |
| DC-710 | −22.151 | 7.099 | 16.614 | −4.792 | −3.059 |
| Polyphenyl ether | −24.772 | 3.223 | 12.767 | −0.100 | −0.490 |
| Polypropylene sebacate | −20.085 | 3.320 | 11.779 | −2.318 | −0.292 |
| Carbowax 20M | −22.870 | 2.459 | 15.528 | −3.897 | 1.210 |
| Diethylene glycol succinate | −21.537 | 0.169 | 11.865 | −0.934 | 1.979 |
| TCEP | −21.292 | 0.130 | 11.865 | −0.450 | 1.195 |

* Determined by Rohrschneider[111].

$$I_s^{\mathrm{st\,ph}}\,(T) = \sum_{i=1}^{5} I^{\mathrm{st\,ph}}\,(T)\,(i)\,s_i^* + C_R^*\,(T) \tag{128}$$

where $I(i)$ is the retention index of the $i$th standard substance, $s_i^*$ is the $i$th substance specific factor and $C_R^*$ is the Rohrschneider system constant of eqn 128.

Eqn. 128 has great importance in predicting calculations of retention indices. Calculation using eqn. 128 can be illustrated with Tekler's data[1299] as follows:

$$\begin{aligned} b_{100C}^{SP} &= 0.284 \cdot (-0.0004) + 0.320 \cdot 1.2186 + 0.279 \cdot (0.0287) + \\ &+ (-0.3903) + 0.281 \cdot (0.0001) + 0.037 = 0.3068 \end{aligned} \tag{129}$$

The measured $b$ value was 0.3071.

Our research group turned its attention to Rohrschneider's work[111,214–216] and began a broad research study in this field. This work opened up new possibilities for the Rohrschneider concept. It was stated that the concept is generally valid in the GLC and its application in the limited range of GLC is fair, with neither theoretical nor practical obstacles (Rohrschneider in his original equation[111] included retention indices determined at a column temperature of 100°C). It was found that the equation, in accordance with the concept, can be set up with the help of other substances, but it seems advisable to bear in mind Rohrschneider's original selection, which is correct from the viewpoints of both theory and practice. This was done by McReynolds[344] when be selected $n$-butanol instead of ethanol, 2-pentanone instead of methyl ethyl ketone and nitropropane instead of nitromethane. It could also be established from Rohrschneider's work[111] that the minimum number of standard substances is three, the optimum is five but the latter is definitely not the upper limit. For example, McReynolds[344] used ten standard substances.

During research work with the aim of examining the general validity of Rohrschneider's concept, it was found that the reference squalane stationary phase can be replaced by other phases (it is well known that the applicable temperature range of squalane stationary phase is narrow, 25–125°C); moreover, the retention index determined on squalane stationary phase can be replaced, for example, by the value of the vapour pressure index.

The most interesting result of the research work emerged from a series of experiments in which the Rohrschneider concept was used with the ratio of retention indices[360]:

$$I_s^{P}(T) = I_s^{SQ}(T) \sum_{i=1}^{5} f_i s_i \qquad (130)$$

where $i$ = serial number of standards; $f_i = \left(\frac{I^P}{I^{SQ}}\right)_i$, $i$th polarity factor; $s_i$ = $i$th substance-specific factor.

The sum of the substance-specific factors was almost 1, and therefore

$$\sum_{i=1}^{5} s_i = 1.000 \qquad (131)$$

Eqn. 131 was checked with a computer using the possible combinations of numerous retention indices, and if the retention indices were correct, no exceptions were found. The results have been published in several papers[510,557,560,667,669,965].

As an example[557], the calculation of the retention index of *n*-butanol on OV-210 at 120°C using eqn. 130 is as follows

$$\begin{aligned} I^{\text{OV-210}}_{n\text{-butanol}}\,(120^\circ\text{C}) &= 590\,[(1.57097 \cdot -0.31763) + (1.44349 \cdot 0.18757) + \\ &+ (1.29855 \cdot 1.42712) + (1.16993 \cdot 0.66505) + (1.05964 \cdot -0.93935)] \\ &= 830.5 \text{ i.u.} \end{aligned} \qquad (132)$$

where 590 i.u. represents $I^{SQ}_{n\text{-butanol}}$ (120°C). The value as measured by McReynolds[344] was 828.0 i.u.

Let us calculate the sum of substance-specific factors by eqn. 131 as follows:

$$\begin{aligned} \sum_{1}^{5} s_i &= -0.31763 + 0.18757 + 1.42712 + 0.66505 \\ &- 0.93935 = 1.02276 \end{aligned} \qquad (133)$$

The difference between the theoretical and calculated values is 0.02276.

Summarizing this theme, we can say that the most important results of the RIS beginning from its discovery are Rohrschneider's concept[111], its mathematical equations and its calculation methods. See also ref. 429.

## 14 McREYNOLDS SYSTEM. POLARITY OF STATIONARY PHASES

A significant contribution to the further advance of Rohrschneider's concept is that of McReynolds[344]. McReynolds first assumed that ten standard substances may be necessary in order to characterize the polarities of stationary phases. However, on the basis of practical investigations it was concluded that the following five standard substances are sufficient· benzene, *n*-butanol, 2-pentanone, nitropropane and pyridine.

The polarity of stationary phases affects their capability of forming individual relationships. The polarity is immeasurable *per se*, and therefore certain standard substances are used for this purpose (Rohrschneider's standards, McReynolds' standards). With respect to the temperature dependence of the retention indices of the substances used, the polarity values determined with their help are also dependent on the column temperature.

·Theoretically[407], using the five standard substances, the following equation describes the individual interaction-forming capacity (polarity) of the stationary phase:

$$P = \sum_{i=1}^{5} [I^{\mathrm{st\,ph}}(T) - I^{0}(T)]\,i \qquad (134)$$

where $P$ (i.u.) is the polarity of the examined stationary phase. In practice, on squalane as the stationary phase the individual interactions can be neglected, and therefore instead of eqn. 134 the following relationship is valid

$$P_{\mathrm{Mc}} = \sum_{i=1}^{5} [I^{\mathrm{st.ph.}}(T) - I^{\mathrm{SQ}}(T)]\,i \qquad (135)$$

where $P_{\mathrm{Mc}}$ (i u.) is the polarity of the examined stationary phase according to McReynolds[344].

For the characterization of stationary phase polarity, the retention indices of the five standard substances are used, according to eqn. 135. For example, the calculation of the polarity of SP-2250 stationary phase at 120°C using the McReynolds system and eqn. 135 as follows·

$$P^{\mathrm{SP\text{-}2250}}_{120^{\circ}\mathrm{C}} = 119 + 158 + 162 + 243 + 202 = 884 \text{ i.u.} \qquad (136)$$

A series of publications dealing with McReynolds' system began with a paper by Preston[348a]. He made a statistical summary of stationary phases using in 1968 and 1969 and established that there were about 300 different stationary phases in use.

Of these, only 21 stationary phases were used by 20 or more researchers

To Preston's paper were added Haken's[411] and Schupp's[441] remarks, which Preston[436] replied to, and Clutton's[485], and Henly's[610] papers, Bierl *et al*[474], Neff *et al.*[529], Szentirmai and co-workers[557,560], Weiner and Parcher[570], Ashes and Ha-

TABLE 32

McREYNOLDS CONSTANTS OF VARIOUS PHENYL METHYL SILICONES (AFTER ETTRE[711])

| *Stationary phase* | *Phenyl content (mole-%)* | *x'* | *y'* | *z'* | *u'* | *s'* |
|---|---|---|---|---|---|---|
| SE-52 | 5 | 32 | 72 | 65 | 98 | 67 |
| OV-3 | 10 | 44 | 86 | 81 | 124 | 88 |
| OV-7 | 20 | 69 | 113 | 111 | 171 | 128 |
| DC-550 | 25 | 74 | 116 | 117 | 178 | 135 |
| OV-11 | 35 | 102 | 142 | 145 | 219 | 178 |
| OV-17 | 50 | 119 | 158 | 162 | 243 | 202 |
| OV-22 | 65 | 160 | 188 | 191 | 283 | 253 |
| OV-25 | 75 | 178 | 204 | 208 | 305 | 280 |

ken[574], Ettre[595], Keller[618] and Mann and Preston[632] dealt with different theoretical and practical problems of the McReynolds' system

Both theoretically and practically the most important paper connected with the standard stationary phases was published by Huber and Kováts[614]. This work seems to be the precursor of the Apolane-87 stationary phase and of the theoretically based concept of standard mixed stationary phases.

Leary *et al* [628] proposed twelve stationary phases (preferred stationary phases) on the basis of the McReynolds system using the nearest neighbour technique The preferred stationary phases are Squalane, SE-30, OV-3, OV-7, DC-710, OV-22, QF-1, XE-60, Carbowax 20M, DEGA, DEGS and TCEP Their paper gave effective help to practical specialists in finding a way through the jungle of stationary phases.

Novák *et al.*[642] published a thermodynamically formulated concept of the polarity of gas chromatographic stationary phases. Rohrschneider[656] has dealt with the chromatographic characterization of stationary phases and solutes for column selection and identification Supina[666a] discussed interesting topics from the point of view of practical work. He pointed out that the McReynolds constants of the following stationary phases are virtually identical· OV-101, SP-2100, DC-200, SF-96, SE-30, E-301, JXR and UCW-982.

Wold and Andersson[680] gave the most comprehensive evaluation of the McReynolds system. They evaluated all the ten solutes on 226 stationary phases, and gave the data for the pre-calculation of retention indices and stationary phases that cause large deviations.

Ettre[711] evaluated the more important questions in connection with the characterization of stationary phases. Table 32 gives the McReynolds constants of different phenyl-containing stationary phases after Ettre[711].

Hartkopf[718] and Hartkopf *et al.*[719] have summarized the more important problems of the Rohrschneider concept and the McReynolds system. Lowry *et al* [736] described a nearest neighbour technique applied to the reduction of the number of standard solutes needed to characterize gas chromatographic stationary phases. McCloskey and Hawkes[797] evaluated the McReynolds system with a two-dimensional representation. Hawkes *et al* [816] summarized the results in connection with preferred stationary phases Vernon and Edwards[871] gave the McReynolds constants of some fluorinated stationary phases

Wold[876] analysed the similarities and dissimilarities between stationary phases by means of pattern recognition. Chastrette[884] evaluated McReynolds data with a two-dimensional plot and factor analysis.

Parcher and Westlake[945] described polarity programmed GLC. Riedo *et al* [953] discussed the physico-chemical parameters of Apolane-87. They measured the retention indices of many different compound types at different column temperatures and also Rohrschneider and McReynolds constants

Tijssen *et al.*[968] used the solubility parameter for predicting selectivity and retention in GLC. Vigdergauz and Bankovskaya[970] dealt with linear analogues of McReynolds polarity factors of preferred stationary phases West and Hall[972] considered the prediction of resolution from retention indices as an aid to column selection and presented a considerable number of results. Burns and Hawkes[988] reported the dispersion interaction and the polarity and acid–base properties of preferred stationary phases.

Delley and Friedrich[997] presented their GC 72 system, which is based on four preferred stationary phases: OV-101, OV-17, OV-225 and Carbowax 20M. Golovnya and Misharina[1014] reported the characterization of the selectivity of stationary phases from the partial molar free energies of solution of McReynolds standards. Grenier-Loustalot *et al.*[1015] studied different Carbowax stationary phases, and Van Lenten *et al* [1031] investigated McReynolds constants of different polysiloxane stationary phases, Vernon and Ogundipe[1069] different hydrocarbon stationary phases and Chastrette and Tagand[1088,1089] bifunctional stationary phases

Evans[1093a] investigated different theoretical and practical problems of stationary phases[397,491,596,893] and discussed the molecular retention index as an alternative system for the characterization of stationary phases Fellous *et al* [1095] compared the different methods of classification of stationary phases Lindsay Smith *et al* [1147] published a method for classifying the selectivity of porous polyaromatic beads with McReynolds constants. Snyder[1148] dealt with the classification of the solvent properties of common liquids. Vernon and Gopal[1156] determined McReynolds' constants on different stationary phases at 180°C Evans and Osborn[1253] investigated the effects of oxidation on some stationary phases when measuring the retention indices of McReynolds standards.

Golovnya and Misharina[1257 1257a] gave a critical review of this important field from the thermodynamic viewpoint.

Based on the Rohrschneider concept and the McReynolds system, our research group introduced the so-called retention polarity[967]:

$$\bar{P}_{\mathrm{R}}(T) = 20 \sum_{i=1}^{5} \left[ \frac{\Delta I(T)}{\bar{I}^{\mathrm{SQ}}(T)} \right]_i \tag{137}$$

where $\bar{P}_{\mathrm{R}}$ is the retention polarity (polarity units, p.u.). Thus

$$\bar{P}^{\mathrm{OV\text{-}330}}_{\mathrm{R}(120^{\circ}\mathrm{C})} = 20\left(\frac{222}{653} + \frac{391}{590} + \frac{273}{627} + \frac{417}{652} + \frac{368}{699}\right) = 52.08 \text{ p.u} \tag{138}$$

Some retention polarity values are listed in Table 33.

TABLE 33

RETENTION POLARITY OF SOME STATIONARY PHASES[967] AT 120°C, ON THE BASIS OF McREYNOLDS CONSTANTS

| *Stationary phase* | *Retention polarity (p.u.)* | *Stationary phase* | *Retention polarity (p.u.)* |
|---|---|---|---|
| Squalane | 0.00 | OV-202 | 47.18 |
| Apolane-87 | 2.30 | SP-2401 | 47.18 |
| Apiezon L | 4.39 | OV-215 | 47.95 |
| SE-30 | 6.80 | O-330 | 52.08 |
| OV-1 | 6.95 | NPGA | 56.63 |
| SP-2100 | 7.17 | NPGS | 65.23 |
| SE-52 | 10.42 | PEG-20M | 70.25 |
| SP-400 | 10.67 | SP-1000 | 75.02 |
| OV-3 | 13.17 | PEG-1000 | 79.84 |
| OV-7 | 18.39 | PEG-1500 | 80.59 |
| SP-1200 | 22.08 | EGA | 82.61 |
| OV-17 | 27.40 | Reoplex-400 | 85.09 |
| SP-2250 | 27.40 | SP-2310 | 99.31 |
| OV-22 | 33.29 | DEGS | 106.63 |
| OV-25 | 35.86 | SP-2340 | 114.39 |
| OS-138 | 38.06 | SP-216-PS | 122.53 |
| SP-1220 | 39.35 | TCEP | 128.91 |
| QF-1 | 45.99 | BCEF | 144.60 |

Eqn. 137 is suitable in practice, but it is not correct theoretically. The correct equation for the retention polarity is

$$\bar{P}_{Rc}(T) = 20 \sum_{i=1}^{5} \left[ \frac{I^{st\,ph}(T) - I^{0}(T)}{I^{0}(T) - I_{M}} \right]_{i} \tag{139}$$

where $I^0$ = vapour pressure index (i.u.), $I_M$ = molecular index contribution (i.u.); $\bar{P}_{Rc}$ = corrected retention polarity (p.u.).

The corrected retention polarity ($\bar{P}_{Rc}$) also has a physical meaning. it indicates the average individual interaction formed on a stationary phase as a percentage of the general interaction.

The most important aspect of these problems is the use of mixed stationary phases. Over many years several investigators have dealt with the theoretical and practical problems of mixed stationary phases[27,33,343,391,424,532,831,1115]. The three large series of mixed stationary phases are connected with the work of Molera *et al.*[1387–1389], Purnel and co-workers[1390–1392] and Gröbler and Bálizs[1189,1190,1330]. For further information, see also refs. 66, 85, 203, 249, 330, 348a, 407, 575, 597, 613a, 629, 648, 655, 710, 831, 885, 921, 967, 1011, 1012, 1061, 1093a, 1094, 1094a, 1115, 1155, 1157, 1159, 1160, 1189, 1190, 1315, 1343, 1357, 1360 and 1370.

TABLE 34

COMPARISON OF RETENTION INDICES ON SQUALANE AT 70°C MEASURED BY TOURRES[161] AND CALCULATED BY EQN. 140

| *Compound* | *Walraven's[226] code* | *Retention index (i.u.)* | | *Difference* |
|---|---|---|---|---|
| | | *Measured* | *Calculated* | |
| 2,3-Dimethylheptane | 4320 | 856.1 | 856.7 | −0.6 |
| 2,4-Dimethylheptane | 4320 | 821.9 | 823.2 | −1.3 |
| 2,5-Dimethylheptane | 4320 | 833.5 | 834.8 | −1.3 |
| 2,6-Dimethylheptane | 4320 | 827.7 | 829.0 | −1.3 |
| 3,5-Dimethylheptane | 4320 | 834.8 | 835.1 | −0.3 |
| 3,4-Dimethylheptane | 4320 | 860.0 | 859.6 | 0.4 |
| 2-Methyl-4-ethylbenzene | 4320 | 825.4 | 825.3 | 0.1 |
| 2-Methyl-3-ethylbenzene | 4320 | 845.8 | 844.4 | 1.4 |
| 3-Methyl-4-ethylbenzene | 4320 | 857.9 | 854.9 | 3.0 |

## 15. PRE-CALCULATION OF RETENTION INDICES

The desire to pre-calculate retention data is almost as old as GLC itself. The results obtained were very significant, and the regularities developed ensured the progress of this research field for a long time ahead. Many variations of such calculations are known which proved to be adequate in practice. In all instances it is a precondition that the calculations should be carried out using reliable retention indices.

The pre-calculation of retention indices can be divided into three main groups: (1) the application of various plots, (2) calculations based on molecular structure and (3) calculations based on Rohrschneider's concept. Examples are presented below to illustrate the main fields listed above.

The retention indices determined by Tourres[161] at various column tempera-

TABLE 35

COMPARISON OF RETENTION INDICES OF METHYLUNDECANES ON SQUALANE AT 100.0°C MEASURED AND CALCULATED BY SCHOMBURG AND HENNEBERG[220]

| *Compound* | *Retention index (i.u.)* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Calculated* | |
| 2,7-Dimethylundecane | 1215.8 | 1217.6 | −1.8 |
| 2,8-Dimethylundecane | 1221.2 | 1222.6 | −1.4 |
| 2,9-Dimethylundecane | 1232.6 | 1233.6 | −1.0 |
| 2,10-Dimethylundecane | 1227.3 | 1228.0 | −0.7 |
| 3,8-Dimethylundecane | 1226.8 | 1228.2 | −1.4 |
| 3,9-Dimethylundecane | 1238.1 | 1239.2 | −1.1 |
| 4,4-Dimethylundecane | 1214.6 | 1217.2 | −2.6 |

TABLE 36

COMPARISON OF RETENTION INDICES ON SQUALANE AT 80.0°C MEASURED AND CALCULATED BY PIRINGER[349]

| *Compound* | *Retention index (i.u.)* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Calculated* | |
| 2-Methylpropane | 366.0 | 363.2 | +2.8 |
| 2-Methylbutane | 475.5 | 476.2 | −0.7 |
| 2,2-Dimethylpropane | 413.4 | 413.0 | +0.4 |
| 2-Methylpentane | 570.3 | 570.2 | +0.1 |
| 3-Methylpentane | 686.0 | 685.8 | +0.2 |
| 2,3-Dimethylhexane | 762.0 | 762.4 | −0.4 |
| 2,2,3,3-Tetramethylbutane | 753.0 | 755.8 | −2.8 |

TABLE 37

COMPARISON OF RETENTION INDICES OF HYDROCARBONS AT 80°C ON SQUALANE MEASURED BY DIMOV *et al*[487a] WITH CALCULATED VALUES

| *Compound* | *Retention index (i.u.)* | | | *Difference (i.u.)* | |
|---|---|---|---|---|---|
| | *Exptl.* | *PCI** | *Calc* | *Exptl − PCI* | *Exptl − Calc* |
| 2-Methylnonane | 964.0 | 965.7 | 964.0 | −1.7 | 0.0 |
| 3-Methylnonane | 970.4 | 970.6 | 971.1 | −0.2 | −0.7 |
| 4-Methylnonane | 960.4 | 959.5 | 959.2 | +0.9 | +1.2 |
| 5-Methylnonane | 957.4 | 956.8 | 958.6 | +0.6 | −1.2 |
| 2,2-Dimethyloctane | 917.9 | 916.6 | 915.2 | +1.3 | +2.7 |
| 2,3-Dimethyloctane | 953.1 | 952.0 | 953.5 | +1.1 | −0.4 |
| 2,4-Dimethyloctane | 916.0 | 916.3 | 917.3 | −0.3 | −1.3 |
| 2,5-Dimethyloctane | 922.8 | 922.4 | 923.4 | +0.4 | −0.6 |
| 2,6-Dimethyloctane | 932.8 | 931.8 | 933.9 | +1.0 | −1.1 |
| 2,7-Dimethyloctane | 929.5 | 930.5 | 931.5 | −1.0 | −2.0 |

* PCI = physico-chemical index

TABLE 38

COMPARISON OF RETENTION INDICES MEASURED AND CALCULATED BY WEINER AND HOWERY[568] ON DIFFERENT STATIONARY PHASES AT 100.0°C

| *Compound* | *Stationary phase* | *Retention index (i.u.)* | | *Difference* |
|---|---|---|---|---|
| | | *Measured* | *Calculated* | |
| Toluene | DC-200 | 767 | 768 | −1.0 |
| | PEG-20M | 1066 | 1065 | +1.0 |
| 1-Propanol | Squalane | 485 | 486 | −1.0 |
| | DC-200 | 548 | 548 | 0 |
| | Apiezon L | 523 | 523 | 0 |
| | PEG-20M | 1021 | 1022 | −1.0 |
| | Reoplex-400 | 1062 | 1064 | −2.0 |
| | DEGS | 1240 | 1241 | −1.0 |

TABLE 39

COMPARISON OF MEASURED AND PRE-CALCULATED RETENTION INDICES ON DC-550 STATIONARY PHASE AT 150°C ACCORDING TO EVANS[596]

| *Compound* | *Retention index (i.u.)* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Pre-calculated* | |
| Anisole | 1006.6 | 1006.1 | +0.5 |
| Mesitylene | 1037.2 | 1037.5 | −0.3 |
| N,N-Dimethylaniline | 1195.0 | 1193.9 | +1.1 |

tures on squalane as the stationary phase can be used as an example to demonstrate the application of different graphical plots. From $I^{SQ}$ (30°C) *versus* $I^{SQ}$ (70°C) plots, a straight line was selected, the equation of which is

$$I^{SQ}(70^\circ C) = 1.036 \cdot I^{SQ}(30^\circ C) - 27.5 \tag{140}$$

Walraven's code[226] of the straight line is 4320. The calculated and measured data are given in Table 34.

Same examples of predicting calculations based on molecular structure are shown in Tables 35–40.

TABLE 40

COMPARISON OF THE $I^{PPG}-I^{SQ}$ VALUES AT 80°C BY SCHOMBURG AND DIELMANN[660]

| *Compound* | $I^{PPG}-I^{SQ}$ *(i.u.)* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Calculated* | |
| 1,7-Octadiene | 56.0 | 55.3 | +0.7 |
| 5-Hexenylcyclopropane | 59.3 | 58.4 | +0.9 |
| 5,6-Methylenehexylcyclopropane | 61.2 | 61.5 | −0.3 |
| 5-Methylhexylcyclopropane | 27.4 | 27.1 | +0.3 |
| 4-Methylhexylcyclopropane | 29.5 | 29.6 | −0.1 |

Although graphical presentation and pre-calculation on the basis of molecular structure give numerous opportunities for the pre-calculation of retention indices, the practical application of Rohrschneider's concept using a computer has the greatest potential. Based on Rohrschneider's data[111], some retention indices were compared with measured values (Table 41).

Two calculation series are presented, based on McReynolds' data[344], from the results of our research group. *n*-Butanol was the substance to be examined and benzene, 2-pentanone, nitropropane, pyridine and 2-methyl-2-pentanol were used as

TABLE 41

COMPARISON OF MEASURED AND CALCULATED RETENTION INDICES OF 2-ETHYL-1-HEXENE AT 100°C ON DIFFERENT STATIONARY PHASES (AFTER ROHRSCHNEIDER[111])

| *Stationary phase* | *Retention index (i.u.)* | | *Difference* |
|---|---|---|---|
| | *Measured* | *Calculated* | |
| Squalane | 780 | – | – |
| DC-200 | 788 | 792 | −4 |
| Apiezon L | 783 | 779 | 4 |
| Diethylhexyl sebacate | 796 | 796 | 0 |
| Celanese ester No. 9 | 799 | 799 | 0 |
| Diisodecyl phthalate | 799 | 799 | 0 |
| DC-710 | 806 | 808 | −2 |
| QF-1 | 807 | 805 | 2 |
| PPG | 803 | 802 | 1 |
| Acetylbutyl citrate | 814 | 818 | −4 |
| Tricresyl phosphate | 820 | 821 | −1 |
| Polyphenyl ether | 821 | 823 | −2 |
| Marlophen-87 | 817 | 816 | 1 |
| Polypropylene sebacate | 823 | 824 | −1 |
| Marlophen-814 | 826 | 825 | 1 |
| NPGS | 841 | 843 | −2 |
| XF-1150 | 844 | 843 | 1 |
| PEG-20M | 847 | 847 | 0 |
| PEG-4000 | 848 | 848 | 0 |
| Reoplex-400 | 858 | 859 | −1 |
| DEGS | 890 | 892 | −2 |
| Ethylene glycol biscyanoethyl ether | 898 | 900 | −2 |
| 1,2,3-Tris-2-cyanoethoxypropane | 916 | 919 | −3 |

*standard substances. The equation for the ratio of retention indices*[360,557,965] *has* the following form:

$$I^{\text{st ph}}_{n\text{-butanol}}\,(120^\circ\text{C}) = 590 \sum_{i=1}^{5} f_i s_i \qquad (141)$$

where $s_1 = -0.56112$, $s_2 = -0.45521$, $s_3 = 0.49807$, $s_4 = 0.48717$ and $s_5 = 1.03353$;

$$f_1 = \frac{I_1^{\text{st ph}}(120^\circ\text{C})}{653}\ (\text{benzene}) \qquad (142)$$

$$f_2 = \frac{I_2^{\text{st ph}}(120^\circ\text{C})}{627}\ (\text{2-pentanone}) \qquad (143)$$

$$f_3 = \frac{I_3^{\text{st ph}}(120^\circ\text{C})}{652}\ (\text{nitropropane}) \qquad (144)$$

$$f_4 = \frac{I_4^{\mathrm{st\ ph}}(120^\circ\mathrm{C})}{699}\ \text{(pyridine)} \tag{145}$$

$$f_5 = \frac{I_5^{\mathrm{st.ph.}}(120^\circ\mathrm{C})}{690}\ \text{(2-methyl-2-pentanol)} \tag{146}$$

The Tekler equation[1299] for retention indices is as follows

$$I_{n\text{-butanol}}^{\mathrm{st\ ph}}(120^\circ\mathrm{C}) = \sum_{i=1}^{5} I_i^{\mathrm{st.ph}}\ s_i^* \tag{147}$$

where $s_1^* = -0\ 50700$, $s_2^* = -0.42836$, $s_3^* = 0.45072$, $s_4^* = 0.41121$ and $s_5^* = 0\ 88378$; $i$ = serial number of standards 1 = benzene, 2 = 2-pentanone, 3 = nitropropane, 4 = pyridine and 5 = 2-methyl-2-pentanol In this instance the Rohrschneider system constant was zero[1299].

As substance-specific factors must satisfy theoretically the original Rohrschneider equation (eqn. 120), we can write

$$\Delta I_s(T) = \sum_{i=1}^{5} \Delta I(i) s_i^* \tag{148}$$

Eqn 148 makes calculations easier, as the McReynolds system contains $\Delta I$ values.

The pre-calculation of $\Delta I$-value on Reoplex-400 at 120°C by using eqn. 148 is illustrated as follows

$$\Delta I_{n\text{-butanol}}^{\text{Reoplex-400}}(120^\circ\mathrm{C}) = 364 \cdot (-0\ 50700) + 449 \cdot (-0.42836) +$$

$$+ 647\quad 0.45072 + 671\quad 0.41121 + 482 \cdot 0\ 88378 = 616\ 7\ \text{i.u} \tag{149}$$

The measured retention index difference obtained by McReynolds[344] was 619.0 i.u.

Summarizing the main results of the field discussed, it can be said that to fulfill scientific accuracy in the pre-calculation of retention indices Tekler's equation[1299] has to be applied; this equation is based on Rohrschneider's concept. However, for practical calculations, it seems to be sufficient to apply the version reduced to three terms and system constants.

For further details, see refs. 35, 36, 66, 113, 115, 125, 169, 172, 178, 179, 220, 221, 339, 443, 532, 581, 584, 607, 667, 669, 679, 698, 700, 721, 722, 765, 776, 777, 859, 860, 875, 885, 887, 918, 919, 968, 972, 979, 998, 1001, 1060, 1096, 1254, 1315, 1339, 1356 and 1377

## 16 CALCULATION OF RETENTION INDICES BY PROGRAMMABLE CALCULATOR OR COMPUTER

It seems unbelievable today, but in the first half of the 1960s the spread of RIS

TABLE 42

REFERENCES TO RETENTION INDICES OF DIFFERENT COMPOUNDS

| *Compound type* | *References* |
|---|---|
| Acetylenes | 181, 1143 |
| Acetals | 112, 503, 1350b |
| Acids | 238, 256, 429, 454, 507, 590, 661, 697, 714, 793, 869, 871, 935, 975, 1039, 1210, 1298a, 1298b, 1351 |
| Acid derivatives | 21, 52, 115, 119, 121, 165, 238, 243, 248, 274, 277, 286, 373, 377, 503, 514, 652, 661, 714, 765, 814, 850, 852, 853, 899, 906, 922, 953, 959, 982, 983, 1007, 1012, 1046, 1047, 1056, 1065, 1077, 1111, 1236, 1244, 1255 |
| Acetates | 238 |
| Acylglycines | 1286 |
| Adamantanes | 235, 280, 319, 385, 725, 829, 929, 930, 1201 |
| Alcohols | 1, 6, 9, 15, 21, 23, 39, 46, 54, 55, 62, 79, 84, 89, 103, 111, 125, 133, 138, 165, 169, 170, 178, 183, 238, 277, 345, 373, 455, 544, 567, 767, 824, 844, 913, 953, 991, 1026, 1060, 1087, 1088, 1173, 1191, 1232, 1253, 1256, 1303, 1315, 1315b, 1317, 1350b, 1353a, 1357, 1373 |
| Aldehydes and ketones | 1, 15, 21, 39, 53, 80, 94, 101, 110, 111, 118, 119,. 121, 133, 138, 144, 153, 165, 172, 183, 196, 238, 246, 247, 277, 345, 474, 554, 574, 585, 889, 910, 953, 1191, 1256, 1293, 1302, 1315b, 1348, 1353a, 1357 |
| Alkaloids | 118, 1120, 1121 |
| Alkynes | 949, 953 |
| Alkanes | 33, 34, 38, 39, 53, 55, 57, 63, 78, 84, 88, 89, 94, 111, 113, 114, 120, 130, 141, 155, 160, 161, 163, 165, 181, 198, 204, 205, 206, 208, 211, 212, 214, 220, 221, 222, 223, 225, 227, 230, 288, 301, 354, 412, 419, 428, 437, 439, 462, 476, 493, 506, 519, 536, 545, 550, 555, 611a, 624, 646, 654, 659, 701, 733, 757, 759, 761, 770, 854, 859, 871, 872, 885, 911, 953, 960, 961, 994, 1011, 1043, 1060, 1080, 1097, 1162, 1170, 1173, 1175, 1176, 1194, 1223, 1235, 1256, 1276, 1279, 1294, 1295, 1296, 1314, 1315, 1317, 1338, 1339, 1342, 1346, 1349, 1353, 1361c |
| Alkenes | 33, 34, 38, 39, 56, 63, 78, 113, 141, 157, 160, 178, 179, 181, 198, 204, 205, 206, 221, 223, 333, 339, 372, 439, 474, 490, 520, 531, 534, 536, 537, 546, 548, 588, 611a, 622, 625, 651, 659, 663, 695, 770, 795, 891, 911, 953, 994, 1042, 1097, 1127, 1151, 1162, 1173, 1212, 1234, 1235, 1342, 1249, 1256, 1320, 1338, 1349 |
| Alkynes | 181, 198, 531, 534, 949, 950, 951, 1077 |
| Amides | 825 |
| Amines | 46, 92, 232, 238, 245, 413, 488, 565, 713, 785, 800, 811, 907, 976, 977, 996 |
| Amino acids | 248, 1359 |
| Aminonitriles | 1076 |
| Anilines | 95, 238, 785, 1086, 1271 |
| Arenes | 1233 |
| Aromatic compounds | 3, 6, 9, 23, 25, 38, 42, 45, 51, 53, 56, 58, 65, 78, 79, 80, 84, 89, 90, 91, 95, 111, 114, 119, 123, 125, 127, 135, 139, 165, 170, 172, 192, 196, 198, 212, 223, 224, 237, 263, 275, 314, 331, 332, 393, 407, 423, 424, 443, 454, 459, 467, 483, 486, 493, 500, 511, 512, 517, 518, 519, 536, 549, 574, 602, 624, 673, 674, 701, 703, 761, 776, 777, 786, 820, 875, 947, 953, 964, 892, 998, 1057, 1058, 1059, 1093, 1109, 1173, 1240, 1268, 1276, 1277, 1303, 1339, 1349, 1357, 689, 731, 841, 843, 844, 845, 863, 868, 872, 910, 923, 927, 1003, 1077, 1097, 1106, 1149, 1161, 1166, 1169, 1225, 1233, 1271, 1278, 1328, 1345, 1347, 1348, 1365 |

(*Continued on p. 272*)

TABLE 42 (*continued*)

| *Compound type* | *References* |
|---|---|
| Barbiturates | 1377 |
| Bile acids | 1350 |
| Biphenyls | 443, 923, 979, 1324, 1335, 1364 |
| Carbohydrates | 27, 323, 417, 453, 510, 1379 |
| Cyanides | 89, 183 |
| Cycloalkanes | 63, 114, 157, 206, 301, 412, 428, 442, 536, 708, 752, 847, 866, 883, 890, 896, 1003, 1008, 1016,, 1022, 1030, 1043, 1091, 1093, 1097, 1173 |
| Cycloalkenes | 24, 114, 157, 206, 387, 393, 531, 536, 654, 659, 660, 701, 847, 908, 1003, 1008, 1151 |
| Dialkylthiocarbamates | 1205 |
| Drugs | 388, 580, 744, 832, 837, 986, 1092, 1197, 1329, 1365a |
| Essential oils | 3, 8, 25, 26, 47, 76, 159, 177, 260, 383, 449, 460, 497, 609, 836, 1154, 1327, 1352 |
| Esters | 3, 6, 15, 18, 19, 21, 23, 25, 26, 27, 36, 39, 42, 49, 53, 55, 58, 89, 101, 112, 115, 144, 158, 197, 243, 244, 287, 345, 373, 376, 377, 416, 455, 661, 672, 698, 713, 765, 853, 857, 913, 1026, 1077, 1088, 1089, 1124, 1173, 1182, 1198, 1244, 1256, 1316, 1317, 1326, 1332, 1333, 1351, 1361b, 1363 |
| Ethers | 80, 144, 238, 274, 345, 373, 377, 391, 503, 828, 928, 1350b, 1376 |
| Furans | 60, 80, 98, 374 |
| Halogenated compounds | 1, 30, 34, 56, 82, 89, 111, 178, 225, 236, 280, 329, 337, 373, 423, 424, 464, 471, 498, 511, 512, 577, 581, 582, 625, 654, 724, 795, 828, 871, 872, 885, 891, 936, 943, 953, 1019, 1253, 1300, 1321, 1334 |
| Heterocyclic compounds | 60, 374, 451, 498, 517 |
| Hydrazones | 1302 |
| Hydrocarbons | 88, 160, 358, 445, 454, 483, 500, 540, 578, 643, 674, 688, 740, 741, 846, 853, 860, 979, 985, 1134, 1138 |
| Hydroxyl-containing compounds | 1, 21, 238, 283, 336, 345, 373, 391, 474, 484, 497, 511, 542, 562, 579, 603, 631, 685, 723, 733, 767, 809, 913, 953, 991, 1026, 1060 |
| Indoles | 281, 451, 517, 1122 |
| Ketones | 1, 9, 21, 42, 53, 80, 97, 138 |
| Lutidine | 212 |
| Mycotoxins | 1313a, 1350a |
| Mercaptans | 251 |
| Naphthalenes | 1237a, 1245a, 1313, 1315a, 1352a |
| Nitro-containing compounds | 82, 89, 99, 118, 133, 145, 152, 281, 498, 514, 518, 535, 676, 686, 780, 953, 976, 977, 1000, 1045, 1121, 1356 |
| Nucleosides | 1083 |
| Olefins see alkenes | |
| Organotins | 74, 75, 134, 488, 506, 707 |
| Oxazines | 173, 175 |
| PAHs | 127, 368, 454, 500, 1082, 1345 |
| Pesticides | 943 |
| Paraffins: see alkanes | |
| Peptides | 477 |
| Pharmaceutical substances | 235, 319, 375, 477, 591, 617, 734, 837, 838, 888, 935, 839, 971, 986 |
| Phenols | 80, 149, 295, 518, 800, 927, 928, 989, 1066, 1085, 1124, 1259, 1271, 1345, 1362, 1378 |
| Phosphorus-containing compounds | 590, 678 |
| Piperazines | 1104 |
| Polymers | 509, 552, 554, 640, 1029, 1065, 1164 |
| Porphyrins | 1366, 1367, 1368 |

TABLE 42 (*continued*)

| *Compound type* | *References* |
|---|---|
| Prostaglandins | 963, 1044, 1084, 1099, 1301 |
| Pyrans | 98, 212 |
| Pyrazines | 1104 |
| Pyridines | 141, 1033, 1086, 1278, 1280 |
| Pyridopyrimidines | 1266, 1369b, 1377a |
| Pyrimidazes | 1232 |
| Pyrroles | 145 |
| Saccharides | 1308 |
| Selenium-containing compounds | 74, 611, 1335a |
| Silanes | 706, 707, 754, 755, 756 |
| Steroids | 100, 232, 386, 388, 400, 458, 473, 487, 694, 768, 807, 808, 814, 821, 848, 880, 881, 900, 914, 1002, 1028, 1047, 1081, 1130, 1171, 1369 |
| Sterols | 914, 1002, 1024, 1075 |
| Sulphur-containing compounds | 259, 314, 340, 462, 484, 494, 496, 514, 566, 608, 750, 765, 809, 810, 870, 897, 898, 901, 904, 905, 996, 1032, 1101, 1102, 1103, 1133, 1185, 1186, 1205, 1257 |
| Terpenes | 9, 14, 47, 68, 79, 104, 156, 159, 197, 231, 289, 290, 429, 513, 532, 907, 912, 976, 977, 980, 981 |
| Tetralins | 283 |
| Tyrosines | 1371 |
| Toxicological materials | 1287, 1312, 1340 |
| Toxins | 1298, 1313a |
| Uridines | 1284 |
| Xanthine derivatives | 1248 |

was hindered by the presence of logarithms in the basic equation Owing to the rapid spread of desktop computers and calculators, programs were prepared and published not only for the calculation of retention indices but also for all the further calculations needed (pre-calculation, $t_M$ calculation, etc ). Nowadays data banks are organized for the storage of retention indices (for example, Hewlett-Packard), and these may facilitate the identification or elimination of peaks carried out with the application of retention indices.

See also refs. 390, 508, 528, 639, 647, 699, 705, 715, 746, 922, 923, 926, 965, 1025, 1064, 1065, 1080, 1152 and 1361.

## 17 UNIVERSALITY OF THE RETENTION INDEX SYSTEM

The greatest significance of the RIS is that it permits results obtained in other laboratories to be used. This universal role is hindered by different data presentations, including other "index" systems. We consider that the propagation of any system the results of which are not convertible into retention index values retards the development of the universal system, because it divides the forces and divers attention from this important question. However, we cannot deny the restricted advantages of other systems in special cases.

To simplify the work, retention indices are grouped in Table 42 based on the type of chemical compound and in Table 43 based on the type of stationary phase used

TABLE 43

REFERENCES TO RETENTION INDICES MEASURED ON DIFFERENT STATIONARY PHASES

| *Stationary phase* | *References* |
|---|---|
| Acetophenone | 455 |
| Acetyl butyl citrate | 885, 1057 |
| Acetyltributyl citrate | 111 |
| Amin 220 | 1086 |
| Apiezon J | 748 |
| Apiezon L | 1, 6, 9, 14, 23, 46, 47, 51, 65, 67, 68, 74, 75, 78, 79, 97, 98, 104, 111, 113, 114, 115, 125, 134, 165, 169, 173, 175, 177, 231, 274, 283, 289, 290, 295, 319, 340, 345, 416, 417, 424, 429, 431, 443, 462, 486, 490, 518, 532, 580, 581, 582, 600, 650, 661, 673, 706, 754, 756, 767, 824, 859, 872, 873, 875, 883, 888, 912, 949, 976, 977, 994, 1011, 1054, 1069, 1104, 1144, 1173, 1243, 1267, 1297, 1335 |
| Apiezon M | 138, 177, 402, 412, 554, 706, 707, 755, 809, 810, 851, 905, 1014, 1069, 1101, 1102, 1103, 1193, 1244, 1257 |
| Apiezon M, hydrogenated | 1193, 1241, 1253 |
| Apiezon T | 1069 |
| Apiezon W | 1069 |
| Apolane 87 | 953, 1019, 1075, 1171, 1187, 1193, 1244, 1253, 1265, 1296, 1297, 1357 |
| $\beta,\beta'$-Oxydipropionitrile | 67, 169 |
| $\beta,\beta'$-Thiodipropionitrile | 67, 169 |
| Benzophenone | 455 |
| Bentone 34 | 329 |
| Bis(2-ethylhexyl)tetrachlorophthalate | 537 |
| Butanediol 1,4-succinate polyester (BDS) | 27 |
| Chromosorb 102 | 274 |
| DC-200 | 67, 111, 115, 220, 259, 314, 329, 1351 |
| DC-401 | 165 |
| DC-535 | 574 |
| DC-550 | 145, 314, 428, 574 |
| DC-702 | 1303 |
| DC-710 | 33, 111, 231, 911, 1031 |
| DEGA | 859, 1153 |
| DEGS | 80, 165, 231, 289, 290, 416, 431, 474, 514, 824, 1124, 1144, 1244, 1315 |
| Dexsil 300 | 765, 852, 853, 1024, 1047, 1301, 1371 |
| Dexsil 400 | 1347 |
| Dexsil 410 | 1347 |
| Dibutyl phthalate | 53, 67, 462 |
| Didecyl phthalate | 67 |
| Diethylene glycol succinate | 111 |
| Diethylhexyl sebacate | 111 |
| Diethylhexyl phosphinate | 1338 |
| Diisodecyl phthalate | 30, 111, 1016 |
| Di-*n*-butyltetrachlorophthalate | 537, 958 |
| Dinonyl phthalate | 39, 53, 54, 55, 63, 67, 138 |
| Dimethylsulpholane | 34, 38, 227, 230, 994 |
| EGA | 1137 |

TABLE 43 (*continued*)

| *Stationary phase* | *References* |
|---|---|
| EGS | 213, 429, 1124 |
| EGSSX | 417, 698, 757, 913, 1022, 1026 |
| Emulphor O | 1, 6, 33, 47, 49, 65, 78, 79, 114, 157, 163, 424, 429 |
| Ethylene glycol biscyanethylether | 111 |
| Fluorolube 2000 | 871 |
| Marlophen 87 | 111 |
| Marlophen 817 | 111 |
| Mixed phases | 521, 523, 524, 525, 648, 831, 849, 965, 849, 1011, 1056, 1093, 1161, 1174, 1189, 1190, 1223, 1245a |
| NGA | 1259 |
| NGS | 821 |
| Nitrile silicone | 891 |
| NPGA | 18, 36, 42, 49, 111 |
| NPGS | 1356 |
| OS 138 | 27, 113, 115, 868, 1042, 1065 |
| OV-1 | 237, 295, 376, 377, 458, 477, 500, 503, 562, 575, 580, 638, 880, 986, 1039, 1092, 1130, 1175, 1179, 1248, 1266, 1280, 1284, 1286, 1287, 1300, 1312, 1313a, 1315, 1342, 1343, 1349, 1361c, 1366, 1376, 1377a |
| OV-3 | 844, 979 |
| OV-7 | 487, 844, 875, 935, 1191, 1340 |
| OV-17 | 196, 232, 287, 376, 377, 387, 388, 431, 458, 474, 477, 487, 500, 503, 575, 580, 638, 672, 713, 720, 765, 810, 844, 852, 853, 869, 904, 905, 908, 939, 975, 979, 986, 1024, 1039, 1047, 1056, 1082, 1083, 1092, 1101, 1102, 1103, 1109, 1122, 1130, 1154, 1191, 1210, 1244, 1248, 1266, 1280, 1286, 1301, 1313, 1324, 1326, 1336, 1340, 1345, 1351, 1352a, 1357, 1371, 1377a |
| OV-22 | 1191 |
| OV-25 | 287, 376, 503, 695, 844, 875, 881, 897, 908, 927, 1011, 1056, 1083, 1109, 1173, 1191, 1248, 1266, 1332, 1352a, 1369b, 1377a |
| OV-73 | 1314 |
| OV-101 | 248, 487, 800, 881, 879, 1056, 1210, 1220, 1225, 1246, 1281, 1308, 1313, 1324, 1328, 1336, 1356, 1357, 1361c, 1363 |
| OV-210 | 376, 377, 388, 503, 575, 875, 881, 879, 1083, 1324, 1357 |
| OV-215 | 1315a |
| OV-225 | 474, 503, 695, 906, 979, 1007, 1243, 1259, 1324, 1357 |
| OV-275 | 1077 |
| PEG* | 49, 169, 393, 439, 443 |
| PEG 20M* | 14, 21, 57, 62, 111, 118, 119, 144, 156, 158, 163, 164, 178, 231, 235, 290, 333, 373, 429, 451, 510, 542, 562, 581, 674, 698, 703, 767, 785, 912, 913, 991, 1022, 1026, 1065, 1071, 1144, 1154, 1170, 1234, 1263, 1237a, 1267, 1271, 1297, 1315, 1335, 1339, 1342, 1356, 1359, 1369b |
| PEG 200* | 183 |
| PEG 400* | 45, 67, 89, 115, 125, 169, 183, 462, 624, 991, 1153, 1339 |
| PEG 600 | 1150, 1303 |
| PEG 1000* | 183, 402, 496, 767, 809, 811, 904, 905, 907, 976, 977, 991, 1014, 1048, 1101, 1102, 1103, 1104 |
| PEG 1500* | 39, 67, 125, 171, 225, 281, 544, 767, 795, 824, 1361 |
| PEG 1540* | 84, 88, 345, 748, 1315b, 1316, 1317 |
| PEG 4000* | 68, 104, 111, 115, 125, 169, 171, 490, 531, 946, 995, 1042, 1043 |
| PEG 6000* | 535 |

(*Continued on p 276*)

TABLE 43 (*continued*)

| *Stationary phase* | *References* |
|---|---|
| PEGA | 138, 247, 672, 1066, 1379 |
| PEGS | 520 |
| Pluronic F 68 | 1327 |
| PPG-425 | 1104 |
| Polyphenyl ether | 111, 1042 |
| Polypropylene sebacate | 111 |
| Porous polymers | 238, 277, 1334 |
| Polypropylene glycol | 27, 49, 111, 125, 169, 223 |
| QF-1 | 111, 119, 134, 231, 431, 498, 814, 871, 872, 1024, 1039, 1191, 1303 |
| Reoplex 400 | 9, 49, 111, 125, 169, 171, 1170 |
| SDEG | 373 |
| SE-30 | 18, 19, 21, 33, 36, 80, 95, 134, 163, 169, 177, 178, 179, 211, 231, 235, 243, 244, 273, 280, 281, 287, 314, 354, 368, 427, 451, 474, 486, 517, 518, 528, 585, 672, 698, 713, 741, 785, 808, 814, 821, 837, 844, 850, 859, 875, 906, 913, 975, 1007, 1012, 1022, 1024, 1026, 1056, 1071, 1083, 1126, 1171, 1175, 1187, 1243, 1246, 1259, 1287, 1302, 1304, 1308, 1312, 1315a, 1328, 1332, 1339, 1340, 1345, 1358, 1376, 1377, 1379 |
| SE-33 | 1315a |
| SE-52 | 127, 169, 314, 488, 518, 1124, 1210, 1313, 1314, 1315a |
| SE-54 | 1315a, 1359 |
| SF-96 | 156, 231, 290, 513, 1170, 1220 |
| Silar 5CP | 906, 1007, 1012, 1173 |
| Silar 10C | 906, 1007, 1332 |
| Silicone fluid nitrile XF-1150 | 111 |
| SP-2100 | 1315, 1350 |
| SP-2250 | 1315 |
| Squalane | 39, 49, 54, 56, 67, 89, 94, 111, 114, 115, 125, 133, 157, 160, 161, 169, 198, 204, 205, 206, 208, 211, 221, 225, 230, 263, 275, 301, 319, 331, 345, 354, 393, 439, 455, 462, 486, 490, 506, 520, 531, 575, 624, 659, 663, 672, 701, 708, 757, 761, 776, 795, 828, 849, 875, 885, 890, 891, 964, 991, 994, 995, 1003, 1011, 1057, 1069, 1091, 1165, 1137, 1150, 1151, 1162, 1175, 1180, 1187, 1193, 1240, 1249, 1253, 1263, 1275, 1279, 1298, 1315b, 1316, 1338, 1339, 1353, 1357, 1365 |
| TCEP | 34, 163, 1339 |
| Tricresyl phosphate | 111, 236, 675 |
| 1,2,3-Tris(2-cyanoethoxy)propene | 33, 111, 911 |
| Tritolyl phosphate | 67 |
| Triton X-305 | 809, 810, 811, 904, 905, 907, 976, 977, 1101, 1102, 1103 |
| Tween 80 | 101, 1170 |
| Ucon 5100 | 125 |
| Ucon 50 HB 2000 | 169 |
| Ucon 50 HB 5100 | 169 |
| Ucon 50 HB 280X | 171, 890, 1077, 1151 |
| Ucon HB 2000 | 80, 125 |
| Ucon 50 LB 1200X | 263, 345 |
| Ucon oil LB 550X | 80, 123, 223, 703, 1008, 1077, 1151, 1170, 1179, 1240, 1339, 1356 |
| XE-60 | 287, 376, 431, 503, 638, 756, 975, 1077, 1144, 1191, 1324 |
| XF-1150 | 49, 175, 376, 377, 503, 575 |

* PEG = Carbowax

## 18. LIST OF SYMBOLS USED

| | |
|---|---|
| $\alpha$ | relative volatility |
| $A_{\rm L}$ | surface area of the stationary phase ($m^2$) |
| $a$ | intercept on the ordinate |
| $A$ | constant of eqn. 19 |
| $a^{\rm R}$ | substance-specific factor according to Rohrschneider[111] |
| $\beta$ | heating rate (°C/min) |
| $B$ | constant of eqn. 19 |
| $b^{\rm R}$ | substance-specific factor according to Rohrschneider[111] |
| $b$ | slope of plot |
| $C$ | constant of eqn. 19 |
| $c_{\rm H}$ | constant |
| $c_{\rm G}$ | constant |
| $c^{\rm R}$ | substance-specific factor according to Rohrschneider[111] |
| $C_{\rm R}$ | system constant of Rohrschneider of eqn. 130 |
| $C_{\rm R}^*$ | system constant of Rohrschneider of eqn. 128 |
| $d^{\rm R}$ | substance-specific factor according to Rohrschneider[111] |
| $D$ | constant of eqn. 48 |
| $E$ | constant of eqn. 48 |
| $e^{\rm R}$ | substance-specific factor according to Rohrschneider[111] |
| $E_{\rm I}$ | total error of the retention index determination (i.u.) under the conditions of classical gas chromatography |
| $F$ | flow-rate of carrier gas ($cm^3$/min) |
| $f_{\rm c}$ | index units/energy units conversion factor |
| $f_i$ | $i$th polarity factor |
| $F^0$ | free energy of solution |
| $\Delta G$ | partial molar free energy of solution |
| $g$ | serial number |
| $h$ | number of atoms in the molecule of interest |
| $\Delta H$ | heat of solution |
| $H$ | homomorph factor according to Schomburg[113–115] |
| $I$ | isothermal retention index according to Kováts[1] |
| i.u. | index units |
| $I_{\rm B}$ | boiling point index |
| $I_{\rm a}$ | atomic index contribution (i.u.) |
| $I_{\rm b}$ | bond index contribution (i.u.) |
| $I_{\rm M}$ | $= I_{\rm a} + I_{\rm b}$ = molecular index contribution (i.u.) |
| $I_{\rm i}$ | interaction index contribution (i.u.) |
| $I_{\rm ig}$ | general interaction index contribution (i.u.) |
| $I_{\rm ii}$ | individual interaction index contribution (i.u.) |
| $i$ | serial number of standards |
| $i_{\rm a}$ | atomic index increment (i.u.) |
| $i_{\rm b}$ | bond index increment (i.u.) |
| $i_{\rm i}$ | interaction index increment (i u.) |
| $i_{\rm ig}$ | general interaction index increment (i.u ) |
| $i_{\rm ii}$ | individual interaction index increment (i.u.) |

| | |
|---|---|
| $I^0$ | vapour pressure index |
| $j$ | compressibility factor according to James and Martin[1381] |
| $K$ | partition coefficient |
| $K_a$ | adsorption coefficient ($cm^3/m^2$) |
| log | logarithm to base 10 |
| ln | natural logarithm |
| $m$ | number of parallel measurements |
| $n$ | difference in carbon numbers of two alkanes |
| $n$-$P_z$ | general designation of *n*-alkane with carbon number $z$ |
| $N_P$ | non-polar stationary phase |
| $\mu$ | chemical potential |
| p | polar stationary phase |
| $p^0$ | vapour pressure (mmHg) |
| $P$ | polarity of the stationary phase |
| $\bar{P}_{Rc}$ | corrected retention polarity (p.u.) |
| $P_{Mc}$ | McReynolds' polarity of stationary phase |
| $\bar{P}_R$ | retention polarity (p.u.) |
| p.u. | polarity units |
| $p_i$ | inlet pressure of carrier gas (kPa) |
| $Q$ | symbol of molecule's centre of interest |
| $q$ | number of increments in the molecule |
| $g$ | density of stationary phase ($g/cm^3$) |
| $R$ | universal gas constant |
| $R^*,R^0$ | substituents in the molecule |
| st.ph. | stationary phase |
| s | substance of interest |
| $s^R$ | Rohrschneider constant |
| SQ | squalane stationary phase |
| $s_i$ | *i*th substance-specific factor of eqn. 130 |
| $s_i^*$ | *i*th substance-specific factor of eqn 128 |
| $t_R$ | retention time |
| $t_M$ | gas holdup time |
| $t'_R$ | adjusted retention time |
| $\bar{t}_R$ | average retention time (min) |
| $T$ | column temperature |
| $T_R$ | retention temperature |
| $T_0$ | initial temperature |
| TPGC | temperature-programmed gas chromatography |
| $t_b$ | difference of boiling points of two isomers |
| $T_b$ | boiling point |
| $u^R$ | Rohrschneider constant |
| $V_g$ | specific retention volume ($cm^3$ carrier gas/g stationary phase) |
| $V_N$ | net retention volume ($cm^3$ carrier gas) |
| $V'_R$ | adjusted retention volume ($cm^3$ carrier gas) |
| $V_L$ | volume of stationary phase present in the column ($cm^3$) |
| $X$ | retention value used in calculations |
| $x$ | coordinate |

| | |
|---|---|
| $x^R$ | Rohrschneider constant |
| $y$ | coordinate |
| $y^R$ | Rohrschneider constant |
| $z$ | carbon number of *n*-alkane |
| $z^R$ | Rohrschneider constant |

## 19 ACKNOWLEDGEMENTS

The authors express their thanks to those researchers who by sending reprints of their papers significantly helped in the compilation of this review.

## 20 SUMMARY

It is well known in GLC that the published retention data are given in many forms; therefore, the usefulness of these data in various laboratories is limited. Evidently, the elaboration of retention index by E.sz. Kováts in 1958 arouse considerable interest. During the past 25 years the number of papers published dealing with the retention index system and its applications recently exceeded 1500.

Theoretically, the retention index system fulfils the task of a universal data providing system. Practically it can be realized only in the case if a data bank is organized by computer where reliable retention indices are stored and which can be easily recalled by researchers of all the gas chromatographic laboratories of the world by telephone and/or satellite. In the present review the main theoretical and practical questions of the retention index system are summarized.

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