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PREDICTION OF RETENTION INDICES OF AROMATIC HYDROCARBONS

JOSE N. TEJEDOR

Servicio Social de Higiene y Seguridad del Trabajo, Instituto Territorial C/Torrelaguna 73, Madrid-27 (Spain)

SUMMARY

A method for calculating the retention index of aromatic compounds has been developed. The method is based on experimental data and can be applied to any stationary phase whose polarity would lie between that of squalane and that of UCON 50 HB 280 X.

INTRODUCTION

The Kováts retention index system[1] is useful in gas–liquid chromatography (GLC), but the qualitative evaluation of chromatograms is very difficult because of the lack of pure standards. Prediction data are important in GLC for both theoretical and practical problems, and many workers have considered different aspects of this problem.

This problem is particularly difficult for aromatic compounds, of which benzene and alkylbenzenes are the most interesting.

Various workers have attempted to predict how the retention index varies with molecular structure[2–19] and with physical and chemical properties, such as vapour pressure and molar volume[20–24]. Other workers have studied the relationship between the structure of a compound and its retention index, without going as far as predicting the latter in both GLC[25–29] and gas–solid chromatography[30].

However, except for the work of Takacs and co-workers[3–12] and West and Hall[19], most of the procedures developed for the calculation of retention indices apply only to the stationary phase used in the calculation, usually squalane. Only in the papers by Dimov and co-workers[20–24] is there a clear relationship between retention index and temperature.

METHOD OF CALCULATION

Experimental data taken from a paper by Engewald and Wennrich[29] were used in this study. The stationary phases were squalane, Ucon LB 550 X and Ucon 50 HB 280 X and the temperature was 100°. The number of carbon atoms in the

compounds studied ranges from 7 to 14. The method is based on the fact that the retention index of a monosubstituted aromatic hydrocarbon in a stationary phase, the polarity of which lies between that of squalane and that of Ucon 50 HB 280 X, can be expressed as

$$I_x^{\text{st.ph.}} = aS_x + b \tag{1}$$

where I_x is the retention index of the compound C_6H_5X in a stationary phase, S_x is the contribution of the alkyl group X and a and b are constants depending on the stationary phase. The contributions of the substituents (S) are additive and the retention index of a disubstituted derivative of benzene could therefore be expressed as

$$I_{x,y}^{\text{st.ph.}} = a^*(S_x + S_y) + b^* \tag{2}$$

where $I_{x,y}$ is the retention index of the compound XC_6H_4Y in a stationary phase, S_x and S_y are the contributions of the alkyl groups X and Y, respectively, and a^* and b^* are constants depending on the stationary phase and/or the position of the substituents on the ring.

As a basis of calculation, the value of the retention index of benzene in squalane at $100°$ (650.5) has been assigned to the constant a, and the value 1.000 for any stationary phase to the contribution of an ethyl group, S_E. Substituting into eqn. 1 for the value of ethylbenzene:

$$I_{\text{ethylbenzene}}^{\text{st.ph.}(100°)} = I_{\text{benzene}}^{\text{squalane}(100°)} \cdot 1 + b$$

$$b = I_{\text{ethylbenzene}}^{\text{st.ph.}(100°)} - I_{\text{benzene}}^{\text{squalane}(100°)} \tag{3}$$

On this basis, the retention index for any alkylbenzene can be calculated as shown below.

Monoalkyl aromatic compounds
    Linear alkyl group.

$$I_x^{\text{st.ph.}(100°)} = aS_x + b \tag{4}$$

where

$I_x^{\text{st.ph.}(100°)}$ = retention index of an *n*-alkyl aromatic hydrocarbon on any stationary phase, the polarity of which lies between that of squalane and that of Ucon 50 HB 280 X at 100

$a = I_{\text{benzene}}^{\text{squalane}(100°)}$

$b = I_{\text{ethylbenzene}}^{\text{st.ph.}(100°)} - I_{\text{benzene}}^{\text{squalane}(100°)}$

S_x = contribution of the *n*-alkyl group (M = methyl, E = ethyl, nPr = *n*-propyl, nBu = *n*-butyl, n Pe = *n*-pentyl, nHe = *n*-hexyl):

$x = M$ $S_x = 0.867$

$$x = E \qquad S_x = 1.000$$
$$x = nPr \qquad S_x = 1.129$$
$$x = nBu \qquad S_x = 1.282$$
$$x = nPe \qquad S_x = 1.429$$
$$x = nHe \qquad S_x = 1.586$$

Branched alkyl group.

$$I_x^{st.ph.(100°)} = a'S_x + b' \tag{5}$$

where

$I_x^{st.ph.(100°)}$ = retention index of a monoalkyl aromatic hydrocarbon with a branched chain

$$a' = 713.19$$
$$b' = 1.0245b - 73.79 \tag{6}$$

S_x = contribution of the branched alkyl group (tBu = *tert.*-butyl, sBu = *sec.*-butyl, iBu = isobutyl, sPe = *sec.*-pentyl); this contribution varies with the polarity of the stationary phase:

$$x = iPr \qquad S_x = -70.559 \cdot 10^{-6}P^{st.ph.} + 1.089 \tag{7}$$
$$x = tBu \qquad S_x = -99.782 \cdot 10^{-6}P^{st.ph.} + 1.182 \tag{8}$$
$$x = sBu \qquad S_x = -98.736 \cdot 10^{-6}P^{st.ph.} + 1.203 \tag{9}$$
$$x = iBu \qquad S_x = -147.045 \cdot 10^{-6}P^{st.ph.} + 1.206 \tag{10}$$
$$x = sPe \qquad S_x = -122.447 \cdot 10^{-6}P^{st.ph.} + 1.329 \tag{11}$$

The value of $P^{st.ph.}$ is related to the Rohrschneider[31] constant x by

$$P^{st.ph.} = 100x \tag{12}$$

$$P^{st.ph.} = I_{benzene}^{st.ph.(100°)} - I_{benzene}^{squalane\ (100°)}. \tag{13}$$

Dialkyl aromatic compounds
Para compounds (linear and branched groups).

$$I_x^{st.ph.(100°)} = a \Sigma S_x + b_p \tag{14}$$

where

$I_x^{st.ph.(100°)}$ = Retention index of a *p*-dialkyl aromatic hydrocarbon

$$a = I_{benzene}^{squalane(100°)}$$
$$b_p = I_{p\text{-diethylbenzene}}^{st.ph.(100°)} - 2I_{benzene}^{squalane(100°)} \tag{15}$$

The term b_p can also be expressed as a function of the polarity of the stationary phase:

$$b_p = 0.8749P^{st.ph.} - 260.5 \tag{16}$$

Ortho and meta compounds (only linear groups).

$$I_x^{st.ph.(100°)} = a'' \Sigma S_x + b_s \tag{17}$$

where

$I_x^{st.ph.(100°)}$ = retention index of an *o*- or *m*-dialkyl aromatic hydrocarbon

$$a'' = 614.36$$

$$b_0 = 0.9614 P^{st.ph.} - 180.32 \tag{18}$$

$$b_m = 0.8895 P^{st.ph.} - 197.71 \tag{19}$$

Owing to insufficient experimental data, neither *o*- nor *m*-dialkyl aromatic compounds with branched groups nor trisubstituted compounds are considered here.

RESULTS

Using eqns. 1–19 the retention indices of 44 mono- and dialkyl aromatic compounds in squalane, Ucon LB 550 X and Ucon 50 HB 280 X at 100° have been calculated. These retention indices, together with the experimental values and the differences between them, are shown in Table I. The constants a, a', a'', b, b', b_0, b_m and b_p, and the contribution of branched alkyl groups, are listed in Table II.

Only two of 132 calculated values vary by more than 6 index units (i.u.) from the experimental values. For one, 13DnPrB in Ucon LB, the experimental retention index seems anomalous considering that it is lower than that of 1iPr4nPrB, whereas it is 13.6 and 16.8 i.u. higher in squalane and Ucon HN, respectively. It is therefore considered that this anomaly could be due to an error in the original paper[28].

On the other hand, in only seven cases, namely the pairs 1tBu4iBuB/1tBu4s BuB, 14DiBuB/1nPr4nBuB and 14DnBuB/1nPr4nPeB in squalane, nBuB/1M4nPrB and 1sBu4iBuB/14DiBuB in Ucon LB and nPeB/1M4nBuB and nPeB/4DiPrB in Ucon HB, has an inversion of the experimental values with respect to the calculated values been found, whereas the number of inversions in the elution of two compounds in two different phases is much greater.

The equations have also been applied to the calculation of retention indices in other columns of intermediate polarity and at other temperatures. In this instance eqns. 4, 3, 5, 13, 14 and 15 become, respectively

$$I_x^{st.ph.(T)} = aS_x + b \tag{20}$$

$$b = I_{ethylbenzene}^{st.ph.(T)} - I_{benzene}^{squalane(100°)} \tag{21}$$

$$I_x^{st.ph.(T)} = a'S_x + b' \tag{22}$$

$$P^{st.ph.(T)} = I_{benzene}^{st.ph.(T)} - I_{benzene}^{squalane(100°)} \tag{23}$$

$$I_x^{st.ph.(T)} = a \Sigma S_x + b_p \tag{24}$$

$$b_p = I_{p-diethylbenzene}^{st.ph.(T)} - 2I_{benzene}^{squalane(100°)} \tag{25}$$

TABLE I

RETENTION INDICES OF MONO- AND DIALKYLBENZENES ON SQUALANE, UCON LB 550 X AND UCON 50 HB 280 X AT 100°

B = benzene; D = di; all other symbols explained in text (*i.e.* pp. 280–281).

Compound	Squalane			Ucon LB 550X			Ucon 50 HB 280X		
	I_{exp}	I_{calc}	Δ	I_{exp}	I_{calc}	Δ	I_{exp}	I_{calc}	Δ
MB	760.1	763.5	3.4	862.1	863.8	1.7	919.9	920.7	0.8
EB	850.0	850.0	0.0	950.3	950.3	0.0	1007.2	1007.2	0.0
14DMB	864.6	867.5	2.9	960.5	962.4	1.9	1017.2	1017.1	−0.1
13DMB	864.8	867.6	2.8	963.9	964.3	0.4	1021.7	1019.7	−2.0
12DMB	886.0	885.0	−1.0	991.6	989.5	−2.1	1053.4	1049.4	−4.0
iPrB	908.4	907.3	−1.1	1004.4	1004.3	−0.1	1059.5	1059.8	0.3
nPrB	938.0	933.9	−4.1	1036.6	1034.2	−2.4	1092.4	1091.1	−1.3
1M3EB	949.7	949.3	−0.4	1047.5	1046.0	−1.5	1105.4	1101.4	−4.0
1M4EB	954.3	954.0	−0.3	1047.5	1048.9	1.4	1105.4	1103.6	−1.8
1M2EB	966.0	966.7	0.7	1069.0	1071.2	2.2	1130.7	1131.1	0.4
tBuB	971.7	973.6	1.9	1067.7	1068.5	0.8	1123.1	1122.5	−0.6
sBuB	990.2	988.6	−1.6	1085.0	1083.5	−1.5	1138.1	1137.5	−0.6
iBuB	991.3	990.7	−0.6	1083.8	1082.1	−1.7	1133.9	1133.9	0.0
13DEB	1027.4	1031.0	3.6	1125.5	1127.7	2.2	1181.6	1183.1	1.5
nBuB	1036.8	1033.4	−3.4	1134.2	1133.7	−0.5	1191.8	1190.6	−1.2
1M4nPrB	1040.2	1037.9	−2.3	1134.6	1132.8	−1.8	1188.4	1187.5	−0.9
14DEB	1040.5	1040.5	0.0	1135.4	1135.4	0.0	1190.1	1190.1	0.0
1M2nPrB	1045.6	1045.9	0.3	1146.5	1150.4	3.9	1206.7	1210.3	3.6
sPeB	1078.2	1078.4	0.2	1171.5	1171.9	0.4	1224.5	1224.5	0.0
1E4nPrB	1126.4	1124.4	−2.0	1218.5	1219.3	0.8	1272.5	1274.0	1.5
nPeB	1133.0	1129.1	−3.9	1233.2	1229.4	−3.8	1289.0	1286.3	−2.7
1M4nBuB	1138.8	1137.4	−1.4	1233.6	1232.3	−1.3	1288.1	1287.0	−1.1
14DiPrB	1154.1	1156.3	2.1	1239.3	1240.8	1.5	1288.9	1290.3	1.4
liPr4nPrB	1181.9	1182.3	0.4	1269.8	1272.0	2.2	1320.3	1324.1	3.8
13DnPrB	1195.5	1193.9	−1.6	1268.3	1286.2	17.9	1337.1	1341.6	4.5
14DnPrB	1212.5	1208.3	−4.2	1302.2	1303.2	1.0	1352.8	1357.9	5.1
1E4nBuB	1227.1	1223.9	−3.2	1318.8	1318.8	0.0	1371.8	1373.5	1.7
nHeB	1231.0	1231.2	0.2	1331.5	1331.5	0.0	1386.8	1388.4	1.6
1M4nPeB	1236.3	1233.0	−3.3	1330.8	1327.9	−2.9	1384.8	1382.6	−2.2
14DtBuB	1281.3	1277.3	−4.0	1360.2	1357.9	−2.3	1408.5	1404.8	−3.7
ltBu4iBuB	1290.6	1292.9	2.3	1368.3	1370.2	1.9	1412.1	1415.4	3.1
ltBu4sBuB	1290.7	1290.9	0.2	1371.3	1371.5	0.2	1417.9	1418.4	0.5
14DsBuB	1304.3	1304.6	0.3	1383.8	1385.2	1.4	1428.0	1432.1	4.1
lsBu4iBuB	1305.8	1306.6	0.8	1383.5	1383.9	0.4	1425.8	1428.8	3.0
14DiBuB	1307.7	1308.5	0.8	1384.0	1382.6	−1.4	1425.3	1425.6	0.3
lnPr4nBuB	1311.0	1307.9	−3.1	1402.1	1402.8	0.7	1452.0	1457.5	5.5
1E4nPeB	1322.1	1319.6	−2.5	1415.4	1414.5	−.09	1468.1	1469.2	1.1
1M4nHeB	1333.4	1335.2	1.8	1429.0	1430.1	1.1	1481.9	1484.8	2.9
ltBu4nBuB	1345.3	1342.3	−3.0	1428.9	1430.1	1.2	1478.8	1480.9	2.1
lsBu4nBuB	1358.4	1356.0	−2.4	1440.0	1443.7	3.7	1491.5	1494.5	3.0
liBu4nBuB	1359.6	1357.9	−1.7	1442.1	1442.4	0.3	1487.9	1491.3	3.4
14DnBuB	1410.8	1407.4	−3.4	1501.5	1502.3	0.8	1551.5	1557.0	5.5
lnPr4nPeB	1418.9	1403.5	−15.4	1497.5	1498.4	0.9	1547.5	1553.1	5.6
1E4nHeB	1421.1	1421.7	0.6	1511.9	1516.6	4.7	1566.8	1571.3	4.5

The results obtained and the experimental data and the differences between them are given in Table III for squalane at 86° and 115°[32] and for dibutyl phthalte[33], and in Table IV for squalane and acetyltributyl citrate at 80°[25]. The constants used in

TABLE II

RETENTION INDEX OF BENZENE, VALUES OF CONSTANTS, a, b, a', b', a'', b_0, b_m, b_p, AND $P^{pt.ph.}$ AND CONTRIBUTIONS OF BRANCHED ALKYL GROUPS FOR THE STATIONARY PHASES UNDER STUDY

Parameter	Squalane				Dibutyl phthalate (86°)	Ucon LB 550 X (100°)	Acetyltributyl citrate (80°)	Ucon 50 HB 280 X (100°)
	80°	86°	100°	115°				
$I_{benzene}$	644.42	646.6	650.5	653.6	732.9	759.2	780.66	821.5
$P^{pt.ph.}$	−6.08	−3.9	0.0	3.1	82.4	108.7	130.16	171.0
a	650.5	650.5	650.5	650.5	650.5	650.5	650.5	650.5
b	191.6	193.8	199.5	200.8	272.3	299.8	323.02	356.7
a'	713.19	713.19	713.19	713.19	713.19	713.19	713.19	713.19
b'	121.95	124.76	130.60	131.93		233.36	257.15	291.65
a''	614.36	614.36	614.36	614.36	614.36	614.36	614.36	614.36
b_0	−186.17	−184.07	−180.32	−177.34	−101.10	−75.82	−55.18	−15.92
b_m	−203.12	−201.18	−197.71	−194.95	−124.42	−101.02	−81.93	−45.61
b_p	−267.18	−263.91	−260.50	−257.79	−188.41	−165.60	−140.58	−110.90
S_{IIr}	1.089	1.089	1.089	1.089	—	1.081	1.080	1.077
S_{IIIu}	1.183	—	1.182	—	—	1.171	1.170	1.165
S_{SIIu}	1.204	—	1.203	—	—	1.192	1.190	1.186
S_{IIIu}	1.207	—	1.206	—	—	1.190	1.187	1.181
S_{NPe}	1.330	—	1.329	—	—	1.316	1.313	1.308

TABLE III

RETENTION INDICES OF MONO- AND DIALKYLBENZENES ON SQUALANE AT 86° AND 115° AND ON DIBUTYL PHTHALATE AT 86°

Compound	Squalane (86°)			Squalane (115°)			Dibutyl phthalate (86°)		
	I_{exp}	I_{calc}	Δ	I_{exp}	I_{calc}	Δ	I_{exp}	I_{calc}	Δ
MB	754.2	757.8	3.6	760.7	764.8	4.1	836.9	836.3	−0.6
EB	844.3	844.3	0.0	851.3	851.3	0.0	922.8	922.8	0.0
14DMB	858.1	864.1	6.0	864.9	870.2	5.3	936.5	939.6	3.1
13DMB	860.3	864.1	3.8	866.3	870.4	4.1	939.5	940.9	1.4
12DMB	880.1	881.2	1.1	888.1	888.0	−0.1	965.2	964.2	−1.0
iPrB	903.5	901.4	−2.1	910.4	918.6	−1.8	—	—	—
nPrB	932.6	928.2	−4.4	940.2	935.2	−5.0	1011.9	1006.7	−5.2
1M3EB	945.6	945.8	0.2	952.1	952.1	0.0	1024.9	1022.6	−2.3
1M4EB	948.0	950.6	2.6	955.1	956.7	1.6	1026.8	1026.1	−0.7
1M2EB	961.2	962.9	1.7	968.8	969.7	0.9	1046.6	1045.9	−0.7
13DEB	1025.0	1027.5	2.5	—	—	—	—	—	—
nBuB	1033.0	1027.7	−5.3	1040.0	1034.7	−5.3	—	—	—
1M2nPrB	1042.0	1042.2	0.2	1050.5	1048.9	−1.6	—	—	—
nPeB	—	—	—	1137.6	1130.4	−7.2	—	—	—

TABLE IV

RETENTION INDICES OF MONO- AND DIALKYLBENZENES ON SQUALANE AND ACETYLTRIBUTYL CITRATE AT 80

Compound	Squalane			Acetyltributyl citrate		
	I_{exp}	I_{calc}	Δ	I_{exp}	I_{calc}	Δ
MB⁻	751.68	755.04	3.36	882.60	887.00	4.40
EB	841.56	841.56	0.00	973.52	973.52	0.00
14DMB	855.73	860.79	.5.06	980.48	987.39	6.91
13DMB	857.76	862.18	4.42	983.96	983.37	−0.59
12DMB	877.26	879.13	1.87	1010.94	1010.12	−0.82
iPrB	901.00	898.62	−2.38	1032.42	1027.40	−5.02
nPrB	929.74	925.47	−4.27	1059.99	1057.43	−2.56
1M3EB	943.07	943.89	−0.82	1070.53	1065.08	−5.45
1M4EB	945.26	947.30	2.04	1071.02	1073.90	2.88
1M2EB	958.25	960.84	2.59	1092.09	1091.83	−0.26
tBuB	967.08	965.66	−1.42	1099.32	1091.58	−7.74
iBuB	982.35	982.77	0.42	1108.74	1103.71	−5.03
sBuB	983.15	980.63	−2.52	1112.10	1105.85	−6.25
1M4iPrB	1004.39	1005.20	0.81	1129.57	1125.94	−3.63
1M3nPrB	1027.78	1023.14	−4.64	1153.38	1144.33	−9.05
nBuB	1029.42	1025.00	−4.42	1160.26	1156.96	−3.30
1M4nPrB	1033.02	1031.22	−1.80	1157.01	1157.82	0.81
14DEB	1033.82	1033.82	0.00	1160.42	1160.42	0.00
1M2nPrB	1039.41	1040.10	0.69	1171.86	1171.08	−0.78
1M4tBuB	1069.11	1066.35	−2.76	1195.38	1184.49	−10.89
sPeB	1071.59	1070.50	−1.09	1200.61	1193.57	−7.04
nPeB	1128.58	1120.62	−7.96	1257.89	1252.58	−5.31
14DiPrB	1148.55	1149.61	1.06	1274.07	1264.50	−9.57

the calculation and the contributions of the branched alkyl groups for these phases are given in Table II.

In Table III only one case shows a difference of more than 6 i.u. between the experimental and the calculated values and no inversion is observed, whereas in Table IV the differences are higher, probably because of the difference between the temperature used in the calculation and that used in the experiments is greater, although in only one instance is the difference more tqan 10 i.u. The number of inversions between the experimental and the calculated values is still very small, being only one in squalane and three in acetyltributyl citrate.

Neither Apiezon L[33] nor more polar phases such as Carbowax 20M[29] gave good results.

CONCLUSIONS

A method allowing the calculation of the retention indices of mono- and dialkylaromatic compounds at 100° in stationary phases whose polarity lies between that of squalane and that of Ucon 50 HB 280 X and which corresponds to Rohrschneider x constants between 0 and 1.71 has been developed. The method can be applied with good results at temperatures close to 100°. This is shown by the fact that of 213 values calculated in five stationary phases and at four different temperatures, only eleven deviated from the calculated retention indices by more than 6 i.u., and only three by more than 10 i.u.

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