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CORRELATION BETWEEN PHYSICAL PROPERTIES OF HALOGENATED ALKANES AND THEIR GAS CHROMATOGRAPHIC BEHAVIOUR ON POLAR AND NON-POLAR STATIONARY PHASES

II. BRANCHED-CHAIN CHLORO-, BROMO- AND IODOALKANES*

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SUMMARY

Branched-chain chloro-, bromo- and iodoalkanes with four to six carbon atoms have been analysed by gas chromatography on 3-m glass columns packed with 15 % of Apiezon L, tricresyl phosphate and Carbowax 20M, on DMCS-treated Chromosorb W, using a flame ionization detector.

The retention indices relative to normal iodoalkanes were determined for all compounds, and the retention indices relative to normal chloroalkanes and bromoalkanes were measured and theoretically calculated for branched-chain chloroalkanes and bromoalkanes, respectively.

The dependence of the retention values on boiling point, molecular volume, type and position of the halogen atom, structure of the molecule and polarity of the liquid phase was investigated.

INTRODUCTION

The dependence of the retention values of linear haloalkanes on their physical properties was examined in previous work[1]. Three liquid phases of increasing polarity were used. The behaviour of the branched-chain alkyl iodides on tricresyl phosphate was also investigated[2–5].

In this work, the influence of the position of methyl groups and of halogen atoms on the gas chromatographic (GC) separation of branched-chain haloalkanes has been studied. Boiling points, molecular volumes and structures have been correlated with the retention times and indices on Apiezon L (APL), tricresyl phosphate (TCP) and Carbowax 20M (CW20M) at different temperatures.

EXPERIMENTAL

The alkyl chlorides and bromides used are listed in Tables I and II, with their main physical constants. The corresponding data for the alkyl iodides[4,5] and for linear chloro- and bromoalkanes[1] has been reported previously.

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The analyses were carried out by using glass columns and injectors, with a Varian Aerograph Model 1520 gas chromatograph. The columns, 3 m × 1/8 in., were filled with 15 % of liquid phase on DMCS-treated Chromosorb W, acid washed, 80–100 mesh. The analytical procedure has been fully described earlier[1].

TABLE I

PHYSICAL CONSTANTS OF CHLOROALKANES

Chloroalkane	T_b (°C) *at 760 torr*	*Refractive index at 20°*	*Density at 20° (g/ml)*	*Molecular volume (cm^3)*
1-Chloropentane	108.2	1.4128	0.8828	120.64
1-Chloro-2-methylbutane	100.5	1.4124	0.8857	120.24
1-Chloro-3-methylbutane	98.8	1.4084	0.8704	122.36
2-Chloro-2-methylbutane	84.5	1.4052	0.8650	123.12
1-Chlorohexane	134.0	1.4199	0.8784	137.18
1-Chloro-2-methylpentane	—	—	—	—
2-Chloro-2-methylpentane	111.5	1.4126	0.863	139.63
3-Chloro-2-methylpentane	116.7	—	—	—
3-Chloro-3-methylpentane	116.0	1.4210	0.890	135.39
1-Chloro-3,3-dimethylbutane	115	1.4161	0.8670	138.98
2-Chloro-2,3-dimethylbutane	118	1.4162	0.8769	137.41

TABLE II

PHYSICAL CONSTANTS OF BROMOALKANES

Bromoalkanes	T_b (°C) *at 760 torr*	*Refractive index at 20°*	*Density at 20° (g/ml)*	*Molecular volume (cm^3)*
1-Bromopentane	129.6	1.1444	1.2177	124.00
1-Bromo-2-methylbutane	121.6	1.4451	1.2234	123.43
1-Bromo-3-methylbutane	121.5	1.4420	1.2209	123.68
2-Bromo-2-methylbutane	107.5	1.4421	1.199	125.94
2-Bromo-3-methylbutane	116.2	1.4454	—	—
1-Bromohexane	156	1.4478	1.1763	140.27
1-Bromo-2-methylpentane	144.1	1.4495	1.1624	141.95
1-Bromo-4-methylpentane	148	1.4490	1.1683	141.23
2-Bromo-2-methylpentane	129.1	$1.442^{23°}$	—	—
2-Bromo-4-methylpentane	142.5	—	—	—
3-Bromo-3-methylpentane	134.4	—	1.1835	139.42
2-Bromo-2,3-dimethylbutane	133.4	1.4517	1.1772	140.16

RESULTS AND DISCUSSION

The retention indices relative to linear iodoalkanes[1–6] (I_{nI}) were determined by using the above three stationary phases at 75°, 100° and 125°. Their dependence on the column temperature was similar to those of linear haloalkanes[1]. Only the values of I_{nI} at 100° are therefore reported in Table III.

Plots of T_b and I_{nI} values as functions of the positions of the substituents

(similar to those in ref. 5 for alkyl iodides on TCP) show that the position of the methyl group or the halogen atom within the carbon chain strongly influences both the boiling point and the retention time almost independently of the polarity of the stationary phase. A difference of about 100 I_{nt} units is observed between the chloro-, bromo- and iodoalkanes with the same basic structures. The differences in the slopes of the

TABLE III

RETENTION INDICES RELATIVE TO *n*-IODOALKANES (I_{nt}) OF BRANCHED CHLORO-, BROMO- AND IODOALKANES

Haloalkane	*APL*	*TCP*	*CW20M*
1-Chlorobutane	199	199	149
1-Chloropentane	300	301	255
1-Chloro-2-methylbutane	279	270	211.5
1-Chloro-3-methylbutane	269	256	190
2-Chloro-2-methylbutane	220	194	114
1-Chlorohexane	401	403	361
1-Chloro-2-methylpentane	373	358	309
2-Chloro-2-methylpentane	308	316	211.5
3-Chloro-2-methylpentane	351	329.5	270
3-Chloro-3-methylpentane	341	326	261
1-Chloro-3,3-dimethylbutane	333	314	252
2-Chloro-2,3-dimethylbutane	318	301	243
1-Chloroheptane	502	505	467
1-Bromobutane	295	298	295
1-Bromopentane	395	399	398
1-Bromo-2-methylbutane	371	361	337
1-Bromo-3-methylbutane	357	351	325
2-Bromo-2-methylbutane	340	300	264
2-Bromo-3-methylbutane	334	298	255
1-Bromohexane	495	500	500
1-Bromo-2-methylpentane	459	452	430
1-Bromo-4-methylpentane	458	458	445
2-Bromo-2-methylpentane	—	385	342
2-Bromo-4-methylpentane	438	381	389
3-Bromo-3-methylpentane	431	421	389
2-Bromo-2,3-dimethylbutane	405	397	362
1-Bromoheptane	594	601	603
1-Iodopentane	500	500	500
1-Iodo-2-methylbutane	475	467	465
1-Iodo-3-methylbutane	462	451	441.5
2-Iodo-2-methylbutane	451	445	429
2-Iodo-3-methylbutane	451	444	429
1-Iodohexane	600	600	600
1-Iodo-2-methylpentane	559	556	554
1-Iodo-3-methylpentane	568	561	563
1-Iodo-4-methylpentane	564	559	565
2-Iodo-2-methylpentane	545	538	517
2-Iodo-3-methylpentane	548	541.5	523
2-Iodo-4-methylpentane	545	537	517
3-Iodo-2-methylpentane	541	536	517
3-Iodo-3-methylpentane	547	542	527
1-Iodo-3,3-dimethylbutane	520	496	483.5

linear plots of I_{nt} against n, previously observed for various homologous series[1], suggests that the retention of the branched-chain compounds can be related to the corresponding normal haloalkanes containing the same halogen atom. In fact, the δ values (*i.e.*, the differences between the normal and the branched-chain compounds with the same carbon number, n) are, in this instance, less affected by the above differences in slope.

The retention indices relative to normal chloro- and bromoalkanes (I_{nc} and I_{nb}, respectively) were therefore measured by direct comparison of the retention times of the branched-chain compounds with those of the corresponding normal isomers. The resulting values are reported in Table IV.

TABLE IV

RETENTION INDICES RELATIVE TO 1-CHLOROALKANES (I_{nc}) AND TO 1-BROMOALKANES (I_{nb}) OF BRANCHED CHLORO- AND BROMOALKANES, RESPECTIVELY

Haloalkane	*Chlorides* (I_{nc})			*Bromides* (I_{nb})		
	APL	*TCP*	*CW20M*	*APL*	*TCP*	*CW20M*
2-Halopentane	463	441	427	453	446	413.5
3-Halopentane	464	445	437	457	443	411
1-Halo-2-methylbutane	479	470	459	476	462	441
1-Halo-3-methylbutane	469	456	439	462	452	429
2-Halo-2-methylbutane	421	395	367	445	402	370
2-Halo-3-methylbutane	—	—	—	439	400	361
2-Halohexane	550	539	522	548	542	509
3-Halohexane	559	540.5	530	551	538	507
1-Halo-2-methylpentane	572	556	551	564	552	531
1-Halo-3-methylpentane	—	—	—	—	—	—
1-Halo-4-methylpentane	—	—	—	563	558	546
2-Halo-2-methylpentane	508	515	459	—	486	446
2-Halo-3-methylpentane	—	—	—	—	—	—
2-Halo-4-methylpentane	—	—	—	543	482	491
3-Halo-2-methylpentane	550	528	514	—	—	—
3-Halo-3-methylpentane	540	524	506	536	522	491
1-Halo-3,3-dimethylbutane	533	513	497	—	—	—
2-Halo-2,3-dimethylbutane	518	500	489	510	498	465

The linearity of the plots of log t_r against n enables every homologous series to be identified by using the slope A_1 and the retention index of a member of the series (for example, the compound with six carbon atoms)[1]. The conversion of the I_{nt} values into I_{nc} values can therefore be achieved by using the following equation, which is similar to the well known equation for the calculation of retention indices:

$$I_{nc}(\mathrm{S}) = 100n + \frac{I_{nt}(\mathrm{S}) - I_{nt}(\text{1-Cl-C}_n)}{I_{nt}(\text{1-Cl-C}_{n+1}) - I_{nt}(\text{1-Cl-C}_n)}$$

$$= 100n + \frac{I_{nt}(\mathrm{S}) - I_{nt}(\text{1-Cl-C}_n)}{A_1(\text{1-Cl-alkanes})}$$

Similar equations can be obviously used to convert I_{nt} into I_{nb} or I_{nb} into I_{nc} values. The values obtained by the application of the above equation correspond exactly to the values calculated directly by using the experimental retention data (Table IV).

From Tables III and IV, it can be seen that by using the I_{nc} and I_{nb} values for chlorides and bromides, respectively, the behaviour on different stationary phases of compounds with similar structures is directly comparable. A graphical representation can be made by using triangular graphs, where the retention index fractions for every compound are reported[7]. With this notation, for a given compound S, the retention index fraction on Apiezon, F_{APL} (S), for example, is given by:

$$F_{APL}(S) = \frac{I_{nh}^{APL}}{I_{nh}^{APL} + I_{nh}^{TCP} + I_{nh}^{CW20M}}$$

where h (halo) may indicate c, b or i, depending on the halogen (chloro-, bromo- or iodo-, respectively).

All of the 1-haloalkanes are represented in Fig. 1 by a triangle in the centre of the diagram, which has been displaced for graphical reasons. Three regions are clearly identified, where the iodides, bromides and chlorides, respectively, are grouped.

A direct comparison of compounds with the same skeletal structure but containing different halogen atoms confirms that all of the branched-chain compounds have a retention lower than the normal compounds, *i.e.*, their rate of elution is higher, and this rate increases from CW20M to TCP to APL. Comparing these three stationary phases, it can be observed that on APL the rate of elution increases from bromides to chlorides and iodides, on TCP the rate increases from bromides to iodides and chlorides, and on CW20M the rate increases from iodides to chlorides and bromides. This general behaviour is confirmed by the values of δI_{nh} reported in Table V, which also reports the δT_b values and the $\delta I_{nh}/\delta T_b$ ratios.

If the retentions of the isomeric compounds are correlated with their boiling points, one would expect[1,2,6] a constant value of the ratio $\delta I_{nh}/\delta T_b$ but this is not confirmed by the experimental data reported in Table V, which show that this ratio also depends on molecular structure and steric hindrance.

In previous work[5], by multiplying the boiling points of some alkyl iodides by

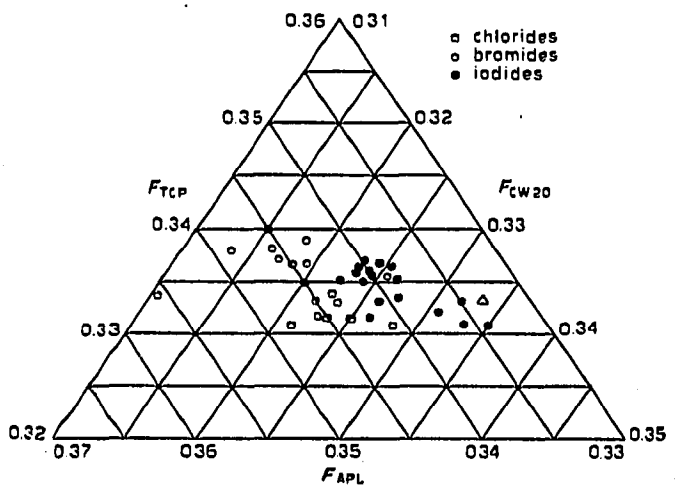

Fig. 1. Retention index fractions of chloro-, bromo- and iodoalkanes on APL, TCP and CW20M. (The centre of the figure, at 0.3–0.3–0.3, has been displaced so as to allow a larger scale representation.)

TABLE V

δI_{nh}, δT_b, AND THEIR RATIO FOR LINEAR AND BRANCHED HALOPENTANES AND HALOHEXANES

Haloalkane	*Chlorides*				*Bromides*				*Iodides*			
	δI_{nc}			δT_b	δI_{nb}			δT_b	δI_{ni}			δT_b
	APL	*TCP*	*CW20M*		*APL*	*TCP*	*CW20M*		*APL*	*TCP*	*CW20M*	
2-Halopentane	37	59	73	11.7	47	54	86.5	12.1	45	58	67	11.2
3-Halopentane	36	55	63	10.2	43	57	89	11.4	50	53	72	10.2
1-Halo-2-methylbutane	21	30	41	7.7	24	38	59	8.0	25	33	35	7.7
1-Halo-3-methylbutane	31	44	61	9.4	38	48	71	8.1	38	49	58.5	8.7
2-Halo-2-methylbutane	79	105	133	23.7	55	98	130	22.1	49	55	71	31.2
2-Halo-3-methylbutane	—	—	—	—	61	100	139	13.4	49	56	71	16.7
2-Halohexane	42	61	78	11.5	52	58	91	12.0	51	60	70	11.4
3-Halohexane	41	59.5	70	11	49	62	93	11.4	54.5	59.5	76	10.0
1-Halo-2-methylpentane	28	44	49	—	36	48	69	11.9	41	44	46	9.0
1-Halo-3-methylpentane	—	—	—	—	—	—	—	—	32	39	37	7.0
1-Halo-4-methylpentane	—	—	—	—	37	42	54	8.0	36	41	35	7.3
2-Halo-2-methylpentane	92	85	141	22.5	—	114	154	26.9	55	62	83	43
2-Halo-3-methylpentane	—	—	—	—	—	—	—	—	52	58.5	77	17
2-Halo-4-methylpentane	—	—	—	—	57	118	109	13.5	55	63	83	17
3-Halo-2-methylpentane	50	72	86	17.5	—	—	—	—	59	64	83	30
3-Halo-3-methylpentane	60	76	94	18	64	78	109	21.6	53	58	73	30
1-Halo-3,3-dimethylbutane	67	87	103	19	—	—	—	—	80	104	116.5	15
2-Halo-2,3-dimethylbutane	82	100	111	16	90	102	135	22.6	—	—	—	—

TABLE V (*continued*)

Haloalkane	*Chlorides*, $\delta I_{nc}/\delta T_b$			*Bromides*, $\delta I_{nb}/\delta T_b$			*Iodides*, $\delta I_{ni}/\delta T_b$		
	APL	*TCP*	*CW20M*	*APL*	*TCP*	*CW20M*	*APL*	*TCP*	*CW20M*
2-Halopentane	3.16	5.04	6.24	3.88	4.46	7.15	4.02	5.18	5.98
3-Halopentane	3.53	5.39	6.18	3.77	5.00	7.81	4.90	5.20	7.06
1-Halo-2-methylbutane	2.73	3.90	5.32	3.00	4.75	7.37	3.25	4.28	5.98
1-Halo-3-methylbutane	3.30	4.68	6.49	4.69	5.92	8.76	4.37	5.63	6.72
2-Halo-2-methylbutane	3.33	4.43	5.61	2.49	4.64	5.88	1.57	1.76	2.27
2-Halo-3-methylbutane	—	—	—	4.55	7.46	10.37	2.93	3.35	4.25
2-Halohexane	3.65	5.30	6.78	4.33	4.83	7.58	4.47	5.26	6.14
3-Halohexane	3.73	5.41	6.36	4.30	5.44	8.16	5.45	5.95	7.60
1-Halo-2-methylpentane	—	—	—	3.02	4.03	5.80	4.55	4.89	5.11
1-Halo-3-methylpentane	—	—	—	—	—	—	4.57	5.57	5.28
1-Halo-4-methylpentane	—	—	—	4.62	5.25	6.75	4.93	5.62	4.79
2-Halo-2-methylpentane	4.09	3.77	6.27	—	4.24	5.72	1.28	1.44	1.93
2-Halo-3-methylpentane	—	—	—	—	—	—	3.06	3.44	4.53
2-Halo-4-methylpentane	—	—	—	4.22	8.74	8.07	3.23	3.70	4.88
3-Halo-2-methylpentane	2.86	4.11	4.91	—	—	—	1.97	2.13	2.77
3-Halo-3-methylpentane	3.33	4.22	5.22	2.96	3.61	5.05	1.77	1.93	2.43
1-Halo-3,3-dimethylbutane	3.53	4.58	5.42	—	—	—	5.33	6.93	7.77
2-Halo-2,3-dimethylbutane	5.12	4.25	6.94	3.98	4.51	5.97	—	—	—

TABLE VI

BOILING POINTS (°K), MOLECULAR VOLUMES AND RATIOS $I_{nh}/T_b \cdot V_m$ FOR LINEAR AND BRANCHED-CHAIN HALOPENTANES AND HEXANES

Haloalkane	*Chlorides*				*Bromides*				*Iodides*			
	$T_b \cdot V_m \cdot 10^{-3}$	$I_{nc}/T_b \cdot V_m \cdot 10^{-3}$			$T_b \cdot V_m \cdot 10^{-3}$	$I_{nb}/T_b \cdot V_m \cdot 10^{-3}$			$T_b \cdot V_m \cdot 10^{-3}$	$I_{ni}/T_b \cdot V_m 10^{-3}$		
		APL	*TCP*	*CW20M*		*APL*	*TCP*	*CW20M*		*APL*	*TCP*	*CW20M*
1-Halobutane	36.68	10.90	10.90	10.90	40.19	9.95	9.95	9.95	46.12	8.67	8.67	8.67
1-Halopentane	46.01	10.87	10.87	10.87	49.94	10.01	10.01	10.01	56.25	8.88	8.88	8.88
2-Halopentane	45.09	10.27	9.78	9.47	48.94	9.26	9.11	8.45	54.06	8.42	8.18	8.01
3-Halopentane	45.31	10.24	9.82	9.64	48.56	9.41	9.12	8.46	54.55	8.25	8.19	7.85
1-Halo-2-methylbutane	44.93	10.66	10.46	10.21	48.72	9.77	9.48	9.05	54.54	8.71	8.57	8.53
1-Halo-3-methylbutane	45.52	10.30	10.02	9.64	48.81	9.46	9.26	8.79	55.11	8.38	8.18	8.01
2-Halo-2-methylbutane	44.03	9.56	8.97	8.33	47.94	9.16	8.34	7.53	52.86	8.53	8.42	8.12
2-Halo-3-methylbutane	44.90	—	—	—	—	—	—	—	54.57	8.26	8.14	7.86
1-Halohexane	55.58	10.79	10.79	10.79	60.20	9.97	9.97	9.97	66.29	9.05	9.05	9.05
2-Halohexane	54.77	10.19	9.84	9.53	59.04	9.28	9.18	8.62	65.23	8.42	8.41	8.12
3-Halohexane	54.87	10.19	9.85	9.66	58.44	9.43	9.21	8.68	64.40	8.47	8.39	8.14
1-Halo-2-methylpentane	—	—	—	—	59.23	9.52	9.32	8.96	64.96	8.60	8.56	8.53
1-Halo-3-methylpentane	—	—	—	—	—	—	—	—	64.36	8.82	8.72	8.75
1-Halo-4-methylpentane	—	—	—	—	59.48	9.46	9.38	9.18	66.19	8.52	8.44	8.57
2-Halo-2-methylpentane	53.71	9.46	9.59	8.54	—	—	—	—	62.44	8.73	8.62	8.28
2-Halo-3-methylpentane	—	—	—	—	—	—	—	—	63.35	8.65	8.55	8.26
2-Halo-4-methylpentane	53.75	—	—	—	—	—	—	—	62.49	8.72	8.59	8.27
3-Halo-2-methylpentane	—	—	—	—	—	—	—	—	68.01	7.95	7.88	7.60
3-Halo-3-methylpentane	52.69	10.25	9.94	9.60	56.82	9.43	9.19	8.64	65.04	8.41	7.97	8.10
1-Halo-3,3-dimethylbutane	53.95	9.88	9.51	9.21	58.70	—	—	—	68.85	7.55	7.20	7.02
2-Halo-2,3-dimethylbutane	53.75	9.64	9.30	9.10	56.98	8.95	8.74	8.16	—	—	—	—

their molecular volumes, values roughly proportional to the retention indices on TCP columns were obtained. This correlation was found not to be completely correct when many consecutive members of a homologous series were considered[1], owing to the curvature of the plot of T_b against n, but, within the limits of a small variation of n, and especially when isomeric compounds are involved, a reasonable constancy of the $\delta I_{nh}/\delta T_b \cdot V_m$ values can be observed (see Table VI). These values, however, have a "fine structure", as can be seen from Fig. 2, where the $I_{nh}/T_b \cdot V_m$ values of 1-halo-X-methylbutanes are reported (X indicates the position of the methyl group in the molecule, and it should be noted that, in order to emphasize the importance of the position of the methyl group, standard chemical nomenclature is not followed completely; in fact, 1-halobutanes are indicated as 1-halo-zero-methylbutanes, 2-halopentanes as 1-halo-1-methylbutanes, etc.).

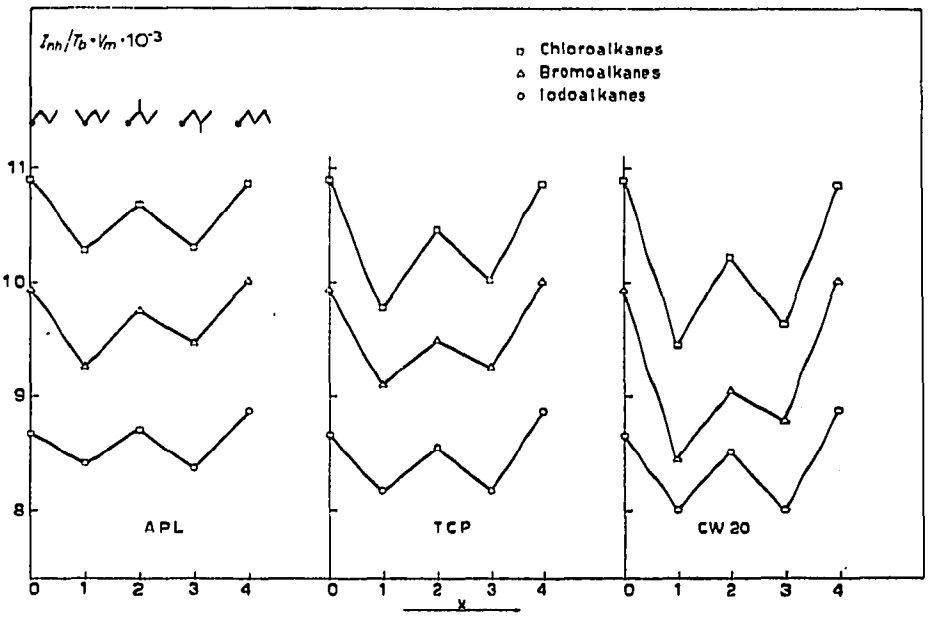

Fig. 2. Values of $I_{nh}/T_b \cdot V_m$ of 1-halo-X-methylbutanes on APL, TCP and CW20M, as a function of the value of X (position of the additional methyl group in the molecule). □, Chloroalkanes; △, bromoalkanes; ○, iodoalkanes.

Similar compounds show similar behaviour, depending on the structure of the molecule and on the polarity of the liquid phase; as the I_{nh} values correlate the GC behaviour of the branched-chain isomers with that of the normal isomers having the same halogen, number of carbon atoms and molecular weight, while $T_b \cdot V_m$ is correlated with the physical properties, one term tends to be compensated by the other (as can be seen from the nearly constant value of $T_b \cdot V_m$, while the ratios I_{nh}/V_m and I_{nh}/T_b vary considerably for different isomers).

The regular behaviour of this "fine structure" permits the calculation of the retention indices of some compounds by mathematical or graphical interpolation. As an example, Fig. 3 shows how the I_{nb} values of 1-bromo-3-methylpentane on the three liquid phases can be calculated from the interpolated values of $I_{nh}/T_b \cdot V_m$.

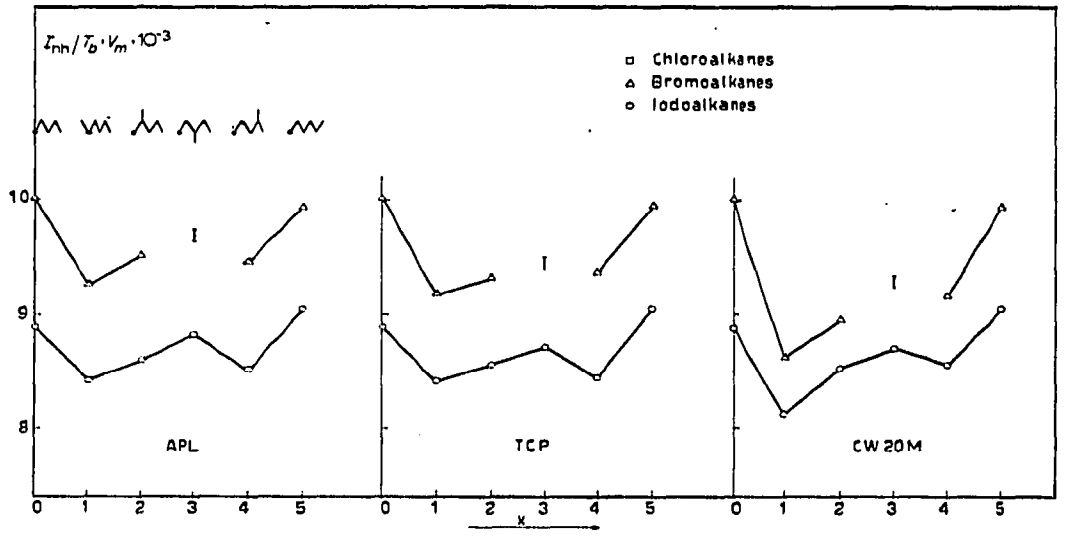

Fig. 3. Values of $I_{nh}/T_b \cdot V_m$ of 1-iodo-X-methylpentanes on APL, TCP and CW20M (○); of five 1-bromo-X-methylpentanes (△); and interpolated values for the calculation of the retention of 1-bromo-3-methylpentane (see text).

Assuming, for this compound, a T_b value of 148.6, a density of 1.1829 and an uncertainity of 0.1 in the interpolated values of $I_{nb}/T_b \cdot V_m$, the I_{nb} values on the three liquid phases will lie in the following ranges: APL, 568–574; TCP, 553–559; and CW20M, 544–550. The reliability of this calculation can be increased if the retention values for higher homologues are known, but the reported approximate values permit the elution order of a missing isomer to be predicted.

The similar values of $I_{nh}/T_b \cdot V_m$ for compounds with the same skeleton and different positions of the halogen atom (see, for example, the X-halo-2-methylpentanes in Table VI and Fig. 3) seem to indicate a major effect of the skeletal structure, thus permitting the calculation of constant additive contributions characteristic of each structural group in a molecule, as in the case of the branched-chain alkanes[8]. In addition, the nature and position of the halogen atom should be taken into account. For this type of semi-empirical approach to the problem of the identification of branched-chain haloalkanes on the basis of their molecular structures and physical properties, several higher homologues would be analysed on different liquid phases.

The retention index of a given haloalkane will therefore be calculated by the algebraic sum of positive and negative contributions from the following structural factors, listed in order of decreasing importance:

(a) number of carbon atoms;
(b) type of the substituted halogen atom;
(c) number of substituted halogen atoms;
(d) position, number and type of the characteristic groups and branches of the skeleton;
(e) position of the substituted halogen atom;
(f) relative positions of the halogen and of the branches.

The values of these structural factors will also depend on the polarity of the liquid phase used.

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