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GAS-LIQUID CHROMATOGRAPHIC ANALYSES

L^a. RETENTION, DISPERSION AND SELECTIVITY INDICES OF POLY-CHLORINATED DIBENZO-*p*-DIOXINS AND DIBENZOFURANS

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SUMMARY

The retention (I), dispersion (I_M) and selectivity (I^*) indices of sixteen polychlorinated dibenzo-*p*-dioxins and fourteen polychlorinated dibenzofurans were determined on a low-polarity HP-5 capillary column using a gas chromatograph connected with an ion-selective detector. I_M and I^* values were also calculated for all 73 dibenzo-*p*-dioxins from the di- to the octachloro isomer and for all possible 135 chlorinated dibenzofurans based on the predicted retention index data reported earlier. The effect of the position of chlorination is shown and the results are compared with those for several series of chlorinated aromatics.

INTRODUCTION

Previously, the gas chromatographic (GC) retention behaviours of the following series of aromatics were studied in our laboratory: chlorinated benzenes¹⁻³, phenols^{1,3-7}, catechols (1,2-dihydroxybenzenes)⁴, salicylaldehydes (2-hydroxybenzaldehydes)^{7,8}, 4-hydroxybenzaldehydes^{7,9}, anisoles (methoxybenzenes)^{3,6,10,11} and veratroles (1,2-dimethoxybenzenes)^{11,12}; the acetate esters of chlorinated phenols^{3,6,10,13-16}, catechols¹⁴ and guaiacols (1-hydroxy-2-methoxybenzenes)¹⁴; several esters of monochloro-¹⁶⁻²², pentafluoro-²¹⁻²⁴ and nitro-^{21,22,25-27} benzoic acids; nitrated polynuclear aromatic hydrocarbons (PAHs)²⁸; and polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs)²⁹. Most frequently these studies concerned GC separations of complex mixtures on capillary columns with different polarities and the relative retention data, most also the retention indices together with the retention index increments due to substitution and some the lipophilicity of the compounds of interest. Later the dispersion and selectivity indices in the GC of various groups of the above compounds based on our retention index data were determined and reported by Evans and Haken^{30,31}.

* For Part XLIX, see ref. 29.

As a continuation of our GC studies on harmful organochlorine compounds occurring in the environment³², this investigation deals with the GC retention, dispersion and selectivity indices of PCDDs and PCDFs obtained on a low-polarity HP-5 capillary column with suitable temperature programming. The dispersion and selectivity indices were also determined for almost all possible chlorinated congeners of interest based on the predicted retention index data reported earlier³³⁻³⁵. The effect of the position of chlorination is shown, and the results are compared with those for several series of chlorinated aromatics.

EXPERIMENTAL

Materials

The PCDDs and PCDFs investigated were obtained from Wellington Labs. (Guelph, Canada) or CIL (Cambridge Isotope Labs., Woburn, MA, U.S.A.)²⁹, the ¹³C-labelled isomers originating from CIL. Mixtures of *n*-alkanes were obtained from SGE (North Melbourne, Australia).

Methods

A Hewlett-Packard 5890/5970 gas chromatograph-mass spectrometer system with an HP-5 fused-silica wall-coated open-tubular (WCOT) column (25 m × 0.20 mm I.D., film thickness 0.11 μm), operated in the splitless injection mode was used. The HP-5 stationary phase (5% diphenyl-95% dimethylpolysiloxane) is very similar to DB-5, BP-5, SPB-5, GC-5, CP-SIL 8, 007-2, RSL-200, SE-52, OV-73 and SE-54 based on the McReynolds' constants. The temperatures of the injector and transfer-line were 250 and 300°C, respectively. The column temperature was held at 100°C for 1 min, then programmed to 180°C at 20°C min⁻¹ and to 290°C at 5°C min⁻¹ and held at 290°C until elution of peaks had ceased. Helium was used as the carrier gas at 7.0 p.s.i. head pressure, the column being coupled directly to the ion source of the mass spectrometer. The selected-ion monitoring mode was used to follow ions characteristic of PCDDs, PCDFs and *n*-alkanes.

The Kováts retention indices were calculated off-line as described earlier³⁶ and the dispersion and selectivity indices as reported previously by Evans *et al.*³⁷.

RESULTS AND DISCUSSION

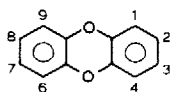
Tables I and II show the retention indices for PCDDs and PCDFs, respectively, obtained on the HP-5 capillary column. The total ion chromatograms of these two groups of components are illustrated in Figs. 1 and 2, respectively. Data for some ¹³C-labelled substances are given in Table III.

Our earlier studies²⁹ showed the GC separation of the title compounds on low-polarity NB-54 and NB-1701 capillary columns using a gas chromatograph with electron-capture detection (ECD). As the substances of interest are very insensitive to flame ionization detection (FID), particularly with the low concentrations used, and as the *n*-alkanes used as reference compounds are not suitable for analysis with ECD, the Kováts retention indices could not be determined by that method. However, by using the technique given above, the retention indices can be determined without problems.

TABLE I

RETENTION (I), DISPERSION (I_M) AND SELECTIVITY (I^*) INDICES FOR PCDDs ON AN HP-5 CAPILLARY COLUMN

Conditions as in Fig. 1.



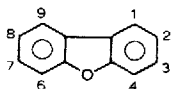
Systematic No. ^a	Compound ^b	I	I_M	I^*
42	1,3,6,8-	2262.3	2281.2	-18.9
48	2,3,7,8-	2353.1	2281.2	71.9
41	1,2,8,9-	2393.8	2281.2	112.6
58	1,2,4,6,8-	2464.3	2526.8	-62.5
61	1,2,4,7,9-	2464.3	2526.8	-62.5
54	1,2,3,7,8-	2554.8	2526.8	28.0
56	1,2,3,8,9-	2579.5	2526.8	52.7
71	1,2,4,6,7,9-	2668.3	2772.3	-104.0
72	1,2,4,6,8,9-	2668.3	2772.3	-104.0
66	1,2,3,4,7,8-	2741.8	2772.3	-30.5
67	1,2,3,6,7,8-	2746.9	2772.3	-25.4
70	1,2,3,7,8,9-	2762.0	2772.3	-10.3
63	1,2,3,4,6,7-	2762.0	2772.3	-10.3
74	1,2,3,4,6,7,9-	2893.9	3017.9	-124.0
73	1,2,3,4,6,7,8-	2937.2	3017.9	-80.7
75	1,2,3,4,6,7,8,9-	3132.6	3263.5	-130.9

^a From ref. 38.^b Numbers indicate the chlorinated positions.

TABLE II

RETENTION (I), DISPERSION (I_M) AND SELECTIVITY (I^*) INDICES FOR PCDFs ON AN HP-5 CAPILLARY COLUMN

Conditions as in Fig. 2.



Systematic No. ^a	Compound ^b	I	I_M	I^*
69	1,3,6,8-	2216.1	2167.1	49.0
83	2,3,7,8-	2324.0	2167.1	156.9
63	1,2,8,9-	2391.0	2167.1	223.9
106	1,3,4,6,8-	2404.0	2412.7	-8.7
94	1,2,3,7,8-	2496.3	2412.7	83.6
114	2,3,4,7,8-	2535.7	2412.7	123.0
96	1,2,3,8,9-	2583.4	2412.7	170.7
116	1,2,3,4,6,8-	2635.6	2658.3	-22.7
118	1,2,3,4,7,8-	2692.8	2658.3	34.5
121	1,2,3,6,7,8-	2699.2	2658.3	40.9
130	2,3,4,6,7,8-	2732.4	2658.3	70.1
120	1,2,3,4,8,9-	2778.8	2658.3	120.5
131	1,2,3,4,6,7,8-	2873.9	2903.8	-29.9
134	1,2,3,4,7,8,9-	2956.7	2903.8	52.9

^a From ref. 38.^b Numbers indicate the chlorinated positions.

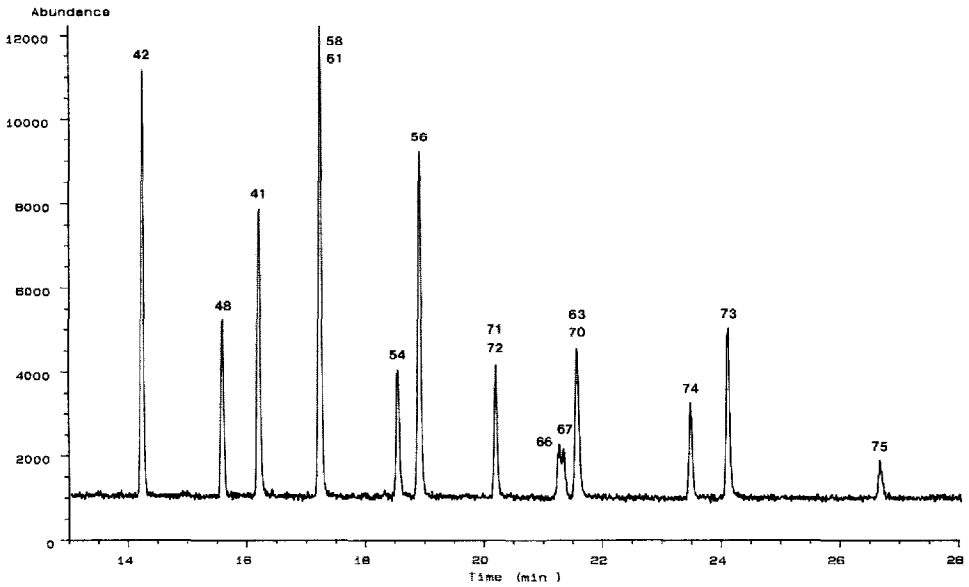


Fig. 1. Total ion chromatogram of a mixture of PCDDs, obtained under the conditions given under Experimental section. For numbering of peaks, see Table I.

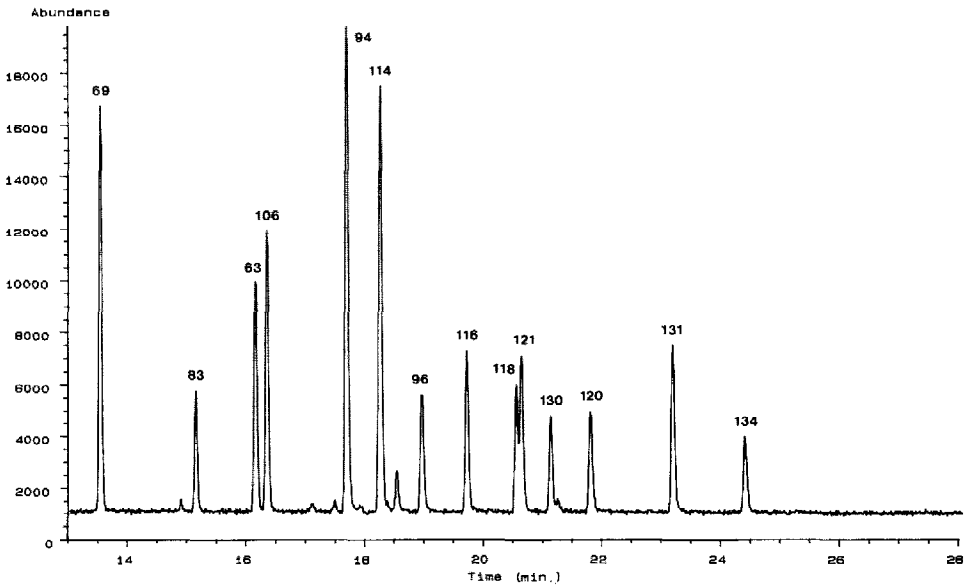


Fig. 2. Total ion chromatogram of a mixture of PCDFs. Conditions as in Fig. 1. For numbering of peaks, see Table II.

A separation of the individual compounds in two mixtures almost identical with that obtained earlier on NB-54²⁹ capillary column was obtained on HP-5 (Figs. 1 and 2). The retention indices in Tables I and II seem to be lower than those obtained earlier on DB-5 capillary columns³³⁻³⁵. This is due mainly to the different stationary phases, but also to the different operating conditions used. From an environmental analysis point of view, important ¹³C-labelled reference compounds are eluted on HP-5 earlier than the corresponding unlabelled substances, showing a difference from -0.2 to -0.5 retention index units (i.u.) (Table III). It should be noted that deuterium labelling causes a considerable reduction in retention so that, for example, perdeuterated aliphatic long-chain hydrocarbons and esters can be completely separated from their non-deuterated homologue on a low-polarity OV-1 capillary column^{39,40}. Our previous studies with partially deuterated chlorinated esters⁴¹⁻⁴³ and some aromatic compounds, *viz.*, chlorinated guaiacols⁴⁴ and veratroles^{45,46}, also showed a slightly earlier elution of a deuterated congener. The difference observed is negligible, however, even on highly polar stationary phases, where generally the structural effects are maximized. Hence it seems evident that the differences in Table III would be greater on analysing the components on a polar column.

The dispersion (I_M) and selectivity (I^*) indices developed recently by Evans and co-workers^{30,31,37,47,48} are given in Tables I and II for PCDDs and PCDFs on HP-5, respectively. For comparison, the corresponding data for almost all possible chlorinated congeners are given in Tables IV and V, based on the predicted retention indices reported earlier^{33,34}. These predicted indices had to be used as measured indices were not available for all isomers. However, the results show that these predicted indices are very close to the experimental values obtained on DB-5 capillary columns, *i.e.*, the difference varies from -11 to +12 i.u. for chlorinated dibenzo-*p*-dioxins³³ and from -15 to +19 i.u. for chlorinated dibenzofurans³⁴. The former contains 41 measured values (73) and the latter 115 (135) (calculated values in parentheses).

As is evident, the I_M values increase with an increase in the degree of chlorination, *i.e.*, the molecular weight. As a consequence of the lower retention indices, the selectivity indices presented in Tables I and II are lower than those in Tables IV

TABLE III

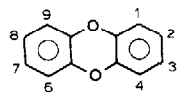
RETENTION INDICES FOR SOME ¹³C-PERLABELLED PCDDs AND PCDFs ON AN HP-5 CAPILLARY COLUMN

Conditions as in Figs. 1 and 2.

Compound ^a	Retention index, <i>I</i>	Difference, $I(^{13}\text{C}) - I(^{12}\text{C})^b$
2,3,7,8-D	2352.7	-0.4
1,2,3,7,8-D	2554.5	-0.3
1,2,3,4,7,8-D	2741.3	-0.5
1,2,3,4,6,7,8,9-D	3132.4	-0.2
2,3,7,8-F	2323.6	-0.4
1,2,3,7,8-F	2495.9	-0.4

^a Numbers indicate the chlorinated positions; D = Dioxin, F = furan.^b Retention indices for parent substances given in Tables I and II.

TABLE IV

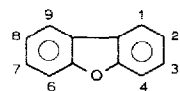
RETENTION (I), DISPERSION (I_M) AND SELECTIVITY (I^*) INDICES FOR CHLORINATED DIBENZO-*p*-DIOXINS

Isomer ^a	I^b	I_M	I^*	Isomer ^a	I^b	I_M	I^*
1,3-	1936	1790	146	1,2,6,9-	2378	2281	97
1,4-	1951	1790	161	1,2,3,4-	2379	2281	98
1,6-	1968	1790	178	1,2,3,7-	2382	2281	101
1,7-	1972	1790	182	1,2,3,8-	2382	2281	101
1,8-	1972	1790	182	2,3,7,8-	2386	2281	105
2,7-	1976	1790	186	1,2,3,9-	2392	2281	111
2,8-	1976	1790	186	1,2,7,8-	2397	2281	116
1,9-	1982	1790	192	1,2,6,7-	2408	2281	127
2,3-	1984	1790	194	1,2,8,9-	2422	2281	141
1,2-	1995	1790	205	1,2,4,7,9-	2511	2527	-16
1,3,6-	2129	2036	93	1,2,4,6,8-	2511	2527	-16
1,3,7-	2133	2036	97	1,2,4,6,9-	2533	2527	6
1,3,8-	2133	2036	97	1,2,3,6,8-	2539	2527	12
1,3,9-	2143	2036	107	1,2,4,7,8-	2545	2527	18
1,2,4-	2143	2036	107	1,2,3,7,9-	2553	2527	26
1,4,8-	2148	2036	112	1,2,3,6,9-	2568	2527	41
1,4,9-	2158	2036	122	1,2,4,6,7-	2570	2527	43
1,7,8-	2177	2036	141	1,2,4,8,9-	2570	2527	43
2,3,7-	2181	2036	145	1,2,3,4,7-	2576	2527	49
1,2,3-	2185	2036	149	1,2,3,4,6-	2586	2527	59
1,2,6-	2188	2036	152	1,2,3,7,8-	2587	2527	60
1,2,7-	2192	2036	156	1,2,3,6,7-	2598	2527	71
1,2,8-	2192	2036	156	1,2,3,8,9-	2612	2527	85
1,2,9-	2202	2036	166	1,2,4,6,8,9-	2725	2772	-47
1,3,6,8-	2290	2281	9	1,2,4,6,7,9-	2725	2772	-47
1,3,7,9-	2304	2281	23	1,2,3,4,6,8-	2747	2772	-25
1,3,6,9-	2319	2281	38	1,2,3,6,8,9-	2760	2772	-12
1,3,7,8-	2338	2281	57	1,2,3,6,7,9-	2760	2772	-12
1,2,4,7-	2340	2281	59	1,2,3,4,6,9-	2769	2772	-3
1,2,4,8-	2340	2281	59	1,2,3,4,7,8-	2781	2772	9
1,4,6,9-	2341	2281	60	1,2,3,6,7,8-	2788	2772	16
1,2,6,8-	2349	2281	68	1,2,3,7,8,9-	2802	2772	30
1,2,4,6-	2350	2281	69	1,2,3,4,6,7-	2806	2772	34
1,2,4,9-	2350	2281	69	1,2,3,4,6,7,9-	2961	3018	-57
1,4,7,8-	2353	2281	72	1,2,3,4,6,7,8-	2996	3018	-22
1,2,7,9-	2363	2281	82	1,2,3,4,6,7,8,9-	3197	3263	-66
1,2,3,6-	2378	2281	97				

^a Numbers indicate the chlorinated positions.^b Calculated retention indices taken from ref. 33.

and V. As with other groups of chlorinated aromatics³¹, viz., benzenes, anisoles, veratroles, phenols, phenyl acetates and 2- and 4-hydroxybenzaldehydes, the I^* values of the compounds in the present investigation decrease with increasing degree of chlorination. This is due to the effect of the screened electrons of the halogen atoms,

TABLE V

RETENTION (I), DISPERSION (I_M) AND SELECTIVITY (I^*) INDICES FOR CHLORINATED DIBENZOFURANS

Isomer ^a	I^b	I_M	I^*	Isomer ^a	I^b	I_M	I^*
1-	1728	1430	298	1,2,3,4-	2318	2167	151
3-	1745	1430	315	1,2,3,6-	2318	2167	151
2-	1751	1430	321	2,3,4,9-	2318	2167	151
4-	1761	1430	331	1,4,6,9-	2319	2167	152
1,3-	1875	1676	199	1,2,7,8-	2324	2167	157
1,4-	1901	1676	225	1,3,4,9-	2333	2167	166
1,7-	1908	1676	232	1,2,6,7-	2335	2167	168
1,8-	1913	1676	237	2,3,4,7-	2336	2167	169
2,4-	1914	1676	238	1,2,7,9-	2338	2167	171
1,6-	1923	1676	247	1,2,4,9-	2339	2167	172
3,7-	1925	1676	249	2,3,4,8-	2342	2167	175
2,7-	1931	1676	255	2,3,4,6-	2342	2167	175
1,2-	1936	1676	260	2,3,7,8-	2342	2167	175
2,8-	1936	1676	260	2,3,6,7-	2353	2167	186
3,6-	1941	1676	265	3,4,6,7-	2353	2167	186
2,6-	1946	1676	270	1,2,6,9-	2364	2167	197
4,6-	1947	1676	271	1,2,3,9-	2377	2167	210
2,3-	1954	1676	278	1,2,8,9-	2398	2167	231
3,4-	1964	1676	288	1,3,4,6,8-	2418	2413	5
1,9-	1982	1676	306	1,2,4,6,8-	2423	2413	10
1,3,7-	2055	1922	133	2,3,4,7,9-	2466	2413	53
1,3,8-	2060	1922	138	1,3,4,7,8-	2468	2413	55
1,3,6-	2071	1922	149	1,3,4,6,7-	2468	2413	55
2,4,9-	2076	1922	154	1,2,3,6,8-	2471	2413	58
1,3,4-	2079	1922	157	1,2,4,7,8-	2473	2413	60
1,4,7-	2081	1922	159	1,2,4,6,7-	2473	2413	60
1,2,4-	2084	1922	162	1,3,4,7,9-	2481	2413	68
1,4,8-	2087	1922	165	2,3,4,6,9-	2482	2413	69
1,4,6-	2087	1922	165	1,2,4,7,9-	2486	2413	73
2,4,7-	2094	1922	172	2,3,4,6,8-	2495	2413	82
2,4,8-	2099	1922	177	1,3,4,6,9-	2497	2413	84
2,4,6-	2100	1922	178	1,2,3,4,7-	2498	2413	85
2,3,9-	2116	1922	194	1,2,4,6,9-	2502	2413	89
1,2,7-	2116	1922	194	1,2,3,4,8-	2503	2413	90
1,2,8-	2121	1922	199	1,2,3,4,6-	2504	2413	91
1,2,3-	2123	1922	201	1,2,3,7,8-	2511	2413	98
3,4,9-	2126	1922	204	1,2,3,6,7-	2522	2413	109
1,3,9-	2129	1922	207	1,2,3,7,9-	2524	2413	111
1,2,6-	2132	1922	210	2,3,4,8,9-	2527	2413	114
2,3,7-	2134	1922	212	1,3,4,8,9-	2542	2413	129
2,3,8-	2139	1922	217	2,3,4,7,8-	2545	2413	132
3,4,7-	2144	1922	222	2,3,4,6,7-	2545	2413	132
3,4,8-	2149	1922	227	1,2,4,8,9-	2547	2413	134
2,3,6-	2149	1922	227	1,2,3,6,9-	2551	2413	138

(Continued on p. 334)

TABLE V (continued)

Isomer ^a	<i>I</i> ^b	<i>I</i> _M	<i>I</i> *	Isomer ^a	<i>I</i> ^b	<i>I</i> _M	<i>I</i> *
3,4,6-	2150	1922	228	1,2,3,4,9-	2572	2413	159
1,4,9-	2155	1922	233	1,2,3,8,9-	2585	2413	172
2,3,4-	2156	1922	234	1,2,3,4,6,8-	2656	2658	-2
1,2,9-	2190	1922	268	1,3,4,6,7,8-	2660	2658	2
1,3,6,8-	2224	2167	57	1,2,4,6,7,8-	2665	2658	7
1,4,6,8-	2240	2167	73	1,3,4,6,7,9-	2675	2658	17
2,4,6,8-	2252	2167	85	1,2,4,6,7,9-	2680	2658	22
1,3,4,7-	2259	2167	92	1,2,4,6,8,9-	2685	2658	27
1,3,7,8-	2264	2167	97	1,2,3,4,7,8-	2706	2658	48
1,2,4,7-	2264	2167	97	1,2,3,4,6,7-	2707	2658	49
1,3,4,8-	2264	2167	97	1,2,3,6,7,8-	2714	2658	56
1,3,4,6-	2265	2167	98	1,2,3,4,7,9-	2719	2658	61
1,2,4,8-	2270	2167	103	1,2,3,6,7,9-	2728	2658	70
1,2,4,6-	2270	2167	103	1,2,3,6,8,9-	2734	2658	76
1,3,6,7-	2274	2167	107	1,2,3,4,6,9-	2736	2658	78
1,3,7,9-	2277	2167	110	2,3,4,6,7,8-	2737	2658	79
1,2,6,8-	2284	2167	117	1,2,3,7,8,9-	2772	2658	114
1,4,7,8-	2290	2167	123	1,2,3,4,8,9-	2780	2658	122
1,4,6,7-	2290	2167	123	1,2,3,4,6,7,8-	2899	2904	-5
2,3,6,8-	2302	2167	135	1,2,3,4,6,7,9-	2914	2904	10
2,4,6,7-	2303	2167	136	1,2,3,4,6,8,9-	2919	2904	15
1,3,6,9-	2303	2167	136	1,2,3,4,7,8,9-	2967	2904	63
1,2,3,7-	2303	2167	136	1,2,3,4,6,7,8,9-	3152	3149	3
1,2,3,8-	2308	2167	141				

^a Numbers indicate the chlorinated positions.

^b Calculated retention indices taken from ref. 34.

as clearly shown in Fig. 3, which indicates a dependence of the selectivity indices of compounds on the degree of chlorine substitution. The values are considerably higher as with other aromatics, where negative *I** values obtained on a low-polarity stationary phase seem to be usual. Tables IV and V show that the negative values for *I** are very rare, particularly with dibenzofurans having lower molecular weights than the corresponding dibenzo-*p*-dioxins. This results in lower *I*_M values for dibenzofurans, although the retention indices of analogous isomers in both groups would be near to each other. As with chlorobenzenes, the greatest retention occurs with the isomers where the chlorine substituents are closest together and decreases as the position around the ring allows greater separation². Thus, for example, the 1,3,6,8- is the first tetrachloro isomer to elute and 1,2,8,9- is the last in both groups of compounds.

The relationship between the retention characteristics on a gas chromatographic column and the molecular structure of the chlorinated dibenzo-*p*-dioxins³³ and dibenzofurans^{34,35} have been thoroughly discussed and the trends are evident from the data in Tables I-V. It is apparent that the models developed can be applied to other similar systems.

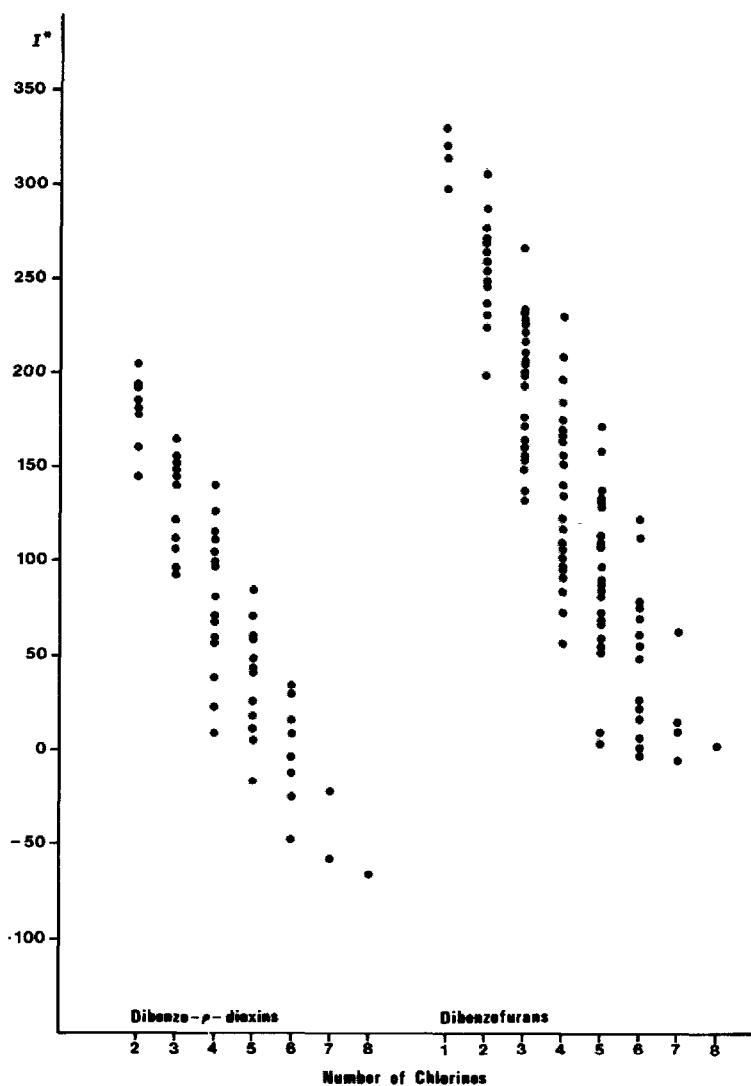


Fig. 3. Dependence of selectivity indices of compounds of interest on the degree of chlorine substitution (Tables IV and V).

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