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GAS CHROMATOGRAPHY OF ALKENYL CHLORIDES

A great number of tabulated Kovats indices have been published during the last twenty years, but only few data are available on the retention indices of alkyl chlorides [1-3]. This paper presents the results of gas chromatographic treatment of various alkenyl chlorides (6-chloro-6-methyl-2-alkenes and related compounds) on nonpolar methyl silicone OV-101, high polar 1,2,3-*tris*-(2'-cyanoethyl)propane (TCEP), and the mixture of TCEP with diethylene glycol succinate (DEGS) liquid phases using capillary columns.

The compounds studied were obtained by addition of allylic chlorides (or alkoxychloromethanes) to isoalkenes (isoolefins, chloromethyl-substituted isoolefins, and isoprene). Their structures were established earlier by ¹H and ¹³C NMR spectra [⁴⁻⁸].

 $R^{2} + R^{4} + R^{1}, R^{2}, R^{3}, R^{5} = H \text{ or } CH_{3}$ $R^{6} + R^{6} + R^{4} = H, CH_{3} \text{ or } CH_{2}Cl$ $R^{6} = CH_{3}, C_{2}H_{5}, C_{3}H_{7}, CH(CH_{3})_{2},$ $R^{1} + R^{3} + Cl + CH_{2}Cl + CH_{2}Cl$ Left part: Right part:

from allylic from isoalkene chloride

In addition to the retention data of the above tertiary chlorides data for some primary allylic and cyclic chlorides (formed as isomeric adducts in case of isoprene [2,7]), as well as for *o*-dichlorobenzene and benzyl chloride as reference compounds were included.

The increments of the molantimeration and he found by com-

The measurements were made on a Chrom 5 Gas Chromatograph (Laboratorne Pristroje, Czechoslovakia) with FID and an electronic fixation of the peak retention time. The GC conditions are summarized in Table 1.

Columns 1 and 2 were made in the Design Office, Estonian Academy of Sciences. Column 3 is self-made with $BaCO_3$ sedimentation on the inner surface and a dynamic coating with a 15% solution of liquid phases (TCEP-DEGS ratio 78:22) in chloroform using a mercury "plug" to get a more uniform and thin film.

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15 Косерлан А. Аллон	K.J. Penes & J. A.	Column number	oros los institution rop-
Column characteristics	interest and the second	2	3
Material	Fused silica	Glass (pyrex)	Glass (pyrex)
Length, m	23	24	37
Inside diameter, mm	0.22	0.27	0.3
Liquid phase	OV-101 (fixed)	TCEP	TCEP 78% DEGS 22%
HETP, mm	0.34	0.92	0.99
Flow rate, cm ³ /min Column oven	1.0	1.5	0,410 1.5
temperature, °C Glass-lined injection	80±0.1	80±0.1	80±0.1
port temperature, °C	160 ± 2	160 ± 2	160 ± 2

Gas chromatography conditions

The parameters of the columns used during the experiments carried out during three months did not change. The dead volumes and retention indices of alkenyl chlorides were calculated via the retention times of *n*-alkanes [9]. The standard deviation of the values of *I*, calculated on the basis of at least four measurements for each compound, was less than 0.5 index units (i.u.) for the nonpolar OV-101 column and 0.7 i.u. for polar columns.

Results and Discussion

In Table 2, relative retention times to o-dichlorobenzene on polar columns, retention indices (I), and values of ΔI^{P-NP} (differences in I on polar and nonpolar phases) for 57 alkenyl chlorides on nonpolar OV-101, and highly polar TCEP and TCEP/DEGS phases are presented.

A comparison of the values of I of various compounds with the same code letter shows that the retention increment values for substituents in the left part of the molecule can easily be obtained. So the average value for the 3-methyl-group increment on OV-101 column is:

- $I^{4b} I^{2b} = 1219.7 1128.4 = 91.3$ $I^{4f} - I^{2f} = 1321.9 - 1235.4 = 86.5$
- $I^{4e} I^{2e} = 1318.9 1232.8 = 86.1$
 - average: 87.6 i.u. $I^{4g} - I^{2g} = 1294.5 - 1208.2 = 86.3$

The increments of the molecule's right part may be found by comparing indices of compounds with the same code number. So, for the -CH2-CH2-methylene groups the increment values on TCEP column are: de de de de de de de la

 $I^{1e} - I^{1a} = 1170.6 - 989.2 = 181.4$

- $I^{3e} I^{3a} = 1226.4 1049.5 = 176.9$ average: 179.2 i.u.
- $I^{2e} I^{2a} = 1232.8 1039.6 = 193.2$
- $I^{4e} I^{4a} = 1318.9 1129.5 = 189.4$ average: 191.8 i.u.

In this case, the average value must be taken for I^1 and I^3 as well as for I^2 and I^4 increments separately, because in the latter cases, the 4-methyl group decreases the index value $\begin{bmatrix} 1, 10 \end{bmatrix}$.

To draw more detailed conclusions, these data should be subjected to further regression analysis. and a good a determined of the second of the

-		10 1	/-101		TC	EP		_	TCEP/DE	3S
Code*	Structure	1.01	I [1, 2]	Vrel	I SI	I [1, 2]	AIP-NP	0°Vrel	LTIT	AIP-NP
112	2	3	11440 [6]	5	. 9	7	351.8	6110	113010	287 11
(g) (g)	o-dichlorobenzene (DCIB)	1016.7	11543	1.00	1391.2	[4] 0.0001 138818 [4]	374.5	1.00	1483.7	467.0
	benzylchloride (BCI)	990.2		1.75	1476.5		486.3	1.83	1567.0	576.8
Ia	~~~~	989.2	[1] 9.986	0.23	1166.0	1160.0 [1]	176.8	0.10	1205.4	216.2
**41		1095.1	1094.3 [¹] 1118.1 [¹]	0.42 0.53	1260.7 1294.3	1257.3 [¹] 1292.3 [¹]	165.6 176.2	0.19 0.34	1299.0 1343.9	203.9 225.8
Ic	- Are		1165.0 [1]			1337.6 [1]	172.6			
le**	- The	1167.9		0.63	1322.8		152.2	0.36	1351.0	180.4
**JI	- Arrel	1174.6	1140 4 [6]	0.59	1310.2		135.6	0.39	1360.2	196.9
18**	- Add	1139.8	1241 M P	0.85	1367.5	1210 2 12021	227.7 945.4	0.66	1428.0	288.2
**4I	ALLER OF	1265.4 1279.1	1198861	2.38	1523.7 1547.5	N-Fenger	258.3 268.4	2.47	1588.1 1610.6	322.7 331.5
li	· · · · · · · · · · · · · · · · · · ·	1086.4	14 Alettor	0.44	1269.5	1 H H Knor	183.1	0.31	1316.4	230.0
li(E)	and a	1173.5	1173.6 [2]	1.09	1401.5	1472.1 [2]	228.0	0.85	1460.2	286.7

	2	3	4	5	9	1 2	8	6	10	11
14 In and	- D	iner the		-460	- 130a2		m JEan		12164 3	0.052
cas			1039.6 [1]	100352	- ACHAR	1230.1 [1]	190.5		8,0131	
2268 2322 Lats the incr	- You	1128.4	1126.6 [1]	0.55	1301.2	1301.9 [1]	172.8	0.37	1352.8	224.4
9 1 9 1 remen	· Net	1210.7	1208.4 [1]	0.88	1372.6	1374.2 [1]	161.9	0.65	1424.9	214.2
129.1 129.1 129.1 tage nts " s	- Tel	1150.6	1149.4 [1]	0.66	1327.0	1325.5 [1]	176.4	0.45	1376.2	225.6
Valu	- York	1232.8		1.04	1398.0		165.2	0.75	1444.1	211.3
	× rci	1235.4	E Hean	1.04	1397.4	1331.6 [1]	162.0	0.78	1446.2	213.8
ave	- face	1208.2	S [1] S.HOT	1.61	1464.4	1385'3 [1]	256.2	1.45	1534.9	326.7
rage take	XXXCI	1297.3	1299.4 [2]	3.11	1565.0	1642.1 [2]	267.7	2.77	1626.4	329.1
E DEUX	CU ABCI	8000		1.72			1.96.3 C	1.83		a ora
lor line	IN ACT DOLE	1124.8	1124.9 [2]	0.68	1332.7	1399.9 [2]	207.9	0.49	1388.8	264.0
an Tel		03.6	1148.9 [2]	0.89	1373.1	1448.1 [2]	224.2	0.71	1436.8	287.4
T ^a a	CC CC	1150.0	1151.8 [2]	0.80	1358.3	1428.3 [2]	208.3	0.61	1417.9	267.9
			101 - 101 -		OT E LE CA		0.1		TCEPADEO	~
ALCENDE	Co Cl or allowy	1195.6	0.1197.0 [2]	1.20	a 1420.0 F	1507.0 [2]	224.4	1.10	1496.6	301.0
Retention indi	ter .	1200.4	1201.5 [2]	1.27	1428.8	1516.7 [2]	228.4	1.17	1505.4	305.0

3 4 5 1209.0 1209.6 1.48 1209.0 1209.6 1.48 1218.5 1218.9 1.43 1218.5 1218.9 1.43 1227.6 1227.6 1.43 1227.6 1227.6 1.69 1049.5 1048.3 1.169 1157.2 1155.6 1.1 1183.9 1182.1 0.59 1232.4 0.79 0.79 1232.4 0.79 0.79 1197.0 1.50	6 1 1450.6 1446.2 1471.4 1204.3 1298.7 1336.3 1354.9	7 1528.9 [2] 1521.6 [2] 1550.0 [2] 1550.0 [2] 1200.3 [4] 1200.3 [4] 1335.7 [4]	8 241.6 227.7 243.8 154.8 154.8 154.2 154.2	9 9 1.30 1.23 1.50	1520.3	11
1209.0 1209.6 [2] 1.48 1218.5 1218.9 [2] 1.43 1218.5 1218.9 [2] 1.43 1227.6 1227.6 [2] 1.69 1227.6 1227.6 [2] 1.69 1049.5 1048.3 [1] 0.29 1157.2 1155.6 [1] 0.54 1183.9 1182.1 [1] 0.69 1232.4 0.182.1 [1] 0.79 1232.4 0.70 0.79 0.79 1197.0 1.50 0.64 0.79	1450.6 1446.2 1471.4 1204.3 1298.7 1336.3 1354.9	1528.9 [2] 1521.6 [2] 1550.0 [2] 1200.3 [4] 1335.7 [4]	241.6 227.7 243.8 154.8 154.8 154.2	1.30 1.23 1.50	1520.3	
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1218.5 1218.9 [2] 1.43 1227.6 1227.6 [2] 1.69 1227.6 1227.6 [2] 1.69 1049.5 1048.3 [1] 0.29 1157.2 1155.6 [1] 0.54 1183.9 1182.1 [1] 0.69 1232.4 0.79 0.79 1232.4 0.83 1.50	1446.2 1471.4 1204.3 1298.7 1336.3 1354.9	1521.6 [²] 1550.0 [²] 1200.3 [⁴] 1297.1 [⁴] 1335.7 [⁴]	227.7 243.8 154.8 154.8 154.8	1.23		0.110
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1049.5 1048.3 [1] 0.29 1157.2 1155.6 [1] 0.54 1183.9 1182.1 [1] 0.69 1232.4 0.79 0.79 1232.4 0.83 0.83 1197.0 1.50 0.69	1204.3 1298.7 1336.3 1354.9	1200.3 [1] 1297.1 [1] 1335.7 [1]	154.8 143.1 154.2		1539.9	312.3
1049.5 1048.3 [1] 0.29 1157.2 1155.6 [1] 0.54 1183.9 1182.1 [1] 0.69 1220.4 0.79 0.79 1232.4 0.83 1197.0 1.50	1204.3 1298.7 1336.3 1354.9	1200.3 [1] 1297.1 [1] 1335.7 [1]	154.8 143.1 154.2			
1157.2 1155.6 [*] 0.54 1183.9 1182.1 [*] 0.69 1220.4 0.79 0.79 1232.4 0.83 1197.0 1.50	1298.7 1336.3 1354.9	1297.1 [¹] 1335.7 [¹]	143.1	0.13	1236.3	186.8
1183.9 1182.1 [1] 0.69 1220.4 0.79 1232.4 0.83 1197.0 1.50	1336.3	1335.7 [1]	154 2	0.33	1337.3	180.1
1220.4 0.79 1232.4 0.83 1197.0 1.50	1354.9	P T		0.46	1380.0	196.1
1232.4 0.83 1197.0 1.50	0 0000		134.5	0.44	1374.5	154.1
1197.0	1363.6		131.2	0.48	1384.5	152.1
	1453.1		256.1	1.09	1495.4	298.4
1220.9 1.84	1484.2		263.3	1.42	1532.5	311.6
1378.8 4.80	1617.3		238.5	4.11	1683.0	308.2
1391.3 5.67	1641.3		250.2	4.90	1708.2	316.9
1125.3 0.75	1348.6		223.3	0.58	1406.1	280.8
			534 3		1 000.	0100
1236.9 1241.8 [2] 1.40	1440.6	1503.7 [2]	203.7	1.05	1489.5	252.6
			110.9			
1129.5 [1]		1305.2 [1]	175.7			
1219.7 1217.7 0.91	1377.6	1378.5	159.9	0.68	1432.0	214.3
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									Table	2 continued
1	2	3	4	5	6	7	8	6	10	11
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4[where we have a state of the st	1321.9		1.63	1466.9		145.0	1.22	1510.8	188.9
	tot -	00.00			0 100 1		1		1 EAF A	1021
4e	1	1318.9		1.63	1465.6		C.041	1.17	0.6061	1.001
48	XXXCla	1294.5		2.51	1531.8		237.3	2.22	1595.0	300.5
35	A LOL				- a ANEL					
4i	2	1214.7		1.10	1405.2		190.5	0.81	1453.2	238.5
A:R		1011 4		1 96	0 2 6 4 1		102 4	1 02	1487.0	9 0 4 9 6
CIT	/	1244.4		00.1	0.1641		1.001	00.1	O. TOFT	0.71.7
4iC	10×Col	1271.5		1.80	1478.6		207.1	1.52	1542.8	271.3
	11/10	PLOAN								
4iD	- The	1277.9		1.96	1492.2		214.3	1.67	1555.1	277.2
	NA AN				1339'3					
4iF		1307.2	Treetd	2.35	1520.3		213.1	1.88	1571.5	264.3
4i(E)	10 AC	1319.3		2.88	1549.5		230.2	2.34	1602.1	282.8
	~ You	124.8								
56		1132.6	1131.2 [1]	0.57	1309.4	1306.5 [1]	176.8	0.40	1361.3	228.7
	A N KCI		11213 2.1.1			1 a leat	E.fer I			
20			1226.2 [1]			1395.5 [1]	169.3			
64	· rci ·	036.6	TANK A	0 00 0	1000 4		971.0	000	1901 9	2447
	x La	0.000	3	67.0	1200.4		6.112	77.0	7.1071	1.440
61	"~ Yoi	878.0		0.58	1308.5		430.5	0.99	13946	446.6
and the second s	2	0.010	101 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1	000	0.0001	Tes o asal	0.004	67.0	0.7201	0.011

tinued	=	11 mo	9.8).2	3.3	0.	6.	
le 2 con	1 25	479	496	249	313	371	387	
Tab	10	1522.6	1556.0	1295.5	1369.3	1519.3	1555.0	
ISC ISC	6	1.32	1.68	0.23	0.42	1.29	1.67	
191	8	393.3	409.7	205.6	256.6	302,5	316.8	
and the second	7					1250		
n n n	9	1436.8	1468.9	1251.9	1312.6	1450.8	1483.9	
A	5	1.34	1.66	0.40	0.60	1.48	1.83	
Sa an	4							
「 一 一 一 一 一 一 一 一 一 一 一 一 一 一 一 一 一 一 一	3	1043.5	1059.2	1046.3	1056.0	1148.3	1167.1	
2 1 1 1 S	of <u>es</u> poceo atin.							22 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2
.u. 941	2	10	1-0	Yor	-Y-Y	July 1	Tor	
12 m 0 1 m	истона 16-2 16-2 16-2 10-2 10-2 10-2 10-2 10-2 10-2 10-2 10	6i(Z)	6i(E)	42	7.	7i(Z)	7i(E)	

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* Code of a tertiary chloride consists of a number designating the left part of the compound and of a letter, designating the right part; *i(E)* and *i(Z)* designate primary allylic isomers according to their configurations and iA...iF designate other isomeric compounds. ** Diastereomers. The structural increment, ΔI^{P-NP} , was determined from the differences in *I* on polar TCEP (or TCEP/DEGS) and nonpolar OV-101 columns (Table 2). The $I^{TCEP} - I^{OV-101}$ values for tertiary alkenyl chlorides vary from 135 to 180 i.u., while those of $I^{TCEP/DEGS} - I^{OV-101}$ are from 155 to 225 i.u. The corresponding values for dichlorides range from 227 to 267 i.u. and from 288 to 329 i.u., respectively. For primary alkadienyl chlorides they vary from 220 to 245 i.u. and from 287 to 312 i.u. respectively. The $I^{TCEP} - I^{OV-101}$ values of the alkoxy-substituted compounds (6, 7) vary from 205 to 410 i.u. and $I^{TCEP/DEGS} - I^{OV-101}$ values from 250 to 497 i.u.

It must be pointed out that the values of I^{TCEP} are lower than those presented in [2]. This may be explained by a very low film thickness (to get a more stable film). We stabilized the TCEP film with a low amount of DEGS. We have been using this column for three years without a considerable decrease in efficiency. At the same time, other resolution parameters are very similar to those of a pure TCEP column.

Conclusions

The relative GC retention data, retention indices, and values of ΔI^{P-NP} for 57 6-chloro-6-methyl-2-alkenes and related compounds on nonpolar methyl silicone OV-101, high polar TCEP and TCEP/DEGS liquid phases using capillary columns have been presented. These data complement the set of Kovats indices and can contribute to the deduction of increments to predict retention parameters of unknown compounds.

A mixed polar coating of TCEP/DEGS has been suggested.

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Ants ERM, Elvi MUKS, Ilme LÕIVEKE

ALKENÜÜLKLORIIDIDE GAASIKROMATOGRAAFIA

On esitatud viiekümne seitsme 6-kloor-6-metüül-2-alkeeni või neile isomeersete ühendite gaasikromatograafilised suhtelised väljumisajad, retentsiooniindeksid ja inkremendi ΔI^{P-NP} väärtused, määratuna 80°C juures mittepolaarsel metüülsilikoonil OV-101, tugevalt polaarsetel 1,2,3-*tris*-(2'-tsüaanoetoksü)-propaanil (TCEP) ja selle segul dietüleenglükooli suktsinaadiga (DEGS), kasutades kapillaarkolonne. Saadud andmed täiendavad retentsiooniindeksite andmepanka ja on kasutatavad indeksite arvutamiseks struktuuriparameetrite alusel.

On leitud, et TCEP/DEGS-seguga impregneeritud kapillaarkolonn on kasutatav suhteliselt ebastabiilse TCEP-kolonni asemel.

Антс ЭРМ, Эльви МУКС, Ильме ЛЫЙВЕКЕ

ГАЗОВАЯ ХРОМАТОГРАФИЯ АЛКЕНИЛХЛОРИДОВ

Представлены хроматографические относительные времена удерживания, индексы удерживания и величины инкремента ΔI^{P-NP} для 57 6-хлор-6-метил-2-алкенов или изомерных им соединений, определенные при 80°С на неполярной метилсиликоновой фазе OV-101, на сильнополярных 1,2,3-*трис*-(2'-цианоэтокси)пропане (TCEP) и смеси его с сукцинатом диэтиленгликоля (DEGS) в капиллярных колонках. Полученные данные могут быть использованы для расчета индексов удерживания близких соединений на основе структурных параметров. Найдено, что пропитанная смесью TCEP/DEGS-колонка может заменять TCEP-колонку вследствие большей стабильности фазы.

The purification of such multicomponent waste waters requires the use of a complicated set of physical and chemical methods. They may build abolicated set of physical and chemical methods. They may flotation cost the submide decompose partially and toxic hydrogen submide is emitted into the arrownere far submide ions are toxic also for acrobic bacteria in the mopunification process. Therefore it is necessary to decrease the supplide concentration of waste waterbefore it is directed to acration and Totation.

but often they are not selective enquelt for example, by the oxidation, of sulphides in waste water with an oxidizer, such as hydrogen peroxide, the bulk of the reagent is consumed in the oxidation of organic matter. This makes the oxidation process very expensive as compared with biochemical oxidation.

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