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Note

Gas-liquid chromatography of some *o*-xylene, indane and tetralin derivatives

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In connection with a study of the chlorination reactions of *o*-xylene, indane and tetralin, we have investigated the gas-liquid chromatography (GLC) of isomeric nitro-*o*-xylenes, nitroindanes, monochloroindanes, dichloroindanes and trichloroindanes, monochlorotetralins and monochloro-*o*-xylenes.

Nitro-*o*-xylenes were prepared by nitration of *o*-xylene and by separation of the two isomers using double vacuum distillation; the two isomeric nitroindanes were prepared by the same method. From the separated nitro derivatives of *o*-xylene and indane, isomeric monochloro-*o*-xylenes and monochloroindanes¹ were prepared by a sequence of reactions involving reduction of the nitro compounds to amines, diazotisation and decomposition of the stabilized diazonium salts. A similar method was used to prepare isomeric chlorotetralins from the corresponding aminotetralins.

Isomeric dichloroindanes were isolated from the mixture of dichloroindanes obtained by chlorination of indane to the second degree by vacuum distillation. 4,5,7-Trichloroindane was obtained by chlorination of 4,7-dichloroindane, 4,5,6-trichloroindane was prepared (although not in absolutely pure form) by nitration of 5,6-dichloroindane, reduction of the nitro derivative, diazotisation of the resulting 4-amino-5,6-dichloroindane and decomposition of the stabilized diazonium salt.

Elemental analyses of the compounds obtained were carried out, and their structures were confirmed by infrared and nuclear magnetic resonance spectroscopy (except for the isomeric trichloroindanes)¹.

EXPERIMENTAL

All chromatographic analyses were carried out with a Fractovap GV 200 apparatus (Carlo Erba, Milan, Italy) equipped with a flame ionization detector; nitrogen was used as carrier gas. Unless otherwise stated, all GLC supports and stationary phases were obtained from Carlo Erba.

The nitro-*o*-xylenes and chlorinated indanes were chromatographed in a glass column (4 m × 5 mm I.D.) packed with Chromosorb A coated with 25% of silicone elastomer SE-52 (this is system A).

Chlorinated indanes were also separated in a stainless-steel column (3 m × 5 mm I.D.) packed with Embacel supporting 10% of Apiezon N (this is system B). The isomeric nitroindanes, chloroindanes and chlorotetralins were separated in a stainless-

steel column (2.8 m × 5 mm I.D.) packed with silanized Chromosorb W impregnated with 15% of ethylene glycol succinate (Lachema, Brno, Czechoslovakia) (this is system C).

A column (3.65 m × 4 mm I.D.) packed with silanized Chromosorb W coated with 20% of dodecyl phthalate-Bentone 34 (1:1) was used for the analysis of isomeric chloro-*o*-xylenes (this is system D).

RESULTS

Results for the separation of *o*-xylene, indane and tetralin derivatives are presented, as elution ratios, in Tables I and II.

TABLE I
GLC OF ISOMERIC NITRO-*o*-XYLENES, NITROINDANES AND CHLORO-*o*-XYLENES

Compound	GLC system*	Column temperature (°C)	Carrier gas pressure (kp/cm ²)	Elution ratio
3-Nitro- <i>o</i> -xylene	A	230	1.00	0.98**
4-Nitro- <i>o</i> -xylene	A	230	1.00	1.28**
4-Nitroindane	C	190	1.00	4.83***
5-Nitroindane	C	190	1.00	5.62***
3-Chloro- <i>o</i> -xylene	D	100	1.50	4.82 [§]
4-Chloro- <i>o</i> -xylene	D	100	1.50	5.24 [§]

* See text.

** Naphthalene as standard (retention time 7.28 min).

*** Naphthalene as standard (retention time 1.31 min).

[§] *o*-Xylene as standard (retention time 14.00 min).

TABLE II
GLC OF CHLORINATED INDANES AND TETRALINS

Compound	Elution ratio in GLC system*			
	C**	A***	A [§]	B ^{§§}
4-Chloroindane	9.95	1.13	—	0.89
5-Chloroindane	11.80	1.29	—	1.00
5-Chlorotetralin	21.30	—	—	—
6-Chlorotetralin	23.60	—	—	—
4,7-Dichloroindane	—	—	1.75	2.22
4,5-Dichloroindane	—	—	1.96	2.89
5,6-Dichloroindane	—	—	2.02	3.22
4,5,7-Trichloroindane	—	—	2.84	5.67
4,5,6-Trichloroindane	—	—	3.34	8.44

* See text.

** Column temperature, 115°; carrier gas pressure, 1.15 kp/cm²; chlorobenzene as standard (retention time 0.63 min).

*** Column temperature, 220°; carrier gas pressure, 0.90 kp/cm²; naphthalene as standard (retention time 7.28 min).

[§] Column temperature, 290°; carrier gas pressure, 0.90 kp/cm²; naphthalene as standard (retention time 2.67 min).

^{§§} Column temperature, 170°; carrier gas pressure 1.20 kp/cm²; 5-chloroindane as standard (retention time 6.00 min).

CONCLUSIONS

Various monochlorinated derivatives of *o*-xylene, indane and tetralin, and some dichlorinated indanes and trichlorinated tetralins containing chlorine in the aromatic ring, have been separated by GLC, and the technique has been utilised for identifying nitroindanes and nitro-*o*-xylenes. However, GLC cannot be used for the identification of indane and tetralin derivatives chlorinated in the aliphatic ring because of the thermal lability of these compounds and the possibility of polymerization of the resulting decomposition products during analysis.

REFERENCE

- 1 J. Novročík and J. Poskočil, *Collect. Czech. Chem. Commun.*, in press.