Journal of Chromatography, 81 (1973) 142–143

CHROM. 6704

Note

Gas chromatographic separation of 1- and 2-halogenodecaboranes

JIŘÍ STUCHLÍK

Institute of Inorganic Chemistry, Czechoslovak Academy of Sciences, Rez, near Prague (Czechoslovakia)

(Received March 22nd, 1973)

In the preparation of pure 1- and 2-isomers of halogenodecaboranes ($XB_{10}H_{13}$)[1], where X is chlorine, bromine or iodine, the analytical separation of the synthesized substances is necessary. As we did not succeed in separating them by thin-layer chromatography, it seemed promising to attempt to separate them by gas chromatography as this technique had been used successfully for the separation of other boron compounds[2–7].

The synthesis of halogenoboranes was carried out according to the literature[8–12], and the products were sublimed in a vacuum of 10^{-4} torr.

The chromatographic separation was performed on Chrom-3 apparatus (Laboratory Equipment N.E., Prague, Czechoslovakia) equipped with a heat conductivity detection cell and operated at 140–170°. Electrolytic hydrogen was used as the carrier gas, and was dried by means of a molecular sieve trap. Samples were applied as 10% solutions in toluene with a 10-μl Hamilton syringe.

A stainless-steel column of length 2.4 m and I.D. 6 mm was packed with 60–80 mesh silanized Chromosorb W coated with 3% (w/w) of QF-1 silicone oil.

Table I gives the specific retention volumes, V_g, of the $XB_{10}H_{13}$ isomers at 161, 151 and 141° and Kovats' retention indices, I, at 151°.

Fig. 1 shows the gas–liquid chromatographic separation of a mixture of all

TABLE I

SPECIFIC RETENTION VOLUMES AND KOVATS' RETENTION INDICES

Substance	V_g			I
	161°	151°	141°	
$B_{10}H_{14}$	7.50	10.23	17.28	1447
1-$ClB_{10}H_{13}$	26.25	36.20	72.32	1751
2-$ClB_{10}H_{13}$	30.00	41.50	85.25	1784
1-$BrB_{10}H_{13}$	38.75	56.70	112.6	1859
2-$BrB_{10}H_{13}$	46.20	67.0	133.1	1899
1-$IB_{10}H_{13}$	40 0	52.7	115.2	1841
2-$IB_{10}H_{13}$	63.0	100.2	185.6	1996
$C_{15}H_{32}$	—	10.65	—	1500
$C_{20}H_{42}$	—	102.0	—	2000

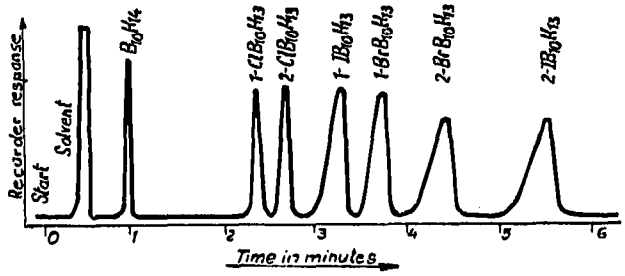

Fig. 1. Chromatogram of 1- and 2-$XB_{10}H_{13}$ isomers on a 2.4-m stainless-steel column packed with 60–80 mesh silanized Chromosorb W coated with 3% (w/w) of QF-1 silicone oil. Column temperature 151°.

of the 1- and 2-$XB_{10}H_{13}$ isomers. The separation was performed at 151° with hydrogen at the flow-rate of 105 ml/min. It is evident from the V_g values and from Fig. 1 that the separation of all of the individual 1- and 2-isomers is satisfactory and is achieved at an adequate speed. Similar separations on columns packed with Apiezon L or SE-30 do not take place and polar phases such as Carbowax 20M or polyesters react with the substances being separated.

ACKNOWLEDGEMENT

The author thanks Mrs. M. Šotolová for her technical help.

REFERENCES

1 J. Stuchlík, S. Heřmánek, J. Plešek and B. Štíbr, *Collect. Czech. Chem. Commun.*, 35 (1970) 339.
2 E. J. Sowinski and I. H. Suffet, *J. Chromatogr. Sci.*, 9 (1971) 632.
3 L. J. Huns, R. S. Braman and J. E. Graham, *Anal. Chem.*, 34 (1962) 1700.
4 N. J. Blay, J. Williams and R. L. Williams, *J. Chem. Soc.*, (1960) 424.
5 J. Dunstan, R. L. Williams and N. J. Blay, *J. Chem. Soc.*, (1960) 5012.
6 R. L. Williams, I. Dunstan and N. J. Blay, *J. Chem. Soc.*, (1960) 5006.
7 J. J. Kaufman, J. E. Tood and W. S. Koski, *Anal. Chem.*, 29 (1957) 1032.
8 R. Schaeffer, J. N. Schoolery and R. Jones, *J. Amer. Chem. Soc.*, 80 (1958) 2670.
9 M. Hilman, *J. Amer. Chem. Soc.*, 82 (1960) 1096.
10 *U.S. Pat.*, 3,046,086 (1962).
11 *U.S. Pat.*, 3,106,446 (1962).
12 L. I. Zakharkin and V. N. Kalinin, *Zh. Obshch. Khim.*, 36 (1966) 2160.