Synthesis and Characterisation of a Tetrahydrofuran Derivative of Scandium Tetrahydroborate

By J. H. MORRIS* and W. E. SMITH

(*Department of Pure and Applied Chemistry, University of Strathclyde, George Street, Glasgow, C.*1)

Summary A tetrahydrofuran derivative of scandium tetrahydroborate $Sc(BH_4)_3$·THF has been prepared and some of its spectroscopic properties investigated.

THERE has been considerable recent interest in covalent metal hydroborates and their derivatives with ligands, in particular derivatives of aluminium, titanium, zirconium, and hafnium although neither the corresponding scandium tetrahydroborate nor its derivatives have been reported.[1,2] We now describe the first hydroborate derivative of scandium, $Sc(BH_4)_3$·THF.

Dry tetrahydrofuran was cold-distilled under vacuum on to anhydrous scandium trichloride and lithium tetrahydroborate (in a slight excess). After standing for some days, the mixture was filtered, solvent was removed under vacuum, and the resultant white solid was purified by rapid sublimation at *ca.* 80° on to an ice–salt probe. The extremely moisture-sensitive sublimate contained no chlorine, and appeared to be thermally stable at room temperature.

The major features of the Raman and i.r. spectra of the compound are:—

Raman†		2899	2522	2422	2372	2249	2164		1484	1455
I.r.‡	2995	2905	2510	2425	2370	2240	2150	2058		1460
	C–H str		B–H str							

Raman†	1370	1254			1042		926	866	844		477	435	377
I.r.‡	1360		1185	1128	1042	1010	920	855		678	450		

† Spectrum recorded on solid sample.
‡ Spectrum recorded on Nujol and hexachlorobutadiene mulls.

The i.r. bands in both the C–H and B–H stretching regions were characteristic of the complex, and repeated sublimation did not change their relative intensities, thus indicating that the compound was free from unco-ordinated solvent. The frequencies and contours of most of the bands of tetrahydrofuran[3] can be identified in the spectrum of the compound, although the strong band near 1080 cm^{-1} is missing or considerably shifted. A broad, strong band at about 450 cm^{-1} is tentatively assigned to a metal–oxygen stretching vibration, suggesting that the tetrahydrofuran is co-ordinated to the scandium atom in a similar manner to the ligand–metal bond in $Al(BH_4)_3$·NMe_3.[4]

The nature of the compound is established by an examination of its 70 ev mass spectrum at 90° and 2×10^{-6} mm. The highest *m/e* values of any group of significant peaks occur in the range 156—162, and may be correlated with the molecular ion $[Sc(BH_4)_3\cdot THF]^+$, although the intensity distribution indicates considerable contribution from species with fewer hydrogens. The first major group of fragment ions are observed in the range *m/e* 142—149 and mass measurement of the major peak at *m/e* 147 gave 147·09525 a.m.u. {theor. for $[Sc(^{11}BH_4)_2\cdot THF]^+$, 147·09465 a.m.u.}. The second major group of fragment ions occur in the range *m/e* 128—134 with predominant features at 131 and 132, and mass measurement of the latter gave 132·054355 a.m.u. {theor. for $[Sc(^{11}BH_4)\cdot THF]^+$, 132·054040 a.m.u.}. Other major ions are found near *m/e* 118, 90, 72, and 42.

The likely formula for the compound is therefore $Sc(BH_4)_3$·THF and this was confirmed by analysis for scandium by atomic absorption spectrophotometry.

(*Received, January 8th,* 1970; *Com.* 033.)

---

[1] (a) H. G. Heal, Recent Studies in Boron Chemistry, R.I.C. Monograph, 1960, no. 1; (b) "Boron, Metalloboron Compounds and Boranes," ed. R. M. Adams, Interscience, London, 1964, p. 373.

[2] (a) N. Davies, B. D. James, and M. G. H. Wallbridge, *J. Chem. Soc.* (*A*), 1969, 2601, and references therein; (b) B. D. James, R. K. Nanda, and M. G. H. Wallbridge, *J. Chem. Soc.* (*A*), 1966, 182, and references therein.

[3] N. Baggett, S. A. Barker, A. B. Foster, R. H. Moore, and D. H. Whiffen, *J. Chem. Soc.*, 1960, 4565, and references therein.

[4] N. A. Bailey, P. H. Bird, and M. G. H. Wallbridge, *Inorg. Chem.*, 1968, 7, 1575.