The Reaction of Pentaborane(9) with Small Carbon Molecules

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SKELL, *et al.*[1] in 1965, used a carbon arc to prepare highly reactive carbon vapour and have studied its reactions with various organic compounds.[1,2] In general, addition compounds are formed (*e.g.* olefins form spiropentanes or bisethanoallenes) but insertion reactions are also known. It would appear interesting to study the reaction of small carbon molecules with many inorganic substrates including boron compounds.

We have used a brass reactor of Skell's basic design[1] operated with a 100 ampere D.C. source to produce the usual mixture of carbon molecules and have studied their reaction with pentaborane(9). In a typical run, 6 g. of B_5H_9 was added to the cell in 40 min. (The arc was cycled on and off in a 1 : 3 second cycle to allow the evolved hydrogen to be pumped off.) Of the B_5H_9 added, approximately 0·45 g. reacted to form volatile products and a non-volatile, slightly pyrophoric and, as yet, intractable material. The volatile products were separated from the majority of the B_5H_9 by conventional high-vacuum techniques and were further purified by gas chromatography.

The major volatile component (2·7% of the B_5H_9 consumed) has a parent mass envelope at *m/e* of 65—74. Exact mass measurements on the 74 peak showed it to be due to $^{11}B_5{}^{12}C^{1}H_7^+$ (exp. 74·10170; calc. 74·10133). The ^{11}B n.m.r. of the material agrees with that of monocarbonhexaborane(7), B_5CH_7, prepared by Onak *et al.*[3]

The second and third products (parent peaks at *m/e* 86 and 101) have not yet been isolated in pure form. However, absolute mass measurements have shown the parent peak of the second component to be due to $^{11}B_5{}^{12}C_2{}^{1}H_7^+$ (exp. 86·10154; calc. 86·10131).

Study of the chemistry of B_5CH_7 has been started and in the initial experiment, 0·18 mmole of B_5CH_7 was allowed to react with excess NaH in tetrahydrofuran and 0·18 mmole of hydrogen was released. The ^{11}B n.m.r. spectrum consisted of two sets of doublets ($\delta = +2{\cdot}2$, *J* 133; $\delta = +15{\cdot}5$ p.p.m., *J* 136 Hz) in the ratio of 1 : 4. A sample of $B_5CH_6^-$ was reacted with excess anhydrous HCl regenerating B_5CH_7. This data confirms B_5CH_7 to be the conjugate acid of $B_5CH_6^-$ as suggested by Onak *et al.*[3] and also confirms the ion to have a near octahedral structure. The position of the bridge proton still remains to be determined. There is no evidence at present for the existence of more than a single isomer of B_5CH_7 although two are possible.

We thank the National Science Foundation for financial support (S.R.P.).

(*Received, February* 19*th,* 1968; *Com.* 201.)

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