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Stoichiometric Evidence for the Dihydrate of Diborane

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Summary The dihydrate of diborane has been prepared by the reaction of diborane with water at -130° .

It is well known that diborane forms adducts with various Lewis bases, including ammonia.¹ However, no adduct with water has previously been isolated because of water's relatively high reactivity as a protonic acid; even at -80° diborane undergoes hydrolysis with hydrogen evolution.² By the following technique, we have achieved hydrate formation without hydrolysis. Diborane and water were cocondensed as an intimate solid mixture at -196° by allowing their vapors to separately but simultaneously effuse into a glass vessel cooled in liquid nitrogen. The mixture was warmed to the highest temperature at which hydrogen evolution was negligible, i.e., -130° ; at this temperature diborane has sufficient vapor pressure (50mm) to facilitate reaction with the water.

In experiments in which an excess of water was used, the reaction mixture was held at -130° for various time intervals, and then any unreacted diborane was pumped out and measured. With a reaction time of 15 min., 14% of the diborane was recovered; with a reaction time of 110 min., only 2% of the diborane was recovered. The fact that

continued pumping for 1 hr. yielded only a trace of diborane is evidence that the unrecovered diborane had reacted with the water.

When excess diborane was used, the unreacted diborane was removed at -130° by pumping after a 2-hr. reaction time. In several experiments, the observed mole ratio of water to consumed diborane ranged from 1.9 to 2.3, thus indicating the empirical formula $B_2H_6 \cdot 2H_2O$ for the reaction product. The results of similar studies of the reactions of diborane with methanol and ethanol indicate that alcoholates of the composition $B_2H_6 \cdot 2ROH$ are formed. When the dihydrate was treated with excess trimethylamine at -130° and then warmed to room temperature, hydrogen, trimethylamine borane, and an uncharacterized residue were formed.

The above data give no information regarding the structure of the dihydrate and dialcoholates of diborane. We hope that, in the case of the dihydrate, low temperature infrared spectrometry and X-ray photoelectron spectroscopy can be used to distinguish between the two likely structures, viz. $BH_2(OH)_2^+ BH_4^-$ and H_2OBH_3 .

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References

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- 2 H. G. Weiss and I. Shapiro, J. Amer. Chem. Soc., 1953, 75, 1221;
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