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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. 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. 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$_2$H$_6$·2H$_2$O 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$_2$H$_6$·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$_2$OBH$_3$.

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References


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