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Notes

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Slow Solution Reaction of Dimeric Dimethylaminoborane with Lithium Hydride

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There are no literature reports on the interaction of an alkali metal hydride with $[(CH_3)_2NBH_2]_2$ under mild conditions. Burg and Randolph[1] showed that $[(CH_3)_2NBH_2]_2$ reacted with NaH (but not LiH) at elevated temperature in the absence of solvent according to

$$[(CH_3)_2NBH_2]_2 + NaH \rightarrow HB[N(CH_3)_2]_2 + NaBH_4 \qquad (1)$$

This reaction probably results from a thermally induced disproportionation of $[(CH_3)_2NBH_2]_2$

$$[(CH_3)_2NBH_2]_2 \rightarrow HB[N(CH_3)_2]_2 + {}^1/_2B_2H_6 \qquad (2)$$

$$NaH + {}^1/_2B_2H_6 \rightarrow NaBH_4 \qquad (3)$$

As part of another project, we have had the opportunity to observe the action of LiH on $[(CH_3)_2NBH_2]_2$ at room temperature in diethyl ether over periods of several months. We find that under these conditions the sole final product is $Li(CH_3)_2N{\cdot}BH_3$.

$$[(CH_3)_2NBH_2]_2 + 2LiH \xrightarrow[25°]{(C_2H_5)_2O} 2Li(CH_3)_2N{\cdot}BH_3 \qquad (4)$$

A typical reaction was 90% complete after 2 months at ambient temperature.

If the system is monitored periodically by boron-11 nmr, weak signals due to an intermediate product can be detected (Figure 1). The nmr parameters compare favorably with those of the ion $(CH_3)_2NBH_2N(CH_3)_2BH_3^-$ [2] (triplet −2.4 ppm, J_{BH} = 92 Hz; quartet 13.1 ppm, J_{BH} = 81 Hz; data for potassium salt in 1,2-dimethoxyethane). The presence of this ion strongly suggests that the main pathway for eq 4 involves ring opening by reaction of LiH with dimethylaminoborane *dimer* followed in a subsequent step by cleavage of the NBNB chain by LiH.

$$[(CH_3)_2NBH_2]_2 + LiH \xrightarrow{(C_2H_5)_2O} Li(CH_3)_2N\text{-}BH_2\text{-}N(CH_3)_2\text{-}BH_3 \qquad (5)$$

$$Li(CH_3)_2N\text{-}BH_2\text{-}N(CH_3)_2\text{-}BH_3 + LiH \xrightarrow{(C_2H_5)_2O} 2Li(CH_3)_2N{\cdot}BH_3 \qquad (6)$$

The cleavage reaction (eq 6) has been demonstrated elsewhere.[2]

Experimental Section

Standard vacuum techniques were used in this work. Lithium hydride (Alfa Inorganics) was used without further purification; $[(CH_3)_2NBH_2]_2$ was prepared by the pyrolysis of $(CH_3)_2NH{\cdot}BH_3$ (Research Organic/Inorganic Chemical Corp.).[3] Diethyl ether was stored in an evacuated bulb over $LiAlH_4$ and when needed was condensed directly into the reaction vessel. Boron-11 nmr spectra were obtained with a Varian HA-100 spectrometer operating at 32.1 MHz. Chemical shifts and coupling constants were measured by the substitution side band method.

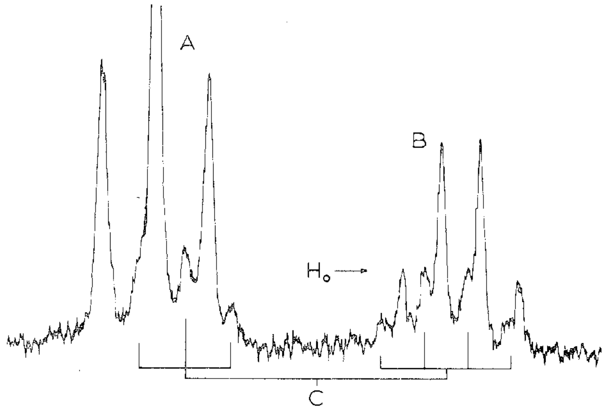

Figure 1. Boron-11 nmr spectrum of the partially complete reaction of $[(CH_3)_2NBH_2]_2$ with LiH after 2.5 weeks: A, triplet, $[(CH_3)_2NBH_2]_2$, −5.1 ppm, J = 113 Hz; B, quartet, $(CH_3)_2NBH_3^-$, 15.0 ppm, J = 83 Hz; C, $(CH_3)_2N\text{-}BH_2\text{-}N(CH_3)_2\text{-}BH_3^-$, triplet, −2.7 ppm, J = 100 Hz; quartet, 14.1 ppm, J = 88 Hz. Chemical shifts are relative to $(C_2H_5)_2O{\cdot}BF_3$.

In a typical experiment an all-Pyrex nmr reaction vessel was charged with 8–10 mmol of LiH, 1–2 mmol of $[(CH_3)_2NBH_2]_2$ and 1 ml of diethyl ether were condensed in, and the vessel was sealed. The mixture was allowed to stand at room temperature for 2–3 months with occasional shaking and periodic monitoring by nmr. Products were identified by their nmr parameters.

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Registry No. $[(CH_3)_2NBH_2]_2$, 23884-11-9; LiH, 7580-67-8; ^{11}B, 14798-13-1.

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Reactions of Coordinated Nickel(II) Complexes Containing Imine and Amine Groups[1]

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Nickel(II) complexes (I, II) containing a Schiff base ligand derived from pyrrole-2-carboxaldehyde and dipropylenetriamine (bis(2-pyrrolyl)-2,6,10-triaza-1,10-undecadiene