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A New Method for the Preparation of B_6H_{10}

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The study of the chemistry of B_6H_{10} has been largely hampered by a lack of a suitable method of preparation, which we now report from reaction of $B_9H_{14}^-$ ion[1,2] with polyphosphoric acid.[3] The yield is only 2.5% based upon $B_9H_{14}^-$, and 14 mole % of the volatile hydrides. Nevertheless, $B_9H_{14}^-$ can be made available in large quantities, the volatile materials are easily separated by use of standard vacuum line techniques, and the reaction is very easily carried out.

The starting material, $(C_2H_5)_3NHB_9H_{14}$, prepared by reaction[2] of $B_{10}H_{14}$ with KOH, was allowed to react at room temperature with polyphosphoric acid in an evacuated flask. The volatile boranes, B_2H_6 (40 mole % yield), B_4H_{10} (37%), B_5H_{11} and B_5H_9 (10%), B_6H_{10} (14%), and traces of B_8H_{12} and $B_{10}H_{14}$, were caught in a series of traps at −196° and then separated in a small number of fractional condensations. Only B_8H_{12} remained with the B_6H_{10}, but it decomposed quantitatively[4] when the B_6H_{10} was warmed to 0° for vapor pressure measurements. Separation of the B_6H_{10}–B_8H_{12} mixture below −20° may also yield reasonable amounts of B_8H_{12} for further study. All hydrides were identified by mass spectrometric methods,[5] and, in addition, the B_6H_{10} was shown to have a vapor pressure of 7.2 mm. at 0°.

We believe that this method of preparation is more convenient, in spite of the low yield, than boride hydrolysis,[6,7] discharge methods,[8,9] or base-catalyzed conversion[10,11] from other hydrides.

Experimental

The $(C_2H_5)_3NHB_9H_{14}$ was prepared according to the procedure of Benjamin, Stafiej, and Takacs.[2] Solid $(C_2H_5)_3NHB_9H_{14}$ (1 g., 4.7 mmoles) was then added slowly *in vacuo* to polyphosphoric acid. This mixture was continuously evacuated and stirred with the use of an Asco Laboratory gland stirrer (Arthur F. Smith Co.) which was lubricated with No. 90 Kel-F grease. Volatile products were trapped in three successive U-tubes cooled to −196° with liquid nitrogen. After 3 days the effervescence had stopped, and the gas pressure measured with a McLeod gauge had been constant for 1 day. The $B_{10}H_{14}$ was then separated from more volatile hydrides by passage of the mixture through a U-tube cooled to −23°. Three quick passes through a −63° trap then retained B_6H_{10} and B_8H_{12}, but allowed the B_2H_6, B_4H_{10}, B_5H_{11}, and B_5H_9 to pass through. After the B_6H_{10}–B_8H_{12} mixture was raised to 0° for measurement of its vapor pressure, a considerable pressure of H_2 remained upon recooling to −196°. Since the remaining liquid was pure B_6H_{10}, we concluded that the B_8H_{12} had decomposed. The presence of B_8H_{12} before the sample was warmed to 0° was proved by the observation of its characteristic pattern[4] in the mass spectrum, including, in particular, the maximum peak at 93 and the parent peak at 100 mass units. After the sample had been warmed to 0° this pattern disappeared, leaving only the B_6H_{10} peaks in the mass spectrum. About 0.12 mole of B_6H_{10} remained. Yields of B_2H_6 (0.33 mmole), B_4H_{10} (0.31 mmole), and B_5H_{11} and B_5H_9 (0.08 mmole) were also determined after separation by fractional condensation. The vapor pressure of B_6H_{10} at 0° (7.2 mm.) is in agreement with the value given by Stock,[6] and the mass spectrum was found to be exactly like the published spectrum.[5]

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The Nature of the Nickel Boride Formed by the Action of Sodium Borohydride on Nickel Salts

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Investigations indicate that finely divided nickel boride is useful in catalysts[1,2] and fuel cell electrodes.[3] The action of solutions of sodium or potassium borohydride on solutions of nickel salts has been described as resulting in nickel boride, Ni_2B.[4,5] Though nickel boride formed by the action of sodium borohydride on nickel salts has received considerable attention as a catalyst, no structure study of this nickel boride has

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