

## The Reaction of *NN'N''*-Trimethylborazine with Stannic Halides

By G. A. ANDERSON, and J. J. LAGOWSKI

(Department of Chemistry, The University of Texas, Austin, Texas)

IN the course of a general study of the borazines we have observed that *NN'N''*-trimethylborazine undergoes reaction with stannic halides to form *B*-halogeno-derivatives with the concomitant formation of the corresponding stannous halide and the hydrogen halide-borazine adduct, *i.e.*,  $\text{Me}_3\text{N}_3\text{B}_3\text{H}_3 \cdot 3\text{HX}$ .

Stannic chloride (22.7 g.) reacts immediately with *NN'N''*-trimethylborazine (6.4 g.) at  $-23^\circ$  to yield the substitution products  $\text{Me}_3\text{N}_3\text{B}_3\text{H}_2\text{Cl}$  and  $\text{Me}_3\text{N}_3\text{B}_3\text{HCl}_2$ , and a small amount of  $\text{Me}_3\text{N}_3\text{B}_3\text{Cl}_3$ . Stannous chloride and hydrogen chloride are also formed in the reaction, the latter subsequently reacting with *NN'N''*-trimethylborazine to produce the addition product  $\text{Me}_3\text{N}_3\text{B}_3\text{H}_3 \cdot 3\text{HCl}$  which we have identified by comparison with an authentic sample prepared in this laboratory. The *B*-chloro-substituted borazines ( $\text{Me}_3\text{N}_3\text{B}_3\text{H}_2\text{Cl}$ , 20%;  $\text{Me}_3\text{N}_3\text{B}_3\text{HCl}_2$ , 60%;  $\text{Me}_3\text{N}_3\text{B}_3\text{Cl}_3$ , 20%) were separated from the reaction mixture by fractional sublimation and their identities confirmed by mass spectroscopy. With increasing halogen substitution, the infrared absorption spectrum of the *B*-chloroborazines shows a

decrease in intensity of the B-H stretching mode<sup>1</sup> at  $2515\text{ cm}^{-1}$ , and a corresponding increase in intensity of the B-Cl stretching mode<sup>2</sup> at  $975\text{ cm}^{-1}$ .

Stannic bromide (18.9 g.) does not react with *NN'N''*-trimethylborazine (5.8 g) at room temperature but at  $110^\circ$  substituted *B*-bromoborazines analogous to the *B*-chloroborazines described above are produced. In addition, stannous bromide and hydrogen bromide are formed. At elevated temperatures the adduct  $\text{Me}_3\text{N}_3\text{B}_3\text{H}_3 \cdot 3\text{HBr}$  is unstable<sup>3</sup> and hydrogen bromide is evolved from the reaction vessel. The substituted *B*-bromoborazines were separated from the reaction mixture by fractional sublimation and identified by mass spectroscopy. The distribution of products (estimated from mass spectroscopic data) from the reaction of equimolar portions of  $\text{Me}_3\text{N}_3\text{B}_3\text{H}_3$  and  $\text{SnBr}_4$  is:  $\text{Me}_3\text{N}_3\text{B}_3\text{H}_2\text{Br}$ , 10%;  $\text{Me}_3\text{N}_3\text{B}_3\text{HBr}_2$ , 80%; and  $\text{Me}_3\text{N}_3\text{B}_3\text{Br}_3$ , 10%.

The reaction of *NN'N''*-trimethylborazine with stannic iodide does not proceed at room temperature or at  $155^\circ$  (reflux for 2 hr.).

Mercuric chloride does not react with *NN'N''*-trimethylborazine at room temperature, but at

150° a reaction occurs forming *B*-monochloro-, *B*-dichloro-, and *B*-trichloro-*NN'**N''*-trimethylborazine. The mercury(II) was reduced to

a mixture of mercury(I) and free mercury during the course of the reaction.

(Received, August 15th, 1966; Com. 602.)

<sup>1</sup> H. Watanabe, Y. Kuroda, and M. Kubo, *Spectrochim. Acta*, 1961, 17, 454.

<sup>2</sup> H. Watanabe, M. Narisada, T. Nakagawa, and M. Kubo, *Spectrochim. Acta*, 1960, 16, 78.

<sup>3</sup> cf. A. W. Laubengayer, O. T. Beachley, Jr., and Richard F. Porter, *Inorg. Chem.*, 1965, 4, 578.