The enthalpy of dissociation of $CF_3SF_3 \cdot BF_3(s)$ (24.6 kcal/mol) is of the same magnitude as that reported for the $SF_4 \cdot BF_3$ dissociation (25.5,[4] 24.8 kcal/mol[5]) while the entropy of $CF_3SF_3 \cdot BF_3$ dissociation (82.2 eu) is considerably larger than that reported for the $SF_4 \cdot BF_3$ adduct (74.5,[5] 72.5 eu[4]). Consequently the equilibrium dissociation pressure above the CF_3SF_3 adduct at 0° (207 Torr) is greater than that of $SF_4 \cdot BF_3$ at the same temperature (20 Torr).[5]

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Correspondence

A Potentially Hazardous Reaction between Dimethylmercury and Tetrachlorodiborane(4)

Sir:

At the recent American Chemical Society National Meeting, Kuo and Wartik[1] alluded to a private communication from us describing the preparation of trichloromethyldiborane(4) by the reaction $B_2Cl_4 + Hg(CH_3)_2 \rightarrow B_2Cl_3CH_3 + HgClCH_3$. At the time Kuo and Wartik mentioned this work we had without incident carried out this reaction some 23 times. Each of our next two attempts to repeat the preparation, however, resulted in violent explosions, and we are writing to warn any who heard the talk and might wish to use this reaction that it is potentially hazardous.

The most disturbing aspect of this work is that the reagents and conditions used for the two reactions which culminated in explosions were, to the best of our knowledge, in no way different from those employed earlier. The tetrachlorodiborane(4) used[2] in the explosive reactions was taken from the same supply used for prior successful preparations. All of the dimethylmercury used for these studies was prepared at one time according to the method of Marvel and Gould.[3] The identity and purity of the diboron compound were confirmed by its infrared spectrum and its vapor pressure; those of the mercurial were established by its infrared spectrum and by gas chromatographic analysis.

In all cases, equimolar quantities (from 1 to 4 mmol) of the two reagents were condensed at liquid nitrogen temperatures into Pyrex reaction vessels whose volumes varied from about 15 to 60 ml, depending on the particular experiment. (The two ill-starred reactions involved about 4 mmol of each reagent, and the volumes of the reaction vessels were about 30 ml.) After they had been sealed from the vacuum system, the reaction vessels were allowed to warm until the dimethylmercury melted (about $-63°$). In the first 23 cases the reaction, which commenced at about $-63°$, was vigorous but well under control, and, about 2 min after the initial melting of the dimethylmercury, the vessel was cooled to liquid nitrogen temperatures prior to opening the tube to the vacuum system for removal and separation of products. Reaction 24 exploded just prior to opening the tube to the vacuum system. Reaction 25 (the last one we have attempted to date) exploded at the onset of melting of the dimethylmercury. Both explosions were accompanied by bright flashes and both were of sufficient force to pose a serious hazard from the standpoint of flying glass, fire, and volatile mercurials.

We are currently attempting to account for this erratic behavior. Until and unless a set of conditions can be specified for which the reaction is entirely safe, we would advise others not to attempt to reproduce this reaction unless they are fully prepared to cope with the explosion that may result.

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[1] H. S. Kuo and T. Wartik, "Some New Boron-Carbon Heterocyclics," presented before the Division of Inorganic Chemistry at the 160th National Meeting of the American Chemical Society, Chicago, Ill., Sept 1970, Abstract INOR 64.

[2] Tetrachlorodiborane(4) was prepared by the method of T. Wartik, R. Moore, and H. Schlesinger, *J. Amer. Chem. Soc.*, **71**, 3265 (1949).

[3] C. S. Marvel and V. L. Gould, *ibid.*, **44**, 153 (1922).