The effect of the trace of tungsten in the uranium dicarbide specimens used in our earlier hydrolysis studies[2] was only slightly larger than the experimental error (±10% of the value reported for hydrogen, the least accurate analysis). It does not account for the discrepancies between our data and those of others, although variations in the tungsten concentration in our specimens may be responsible for some minor variations in our data. The neutron activation analysis for traces of tungsten was not sufficiently accurate to confirm this.

Acknowledgment.—The authors thank R. E. McDonald and L. Queener of the ORNL Metals and Ceramics Division for preparing the specimens, and R. J. Gray and B. C. Leslie for the metallographic examinations. We are also indebted to the ORNL Analytical Chemistry Division, in particular J. H. Hackney for gas chromatographic analyses, R. L. Sherman for X-ray analyses, and the group of W. R. Laing for most of the chemical analyses.

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Carborane Phosphate Exocycle

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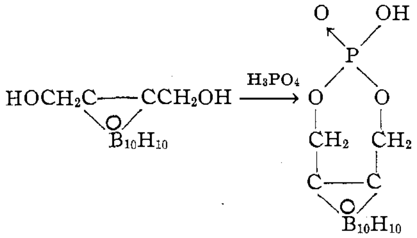
Received July 24, 1964

We wish to report the formation of a carborane exocycle prepared from 1,2-bis(hydroxymethyl)carborane and polyphosphoric acid.

Disubstituted carborane compounds have been reported to form 1,2-exocyclic compounds with ease. Numerous examples of the participation of the two carbon atoms of the carborane nucleus in five-membered rings have been reported.[1-4] The formation of six-membered[5] compounds (two carboranes contribute to ring) and a seven-membered[6] exocyclic compound has also been reported. This communication describes the formation of another seven-membered exocyclic compound.

Experimental

A mixture of 5.5 g. (0.03 mole) of 1,2-bis(hydroxymethyl)-carborane and 60 g. of polyphosphoric acid was allowed to react at 100–110° for 12–14 hr. Approximately 300 ml. of water was added to the slurry and then filtered to yield 9.4 g. of a solid. The solid was dissolved in ether and the solution was dried over magnesium sulfate. Evaporation of the ether yielded a white, crystalline solid which was recrystallized from hot benzene to give a solid, m.p. 259–260°. The product was soluble in acetone and hot acetonitrile, slightly soluble in benzene, and insoluble in pentane. The mass spectrum showed the most intense peaks in the m/e 262 to 268 region. The infrared spectrum (Figure 1) of the product contained absorption bands at 2899 (C–H), 2584 (B–H), 1653 (>P(O)(OH)), 1468 (CH_2), 1290 (PO), 1176 ($-CH_2OP$), 1081 (POC), 1026 (BH_2), and 727 cm.$^{-1}$.



Anal. Calcd. for $C_4H_{15}B_{10}O_4P$ (266.25): C, 18.05; H, 5.68; B, 40.61; P, 11.63. Found: C, 18.04; H, 5.62; B, 40.50; P, 11.82.

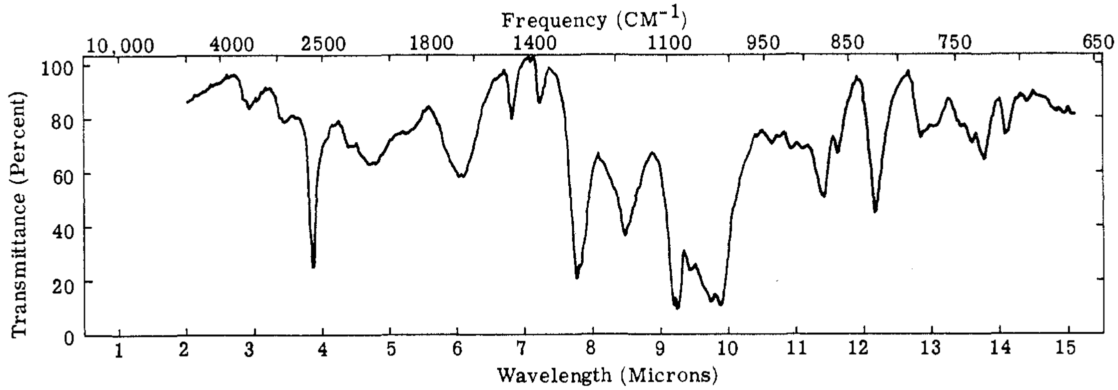


Figure 1.—Carborane phosphate exocycle.

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