"soluble"-ribonucleic acid. The partition chromatographic procedure has the great advantage of simplicity.

Experiments are underway to improve and extend these results.

Acknowledgment.—This work was supported in part by a grant from the National Science Foundation.

U. S. PLANT, SOIL AND NUTRITION LABORATORY
AGRICULTURAL RESEARCH SERVICE, U.S.D.A., AND
DEPARTMENT OF BIOCHEMISTRY GEORGE A. EVERETT
CORNELL UNIVERSITY SUSAN H. MERRILL
ITHACA, NEW YORK ROBERT W. HOLLEY

RECEIVED SEPTEMBER 19, 1960

THE STRUCTURE OF THE $B_3H_8^-$ ION

Sir:

An X-ray diffraction crystal structure study of the diammoniate of tetraborane, $B_4H_{10}\cdot 2NH_3$, conducted at room temperature as well as at −100° shows that the crystals are orthorhombic with room temperature unit cell parameters a = 9.25, b = 9.42 and c = 8.23 Å. The space group is Pbnm and the cell contains four formula units.

Analysis of the two independent sets of diffraction data comprising 433 and 534 reflections, respectively, confirms the ionic structure $[(H_3N)_2BH_2^+][B_3H_8^-]$.[1] Fig. 1(a) is a composite electron density map of the $B_3H_8^-$ ion, for which several different structures have been proposed,[1-5] including the one supported by our results.[1-3] The boron atoms form an isosceles triangle of 1.80 Å. base and 1.77 Å. sides, the latter being B–H–B bridges. Each boron atom has two "regular" (*i.e.*, non-bridge) hydrogens on opposite sides of the boron plane. All B–$H_{regular}$ distances are in the range 1.05–1.20 Å. Approximate bridge B–H distances are B_{apex}–H_{bridge} = 1.5 Å. and B_{base}-H_{bridge} = 1.2 Å. The bridge hydrogen atom appears somewhat elongated at both temperatures. The over-all agreement is $R = \Sigma||F_0|-|F_c||/\Sigma|F_0|$ = 0.084 and 0.077 for observed reflections at −100° and room temperature, respectively.

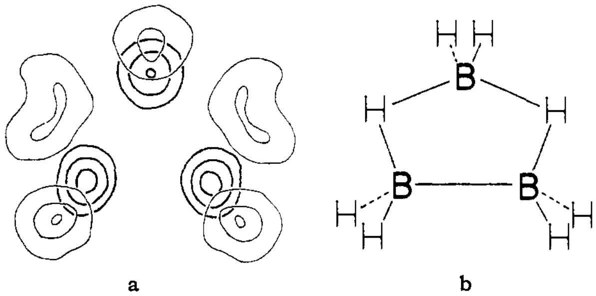

Fig. 1(a).—Composite electron density map of $B_3H_8^-$: heavy contours are at intervals of 2.5 e.Å^{-3}. Light contours showing the hydrogen atoms in a difference Fourier synthesis are drawn at intervals of 0.24 e.Å^{-3}. The three "regular" hydrogen atoms behind the boron plane have been omitted. (b) Schematic representation of the $B_3H_8^-$ ion.

The ion as shown in Fig. 1 has a vertical mirror plane demanded by the space group. Another mirror plane in the plane of the boron triangle is satisfied within experimental error. Thus the ion apparently has C_{2v} symmetry. The alternative space group $Pbn2_1$, in which the ion would lack a mirror plane, is ruled out by the failure of least squares refinement to yield significantly different parameters.

We are indebted to Dr. Goji Kodama for providing us with samples of the compound. The work was conducted, in part, under a grant from the National Science Foundation and, in part, under Contract AF33(616)-5874 with the U. S. Air Force, the sponsoring agency being the Aeronautical Research Laboratory of the Wright Air Development Center, Air Research and Development Command.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF MICHIGAN CHARLES R. PETERS
ANN ARBOR, MICHIGAN C. E. NORDMAN

RECEIVED SEPTEMBER 24, 1960

(1) G. Kodama, R. W. Parry and J. C. Carter, THIS JOURNAL, **81**, 3534 (1959).

(2) W. N. Lipscomb, *J. Phys. Chem.*, **62**, 381 (1958).

(3) W. N. Lipscomb in "Advances in Inorganic Chemistry and Radiochemistry," Vol. I, Academic Press, Inc., New York, N. Y., 1959, pp. 132–133.

(4) W. D. Phillips, H. C. Miller and E. L. Muetterties, THIS JOURNAL, **81**, 4496 (1959).

(5) L. Pauling, "The Nature of the Chemical Bond," Cornell University Press, Ithaca, N. Y., 1960, p. 378.

CARBENE BY THE DEHYDROHALOGENATION OF METHYL CHLORIDE

Sir:

Carbene, CH_2:, is formed by photolysis or pyrolysis of ketene[1a,b,c] and diazomethane,[1c,d,e] and presumably as a zinc iodide complex[2] from methylene iodide and zinc–copper couple. Recently it was reported that trimethylaminemethylene[3] is also a source of carbene.

It is now reported that carbene is formed by the dehydrohalogenation (α-elimination) of methyl chloride with strong bases. Thus, methyl chloride reacts with phenyl sodium in the presence of *carbene-acceptors*, such as cyclohexene, isobutylene and *cis*-2-butene to give norcarane, 1,1-dimethylcyclopropane and *cis*-1,2-dimethylcyclopropane, respectively.[4] Apparently carbene generated in this manner undergoes stereospecific *cis* addition.[5] There was no evidence of carbon–hydrogen bond insertion. Methyl chloride (1 mole) was passed into a suspension of phenylsodium (*ca.* 1 mole) in hexadecane (100 ml. and cyclohexene (130 ml.) maintained below 40° (external cooling) to give after carbonation, norcarane (3.2%), benzene (19%), toluene (41%), ethylbenzene (1.9%), *n*-propylbenzene (0.1%), isopropylbenzene (0.08%), benzoic acid (3.0%)

(1) (a) T. G. Pearson, R. H. Purcell and G. S. Saigh, *J. Chem. Soc.*, 409 (1938); (b) M. Burton, T. W. Davis, A. Gordon and H. A. Taylor, THIS JOURNAL, **63**, 1956 (1941); (c) H. M. Frey and G. B. Kistiakowsky, *ibid.*, **79**, 6373 (1957); (d) H. Staudinger and R. Endle, *Chem. Ber.*, **46**, 1437 (1913), and earlier papers; (e) F. O. Rice and A. C. Glasebrook, THIS JOURNAL, **55**, 4329 (1933); **56**, 2381 (1943).

(2) H. E. Simmons and R. D. Smith, *ibid.*, **81**, 4256 (1959).

(3) V. Franzen and G. Wittig, *Angew. Chem.*, **72**, 417 (1960).

(4) Products were characterized and identified by the usual physical properties and/or gas-chromatographic and infrared comparisons with authentic samples.

(5) W. von E. Doering and P. M. LaFlamme, THIS JOURNAL, **78**, 5447 (1956); P. S. Skell and R. C. Woodworth, *ibid.*, **78**, 4496 (1956): W. von E. Doering, R. G. Laughlin, R. G. Buttery and N. Chaudhuri, *ibid.*, **78** 3224 (1956); F. A. L. Anet, R. F. W. Bader and A. Van der Auwera, *ibid.*, **82**, 3217 (1960).