monia.[6] Triphosphonitrilic diamidetetraazide was then prepared by treating the triphosphonitrilic diamidetetrachloride with NaN_3 for one hour at 25–30° in 1:3 water–acetone solution.

$$P_3N_3(NH_2)_2Cl_4 + 4NaN_3 \longrightarrow P_3N_3(NH_2)_2(N_3)_4 + 4NaCl$$

A white solid product separated when the reaction mixture was poured into cold water. However, this product was unstable and has not as yet been identified. Upon evaporation of the acetone in the filtrate, a second solid product was obtained which later was identified as triphosphonitrilic diamidetetraazide. Recrystallization of the latter from an acetone–water mixture gave white needles in 20% yield of triphosphonitrilic diamidetetraazide, m.p. 81–82°.

Identification of the compound is based on infrared absorption data and elemental analysis. Absorption peaks at 2160 cm.$^{-1}$ and 3400 cm.$^{-1}$ suggest the presence of azido and amino groups[7] in the product and strong absorption at 1200 cm.$^{-1}$ indicates retention of the trimeric P_3N_3 ring system.[8] Calcd. for $P_3N_{17}H_4$: P, 27.72; N, 71.06. Found: P, 27.67; N, 67.93; Cl, neg.

We are grateful to Mrs. P. Wheeler for the microanalysis and to Mr. A. S. Tompa for the infrared analysis.

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RESEARCH AND DEVELOPMENT DEPARTMENT
U. S. NAVAL PROPELLANT PLANT M. S. CHANG
INDIAN HEAD, MARYLAND A. J. MATUSZKO

RECEIVED AUGUST 26, 1960

SEPARATION OF AMINO ACID-SPECIFIC "SOLUBLE" RIBONUCLEIC ACIDS BY PARTITION CHROMATOGRAPHY

Sir:

The separation of amino acid-specific "soluble"-fraction ribonucleic acids by countercurrent distribution has been described.[1,2,3,4] This communication describes the separation of these ribonucleic acids by partition chromatography.

The solvent system was prepared by dissolving 278 g. of dipotassium hydrogen phosphate, 435 g. of sodium dihydrogen phosphate monohydrate, and 506 mg. of magnesium chloride hexahydrate in glass-distilled water to give a total volume of 2500 ml., and mixing this solution at room temperature (25°) with 1000 ml. of 2-propanol (Mallinckrodt, reagent) and 250 ml. of formamide (Fisher, reagent). Silicic acid (Mallinckrodt, reagent, 100 mesh, for chromatographic analysis) was washed with 0.001 *M* potassium ethylenediaminetetraacetate, *p*H 7, and with glass-distilled water (fine particles which did not settle rapidly were removed by decantation), and dried overnight at 110°. At room temperature, 300 g. of this silicic acid was mixed thoroughly in a mortar with 200 ml. of the aqueous phase (lower layer) of the solvent system. The resulting free-flowing powder was suspended in the organic phase (upper layer) of the solvent system, and poured into a column 4 cm. by 50 cm. After the silicic acid had settled by gravity, a suspension of 40 mg. of yeast "soluble"-ribonucleic acid[4] in 20 ml. of the organic phase, was placed on the column. A total of 3400 ml. of the organic phase then was allowed to flow through the column (100 ml. per hour). The effluent was collected in fractions totalling 125 ml. each, and these fractions were dialyzed and evaporated.[4] The absorbancies of the fractions at 260 mμ were determined, and the fractions were assayed for amino acid-acceptor activity by standard methods.[4,5]

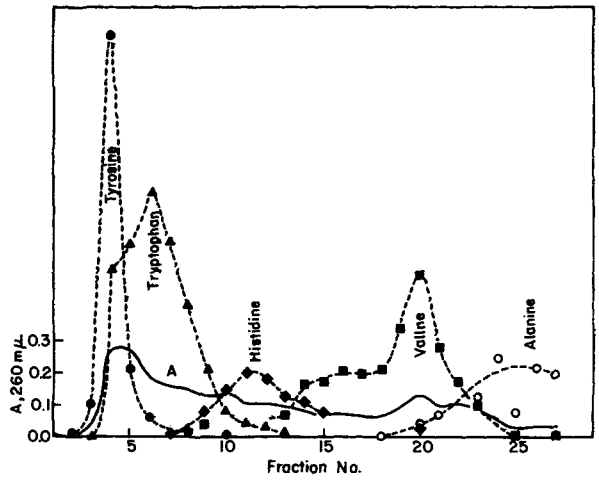

Fig. 1.—Partition chromatography of yeast "soluble"-ribonucleic acids; —, absorbancy at 260 mμ; - - -, amino acid-acceptor activity for the amino acids shown. With the exception of tyrosine, the activities are plotted on a scale such that the activity of the starting material would coincide with its absorbancy, and the specific activities of the fractions relative to the starting material are given by the ratio of activity to absorbancy. The activity for tyrosine is plotted at one-half the scale of the others. The increase in the specific activities of the peak fractions, relative to the starting material, was: tyrosine, nine; tryptophan, four; histidine, two; valine, four; alanine, five. (The starting material gave approximately these counts per minute per mg. under the assay conditions used: tyrosine, 1000; tryptophan, 1000; histidine, 1500; valine, 2000; alanine, 1500.)

The results (Fig. 1) show conclusively that amino acid-specific ribonucleic acids can be separated by partition chromatography. This is the first demonstration that partition chromatography can be used for the fractionation of nucleic acids. The specific activities of the different ribonucleic acids were considerably increased relative to the starting material.

In comparison with countercurrent distribution, the chief disadvantage of this partition chromatographic procedure is the high loss of material (approximately 60%), presumably by adsorption on the silicic acid, but this disadvantage is minimized by the ready availability of the yeast

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"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

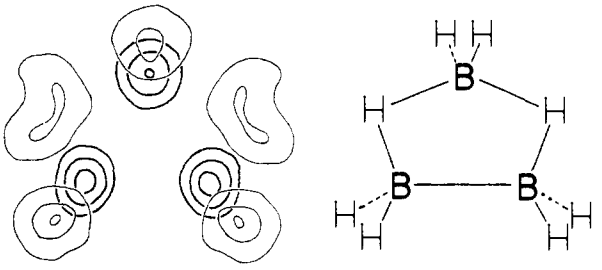
RECEIVED SEPTEMBER 19, 1960

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



a b

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.

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 $R = \Sigma||F_o|-|F_c||/\Sigma|F_o| = 0.084$ and 0.077 for observed reflections at $-100°$ and room temperature, respectively.

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

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%)

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