more substituent atoms attached to the central carbon are highly electronegative ones.

The experimental procedures used in this work were similar to those previously described.[3]

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RECEIVED AUGUST 15, 1962

SYNTHESIS OF A BORON-LABELED TETRABORANE[1]

Sir:

We wish to report the first synthesis of a boron hydride molecule containing a boron isotopic label *in a specific position.* Skeletal labeled boron hydrides promise to be of considerably greater use in the study of mechanisms of reactions than the deuterium labeled materials that have thus far been produced, since the probability of spontaneous rearrangement without skeletal disruption is much lower.

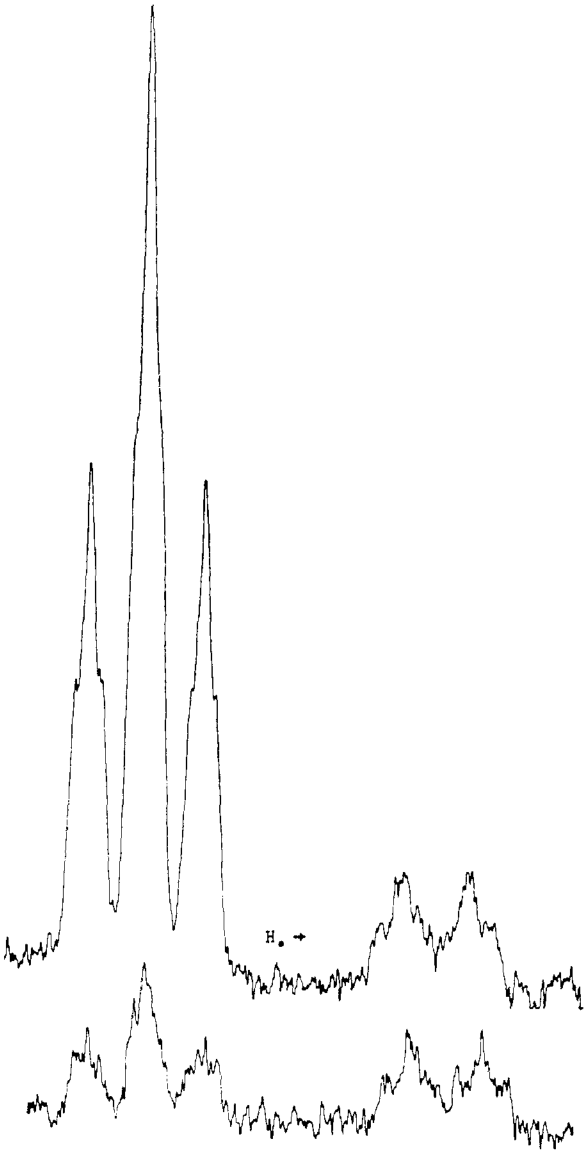

Fig. 1.—^{11}B n.m.r. spectra of tetraboranes: upper trace, ^{11}B enrichment in the 4-position; lower trace, 4% ^{11}B in all positions.

Diborane-^{10}B was prepared by reaction of lithium aluminum hydride with boron trifluoride containing 96% ^{10}B purchased from Oak Ridge National Laboratory. Sodium triborohydride was prepared from the ^{10}B-enriched diborane by reaction with sodium amalgam in diethyl ether.[2] Following isolation and purification by the usual procedure the labeled triborohydride was allowed to react with a mixture of isotopically normal diborane and hydrogen chloride. In a typical reaction 5.39 mmoles of triborohydride was treated for 20 min. at 0° with a mixture of 5.58 mmoles of hydrogen chloride and 5.81 mmoles of diborane. Fractionation of the resulting gaseous mixture by standard techniques resulted in isolation of 2.05 mmoles of tetraborane (−45.2° V.T., 37.5 mm.; lit., interpolated, 37.5 mm.) as well as 5.76 mmoles of diborane and small quantities of hydrogen chloride and higher boranes, including decaborane, $B_{10}H_{14}$. The ^{11}B n.m.r. spectrum of the tetraborane was obtained with a Varian Model 4300B high resolution spectrometer operating at 19.3 Mc./s. and is compared in Fig. 1 with the spectrum of tetraborane-per-96% ^{10}B prepared by the same procedure from diborane-96% ^{10}B and sodium triborohydride-96% ^{10}B. The high field doublet arising from the two B–H groups in the 1,3 positions[3] is of closely comparable intensity in the two spectra (taken under identical instrumental conditions) but the triplet arising from the 2,4 BH_2 positions is considerably more intense in the sample prepared from isotopically normal diborane. Clearly, the ^{10}B is contained in the 4 position almost exclusively except for the four per cent unavoidably introduced into all other positions from the sodium triborohydride.

It has been suggested previously[4] that the pyrolytic conversion of diborane to tetraborane proceeds by formation of triborane-9, conversion of this intermediate to triborane-7 in the rate limiting step, and reaction of triborane-7 with diborane to form tetraborane. We suggest that the present reaction proceeds by the pathway

$$\mathrm{Na^{10}B_3H_8 + HCl \longrightarrow NaCl + {}^{10}B_3H_9} \qquad (1)$$

$$\mathrm{3/2\,{}^{10}B_2H_6 \longleftarrow {}^{10}B_3H_9 \longrightarrow {}^{10}B_3H_7 + H_2} \qquad (2)$$

$$\mathrm{{}^{10}B_3H_7 + {}^{11}B_2H_6 \longrightarrow {}^{10}B_3{}^{11}BH_{10} + {}^{11}BH_3} \qquad (3)$$

Mass spectrometric analysis of the selectively labeled sample showed that it contained about 16% ^{11}B. Assuming that the 1, 2 and 3 positions contain only 4% ^{11}B coming from the triborohydride, the 4 position is estimated to contain 52% ^{11}B. Less than complete ^{11}B labeling in the 4 position arises from two principal causes.

(a) Since isotopically normal diborane used as the source of ^{11}B contains 20% ^{10}B, the lighter iso-

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(1) Interconversion of Boranes. V. For paper IV of this series see G. L. Brennan and R. Schaeffer, *J. Inorg. Nucl. Chem.*, **20**, 205 (1961).

(2) W. V. Hough, L. J. Edwards and A. D. McElroy, *J. Am. Chem. Soc.*, **80**, 1828 (1958).

(3) For the numbering system recommended for boron compounds see the Preliminary Report of the Advisory Committee on the Nomenclature of Organic Boron Compounds, available through *Chemical Abstracts*.

(4) R. E. Enrione and R. Schaeffer, *J. Inorg. Nucl. Chem.*, **18**, 103 (1961).

tope must be introduced to at least this extent in the 4 position even if the mechanism of introduction is highly stereospecific. In addition, any kinetic isotope effect shown in reaction (3) will be in favor of further ^{10}B introduction.

(b) As the reaction of hydrogen chloride with sodium triborohydride proceeds, a fraction of the latter is converted to diborane which in this case contains 96% ^{10}B. The ^{11}B isotope content of the original diborane is thus lowered somewhat, depending on the degree of branching of reaction (2).

A more complete report of this work together with an alternate but less selective synthesis and the implications of the work with respect to the mechanisms of interconversion of boranes will be reported in the near future. Work is also in progress on the mechanisms of tetraborane reactions using the labeled material.

Acknowledgments.—Support of this work by National Science Foundation Grant G-6224 is gratefully acknowledged. The initial study of the reaction of sodium triborohydride with hydrogen chloride and diborane was carried out by R. O. Buttlar and will be reported elsewhere.

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RECEIVED AUGUST 7, 1962

THE SYNTHESIS OF HEXAPHENYLPENTALENE

Sir:

In the forty years since Armit and Robinson[1] first suggested that pentalene might be an aromatic hydrocarbon, there has accumulated in the chemical literature a wealth of information concerned with unsuccessful attempts to synthesize[2] and describe theoretically[3] this pseudoaromatic[4] and its derivatives.[5] Reported here is the synthesis of hexaphenylpentalene I, the first derivative of pentalene free of fused rings.[5]

The fluoride-catalyzed (potassium fluoride in dimethyl sulfoxide) condensation[6] of 1,2,3-triphenylcyclopentadiene[8] with 1,2,3-triphenylpropenone[9] *via* a Michael–Knoevenagel dehydration reaction sequence[10] afforded in 53% yield the dihydrohexaphenylpentalene, II [bright yellow needles (methanol–chloroform) m.p. 266–267°. Calcd. for $C_{44}H_{32}$: C, 94.25; H, 5.75; mol. wt., 560.7. Found: C, 94.26, 94.11; H, 5.85, 6.03; mol. wt., 556; ultraviolet (acetonitrile) λ_{max} mμ (log ϵ): 242 (4.39), 296 (4.24), 355 (4.14); infrared (KBr): 3.27, 3.30, 6.24, 6.72, 6.90, 9.30, 9.72, 13.20, 13.65, 14.40 μ].[11]

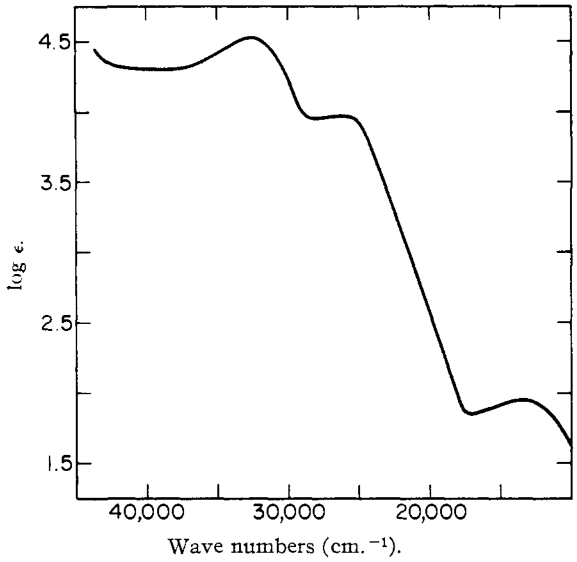

Fig. 1.—Ultraviolet, visible, and near-infrared spectrum of hexaphenylpentalene.

II undergoes rapid dehydrogenation by N-bromosuccinimide in refluxing deoxygenated carbon tetrachloride to give in 77% yield hexaphenylpentalene, I [green-brown needles (methanol–chloroform), m.p. 273–276°. Calcd. for $C_{44}H_{30}$: C, 94.59; H, 5.41; mol. wt., 558.7. Found: C, 94.53, 94.51; H, 5.51, 5.65; mol. wt., 527. Ultraviolet–visible spectrum (dioxane) λ_{max} mμ (cm.$^{-1}$, log ϵ): 310 (32,300 cm.$^{-1}$, 4.52), 380 (26,400 cm.$^{-1}$, 3.99), 720 (13,900 cm.$^{-1}$, 1.95) see Fig. 1. Near-infrared (carbon tetrachloride): 800 mμ (12,500 cm.$^{-1}$) to 1.5 μ (6700 cm.$^{-1}$) broad tailing of 720 mμ (13,900 cm.$^{-1}$) peak. Infrared (KBr): 3.27, 6.25, 6.26, 6.27, 6.66, 6.73, 6.91, 9.31, 9.79, 12.81, 12.97, 13.47, 13.88, 14.37].[11]

Hexaphenylpentalene also was prepared in low yields by iodine oxidation of the deep red solution (λ_{max} 489 mμ) resulting from the reaction of II with butyllithium in tetrahydrofuran.

While solutions of I are air sensitive, the crystalline solid appears to be quite stable. Both I and II give deep green solutions with bromine (CCl_4) and

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(1) J. W. Armit and R. V. Robinson, *J. Chem. Soc.*, **127**, 828 (1922).

(2) For original references see the reviews of: (a) E. D. Bergmann in "Non-benzenoid Aormatic Compounds" (D. Ginsburg editor), Interscience Publishers, New York, N. Y., 1959; (b) M. E. Vol'pin, *Uspekhi Khim.*, **29**, 298 (1960); (c) H. Paul, *Chem. Techn.*, **8**, 189 (1956).

(3) See the reviews in reference 2. Also: (a) A. Streitwieser, "Molecular Orbital Theory for Organic Chemists," John Wiley and Sons, New York, N. Y., 1961; (b) H. J. Dauben, Jr., and D. J. Bertelli, *J. Am. Chem. Soc.*, **83**, 4659 (1962).

(4) For pertinent discussions of pseudoaromaticity see: (a) D. P. Craig, *J. Chem. Soc.*, 3175 (1951); also, see chapter by D. P. Craig in reference 2a; (b) W. von E. Doering and D. W. Wiley, *Tetrahedron*, **11**, 183 (1960).

(5) Several fused ring derivatives of pentalene have been reported: (a) 5,10-disubstituted dibenzo[a,e]pentalene, K. Brand, *Ber.*, **45**, 3071 (1912); (b) dibenzo[a,e]pentalene, C. T. Blood and R. P. Linstead, *J. Chem. Soc.*, 2263 (1952); C. C. Chuen and S. W. Fenton, *J. Org. Chem.*, **23**, 1538 (1958); (c) 3,5-dimethylcyclohepta[c,d]pentalene, K. Hafner and J. Schneider, *Ann.*, **624**, 37 (1952); (d) 1,2,3-triphenylbenzopentalene, E. Le Goff, *J. Am. Chem. Soc.*, **84**, 1505 (1962).

(6) The ability of F^- to form the strongest known hydrogen bond[7] in HF_2^- presumably accounts for its functioning as a Lewis base: $>HC{-}H + 2F^- \longrightarrow >HC^- + FHF^-$.

(7) L. Pauling, "The Nature of the Chemical Bond," Cornell University Press, Ithaca, N. Y., 1960, p. 460.

(8) P. L. Pauson and B. J. Williams, *J. Chem. Soc.*, 4153 (1961).

(9) A. Dornow and F. Boherg, *Ann.*, **578**, 101 (1952).

(10) Potassium fluoride in ethanol, ether and benzene previously has been reported to catalyze Knoevenagel reactions: A. Sakurai, *Sci. Paper Inst. Phys. Chem. Research (Tokyo)*, **53**, 250 (1959); M. Igarashi, H. Midorikawa and S. Aoyama, *J. Sci. Research Inst. (Tokyo)*, **52**, 151 (1958); H. Baba, H. Midorikawa and S. Aoyama, *ibid.*, **52**, 99–117 (1958).

(11) Melting points are corrected; molecular weight was determined with vapor pressure osmometer; the infrared spectrum, for which the more prominent absorption peaks are recorded, was consistent with the proposed structure.