The weight of the cesium salt was 0.6 g. (0.00136 mole). The conversion was 46% and the yield was 60%.

A solution of 0.0033 mole of $B_{10}H_{13}P(C_6H_5)_2$ in 25 ml. of water, 120 ml. of dioxane, and 5 ml. of concentrated hydrochloric acid was allowed to stand for 7 hr. The total hydrogen evolved was 0.041 l. Then 50 ml. of water was added and the solution was evaporated to 1/4 the original volume. The resulting slurry was filtered to give 0.0005 mole of the insoluble $B_9H_{14}P(C_6H_5)_2$. The filtrate on addition of $(CH_3)_4NCl$ yielded 0.0025 mole of $(CH_3)_4NB_{10}H_{12}P(C_6H_5)_2$.

Reaction of $B_{10}H_{13}P(C_6H_5)_2$ with Amines.—Addition of 10 ml. of triethylamine to a solution of 0.01 mole of $B_{10}H_{13}P(C_6H_5)_2$ in dichloromethane resulted in heat evolution and slow separation of a solid. No hydrogen evolved. The solid was repeatedly recrystallized from hot toluene–dichloromethane, m.p. 216–217°.

Anal. Calcd. for $(C_2H_5)_3NHB_{10}H_{12}P(C_6H_5)_2$: B, 26.6; P, 7.63. Found: B, 26.3; P, 7.89.

The infrared spectrum of this compound was essentially identical to that of crystalline solid obtained by adding $(C_2H_5)_3NHCl$ to a solution containing $B_{10}H_{12}P(C_6H_5)_2^-$.

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Chemistry of Boranes. V.[1] First Example of a B_{11} Hydride, the $B_{11}H_{14}^-$ Anion

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Ethereal decaborane reacts with metal borohydrides at 90° to give $M^+B_{11}H_{14}^-$. Spectral data suggest that the anion $B_{11}H_{14}^-$ has high symmetry and appear consistent with the $B_{11}H_{14}^-$ structure hypothesized by Moore, Lohr, and Lipscomb. In strong base, $B_{11}H_{14}^-$ ionizes and salts of $B_{11}H_{13}^{-2}$ can be isolated from these solutions.

We wish to report the synthesis of a new boron hydride derivative, the first example of a B_{11} species, which has been isolated in the form of a salt $M^+B_{11}H_{14}^-$ from the essentially quantitative reaction of decaborane and sodium or lithium borohydride at elevated temperature

$$\mathrm{MBH_4} + \mathrm{B_{10}H_{14}} \xrightarrow[\mathrm{R_2O}]{90^\circ} \mathrm{MB_{11}H_{14}} + 2\mathrm{H_2} \qquad (1)$$

We have confirmed the findings of Hough and Edwards,[2] who reported that $NaB_{10}H_{13}$, diborane, and hydrogen are the sole products of reaction 1 when effected at 25°.

$$\mathrm{MBH_4} + \mathrm{B_{10}H_{14}} \xrightarrow[\mathrm{R_2O}]{25^\circ} \mathrm{MB_{10}H_{13}} + \begin{array}[t]{c}\mathrm{H_3B{\cdot}OR_2}\\ \uparrow\downarrow\\ \mathrm{B_2H_6} + \mathrm{R_2O}\end{array} + \mathrm{H_2} \qquad (2)$$

The obvious implication to be drawn from these data is that reaction 1 involves a sequence of steps including reactions 2 and 3, and, in fact, we

$$\mathrm{R_2O{\cdot}BH_3} + \mathrm{MB_{10}H_{13}} \xrightarrow{90^\circ} \mathrm{MB_{11}H_{14}} + \mathrm{H_2} + \mathrm{R_2O} \qquad (3)$$

found that ethereal B_2H_6 and $NaB_{10}H_{13}$ do form $NaB_{11}H_{14}$ at 45°. The $B_{11}H_{14}^-$ salts were isolated from the reaction mixtures as etherates, $NaB_{11}H_{14}{\cdot}2.5$dioxane and $LiB_{11}H_{14}{\cdot}2$dioxane. These etherates dissolve readily in water and from metathesis reactions in such aqueous solutions, crystalline, non-solvated cesium, tetramethylammonium, trimethylsulfonium, and triethylammonium salts of $B_{11}H_{14}^-$ have been isolated.

The infrared spectrum of $B_{11}H_{14}^-$ is quite simple and suggests a high symmetry for this anion. The B–H stretch at 2530 cm.$^{-1}$ is structureless, and there is no bridge B–H absorption. No contemporary structure appears consistent with these data, but such a possibility cannot be excluded from consideration.

The proton magnetic resonance spectrum of aqueous solutions of $NaB_{11}H_{14}$ consists of a water resonance and a broad multiplet which has a high field shoulder. Effective spin decoupling of the boron and hydrogen nuclei by superposition of a saturating boron rf. field yields a proton spectrum consisting of the water resonance, two very closely spaced peaks of similar intensity (total relative

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[1] Paper IV, E. L. Muetterties and V. D. Aftandilian, *Inorg. Chem.*, **1**, 731 (1962).

[2] W. V. Hough and L. J. Edwards, 133rd National ACS Meeting, Division of Inorganic Chemistry, Paper No. 69, San Francisco, Calif., 1958.

intensity of 11), and a high field peak of relative intensity 3. Since the line width of the high field peak does not undergo any significant change as the frequency of the saturating B^{11} field is varied, the protons giving rise to this peak are presumed to be at most very weakly spin coupled with any boron nuclei. The lower field pair must have relative intensities six and five but they are so closely spaced that the individual curves cannot be accurately integrated. Integration of these two curves gives a total intensity of eleven relative to three for the high field curve. From the irradiation frequencies required for maximum sharpness of each of the low field H^1 peaks, it is estimated that the two different types of boron nuclei have a chemical shift of only about 0.5 p.p.m. This is too small a shift to detect, as is the case in the B^{11}-spectrum because of the inherently large line widths for boron resonances. With superposition of a saturating H^1 field, the B^{11} spectrum of $B_{11}H_{14}^-$ is a single resonance with a line width of about 5/3 the B^{11} line width of $B_{12}H_{12}^{-2}$. In the absence of the saturating H^1 field, B^{11} resonance consists of a symmetrical doublet; the estimated average BH coupling constant is 130 c.p.s., similar to A_{BH} in $B_{12}H_{12}^{-2}$ (129 c.p.s.).

The n.m.r. data establish: (1) two boron environments containing similar numbers of boron atoms, *i.e.*, six and five; (2) each boron atom is strongly coupled with one hydrogen atom; (3) two proton environments containing similar numbers of hydrogen atoms which must represent the terminal B–H protons for the two types of boron atoms; and (4) three unique protons that do not appear to be strongly bonded to any boron atoms.

These data and the infrared data are wholly consistent with but do not uniquely determine a pseudo-B_{12} icosahedral model which has been proposed by Moore, Lohr, and Lipscomb.[3] These authors constructed $B_{11}H_{14}^-$ from $B_{12}H_{12}^{-2}$ by replacement of a BH group with an H_3^+ group which comprises a triangle perpendicular to the molecular axis (Fig. 1). The unique B^c nucleus is not truly environmentally equivalent to the B^a set; however, all these, the five B^a atoms and one B^c atom, have equivalent short range environments in that they each are bonded to five boron atoms. These then comprise the one set of spectroscopically equivalent boron atoms, and the five B^b boron atoms, each of which has only four nearest boron atoms, comprise the other set. The three proton environments are then the H^a and H^c set (six), the H^b set (five), and the unique H_3 set. It is true that in a static model the threefold H_3 axis makes the B^b set of boron atoms non-equivalent; however, it is quite reasonable[4] that there is a rapid tunneling through the barrier to free rotation of the H_3 group about the molecular axis to give spectroscopic equivalence of the B^b set of boron atoms.

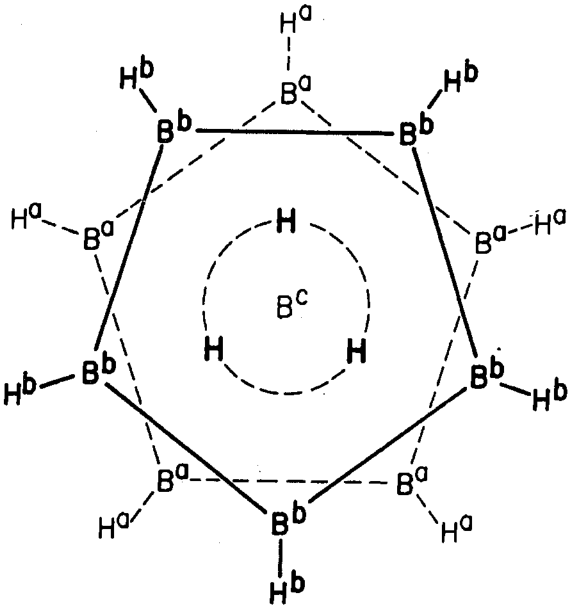

Fig. 1.—Possible structure for $B_{11}H_{14}^-$ is presented looking down symmetry axis through B^c and H^c; the H^c hydrogen atom is not depicted. The H_3 group may be effectively freely rotating about the molecular axis.

In solutions of $B_{11}H_{14}^-$ in D_2O, there is exchange of B–H for B–D as evidenced by infrared data. There is also a very rapid exchange of deuterium with protons in the unique H_3 set; fresh solutions of $NaB_{11}H_{14}$ in D_2O show no evidence of a proton n.m.r. resonance in the H_3 region.

Moore, Lohr, and Lipscomb[3] mention the possible existence of $B_{11}H_{13}^{-2}$ and indicate that it would possibly be less stable than $B_{11}H_{14}^-$. We have found that from highly basic solutions, sparingly water soluble $B_{11}H_{13}^{-2}$ salts of $Zn(NH_3)_4^{+2}$, $(CH_3)_3S^+$, and $(CH_3)_4N^+$ are readily isolated. The infrared spectrum of $B_{11}H_{13}^{-2}$ is distinguishable from that of $B_{11}H_{14}^-$ in the long wave length region. The ready reversibility of

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(3) E. B. Moore, Jr., L. L. Lohr, Jr., and W. N. Lipscomb *J. Chem. Phys.*, **35**, 1329 (1961).

(4) In $Al(BH_4)_3$, quantum mechanical tunneling of the barrier to rotation of BH_4 groups has been proposed to account for the n.m.r. spectral data: R. A. Ogg and J. D. Ray, *Discussions Faraday Soc.*, **19**, 239 (1955). Also, an intramolecular tunneling process has been suggested to account for boron atom and hydrogen atom equivalences in $B_3H_8^-$: W. N. Lipscomb, *Advan. Inorg. Chem. Radiochem.*, **1**, 132 (1959).

$$B_{11}H_{14}^{-} \rightleftharpoons B_{11}H_{13}^{-2} + H^{+}$$

is evident by examination of the infrared spectrum of the $(CH_3)_4N^+$ salt upon recrystallization alternately from water and aqueous $(CH_3)_4NOH$. The lack of suitable solvents for the $B_{11}H_{13}^{-2}$ salts precluded examination of the B^{11} resonance spectrum of pure derivatives of the dianion.

Experimental

Reagents.—Decaborane purchased from Olin Mathieson was recrystallized from heptane. Lithium and sodium borohydrides, from Metal Hydrides, were used without purification. All ethers employed in this work were dried with $LiAlH_4$ and distilled prior to use.

I. Synthesis of $LiB_{11}H_{14}\cdot 2.0$Dioxane.—A 100-ml. stainless steel pressure vessel was charged with decaborane (1.78 g., 0.0145 mole) and lithium borohydride (0.32 g., 0.0145 mole). The reactor was cooled to −80°, evacuated, and 30 ml. of freshly distilled 1,2-dimethoxyethane sucked in through the valve. The reactor then was heated with shaking to 90° for 10 hr. After cooling to room temperature, the vessel was opened to the vacuum train. Gaseous products were bled through a liquid N_2 trap and the non-condensable gas was transferred to a known volume with a Töpler pump. There was 0.027 mole (93% of that required for $2H_2/B_{10}H_{14}$) of non-condensable gas which, by mass spectral analysis, was shown to be 98.2% hydrogen, 0.77% nitrogen and/or carbon monoxide, and about 0.5% methane. The liqud nitrogen trap contained a small amount of liquid. Its vapor pressure of 71 mm. at 25°, as compared with 75 mm. at 25° for 1,2-dimethoxyethane, was taken as evidence that no more than traces of diborane were present. The reactor contained a yellow liquid. It was clarified by filtration and diluted with about 100 ml. of dioxane to precipitate a yellow solid which was collected, washed with dioxane, and dried to give 3.41 g. of $LiB_{11}H_{14}\cdot$2dioxane (73% based on reactants charged).

Anal. Calcd. for $LiB_{11}H_{14}\cdot 2C_4H_8O_2$: Li, 2.19; B, 37.6; C, 30.4; H, 9.86. Found: Li, 2.0; B, 38.3; C, 30.1; H, 9.36.

II. Synthesis of $NaB_{11}H_{14}\cdot 2.5$Dioxane. A. From $NaBH_4$ and $B_{10}H_{14}$.—The sodium salt can be obtained as described above for lithium or, more conveniently, by carrying out the reaction at atmospheric pressure in dioxane. A mixture of dioxane (100 ml.), decaborane (3.8 g., 0.031 mole), and $NaBH_4$ (0.95 g., 0.025 mole) was refluxed with stirring in a N_2 atmosphere for 16 hr. until hydrogen evolution had completely stopped. A yellow solid separated from the hot solution. The mixture was chilled, the yellow solid collected, washed with dioxane, and vacuum dried to leave 7.36 g. (78%) of $NaB_{11}H_{14}\cdot 2.5\ C_4H_8O_2$.

Anal. Calcd. for $NaB_{11}H_{14}\cdot 2.5C_4H_8O_2$: Na, 6.11; B, 31.6; C, 31.9; H, 9.11. Found: Na, 6.6; B, 31.1; C, 31.4; H, 9.10.

The Li^+ and Na^+ salts described above are brilliant yellow. They can be recrystallized from mixtures of dioxane with tetrahydrofuran or 1,2-dimethoxyethane to give yellow platelets. The yellow color is, however, attributable to a trace impurity. If the yellow sodium salt is boiled with 100 ml. of dioxane and filtered hot, addition of petroleum ether precipitates a nearly white solid having infrared and B^{11} resonance spectra identical to those of the yellow material. The sparingly water soluble salts described below are yellow initially, but, if on recrystallization from boiling water the solution is boiled sufficiently, pure white derivatives are obtained. Decomposition of the yellow impurity is accelerated by traces of strong acids. The white salts are identical to the yellow salts by infrared analysis, B^{11} resonance, and X-ray powder pattern. These salts are of limited stability. On storage at room temperature, the sodium salt, initially completely soluble in tetrahydrofuran or 1,2-dimethoxyethane, becomes increasingly insoluble in these solvents over several weeks, and decreasing amounts of the original material can be recovered as the dioxanate. Aqueous solutions reduce silver nitrate solution and slowly evolve hydrogen at room temperature. Hydrogen evolution is brisk upon acidification.

B. From $NaB_{10}H_{13}$ and B_2H_6.—A solution of 9.2 mmoles of $NaB_{10}H_{13}$[5] in 12 ml. of bis-(2-methoxyethyl) ether in a vacuum train reaction flask was stirred with 14.5 mmoles of diborane at 45° for 2.5 hr. and at room temperature overnight. Much non-condensable gas was evolved. Careful fractionation of the condensable gases gave 6.5 mmoles of unchanged diborane. The solvent was evaporated under reduced pressure and the solid yellow residue was dissolved in water. Addition of tetramethylammonium chloride solution gave an off-white precipitate. Recrystallization from hot water gave pure white $(CH_3)_4NB_{11}H_{14}$ with an infrared spectrum identical to that of material obtained by metathesis of $(CH_3)_4NCl$ with $NaB_{11}H_{14}$ prepared as described above.

III. Sparingly Water Soluble $B_{11}H_{14}^-$ and $B_{11}H_{13}^{-2}$ Salts.—The various salts of $B_{11}H_{14}^-$ were prepared from an aqueous solution of $NaB_{11}H_{14}$ which first had been freed of dioxane by dissolving the sodium salt dioxanate in water and evaporating to dryness at room temperature in a rotating evaporator. The crude $B_{11}H_{14}^-$ salts were precipitated upon adding to aqueous $NaB_{11}H_{14}$ an aqueous solution of a suitable salt of the cation, *e.g.*, RbF, CsCl, $(CH_3)_4NCl$, $(CH_3)_3SI$, $(C_2H_5)_3NHCl$, or $(CH_3)_3NHCl$. The resulting salts all were recrystallizable from water. In addition, $CH_2Cl_2/CHCl_3$ and $CHCl_3/CH_3OH$ are suitable recrystallization solvents for the $(C_2H_5)_3NH^+$ and $(CH_3)_3NH^+$ salts, respectively. The $B_{11}H_{13}^{-2}$ salts separated from solutions which had been made strongly basic with aqueous NaOH (or aqueous ammonia, in the case of the $Zn(NH_3)_4^{+2}$ salt).

$(C_2H_5)_3NH^+B_{11}H_{14}^-$.—*Anal.* Calcd.: C, 30.7; H, 12.9; N, 5.96; B, 50.6; mol. wt., 234. Found: C, 31.4; H, 13.5; N, 5.97; B, 49.7; mol. wt., 231 (ebullioscopic determinations in CH_2ClCH_2Cl at varying concentrations extrapolated to infinite dilution).

$Cs^+B_{11}H_{14}^-$.—*Anal.* Calcd.: Cs, 50.0; B, 44.7; H, 5.31; mol. wt., 266. Found: Cs, 50.4; B, 44.4; H, 5.37; mol. wt., 265. (X-Ray molecular weight. Single crystal analysis showed an orthorhombic cell with the constants: $a = 7.35$ Å., $b = 11.28$ Å., and $c = 12.85$ Å.; density by floatation = 1.66; thus mol. wt. = 265 if $z = 4$.)

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(5) M. F. Hawthorne, A. R. Pitochelli, R. D. Strahm, and J. J. Miller, *J. Am. Chem. Soc.*, **82**, 1825 (1960).

$(CH_3)_3PC_2H_4P(CH_3)_3{}^{+2}B_{11}H_{13}{}^{-2}\cdot H_2O$.—*Anal.* Calcd.: C, 29.1; H, 11.3; P, 18.8; B, 36.0. Found: C, 28.2; H, 11.1; P, 19.3; B, 36.0.

$Zn(NH_3)_4{}^{+2}B_{11}H_{13}{}^{-2}$.—*Anal.* Calcd.: Zn, 24.6; N, 21.2; H, 9.51; B, 44.9; mol. wt., 266. Found: Zn, 24.3; N, 19.9, 20.1; H, 9.59; B, 44.1; mol. wt., 268. (X-Ray molecular weight. Single crystal analysis showed an orthorhombic cell with a = 10.75 Å., b = 14.15 Å., and c = 8.85 Å.; density by floatation = 1.32; thus mol. wt. = 268 if z = 4).

$[(CH_3)_3S^+]_2B_{11}H_{13}{}^{-2}$.—*Anal.* Calcd.: C, 25.2; H, 10.9; S, 22.4; B, 41.5. Found: C, 23.0; H, 10.3; S, 22.4; B, 40.0.

$Rb^+{}_2B_{11}H_{13}{}^{-2}$.—*Anal.* Calcd.: mol. wt., 303. Found: mol. wt., 309. (X-Ray molecular weight. Single crystal analysis showed a body centered tetragonal cell of a = 7.70 Å. and c = 10.84 Å.; density by floatation = 1.86; thus mol. wt. = 309 if z = 2).

IV. Spectral Data. A. Infrared.—The infrared spectrum shows only three absorption peaks: 2530 cm.$^{-1}$ (very sharp), 1042 cm.$^{-1}$ (sharp), and ~720 cm.$^{-1}$ (broad) of relative intensities on the order of 15:3:1.

The B–H stretch at 2530 cm.$^{-1}$ occurs as a single sharp band ($NaB_{11}H_{14}$ in water, crystalline $(CH_3)_3SB_{11}H_{14}$, or $(C_2H_5)_3NHB_{11}H_{14}$ as Nujol mulls). Although solutions show a structureless B–H absorption, certain salts, as crystalline solids in a Nujol mull, show a more complicated B–H stretch spectrum presumably due to coupling of the BH stretching vibration with lattice vibrations. For example, $CsB_{11}H_{14}$ shows three bands of nearly equal intensity at 2538, 2500, and 2487 cm.$^{-1}$. $[(CH_3)_3S]_2B_{11}H_{13}$ has two strong bands at 2487 and 2457 cm.$^{-1}$ with a suggestion of weaker shoulders at 2439, 2403, and 2375 cm.$^{-1}$. The $Zn(NH_3)_4B_{11}H_{13}$ spectrum shows three strong bands at 2475, 2443 (a doublet?), and 2427 cm.$^{-1}$ with shoulders at 2525, 2500, and 2392 cm.$^{-1}$.

The $B_{11}H_{14}{}^-$ cage absorption at 1042 cm.$^{-1}$ has very weak shoulders at 1075 and 1010 cm.$^{-1}$. The $B_{11}H_{13}{}^{-2}$ ion is distinguishable from $B_{11}H_{14}{}^-$ by a sharp band of medium intensity occuring at 1117 cm.$^{-1}$ in the $B_{11}H_{13}{}^{-2}$ spectrum. The dinegative anion also shows absorption at 1053 cm.$^{-1}$ (strong) with a 1020 cm.$^{-1}$ shoulder, and at 720 cm.$^{-1}$ (medium).

Recrystallization of $CsB_{11}H_{14}$ from D_2O gave a product having an infrared spectrum like that of the starting material except for the appearance of a new band at 1887 cm.$^{-1}$ (B–D stretch) to show that some exchange of D for H had occurred.

B. Ultraviolet and Visible.—Pure $(C_2H_5)_3NHB_{11}H_{14}$ in CH_2Cl_2 solutions cuts off in the ultraviolet region at about 2500 Å. and shows a shoulder at about 2750 Å. having a molar extinction coefficient on the order of 1000. The yellow impure $(C_2H_5)_3NHB_{11}H_{14}$, of identical X-ray pattern, infrared spectrum, and B^{11} resonance spectrum, had an identical spectrum in the 2500 to 2750 Å. region and in addition showed weaker absorption as a shoulder at 3150 Å. and a peak at 4500 Å. This absorption at 3150 and 4500 Å. is the only measurable property in which the yellow impure salt differs from the purified white salt.

Pure $CsB_{11}H_{14}$ in water is transparent, showing only a weak shoulder at 3150 Å.

C. N.m.r. Data.—Spectra were obtained on a Varian spectrometer (Model V4300 and associated magnet). Boron spectra were examined at 10, 14.2, and 19.2 Mc., and proton spectra were examined at 60 Mc. An NMR Specialties spin decoupler (Model SD60) was employed for observation of the B^{11} resonance at 19.2 Mc. and of the proton resonance at 60 Mc. with saturation of the hydrogen and boron nuclei, respectively. Four solutions were examined: (a) a concentrated solution of $NaB_{11}H_{14}$ free of dioxane prepared by two room temperature evaporations of a 3.0-g. sample of $NaB_{11}H_{14}$ dioxanate in successive 50-ml. portions of water; (b) a saturated CH_2Cl_2 solution of $(C_2H_5)_3NHB_{11}H_{14}$; (c) a D_3CCN solution of $(CH_3)_3NHB_{11}H_{14}$; and (d) a concentrated solution of the sodium salt in D_2O prepared as in (a) using D_2O.

The $B_{11}H_{14}{}^-$ proton spectra were identical for solutions (a), (b), and (c). They consisted of a broad signal covering a span of about 420 c.p.s. The area showed four diffuse maxima about equally spaced at about 140 c.p.s. On the high field side of this diffuse quartet was a broad shoulder. In each solution, different areas of this B–H signal were obscured by the resonance of the solvent protons. Upon effective decoupling of the boron and hydrogen nuclei by irradiation at 19.2 Mc., the diffuse proton quartet collapsed into two sharp singlets of about equal intensity located at +2.37 and +2.92 p.p.m. from the water resonance. The relative heights and sharpness of these peaks changed with only very slight variation in the frequency of the saturating B^{11} field. Less sensitive to the frequency of irradiation was a weaker, broader high field peak at +7.62 p.p.m. from water coinciding with the high field shoulder on the original quartet. The closely spaced low field peaks could not be integrated separately but the ratio of the sum of their areas to the area of the weaker high field peak was 10.6/3 for solution (a) and 11/3 for solution (b) after correction in the latter case for the ethyl CH_3 resonance which coincided with the +2.92 p.p.m. resonance. Solution (d), the sodium salt in D_2O, showed the same quartet as the other solutions but the high field shoulder was absent. Irradiation collapsed the quartet into two closely-spaced, sharp peaks but there was no indication of the weaker, high field resonance at +7.62 p.p.m.

The B^{11} spectrum of all solutions of $B_{11}H_{14}{}^-$ consisted of a symmetrical doublet. Within experimental error, the doublet separation of about 140 c.p.s. was independent of frequency (10 to 19.2 Mc.). This separation is the maximum value for the B–H coupling constants in $B_{11}H_{14}{}^-$. Since the other data indicate that there are two major chemically shifted boron resonances of approximately 10 c.p.s. separation (at 19.2 Mc.), a reasonable average value for A_{BH} is ~130 c.p.s. On irradiation at 60 Mc., the 19.2 Mc. B_{11} resonance consisted of a single peak. The half-height width of the singlet at the optimum H^1 saturating frequency was 50 c.p.s. The width of the boron resonance of the $B_{12}H_{12}{}^{-2}$ decoupled from H^1 was found to be 30 c.p.s. Thus the decoupled B_{11} resonance of $B_{11}H_{14}{}^-$ suggests that there are two chemically shifted peaks too closely spaced to be resolved and this is consistent with the interpretation of the proton spectra.