$[(CH_3)_4N]_2$-1,10-$B_{10}Br_8(COC_6H_5)_2$ (decomposes at 342–344°). *Anal.* Calcd for $[(CH_3)_4N]_2B_{10}Br_8(COC_6H_5)_2$: C, 23.9; H, 3.1; B, 9.8; Br, 57.8; N, 2.5. Found: C, 23.2; H, 3.2; B, 9.4; Br, 58.5; N, 2.9.

The infrared spectrum in a potassium bromide wafer included absorptions at 3030 (w), 1610 (m, C=O), 1590 (w), 1575 (w), 1480 (ms), 1260 (m), 1150 (w), 950 (s), 835 (m), 780 (m), 700 (m), and 665 (w) cm^{-1}.

1,10-$B_{10}H_8(CH_3)_2{}^{2-}$. A solution of 1,10-$B_{10}H_8(CO)_2$ (3 g, 17 mmoles) in 50 ml of ether was added slowly to ether (50 ml) containing lithium aluminum hydride (2 g, 53 mmoles). The mixture was refluxed for 2 hr and then hydrolyzed with wet ether followed by 10 ml of water. The ether was decanted from the coagulated solid and the solid was washed with water until the washing gave no precipitate with cesium fluoride. The addition of cesium fluoride to the washings precipitated Cs_2-1,10-$B_{10}H_8(CH_3)_2$ (1.8 g, 26%, no melting point to 400°). *Anal.* Calcd for $Cs_2B_{10}H_8(CH_3)_2$: C, 5.8; H, 3.4; B, 26.3. Found: C, 5.8; H, 3.4; B, 25.8. Ultraviolet: no maximum.

The infrared spectrum in a potassium bromide wafer included absorptions at 2900 (w), 2830 (w), 2440 (s), 1440 (w), 1390 (w), 1210 (m), 1175 (m), 1025 (m), and 930 (m) cm^{-1}. The B^{11} mnr spectrum of the corresponding sodium salt in water consisted of a symmetrical doublet at 43 ppm (J = 112 cps) and a singlet at 14 ppm, referred to methyl borate. The proton spectrum of a similar solution consisted of a single peak at +3.1 ppm from internal water.

Chemistry of Boranes. XXXI.[1]
1,10-Bis(hydroxymethyl)octachlorodecaborate(2−)

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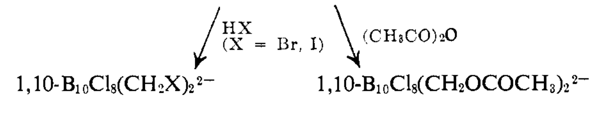
Abstract: Reduction of 1,10-$B_{10}Cl_8(CO)_2$ with lithium borohydride gives 1,10-$B_{10}Cl_8(CH_2OH)_2{}^{2-}$. This anion is readily acylated and reacts with halogen acids to form the corresponding bis(halomethyl) anions. The latter have been converted to cyanomethyl, carboxymethyl, aminomethyl, and other derivatives of $B_{10}H_{10}{}^{2-}$.

Previous work[1-3] has shown that carbonyl derivatives of $B_{10}H_{10}{}^{2-}$ and $B_{12}H_{12}{}^{2-}$ are versatile intermediates. Conversions of the carbonyl groups to amine, isocyanate, carboxyl, nitrile, alkyl, and other groups have been described. The scope of $B_{10}H_{10}{}^{2-}$ derivative chemistry has now been further extended by the reduction of 1,10-$B_{10}Cl_8(CO)_2$ to 1,10-$B_{10}Cl_8(CH_2OH)_2{}^{2-}$ (Figure 1) and the demonstration of preparative utility for this species.

The reduction to 1,10-$B_{10}Cl_8(CH_2OH)_2{}^{2-}$ is accomplished in good (60–90%) yield with lithium borohydride in glyme. The hydroxy groups are readily acylated by acetic anhydride and are easily replaced by bromine or iodine upon reaction with the corresponding halogen acid.



$$\begin{tikzcd}[column sep=small] \text{1,10-}B_{10}Cl_8(CO)_2 \arrow[r, "LiBH_4"] & \text{1,10-}B_{10}Cl_8(CH_2OH)_2{}^{2-} \arrow[dl, "\begin{matrix}HX\\(X = Br,\ I)\end{matrix}"] \arrow[dr, "(CH_3CO)_2O"] & \\ \text{1,10-}B_{10}Cl_8(CH_2X)_2{}^{2-} & & \text{1,10-}B_{10}Cl_8(CH_2OCOCH_3)_2{}^{2-} \end{tikzcd}$$

The halogen atoms bonded to carbon in 1,10-$B_{10}Cl_8(CH_2X)_2{}^{2-}$ are readily replaced by nucleophiles. In contrast, the chlorine atoms bonded to boron are quite inert, which is consistent with earlier observations on the lack of reactivity of B-halogenated derivatives of $B_{10}H_{10}{}^{2-}$.[4] This difference in halogen reactivity is similar to that which exists between phenyl and benzyl halides. This similarity is noteworthy because of other parallels[5,6] between $B_{10}H_{10}{}^{2-}$ chemistry and the chemistry of organic aromatic systems. It is emphasized, however, that no kinetic or other mechanistic studies on the reactions reported here have been made; extension of the observed parallels in reactivity to speculative parallels in causative factors, though inviting, would be premature.

The bromine atoms in 1,10-$B_{10}Cl_8(CH_2Br)_2{}^{2-}$ are hydrolyzed easily to regenerate 1,10-$B_{10}Cl_8(CH_2OH)_2{}^{2-}$. They are readily replaced by $CH_2SOCH_3{}^-$ in dimethyl sulfoxide and by cyanide in refluxing aqueous sodium cyanide. The nitrile groups in the product from the latter reaction have been hydrolyzed to form the corresponding dicarboxylic acid.

$$\text{1,10-}B_{10}Cl_8(CH_2Br)_2{}^{2-} \xrightarrow[(CH_3)_2SO]{CH_2SOCH_3{}^-} \text{1,10-}B_{10}Cl_8(CH_2CH_2SOCH_3)_2{}^{2-}$$

$$\text{1,10-}B_{10}Cl_8(CH_2Br)_2{}^{2-} \xrightarrow[H_2O,\ \text{reflux}]{CN^-}$$

$$\text{1,10-}B_{10}Cl_8(CH_2CN)_2{}^{2-} \xrightarrow[(3)\ H^+]{\begin{matrix}(1)\ H^+\\(2)\ OH^-\end{matrix}} \text{1,10-}B_{10}Cl_8(CH_2COOH)_2{}^{2-}$$

Titration in aqueous solution indicates a pK_a of approximately 6.9 for $Na_2B_{10}Cl_8(CH_2COOH)_2$, compared to approximately 7.5 for $Na_2B_{10}Cl_8(COOH)_2$.[1]

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[1] Paper XXX: W. H. Knoth, J. C. Sauer, J. H. Balthis, H. C. Miller, and E. L. Muetterties, *J. Am. Chem. Soc.*, **89**, 4842 (1967).

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[5] W. R. Hertler and M. S. Raasch, *J. Am. Chem. Soc.*, **86**, 3661 (1964).

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Reaction of $1,10\text{-}B_{10}Cl_8(CH_2Br)_2{}^{2-}$ with ammonia and with dimethylamine at 100° gives the corresponding aminomethyl species.

$$1,10\text{-}B_{10}Cl_8(CH_2Br)_2{}^{2-} \xrightarrow[100^\circ]{NH_3} 1,10\text{-}B_{10}Cl_8(CH_2NH_3)_2 \equiv 1,10\text{-}H_2B_{10}Cl_8(CH_2NH_2)_2$$

The pK_a in acetonitrile of $1,10\text{-}B_{10}Cl_8(CH_2NH_3)_2$ is 10.4 compared to 10.9 for $1,10\text{-}B_{10}Cl_8(NH_3)_2$.[7] The increase in acidity of the carboxymethyl and ammoniomethyl derivatives over their lower homologs presumably results from the insulating effect of the methylene group between the functional group and the electron-donating B_{10} polyhedron.[2]

Reaction of $1,10\text{-}B_{10}Cl_8(CH_2I)_2{}^{2-}$ with triphenylphosphine gives $1,10\text{-}B_{10}Cl_8[CH_2P(C_6H_5)_3]_2$.

These reactions show that the $1,10\text{-}B_{10}Cl_8(CH_2\!-\!)_2$ unit could be incorporated into many organic structures by classical reactions beginning with $1,10\text{-}B_{10}Cl_8(CH_2OH)_2{}^{2-}$. All the derivatives reported here show excellent thermal and oxidative stabilities; the limits are usually determined by the properties of the organic substituent.

Experimental Section

$1,10\text{-}B_{10}Cl_8(CH_2OH)_2{}^{2-}$. A solution of $1,10\text{-}B_{10}Cl_8(CO)_2$[1] (3.0 g, 6.7 mmoles) in glyme (50 ml) was added, with stirring in a nitrogen atmosphere, to lithium borohydride (1.5 g, 69 mmoles) in glyme (50 ml). The resulting mixture was refluxed for 1 hr and then cooled; the supernatant liquid was decanted leaving a viscous colorless oil. This was dissolved in water (80 ml). The addition of tetramethylammonium chloride precipitated $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OH)_2$ which recrystallized from water as glistening colorless platelets (2.4 g, 59.5% yield, no melting point to 360°). In another run, scaled up to 13.5 g of $1,10\text{-}B_{10}Cl_8(CO)_2$, the product was digested in 150 ml of water in lieu of recrystallization. The yield was 16.4 g, 90.5%; the infrared spectrum was identical with that of recrystallized material. *Anal.* Calcd for $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OH)_2$: C, 19.9; H, 5.0; B, 18.0; N, 4.6. Found: C, 19.9; H, 4.6; B, 17.6; N, 4.7.

The infrared spectrum in a Nujol mull includes absorptions at 3650 (w), 3450 (w), 1550 (w), 1100 (m), 1000 (s), 945 (m), and 870 cm^{-1}.

$1,10\text{-}B_{10}Cl_8(CH_2OCOCH_3)_2{}^{2-}$. A mixture of $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OH)_2$ (1 g, 1.7 mmoles) and acetic anhydride (25 ml) was refluxed for 1 hr, forming a clear solution. Water (50 ml) and excess 10% aqueous sodium hydroxide were added. The solution was chilled in a water–ice bath; the addition of aqueous tetramethylammonium chloride precipitated $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OCOCH_3)_2$ (decomposes at 294°) which was recrystallized from water. *Anal.* Calcd for $[(CH_3)_4N]_2B_{10}Cl_8(CH_2OCOCH_3)_2$: C, 24.5; H, 5.0; B, 15.8. Found: C, 24.8; H, 5.1; B, 15.7.

The infrared spectrum in a Nujol mull includes absorptions at 1700 (s), 1265 (ms), 1220 (ms), 1020 (s), 980 (m), 945 (m), 905 (mw), and 840 (mw) cm^{-1}.

$1,10\text{-}B_{10}Cl_8(CH_2Br)_2{}^{2-}$. A mixture of $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OH)_2$ (5.0 g, 8.3 mmoles) and 48% aqueous hydrobromic acid (75 ml) was refluxed for 2 hr and filtered hot. The crude $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2Br)_2$ (4.7 g, 77%) was recrystallized from aqueous acetonitrile to obtain 1.4 g (23%) of product (darkens at 305°). The crude material is sufficiently pure for preparative purposes. *Anal.* Calcd for $[(CH_3)_4N]_2B_{10}Cl_8(CH_2Br)_2$: C, 16.5; H, 3.9; B, 14.9; Br, 21.8; Cl, 39.0. Found: C, 17.4; H, 3.9; B, 14.7; Br, 22.1; Cl, 38.3. Ultraviolet: $\lambda_{max}^{CH_3CN}$ 234 mμ (ϵ 14,200). This product was converted to the corresponding sodium salt by acidic ion exchange in aqueous acetonitrile solution followed by removal of the acetonitrile and neutralization with sodium hydroxide. Aqueous solutions of $Na_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2Br)_2$ slowly liberated bromide ion and became acidic when heated on a steam bath, suggesting hydrolysis to the hydroxymethyl precursor. A solution of $Na_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2Br)_2{}^{2-}$ in 10% aqueous sodium hydroxide was refluxed for 1.5 hr. The addition of tetramethylammonium chloride precipitated $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OH)_2$, identified by infrared analysis.

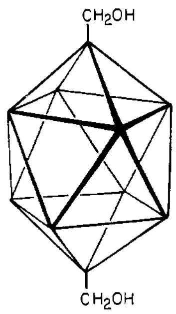

Figure 1. Structure of $1,10\text{-}B_{10}Cl_8(CH_2OH)_2{}^{2-}$ (chlorine atoms not shown).

The infrared spectrum of $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2Br)_2$ in a potassium bromide wafer includes absorptions at 3030 (w), 2920 (w), 1480 (s), 1450 (w), 1420 (w), 1400 (w), 1290 (w), 1160 (m), 1100 (w), 1000 (s), 975 (m), 950 (ms), 850 (m), and 790 (w) cm^{-1}.

$1,10\text{-}B_{10}Cl_8(CH_2I)_2{}^{2-}$. A mixture of $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OH)_2$ (0.5 g, 0.8 mmole) and concentrated iodine-free hydriodic acid (75 ml) was refluxed for 1.5 hr, cooled, and filtered. The filter cake was recrystallized from aqueous acetonitrile to obtain 3.9 g (57%) of colorless $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2I)_2$ (no melting point to 400°, darkens at 290–310°). The analytical sample was recrystallized a second time. *Anal.* Calcd for $[(CH_3)_4N]_2B_{10}Cl_8(CH_2I)_2$: C, 14.6; H, 3.4; B, 13.2; Cl, 34.5; I, 30.9; N, 3.4. Found: C, 14.7; H, 4.9; B, 13.4; Cl, 34.3; I, 29.6; N, 3.3. Ultraviolet: $\lambda_{max}^{CH_3CN}$ 242 mμ (ϵ 10,700) and 275 mμ (sh, ϵ 7050).

The infrared spectrum closely resembles that of the bromomethyl analog above, except that the bands at 1160 and 1100 cm^{-1} in the latter are shifted to 1120 and 1080 cm^{-1}, respectively.

$1,10\text{-}B_{10}Cl_8(CH_2CN)_2{}^{2-}$. $[CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2OH)_2$ (11 g, 18.3 mmoles) was converted to the corresponding sodium salt by acidic ion exchange followed by neutralization with sodium hydroxide and evaporation of the solution on a steam bath. A mixture of the sodium salt and 75 ml of 48% hydrobromic acid was refluxed for 2 hr. The addition of trimethylammonium chloride precipitated $[(CH_3)_3NH]_2B_{10}Cl_8(CH_2Br)_2$ which was removed and washed with water until it was free of acid. Three-quarters of this product was mixed with sodium cyanide (20 g, 0.43 mole) in water (50 ml); the mixture was refluxed for 30 min. The addition of excess cesium chloride precipitated $Cs_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2CN)_2$ as an off-white solid which was recrystallized twice from water (2.4 g, 24% yield, decomposes at 394°). *Anal.* Calcd for $Cs_2B_{10}Cl_8(CH_2CN)_2$: C, 6.5; H, 0.5; B, 14.6; N, 3.8. Found: C, 6.9; H, 1.3; B, 15.3; N, 3.9. Ultraviolet: $\lambda_{max}^{CH_3CN}$ 220 mμ (ϵ 10,850) and 260 mμ (sh, ϵ 460).

The infrared spectrum in a potassium bromide wafer includes absorption bands at 2940 (w), 2900 (w), 2240 (m, $C{\equiv}N$), 1400 (w), 1260 (m), 1180 (m), 1010 (s), 980 (ms), 850 (ms), 810 (w), 715 (w), and 690 (w) cm^{-1}.

$1,10\text{-}Br_{10}Cl_8(CH_2COOH)_2{}^{2-}$. A solution of $Cs_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2Br)_2$ (30 g, 35 mmoles) and sodium cyanide (30 g, 612 mmoles) in water (100 ml) was refluxed for 30 min, cooled, and filtered. The filter cake $[Cs_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2CN)_2]$ was washed with a little cold water and then passed, in aqueous solution, through a strongly acidic ion-exchange column. The effluent was evaporated to dryness on a steam bath. The residual solid was not fully characterized: infrared analysis suggested it was $Cs_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2CONH_2)_2$. This solid was dissolved in 100 ml of 10% aqueous sodium hydroxide; the solution was refluxed for 6 hr. Acidification with hydrochloric acid followed by the addition of tetramethylammonium chloride precipitated $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2COOH)_2$ which was recrystallized from water (14 g, 60% yield, slowly darkened above 300°). *Anal.* Calcd for $[(CH_3)_4N]_2B_{10}Cl_8(CH_2COOH)_2$: C, 21.9; H, 4.6; B, 16.4; Cl, 43.2. Found: C, 20.5; H, 4.7; B, 16.8; Cl, 43.3.

The neutral equivalent found by passing an aqueous solution of $[(CH_3)_4N]_2\text{-}1,10\text{-}B_{10}Cl_8(CH_2COOH)_2$ through a strongly acidic

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[7] J. Fok, unpublished.

ion-exchange column and titrating the effluent was 331 (calculated 329). The titration curve showed the presence of weakly as well as strongly acidic protons as required for $H_2B_{10}Cl_8(CH_2COOH)_2$.

The infrared spectrum of the tetramethylammonium salt in a potassium bromide wafer includes absorption bands at 1690 (s, C═O), 1480 (s), 1420 (m), 1300 (ms), 1230 (m), 1110 (mw), 1005 (s), 975 (m), 950 (s), and 860 (m) cm^{-1}.

1,10-$B_{10}Cl_8(CH_2CH_2SOCH_3)_2^{2-}$. Dimethyl sulfoxide (100 ml) and 53% sodium hydride dispersion in oil (6 g, 138 mmoles) were heated at 65–75° for 1.5 hr in a nitrogen atmosphere. The mixture was cooled to 30° and Cs_2-1,10-$B_{10}Cl_8(CH_2Br)_2$ (8.7 g, 10.3 mmoles) was added. The temperature rose to 40°; the mixture was stirred into water (500 ml) and the oil which separated was removed. Concentrated hydrochloric acid (12 *M*) was added until a white solid separated. This was dissolved in dilute sodium hydroxide; the addition of tetramethylammonium hydroxide precipitated $[(CH_3)_4N]_2$-1,10-$B_{10}Cl_8(CH_2CH_2SOCH_3)_2$ which was recrystallized from water (turns red at 184°, foams 360°).

The infrared spectrum in a Nujol mull includes a broad, strong absorption band centered at 1020 cm^{-1} and other bands at 945 (m), 900 (mw), and 850 (w) cm^{-1}. *Anal.* Calcd for $[(CH_3)_4N]_2$-$B_{10}Cl_8(CH_2CH_2SOCH_3)_2$: C, 23.2; H, 5.3; B, 15.0; Cl, 39.4; S, 8.9. Found: C, 22.7; H, 5.6; B, 14.9; Cl, 39.9; S, 8.9. A sample of $[(CH_3)_4N]_2$-1,10-$B_{10}Cl_8(CH_2CH_2SOCH_3)_2$ was passed through an acidic ion-exchange column. The effluent was evaporated to leave a solid residue which was recrystallized from 10% hydrochloric acid. The product was $(H_3O)_2B_{10}Cl_8(CH_2CH_2SOCH_3)_2$ (decomposes at 270°). *Anal.* Calcd for $(H_3O)_2B_{10}Cl_8(CH_2CH_2SOCH_3)_2$: C, 11.8; H, 3.3; B, 17.6; S, 10.5. Found: C, 11.6; H, 3.5; B, 17.4; S, 10.4.

1,10-$B_{10}Cl_8(CH_2NH_3)_2$. A mixture of ammonia (40 g, 2.4 moles) and Cs_2-1,10-$B_{10}Cl_8(CH_2Br)_2$ (15 g, 17.7 mmoles) was heated at 100° for 3 hr in a glass-lined pressure vessel. The ammonia was evaporated; the residue was dissolved in water, and the solution was passed through a strongly acidic ion-exchange column. The effluent was evaporated to dryness, leaving 6.9 g (86%) of crude 1,10-$B_{10}Cl_8(CH_2NH_3)_2$. This was recrystallized from water to obtain 3.2 g (40%) of pure material (no melting point to 400°). In a second run, a 78% yield of recrystallized product was obtained. *Anal.* Calcd for $B_{10}Cl_8(CH_2NH_3)_2$: C, 5.3; H, 1.8; B, 23.9; Cl, 62.9; N, 6.2. Found: C, 5.9; H, 2.8; B, 23.9; Cl, 63.1; N, 6.2. Ultraviolet: $\lambda_{max}^{H_2O}$ 255 mμ (sh, ε 555) and 221 mμ (ε 10,000).

The infrared spectrum in a Nujol mull includes absorption bands at 3570 (m), 3510 (w), 3280 (m), 1610 (m), 1090 (m), 1060 (s), 975 (s), 835 (m), and 800 (w) cm^{-1}.

1,10-$B_{10}Cl_8[CH_2N(CH_3)_2H]_2$. A mixture of dimethylamine (30 g, 0.67 mole) and Cs_2-1,10-$B_{10}Cl_8(CH_2Br)_2$ (12.5 g, 14.8 mmoles) was heated at 100° for 4 hr in a glass-lined pressure vessel. Evaporation of the excess dimethylamine left a white solid which was recrystallized from aqueous alcohol to obtain 4.4 g (69%) of 1,10-$B_{10}Cl_8[CH_2N(CH_3)_2H]_2$. *Anal.* Calcd for $B_{10}Cl_8[CH_2N(CH_3)_2H]_2$: C, 14.1; H, 3.5; B, 21.2; Cl, 55.5; N, 5.5. Found: C, 14.3; H, 3.4; B, 21.0; Cl, 55.5; N, 5.6. Ultraviolet: $\lambda_{max}^{CH_3CN}$ 250 mμ (ε 750) and 222 mμ (ε 12,000).

The infrared spectrum in a Nujol mull includes absorptions at 3180 (s), 1160 (w), 1120 (w), 1010 (s), 985 (s), 875 (m), and 816 (w) cm^{-1}.

1,10-$B_{10}Cl_8[CH_2P(C_6H_5)_3]_2$. A solution of $[(CH_3)_4N]_2$-1,10-$B_{10}Cl_8(CH_2I)_2$ (7.8 g, 9.5 mmoles) and triphenylphosphine (6 g, 23 mmoles) in dimethylformamide (100 ml) was refluxed for 24 hr. The solution was filtered into water (400 ml); a solid precipitated and was removed by filtration. This solid was washed with ethanol, leaving 8.2 g of crude 1,10-$B_{10}Cl_8[CH_2P(C_6H_5)_3]_2$. Of this amount, 7.5 g was extracted with 250 ml of acetonitrile in a Soxhlet extractor for 4 days. One gram (12%) of crystalline 1,10-$B_{10}Cl_8[CH_2P(C_6H_5)_3]_2$ (no melting point to 360°) separated from the extract. *Anal.* Calcd for $B_{10}Cl_8[CH_2P(C_6H_5)_3]_2$: C, 48.3; H, 3.6; B, 11.4; Cl, 30.0; P, 6.6. Found: C, 48.4; H, 4.0; B, 11.5; Cl, 30.3; P, 6.6.

The infrared spectrum in a potassium bromide wafer includes major absorption bands at 1600 (m), 1480 (m), 1440 (s), 1125 (s), 1105 (s), 1000 (s), 960 (m), 840 (m), 800 (s), 770 (s), 740 (vs), 720 (s), and 695 (vs) cm^{-1}.