does not apply. It should also be noted that, although a very strong peak at *m/e* 100, corresponding to $Ti(NC)_2^+$, is present in the titanium(IV) cyanate spectrum, it also is not part of the typical grouping of five peaks due to the titanium isotope distribution, and the $Ti(NC)_2^+$ assignment is most probably incorrect. Three other strong peaks are significant: that at *m/e* 207 in the vanadium(IV) thiocyanate spectrum, corresponding to $V(C_5H_5)_2NC^+$, and the *m/e* 213 and 303 peaks, both part of characteristic isotope patterns, in the zirconium(IV) and hafnium(IV) cyanate spectra, corresponding to $M(C_5H_5)(OCN)O^+$.

It would appear that the difference in the bonding modes indicated by the infrared and mass spectral data for the metallocene cyanate complexes may be attributed to the presence of the 3d electron in the titanium(III) and vanadium(IV) complexes, since it can participate in $d_\pi \rightarrow \pi^*$ bonding with the larger lobes of the vacant π^* cyanate orbitals located on the nitrogen atom. π bonding involving π(oxygen) $\rightarrow d_\pi$ overlap would be expected to predominate in the d^0 titanium(IV), zirconium(IV), and hafnium(IV) complexes. It will therefore be of considerable interest to learn if the same bonding distinction will be made in the corresponding complexes of niobium and tantalum in their IV and V oxidation states.

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π-Phosphacarbollyl Derivatives of Manganese, Iron, and Cobalt[1]

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The phosphacarboranes 7,8- and 7,9-$B_9H_{10}CHP^-$ and the corresponding $B_9H_{10}CHPCH_3$ derivatives are deprotonated with basic reagents to form "phosphacarbollide" ions. Sandwich-bonded transition metal compounds are formed with all four of the phosphacarbollide ligands. The syntheses, characterization, structures, and a limited number of reactions of some manganese, iron, and cobalt complexes are discussed.

In the past few years a large number of metal and nonmetal elements have been incorporated into various boron hydride cage structures.[3,4] The first example of phosphorus atom insertion into a borane structure involved reaction of $Na_3B_{10}H_{10}CH$ with phosphorus trichloride to form 1,2-$B_{10}H_{10}CHP$.[5] This phosphacarborane has chemical and physical properties quite similar to those of the better known isoelectronic species, 1,2-$B_{10}H_{10}C_2H_2$. Thermal rearrangement of 1,2-$B_{10}H_{10}CHP$ yields both 1,7- and 1,12-$B_{10}H_{10}CHP$.[6] Piperidine at reflux abstracts a boron atom from 1,2- and 1,7-$B_{10}H_{10}CHP$ to give the 7,8- and 7,9-$B_9H_{10}CHP^-$ ions, respectively. The phosphacarborane ions react with methyl iodide at the phosphorus atom to give the neutral derivatives 7,8- and 7,9-$B_9H_{10}CHPCH_3$.[6] The four 11-atom phosphacarboranes are isoelectronic with the previously reported 1,2- and 1,7-$B_9C_2H_{12}^-$ ions.[7]

In this paper we present our results to date on the manganese, iron, and cobalt complexes derived from the $B_9H_9CHP^{2-}$ and $B_9H_9CHPCH_3^-$ ions.

Experimental Section

Apparatus and Materials.—The infrared spectra for which a solvent is not indicated have been recorded as KBr disks. All infrared spectra were measured on a Perkin-Elmer 521 or 621 instrument. Intensities of absorptions are reported as strong (s), medium (m), or weak (w). Low-resolution mass spectra were obtained with an Atlas CH-4 instrument. High-resolution mass spectra were obtained with an AEI MS-9 instrument. Boron (^{11}B) nmr spectra at 32.1 MHz were obtained with a Varian HA-100 spectrometer. Spectral integrals were measured with a planimeter. Boron chemical shift data were measured relative to external trimethyl borate. All data presented in this paper are referenced to the $BF_3 \cdot (C_2H_5)_2O$ standard by addition of −18.15 ppm to the trimethyl borate chemical shift value. Proton nmr spectra were obtained with a Varian A-60, A-60A, or HA-100 spectrometer. Electronic spectra were obtained with a Cary 14 spectrometer. Melting points were obtained in evacuated sealed capillaries and are uncorrected. Elemental analyses (C, H, N, metal) were obtained from the University of Illinois Microanalytical Laboratory. All reactions were carried out under an atmosphere of prepurified nitrogen or argon. Magnetic susceptibilities were measured by the Faraday method using a Cahn Model G electrobalance and a Varian Associates D-4004

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electromagnet. Controlled-potential cyclic voltammetry was accomplished using a solid-state version of an instrument based of the design of DiSalvo and Schaap.[8] Potentials were measured at a platinum electrode *vs.* a saturated calomel electrode. All measurements were made in acetonitrile solution containing 0.1 *N* tetraethylammonium perchlorate as supporting electrolyte and an approximately 0.001 *M* concentration of metal complex.

$[(CH_3)_4N]_2Fe(1,2\text{-}B_9H_9CHP)_2$.—A mixture of $C_5H_{10}NH_2(7,8\text{-}B_9H_{10}CHP)$[6] (0.50 g, 0.0021 mol) in 50 ml of tetrahydrofuran (THF) was treated with excess sodium hydride at reflux for 1 hr. The supernatant liquid was transferred *via* syringe to a flask containing a slurry of anhydrous ferrous chloride (0.21 g, 0.0016 mol) in THF at reflux and reflux was continued for 12 hr. The solvent was removed under vacuum after adding a small amount of 100–200 mesh silica gel to the mixture. The products were chromatographed on a silica gel column and a red fraction was eluted with acetone. This fraction was treated with aqueous tetramethylammonium chloride. The crude product was crystallized from acetone–methanol to give 0.44 g (83% yield) of $[(CH_3)_4N]Fe(1,2\text{-}B_9H_9CHP)_2$, mp 294–295°. The infrared spectrum included absorptions at λ_{max} 3030 (m), 2950 (w), 2500 (s), 1430 (s), 1445 (m), 1410 (w), 1290 (w), 1170 (w), 1080 (m), 1000 (s), 950 (s), 890 (m), 750 (m), 730 (w), and 700 (w) cm^{-1}.

$[(CH_3)_4N]_2Fe(1,7\text{-}B_9H_9CHP)_2$.—A 4.00-g (0.017-mol) sample of $C_5H_{10}NH_2(7,9\text{-}B_9H_{10}CHP)$ in 25 ml of THF containing 5.0 ml of triethylamine was added dropwise to a stirred slurry of 3.0 g (0.023 mol) of ferrous chloride in 25 ml of THF at reflux. The red-violet solution was refluxed for 14 hr. The solvent was removed *in vacuo* and the residues were extracted with acetone. Aqueous tetramethylammonium chloride was added to the extract until precipitation was complete. The crude product was crystallized from acetone–methanol to give 3.40 g (80% yield) of $[(CH_3)_4N]_2Fe(1,7\text{-}B_9H_9CHP)_2$. The infrared spectrum contains absorptions at λ_{max} 3030 (w), 1481 (s), 1093 (m), 1040 (m), 995 (s), 946 (s), 945 (s), 752 (m), 742 (m), and 621 (w) cm^{-1}.

$[(CH_3)_4N]Fe(1,7\text{-}B_9H_9CHP)_2$.—A 0.59-g (0.01-mol) sample of $Cs_2Fe(1,7\text{-}B_9H_9CHP)_2$ was dissolved in water and oxidized by dropwise addition of an aqueous solution containing 0.60 g of ceric ammonium sulfate. The green precipitate was collected, dissolved in methanol, and treated with aqueous tetramethylammonium chloride until precipitation was complete. The dried residues were extracted with methylene chloride. The extract was partly evaporated and cooled in an ice bath until crystallization occurred. A 21% yield (0.09 g) of $[(CH_3)_4N]Fe(1,7\text{-}B_9H_9CHP)_2$ was obtained as green plates. The infrared spectrum contains λ_{max} at 3030 (m), 2050 (vs), 1481 (s), 1448 (w), 1416 (w), 1097 (m), 1033 (m), 986 (s), 946 (s), 734 (m), and 625 (w) cm^{-1}.

$Fe(1,2\text{-}B_9H_9CHPCH_3)_2$.—A 1.00-g (0.006-mol) sample of $7,8\text{-}B_9H_{10}CHPCH_3$[6] was deprotonated by reaction with excess sodium hydride in 25 ml of THF at reflux for 1 hr. This solution was filtered under nitrogen and added dropwise to a refluxing slurry of 1.2 g (0.0093 mol) of ferrous chloride in 25 ml of THF. The mixture was refluxed for 10 hr and then the solvent was removed under vacuum. The residue was chromatographed on a silica gel column using benzene as the eluent. Crystallization from benzene–heptane gave 0.22 g (19% yield) of $Fe(1,2\text{-}B_9H_9CHPCH_3)_2$, mp 325° dec. The infrared spectrum contains absorptions at λ_{max} 3020 (w), 2920 (m), 2560 (s), 1390 (w), 1260 (w), 1070 (m), 1020 (m), 990 (s), 945 (w), 895 (m), 800 (w), 775 (m), 750 (m), 735 (m), 720 (w), and 680 (w) cm^{-1}.

$Fe(1,7\text{-}B_9H_9CHPCH_3)_2$.—A 1.66-g (0.01-mol) sample of $7,9\text{-}B_9H_{10}CHPCH_3$ was treated with sodium hydride (0.01 mol) in THF at reflux and the solution was transferred to a dropping funnel fitted to a three-necked flask containing 1.9 g (0.015 mol) of anhydrous ferrous chloride and 50 ml of THF at reflux. The solution was added over 15 min and the mixture was stirred at reflux for 24 hr. The solution was removed *in vacuo* and the solids were chromatographed on a 20 × 0.75 in. column of silica gel. Elution with carbon tetrachloride gave two bands. The first band was collected, the solvent was evaporated, and the solids were crystallized from high-boiling petroleum ether (bp 90–110°) to give 0.60 g of red needles (isomer I), mp 239–240°. The second band was treated similarly to the first, except that the solid was crystallized from high-boiling petroleum ether with a small quantity of benzene added. Orange needles (0.53 g) of another compound (isomer II), mp 233–234°, were obtained. The infrared spectrum of isomer I contains absorptions at λ_{max} 3000 (w), 2920 (w), 2500 (vs), 1395 (m), 1283 (w), 1140 (w), 1132 (w), 1113 (w), 1102 (m), 1032 (m), 994 (s), 914 (w), 898 (w), 883 (w), 780 (m), 755 (w), 732 (m), 617 (w), 520 (w), and 409 (s) cm^{-1}. The infrared spectrum of isomer II contains absorptions at λ_{max} 2996 (w), 2019 (w), 2970 (vs), 1399 (m), 1282 (w), 1138 (w), 1109 (s), 1031 (m), 992 (s), 913 (w), 895 (w), 880 (w), 774 (m), 750 (w), 742 (m), 619 (w), 515 (w), and 404 (s) cm^{-1}. The low-voltage mass spectra of both isomers cut off at *m/e* 390 corresponding to the parent ion $^{56}Fe^{11}B_{18}{}^{1}H_{26}{}^{12}C_4{}^{31}P_2{}^+$.

$(CH_3)_4N[(1,7\text{-}B_9H_9CHP)Fe(1,7\text{-}B_9H_9CHPCH_3)]$.—A mixture of sodium hydride, 0.04 g, and $Fe(7,9\text{-}B_9H_9CHPCH_3)_2$ (isomer II), 0.1 g, was refluxed in 35 ml of 1,2-dimethoxyethane for 24 hr. The solvent was removed under vacuum. The products were dissolved in water and tetramethylammonium chloride solution was added until precipitation was complete. The orange-red product was recrystallized twice from acetone–methanol, mp 293–295°. The infrared spectrum contains absorptions at λ_{max} 2910 (w), 2530 (s), 1095 (m), 1028 (m), 985 (m), 885 (w), 875 (w), 768 (w), and 740 (w) cm^{-1}.

$(\pi\text{-}C_5H_5)Fe(1,7\text{-}B_9H_9CHPCH_3)$.—A tetrahydrofuran solution containing 0.82 g (0.005 mol) of $7,9\text{-}B_9H_{10}CHPCH_3$ and 1.65 g (0.025 mol) of freshly cracked cyclopentadiene was treated with excess sodium hydride. After reaction, the above solution was filtered under nitrogen into a dropping funnel. The solution containing NaC_5H_5 and $Na(7,9\text{-}B_9H_9CHPCH_3)$ was added to a mixture of 3.85 g (0.03 mol) of ferrous chloride in 100 ml of refluxing tetrahydrofuran. After a 16-hr reflux period, the solvent was removed under vacuum. Sublimation of the residues at 70° (10^{-2} mm) gave 1.46 g (0.0079 mol) of ferrocene. Further sublimation at 120° gave another material which was crystallized from methylene chloride–high boiling petroleum ether (bp 90–110°) to give 0.43 g (30% yield) of $(\pi\text{-}C_5H_5)Fe(1,7\text{-}B_9H_9CHPCH_3)$, mp 165–167°. The low-voltage mass spectrum of this new compound cut off sharply at *m/e* 388 corresponding to the parent ion $^{56}Fe^{11}B_9{}^{12}C_7{}^{1}H_{18}{}^{31}P^+$. The infrared spectrum (chloroform solution) shows absorptions at λ_{max} 3006 (w), 2920 (w), 2560 (vs), 1410 (m), 1283 (w), 1104 (m), 1030 (m), 922 (s), and 838 (s) cm^{-1}.

$[(CH_3)_4N]Co(1,2\text{-}B_9H_9CHP)_2$.—A 0.50-g (0.0021-mol) sample of $C_5H_{10}NH_2(7,8\text{-}B_9H_{10}CHP)$ was mixed with 1 equiv of cobalt(II) chloride in 50 ml of tetrahydrofuran. Triethylamine (0.0045 mol) was added and the mixture was refluxed for 15 hr. The solvent was removed under vacuum and the products were chromatographed on silica gel. The fraction eluted with methanol was treated with aqueous tetramethylammonium chloride until precipitation was complete. The crude product was crystallized from ethylene dichloride to give 0.059 g (13% yield) of orange $[(CH_3)_4N]Co(1,2\text{-}B_9H_9CHP)_2$. The infrared spectrum showed absorptions at λ_{max} 3010 (m), 2540 (s), 1480 (s), 1420 (w), 1410 (w), 1400 (w), 1310 (w), 1280 (w), 1080 (s), 1010 (m), 985 (s), 940 (s), 880 (m), 750 (w), 720 (m), 680 (w), 660 (w), 635 (w), and 610 (w) cm^{-1}.

$[(CH_3)_4N]Co(1,7\text{-}B_9H_9CHP)_2$. —One gram of $(CH_3)_3NH(7,9\text{-}B_9H_{10}CHP)$ was dissolved in 40 ml of tetrahydrofuran in a dropping funnel and 1.5 ml of trimethylamine was added. This solution was added over a 30-min period to 0.6 g of cobalt(II) chloride in 50 ml of refluxing tetrahydrofuran. Reflux was continued for 20 hr and then 0.39 g of anhydrous ferric chloride was added and the mixture was refluxed 1 hr more. The solvent was removed under vacuum and the residues were extracted with methanol. The extract was treated with aqueous tetramethylammonium chloride until precipitation was complete. The crude product was crystallized from ethylene dichloride to give 0.79 g (77% yield) of $[(CH_3)_4N]Co(1,7\text{-}B_9H_9CHP)_2$. The infrared

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[8] J. DiSalvo and W. B. Schaap, Abstracts, 153rd National Meeting of the American Chemical Society, Miami Beach, Fla., 1967, No. B-54.

spectrum contains absorptions at λ_{max} 2540 (s), 1101 (m), 1036 (m), 997 (s), 945 (w), and 735 (m) cm^{-1}.

$Co(1,2\text{-}B_9H_9CHPCH_3)_2$.—A mixture of $7,8\text{-}B_9H_{10}CHPCH_3$, 0.5 g (0.003 mol), cobalt(II) chloride, 0.2 g (0.0015 mol), and triethylamine, 0.5 ml (0.0053 mol), was refluxed in 30 ml of tetrahydrofuran for 12 hr. After removal of the solvent under vacuum, the products were separated by chromatography on a silica gel column using benzene as eluent to give 0.17 g (29% yield) of $Co(1,2\text{-}B_9H_9CHPCH_3)_2$, mp 276–278° dec. The infrared spectrum of this compound contains absorptions at λ_{max} 2960 (w), 2920 (m), 2850 (w), 2560 (s), 1450 (w), 1390 (w), 1280 (w), 1260 (m), 1090 (s), 1040 (s), 980 (s), 950 (w), 910 (w), 890 (w), 880 (w), 870 (w), 795 (m), and 775 (m) cm^{-1}.

$Co(1,7\text{-}B_9H_9CHPCH_3)_2$.—The same general procedure described for the preparation of $Fe(7,9\text{-}B_9H_9CHPCH_3)_2$ was used here. The separation of the isomers was accomplished by chromatography on a silica gel column. The two isomers were eluted from the column with carbon tetrachloride and methylene chloride. Both isomers were crystallized from methylene chloride–petroleum ether (bp 90–110°). Thus reaction of 1.83 g (0.01 mol) of $Na[7,9\text{-}B_9H_9CHPCH_3]$ and 1.30 g (0.01 mol) of cobalt(II) chloride gave 0.57 g (29% yield) of isomer I, mp 232–234°, and 0.59 g (30% yield) of isomer II, mp 278–279°. The infrared spectrum of isomer I shows absorptions at λ_{max} 3036 (w), 3005 (w), 2923 (w), 2560 (v), 1395 (m), 1286 (w), 1145 (w), 1136 (w), 1098 (w), 1087 (w), 1046 (m), 984 (s), 864 (w), 780 (m), 733 (m), 465 (w), 412 (w), and 381 (w) cm^{-1}. The infrared spectrum of isomer II shows absorption at λ_{max} 3018 (w), 2925 (m), 2580 (vs), 1396 (m), 1288 (w), 1146 (m), 1094 (s), 1048 (s), 985 (s), 960 (sh), 920 (m), 899 (m), 873 (m), 770 (s), and 733 (s) cm^{-1}.

$(1,7\text{-}B_9H_9CHP)Co(1,7\text{-}B_0H_9CHPCH_3)$.—A sample of $(CH_3)_4NCo(1,7\text{-}B_9H_9CHP)_2$, 0.18 g (0.0041 mol), was treated with excess methyl iodide at reflux in tetrahydrofuran for 24 hr. The solvent was removed under vacuum and the residues were heated in a vacuum sublimator at 130° to give 0.084 g (56% yield) of an orange crystalline sublimate, mp 181–183°. The low-voltage mass spectrum of the product cuts off at *m/e* 378 corresponding to the $^{59}Co^{11}B_{18}{}^{12}C_3{}^1H_{23}{}^{31}P_2{}^+$ ion. The infrared spectrum contains absorptions at λ_{max} 3025 (w), 2920 (w), 2580 (s), 1395 (m), 1285 (w), 1262 (m), 1130 (m), 1103 (s), 1080 (s), 985 (s), 944 (m), 915 (m), 890 (m), 798 (m), and 730 (s) cm^{-1}.

$(CH_3)_4N[1,7\text{-}B_9H_9CHP)Mn(CO)_3]$.—One gram of $(CH_3)_4N[7,9\text{-}B_9H_{10}CHP]$ (0.0045 mol) in 25 ml of tetrahydrofuran was treated with 3 ml of a 1.6 *N* solution of butyllithium in hexane and the mixture was refluxed briefly. Then $BrMn(CO)_5$, 1.32 g (0.0048 mol) was added and reflux was continued for 24 hr. The solvent was removed under vacuum and the residues were extracted with methanol. The extract was treated with aqueous tetramethylammonium chloride solution to give a bright yellow precipitate. The product was recrystallized twice from methylene chloride–hexane to give 0.08 g (5% yield) of light yellow $(CH_3)_4N[1,7\text{-}B_9H_9CHP)Mn(CO)_3]$. The infrared spectrum of the compound in acetonitrile solution contains peaks at 2025 and 1955 cm^{-1} and in KBr disk contains absorptions at λ_{max} 1157 (m), 1065 (m), 1020 (m), 915 (w), and 771 (w) cm^{-1}. The electronic spectrum (acetonitrile solution) contains λ_{max} (ϵ) at 354 (705) and 206 (21,000) mμ.

$(1,7\text{-}B_9H_9CHPCH_3)Mn(CO)_3$.—A 0.48-g (0.0029-mol) sample of $7,9\text{-}B_9H_{10}CHPCH_3$ was allowed to react with excess sodium hydride in tetrahydrofuran solution. This solution was filtered into a second reaction vessel and 0.80 g (0.0029 mol) of $BrMn(CO)_5$ was added. The mixture was refluxed for 2 hr and then the solvent was removed *in vacuo* at room temperature. Unreacted $BrMn(CO)_5$ was removed by room temperature sublimation of the residues. A second fraction was obtained by sublimation at 40° (10^{-2} mm) and further purified by chromatography on an alumina column with hexane as eluent to give 0.24 g (27% yield) of $(1,7\text{-}B_9H_9CHPCH_3)Mn(CO)_3$, mp 99.5–100°. The infrared spectrum (cyclohexane solution) contains absorptions at λ_{max} 3000 (w), 2920 (w), 2590 (s), 2051 (s), 1984 (s), 1963 (s), 1402 (w), 1289 (w), 1100 (m), 998 (s), 889 (m), 647 (s), and 633 (s) cm^{-1}. The osmometric molecular weight in acetone solution was 300 (calcd for $(B_9H_9CHPCH_3)Mn(CO)_3$, 304.4).

TABLE I

ELECTRONIC SPECTRAL DATA FOR THE PHOSPHACARBOLLYLIRON(II), -IRON(III), -COBALT(II), AND -COBALT(III) DERIVATIVES

Compound	Solvent	λ_{max}, mμ (ϵ)
$[(CH_3)_4N]_2Fe(1,2\text{-}B_9H_9CHP)_2$	CH_3CN	288 (10,300) 406 (292) 480 (215)
$[(CH_3)_4N]_2Fe(1,7\text{-}B_9H_9CHP)_2$	CH_3CN	289 (11,200) 406 (124) 513 (220)
$Fe(1,2\text{-}B_9H_9CHPCH_3)_2$	CH_3CN	255 (8950) 270 sh (7800) 445 (318)
$Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ (isomer I)	CH_3OH	270 (12,100) 383 (130) 465 (805)
$Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ (isomer II)	CH_3OH	281 (13,000) 377 (130) 475 (590)
$(\pi\text{-}C_5H_5)Fe(1,7\text{-}B_9H_9CHPCH_3)$	CH_3OH	258 (7100) 374 (190) 345 (145) 436 (260)
$(CH_3)_4N[Fe(1,7\text{-}B_9H_9CHP)_2]$	CH_3OH	289 (12,540) 270 sh (11,520) 335 (8840) 500 (203)
$(CH_3)_4N[Co(1,2\text{-}B_9H_9CHP)_2]$	CH_3CN	210 (15,000) 245 (9230) 328 (15,300) 370 sh (2270) 430 (456)
$(CH_3)_4N[Co(1,7\text{-}B_9H_9CHP)_2]$	CH_3OH	273 (15,760) 330 (12,950) 451 (357)
$Co(1,2\text{-}B_9H_9CHPCH_3)_2$	CH_3CN	292 (10,700) 338 (8760) 410 sh (913) 518 (262)
$Co(1,7\text{-}B_9H_9CHPCH_3)_2$ (isomer I)	CH_3OH	292 (10,000) 338 (8150) 410 sh (900) 517 (240)
$Co(1,7\text{-}B_9H_9CHPCH_3)_2$ (isomer II)	CH_3OH	215 (18,600) 298 (8100) 355 (7900)[a] 500 sh (46)[a]

[a] These two peaks were measured in acetone solution.

Results and Discussion

Preparation and Characterization of Phosphacarbollyliron Derivatives.—Deprotonation of $7,8\text{-}B_9H_{10}CHP^-$ with either sodium hydride or triethylamine in tetrahydrofuran solution generated the phosphacarbollyl anion $7,8\text{-}B_9H_9CHP^{2-}$.[9] The other three phosphacarbollyl anions used in this study, $7,9\text{-}B_9H_9\text{-}$

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(9) The numbering system employed in this article follows the new nomenclature rules which recently appeared in *Inorg. Chem.*, **7**, 1945 (1968). Unfortunately, two numbering systems have been suggested for *closo* translation metal complexes. One method suggests that the *nido* borane ligand be numbered prior to (rule 2.321) incorporation into the metal complex [*e.g.*, $Fe(7,9\text{-}B_9H_9CHPCH_3)_2$]. The other method numbers the *closo* complex considering the metal atom as part of the cage [*e.g.*, $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$]. The latter method avoids the difficulty of translating from one numbering system to another when comparing closely related icosahedral systems. It is hoped that the nomenclature committee will rapidly clarify this numbering system ambiguity.

TABLE II

ANALYTICAL DATA FOR π-PHOSPHACARBOLLYL TRANSITION METAL DERIVATIVES

	% calcd				% found			
Compound	C	H	N	Metal	C	H	N	Metal
$[(CH_3)_4N]_2Fe(1,2-B_9H_9CHP)_2$	23.79	8.78	5.55	11.06	24.05	8.91	6.02	10.72
$[(CH_3)_4N]Fe(1,7-B_9H_9CHP)_2$	16.73	7.49	3.25	12.97	17.11	7.05	3.04	12.37
$[(CH_3)_4N]Fe(1,7-B_9H_9CHP)-(1,7-B_9H_9CHPCH_3)$	18.86	7.91	3.14	12.53	19.56	8.03	3.85	12.12
$Fe(1,7-B_9H_9CHPCH_3)_2$[a]	12.43	6.78	...	14.44	13.10	6.78	...	14.51
$[(CH_3)_4N]Co(1,2-B_9H_9CHP)_2$	16.61	7.44	3.23	13.58	16.78	7.91	...	12.71
$Co(1,2-B_9H_9CHPCH_3)_2$	12.32	6.74	...	15.11	12.84	6.53	...	15.13
$(1,7-B_9H_9CHP)Co(1,7-B_9H_9CHPCH_3)$	9.61	6.18	...	58.93	10.49	6.54	...	58.73
$(CH_3)_4N[(CO)_3Mn(1,7-B_9H_9CHP)]$	26.43	6.10	3.85	15.12	26.25	6.06	3.79	15.36

[a] The melting point of this isomer is 233–234°.

TABLE III

^{1}H NMR DATA OF 1,2- AND 1,7-PHOSPHACARBOLLYLIRON AND -COBALT DERIVATIVES

Compound	Solvent	Resonance, τ value (rel area)	Assignment
$[(CH_3)_4N]_2(1,2-B_9H_9CHP)_2Fe$	Dimethyl sulfoxide-d_6	Sharp singlet, 6.85 (12)	Methyl protons of cation
		Broad singlet, 7.48 (1)	Carborane C–H
$[(CH_3)_4N]_2(1,7-B_9H_9CHP)_2Fe$	Acetone-d_6	Sharp singlet, 6.86 (12)	Methyl protons of cation
		Broad singlet, 8.0 (~1)	Carborane C–H
$Fe(1,2-B_9H_9CHPCH_3)_2$	Dimethyl sulfoxide-d_6	Sharp doublet, 6.72 Sharp doublet, 6.99 (~3)	P–CH_3 (J = 12 Hz)
		Broad singlet, 7.0 Broad singlet, 7.3 (~1)	Carborane C–H
$Fe(1,7-B_9H_9CHPCH_3)_2$ (isomer I)	Acetone-d_6	Sharp doublet, 7.2 (3)	P–CH_3 (J = 13 Hz)
		Broad singlet, ~7.3 (1)	Carborane C–H
$Fe(1,7-B_9H_9CHPCH_3)_2$ (isomer II)	Acetone-d_6	Broad singlet, 6.2 (1)	Carborane C–H
		Sharp doublet, 7.34 (3)	P–CH_3 (J = 13 Hz)
$(\pi-C_5H_5)Fe(1,7-B_9H_9CHPCH_3)$	$CDCl_3$	Sharp doublet, 5.18 (5)	Cyclopentadienyl protons (J = 1 Hz)
		Broad singlet, 7.3 (1)	Carborane C–H
		Sharp doublet, 7.7 (3)	P–CH_3 (J = 13 Hz)
$(CH_3)_4N[(1,7-B_9H_9CHP)Fe(1,7-B_9H_9CHPCH_3)]$	Acetone-d_6	Sharp singlet, 6.7 (12)	Methyl protons of cation
		Broad singlet, 6.8 (2)	Carborane C–H
		Sharp doublet, 7.47 (3)	P–CH_3 (J = 13 Hz)
$[(CH_3)_4N](1,2-B_9H_9CHP)_2Co$	Acetone-d_6	Sharp singlet, 6.56 (12)	Methyl protons of cation
		Broad doublet,[a] 7.11 (2)	Carborane C–H
$[(CH_3)_4N](1,7-B_9H_9CHP)_2Co$	Acetone-d_6	Sharp singlet, 6.6 (12)	Methyl protons of cation
		Broad singlet, 6.9 (2)	Carborane C–H
$(1,7-B_9H_9CHP)Co(1,7-B_9H_9CHPCH_3)$	$CDCl_3$	Broad singlet, 5.8 Broad singlet, 6.2 [2]	Carborane C–H Carborane C–H
		Sharp doublet, 7.30 Sharp doublet, 7.33 [3]	P–CH_3 (J = 13 Hz) P–CH_3 (J = 13 Hz)

[a] The two peaks assigned to the carborane CH protons are separated by 15 Hz.

CHP^{2-} and 7,8- and 7,9-$B_9H_9CHPCH_3^-$, were formed *in situ* in the same manner. When 7,8-$B_9H_9CHP^{2-}$ was treated with anhydrous iron(II) chloride in tetrahydrofuran solution the red complex ion, $Fe(1,2-B_9H_9CHP)_2^{2-}$ was obtained. The $Fe(1,7-B_9H_9CHP)_2^{2-}$ ion was prepared similarly. Iron(II) derivatives were isolated from aqueous solution as the tetramethylammonium salts in good yield. The elemental analyses of all compounds reported here are given in Table II or have been reported in a preliminary communication.[10] Treatment of $[(CH_3)_4N]_2Fe(1,7-B_9H_9CHP)_2$ with methyl iodide in tetrahydrofuran solution produced a sublimable red-orange product. Chromatography on a silica gel column with carbon tetrachloride as eluent gave two components, isomer I (mp 239.5–240.5°) and isomer II (mp 233–234°). The two isomers described above as well as the $(1,2-B_9H_9CHPCH_3)_2Fe$ derivatives were also obtained by reaction of the appropriate $B_9H_9CHPCH_3^-$ ion with iron(II) chloride. Separation of the $(1,2-B_9H_9CHPCH_3)_2Fe$ isomers was not accomplished but the presence of the two isomers was observed in the proton nmr spectrum of the mixture (see Table III). The mass spectrum of each isomer cuts off at m/e 390 corresponding to the parent ion, $^{56}Fe^{11}B_{18}{}^{12}C_4{}^1H_{26}{}^{31}P_2^+$.[11] Iron complexes of the

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(10) L. J. Todd, I. C. Paul, J. L. Little, P. S. Welcker, and C. R. Peterson, *J. Am. Chem. Soc.*, **90**, 4439 (1968).

(11) In some samples a weak multiplet was observed at 14 mass units above the parent ion. This was identified as a molecule with one extra methyl group by measuring the most intense peak of the multiplet as m/e 401.2802 (calculated for $^{56}Fe^{11}B_{15}{}^{10}B_3{}^{12}C_5{}^1H_{28}{}^{31}P_2$, 401.2799). This is also reflected in the slightly high carbon analysis observed with several of the compounds.

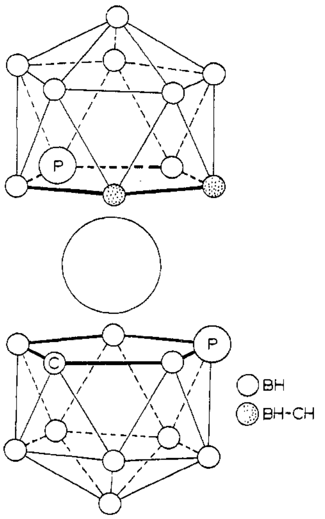

Figure 1.—Schematic drawing of $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$; the methyl groups attached to the phosphorus atoms are not shown.

7,9-dicarbollide ion have not been successfully prepared as yet[4] and in general the 7,9-dicarbollide complexes are less stable than the corresponding 7,8 isomers.[12] As is seen above, this limitation does not apply to the phosphacarbollide complexes. Isomer I was partially resolved by chromatography on a lactose hydrate column employing heptane as eluent to give a rotation $[\alpha]^{25}{}_{5000}$ +69°. A *dl* pair of $B_9H_{10}CHP^-$ ions will be generated by base abstraction of either of the two equivalent boron atoms adjacent to both the carbon and the phosphorus atoms in $1,7\text{-}B_{10}H_{10}\text{-}CHP$.[6] Isomers I and II are then a *dd,ll* racemate and a *dl,meso* form, respectively. A cocrystallite containing both isomers has been analzyed by X-ray methods.[10] The distances between the iron atom and the atoms of the open pentagonal faces of the two phosphacarbollide ions are nearly equal. Thus this is a symmetrical sandwich-bonded complex similar to the previously reported iron and cobalt dicarbollyl compounds.[12] Although the two pentagonal rings directly π bonded to the iron atom are within 2° of being parallel, there is a significant distortion of the icosahedral structure caused by the phosphorus atoms. An idealized view of the molecular structure of $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ is presented in Figure 1. The dotted atoms in the structure represent the two positions partly occupied by both carbon and boron atoms in the cocrystallite.

The ^{11}B nmr spectra of the two $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ isomers are given in Figure 2. It is seen that the two spectra are quite similar but possess differences in detail which allow one to distinguish the two isomers. The boron spectra of all of the diamagnetic phosphacarbollylmetal complexes presented in this paper have an envelope which is approximately 30 ppm wide but are uninformative because of extensive overlap of the individual resonance lines. The electronic spectral data of the iron derivatives are presented in Table I without interpretation.

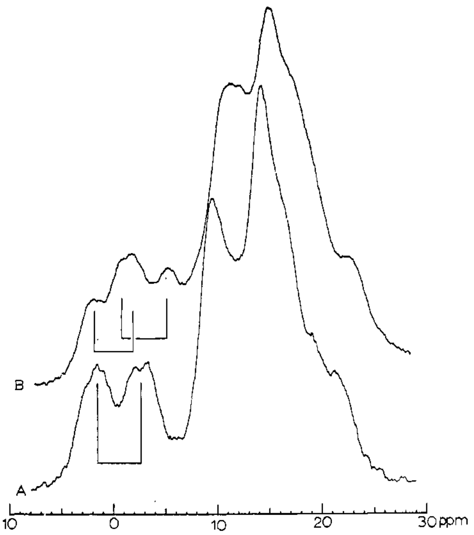

Figure 2.—The ^{11}B nmr spectra of the $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ isomers in tetrahydrofuran solution referenced to $BF_3\cdot(C_2H_5)_2O$: A, isomer II; B, isomer I.

During the separation of the two isomers of $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ by column chromatography, a small quantity of a third component was eluted with acetone. This was identified as the $(1,7\text{-}B_9H_9CHP)Fe(1,7\text{-}B_9H_9\text{-}CHPCH_3)^-$ ion. To test the possibility that this ion could be generated by demethylation of a neutral iron complex, $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ (isomer II) was treated with sodium hydride in monoglyme at reflux for 24 hr giving $(CH_3)_4N[(1,7\text{-}B_9H_9CHP)Fe(1,7\text{-}B_9H_9\text{-}CHPCH_3)]$. The integral ratios observed in the ^1H nmr spectrum of this compound (Table III) are in agreement with this formulation.

In contrast to the bis(carbollyl)- and bis(dicarbollyl)-iron(II) complexes, the $Fe(B_9H_9CHP)_2{}^{2-}$ ions are not susceptible to air oxidation in aqueous solution. The reduction potential data presented in Table IV clearly indicate the relative susceptibility of these iron(II) complexes to ($d^6 \rightarrow d^5$) oxidation. Increased methylation of the phosphacarbollyliron complexes appears to decrease the ease of electrochemical oxidation. It is also of interest to note that the $(B_{10}H_{10}S)_2Fe^{2-}$ ion is more stable in the +2 formal oxidation state.

Oxidation of $Cs_2[Fe(1,7\text{-}B_9H_9CHP)_2]$ with ceric ion produced the green paramagnetic $Fe(1,7\text{-}B_9H_9CHP)_2{}^-$ ion in low yield. Figure 3 presents the ^{11}B nmr spectrum of this iron(III) complex. The individual resonance lines are broad, do not appear to exhibit directly bonded $^{11}B\text{-}^1H$ spin coupling, and span a large chemical shift range (360 ppm). The approximate integral ratio of the low-field multiplet to the broad upfield resonance is 6:3. Upon expanding the low-field regions (−24 to +29 ppm), the four peaks in this area have the integral ratio 1:2:1:2, reading upfield. The high-field resonance of weight 3 may thus be assigned to the three boron atoms in the open face of the 7,9-phosphacarbollide ion, since they would be most affected by the unpaired electron on the iron atom. Simi-

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[12] M. F. Hawthorne, D. C. Young, T. D. Andrews, D. V. Howe, R. L. Pilling, A. D. Pitts, M. Reintjes, L. F. Warren, Jr., and P. A. Wegner, *J. Am. Chem. Soc.*, **90**, 879 (1968).

TABLE IV

REDUCTION POTENTIALS FOR OLLYLIRON AND -COBALT DERIVATIVES

Compound	Method	$E_{1/2}$ vs. sce	
		$M^{III} + e^- \rightarrow M^{II}$	$M^{II} + e^- \rightarrow M^{I}$
$[(CH_3)_4N]_2(1,2\text{-}B_9H_9CHP)_2Fe$	cv[a]	+0.08	. . .
$[(CH_3)_4N]_2(1,7\text{-}B_9H_9CHP)_2Fe$	cv	+0.05	. . .
$(CH_3)_4N[(1,7\text{-}B_9H_9CHP)Fe(1,7\text{-}B_9H_9CHPCH_3)]$	cv	+0.74	. . .
$(1,2\text{-}B_9H_9CHPCH_3)_2Fe$	cv	+1.6	. . .
$(1,7\text{-}B_9H_9CHPCH_3)_2Fe$ (isomer I)	cv	+1.46	. . .
$[(CH_3)_4N](1,2\text{-}B_9H_9C_2H_2)_2Fe$	Polarography[b]	−0.424	. . .
$[(CH_3)_4N]_2(B_{10}H_{10}S)_2Fe$	Polarography[c]	+0.2	. . .
$(1,2\text{-}B_9H_9CHPCH_3)_2Co$	cv	+0.43	−0.78
$(1,7\text{-}B_9H_9CHPCH_3)_2Co$ (mp 278–279°)	cv	+0.41	−0.74

[a] Cyclic voltammetry. [b] See ref 12. [c] W. R. Hertler, F. Klanberg, and E. L. Muetterties, *Inorg. Chem.*, **6**, 1696 (1967).

lar paramagnetic contact or pseudocontact shifts have been observed in the ^{11}B nmr spectra of the 1,2-dicarbollyliron(III) derivatives.[12]

The effective magnetic moment of the $Fe(1,7\text{-}B_9H_9CHP)_2^-$ ion (see Table V) was measured by the Faraday technique.[13] German and Dyatkina[14] have calculated μ_{eff} of 2.38 BM for $Fe(1,2\text{-}B_9H_9C_2H_2)_2^-$ using the g values measured earlier in an electron spin resonance study by Maki and Berry.[15] We also measured the magnetic moment of $[(CH_3)_4N]Fe[1,2\text{-}B_9H_9C_2(CH_3)_2]_2$ and obtained a higher value than previously reported.[12,16] Thus all of the sandwich-bonded iron(III) complexes studied to date have an effective magnetic moment of about 2.3 BM.

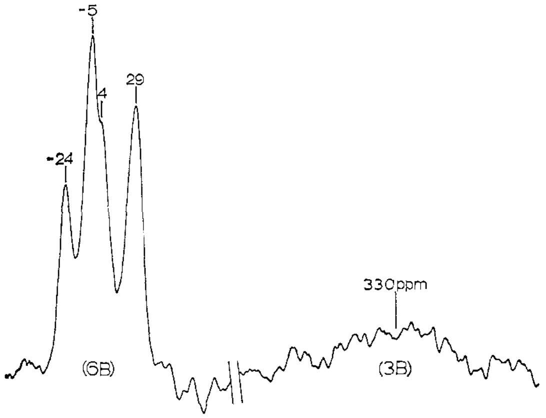

Figure 3.—The ^{11}B nmr spectrum of $[(CH_3)_4N]Fe(1,7\text{-}B_9H_9CHP)_2$ in acetone solution referenced to $BF_3\cdot(C_2H_5)_2O$.

π-Cyclopentadienyl-1,7-phosphacarbollyliron.—The neutral cyclopentadienyliron(II) complex was obtained in 30% yield (based on the amount of starting phosphacarbollide ion) by reaction in tetrahydrofuran of $C_5H_5^-$, $7,9\text{-}B_9H_9CHPCH_3^-$, and ferrous chloride in a 5:1:3 mole ratio, respectively. The ^1H nmr spectrum of this compound exhibited a sharp doublet at τ 7.47 (J = 13 Hz) of intensity 3 which was assigned to the P–CH_3 protons. A ^{31}P–C–^1H coupling constant of about 13 Hz has been observed in all complexes involving the $B_9H_9CHPCH_3^-$ ions. A sharp doublet of intensity 5 at τ 5.18 in the above spectrum was assigned to the cyclopentadienyl protons and the 1-Hz splitting was attributed to long-range spin coupling with the phosphorus atom. The ^{11}B nmr spectrum of this mixed-ligand complex closely resembles those of the $Fe(1,7\text{-}B_9H_9CHPCH_3)_2$ isomers.

Phosphacarbollylcobalt Complexes.—Reactions of $7,8\text{-}B_9H_9CHPCH_3^-$ with anhydrous cobalt(II) chloride gave orange-red $(1,2\text{-}B_9H_9CHPCH_3)_2Co$ in moderate yield. The two isomers could not be separated in this case. A similar reaction employing the 7,9 ligand produced both $(1,7\text{-}B_9H_9CHPCH_3)_2Co$ isomers. These complexes have cobalt in the formal +2 oxidation state but are not as susceptible to oxidation as cobaltocene. They can be recrystallized from boiling solvent in the air without detectable oxidation. In contrast, the reaction of 7,8- or $7,9\text{-}B_9H_9CHP^{2-}$ with anhydrous cobalt(II) chloride gave directly the corresponding $(B_9H_9CHP)_2Co^-$ complexes accompanied by formation of cobalt metal. Cobalt complexes having a formal +3 oxidation state have also been obtained by reaction of cobalt(II) chloride with the 7,8- and $7,9\text{-}B_9H_9C_2H_2^{2-}$ and $B_{10}H_{10}CH^{3-}$ ions.[12,17] The over-all charge on the carborane–transition metal

TABLE V

EFFECTIVE MAGNETIC MOMENTS OF π-PHOSPHACARBOLLYL-, π-DICARBOLLYL-, AND π-CYCLOPENTADIENYLIRON(III) AND -COBALT DERIVATIVES

Compound	μ_{eff}, BM
$(CH_3)_4N[Fe(1,7\text{-}B_9H_9CHP)_2]$	2.34
$[(CH_3)_4N][Fe[1,2\text{-}B_9H_9C_2(CH_3)_2]_2$	2.45 1.99[a]
$[(CH_3)_4N]Fe[1,2\text{-}B_9H_9C_2H_2]_2$	2.10[a]
$[ClO_4]Fe(\pi\text{-}C_5H_5)_2$	2.34
$(1,2\text{-}B_9H_9CHPCH_3)_2Co$[b]	1.68
$(1,7\text{-}B_9H_9CHPCH_3)_2Co$ (mp 278–279°)	1.89
$(1,7\text{-}B_9H_9CHPCH_3)_2Co$ (mp 231–234°)	1.82
$(\pi\text{-}C_5H_5)_2Co$	1.76 ± 0.07[c]

[a] See ref 12. [b] χ_m has been corrected for a diamagnetic contribution of 153 × 10^{-6} cgsu using the susceptibility of $(1,2\text{-}B_9H_9CHPCH_3)_2Fe$. [c] E. O. Fischer and H. P. Fritz, *Advan. Inorg. Chem. Radiochem.*, **1**, 55 (1959).

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(13) $\mu_{eff} = 2.83\sqrt{\chi_m T}$ BM, where χ_m has been corrected for a diamagnetic contribution of 384 × 10^{-6} cgsu using the susceptibility of $(CH_3)_4N[Co(1,7\text{-}B_9H_9CHP)_2]$.

(14) E. D. German and M. E. Dyatkina, *Zh. Strukt. Khim.*, **7**, 866 (1966).

(15) A. H. Maki and T. E. Berry, *J. Am. Chem. Soc.*, **87**, 4437 (1965).

(16) In this case χ_m has been corrected for a diamagnetic contribution of 203 × 10^{-6} cgsu using the susceptibility of $(CH_3)_4N[Co(1,2\text{-}B_9H_9C_2H_2)_2]$ and Pascal's constants.

(17) D. E. Hyatt, J. L. Little, J. T. Moran, F. R. Scholer, and L. J. Todd, *J. Am. Chem. Soc.*, **89**, 3342 (1967).

complex appears to have an important influence upon the redox behavior of the molecule. In a cyclic voltammetry study the $(B_9H_9CHPCH_3)_2Co$ species (*i.e.*, the 1,2 and 1,7 derivatives) contained reversible oxidation and reduction waves as seen in Table IV. However, the $(B_9H_9CHP)_2Co^-$ species (*i.e.*, the 1,2 and 1,7 derivatives) show no oxidation or reduction waves between +1.5 and −1.0 V (*vs.* sce). The reversible redox reaction [Co(II)–Co(III)], observed by cyclic voltammetry for $(1,2\text{-}B_9H_9CHPCH_3)_2Co$ at +0.43 V (*vs.* sce), suggests that the cationic Co(III) complex might be isolable. Attempts to date have not been successful.

The effective magnetic moments of the cobalt(II) complexes are given in Table V. The average value is 1.79 BM which compares well with the magnetic moment for cobaltocene and corresponds to a spin-only formulation of one unpaired electron.

Reaction of $(1,7\text{-}B_9H_9CHP)_2Co^-$ with methyl iodide in tetrahydrofuran solution gave a 56% yield of orange, sublimable $(1,7\text{-}B_9H_9CHPCH_3)Co(1,7\text{-}B_9H_9CHP)$. The proton nmr spectrum contains two P–CH_3 and two carborane C–H resonances indicating that the above compound is a mixture of two isomers as expected.

The electronic spectra of the phosphacarbollylcobalt derivatives are presented in Table I without interpretation. The ^1H nmr data of the cobalt complexes are given in Table III. Both ^{11}B and ^1H nmr spectral data of the paramagnetic cobalt(II) complexes were difficult to obtain because of the insolubility of the compounds in suitable solvents and the broadness of the resonance lines. The ^1H nmr spectrum of $(1,2\text{-}B_9H_9CHPCH_3)_2Co$ and $(1,7\text{-}B_9H_9CHPCH_3)_2Co$ (isomer I) (acetone-d_6) contained an intense, broadened singlet at τ 6.76 and 7.25, respectively, which may be due to the methyl group attached to phosphorus. The carborane C–H could not be detected.

Phosphacarbollylmanganese Tricarbonyl Derivatives.—Reaction of $7,9\text{-}B_9H_9CHPCH_3^-$ or $7,9\text{-}B_9H_9CHP^{2-}$ with 1 equiv of $BrMn(CO)_5$ at reflux in tetrahydrofuran resulted in carbon monoxide evolution over a 2-hr period and produced low yields of $(1,7\text{-}B_9H_9CHPCH_3)Mn(CO)_3$ and $(1,7\text{-}B_9H_9CHP)Mn(CO)_3^-$, respectively. The infrared spectrum of $(1,7\text{-}B_9H_9CHP)Mn(CO)_3^-$ (acetonitrile solution) contains A and E carbonyl stretching modes at 2025 and 1955 cm^{-1}, respectively. The infrared spectrum of $(1,7\text{-}B_9H_9CHPCH_3)Mn(CO)_3$ (CS_2 solution) contains a carbonyl band (A mode) at 2045 cm^{-1}. However, the E mode predicted for C_{3v} local symmetry is apparently no longer degenerate but is split into two bands at 1984 and 1963 cm^{-1}. The ^1H nmr spectrum ($CDCl_3$ solution) of $(1,7\text{-}B_9H_9CHPCH_3)Mn(CO)_3$ contained a sharp doublet at τ 7.82 (J = 13 Hz) and a broad singlet at τ 8.2 assigned to the methyl group on phosphorus and the carborane C–H, respectively. The ^{11}B nmr spectra of these two compounds extended over approximately 30 ppm but were uninterpretable.

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1,2,4,5-Tetraaza-3,6-diborinane Monomers and Dimers

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The thermal decomposition of hydrazine–*t*-butylborane has been examined and the product has been identified as a polyhedral "cage" compound, a dimer of 3,6-di-*t*-butyl-1,2,4,5-tetraazadiborinane (VIII). Nuclear magnetic resonance data for this compound and its derivatives with methyl isocyanate are presented and discussed in relation to their structures. Proton-exchange studies of VIII and its derivatives were monitored by nmr. Attempts to dimerize 3,6-diphenyl-1,2,4,5-tetraaza-3,6-diborinane (I, R = C_6H_5) provided the same product as thermal decomposition of hydrazine–phenylborane. This product is not a polyhedral cage compound.

Introduction

Initial attempts at the preparation of hydrazinoboranes[1,2] were unsuccessful and it was not until 1961 that the first compound of this type, diborylhydrazine, $H_2B\text{–}NH\text{–}NH\text{–}BH_2$, was reported; it was prepared by the pyrolysis of a diborane–hydrazine adduct.[3] Alkylated compounds of similar structure were obtained by treatment of tetraalkyldiboranes with hydrazine at 100–150°.[4] Formation of compounds of type I from the reaction of bis(amino)boranes with hydrazine have been reported[5,6] and this type of ring structure

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[1] H. J. Emeléus and F. G. A. Stone, *J. Chem. Soc.*, 840 (1951).
[2] J. Steindler and H. I. Schlesinger, *J. Am. Chem. Soc.*, **75**, 756 (1953).
[3] J. Goubeau and E. Ricker, *Z. Anorg. Allgem. Chem.*, **310**, 123 (1961).
[4] H. Noth, *Z. Naturforsh.*, **16b**, 471 (1961).
[5] K. Niedenzu, H. Beyer, and J. W. Dawson, *Inorg. Chem.*, **1**, 738 (1962).
[6] H. Noth and W. Regnet, "Boron–Nitrogen Chemistry," Advances in Chemistry Series, No. 42, American Chemical Society, Washington, D. C., 1964, p 166.