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REACTION OF (IR(CYCLO-OCTA-1,5-DIENE)(P(P-TOLYL)3)2) (NIDO-7,8---ETC(U)
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6 Reaction of [Ir(cyclo-octa-1,5-diene){P(p-tolyl)$_3$}$_2$] [nido-7,8-$C_2B_9H_{12}$] with Hydrogen; Synthesis and X-Ray Structure of 3,9-[(H)$_2${P(p-tolyl)$_3$}$_2$Ir]-3,9-μ-(H)$_2$-7,8-$C_2B_9H_{10}$.

By

10 James A. Doi, Raymond G. Teller[redacted]d M. Frederick Hawthorne*

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Reaction of $[Ir(cyclo\text{-}octa\text{-}1,5\text{-}diene)(P(p\text{-}tolyl)_3)_2][\underline{nido}\text{-}7,8\text{-}C_2B_9H_{12}]$ with Hydrogen; Synthesis and X-ray Structure of $3,9\text{-}\{(H)_2(P(p\text{-}tolyl)_3)_2Ir\}\text{-}3,9\text{-}\mu\text{-}(H)_2\text{-}7,8\text{-}C_2B_9H_{10}$

By

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SUMMARY

Treatment of the ion pair, $[\mathrm{Ir(cod)(PR_3)_2}]$[*nido*-7,8-$\mathrm{C_2B_9H_{12}}$] (cod=cyclo-octa-1,5-diene; R=phenyl, p-tolyl) with hydrogen gave *closo*-3,3-$\mathrm{(PR_3)_2}$-3-H-3,1,2-$\mathrm{IrC_2B_9H_{11}}$ and the novel *nido*-metallocarborane, 3,9-{cis-$\mathrm{(H)_2}$-trans-$\mathrm{(PR_3)_2}$-Ir}-3,9-μ-$\mathrm{(H)_2}$-*nido*-7,8-$\mathrm{C_2B_9H_{10}}$.

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During our continuing investigation of the chemistry of the known alkene hydrogenation and isomerization catalyst; closo-3-3-$(PPh_3)_2$-3-H-3,1,2-$RhC_2B_9H_{11}$, we undertook the preparation of the iridium congener; closo-3-3-$(PPh_3)_2$3-H-3,1,2-$IrC_2B_9H_{11}$ (Ia). Preparation of Ia is conveniently effected by treatment of $[Ir(cod)(PPh_3)_2]$[nido-7,8-$C_2B_9H_{12}$] with 1 atm. of hydrogen in cyclohexane heated to reflux. Characterization of Ia via nmr, infrared and elemental analysis was unequivocal.[1]

When the same reaction is carried out at -22°C in pentane, a mixture of products is formed. In addition to Ia, a pale yellow crystalline complex (IIa) is found in 40% yield. Infrared, ^1H, ^{31}P and ^{11}B nmr and microanalysis are consistent with the formulation; $(H)_2(PPh_3)_2Ir(C_2B_9H_{12})$. In particular, the ^1H nmr displays an envelope of broad signals between τ 13 ppm and τ 18 ppm, assigned to bridging Ir-H-B and B-H-B protons and high field signals at τ 28.0 and τ 28.5 ppm which are assigned to terminal metal hydrides. In addition, the infrared spectrum shows absorptions consistent with bridging Ir-H-B groups (ν 1965vw cm^{-1}) and terminal Ir-H groups (ν 2249w cm^{-1}).

Since a myriad of linkages between the transition metal and carborane cluster is possible, an X-ray structure analysis of the related $(H)_2(P(p\text{-tolyl})_3)_2Ir(C_2B_9H_{12})$ (IIb) was carried out.

Crystal Data: $Ir[P(C_7H_7)_3]_2(H)_2(C_2B_9H_{12})\cdot C_7H_8$ M = 1028.5, triclinic, space group $P\bar{1}$, a = 12.292(4), b = 12.854(3), c = 16.169(4)Å, $\alpha = 78.41(1)^1$, $\beta = 94.88(1)^\circ$, $\gamma = 102.04(1)^\circ$, Z = 2, ρ(calc 119K) = 1.40 g/cm^3, ρ(obs) = 1.32 g/cm^3, μ = (MoKα) = 31.3 cm^{-1}. Data were collected on a Picker FACS-I automated diffractometer at -154°C. The structure was solved by standard Patterson-Fourier techniques. Full matrix least squares refinements of all atoms[2] (including the hydrogens bridging the Ir-B bonds) has converged to an agreement factor of 0.044.[3]

The molecule is illustrated in Figure 1, with a summation of bond distances and angles. The crystal structure formally consists of a discrete cis-$[(H)_2[(p\text{-tolyl})_3P]_2Ir]^+$ fragment bonded to two terminal B-H bonds of a $[C_2B_9H_{12}]^{-1}$ anion.

Figure 1

The crystal displays an unusual disorder featuring the carborane cluster. Packing within the crystal is apparently determined by the tri-p-tolyl-phosphine ligands, leaving the carborane cluster free to adopt 1 of 2 different orientations in a 60:40 ratio. Each orientation of the carborane cluster however is constrained so that the Ir atom is bound to the same two B-H bonds; additionally the carbon atom positions within the carborane are also constrained to be identical in the two orientations. Consequently, the two species present in the crystal are chemically equivalent.

An especially noteworthy feature of the complex is the mode of interaction of the $[(H)_2L_2Ir]^+$ fragment with the $[C_2B_9H_{12}]^-$ moiety. The iridium atom interacts with the B(3)-H(3) and B(9)-H(9) bonds thereby completing the octahedral coordination about the metal atom. Exopolyhedral bonding of a transition metal to a borane cluster has been previously observed in $[(Ph_3P)_2Cu]_2B_{10}H_{10}\cdot CHCl_3$,[4] (Cu-H = 1.97 (6) Å, Cu-B = 2.30(1) Å).

Comparison of Ir-B and Ir-H distances in IIb with those in $Ir(B_5H_8)(CO)(PPh_3)_2$ (Ir-B (H bridged) = 2.250(6) Å, Ir-H = 1.73 Å),[5] a nido-pentagonal pyramid metalloborane, indicate a weaker Ir-carborane interaction in IIb (Ir-B = 2.46(1) Å, Ir-H = 1.94(8) Å). Note that this Ir-H bond length is also longer than the Ir-H bridging bond lengths found in the recently reported

$[(C_5Me_5)_2Ir_2(\mu\text{-}H)_3]BF_4$ (Ir-H 1.75(1) Å, neutron diffraction).[6]

Although the terminal Ir-H hydride ligands of IIb were not located in the structure analysis the geometry about the Ir atom is clearly that of a distorted octahedron with the symmetric hydrides occupying the two cis "vacant" positions.

Three center M-H-B (M = transition metal) bonding of exopolyhedral B-H units is seen in a variety of metallocarborane complexes.[7] However, all of these systems involve closo carboranyl or metallocarboranyl moieties. Previous to this work, the only M-H-B bonding of exopolyhedral B-H units of a nido carboranyl moiety were found in the main group compounds; 7,8-μ-dialkylalumino-1,2-dicarba-nido-undecaborane (13) (alkyl = CH_3, C_2H_5) and its gallium analogues[8,9] in which the M-H-B bonds can be viewed as formally donating two electrons each to the aluminum or gallium to satisfy its "octet". The same formalism allows IIa to be seen as a stable eighteen electron organometallic compound.

Figure 2

Heating the nido compounds IIa or IIb, in benzene, gave the thermodynamically favored closo compounds Ia and its tri-p-tolylphosphine analogue Ib (both in 90% yield). This behavior is analogous to that of the aluminum and gallium nido-carborane systems.[8]

As previously noted, heating $[Ir(cod)(PPh_3)_2][nido\text{-}7,8\text{-}C_2B_9H_{12}]$ in cyclohexane in the presence of hydrogen gave primarily the closo compound Ia. However, trace amounts of the nido compound IIa could also be isolated under those reaction conditions. Heating the $[Ir(cod)(PR_3)_2][nido\text{-}7,8\text{-}C_2B_9H_{12}]$ (R=phenyl and p-tolyl) compounds in cyclohexane without hydrogen, gave only the closo compounds Ia and Ib, with no trace of the nido compounds.

Acknowledgements

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2. The p-tolyl rings were refined as rigid groups. After the location and refinement of all atoms except the four hydrogens bound to the Ir atom and the two partially occupied carborane hydrogens a difference-Fourier snythesis was calculated. The two tallest peaks in the map were attributed to the bridging hydrogen atoms. Their final refined positions are less than two standard deviations away from their Fourier map positions. The only other significant peak in the map in the vicinity of the Ir atom was in the proper position for a terminal hydride atom. Since the second hydride atom was not located (even in maps of decreasing $\sin\theta/\tau$ cutoff) this atom was not included in the final cycles of refinement. The successful refinement of these bridging hydrogen atoms is due at least in part to their distance from the metal atom.

3. The crystal used for data collection was approximately equidimensional (0.10mm x 0.12mm x 0.14mm). An analytical absorption correction was made, the minimum and maximum absorption coefficients were 0.7462 and 0.8407 respectively. Structure factors were weighted by $1/\sigma^2$ where σ is the standard deviation of F. In the final cycle of least squares no shift was larger than one-tenth of its corresponding estimated standard deviation.

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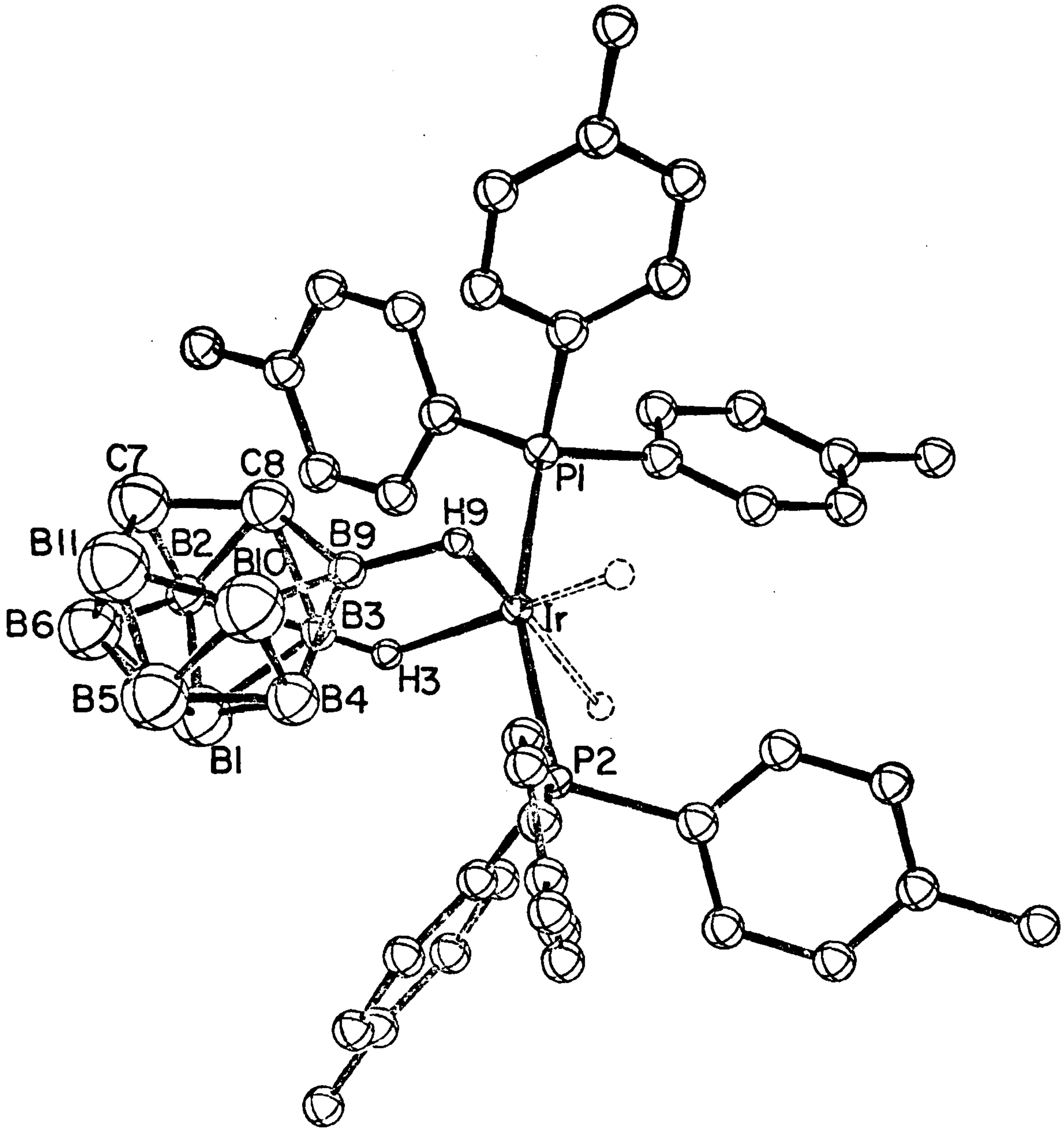
9. a) The cage numbering system in this paper is consistent with that favored by IUPAC for nido carboranes, and is different from the numbering scheme in ref. 6.

   b) Pure Appl. Chem.; 30, 683 (1972).

Figure 1 The molecular structure of $3,9\text{-}\{(H)_2(P(p\text{-tolyl})_3)_2Ir\}\text{-}3,9\text{-}\mu\text{-}(H)_2\text{-}7,8\text{-}C_2B_9H_{10}$. Some important bond lengths are: Ir-P(1), 2.312(3); Ir-P(2), 2.301(3); Ir-H(3); 1.85(7); Ir-H(9), 2.02(10); Ir-B(3), 2.480(11); Ir-B(9), 2.452(11); B(3)-H(3), 1.4(1); B(9)-H(9), 1.3(1) Å; bond angles: P(1)-Ir-P(2), 158.9(1); P(1)-Ir-H(3), 102(2); P(1)-Ir-H(9), 94(3); P(2)-Ir-H(3), 94(2); P(2)-Ir-H(9), 95(3); H(3)-Ir-H(9), 104(4); B(3)-Ir-B(9), 40.8(4); Ir-H(3)-B(3), 98(4); Ir-H(9)-B(9), 94(6)°.



C7
C8
B11
B2
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H9
P1
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Ir
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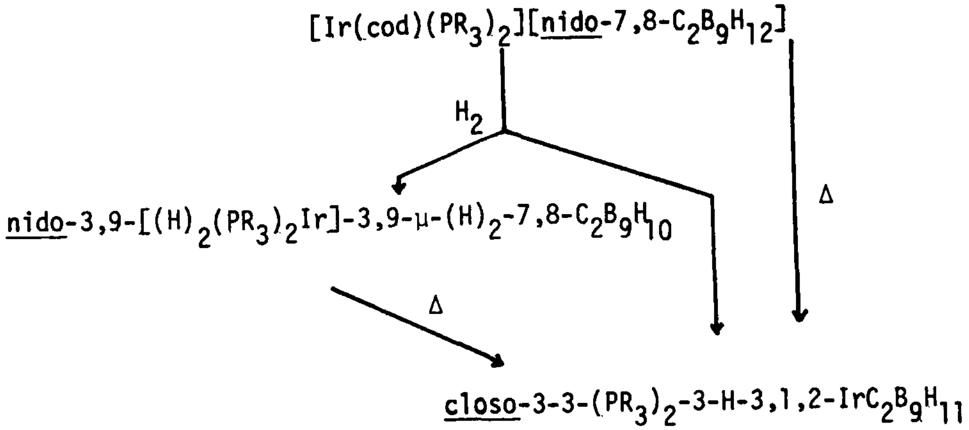
B1
P2



Cod = cyclo-octa-1,5-diene

a) R = Ph

b) R = p-tolyl

Figure 2

Some Distances (in Å) and Angles (in degrees) in 3,9-$\{(H)_2(P(p\text{-tolyl})_3)_2Ir\}$-3,9-μ-$(H)_2$-7,8-$C_2B_9H_{10}$.

Distances about the Ir and P atoms

Ir-P(1)	2.312(3)	P(1)-Group 1	1.807
Ir-P(2)	2.301(3)	P(1)-Group 2	1.828
Ir-H(3)	1.85(7)	P(1)-Group 3	1.829
Ir-H(9)	2.02(10)	P(2)-Group 4	1.831
Ir-B(3)	2.480(11)	P(2)-Group 5	1.814
Ir-B(9)	2.452(11)	P(2)-Group 6	1.859

Distances within the carborane fragment[a]

B(1)-B(2)	1.56(3)	B(5)-B(6)	1.68(3)
B(1)-B(3)	1.73(2)	B(5)-B(10)	1.70(3)
B(1)-B(4)	1.72(2)	B(5)-B(11)	1.71(3)
B(1)-B(5)	1.81(3)	B(6)-C(7)	1.70(2)
B(1)-B(6)	1.79(3)	B(6)-B(11)	1.77(3)
B(2)-B(3)	1.77(2)	C(7)-C(8)	1.58(1)
B(2)-B(6)	1.60(3)	C(7)-B(11)	1.67(2)
B(2)-C(7)	1.73(3)	C(7)-B(2)'	1.71(3)
B(2)-C(8)	1.73(3)	C(8)-B(9)	1.72(2)
B(3)-B(4)	1.71(2)	C(8)-B(2)'	1.73(3)
B(3)-C(8)	1.69(2)	B(9)-B(10)	1.78(2)
B(3)-B(9)	1.72(2)	B(9)-B(2)'	1.76(2)
B(4)-B(5)	1.67(2)	B(10)-B(11)	1.75(2)
B(4)-B(9)	1.72(2)	B(10)-B(2)'	1.69(3)
B(4)-B(10)	1.72(2)	B(11)-B(2)'	1.63(3)
B(1)-H(1)	1.3(2)	C(7)-H(7)	1.0(3)
B(3)-H(3)	1.4(1)	C(8)-H(8)	1.2(1)
B(4)-H(4)	1.2(2)	B(9)-H(9)	1.3(1)
B(5)-H(5)	0.9(1)	B(10)-H(10)	1.2(1)
B(6)-H(6)	1.0(1)	B(11)-H(11)	1.0(2)

Angles

P(1)-Ir-P(2)	158.9(1)	H(3)-Ir-H(9)	104(4)
P(1)-Ir-H(3)	102(2)	B(3)-Ir-B(9)	40.8(4)
P(1)-Ir-H(9)	94(3)	Ir-H(3)-B(3)	98(4)
P(2)-Ir-H(3)	94(2)	Ir-H(9)-B(9)	94(6)
P(2)-Ir-H(9)	95(3)		

a) A numbering system consistent with that of figure 1 is used. B(2) and B(2)' are one half weighted atoms.

1. Positional (in fractional coordinates) and Thermal Parameters[a] for the Nongroup Atoms of 3,9-$\{(H)_2(P(p\text{-tolyl})_3)_2Ir\}$-3,9-μ-$(H)_2$-7,8-$C_2B_9H_{10}$.

Atom[b]	x	y	z	B(Å^2)
Ir	.17641(3)	.40184(3)	.32659(3)	c
P(1)	.2165(2)	.5270(2)	.2034(2)	c
P(2)	.1783(2)	.3285(2)	.4686(2)	c
B(1)[B(10)]	.0291(16)	.1891(15)	.1765(13)	5.5(4)
B(2)[---]	-.0043(19)	.1140(18)	.2624(15)	1.6
B(3)[B(9)]	.0743(9)	.2457(9)	.2646(7)	1.7(2)
B(4)[B(4)]	.0101(12)	.3170(12)	.1805(9)	3.2(3)
B(5)[B(5)]	-.0985(16)	.2369(16)	.1418(14)	5.2(4)
B(6)[B(11)]	-.1074(17)	.1088(18)	.1950(13)	5.6(4)
C(7)[C(7)]	-.1344(12)	.1169(12)	.2944(9)	4.4(3)
C(8)[C(8)]	-.0311(10)	.1924(10)	.3309(8)	3.3(2)
B(9)[B(3)]	-.0144(9)	.3259(9)	.2820(7)	1.5(2)
B(10)[B(1)]	-.1223(15)	.3244(15)	.2016(11)	4.5(4)
B(11)[B(6)]	-.1974(15)	.1910(14)	.2155(11)	4.5(4)
---[B(2)]	-.1489(19)	.2455(18)	.2972(15)	1.6
H(1)[H(10)]	.081(9)	.175(9)	.111(7)	4.(3)
H(3)[H(9)]	.184(6)	.273(6)	.293(5)	1.(2)
H(4)[H(4)]	.060(12)	.391(12)	.131(10)	8.(4)
H(5)[H(5)]	-.110(9)	.267(9)	.090(7)	2.(3)
H(6)[H(11)]	-.119(8)	.032(9)	.211(6)	2.(2)
H(7)[H(7)]	-.196(21)	.060(20)	.320(16)	13.(8)
H(8)[H(8)]	-.010(7)	.159(6)	.403(5)	1.(2)
H(9)[H(3)]	.012(9)	.405(9)	.318(7)	3.(3)
H(10)[H(1)]	-.160(8)	.396(8)	.165(6)	2.(2)
H(11)[H(6)]	-.277(18)	.168(17)	.217(13)	16.(7)

2. Anisotropic Temperature Factors[d]

Atom	$10^5\beta_{11}$	$10^5\beta_{22}$	$10^5\beta_{33}$	$10^5\beta_{12}$	$10^5\beta_{13}$	$10^5\beta_{23}$
Ir	228(3)	206(3)	82(2)	34(2)	23(2)	-14(1)
P(1)	277(20)	213(18)	144(11)	47(15)	64(12)	-17(11)
P(2)	294(20)	266(18)	97(11)	36(15)	15(12)	-41(11)

3. Rigid Group Parameters[e]

Group	x	y	z	Phi	Theta	Rho	B(Å^2)
Ptolyl 1	.2529(5)	.2154(4)	.5000(4)	2.436(3)	2.614(4)	2.783(3)	2.14(7)
Ptolyl 2	.0446(4)	.2833(5)	.5189(4)	-2.642(3)	-2.561(4)	0.504(4)	2.28(9)
Ptolyl 3	.2516(4)	.4290(4)	.5336(4)	1.917(5)	-2.382(3)	0.925(4)	1.87(7)
Ptolyl 4	.1013(4)	.5966(4)	.1689(4)	-3.138(7)	2.042(4)	-.586(6)	2.07(7)
Ptolyl 5	.2633(5)	.4906(4)	.1101(3)	0.234(4)	2.585(3)	-1.122(3)	1.84(7)
Ptolyl 6	.3347(4)	.6376(4)	.2172(4)	-2.351(3)	-2.556(4)	3.128(3)	2.00(7)
Ptolyl 7[f]	.4837(8)	.1341(13)	.1055(9)	.54(2)	-1.95(1)	3.18(2)	2.8(2)
Ptolyl 8[f]	.4679(13)	.1412(17)	.1539(13)	-1.80(2)	2.13(1)	-.75(2)	5.0(3)

a) Estimated standard deviations in the least significant figures are given in parentheses.

b) Alternate atomic designations for the carborane cage atoms are given in brackets. Note that B(2) is a partially weighted atom.

c) Anisotropic thermal parameters.

d) The form of the anisotropic thermal ellipsoidal is:

$$\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}\ell^2 + 2\beta_{12}hk + 2\beta_{13}h\ell + 2\beta_{23}k\ell)]$$

e) x, y, and z are the fractional coordinates of the origin of the rigid group and Phi, theta, and rho the rotation angles (in radians)

f) One half weighted groups.

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Treatment of the ion pair, [Ir(cod)(PR$_3$)$_2$][*nido*-7,8-$C_2B_9H_{12}$] (cod=cyclo-octa-1,5-diene; R=phenyl, p-tolyl) with hydrogen gave *closo*-3,3-(PR$_3$)$_2$-3-H-3,1,2-$IrC_2B_9H_{11}$ and the novel *nido*-metallocarborane,3,9-{*cis*-(H)$_2$-*trans*-(PR$_3$)$_2$-Ir}-3,9-μ-(H)$_2$-*nido*-7,8-$C_2B_9H_{10}$.

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