

POLYHEDRAL IRIDAMONOCARBABORANE CHEMISTRY\*.  
THE ISOLATION AND CHARACTERISATION OF THE NINE-VERTEX  
*closo*-TYPE SPECIES  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$  AND THE PARTIALLY OPEN  
STRUCTURES OF FORMALLY *closo* NINE-VERTEX CLUSTERS

Bohumil ŠTÍBR<sup>a</sup>, John D. KENNEDY<sup>b</sup>, Mark THORNTON-PETT<sup>b</sup>, Eva DRDÁKOVÁ<sup>a</sup>,  
Tomáš JELÍNEK<sup>a</sup> and Jaromír PLEŠEK<sup>a</sup>

<sup>a</sup>Institute of Inorganic Chemistry,

Czechoslovak Academy of Sciences, 25068 Řež u Prahy, Czechoslovakia

<sup>b</sup>School of Chemistry, University of Leeds, Leeds LS2 9JT, England

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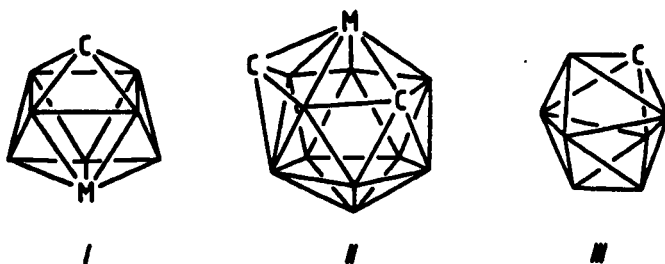
Reaction of  $[\text{Ir}(\text{CO})\text{Cl}(\text{PPh}_3)_2]$  with the  $[\textit{closo}\text{-CB}_7\text{H}_8]^-$  anion in dichloromethane solution at room temperature results in oxidative insertion into the cluster to give the novel nine-vertex *closo*-type species  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$  in 87% yield as a pale-yellow air-stable solid. Crystals (from hexane-benzene) are orthorhombic, space group  $P2_12_12_1$ , with  $a = 1056.7(1)$ ,  $b = 1597.6(2)$ ,  $c = 2150.5(2)$  pm, and  $Z = 4$ . The crystal and molecular structure was refined to  $R (R_w) = 0.0244 (0.0250)$  for 3 338 observed reflections with  $F_0 > 4.0\sigma(F_0)$ . The cluster structure is based on a tricapped trigonal prismatic arrangement of the  $\{\text{IrCB}_7\}$  atoms, with the iridium atom at a five-connected cluster-vertex position, and the carbon at a non-adjacent four-connected capping site. In this formally *closo* cluster, the distance between the two boron atoms that are bound to both iridium and carbon atoms is markedly long at 219.0(14) pm, an instance of a generally unrecognized phenomenon which is briefly discussed in the context of other partially opened, though formally *closo*, nine- ten- and eleven-vertex boron-containing cluster compounds.

Although dicarbaborane chemistry is well represented in the literature, polyhedral monocarbaborane chemistry is much less well examined. For example, the only previously reported nine-vertex *closo*-metallamonocarbaboranes<sup>1,2</sup> are two monoanionic species  $[(\text{C}_5\text{H}_5)\text{CoCB}_7\text{H}_8]^-$  and  $[(\text{C}_5\text{H}_5)\text{CoCMeB}_7\text{H}_7]^-$ . These have the schematic cluster configuration *I* that is based on a tricapped trigonal prismatic arrangement of the  $\{\text{CoCB}_7\}$  cluster atoms.

These two compounds were formed in an interesting though quasi-serendipitous manner by the degradation by base of  $[(\text{C}_5\text{H}_5)\text{CoC}_2\text{B}_{10}\text{H}_{12}]^-$  and  $[(\text{C}_5\text{H}_5)\text{CoC}_2\text{MeB}_{10}\text{H}_{11}]^-$  (both of schematic cluster structure *II*), respectively<sup>1,2</sup>. We now report the preparation

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and characterisation of the first non-ionic example of this basic compound type, the neutral species  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$ . This we have prepared by a direct route that involves the straightforward redox penetration reaction of the  $[\textit{closo-1-CB}_7\text{H}_8]^-$  anion (a fluxional species of presumed ground-state configuration *III*) (ref.<sup>3</sup>) by the formal  $\{\text{Ir}(\text{CO})(\text{PPh}_3)_2\}^+$  moiety. The cluster structure of the new compound reveals a partial cluster opening that is apparently characteristic of many other formally *closo* nine-vertex clusters, a phenomenon of which the generality has not previously been noted.



## RESULTS AND DISCUSSION

Reaction between  $[\text{Ir}(\text{CO})\text{Cl}(\text{PPh}_3)_2]$  and the  $[\textit{closo-1-CB}_7\text{H}_8]^-$  anion in dichloromethane solution at room temperature, followed by filtration and recrystallisation, resulted in the isolation of a yellow crystalline solid identified as  $[\textit{7,7,7-(CO)(PPh}_3)_2\text{-closo-7,1-IrCB}_7\text{H}_8]$ . The yield was good (87%; reaction scale ca 90  $\mu\text{mol}$ ), the stoichiometry being as in Eq. (A):



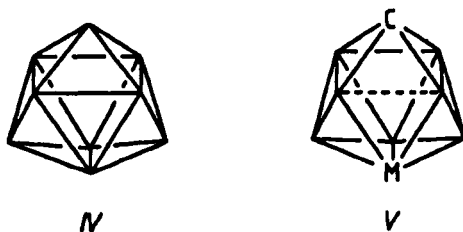
The compound is air-stable in its pure crystalline form, although it is less stable in solution. The molecular structure of the molecule has been determined by single-crystal X-ray diffraction methods and is shown in Fig. 1. Interatomic distances and angles are in Tables I and II. The cluster structure of the molecule is seen to be based on a closed tricapped trigonal prismatic arrangement of the nine  $\{\text{IrCB}_7\}$  cluster atoms, with the carbon atom occupying a low-coordinate capping position (of cluster connectivity four) and the iridium a non-adjacent higher coordinate position (of cluster connectivity five). This is in accord with general observations that carbon atoms will tend to occupy sites of lower connectivity, and metal atoms sites of higher connectivity, in metallacarborane clusters<sup>4</sup>. The positioning of these cluster constituents is the same as that in the previously reported<sup>1,2</sup> anionic analogue  $[(\text{C}_3\text{H}_5)\text{CoCB}_7\text{H}_8]^-$ .

The overall similarities between the latter ion and the neutral iridium congener reported here are also confirmed by the similarities of the measured <sup>11</sup>B NMR shielding patterns (Fig. 2 and Table III), a phenomenon which is often taken to indicate electronic similarities. The <sup>11</sup>B and <sup>1</sup>H-<sup>11</sup>B(selective)} NMR spectra of the iridium complex

each reveal a 2 : 2 : 2 : 1 pattern of resonances associated with the cluster {BH} groups, while the corresponding  $^{31}\text{P}$ -{ $^1\text{H}$ (complete)} NMR spectrum exhibits only one singlet. These NMR data are consistent with a solution structure in which a time-average mirror plane intersects the C(1), B(9), and Ir(7) cluster vertices.

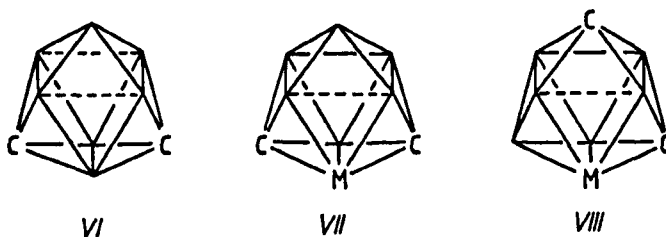
The compound can be regarded initially as being in accord with the Williams–Wade cluster-geometry<sup>4</sup> and electron-counting<sup>5</sup> formalism. In this approach, the {CH} unit and the {Ir(CO)(PPh<sub>3</sub>)<sub>2</sub>} octahedral iridium(III) centre each formally contribute three orbitals and three electrons to the cluster bonding scheme. The compound is thus formally equivalent to the *closo* binary borane anion [B<sub>9</sub>H<sub>9</sub>]<sup>2-</sup>, which exhibits<sup>6</sup> the model tricapped trigonal prismatic structural motif (structure IV). In metal-ligand terms the compound is formally an octahedral eighteen-electron iridium(III) complex, and in this approach there will be predominantly a conventional three-orbital metal-to-cluster bonding interaction. The preparative reaction (Eq. (A) above) can thereby be regarded as an oxidative insertion of iridium(I) into the cluster, generating an effective tridentate  $\eta^5$ -[*nido*-CB<sub>7</sub>H<sub>8</sub>]<sup>3-</sup> ligand by reduction of the [*closo*-CB<sub>7</sub>H<sub>8</sub>]<sup>-</sup> starting substrate.

Within the terms of the formally closed, tricapped trigonal prismatic, geometry (structure IV), however, the long distance of 219.0(14) pm between B(3) and B(4), flanking the C(1) cap and adjacent to iridium (structure V), is very noteworthy, because this distance must represent only a very weak bonding interaction at best. A similar lengthening, but not nearly so severe [202.5(14) pm], occurs in the previously mentioned cobalt<sup>2</sup> analogue [(C<sub>5</sub>H<sub>5</sub>)CoCB<sub>7</sub>H<sub>8</sub>]<sup>-</sup>, and in this context it is now interesting that a survey of known, formally closed, nine-vertex structures in fact reveals that all these previously reported heteroatom boron-containing species also exhibit a noticeable weakening of at least one interboron linkage.

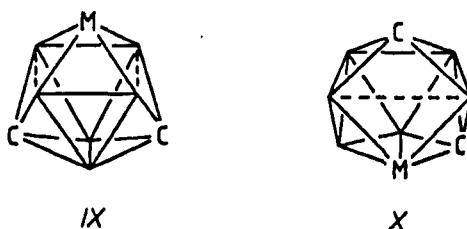


Thus, although in the unsubstituted parent model binary anion [B<sub>9</sub>H<sub>9</sub>]<sup>2-</sup> (structure IV) the interboron distances flanking the lower-connectivity caps are at the higher end of typical<sup>7</sup> intercluster ranges at 185.0(15) to 193.0(15) pm (ref.<sup>6</sup>), they are nevertheless within bonding range. By contrast, all nine-vertex formally *closo* heteroboranes structurally examined so far have at least one such interboron distance at least as large as the arbitrary value of 200 pm that can be chosen as a weak-bonding limit. Even in non-hetero *closo* nine-vertex nine-boron clusters it is apparent that any asymmetry induces

such lengthening, and [1,5-(SMe<sub>2</sub>)-*closo*-B<sub>9</sub>H<sub>7</sub>] has two such linkages over 200 pm, at 203(1) and 202(1) pm (ref.<sup>8</sup>).

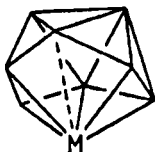


The dicarbaborane [1,6-Me<sub>2</sub>-*closo*-1,6-C<sub>2</sub>B<sub>7</sub>H<sub>7</sub>] (structure VI) similarly exhibits a lengthening of two such linkages, to 200(1) and 201(1) pm (ref.<sup>9</sup>), and the two metalladiborane species [(CO)<sub>3</sub>MnC<sub>2</sub>B<sub>6</sub>H<sub>8</sub>]<sup>-</sup> and [(PEt<sub>3</sub>)<sub>2</sub>HCoC<sub>2</sub>B<sub>6</sub>H<sub>8</sub>] (structures VII and VIII, respectively) exhibit lengthenings to 212.2(7) and 203(1) pm, respectively<sup>10,11</sup>. In these last two compounds the lengthening is of the linkage that divides the (five-connected) metal vertex and the non-adjacent four-connected vertex, as also observed in the new iridium compound reported in this work (structure V above).



In this context should also be mentioned the platinum compounds [(PMe<sub>3</sub>)<sub>2</sub>PtC<sub>2</sub>B<sub>6</sub>H<sub>8</sub>] and [(PMe<sub>3</sub>)<sub>2</sub>PtC<sub>2</sub>Me<sub>2</sub>B<sub>6</sub>H<sub>6</sub>] (ref.<sup>12</sup>, schematic structure IX), in each of which the metal atom (uniquely in examples examined so far) occupies a position of cluster connectivity four. Here the interboron lengthening, to 224(8) and 195(2) pm, respectively, now occurs for the linkages dividing the platinum atom from the two four-connected carbon vertices. However, in this case there will be substantial *d*<sup>8</sup> platinum(II) square-planar character, and consequently strong contributions from a two-orbital metal-cluster interaction, so different cluster bonding criteria may well apply (see, for example, refs<sup>13-16</sup>); in this last regard, it is relevant that [(PEt<sub>3</sub>)<sub>2</sub>PtC<sub>2</sub>Me<sub>2</sub>B<sub>6</sub>H<sub>6</sub>] (ref.<sup>17</sup>) exhibits a definitive cluster "slippage" (refs<sup>18-20</sup>), resulting in a four-membered open face that has an interboron cross-face distance of 241(5) pm (schematic structure X). A complete diamond-square-diamond process involving the face in question results in the so-called *isocloso* cluster structure XI, exhibited by [(PMe<sub>3</sub>)<sub>2</sub>HIrB<sub>8</sub>H<sub>7</sub>Cl] (ref.<sup>21</sup>), in which the interboron distance is now 305(2) pm, but again different metal-to-cluster bonding criteria, in this case possibly involving *d*<sup>4</sup>

iridium(V) "capped octahedral" bonding character and a concomitant predominantly four-orbital metal-cluster interaction<sup>22,23</sup> are believed to apply.



XI

The ubiquity of this cluster-opening phenomenon (structures *V* – *VIII*) in most of the structurally characterised formally *closo* nine-vertex boron-containing species is, as far as we are aware, not generally recognized, although O'Neill and Wade have considered electronic factors that may influence relative interatomic distances in *closo* nine-vertex systems, and have noted the long linkage<sup>9</sup> in [1,6-Me<sub>2</sub>-*closo*-1,6-C<sub>2</sub>B<sub>7</sub>H<sub>7</sub>] in this context<sup>24</sup>. Very recently we have also noted cluster-opening behaviour in eleven-vertex

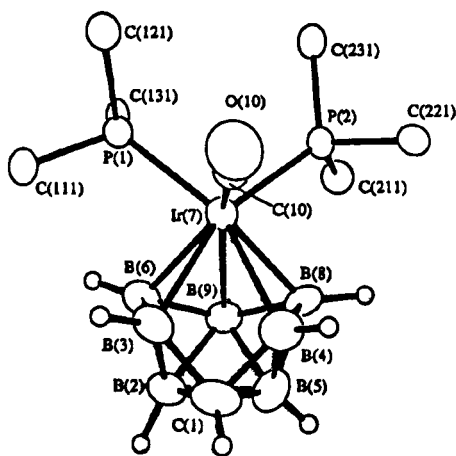


FIG. 1.

ORTEP-type diagram of the crystallographically determined molecular structure of [(CO)(PPh<sub>3</sub>)<sub>2</sub>IrCB<sub>7</sub>H<sub>8</sub>] with selected P-organyl group atoms omitted for clarity

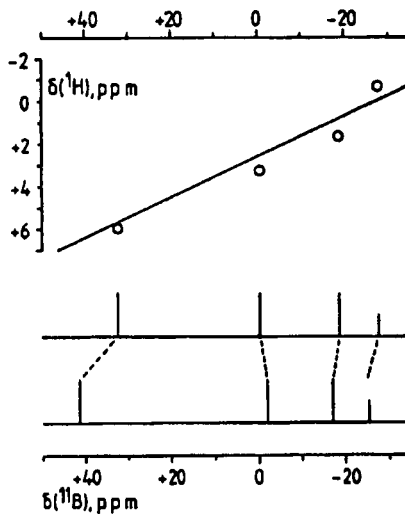


FIG. 2

Lower diagrams: Stick representations of the chemical shifts and relative intensities in the <sup>11</sup>B NMR spectra of [(CO)(PPh<sub>3</sub>)<sub>2</sub>IrCB<sub>7</sub>H<sub>8</sub>] and the [(C<sub>5</sub>H<sub>5</sub>)CoCB<sub>7</sub>H<sub>8</sub>]<sup>-</sup> anion. Upper diagram [same scale in δ(<sup>11</sup>B)]: plot of δ(<sup>11</sup>B) versus δ(<sup>1</sup>H) for {B11} units of [(CO)(PPh<sub>3</sub>)<sub>2</sub>IrCB<sub>7</sub>H<sub>8</sub>]; the line drawn has slope δ(<sup>11</sup>B):δ(<sup>1</sup>H) of 10.5, intercept +2.66 ppm in δ(<sup>1</sup>H)

*closo*-type species<sup>16,25 - 27</sup> and there are recent indications that it may be prevalent in ten-vertex chemistry as well<sup>28 - 29</sup>. There are probably several factors that can contribute to this cluster-opening, including electron-pair localisation among heteroatoms or at bridging atoms, changes in metal oxidation state, heteroatom-to-cluster bonding interactions that may involve other than three orbitals, larger cluster atoms that force apart already weak interboron linkages, etc. In [1,6-Me<sub>2</sub>-*closo*-1,6-C<sub>2</sub>B<sub>7</sub>H<sub>7</sub>] the lengthening has been attributed to a draining of electron density towards the carbon atoms<sup>24</sup> but it is not clear whether similar factors would be dominant for the much larger distortion observed in the iridacarborane reported here.

As more experimental data become available within nine, ten, and eleven-vertex areas (and possibly also within the eight-vertex area)<sup>30</sup>, it is becoming apparent that the geometric and electronic components that underpin the simple Wade-Williams formalism may in many cases be critically dependent upon variable combinations of the factors mentioned above. It would also seem that balance of the components can readily

TABLE I  
Selected interatomic distances (pm) for [(CO)(PPh<sub>3</sub>)<sub>2</sub>IrCB<sub>7</sub>H<sub>8</sub>], with estimated standard deviations (e.s.d.s) in parentheses

Atoms	Distance	Atoms	Distance
P(1)-Ir(7)	242.1(4)	P(2)-Ir(7)	240.8(4)
C(10)-Ir(7)	187.0(9)	B(3)-Ir(7)	237.9(11)
B(4)-Ir(7)	244.8(11)	B(6)-Ir(7)	218.9(11)
B(8)-Ir(7)	217.9(10)	B(9)-Ir(7)	235.0(9)
C(111)-P(1)	182.1(6)	C(211)-P(2)	181.7(6)
C(121)-P(1)	185.6(6)	C(221)-P(2)	182.4(5)
C(131)-P(1)	182.7(6)	C(231)-P(2)	184.0(6)
O(10)-C(10)	113.7(9)		
B(2)-C(1)	159.7(16)	B(3)-C(1)	161.7(15)
B(4)-C(1)	158.2(15)	B(5)-C(1)	154.5(16)
B(3)-B(2)	176.2(16)	B(5)-B(2)	187.1(15)
B(6)-B(2)	166.1(15)	B(9)-B(2)	190.0(15)
B(6)-B(3)	170.9(16)	B(5)-B(4)	172.4(18)
B(8)-B(4)	173.9(15)	B(8)-B(5)	166.5(16)
B(9)-B(5)	190.4(16)	B(9)-B(6)	171.1(14)
B(9)-B(8)	172.8(13)		
H(1)-C(1)	99.9	H(2)-B(2)	113.3
H(3)-B(3)	113.3	H(4)-B(4)	105.7
H(5)-B(5)	100.4	H(6)-B(6)	114.7
H(8)-B(8)	120.9	H(9)-B(9)	122.9

be upset by even quite subtle changes in cluster constituents and substituents. A more extensive investigation of these areas is therefore necessary so that, with a greater experimental base and concomitant theoretical work, these various factors can be better delineated and insights as to the electronic origins of the observed effects can be developed. We hope to direct some of our future work into these areas.

TABLE II  
Selected angles ( $^{\circ}$ ) between interatomic vectors for  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$ , with estimated standard deviations (e.s.d.s) in parentheses

Atoms	Angle	Atoms	Angle
P(2)-Ir(7)-P(1)	100.0(2)	C(10)-Ir(7)-P(1)	94.4(3)
C(10)-Ir(7)-P(2)	96.7(3)	B(3)-Ir(7)-P(1)	100.3(3)
B(3)-Ir(7)-P(2)	158.9(2)	B(3)-Ir(7)-C(10)	87.5(4)
B(4)-Ir(7)-P(1)	153.7(2)	B(4)-Ir(7)-P(2)	106.3(3)
B(4)-Ir(7)-C(10)	80.5(4)	B(4)-Ir(7)-B(3)	53.9(4)
B(6)-Ir(7)-P(1)	79.1(4)	B(6)-Ir(7)-P(2)	136.5(3)
B(6)-Ir(7)-C(10)	126.8(4)	B(6)-Ir(7)-B(3)	43.7(4)
B(6)-Ir(7)-B(4)	83.4(5)	B(8)-Ir(7)-P(1)	150.8(2)
B(6)-Ir(7)-P(2)	76.1(3)	B(8)-Ir(7)-C(10)	114.8(4)
B(8)-Ir(7)-B(3)	83.4(4)	B(8)-Ir(7)-B(4)	43.7(3)
B(8)-Ir(7)-B(6)	84.3(4)	B(9)-Ir(7)-P(1)	109.5(3)
B(9)-Ir(7)-P(2)	98.9(3)	B(9)-Ir(7)-C(10)	148.6(3)
B(9)-Ir(7)-B(3)	68.7(4)	B(9)-Ir(7)-B(4)	69.0(4)
B(9)-Ir(7)-B(6)	44.1(3)	B(9)-Ir(7)-B(8)	44.7(3)
O(10)-C(10)-Ir(7)	170.8(6)		
B(3)-C(1)-B(2)	66.5(7)	B(4)-C(1)-B(2)	116.1(8)
B(4)-C(1)-B(3)	86.4(7)	B(5)-C(1)-B(2)	73.1(7)
B(5)-C(1)-B(3)	113.4(8)	B(5)-C(1)-B(4)	66.9(8)
C(1)-B(3)-Ir(7)	105.4(6)	B(2)-B(3)-Ir(7)	100.4(6)
B(2)-B(3)-C(1)	56.2(7)	B(6)-B(3)-Ir(7)	62.2(5)
B(6)-B(3)-C(1)	107.2(8)	B(6)-B(3)-B(2)	57.2(7)
C(1)-B(4)-Ir(7)	103.6(6)	B(5)-B(4)-Ir(7)	97.8(6)
B(5)-B(4)-C(1)	55.5(7)	B(8)-B(4)-Ir(7)	59.9(5)
B(8)-B(4)-C(1)	106.4(8)	B(8)-B(4)-B(5)	57.5(7)

## EXPERIMENTAL

## General

$[\text{Ir}(\text{CO})\text{Cl}(\text{PPh}_3)_2]$  and  $[\text{NHMe}_3]^+[\text{closo-1-CB}_7\text{H}_8]^-$  were made by literature methods<sup>3,31</sup>. NMR spectroscopy was performed as described in other recent papers from our laboratories<sup>32,33</sup>, with chemical shifts  $\delta$  being given in ppm to high frequency (low field) of  $\Xi = 32.083971$  MHz (nominally  $\text{F}_3\text{BOEt}_2$  in  $\text{CDCl}_3$ ) for  $^{11}\text{B}$  (quoted  $\pm 0.5$  ppm),  $\Xi = 40.480730$  MHz (nominally 85%  $\text{H}_3\text{PO}_4$ ) for  $^{31}\text{P}$  (quoted  $\pm 0.5$  ppm), and  $\Xi = 100$  MHz for  $^1\text{H}$  (quoted  $\pm 0.05$  ppm),  $\Xi$  being defined as in ref.<sup>34</sup>. Spectra were calibrated for  $\delta$  using solvent resonances as internal secondary standards.

*Preparation of  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$ .*  $[\text{Ir}(\text{CO})\text{Cl}(\text{PPh}_3)_2]$  (71.7 mg; 91.8  $\mu\text{mol}$ ) and  $[\text{NHMe}_3]^+[\text{1-CB}_7\text{H}_8]^-$  (46 mg; 245  $\mu\text{mol}$ ) were stirred in dichloromethane (30  $\text{cm}^3$ ) for 24 h at ambient temperature. The mixture was then filtered through silica gel (TLC grade; ca 5 g), and the yellow filtrate reduced in volume to ca 15  $\text{cm}^3$  in vacuo. The solution was overlaid with hexane (15  $\text{cm}^3$ ) and left to crystallise for 12 h. The mother liquors were removed, and the orange crystals dried in vacuo to give the first crop of pure  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$  (54 mg; 70% based on Ir starting complex);  $R_F$  (silica gel;  $\text{CHCl}_3$ ) 0.63. A second crop (13.2 mg; 17%) (total yield 87%) was obtained by HPLC from the mother liquors [250  $\times$  16 mm column (Knauer); Lichrosorb C Si60 7  $\mu\text{m}$  silica gel; separation efficiency ca 4 200 theoretical plates] using  $\text{CH}_2\text{Cl}_2$ -pentane (35:65 v/v) as the mobile phase with UV detection at  $\lambda = 254$  nm. Under these conditions the compound had an HPLC capacity factor  $k'$  1.71 ( $k'$  calculated as in ref.<sup>35</sup>). The NMR data are summarized in Table III.

## Single-Crystal X-Ray Diffraction Analysis

All measurements were carried out on a Nicolet P3/F diffractometer using an  $\omega/2\theta$  scan mode and graphite-monochromated molybdenum  $K_{\alpha}$  radiation ( $\lambda = 71.069$  pm) following a standard procedure<sup>36</sup>. The data set was corrected for absorption empirically<sup>37</sup>. The structure was solved by standard heavy-atom and Fourier difference techniques and was refined by full-matrix least-squares using the SHELX program system<sup>38</sup>. All non-hydrogen atoms were refined anisotropically with phenyl groups treated as rigid bodies with idealised hexagonal

TABLE III  
Measured NMR parameters (ppm) for  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$ , together with those reported<sup>a</sup> for the  $[(\text{C}_5\text{H}_5)\text{CoCB}_7\text{H}_8]^-$  anion (in acetone) for comparison

$[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]^b$ in $\text{CDCl}_3$ at 294 – 297 K			$[(\text{C}_5\text{H}_5)\text{CoCB}_7\text{H}_8]^-$
$\delta(^{11}\text{B})$	$\delta(^1\text{H})$	relative intensity	$\delta(^{11}\text{B})$
+32.3	+5.91	2	+41.1
-0.6	+3.25	2	-2.2
-18.8	+1.66	2	-17.3
-27.2	-0.84	1	-25.2
[CH]	+2.34	1	[CH] <sup>c</sup>

<sup>a</sup> Data from ref.<sup>1</sup>; <sup>b</sup>  $\delta(^{31}\text{P}) + 4.7$  ppm (+6.3 ppm at 218 K); <sup>c</sup>  $\delta(^1\text{H}) + 2.76$  (pyridine solution of  $\text{Cs}^+$  salt).



TABLE IV

Non-hydrogen and cluster-hydrogen fractional atomic coordinates ( $\cdot 10^4$ ) for  $[(\text{CO})(\text{PPh}_3)_2\text{IrCB}_7\text{H}_8]$ , with e.s.d.s in parentheses

Atom	x	y	z	$U_{\text{iso}}/U_{\text{eq}}^a$
Ir(7)	3724.2(2)	4905.8(1)	1158.7(1)	33.2(1)
P(1)	2765(2)	3652(1)	1580(1)	31.6(5)
P(2)	2056(2)	5899(1)	1354(1)	30.1(5)
C(111)	3660(5)	2688(2)	1481(2)	42(2)
C(112)	3104(5)	1944(2)	1278(2)	51(3)
C(113)	3828(5)	1217(2)	1233(2)	67(3)
C(114)	5108(5)	1232(2)	1391(2)	80(4)
C(115)	5665(5)	1976(2)	1594(2)	78(4)
C(116)	4940(5)	2703(2)	1639(2)	57(3)
C(121)	2538(5)	3552(3)	2433(2)	35(2)
C(122)	2783(5)	4214(3)	2838(2)	55(3)
C(123)	2648(5)	4102(3)	3478(2)	72(4)
C(124)	2268(5)	3327(3)	3712(2)	62(3)
C(125)	2023(5)	2665(3)	3307(2)	52(3)
C(126)	2158(5)	2778(3)	2667(2)	48(3)
C(131)	1204(3)	3400(3)	1267(2)	37(2)
C(132)	1092(3)	3203(3)	637(2)	51(3)
C(133)	-97(3)	3040(3)	383(2)	63(3)
C(134)	-1173(3)	3074(3)	757(2)	68(3)
C(135)	-1061(3)	3217(3)	1387(2)	73(4)
C(136)	128(3)	3434(3)	1642(2)	52(3)
C(211)	1002(4)	6032(3)	694(2)	35(2)
C(212)	438(4)	5306(3)	463(2)	47(3)
C(213)	-416(4)	5356(3)	-30(2)	59(3)
C(214)	-706(4)	6131(3)	-293(2)	70(3)
C(215)	-141(4)	6857(3)	-62(2)	69(4)
C(216)	713	6807(3)	431(2)	54(3)
C(221)	2543	6954(2)	1571(2)	38(2)
C(222)	3818	7170(2)	1621(2)	46(2)
C(223)	4159	7979(2)	1799(2)	58(3)
C(224)	3226	8572(2)	1927(2)	60(3)
C(225)	1951	8356(2)	1877(2)	63(3)
C(226)	1609	7547(2)	1699(2)	52(3)
C(231)	942	5729(3)	1999(2)	38(2)
C(232)	-307	5471(3)	1910(2)	53(3)
C(233)	-1117	5391(3)	2419(2)	71(4)
C(234)	-680	5570(3)	3016(2)	72(4)
C(235)	568	5827(3)	3106(2)	76(4)

TABLE IV  
(Continued)

Atom	x	y	z	$U_{iso}/U_{eq}^a$
C(236)	1379	5907(3)	2597(2)	60(3)
C(10)	4665(7)	5135(5)	1875(3)	43(2)
O(10)	5375(6)	5257(4)	2262(3)	70
C(1)	6133(8)	5042(6)	253(4)	67
B(2)	5225(11)	4375(6)	-75(5)	56
B(3)	5603(9)	4333(6)	722(5)	53
B(4)	5466(9)	5700(6)	694(5)	53
B(5)	5075(12)	5542(6)	-76(6)	64
B(6)	4187(10)	3996(6)	432(4)	51
B(8)	3933(8)	5822(5)	415(4)	40
B(9)	3615(8)	4870(5)	68(3)	44
H(1)	7038	5201	190	70
H(2)	5599	4084	-519	70
H(3)	6192	3869	996	70
H(4)	5879	6223	916	70
H(5)	5012	5953	-427	70
H(6)	3711	3352	402	70
H(8)	3419	6487	323	70
H(9)	2609	4781	-211	70

<sup>a</sup>  $U_{eq}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

symmetry (C-C = 139.5 pm). All phenyl hydrogen atoms were included in calculated positions (C-H = 96 pm) and were assigned to an overall isotropic thermal parameter. The cluster hydrogen atoms were located on a Fourier difference synthesis but, since they tended to shift to unreasonable positions during refinement, they were assigned to a fixed overall isotropic thermal parameter and their positional parameters were not refined. The weighting scheme  $w = [\sigma^2(F_o) + 0.0004 (F_o)^2]^{-1}$  was used. Final atomic coordinates are in Table IV.

*Crystal data for [(CO)(PPh<sub>3</sub>)<sub>2</sub>IrCB<sub>7</sub>H<sub>8</sub>].* C<sub>38</sub>H<sub>40</sub>B<sub>7</sub>IrOP<sub>2</sub>,  $M = 842.6$ ,  $a = 1056.7(1)$ ,  $b = 1597.6(2)$ ,  $c = 2150.5(2)$  pm,  $U = 3.6301(7)$  nm<sup>-3</sup>, orthorhombic, space group  $P2_12_12_1$ ,  $Z = 4$ ,  $D_x = 1.54$ ,  $\mu = 36.33$  cm<sup>-1</sup>, and  $F(000) = 1675.81$ .

*Data collection.* Scan speeds 2.0 – 29.3° min<sup>-1</sup>, scan widths 2.0° +  $\alpha$ -doublet splitting,  $4.0 < 2\theta < 50.0^\circ$ ,  $T = 290$  K.

*Structure refinement.* Number of data collected 3 712, number observed  $\{ (F > 4.0\sigma(F)) \} = 3\ 338$ ,  $R = 0.0244$ ,  $wR = 0.0250$ , number of parameters 372.

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