Polyhedral Organoiridaborane Chemistry. The Reaction of MeNC with [6-(η^5-C_5Me_5)-*nido*-6-IrB_9H_{13}]. Nuclear Magnetic Resonance Studies and the Crystal and Molecular Structure of [4,4-(η^5-C_5Me_5)(MeNC)-*arachno*-4-IrB_8H_{12}] *

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The reaction between MeNC and [6-(η^5-C_5Me_5)-*nido*-6-IrB_9H_{13}] results in the formation of two new yellow air-stable *arachno* metallaboranes, the ten-vertex compound [6-(η^5-C_5Me_5)-6-(*endo*-MeNC)-*arachno*-6-IrB_9H_{11}-9-(*exo*-MeNC)], and the nine-vertex compound [4-(η^5-C_5Me_5)-4-(*endo*-MeNC)-*arachno*-4-IrB_8H_{12}]. Both new species are characterized by multielement and multiple resonance n.m.r. spectroscopy and the nine-vertex species also by single-crystal *X*-ray diffraction analysis. Crystals were monoclinic, space group $P2_1/n$, with a = 1 392.0(2), b = 1 010.6(2), c = 1 480.8(2) pm, β = 113.01(1)°, and Z = 4. The structure refined to R = 0.0210, R' = 0.0212. The *endo*-type hydrogen atoms on B(6) and B(8) were located and shown to have partial bridging character to B(7).

MacKinnon and co-workers[1] have recently reported the reaction between the *nido* ten-vertex rhodaborane [6-(η^5-C_5Me_5)-*nido*-6-RhB_9H_{13}] and methyl isocyanide to give the *arachno* ten-vertex nine-boron species [6-(η^5-C_5Me_5)-6,9-$(MeNC)_2$-*arachno*-6-RhB_9H_{11}] [equation (1)]. Here we report

$$[(C_5Me_5)RhB_9H_{13}] + 2MeNC \longrightarrow [(C_5Me_5)(MeNC)RhB_9H_{11}(MeNC)] + H_2 \quad (1)$$

that the analogous reaction with the iridaborane congener [6-(η^5-C_5Me_5)-*nido*-6-IrB_9H_{13}] (**1**)[2] not only yields the corresponding *arachno* ten-vertex product [6-(η^5-C_5Me_5)-6,9-$(MeNC)_2$-*arachno*-6-IrB_9H_{11}] (**2**) but also the nine-vertex eight-boron species [4-(η^5-C_5Me_5)-4-(MeNC)-*arachno*-4-IrB_8H_{12}] (**4**) which we have characterized by single-crystal *X*-ray diffraction analysis. The numbering schemes used are as in Figures 1 and 2.

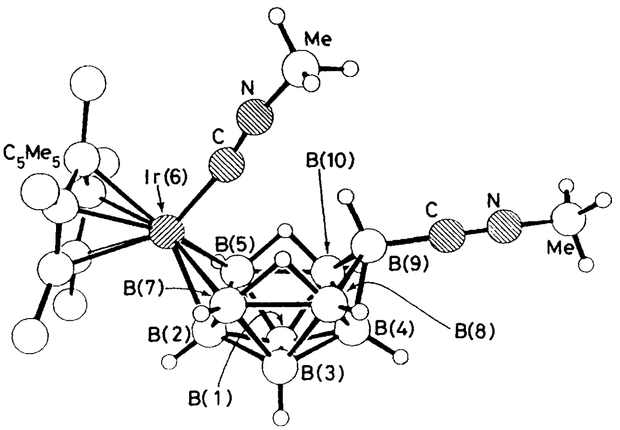

Figure 1. Representation of the proposed molecular structure of [6-(η^5-C_5Me_5)-6-(MeNC)-*arachno*-6-IrB_9H_{11}-9-(MeNC)] (**2**)

Results and Discussion

Reaction between an excess of MeNC and [6-(η^5-C_5Me_5)-*nido*-6-IrB_9H_{13}] (**1**) in dichloromethane solution for *ca.* 3 h, followed by chromatographic separation, yielded two air-stable metallaborane products, compounds (**2**) and (**3**). Their measured n.m.r. data are summarized in Table 1, which also contains data for the recently reported[1] rhodium species [6-(η^5-C_5Me_5)-6,9-$(MeNC)_2$-*arachno*-6-RhB_9H_{11}] for comparison. The extreme similarity between the ^{11}B and ^1H data for compound (**1**) and the rhodaborane, together with the identity of [^{11}B–^{11}B]-COSY correlation patterns (not presented in the Table), readily identify compound (**2**) as the iridium analogue of the rhodaborane, *i.e.* as [6-(η^5-C_5Me_5)-6,9-$(MeNC)_2$-*arachno*-6-IrB_9H_{11}], of schematic structure as shown in Figure 1.

The principal n.m.r. differences are those for $\delta(^{11}B)$ at the B(2) and B(5,7) positions next to the metal atom, those for the iridaborane being some 6—7 p.p.m. more shielded than for the rhodaborane (compare *nido* species in ref. 2). As with the rhodaborane, the *endo*-9 proton is significantly deshielded by the adjacent *endo*-6-MeNC grouping. There may also be a slight antipodal deshielding of some 0.8 p.p.m. for $^1H(4)$ in compound (**2**) arising from the heavier third-row transition element (iridium *versus* rhodium) at the antipodal (6) position.

The similarity of the n.m.r. parameters for the second product, compound (**3**), with those of the previously reported[3] nine-vertex eight-boron *arachno*-4-iridanonaboranes [4-(CO)-4,4-$(PMe_3)_2$-4-H-*arachno*-4-IrB_8H_{12}] and [4,4,4-$(PMe_3)_3$-4-H-*arachno*-4-IrB_8H_{12}] suggested that compound (**3**) was of similar formulation, with the *endo*-MeNC configuration about the metal centre in compound (**2**) further suggesting that this also obtained for compound (**3**); the latter is then formulated as [4-(η^5-C_5Me_5)-*endo*-4-(MeNC)-*arachno*-4-IrB_8H_{12}]. This formulation was confirmed by the results of a single-crystal *X*-ray diffraction analysis, in which all borane cluster hydrogen atoms were located and refined, a result that was not realisable with the two *arachno*-4-metallanonaboranes previously investigated by single-crystal *X*-ray diffraction techniques, *viz.* [4,4-$(PMe_2Ph)_2$-*arachno*-4-PtB_8H_{12}][4] and [4-(CO)-4,4-$(PMe_3)_2$-4-H-*arachno*-4-IrB_8H_{11}-1-Cl].[3,5]

Crystals were obtained *via* pentane diffusion into a solution of compound (**3**) in chloroform. The crystals were particularly well formed pale yellow prisms which enabled the collection of a

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* *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1989, Issue 1, pp. xvii—xx.

Table 1. Measured ^{11}B and ^{1}H n.m.r. parameters for $[(C_5Me_5)(MeNC)IrB_9H_{11}(MeNC)]$ (**2**), $[(C_5Me_5)(MeNC)RhB_9H_{11}(MeNC)]$ (data from ref. 1), and $[(C_5Me_5)(MeNC)IrB_8H_{12}]$ (**3**) at 294 K

Compound (**2**), $CDCl_3$ solution			$[(C_5Me_5)(MeNC)RhB_9H_{11}(MeNC)]$, CD_2Cl_2 solution		
$\delta(^{11}B)^a$/p.p.m.	$^1J(^{11}B-^1H)^b$/Hz	$\delta(^1H)^{c,d}$	Assignment ^{e}	$\delta(^{11}B)^a$/p.p.m.	$\delta(^1H)^c$
+10.4 (1B)	140	+3.83	(2)	+16.3	+3.36
+8.1 (1B)	130	+3.99	(4)	+8.2	+3.12
−11.7 (2B)	116	+2.00	(5),(7)	−2.1	+2.14
−20.7 (2B)	130	+2.02	(8),10)	−18.2	+1.86
−34.7 (2B)	139	+0.85	(1),(3)	−31.7	+0.66
−39.8 (1B)	117	+0.32	(9)	−37.9	+0.47
—	—^{h}	−3.55	(7,8)(5,10)(bridge)	—	−3.17
—	—^{h}	—	—	—	—

Compound (**3**), $CDCl_3$ solution				
Assignment ^{e}	$\delta(^{11}B)^a$/p.p.m.	Observed $[^{11}B-^{11}B]$ COSY correlations ^{f}	$^1J(^{11}B-^1H)^b$/Hz	$\delta(^1H)^{c,g}$
(1)	+6.8	(5,9)s (2,3)s	137	+3.11
(7)	+20.6	—	—^{h}	+4.46
(5),(9)	−8.5	(1)s (2,3)w	143	+2.28
(6),(8)	−28.6	(2,3)s	151	+2.00
(2),(3)	−34.8	(1)s (5,9)w (6,8)s	144	+0.78
—	—	—	—	—
(5,6),(8,9)(bridge)	—	—	—^{h}	−3.15
(6,7),(7,8)(*endo*)	—	—	—^{h}	−0.96^i

[a] ±0.5 p.p.m. to high frequency (low field) of $BF_3(OEt_2)$. [b] ±8 Hz; from ^{11}B spectra with resolution enhancement. [c] ±0.05 p.p.m. to high frequency (low field) of $SiMe_4$; $\delta(^1H)$ related to directly bound B positions in ^1H-$\{^{11}B\}$ selective experiments. [d] Additional ^1H data: $\delta(C_5Me_5)$ +1.85, δ(MeNC) +3.45(sharper) and +3.41(broader). [e] By $[^{11}B-^{11}B]$-COSY correlations and parallels with other *arachno*-4-metallanonaboranes in refs. 3, 4, and 8. [f] Measured under conditions of complete ^1H decoupling. [g] Additional ^1H data: $\delta(C_5Me_5)$ +1.98, δ(MeNC) +3.36. [h] Not measured due to broadness of signals relative to *J*. [i] Selectively sharpened by $\nu[^1H(6)(8)]$ only in ^1H-$\{^{11}B(selective)\}$ experiments.

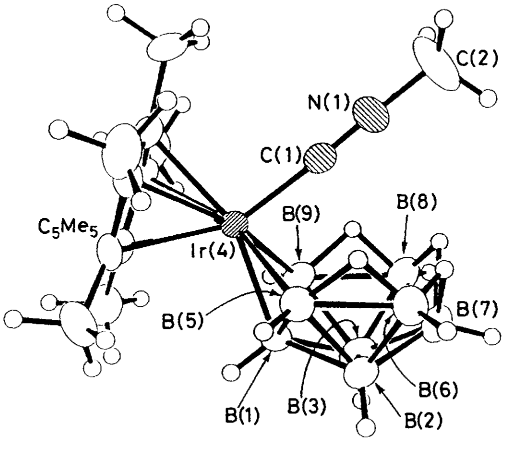


Figure 2. Crystallographically determined molecular structure of $[4-(\eta^5-C_5Me_5)-4-(MeNC)$-*arachno*-$4-IrB_8H_{12}]$ (**3**)

high-quality data set. Consequently, once all the non-hydrogen atoms had been located and refined anisotropically, the borane hydrogen atoms appeared as the highest peaks in the subsequent Fourier difference map. These all refined to the reasonable final positions presented in this paper. Selected interatomic distances and angles are presented in Tables 2 and 3 respectively, and a drawing of the molecular structure is in Figure 2.

The cluster structure is readily seen to be very similar to that previously established for $[(CO)(PMe_3)_2HIrB_8H_{11}Cl]$,[3,5] *viz.* an *arachno* nine-vertex cluster of the *iso*-$B_9H_{15}/B_9H_{14}^-$ family with a metal atom in one of the three outer open-face positions. The iridium-to-boron distances are however somewhat shorter for compound (**3**), compared to the $(CO)(PMe_3)_2Ir$ compound, which may reflect relative *trans* influences, although the comparison is rendered difficult because comparative interatomic distances will be influenced by the inclusion of borane hydrogen atoms in the refinement of compound (**3**) but their exclusion in the final published refinement[3] of $[(CO)(PMe_3)_2HIrB_8H_{11}Cl]$.

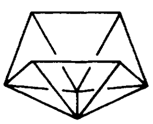
The metal centre in compound (**3**) [and also in compound (**2**)] can be regarded as relatively straightforward 18-electron d^6 'octahedral' iridium(III) with three valence orbitals involved in bonding with the C_5Me_5 moiety, one with the methyl isocyanide, and two with the cluster. In cluster terms the neutral $\{(C_5Me_5)(MeNC)Ir\}$ metal centre contributes two electrons and two orbitals to the cluster bonding proper, just like {BHL} in compounds such as $B_9H_{13}L$, where L = MeCN, Me_2S, *etc.*, and is therefore in accord with the Williams–Wade[6,7] cluster geometry and electron-counting rules, but with the proviso that the two electrons in the *endo* Ir(4)–C(4)(MeNC) bond are included in the cluster-electron *count* (even though they are not involved in the cluster bonding proper) thus notionally taking the place of the two electrons in an *endo*-B–H bond in a binary borane analogue.

The location of the cluster hydrogen atoms in compound (**3**) is of interest because previous crystallographic investigations did not reveal them accurately in $[(CO)(PMe_3)_2HIrB_8H_{11}Cl]$[5] and $[(PMe_2Ph)_2PtB_8H_{12}]$[4] and also because there has been some discussion in the literature concerning the *endo*-terminal '*versus*' pseudo-bridging character of the *endo*-bound hydrogen atoms associated with B(6), B(7), and B(8) in eight-boron and nine-boron structures of this type.[3,4,8–10] Thus for compound (**3**) these well located formally *endo*-terminal hydrogen atoms on B(6) and B(8) are of interest in that they are sufficiently close (*ca.* 160 pm) to B(7) to suggest some bonding interaction and thereby merit the 'pseudo-bridging' descriptor given to *endo*-hydrogen atoms of this type, for example in *arachno*-B_8H_{14}.[9] However, the difference in energetic terms between these two modes is often not great, as evidenced by the ready *endo*/bridging hydrogen-atom fluxionality of the closely related

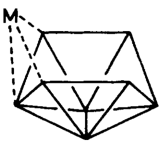
Table 2. Selected interatomic distances (pm) for $[(C_5Me_5)(MeNC)IrB_8H_{12}]$ (**3**) with estimated standard deviations (e.s.d.s) in parentheses *

(*i*) From the iridium atom			
Ir(4)–B(5)	221.6(7)	Ir(4)–B(9)	220.8(7)
Ir(4)–B(1)	216.9(6)	—	—
Ir(4)–C(3)	225.8(6)	Ir(4)–C(6)	225.4(6)
Ir(4)–C(4)	225.5(5)	Ir(4)–C(7)	224.4(6)
Ir(4)–C(5)	224.4(6)	—	—
Ir(4)–C(1)(MeNC)	189.6(6)	—	—
(*ii*) Boron–boron			
B(1)–B(2)	177.4(8)	B(1)–B(3)	174.8(9)
B(1)–B(5)	179.9(9)	B(1)–B(9)	176.9(9)
B(2)–B(3)	176.3(9)	—	—
B(2)–B(5)	181.2(9)	B(3)–B(9)	181.1(9)
B(2)–B(6)	172.8(9)	B(3)–B(8)	173.5(9)
B(2)–B(7)	175.4(9)	B(3)–B(7)	177.5(9)
B(5)–B(6)	186.2(9)	B(8)–B(9)	190.0(9)
B(6)–B(7)	185.5(10)	B(7)–B(8)	185.2(10)
(*iii*) Boron–hydrogen			
B(1)–H(1)	100(4)	—	—
B(2)–H(2)	96(5)	B(7)–H(3)	117(4)
B(5)–H(5)	113(4)	B(9)–H(9)	109(5)
B(5)–H(5,6)(bridge)	130(4)	B(9)–H(8,9)(bridge)	135(5)
B(6)–H(6)(*exo*)	112(4)	B(8)–H(8)(*exo*)	112(5)
B(6)–H(5,6)(bridge)	120(4)	B(8)–H(8,9)(bridge)	126(5)
B(6)–H(6,7)(*endo*)	102(5)	B(8)–H(7,8)(*endo*)	116(5)
B(7)–H(7)	100(5)	—	—
B(7)–H(6,7)(*endo*)	151(5)	B(7)–H(7,8)(*endo*)	169(5)
(*iv*) Other			
C(1)–N(1)	115.5(6)		—
N(1)–C(methyl)	142.5(7)		—
C(aromatic)–C(aromatic)	140.4(7)—144.3(7)		
C(aromatic)–C(methyls)	148.2(7)—152.3(8)		

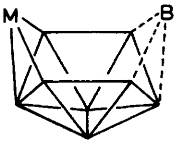
* The two columns contain pairs of dimensions that would be identical if the compound conformed to the mirror-plane symmetry that it approximates to in the solid state and that it exhibits on time-average in solution.



(I)



(II)



(III)

arachno nine-vertex nine-boron species $B_9H_{14}^-$ and $B_9H_{13}L^-$ (where $L^- = NCS^-$, *etc.*).[10,11]

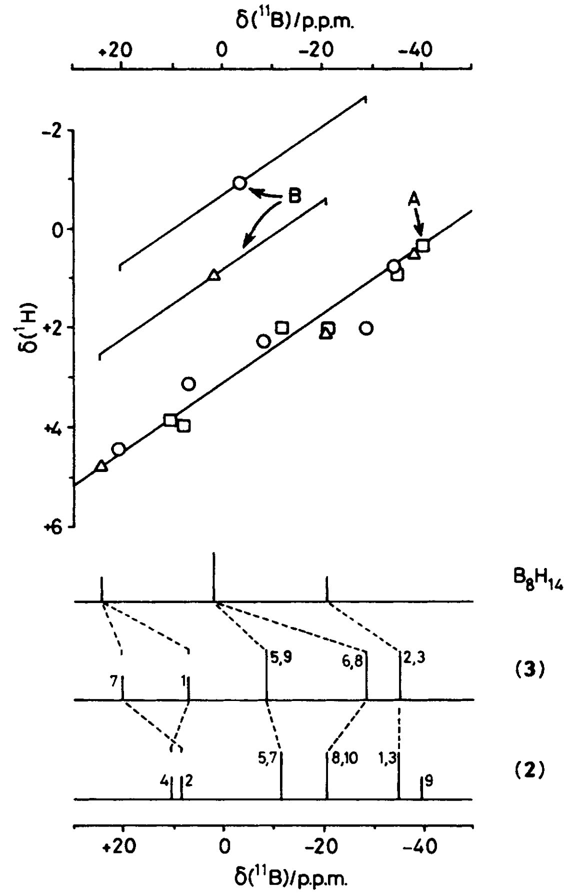


Figure 3. The uppermost diagram shows plots of $\delta(^1H)$ *versus* $\delta(^{11}B)$ for $[(C_5Me_5)(MeNC)IrB_9H_{11}(MeNC)]$ [(**2**), □], $[(C_5Me_5)(MeNC)IrB_8H_{12}]$ [(**3**), ○], and *arachno*-B_8H_{14} (△, data from ref. 9) (with the bridging hydrogen atoms omitted). The line drawn has slope $\delta(^{11}B):\delta(^1H)$ 14.5:1 with intercept +3.1 in $\delta(^1H)$. Point A is that for $(^{11}B,^1H)(9)$ in compound (**2**) (see text), and points B are the *endo*-terminal/pseudo-bridging positions. The lines drawn that are associated with points B show the ^{11}B chemical shift positions of the two boron atoms that are bridged in each case. The lower three diagrams are stick representations of the relative intensities and chemical shifts in the ^{11}B spectra of the above three compounds. Hatched lines connect corresponding positions in the *arachno*-shaped eight-boron fragments in the three species

In gross structural terms compounds (**3**) and (**2**) respectively can be regarded as being derived by the addition of successive metal [schematic structure (**II**)] and boron [schematic structure (**III**)] vertices to a basic *arachno* eight-boron skeleton [schematic structure (**I**)]. The columns in the n.m.r. Table 1 are arranged so that equivalent positions in compounds (**2**) and (**3**) are in equivalent rows, and it can be seen that there is a similarity of chemical shifts for equivalent positions in the two molecules indicating that they have closely related electronic structures. This is better illustrated in Figure 3 which also contains data[9] for the *arachno* eight-boron species B_8H_{14} for comparison. The bottom three diagrams in Figure 3 are stick representations of the intensities and chemical shifts in the ^{11}B spectra of B_8H_{14}, compound (**3**), and compound (**2**), with resonance lines for equivalent positions in the three compounds joined by hatched lines. It can be seen that the basic *arachno* octaboranyl character of the B_8H_{14} molecule is retained throughout the successive addition of metal and boron vertices (**I**) ⟶ (**II**) ⟶ (**III**), which in turn suggests that the binding of these metal and boron vertices mimics to some extent the bonding of the *endo* terminal/pseudo-bridging hydrogen atoms to the cluster; *viz.* they have somewhat stronger interaction with the adjacent open-face boron atoms in (**2**) and (**3**) than they have with the apical B(2) atom. It is however likely that variation in the character of this metal atom can significantly affect this simple picture as discussed elsewhere.[3,4,12,13]

The uppermost diagram in Figure 3 consists of a plot of $\delta(^{11}B)$ *versus* $\delta(^1H)$ for directly bound atoms in compounds (**2**), (**3**), and *arachno*-B_8H_{14} (with the true bridging hydrogen atoms omitted). The *exo*-hydrogen data for all three compounds fall close to the same correlation line, again emphasising the electronic and structural similarities, with the pseudo-bridging hydrogen nuclei in B_8H_{14} and compound (**3**) somewhat more

Table 3. Selected interatomic angles (°) for compound (**3**) with e.s.d.s in parentheses *

(*i*) At the iridium atom			
C(C_5 centroid)–Ir(4)–C(1)	126.1	—	
C(C_5 centroid)–Ir(4)–B(1)	124.3	—	
C(C_5 centroid)–Ir(4)–B(5)	128.9	C(C_5 centroid)–Ir(4)–B(9)	127.6
C(1)–Ir(4)–B(1)	109.6(3)	—	
C(1)–Ir(4)–B(5)	85.6(3)	C(1)–Ir(4)–B(9)	88.4(3)
B(1)–Ir(4)–B(5)	48.4(2)	B(1)–Ir(4)–B(9)	47.7(2)
B(5)–Ir(4)–B(9)	86.2(3)		
(*ii*) Iridium–boron–boron			
Ir(4)–B(1)–B(2)	120.7(4)	Ir(4)–B(1)–B(3)	121.3(4)
Ir(4)–B(1)–B(5)	67.2(3)	Ir(4)–B(1)–B(9)	67.3(3)
Ir(4)–B(5)–B(1)	64.4(3)	Ir(4)–B(9)–B(1)	65.0(3)
Ir(4)–B(5)–B(2)	116.5(4)	Ir(4)–B(9)–B(3)	116.4(4)
Ir(4)–B(5)–B(6)	124.0(4)	Ir(4)–B(9)–B(8)	122.8(4)
(*iii*) Other			
Ir(4)–C(1)–N(1)	177.1(4)	—	
C(1)–N(1)–C(methyl)	174.2(5)	—	
B(5)–H(5,6)–C(methyl)	174.2(5)	—	
B(5)–H(5,6)–B(6)	96(3)	B(9)–H(8,9)–B(8)	93(3)
B(6)–H(6,7)–B(7)	91(4)	B(8)–H(7,8)–B(7)	79(3)
Angle between C_5 plane and B(1)B(5)B(9) plane 9.1			

* As for Table 2.

shielded than the general trend. An interesting feature is that the *endo*-terminal hydrogen atom on B(9) in compound (**2**) is *not* more shielded than the general trend. This is due to the magnetic anisotropy of the Ir(6)-bound MeNC group and the effect has been discussed in more detail in ref. 1.

Experimental

Reaction between nido-[6-(η^5-C_5Me_5)IrB_9H_{13}] (**1**) *and* MeNC.—Dried and degassed solvents were used throughout. The initial reaction was carried out under dry nitrogen, but subsequent manipulations and chromatography were carried out in air. A solution of MeNC[14] (1.7 cm^3 of a 0.432 mol dm^{-3} solution in CH_2Cl_2; 350 μmol) was added to a solution of compound (**1**) (prepared as in ref. 2; 60 mg, 140 μmol) in CH_2Cl_2 (40 cm^3). The resulting solution was stirred for 3.5 h during which time a colour change from orange to yellow occurred. Filtration of the reaction mixture gave a pale yellow solid. This was treated with CH_2Cl_2–hexane (60:40; 10 cm^3), yielding a pale yellow solid and a yellow solution; further filtration and washing with CH_2Cl_2-hexane (60:40) yielded pure compound (**2**), as a pale yellow solid, identified as described in the text (45 mg, 90 μmol; 62%) ($m/e_{max.}$ 520. $^{12}C_{14}{}^1H_{32}{}^{11}B_9{}^{193}Ir^{14}N_2$ requires *m/e* 520). The combined liquid filtrates were evaporated to dryness (rotary evaporator, water-pump pressure, bath temperature ⩽40 °C), redissolved in CH_2Cl_2-hexane (60:40; 10 cm^3), and purified using high-pressure liquid chromatography [CH_2Cl_2–hexane (60:40) as liquid phase, flow rate 10 cm^3 min^{-1}; silica column (Knauer, 250 × 16 mm diameter; Lichrosorb Si60, 7 μm); 5 × 2 cm^3 liquid samples; detection by u.v. absorption at 254 nm] to give pure compound (**3**) as a very pale yellow solid (retention time 13.6 min) (12 mg, 30 μmol; 18%), identified by n.m.r. spectroscopy and single-crystal *X*-ray diffraction analysis. Crystals for the diffraction analysis were grown by diffusion of pentane into a solution of compound (**3**) in chloroform.

N.M.R. Spectroscopy.—N.m.r. spectroscopy was performed at 2.35 and 9.40 T on JEOL FX100 and Bruker AM400 instrumentation, with the general techniques, and also ^1H-{^{11}B}[15] and [^{11}B–^{11}B]-COSY[16,17] experiments, essentially as described and illustrated in other recent papers from our laboratories.[18–20] It may be noted that in the ^1H-{^{11}B} experiments use was made of the technique in which a ^1H-{^{11}B(40 kHz, off-resonance)} spectrum is subtracted from a ^1H-{^{11}B(on-resonance)} spectrum in order to remove resonance lines from protons not coupled to the ^{11}B nuclei of interest.[4,21] Chemical shifts δ are given in p.p.m. to high frequency (low field) of Ξ 100 ($SiMe_4$) for ^1H and Ξ 32.083 971 MHz [nominally $BF_3(OEt_2)$ in $CDCl_3$] for ^{11}B, Ξ being defined as in ref. 22; they were measured using solvent deuteron or residual proton resonances as internal secondary standards.

X-Ray Crystallography.—All crystallographic measurements were made on a Nicolet P3/F diffractometer operating in the ω scan mode using graphite-monochromatised Mo-K_α radiation (λ = 71.069 pm) following a standard procedure described elsewhere.[23] The data set was corrected for absorption empirically once the structure had been determined.[24] The structure was solved *via* standard heavy-atom methods and refined by full-matrix least squares using the SHELX program system.[25] All non-hydrogen atoms were refined with anisotropic thermal parameters. The methyl hydrogen atoms were included in calculated positions (C–H 108 pm) and were assigned an overall isotropic thermal parameter. The borane hydrogen atoms were located in a Fourier difference map and were freely refined with individual isotropic thermal parameters. The weighting scheme $w = [\sigma^2(F_o) + g(F_o)^2]^{-1}$ was used at the end of refinement in which the parameter *g* was included in refinement in order to obtain satisfactory agreement analyses.

Crystal data. $C_{12}H_{30}B_8IrN$, *M* = 467.08, monoclinic, space group $P2_1/n$, a = 1 392.0(2), b = 1 010.6(2), c = 1 480.8(2) pm, β = 113.01(1)°, *U* = 1.9173(4) nm^3, *Z* = 4, μ = 66.89 cm^{-1}, *F*(000) = 904.

Data collection. Scan widths 1.0° + α-doublet splitting, scan speeds 2.0—29.3° min^{-1} and 4.0 < 2θ < 50.0°. Total data collected = 3 408, observed = 3 052 [$I > 2.0\sigma(I)$].

Structure refinement. Number of parameters = 267, weighting factor *g* = 0.0001, *R* = 0.0210, *R'* = 0.0212.

Relevant non-hydrogen and hydrogen atomic co-ordinates are in Table 4.

Additional material available from the Cambridge Crystallo-

Table 4. Fractional atomic co-ordinates (× 10^4) for compound (**3**)

Atom	*x*	*y*	*z*
Ir(4)	10 145.7(1)	257.8(1)	2 416.4(1)
C(1)	8 746(3)	297(4)	1 486(3)
N(1)	7 896(3)	263(4)	916(3)
C(2)	6 817(4)	286(6)	286(4)
C(3)	10 523(3)	−1 809(4)	3 032(3)
C(4)	11 375(3)	−971(4)	3 563(3)
C(5)	11 799(3)	−453(4)	2 898(3)
C(6)	11 205(3)	−989(4)	1 937(3)
C(7)	10 434(3)	−1 809(4)	2 028(3)
C(8)	9 865(4)	−2 609(5)	3 451(4)
C(9)	11 811(4)	−734(6)	4 656(3)
C(10)	12 756(4)	402(5)	3 171(4)
C(11)	11 462(5)	−790(6)	1 065(4)
C(12)	9 646(4)	−2 629(5)	1 216(4)
B(1)	10 546(3)	2 156(5)	3 153(3)
B(2)	9 598(4)	3 093(5)	3 399(4)
B(3)	10 077(4)	3 636(6)	2 523(4)
B(5)	9 543(4)	1 302(5)	3 403(3)
B(6)	8 370(4)	2 388(6)	2 967(4)
B(7)	8 720(4)	3 686(5)	2 263(4)
B(8)	9 245(5)	3 441(5)	1 302(4)
B(9)	10 366(4)	2 242(5)	1 903(4)
H(1)	11 256(30)	2 120(37)	3 694(27)
H(2)	9 799(35)	3 627(47)	3 984(33)
H(3)	10 596(33)	4 584(35)	2 708(27)
H(5)	9 721(29)	890(37)	4 162(27)
H(6)	7 896(26)	2 602(33)	3 408(24)
H(6′)	7 964(40)	2 597(52)	2 239(37)
H(7)	8 225(38)	4 452(44)	2 097(33)
H(8)	9 189(41)	4 149(55)	699(36)
H(8′)	8 386(36)	3 129(46)	1 106(32)
H(9)	10 999(34)	2 285(42)	1 642(30)
H(56)	8 530(30)	1 236(42)	2 919(27)

graphic Data Centre comprises H-atom co-ordinates, thermal parameters, and remaining bond lengths and angles.

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