Journal of Organometallic Chemistry, 390 (1990) 379–387
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands
JOM 20865

Arene–ruthenaborane clusters

I. Products of the reaction between $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_4\}\text{-}RuB_{10}H_8(OEt)_2]$ and carbon monoxide. Nuclear magnetic resonance studies and the crystal structure of $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2]$ *

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(Received November 27th, 1989)

Abstract

Reaction between $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_4\}RuB_{10}H_8(OEt)_2]$ (**1**) and carbon monoxide in dichloromethane gives a 94% yield of the burgundy-coloured complex $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2]$ (**2**) the crystal structure of which has been determined. Each molecule in the unit cell comprises a double cluster composed of a triangular ruthenium cluster and an eleven-vertex "*isocloso*" ruthenaborane sharing a common ruthenium atom vertex; two ruthenium atoms have an $(\eta^6\text{-}C_6Me_6)$ ligand bound to them and are mutually bridged by two carbonyl ligands. Each of these in turn is joined to the ruthenium atom vertex of an *isocloso*-ruthenaundecaborane via a Ru–H–Ru bridge.

Introduction

The metal–borane double cluster compound $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_4\}RuB_{10}H_8(OEt)_2]$ (**1**) has recently been reported [1] as an unexpected product from the reaction of $[2\text{-}(\eta^6\text{-}C_6Me_6)\text{-}2\text{-}Cl\text{-}arachno\text{-}2\text{-}RuB_3H_8]$ and $closo\text{-}[B_{10}H_{10}]^{2-}$ in refluxing ethanol. This triangular metal cluster was the first of its type, and of particular

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* 1-[6′,9′-bis(ethoxy)-*nido*-octahydrodecaborato]-2,3-bis-(η^6-hexamethylbenzene)-di-μ-carbonyl-2,3-di-μ-hydrido-1,2;1,3-*triangulo*-triruthenium.

0022-328X/90/$03.50

interest is the formal electron count of the molecule which differs from those for other types of characterized triruthenium clusters, all of which are carbonyl species and generally contain more cluster electrons. The fair yield of this compound (31% based upon Ru) permits the ready examination of its further chemistry, for example, its reactions with electron donor ligands. We report here the first of these reactions, namely that with carbon monoxide. (The numbering scheme used in this paper is the same as that in Ref. 1.)

Resuls and discussion

The reaction of **1** with carbon monoxide in dichloromethane for 10 min at room temperature, followed by chromatographic separation, resulted in the isolation of a single compound $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt))_2]$ (**2**), in 94% yield (idealized stoichiometry as represented below):

$$\underset{(\mathbf{1})}{\left[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_4\}RuB_{10}H_8(OEt)_2\right]} + 2CO \rightarrow \underset{(\mathbf{2})}{\left[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2\right]} + H_2 \quad (1)$$

The product **2**, an air-stable burgundy-coloured solid, was identified and characterized by NMR and IR spectroscopy, and by a single-crystal X-ray diffraction study. Crystals were grown from CH_2Cl_2/Et_2O by vapour diffusion. The NMR parameters are given in Table 1, together with those previously reported [1,2] for **1** for comparison. The ^{11}B shielding pattern, with two signals at very low field and

Table 1

^{11}B and ^1H NMR data for $[\{(\eta^6\text{-}C_6Me_6)Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2]$ (**2**), along with those for $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_4\}RuB_{10}H_8(OEt)_2]$ (**1**) [1]

Assignment [a]	2 in CD_2Cl_2 at 297 K			1 in CD_3CN at 304 K		
	$\delta(^{11}B)$ (ppm) [b,c]	$(^1J(^{11}B-^1H)$ (Hz))	$\delta(^1H)$ (ppm) [b,d,e]	$\delta(^{11}B)$ (ppm) [b,c]	$(^1J(^{11}B-^1H)$ (Hz))	$\delta(^1H)$ (ppm) [b,d,g]
2,3	(2B) +90.0	(OEt) [f]	(OEt) [f]	(2B) +88.1	(OEt) [h]	(OEt) [h]
4,5,6,7	(4B) +1.9	(140)	(4H) +1.81	(4B) +1.6	(135)	(4H) +2.39
8,9	(2B) +9.0	(135)	(2H) +3.24	(2B) +3.6	(134)	(2H) +3.11
10,11	(2B) +1.9	(140)	(2H) +1.81	(2B) −0.1	(134)	(2H) +1.93

[a] Assignment from relative intensities, $^1H\{^{11}B(\text{selective})\}$, and by comparison with other *isocloso*-1-metallaundecaboranes [2]. [b] Relative intensities in parentheses. [c] ±0.5 ppm to high frequency (low field) of $[BF_3(OEt_2)]$ in $CDCl_3$. [d] ±0.05 ppm to high frequency (low field) of TMS, ^1H resonances related to directly bound B atom positions by selective $^1H\{^{11}B\}$ experiments. [e] $\delta(^1H)$ (C_6Me_6) +2.07 ppm; $\delta(^1H)$ $[Ru_3H_2(CO)_2]$ −15.91 ppm. [f] $\delta(^1H)$ (OEt) +3.99 ppm (quartet) and +1.40 ppm (triplet) $(^3J(^1H-^1H)$ (mean) ca. 7 Hz). [g] $\delta(^1H)$ (C_6Me_6) +2.14 ppm; $\delta(^1H)$ (Ru_3H_4) −13.94 ppm, at lower temperatures this separates into two positions, each of relative intensity (2H), with $\delta(^1H)$ −10.60 and −12.67, ppm at 203 K; coalescence temperature at 270 K at 400 MHz (9.4 T). At extreme low temperatures the higher field resonance (which has a 1/2/1 triplet character, splitting ca. 3.6 Hz) broadens somewhat and shifts slightly $\delta(^1H)$ (CD_2Cl_2) −17.67 ppm. $\omega_{1/2}$ ca. 9 Hz and 203K; $\delta(^1H)$ (CD_2Cl_2) −17.59 ppm and $\omega_{1/2}$ ca. 12.3 Hz at 173 K. [h] $\delta(^1H)$ (OEt) +2.97 ppm (quartet) and +0.90 ppm (triplet) $(^3J(^1H-^1H)$ (mean) ca. 7 Hz).

eight at intermediate field, is diagnostic of the closed eleven-vertex "*isocloso*" type 1-metallaundecaboranes [2]. The similarity of the ^{11}B and ^{1}H NMR data (Table 1) compared with those for **1** clearly shows that **2** is of the same metallaborane structural type. The overall ^{11}B intensity ratio of 2/4/2/2 indicates a time-averaged C_{2v} symmetry. The [^{1}H{selective ^{11}B}] NMR experiments allow the assignment of an exoterminal ^{1}H resonance to all but one of the ^{11}B resonances. This one is the low field resonance of relative intensity two, which is assigned to boron atoms B(2) and B(3), the two ethoxy-substituted borons. The ^{1}H NMR spectrum reveals a proton resonance of relative intensity 2H at −15.91 ppm, which is assigned to two Ru–H–Ru bridging hydrogen atoms, and one resonance of relative intensity 36H at +2.07 ppm, assigned to two hexamethylbenzene ligands. The IR spectrum shows a single band attributable to ν(CO) at 1729 cm^{-1}. This is consistent with either a carbonyl ligand bridging two metal atoms, (expected range 1900–1700 cm^{-1}) [3] or an asymmetric capping carbonyl, similar to that found [4] in $[Ru_6(CO)_{18}]^{2-}$ (ν(CO) 1749 cm^{-1} [5]). The single-crystal X-ray diffraction analysis of **2** revealed the

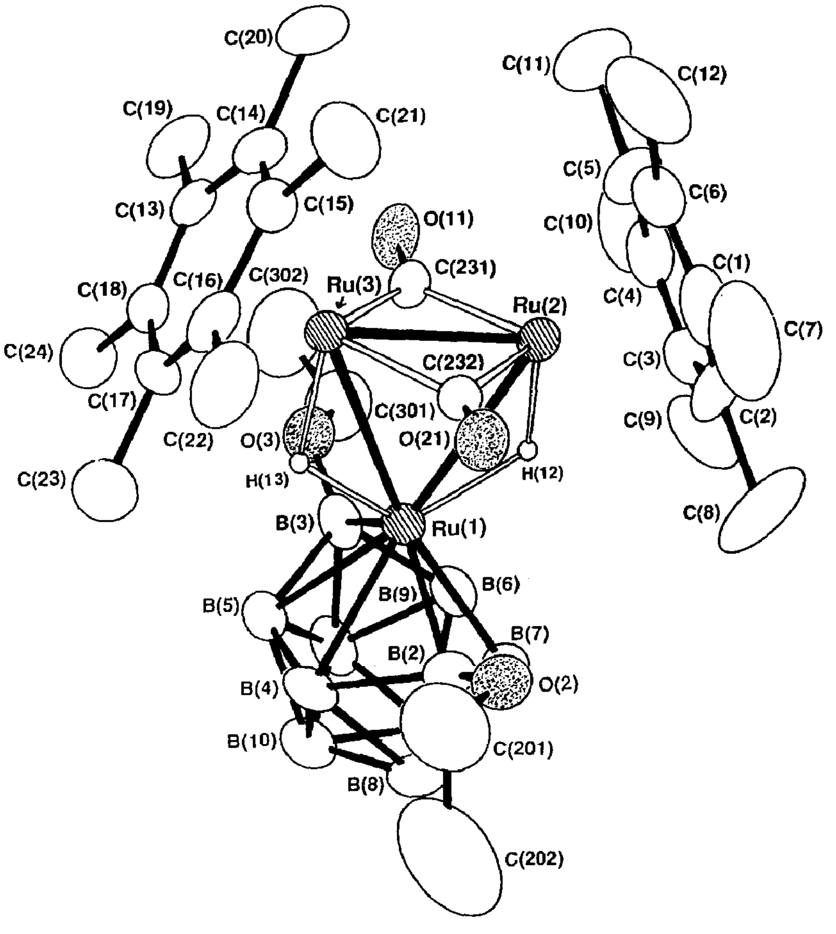

Fig. 1. Structure of $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2]$. Thermal ellipsoids are drawn at the 50% probability level. In this projection B(11) (joined to B(6), B(7), B(8), B(9) and B(10)) is obscured by C(201).

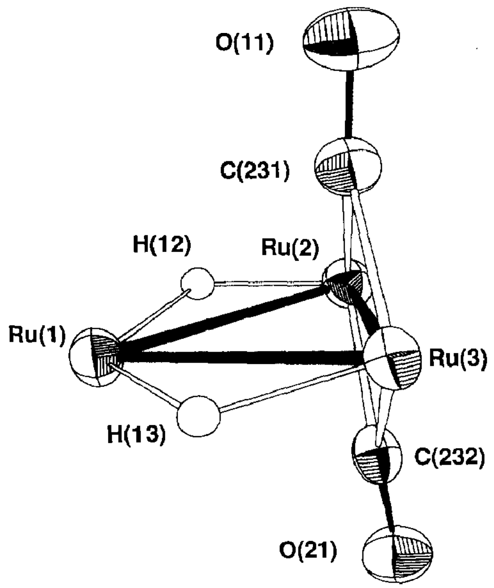

Fig. 2. Diagram showing the binding of carbon monoxide to the Ru_3 triangle in $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2]$ (2). Thermal ellipsoids are drawn at the 50% probability level.

molecular structure shown in Fig. 1 (the $\{Ru_3H_2(CO)_2\}$ subcluster is shown in Fig. 2).

Atomic coordinates, except those of the hexamethylbenzene hydrogens, are given in Table 2. Selected interatomic distances and angles are listed in Tables 3 and 4. The metallaundecaborane subcluster has evidently been retained intact, and the interatomic distances to the Ru(1) metal-atom centre and the boron–boron distances (range 1.686(13)–1.850(12) Å) within the subcluster are normal for this class of compound, and are comparable with those in **1** and other previously reported *isocloso*-metallaundecaboranes of ruthenium [6,7,8,9] and osmium [10]. This subcluster is then bound to two other cojoined ruthenium atoms, Ru(2) and Ru(3), via two Ru–H–Ru bridges. The two hydrides lie on opposite sides, and slightly out of the plane, of the $\{Ru_3\}$ triangle. The dihedral angles between the planes defined by Ru(1)Ru(2)H(12) and Ru(1)Ru(2)Ru(3) and by Ru(1)Ru(3)H(13) and Ru(1)Ru(2) Ru(3) are both about 12°, with H(12) lying to the same side as C(231). The dihedral angle between the planes Ru(1)Ru(2)Ru(3) and Ru(1)B(2)B(3)B(8)B(9) is 12°. These distortions away from the idealized C_{2v} symmetry that is observed in the NMR spectra are probably due to crystal packing forces. The two mutually similar metal atoms Ru(2) and Ru(3) are each bound η^6 to a hexamethylbenzene ligand, and are joined together by two bridging carbonyl ligands. Each of the two hexamethylbenzene ligands shows a slight 'twisted-boat' distortion away from planarity. The associated methyl groups also deviate from the mean planes through each aromatic ring. The displacement is between -0.65 and $+4.08°$ for the methyl groups attached to the arene ring associated with Ru(2), and between -2.64 and $+4.08°$ for those associated with Ru(3). There is no overall gross distortion of the methyl groups from the mean plane of the ring in a direction away from the metal centre, as observed in pentamethylcyclopentadienyl rhodaboranes [11], or towards the metal centre, as observed in polymethylarene cobalta and ferraboranes [12].

Table 2

Atom coordinates ($\times 10^4$) and isotropic temperature factores ($\times 10^2$) for $[\{\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}\text{-}Ru_2B_{10}H_8(OEt)_2]$

Atom	*x*	*y*	*z*	*U*
Ru(1)	4540.1(5)	2459.9(3)	9079.6(2)	
Ru(2)	4560.9(4)	2933.0(3)	7768.5(2)	
Ru(3)	6211.6(4)	1820.8(3)	8270.1(2)	
C(231)	6250(6)	3045(4)	8218(3)	
O(11)	6959(5)	3562(3)	8302(3)	
C(232)	4501(6)	1718(4)	7855(3)	
O(21)	3851(4)	1186(3)	7669(2)	
B(2)	2952(8)	1884(6)	9215(4)	
B(3)	5472(9)	3128(5)	9792(4)	
B(4)	4051(9)	1573(5)	9880(4)	
B(5)	5222(8)	2167(6)	10156(4)	
B(6)	3951(9)	3612(6)	9688(4)	
B(7)	2743(8)	2925(6)	9440(4)	
B(8)	2628(9)	2076(6)	9998(5)	
B(9)	4664(10)	3107(6)	10459(4)	
B(10)	3933(10)	2199(6)	10563(4)	
B(11)	3114(10)	3054(6)	10269(5)	
O(2)	2318(5)	1463(4)	8740(2)	
C(201)	2305(12)	596(8)	8737(5)	
C(202)	1415(15)	261(9)	9075(5)	
O(3)	6541(5)	3461(3)	9711(3)	
C(301)	6808(10)	4290(6)	9826(6)	
C(302)	8035(12)	4464(8)	9858(8)	
C(1)	3280(9)	2870(6)	6830(4)	
C(2)	2735(7)	3328(6)	7289(5)	
C(3)	3371(8)	4022(5)	7561(4)	
C(4)	4492(8)	4228(4)	7369(4)	
C(5)	4981(7)	3733(6)	6938(4)	
C(6)	4369(8)	3059(6)	6663(4)	
C(7)	2498(11)	2177(7)	6542(6)	
C(8)	1520(8)	3131(9)	7518(7)	
C(9)	2849(11)	4558(7)	8041(4)	
C(10)	5171(11)	4968(6)	7621(6)	
C(11)	6178(9)	3937(8)	6700(5)	
C(12)	4836(14)	2546(8)	6148(5)	
C(13)	8304(6)	1750(4)	8337(3)	
C(14)	7796(7)	1596(5)	7694(4)	
C(15)	6985(7)	966(5)	7568(3)	
C(16)	6634(6)	469(4)	8087(4)	
C(17)	7039(6)	683(4)	8714(3)	
C(18)	7904(6)	1323(4)	8834(3)	
C(19)	9236(8)	2401(6)	8481(5)	
C(20)	8229(8)	2086(6)	7153(4)	
C(21)	6504(9)	738(6)	6874(4)	
C(22)	5794(8)	−231(5)	7914(5)	
C(23)	6619(8)	197(5)	9242(4)	
C(24)	8395(8)	1513(7)	9519(4)	
H(12)	4031(65)	3147(41)	8416(33)	4(2)
H(13)	5642(70)	1729(47)	8957(37)	6(2)

Table 3

Selected interatomic distances (Å) for **2**, with estimated standard deviations in parentheses

(a) Ruthenium – ruthenium			
Ru(1)–Ru(2)	2.870(1)	Ru(1)–Ru(3)	2.873(1)
Ru(2)–Ru(3)	2.731(1)		
(b) Ruthenium – carbon			
Ru(2)–C(231)	2.023(6)	Ru(3)–C(231)	2.028(7)
Ru(2)–C(232)	2.020(7)	Ru(3)–C(232)	2.019(6)
Ru(2)–C(1)	2.334(9)	Ru(3)–C(13)	2.333(7)
Ru(2)–C(2)	2.269(8)	Ru(3)–C(14)	2.291(8)
Ru(2)–C(3)	2.253(8)	Ru(3)–C(15)	2.286(8)
Ru(2)–C(4)	2.298(8)	Ru(3)–C(16)	2.326(7)
Ru(2)–C(5)	2.284(9)	Ru(3)–C(17)	2.251(6)
Ru(2)–C(6)	2.320(8)	Ru(3)–C(18)	2.271(6)
(c) Ruthenium – boron			
Ru(1)–B(2)	2.065(9)	Ru(1)–B(5)	2.356(8)
Ru(1)–B(3)	2.049(8)	Ru(1)–B(6)	2.301(9)
Ru(1)–B(4)	2.346(9)	Ru(1)–B(7)	2.359(10)
(d) Ruthenium – hydrogen			
Ru(1)–H(12)	1.84(7)	Ru(2)–H(12)	1.59(7)
Ru(1)–H(13)	1.77(8)	Ru(3)–H(13)	1.65(8)
(e) Boron – boron			
B(2)–B(4)	1.827(11)	B(6)–B(7)	1.696(13)
B(2)–B(7)	1.808(13)	B(6)–B(9)	1.850(12)
B(2)–B(8)	1.757(13)	B(6)–B(11)	1.792(14)
B(3)–B(5)	1.800(12)	B(7)–B(8)	1.845(14)
B(3)–B(6)	1.806(14)	B(7)–B(11)	1.759(13)
B(3)–B(9)	1.754(14)	B(8)–B(10)	1.785(13)
B(4)–B(5)	1.686(13)	B(8)–B(11)	1.779(15)
B(4)–B(8)	1.838(14)	B(9)–B(10)	1.734(15)
B(4)–B(10)	1.787(13)	B(9)–B(11)	1.738(16)
B(5)–B(9)	1.818(14)	B(10)–B(11)	1.758(15)
B(5)–B(10)	1.761(14)		
(f) Others			
B(2)–O(2)	1.350(10)	B(3)–O(3)	1.346(11)
O(2)–C(201)	1.434(14)	O(3)–C(301)	1.418(11)
C(201)–C(202)	1.402(20)	C(301)–C(302)	1.398(18)
C(231)–O(11)	1.165(8)	C(232)–O(21)	1.179(8)

The two bridging carbonyl ligands are both equidistant from Ru(2) and Ru(3) and occupy bridging sites on opposite sides, and almost perpendicular to the Ru(1)Ru(2)Ru(3) plane (the dihedral angles between each of the planes Ru(2)Ru(3)C(231) and Ru(2)Ru(3)C(232), with the plane Ru(1)Ru(2)Ru(3) are ca. 89 and 86°, respectively).

The *closo*-type metallaundecaborane subcluster belongs to a class that is not accommodated by the simple electron counting rules as laid down by Wade [13]. This class has been the subject of some discussion [14–16]. In terms of the "*hypercloso*" view [14] the Ru(1) atom is regarded as a Ru^{II} centre, donating three

Table 4

Selected interatomic angles (°) for **2**, with estimated standard deviations in parentheses.

(a) About the ruthenium atom			
Ru(1)–Ru(2)–Ru(3)	61.6	B(2)–Ru(1)–B(3)	121.4(4)
Ru(2)–Ru(1)–Ru(3)	56.8	B(2)–Ru(1)–B(4)	48.4(3)
Ru(1)–Ru(3)–Ru(2)	61.6	B(2)–Ru(1)–B(5)	87.8(3)
Ru(1)–Ru(2)–C(231)	71.7(2)	B(2)–Ru(1)–B(6)	88.0(4)
Ru(3)–Ru(2)–C(231)	47.7(2)	B(2)–Ru(1)–B(7)	47.7(3)
Ru(1)–Ru(3)–C(231)	71.6(2)	B(3)–Ru(1)–B(4)	87.3(3)
Ru(2)–Ru(3)–C(231)	47.5(2)	B(3)–Ru(1)–B(5)	47.6(3)
Ru(1)–Ru(2)–C(232)	68.8(2)	B(3)–Ru(1)–B(6)	48.7(3)
Ru(3)–Ru(2)–C(232)	47.4(2)	B(3)–Ru(1)–B(7)	88.3(3)
Ru(1)–Ru(3)–C(232)	68.7(2)	B(4)–Ru(1)–B(5)	42.0(3)
Ru(2)–Ru(3)–C(232)	47.5(2)	B(4)–Ru(1)–B(6)	88.1(3)
Ru(1)–Ru(2)–H(12)	36 (2)	B(4)–Ru(1)–B(7)	72.2(3)
Ru(2)–Ru(1)–H(12)	31 (2)	B(5)–Ru(1)–B(6)	72.7(3)
Ru(1)–Ru(3)–H(13)	34 (3)	B(5)–Ru(1)–B(7)	87.2(3)
Ru(3)–Ru(1)–H(13)	32 (3)	B(6)–Ru(1)–B(7)	42.7(3)
(b) About the carbon atom			
Ru(2)–C(231)–Ru(3)	84.8(3)	Ru(2)–C(232)–Ru(3)	85.1(3)
Ru(2)–C(231)–O(11)	136.2(5)	Ru(2)–C(232)–O(21)	137.5(5)
Ru(3)–C(231)–O(11)	137.8(5)	Ru(3)–C(232)–O(21)	136.5(5)
(c) Boron – boron – boron			
B(4)–B(2)–B(7)	99.4(6)	B(3)–B(5)–B(4)	121.3(6)
B(5)–B(3)–B(6)	99.9(6)	B(3)–B(6)–B(7)	122.9(7)
B(2)–B(4)–B(5)	121.9(6)	B(2)–B(7)–B(6)	120.2(7)
(d) Others			
Ru(1)–H(12)–Ru(2)	114(4)	Ru(1)–H(13)–Ru(3)	114(4)
B(2)–O(2)–C(201)	121.5(7)	O(2)–C(201)–C(202)	113.6(1.1)
B(3)–O(3)–C(301)	123.1(7)	O(3)–C(301)–C(302)	113.2(9)

orbitals and two electrons to cluster bonding, thus giving a total of 22 skeletal electrons. This is short of the 24 normally associated with closed 11-vertex geometries, and it is thus termed as being electron *hyper*-deficient. An alternative view is the *isocloso* view of Bould and coworkers [9,16,17], in which the metal centre formally described as Ru^{IV}, donates four orbitals and four electrons to the cluster bonding, thus giving the cluster a total of 24 bonding electrons required for the observed closed 11-vertex geometry.

The total electron count associated with the triangular metal cluster is 46. This is two short of the 48 normally associated with triangular metal clusters [18], and implies at least one sixteen-electron centre. This may be regarded in the same manner as that for the precursor, **1**, with the $\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}$ moiety being considered as a bidentate ligand containing two eighteen electron centres with Ru(2) H(12) and Ru(3) H(13) each forming a two electron bond with Ru(1). This gives Ru(1) an electron count of sixteen, with a bonding orbital description and electron count similar to those for the bisphosphineruthenaundecaborane compound [1,1-$(PPh_3)_2$-2,3-$(OEt)_2$-*isocloso*-1-$RuB_{10}H_8$] [6,8].

Experimental

General

$[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_4\}RuB_{10}H_8(OEt)_2]$ (1) was prepared by as previously described [1]. Carbon monoxide gas was supplied in cylinders from New Zealand Industrial Gases and used without further purification. Preparative thin layer chromatography (TLC) used 1 mm layers of silica (Si60 G, Serva) on plates 20 × 20 cm; these were made in the laboratory from an aqueous slurry, followed by drying in air at ca. 40 °C.

Nuclear magnetic resonance spectroscopy

This was performed at 9.40 Tesla on a Bruker AM400 instrument. The procedure used for $^1H\{^{11}B\}$ spectroscopy has been described elsewhere [19]. Other NMR spectroscopy was straightforward. Chemical shifts $\delta(^1H)$ and $\delta(^{11}B)$ are given in ppm to high frequency (low field) of Ξ100 ($SiMe_4$) and 32.083971 MHz (nominally $BF_3(OEt)_2$ in $CDCl_3$) [2], respectively.

Infrared spectroscopy

Spectra of solutions, with CH_2Cl_2 as solvent, were recorded on a Digilab FTS-40 instrument.

Preparation of $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2]$ (2)

CO was bubbled through a CH_2Cl_2 (10 cm^3) solution of **1** (11.5 mg, 13.6 μmol) for 10 min, the initial claret colour of the solution changing to burgundy. The solution was reduced in volume (rotary evaporator) and subjected to preparative TLC with a $CH_2Cl_2/MeCN$ (9/1) mixture as eluent. One product (R_f 0.76) gave an intense burgundy-coloured TLC band and was identified as described above as $[\{(\eta^6\text{-}C_6Me_6)_2Ru_2H_2(CO)_2\}RuB_{10}H_8(OEt)_2]$ (**2**) (11.4 mg, 12.8 μmol, 94%). Crystals suitable for a single-crystal X-ray diffraction analysis were obtained by vapor diffusion of diethyl ether into a solution of **2** in dichloromethane.

Crystal structure analysis

Prismatic crystals of **2** were assigned to the monoclinic crystal class, space group $P2_1/n$ with a 11.193(2), b 16.535(3), c 21.043(5) Å, β 96.62(2)°, U 3869(2) $Å^3$, $Z = 4$, T 20 °C, D_c 1.53 g cm^{-1}, $F(000) = 1800$, $\mu(\text{Mo-}K_\alpha)$ 89.1 cm^{-1}, crystal size 0.2 × 0.25 × 0.65 mm. Data were collected on a CAD4 diffractometer with Zr-filtered, Mo-K_α radiation (0.71069 Å), and a graphite monochromator. A total of 7068 unique data were measured with the $\omega/2\theta$ scan mode (θ_{max} 25°). The intensities of 3 reflections, monitored throughout data collection, indicated that any decomposition was less than 1%. Empirical absorption corrections were applied [20], maximum and minimum values being 0.9996 and 0.9479 respectively. The structure was solved by direct and Fourier methods and refined by a full-matrix least-squares technique. The final refinement cycle converged to values of 0.042 and 0.044 for R and R_w respectively for the 497 variables and 5207 data for which $F^2 > 3\sigma(F^2)$. The weight, w, is defined as 15.0872 $[\sigma^2(F) + 0.000719F^2]$. Anisotropic thermal motion was assumed for all non-hydrogen atoms. Those hydrogens associated with the hexamethylbenzene groups were assigned to calculated positions (U 0.05 $eÅ^{-2}$); hydrogens associated with the metallaundecaborane moiety and the bridging hydrogens were located from difference electron-density maps and their parameters were refined. Atomic scattering factors were taken from the tabulations in Ref. 21, and anomalous dispersion corrections from those in Ref. 22.

A complete list of bond lengths and angles has been deposited with the Cambridge Crystallographic Data Centre. Lists of thermal and structure factors are available from the authors.

Acknowledgements

We thank the Massey University Research Fund for a post-doctoral Fellowship (MB) and the NZ University Grants Committee for funds for equipment.

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