

# B-frame supported bimetallics. $[(\text{PMe}_2\text{ph})_2\text{PtB}_9\text{H}_{11}\text{Ru}(\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me})]$ and $[(\text{PMe}_2\text{ph})_2\text{PtB}_9\text{H}_9\text{Ru}(\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me})]$ ; an interesting pair of electron-deficient *nido* and *closo* geometries<sup>1</sup>

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## Abstract

$[7,7-(\text{PMe}_2\text{Ph})_2-9-(\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me})-7,9-\text{PtRuB}_9\text{H}_{11}]$  has a formal *closo* Wadlan cluster-electron count, but a *nido* geometry, whereas  $[1-(\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me})-4,4-(\text{PMe}_2\text{Ph})_2-1,4-\text{RuPtB}_9\text{H}_9]$ , which does have a *closo* geometry, has a formal sub-*closo* cluster electron count; both compounds are formed in the reaction between  $[6-(\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me})\text{-nido-6 RuB}_9\text{H}_{13}]$ , KH and  $[\text{PtCl}_2(\text{PMe}_2\text{Ph})_2]$ . © 1998 Elsevier Science S.A.

*Keywords:* *Nido* and *closo* geometries; Electron count; Boron

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## 1. Introduction

In contrast to the chain and ring structures that typify the carbon hydrides, boron hydride chemistry is based on polyhedral cage and cluster structures. The structures of many polyhedral boron-containing compounds and related cluster species are governed by Ken Wade's Rules [1,2]<sup>2</sup> that relate cluster geometrical structural patterns [4,5]<sup>3</sup> to formal cluster-electron counts. These Rules have been exceptionally useful in the development of the area [1,2,4–8,3,9] (<sup>1,2</sup>).<sup>4</sup> Exceptions to these Rules are important, because the delineation of any related series of exceptions should facilitate the recognition and the understanding of new patterns, and of any underlying chemical features that account for them, and pave the way for their logical and useful extension. Particularly interesting deviations can arise

when transition-element centres are also part of the boron-containing clusters [9]. For example, open *nido* or *arachno* geometries are sometimes observed when the formal cluster electron-count dictates *closo* [10–20], and closed cluster geometries can be observed when the formal cluster-electron count is sub-*closo* [20–23]. Sometimes the observed open and closed cluster geometries differ from the normal *closo*, *nido*, etc. associated with the classical Wade's Rules formalism [9,21–28] [29–31], and some of these that appear to fall into alternative structural patterns [9,30,31] have been labelled *isocloso*, *isonido*, etc. There are interesting discussions and tentative rationalisations about some of these anomalies in the literature [9,32–35]. However, although some specific individual deviations are reasonably defined and rationalised [36–38], there are no unequivocal general theoretical interpretations. It is therefore important to have comparative experimental findings for the development and assessment of theory. In this context we here present a preliminary report on two very closely related new anomalous eleven-vertex bimetalaborane cluster systems, represented by  $[7,7-(\text{PMe}_2\text{Ph})_2-9-(\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me})-7,9-\text{PtRuB}_9\text{H}_{11}]$  (compound **1**) and  $[1-(\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me})-4,4-(\text{PMe}_2\text{Ph})_2-1,4-\text{RuPtB}_9\text{H}_9]$  (compound **2**). These two examples differ only by two hydrogen atoms, and thereby

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<sup>1</sup> We are pleased to be able to dedicate this paper to Ken Wade, whose Rules have been instrumental and inspirational in the development of boron-containing cluster chemistry over the quarter century since their original formulation.

<sup>2</sup> See also K. Wade, contribution in Ref. [3].

<sup>3</sup> See also R.E. Williams, contribution in Ref. [3].

<sup>4</sup> See references therein. See also J.D. Kennedy, contribution to [3].

constitute a good comparative pair. Previous comparative work in this area has been limited largely to monometalla systems.

Reaction on a scale of 700  $\mu\text{mol}$  between [6-( $\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me}$ )-*nido*-6-RuB<sub>9</sub>H<sub>13</sub>], KH and [PtCl<sub>2</sub>(PMe<sub>2</sub>Ph)<sub>2</sub>] in THF at room temperature for 24 h, followed by chromatography (TLC, silica gel G, 100% CH<sub>2</sub>Cl<sub>2</sub>) reveals several coloured reaction products. Repeated chromatography has resulted in the ultimate

isolation and identification of two of these, yellow [7,7-(PMe<sub>2</sub>Ph)<sub>2</sub>-9-( $\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me}$ )-7,9-PtRuB<sub>9</sub>H<sub>11</sub>] (*nido* eleven-vertex numbering system; compound **1**) ( $R_F$  ca. 0.3; 8.9%) and green–purple [1-( $\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me}$ )-4,4-(PMe<sub>2</sub>Ph)<sub>2</sub>-1,4-RuPtB<sub>9</sub>H<sub>9</sub>] (*closo* eleven-vertex numbering system; compound **2**) ( $R_F$  ca. 0.5; 8.3%) as air-stable crystalline solids. Both have been characterised by single-crystal X-ray diffraction analysis, (Fig. 1) and NMR spectroscopy.

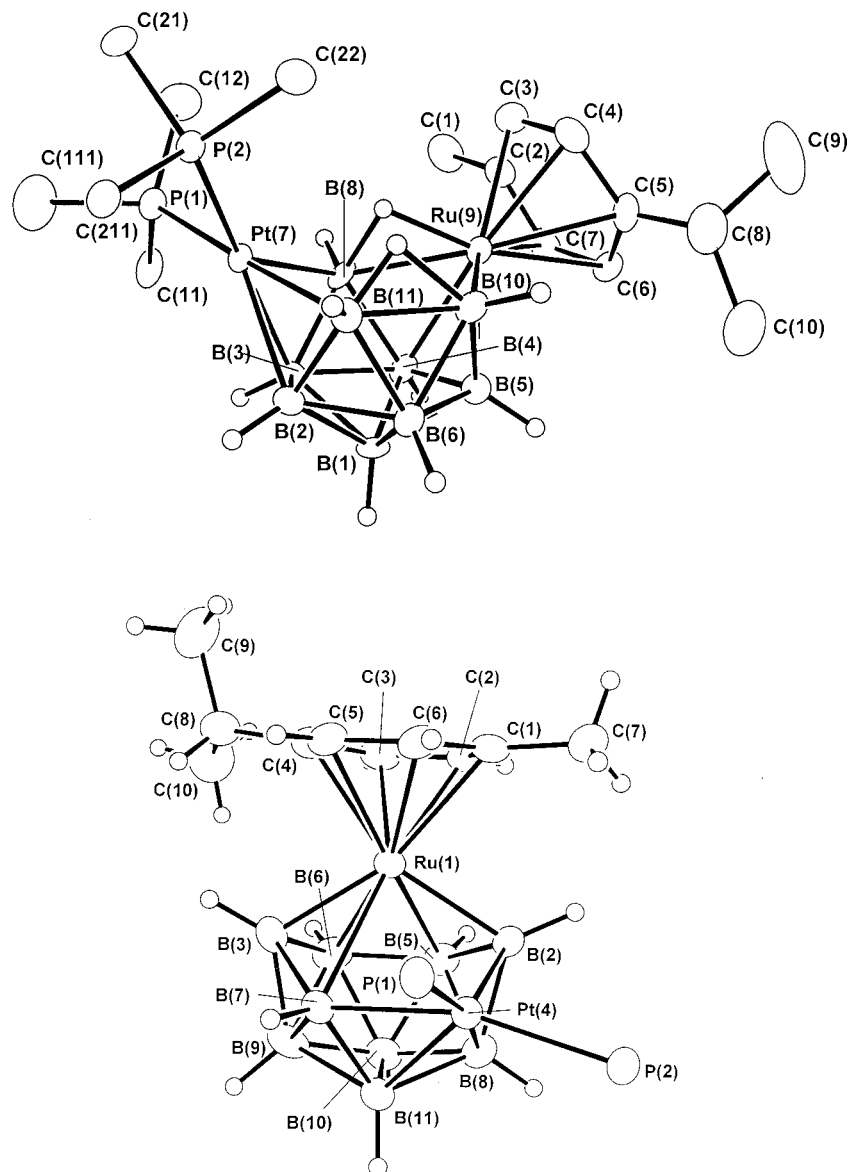


Fig. 1. ORTEP-type [39] drawings for [7,7-(PMe<sub>2</sub>Ph)<sub>2</sub>-9-( $\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me}$ )-7,9-PtRuB<sub>9</sub>H<sub>11</sub>] (compound **1**, upper) and [1-( $\eta^{6-iso}\text{PrC}_6\text{H}_4\text{Me}$ )-4,4-(PMe<sub>2</sub>Ph)<sub>2</sub>-1,4-RuPtB<sub>9</sub>H<sub>9</sub>] (compound **2**, lower). Selected interatomic distances (Å) for compound **1** are: from Pt(7) to P(1) 2.318(2), P(2) 2.328(2), B(2) 2.226(8), B(3) 2.280(8), B(8) 2.317(9) and B(1) 2.305(8); from Ru(9) to B(4) 2.251(8), B(5) 2.236(8), B(8) 2.215(8), B(10) 2.303(8), and to C(2)–C(7) in range 2.196(7) to 2.302(7); for compound **2**: Pt(4) to P(1) 2.354(2), P(2) 2.315(2), B(2) 2.124(10), B(7) 2.198(9), B(8) 2.269(10) and B(1) 2.302(10); from Ru(1) to B(2) 2.168(9), B(3) short at 2.086(10), B(5) 2.254(10), B(6) 2.284(10), B(7) long at 2.604(9) and to C(1)–C(6) exhibiting a larger range than for **1**, viz. 2.207(8) to 2.370(8). In compound **1** the distances Ru(9)–Pt(7) at 3.767(1) and Ru(9)–B(11) at 3.537(8) Å across the open face are clearly non-bonding. In compound **2** the equivalent distances Ru(1)–Pt(4) at 2.964(1) and Ru(1)–B(7) at 2.604(9) Å indicate much more interaction, although they are at the higher end of ranges that are generally thought of as bonding; i.e. even with the sub-*closo* electron count the *closo*-type structure still exhibits some tendency to more open *nido* character (schematic **II**).

## 2. Single crystal X-ray diffraction analysis

Orange crystals of [(PMe<sub>2</sub>Ph)<sub>2</sub>PtB<sub>9</sub>H<sub>11</sub>Ru(η<sup>6</sup>-*iso*PrC<sub>6</sub>H<sub>4</sub>Me)] (compound **1**) were obtained by diffusion of hexane into a solution in dichloromethane, and dark purple crystals of [(PMe<sub>2</sub>Ph)<sub>2</sub>PtB<sub>9</sub>H<sub>9</sub>Ru(η<sup>6</sup>-*iso*PrC<sub>6</sub>H<sub>4</sub>Me)] (compound **2**) were obtained by diffusion of hexane into a solution in toluene.

### 2.1. Crystal data

Compound **1**: C<sub>26</sub>H<sub>47</sub>B<sub>9</sub>P<sub>2</sub>PtRu, *M* = 815.03, monoclinic, *a* = 9.1991(6), *b* = 14.3595(7), *c* = 25.178(2) Å, β = 94.197(8)°, *U* = 3317.0(4) Å<sup>3</sup>, space group P2<sub>1</sub>/c (No. 14), *Z* = 4, *D*<sub>c</sub> = 1.632 Mg m<sup>3</sup>, *F*(000) = 1600, *T* = 210 K, crystal dimensions 0.38 × 0.33 × 0.27 mm; 5971 reflections were measured (5830 unique, *R*<sub>int</sub> = 0.0854) on a Stoe STADI4 four-circle diffractometer in the range 3 < 2θ < 50°, operating in the ω–θ scan mode and using graphite-monochromated Mo–K<sub>α</sub> radiation (λ = 0.71073 Å). Compound **2**: C<sub>26</sub>H<sub>45</sub>B<sub>9</sub>P<sub>2</sub>PtRu, *M* = 813.01, monoclinic, *a* = 10.2509(14), *b* = 13.950(2), *c* = 22.775(5) Å, β = 94.27(2)°, *U* = 3247.8(10) Å<sup>3</sup>, space group P2<sub>1</sub>/c (No. 14), *Z* = 4, *D*<sub>c</sub> = 1.663 Mg m<sup>3</sup>, *F*(000) = 1592, *T* = 220 K, crystal dimensions 0.42 × 0.34 × 0.23 mm; 6083 reflections were measured as for compound **1** (5711 unique, *R*<sub>int</sub> = 0.0417). In each case three standard reflections measured every hour showed a negligible variation in intensity, and data were corrected for Lorentz and polarisation effects and an absorption correction was applied which was based on azimuthal *psi*-scans. Transmission factors were 0.0872–0.2669, μ(Mo–K<sub>α</sub>) = 4.781 mm<sup>–1</sup> for compound **1** and 0.7071 to 0.9865, μ(Mo–K<sub>α</sub>) = 4.883 mm<sup>–1</sup> for compound **2**.

### 2.2. Structure solution and refinement

Both compounds were solved and refined similarly. Platinum and ruthenium atoms were located using Patterson heavy-atom methods (SHELXS-86) [40], subsequent Fourier difference techniques located the remaining heavier atoms, and refinement (SHELXL-93) [41] was by full-matrix least-squares on *F*<sup>2</sup> using all the unique data. All non-hydrogen atoms were assigned anisotropic displacement parameters. Phenyl rings were restrained to be flat and of overall C<sub>2v</sub> symmetry, with their hydrogen atoms included using a riding model with isotropic displacement parameters. Borane terminal hydrogen atoms were located via Fourier difference syntheses, and were assigned isotropic displacement parameters constrained at 1.2 *U*<sub>eq</sub> of their parent boron atoms. All B–H distances in compound **1** were restrained to be 1.1 Å (within an e.s.d of 0.03 Å). For compound **1** the weighting scheme was *w* = 1/[σ<sup>2</sup>(*F*<sub>o</sub><sup>2</sup>) + (0.0403*P*)<sup>2</sup>] where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3;

the final *wR*(*F*<sup>2</sup>) was 0.0917, with a conventional *R*(*F*) 0.0374 based on 4400 observed reflections [with *I* > 2σ(*I*)] (*R* factors defined in Ref. 32) for 394 parameters and 101 restraints, goodness of fit = 1.206, maximum Δ/σ = –0.010, maximum Δ*p* = 1.408 e Å<sup>–3</sup>. For compound **2** the weighting scheme was *w* = 1/[σ<sup>2</sup>(*F*<sub>o</sub><sup>2</sup>) + (0.0254*P*)<sup>2</sup> + 18.4728*P*] where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3; the final *wR*(*F*<sup>2</sup>) was 0.0975, with a conventional *R*(*F*) 0.0402 based on 4093 observed reflections [with *I* > 2σ(*I*)] for 386 parameters and 73 restraints, goodness of fit = 1.011, maximum Δ/σ = +0.014, maximum Δ*p* = 0.721 e Å<sup>–3</sup>.

## 3. NMR data

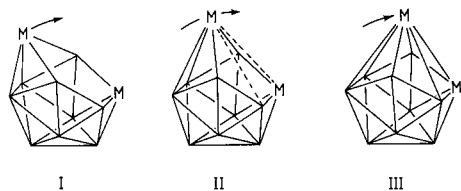
NMR data for [7,7-(PMe<sub>2</sub>h)<sub>2</sub>-9-(η<sup>6</sup>-*iso*PrC<sub>6</sub>H<sub>4</sub>Me)-7,9-PtRuB<sub>9</sub>H<sub>11</sub>] (compound **1**) in CDCl<sub>3</sub>, ordered as tentative assignment δ(<sup>11</sup>B) (rel. BF<sub>3</sub>OEt<sub>2</sub>) [δ(<sup>1</sup>H)] for {BH(*exo*)} units, as follows; BH(8) +34.8 [+5.08, *J*(<sup>195</sup>Pt–<sup>1</sup>H) 19 Hz], BH(6 or 3) ca. +24 [+4.82], BH(3 or 6) ca. +24 [+4.10], BH(5) +13.6 [+3.50], BH(4) +1.1 [+1.97, *J*(<sup>195</sup>Pt–<sup>1</sup>H) 16 Hz], BH(2) –0.8 [+2.39], BH(11) –2.6 [+2.48]. BH(10) –5.6 [+2.42] and BH(1) –24.6 [+1.70], with δ(<sup>1</sup>H) (8,9) –12.24 and (10,11) –2.14; additionally δ(<sup>1</sup>H) (*iso*PrC<sub>6</sub>H<sub>4</sub>Me) at +1.27 and +1.29 (doublets), +2.06 (singlet) +2.74 (septet) and +5.31 to +5.59 ppm (multiplets), and δ(<sup>1</sup>H) (P–methyl) at +1.79, +1.69, +1.58 and +1.50 ppm with doublet splittings ca. 9 Hz), and δ(<sup>31</sup>P) (rel. 85% H<sub>3</sub>PO<sub>4</sub>) +13.7 [<sup>1</sup>*J*(<sup>195</sup>Pt–<sup>31</sup>P) 2676 Hz], +11.9 [<sup>1</sup>*J*(<sup>195</sup>Pt–<sup>31</sup>P) 2529 Hz], <sup>2</sup>*J*(<sup>31</sup>P–<sup>31</sup>P) 29 Hz. For [1-(η<sup>6</sup>-*iso*PrC<sub>6</sub>H<sub>4</sub>Me)-4,4-(PMe<sub>2</sub>Ph)<sub>2</sub>-1,4-RuPtB<sub>9</sub>H<sub>9</sub>] (compound **2**) in CDCl<sub>3</sub>, ordered as tentative assignment δ(<sup>11</sup>B) (rel. BF<sub>3</sub>OEt<sub>2</sub>) [δ(<sup>1</sup>H)] for {BH(*exo*)} units as follows; BH(3 or 2) +95.9 [+8.78], BH(2 or 3) +92.2 [+10.25], BH(8) +35.2 [+4.90], BH(5 or 6) ca. +8.5 [+2.47], BH(6 or 5) ca. +8.5 [+2.60], BH(10 or 9) ca. +3.5 [+3.48, doublet splitting, 19 Hz], BH(9 or 10) ca. +3.5 [+3.86, triplet structure, splitting 31 Hz], BH(11) +1.4 [+2.24], and BH(4) –9.5 [+0.47]; additionally δ(<sup>1</sup>H) (*iso*PrC<sub>6</sub>H<sub>4</sub>Me) at +1.33 (doublets) +2.41 (singlet) +3.37 (septet) and +5.18 to +5.52 ppm (multiplets), δ(<sup>1</sup>H) (P–methyl) at +1.80, +1.76, +1.41 and +1.29 ppm (all with doublet splittings ca. 9 Hz), and δ(<sup>31</sup>P) (rel. 85% H<sub>3</sub>PO<sub>4</sub>) +0.4 [<sup>1</sup>*J*(<sup>195</sup>Pt–<sup>31</sup>P) 2822 Hz], –7.8 [<sup>1</sup>*J*(<sup>195</sup>Pt–<sup>31</sup>P) 2592 Hz], <sup>2</sup>*J*(<sup>31</sup>P–<sup>31</sup>P) 29 Hz.

## 4. Discussion

Yellow compound **1** is seen to be essentially of *nido* open eleven-vertex geometry **I**, and has two bridging hydrogen atoms associated with the open face. It is a direct analogue of *nido*-structured [7,7-(PMe<sub>2</sub>Ph)<sub>2</sub>-7-

PtB<sub>10</sub>H<sub>12</sub>] (compound **3**) [42,43] with the neutral {Ru( $\eta^{6-iso}$ PrC<sub>6</sub>H<sub>4</sub>Me)} unit being formally isolobal and isoelectronic with a neutral cluster {BH} fragment. A formal Wadlan electron-count for compound **1** or compound **3**, with a contribution of two electrons from the {Pt(PMe<sub>2</sub>Ph)<sub>2</sub>} vertex, would give 24 electrons, predicting (2*n* + 2)-electron *closo* geometry for the eleven-vertex cluster system in each case, in apparent conflict with the observed geometries. This is because the platinum centre can be regarded in this compound as a square-planar sixteen-electron platinum(II) centre [9,14–17]. Although the {Pt(PMe<sub>2</sub>Ph)<sub>2</sub>} unit is equivalent to an anionic {BH<sub>2</sub>}<sup>−</sup> unit in terms of cluster bonding, unlike {BH<sub>2</sub>}<sup>−</sup> it does not have two electrons for cluster counting purposes in an *endo* terminal bond to hydrogen, hence the anomaly [9,14–17].

The situation with purple [1-( $\eta^{6-iso}$ PrC<sub>6</sub>H<sub>4</sub>Me)-4,4-(PMe<sub>2</sub>Ph)<sub>2</sub>-1,4-RuPtB<sub>9</sub>H<sub>9</sub>] (compound **2**) appears somewhat more anomalous. Compared to compound **1**, it lacks the two bridging hydrogen atoms, and the cluster now has an eleven-vertex *closo*-type geometry, as opposed to the *nido* geometry of compound **1**. However, with two hydrogen atoms fewer, the formal Wadlan electron count of compound **2** is not *closo*, but sub-*closo* at 2*n*-electron *pileo*. This latter might possibly be expected to be associated with a more compact structure than *closo* itself, for example as observed in the eleven-vertex 'isocloso' monometallaborane [1-( $\eta^{6-iso}$ PrC<sub>6</sub>H<sub>4</sub>Me)-1-RuB<sub>10</sub>H<sub>10</sub>] [20], also of formal *pileo* electron count. However, for compound **2** the Ru(1)–Pt(4) and Ru(1)–B(7) distances of 2.964(1) and 2.604(9) Å respectively are in fact long, indicating an incomplete closure **II** from the *nido* geometry **I**, rather than a complete compact closure as shown in **III**.



This inhibition of complete closure could arise from a second characteristic of platinum(II) square-planar character, viz. a reluctance by the platinum to accept an additional electron pair from the cluster bonding scheme to give it a closed transition-element eighteen-electron shell.

## 5. Conclusion

The comparison of compounds **1** and **2** is in interesting contrast to the mutually isoelectronic pair consisting of neutral *nido*-shaped [(PPh<sub>3</sub>)<sub>2</sub>RhSB<sub>9</sub>H<sub>10</sub>] (compound

**4**) and the *closo*-shaped anion [(PPh<sub>3</sub>)<sub>2</sub>RhSB<sub>9</sub>H<sub>10</sub>]<sup>−</sup> (species **5**) [14–19], effectively duplicated by the neutral pair consisting of *nido*-shaped [(PMe<sub>2</sub>Ph)<sub>2</sub>PtCB<sub>9</sub>H<sub>11</sub>] (compound **6**) and *closo*-shaped [(PMe<sub>2</sub>Ph)<sub>2</sub>PtC<sub>2</sub>B<sub>8</sub>H<sub>10</sub>] (compound **7**) [36–38,44]. All the species **4**, **5**, **6** and **7** have formal *closo* electron counts, and so here the notional **4** → **5** and **6** → **7** conversions are from Wade-anomalous *nido* to Wade-consistent *closo*. These two geometric *nido* to *closo* transitions are associated with the loss of a proton only and no electrons. By contrast, the present (notional) **1** → **2** conversion of the {RuPt} species is from Wade-anomalous *nido* to perhaps a somewhat more Wade-anomalous, partially open, *closo* by the loss of two electrons as hydrogen atoms. Compounds **1** and **2** obviously an interesting pair of compounds, and we currently try to extend these studies to additional eleven-vertex bimetalla-B-frame systems.

## Acknowledgements

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