A Spiked-butterfly Cluster: Crystal and Electronic Structures of $[RhRu_4H(\eta^5\text{-}C_5Me_5)(CO)_{13}BH_2]$†

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The crystal structure of $[RhRu_4H(\eta^5\text{-}C_5Me_5)(CO)_{13}BH_2]$ **2** has been determined: monoclinic, space group $P2_1/c$, $a = 18.118(4)$, $b = 10.823(2)$, $c = 17.432(3)$ Å, $\beta = 118.49(3)°$, $Z = 4$. The confirmation of this geometry corrects a proposal that we have previously made regarding this compound. Compound **2** has a pentametal skeleton which is best described as a 'spiked-butterfly' with the rhodium atom occupying the 'spike' position. Ambiguities concerning an electron-counting scheme for compound **2** have been addressed by using the Fenske–Hall molecular orbital approach to probe the electronic structure.

As part of our current investigations into the chemistry of heterometallic boron-containing clusters, we have reported[1] details of the reactions of $[Ru_3(CO)_9BH_4]^-$ and $[Ru_3(CO)_9B_2H_5]^-$ with $[\{Rh(\eta^5\text{-}C_5Me_5)Cl_2\}_2]$. In each case, the major product of these reactions was the butterfly cluster $[RhRu_3H_2(\eta^5\text{-}C_5Me_5)(CO)_9BH]$ **1**. A purple-red compound **2** was produced in both these reactions in < 5% yield and, on the basis of NMR and IR spectroscopic, and mass spectrometric data we proposed the formula of **2** to be '$[RhRu_4H(\eta^5\text{-}C_5Me_5)(CO)_{12}BH]$'. In particular, we noted that the highest mass envelope in the FAB mass spectrum of this compound was at *m*/*z* 992 and that the isotopic distribution gave a good match to that expected for the given formula. At the time, we were unable to gain X-ray quality crystals of **2**; we suggested that the structure shown in Fig. 1 would be consistent with the 76-valence-electron count of the compound. We commented that the bridging metal fragment could comprise either a ruthenium unit (as shown in Fig. 1) or a rhodium-centred unit.[1]

We have now succeeded in growing suitable crystals of **2** and have undertaken a single crystal X-ray structure determination. We report here our new results which demand a correction, not only to the originally proposed structure of **2**, but also to its formulation. The new structure raises an ambiguity as regards the electron count in compound **2**, and we present an analysis of the electronic structure of the new cluster.

Experimental

Compound **2** was prepared as a minor product (< 5% yield) in reactions previously described.[1]

Crystal Structural Determination.—Suitable single crystals were grown at −25 °C from a CH_2Cl_2 solution of **2** layered with hexane. A crystal was mounted directly from solution under argon using a perfluorocarbon oil which protected it from atmospheric O_2 and moisture. The oil freezes at reduced temperatures and holds the crystal static in the X-ray beam.[2] The crystal was immediately transferred to a Stoe diffractometer equipped with an Oxford Cryosystems Cryostream cooling device operating at 153 K.

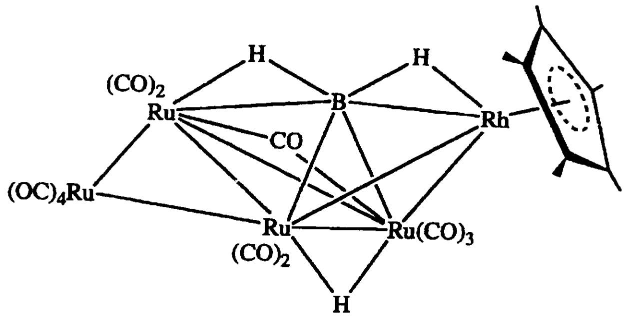

Fig. 1 The proposed structure of '$[RhRu_4H(\eta^5\text{-}C_5Me_5)(CO)_{12}BH_2]$' (ref. 1)

Crystal data. $C_{23}H_{18}BO_{13}RhRu_4$, $M = 1020.37$, monoclinic, space group $P2_1/c$ (no. 14), $T = 153(2)$ K, $a = 18.118(4)$, $b = 10.823(2)$, $c = 17.432(3)$ Å, $\beta = 118.49(3)°$, $U = 3004.3(10)$ Å^3 (by least-squares refinement of diffractometer angles from 25 automatically centred reflections in the range $20 < 2\theta < 25°$), $Z = 4$, $D_c = 2.256$ g cm^{-3}, $F(000) = 1944$, $\lambda(\text{Mo-K}\alpha) = 0.710\,73$ Å, $\mu(\text{Mo-K}\alpha) = 25.64$ cm^{-1}. Dark red block. Crystal dimensions: 0.43 × 0.37 × 0.22 mm, $\mu R = 0.44$.

Data collection and processing. Stoe four-circle diffractometer, 30 step ω–θ scan mode, with scan step 0.04° and step time 0.5–2.0 s per step, graphite-monochromated Mo-Kα radiation. 4048 Reflections measured ($7.0 \leqslant 2\theta \leqslant 45.0°$, $+h$, $-k$, $\pm l$), 3903 unique ($R_{int} = 0.022$) after a semi-empirical absorption correction based on ψ-scan data (maximum and minimum transmission factors, 0.976, 0.556). Three standard reflections measured at hourly intervals throughout the data collection showed no significant variations.

Structure solution and refinement. The structure was solved by direct methods (Ru and Rh atoms) and Fourier-difference techniques for the remaining non-hydrogen atoms. Methyl H atoms were placed in idealised positions and allowed to ride on the relevant C atoms. The H atoms co-ordinated to the B atom were located directly in the electron-density difference map and were refined freely. The hydrido-H atom was located in the difference map but was not refined. The structure was refined by full-matrix least squares on F^2 with all non-hydrogen atoms assigned anisotropic displacement parameters. The weighting scheme $w^{-1} = [\sigma^2F^2 + (0.0841P)^2 + 5.6P]$ where $P = [0$ or $F_o^2 + 2F_c^2]/3$ was introduced and produced a satisfactory agreement analysis. The final converged residuals for 392 parameters were $R_1 = 0.030$, $wR_2 = 0.096$ for 3662 reflections

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† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1995, Issue 1, pp. xxv–xxx.

Table 1 Atomic coordinates (× 10^4) for **2**

Atom	*x*	*y*	*z*
Rh	8 651(1)	−1 668(1)	517(1)
Ru(1)	8 029(1)	690(1)	267(1)
Ru(2)	6 953(1)	2 543(1)	−878(1)
Ru(3)	7 450(1)	2 599(1)	965(1)
Ru(4)	5 747(1)	2 297(1)	−284(1)
B	6 796(5)	1 189(7)	−36(5)
C(1)	8 789(5)	−3 698(7)	580(5)
C(2)	8 150(4)	−3 410(6)	−302(5)
C(3)	7 475(4)	−2 846(7)	−244(5)
C(4)	7 676(4)	−2 824(6)	663(5)
C(5)	8 467(5)	−3 383(6)	1 170(4)
C(6)	9 597(5)	−4 376(7)	829(5)
C(7)	8 202(5)	−3 687(8)	−1 130(5)
C(8)	6 667(5)	−2 403(7)	−994(5)
C(9)	7 091(5)	−2 368(7)	983(5)
C(10)	8 905(5)	−3 600(7)	2 147(5)
C(11)	9 752(5)	−1 196(7)	787(5)
O(11)	10 405(3)	−860(5)	952(4)
C(12)	8 120(4)	−336(7)	−621(5)
O(12)	8 017(3)	−560(5)	−1 313(3)
C(13)	8 639(4)	−306(6)	1 380(5)
O(13)	8 876(3)	−350(5)	2 128(3)
C(101)	9 034(5)	1 593(6)	531(4)
O(101)	9 634(3)	2 117(5)	692(3)
C(201)	7 898(6)	2 817(7)	−1 080(5)
O(201)	8 432(4)	2 969(7)	−1 241(4)
C(202)	6 313(5)	3 939(7)	−1 545(5)
O(202)	5 922(4)	4 720(6)	−1 996(4)
C(203)	6 378(5)	1 406(8)	−1 810(5)
O(203)	6 013(4)	700(6)	−2 351(4)
C(301)	7 310(4)	1 455(7)	1 731(5)
O(301)	7 194(3)	755(5)	2 132(3)
C(302)	7 069(5)	4 057(7)	1 319(5)
O(302)	6 868(4)	4 878(5)	1 584(4)
C(303)	8 631(5)	2 897(7)	1 724(5)
O(303)	9 311(4)	3 086(6)	2 202(4)
C(401)	5 365(4)	2 061(7)	565(5)
O(401)	5 175(4)	1 911(7)	1 089(4)
C(402)	4 706(5)	1 974(8)	−1 321(5)
O(402)	4 079(4)	1 779(7)	−1 912(4)
C(403)	5 588(5)	4 038(7)	−392(5)
O(403)	5 501(4)	5 095(5)	−469(4)

with $I > 2\sigma(I)$ and $R_1 = 0.036$, $wR_2 = 0.127$ (all data); goodness of fit = 1.26. A final difference map showed the highest residual peak of 1.27 e Å^{-3} to be close to the metal atom positions. Final fractional atomic coordinates are listed in Table 1. All calculations were performed on a Viglen 486 personal computer using SHELXTL PLUS[3] and SHELXL 93.[4]

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters and remaining bond lengths and angles.

Molecular Orbital Calculations.—Fenske–Hall calculations[5] were carried out on the model compound **2a** in terms of orbital interactions between the fragments $\{Ru_4H(CO)_{12}BH_2\}$ and $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$. Coordinates determined from the crystal structure were used, but with a C_5H_5 ring replacing the C_5Me_5 ligand. The calculations employed basis functions generated by the numerical Xα atomic orbital program of Herman and Skillman[6] used in conjunction with the Xα-to-Slater basis program of Bursten and Fenske.[7]

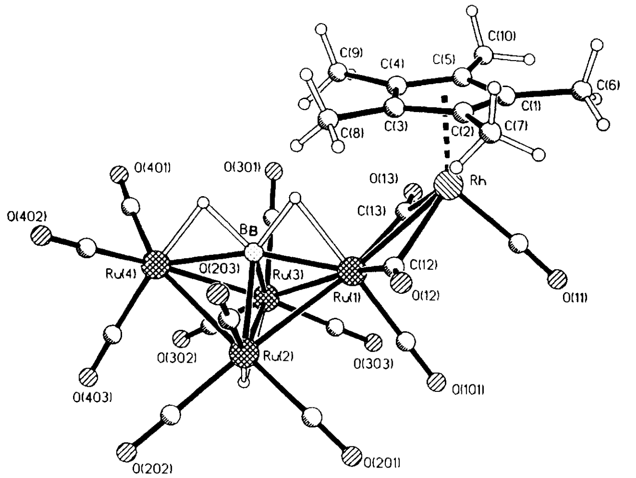


Fig. 2 Molecular structure of $[RhRu_4H(\eta^5\text{-}C_5Me_5)(CO)_{13}BH_2]$ **2**

Table 2 Selected bond distances (Å) and angles (°) for **2**

Ru(1)–Ru(2)	2.8449(10)	Ru(1)–Ru(3)	2.8397(8)
Ru(2)–Ru(3)	2.8937(10)	Ru(2)–Ru(4)	2.8416(10)
Ru(3)–Ru(4)	2.8210(14)	Ru(1)–B	2.105(8)
Ru(2)–B	2.186(7)	Ru(3)–B	2.191(8)
Ru(4)–B	2.110(8)	Ru(1)–Rh	2.7395(8)
Ru(1)–C(12)	1.975(7)	Rh–C(12)	2.263(7)
Ru(1)–C(13)	2.026(7)	Rh–C(13)	2.114(7)
Rh–(C_5 ring centroid)	1.910		
Ru(2)–Ru(1)–Ru(3)	61.20(2)	Ru(1)–Ru(2)–Ru(3)	59.31(3)
Ru(1)–Ru(2)–Ru(4)	93.81(3)	Ru(3)–Ru(2)–Ru(4)	58.92(3)
Ru(1)–Ru(3)–Ru(2)	59.49(2)	Ru(2)–Ru(3)–Ru(4)	59.62(3)
Ru(1)–Ru(3)–Ru(4)	94.37(3)	Ru(2)–Ru(4)–Ru(3)	61.46(3)
Rh–Ru(1)–Ru(3)	144.86(3)	Rh–Ru(1)–Ru(2)	149.37(3)
Ru(1)–B–Ru(4)	160.3(4)	Ru(2)–B–Ru(3)	82.8(3)
Rh–C(12)–O(12)	124.8(5)	Rh–C(13)–O(13)	130.2(5)
Ru(1)–C(12)–O(12)	154.7(6)	Ru(1)–C(13)–O(13)	146.8(5)

Results and Discussion

Molecular Structure of Compound **2**.—The crystal structure of compound **2** shows that the discrete molecular units are separated by normal van der Waals distances. The molecular structure of **2** is shown in Fig. 2, and selected bond distances and angles are listed in Table 2. The results confirm the presence of a pentametal framework as expected. However, the geometry is more open than the proposed structure shown in Fig. 1 and is best described as being a 'spiked butterfly'. Of prime importance is the fact that **2** contains not twelve carbonyl ligands as originally proposed from the mass spectral data, but thirteen. Our original deduction of the formula of **2** was based primarily on mass spectral data. The highest mass peak is due to the parent ion with the loss of one CO ligand; no parent ion is clearly detected.

The central core of the structure of **2** comprises an Ru_4 butterfly. The boron atom resides within the cavity, in a semi-interstitial position; the B atom is within bonding contact of all four ruthenium atoms. Each of atoms Ru(4), Ru(3) and Ru(2) carries three terminal carbonyl ligands whilst atom Ru(1) bears one terminal CO ligand and two carbonyls that are involved in bridging interactions between Ru(1) and the $\{Rh(\eta^5\text{-}C_5Me_5)(CO)\}$ 'spike'. Both of the ligands C(12)O(12) and C(13)O(13) bridge the Ru(1)–Rh edge asymmetrically. The distance Ru(1)–Rh is 2.7395(8) Å and this is indicative of a direct metal–metal bond; the distance is shorter than the average Ru–Ru bond length within the butterfly framework [2.8503(14) Å].

The geometry of the Ru_4B-butterfly core is little perturbed in going from $[Ru_4H(CO)_{12}BH_2]$ **3** to compound **2**. In **3**,[8] the internal dihedral angle of the Ru_4 framework is 118° and is 116.6° in **2**; the height of the boron atom above the $Ru_{wing\text{-}tip} \cdots Ru_{wing\text{-}tip}$ axis is 0.39[9] in **3** and 0.36 Å in **2**.

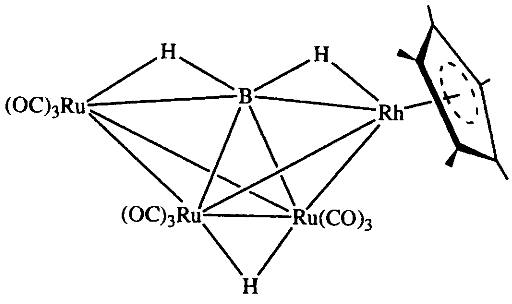
Cluster hydrogen atoms were located in **2**. Their positions are consistent with the solution ^1H NMR spectroscopic signature of the compound; the signal at δ −20.58 is assigned to the hydrogen atom which bridges the edge Ru(2)–Ru(3) and the resonances at δ −8.9 and −10.2 are assigned to bridging hydrogen atoms along edges Ru(4)–B and Ru(1)–B, respectively. The two boron-associated cluster-hydrogen atoms can be assigned by comparison with the parent tetraruthenaborane **3** in which the two Ru–H–B bridging hydrogen atoms are equivalent and exhibit a single resonance (collapsed quartet) at δ −8.4.[8,10] In **2**, one of the Ru–H–B hydrogen atoms remains in an environment akin to that in **3**, whilst the other is significantly perturbed by the introduction of the rhodium fragment.

Bonding Description of Compound **2**.—We have recently reported another spiked-butterfly cluster, $[Ru_5H_2(\eta^5\text{-}C_5Me_5)(CO)_{13}BH_2]$ **4**.[11] The 'spike' in **4** comprises a $\{Ru(\eta^5\text{-}C_5Me_5)(CO)_2\}$ unit, and, as in **2**, it is bonded to a wing-tip metal atom of the Ru_4 butterfly core. Although hydrogen atoms were located in this structure, the estimated standard deviations associated with the hydrogen atom that is shown in structure **4** to bridge the $Ru_{wing\text{-}tip}$–Ru_{spike} edge were such that a complete description remained ambiguous. In order to satisfy the 18-electron rule at the ruthenium 'spike' atom and a 62-electron count for the butterfly portion of **4**, one can assign a localised $Ru_{wing\text{-}tip}$–Ru_{spike} bond and a terminal (to the butterfly) hydride ligand.

It is of interest to compare this bonding description with that suggested by the structure of **2**. The pendant $\{Ru(\eta^5\text{-}C_5Me_5)(CO)\}$ unit in **2** requires two further electrons to become an 18-electron centre. These *could* formally be assigned as one electron from a Ru(1)–Rh bond and one electron from one of the bridging carbonyl ligands. This then gives a 62-electron count for the butterfly part of **2** with one electron from the Ru(1)–Rh bond, one electron from one bridging carbonyl ligand and two electrons from the other μ-CO ligand. This rationale would be particularly appropriate if one of ligands C(12)O(12) or C(13)O(13) were semi-bridging in nature. However, as noted above, both of these ligands bridge the Ru(1)–Rh edge asymmetrically with both carbon atoms being closer to the ruthenium than the rhodium atom [Ru(1)—C(12) 1.975(7), Ru(1)–C(13) 2.026(7), Rh–C(12) 2.263(7) and Rh–C(13) 2.114(7) Å]. This might imply that both are tending towards being semi-bridging in nature and that the primary attachment of the 'spike' to the cluster core is through a two-centre two electron Ru–Rh bond. The experimental bond length of 2.7395(8) Å is consistent with a direct interaction, and electron counts for both portions of the cluster could be satisfied if a dative Ru→Rh bond were invoked. We have recently suggested that the bonding in the related clusters $[RhRu_3H(CO)_9(\eta^5\text{-}C_5Me_5)B(Au_2L_2)(AuCl)]$ [L = PPh_3 or L_2 = 1,1′-bis(diphenylphosphino)ferrocene] might be regarded as donor–acceptor complexes in which the '$[RhRu_3H(CO)_9(\eta^5\text{-}C_5Me_5)B(Au_2L_2)]$' cluster acts as a two-electron donor to the gold(I) chloride unit.[12] However, we have seen no evidence of the cluster $[Ru_4H(CO)_{12}BH_2]$ acting as a donor, as is demanded by the aforementioned bonding description. With these ambiguities in mind, we have carried out an analysis of the bonding in a model compound **2a** using the Fenske–Hall molecular orbital (MO) approach; compound **2a** has the formula $[RhRu_4H(\eta^5\text{-}C_5H_5)(CO)_{13}BH_2]$ and differs from **2** only in having an $\eta^5\text{-}C_5H_5$ ligand in place of the $\eta^5\text{-}C_5Me_5$ group.

The bonding in **2a** is considered in terms of the interaction between the two neutral fragments $\{Ru_4H(CO)_{12}BH_2\}$ and $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$. A significant feature is that the interaction of fragment MOs does *not* lead to a simple bonding picture. There are four major orbital interactions (see below), and together these account for 58% of the total Mulliken-overlap population between the two fragments. The remaining 42% is spread over a further seventeen interactions which involve the five frontier MOs of the $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$ fragments, and fourteen MOs of the butterfly cluster. In order to provide a reasonable discussion of the bonding, we focus on the four major interactions. The remaining contributions augment the same general picture.

The four principal interactions are schematically represented in Fig. 3; the highest occupied MO (HOMO) and lowest



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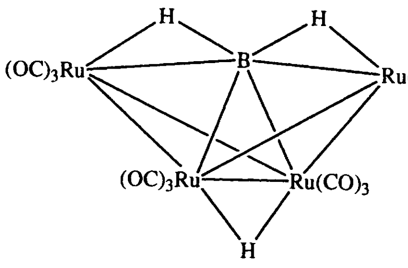

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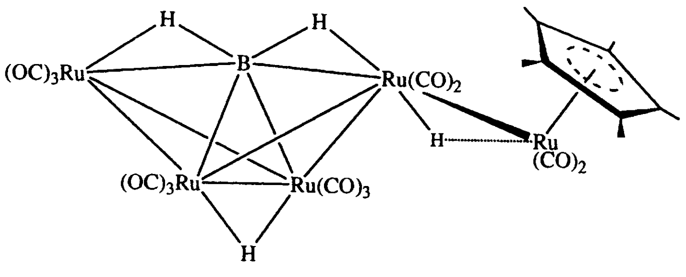

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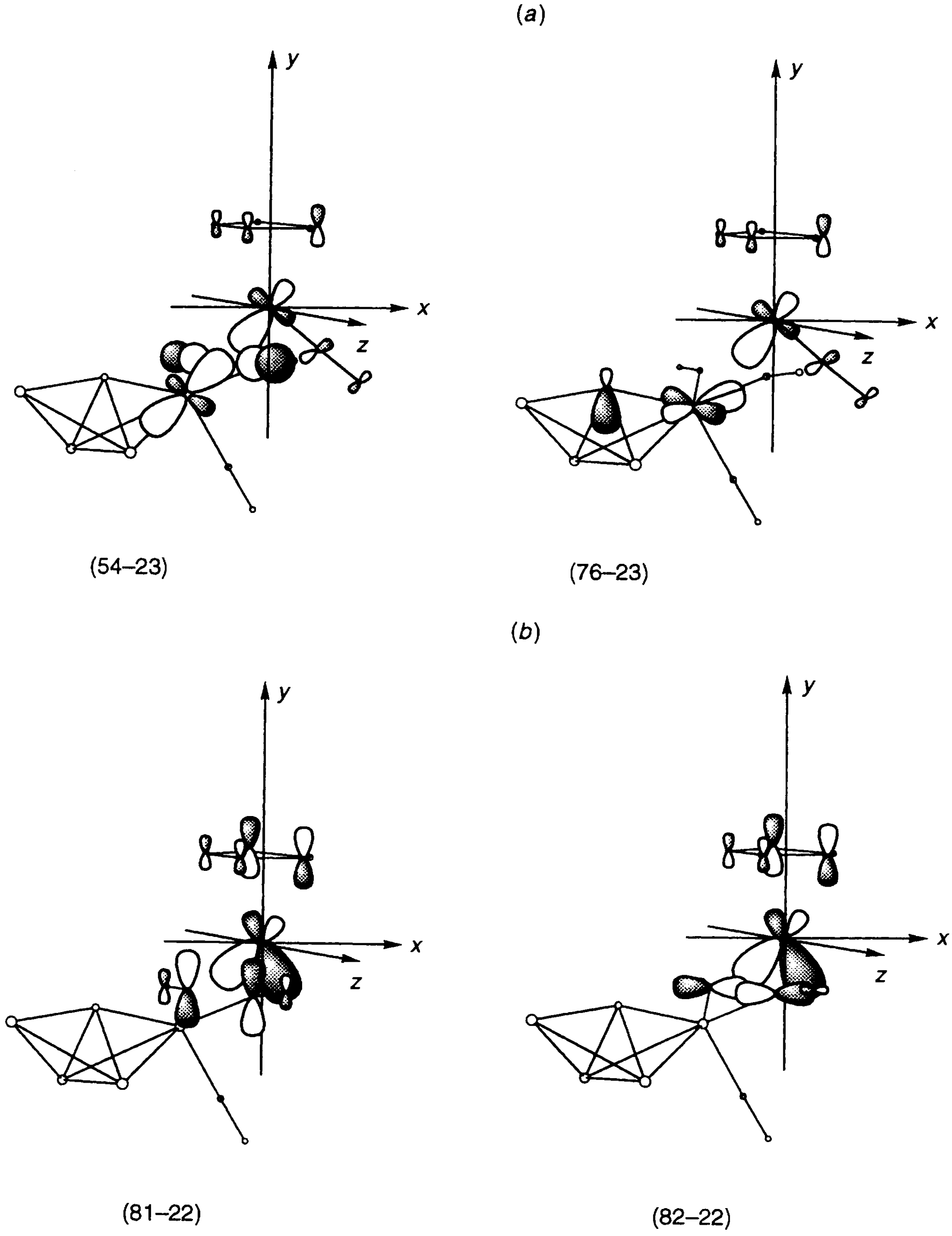

(54–23) (76–23)

(81–22) (82–22)

Fig. 3 Schematic representations of the four major orbital interactions between the fragments $\{Ru_4H(CO)_{12}BH_2\}$ and $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$; (*a*) interactions between two filled MOs of the butterfly cluster and the LUMO of $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$; (*b*) interactions between two unoccupied MOs of the butterfly cluster and the HOMO of $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$. The orbital centred on rhodium is a $d_{yz}p_z$ hybrid as defined by the axis set given. Note that fragment MOs 54, 76, 81 and 82 of $\{Ru_4H(CO)_{12}BH_2\}$ possess contributions from all the Ru atoms but, for clarity, only the dominant contributions are shown

unoccupied MO (LUMO) of $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$ are MOs 22 and 23 respectively, and the HOMO and LUMO of $\{Ru_4H(CO)_{12}BH_2\}$ are MOs 79 and 80 respectively. Both the HOMO and LUMO of the $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$ fragment possess significant orbital contributions from the carbon atoms of the cyclopentadienyl ring. Fig. 3(*a*) shows the $\{Ru_4H(CO)_{12}BH_2\}$–$\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$ orbital interactions (54–23) and (76–23), both of which give rise to transfer of electron density from the butterfly framework to the rhodium centre. Both interactions may be associated with a Ru→Rh dative bond description, although in (54–23) this is augmented with metal–carbon bonding involving the two carbonyl ligands. Fig. 3(*b*) shows the $\{Ru_4H(CO)_{12}BH_2\}$–$\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$ orbital interactions (81–22) and (82–22). The MOs 81 and 82 also possess character from all four Ru atoms (see Fig. 3 caption), but each MO contains ≈40% carbonyl character as indicated. The significant result of orbital interactions (81–22) and (82–22) is a transfer of charge from the HOMO of the rhodium 'spike' to two of the carbonyl ligands on the butterfly wing-tip.

The net charge transfer between the two fragments is from the $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$ unit to the $\{Ru_4H(CO)_{12}BH_2\}$

butterfly; this total includes the effects of *all* the interfragment orbital interactions and not just those shown in Fig. 3. The $\{Rh(\eta^5\text{-}C_5H_5)(CO)\}$ unit carries a net charge of +0.33 electrons in compound **2a** as compared to a zero charge in the isolated fragment. The atomic charges which change the most on the formation of **2a** are those of the carbon atoms in the cyclopentadienyl ring, and the carbon and oxygen atoms in the two bridging-carbonyl ligands. The atomic charges of atoms Ru(1) and Rh (numbering as in Fig. 2) change little as a result of complex formation [Δq for Rh = +0.04 and Δq for Ru(1) = +0.01]. The implication is that it is the ligands, and not the metal framework, that absorb the electronic changes that accompany the addition of the rhodium 'spike' to the butterfly cluster.

Comments on the Formation of **2**.—The formation of **2** was unexpected and the pathway to its formation requires comment. The neutral cluster $[Ru_3(CO)_9BH_5]$ undergoes spontaneous cluster expansion to form **3** as well as $[Ru_6H(CO)_{17}B]$.[13] Similarly, we have observed that the anion $[Ru_3(CO)_9BH_4]^-$ undergoes expansion to give the conjugate base of **3** in addition to $[Ru_6(CO)_{17}B]^-$.[11] It is possible therefore that **2** does not arise directly from the triruthenaborane anion but is formed *via* an intermediate butterfly species. However, the reaction of $[Ru_4H(CO)_{12}BH]^-$ with $[\{Rh(\eta^5\text{-}C_5Me_5)Cl_2\}_2]$ does not produce compound **2**[14] (certainly not as an isolable product). This scenario is not new to us. The photolysis of $[Ru_3(CO)_9BH_5]$ in MeCN in the presence of $M(CO)_6$ (M = Cr, Mo or W) leads to the formation of $[Ru_4H(CO)_{12}BH(\mu\text{-}NCHMe)]$ although addition of MeCN to $[Ru_4H(CO)_{12}BH_2]$ does not occur directly.[15]

In preliminary studies of the reactivity patterns of **2**, we have attempted deprotonation in an initial step towards further investigations. However, prolonged stirring with $[N(PPh_3)_2]Cl$, under conditions that permit ready deprotonation of compound **3**,[10] leave **2** unchanged. Under these conditions, **4** decomposes.

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