Macropolyhedral boron-containing cluster chemistry. Cluster opening and B-frame rearrangement in the reaction of $B_{16}H_{20}$ with $[\{(IrCl_2(\eta^5\text{-}C_5Me_5)\}_2]$. Synchrotron X-ray structures of $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{17}Cl]$ and $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{15}Cl]$

Michael J. Carr,[a] Sarath D. Perera,[a,b] Tomáš Jelínek,[a,c] Colin A. Kilner,[a] William Clegg,[d,e] Bohumil Štíbr[c] and John D. Kennedy[a]

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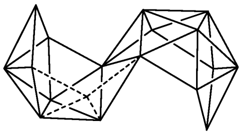
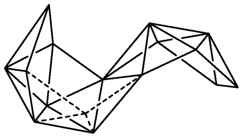
Products from the reaction of + *nido* ten-vertex : *nido* eight-vertex, $B_{16}H_{20}$ 1 with $[\{(IrCl_2(\eta^5\text{-}C_5Me_5)\}_2]$ and tmnd show unanticipated rearrangement of the starting $\{B_{16}\}$ skeleton, as exhibited by + *nido* ten-vertex : *nido* ten-vertex, $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{17}Cl]$ 2 which has a $\{B_2\}$ edge conjunction and by + *nido* ten-vertex : *nido* eleven-vertex, $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{15}Cl]$ 3 which has a $\{B_3\}$ face conjunction.

Extension of the contiguous cluster chemistry of molecular boron hydride species beyond the stable twelve-vertex icosahedral cluster configuration requires either the expansion of single clusters beyond the icosahedron[1,2] or the intimate fusion of two or more clusters to form 'macropolyhedral' species in which the multi-centre cluster-bonding properties extend across the nexus of inter-cluster conjunction.[3] Metallaborane chemistry constitutes a well-examined branch of borane cluster chemistry, and metallaboranes have figured significantly in the development of macropolyhedral borane science. One approach to metallaborane macropolyhedral chemistry derives from the addition of metal centres to pre-formed macropolyhedral binary boranes. The only significant binary borane species previously examined in this context are the *anti* and *syn* isomers of $B_{18}H_{22}$, which engender $\{MB_{18}\}$, $\{M_2B_{18}\}$ and $\{M_3B_{18}\}$ cluster structures in which the starting $B_{18}H_{22}$ configurations are retained, albeit in some cases condensed to varying extents in some reactions products.[3–8] Clusters with $\{MB_{17}\}$ formulations are formed sometimes,[6,7,9] but their eighteen-vertex configurations conform to the two basic $B_{18}H_{22}$ cluster geometries.

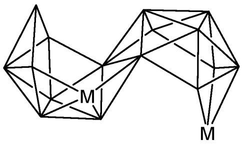
The $B_{18}H_{22}$ isomers have + *nido* ten-vertex : *nido* ten-vertex, constitutions with two boron atoms held in common at the point of fusion (*e.g.* the *anti* isomer, schematic **I**). $B_{16}H_{20}$ (compound **1**) is related to the $B_{18}H_{22}$ isomers in that it has a similar two-borons-in-common fusion, but now the fusion is between *nido* eight-vertex and *nido* ten-vertex subclusters (schematic cluster skeleton **II**; for the significance of the hatched connectivities in **I** and **II**, see below).[10,11] We here report that, in contrast to the $B_{18}H_{22}$ reactions mentioned above, and in contrast to the only other two instances of addition of metal centres to the $B_{16}H_{20}$ skeleton,[11] reaction of $B_{16}H_{20}$ with $[\{(IrCl_2(\eta^5\text{-}C_5Me_5)\}_2]$ in the presence of base results in the formation of macropolyhedral metallaborane species in which there has now been a significant rearrangement of the starting $\{B_{16}\}$ configuration **I**.



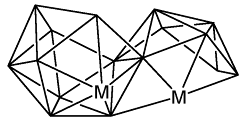
I



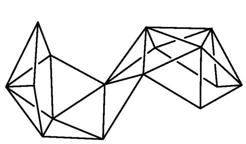
II



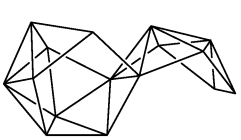
III



IV



V



VI

Thus, reaction of $[\{(IrCl_2(\eta^5\text{-}C_5Me_5)\}_2]$ (165 mg, 210 μmol) with $B_{16}H_{20}$ (compound **1**; 40 mg, 210 μmol) and *N*,*N*,*N*,*N*-tetramethyl-naphthalene-1,8-diamine (tmnd) (90 mg, 420 μmol) in toluene (10 ml) at 100 °C for 3 hours, followed by TLC separation (Silica Gel, Fluka GF_{254}, 50 : 50 CH_2Cl_2–hexane as liquid phase) gave several coloured components, of which we have identified two as of constitutions $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{17}Cl]$ (compound **2**; orange, R_F *ca.* 0.60; 16 mg, 18 μmol, 9%) (Fig. 1, schematic skeleton **III**) and $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{15}Cl]$ (compound **3**; violet, R_F *ca.* 0.30; 3 mg; 3 μmol, 2%) (Fig. 2, schematic skeleton **IV**).[12,13] NMR examination suggested that compound **2** was present in the initial product mixture, although the presence of the low-yield compound **3** was less certain. The molecular structures of both compounds were determined by single-crystal X-ray diffraction

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[a] *The School of Chemistry of the University of Leeds, Leeds, UK LS2 9JT*
[b] *The Chemistry Department of the Open University of Sri Lanka, P.O. Box 21, Nawala, Nugegoda, Sri Lanka*
[c] *The Institute of Inorganic Chemistry of the Academy of Sciences of the Czech Republic, 25068, Řež-by-Prague, The Czech Republic*
[d] *The School of Natural Sciences (Chemistry) of Newcastle University, Newcastle upon Tyne, UK NE1 7RU*
[e] *CCLRC Daresbury Laboratory, Daresbury, Warrington, UK WA4 4AD*

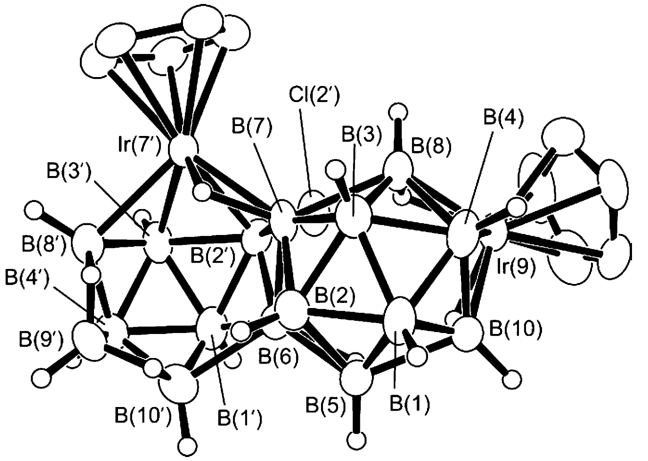


Fig. 1 Crystallographically determined molecular structure of $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{17}Cl]$ **2**,[12] with $\{C_5Me_5\}$ C-methyl groups omitted for simplicity. The two subclusters are fused with two atoms, B(6) and B(7), in common. Selected interatomic distances (Å) are as follows: Ir(7′) to B(2′) 2.193(8), to B(3′) 2.183(9), to B(7) 2.189(9), and to B(8′) 2.268(9); from Ir(9) to B(4) 2.204(10), to B(8) 2.218(8) and to B(10) 2.226(10); with other interboron distances in the range 1.698(12)–1.842(13), except for B(6)–B(10′), B(5)–B(10) and B(7)–B(8), all long at 1.958(15), 1.960(13) and 2.082(12) respectively; Ir(7′)–C(aryl) ranges from 2.218(7) to 2.275(9) and Ir(9)–C(aryl) ranges from 2.178(8) to 2.204(10).

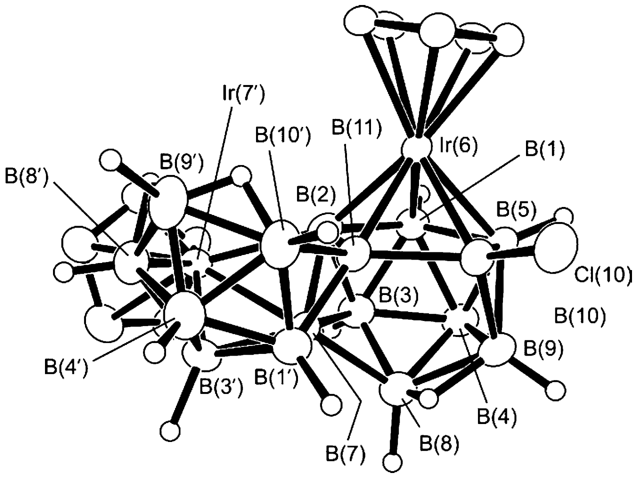
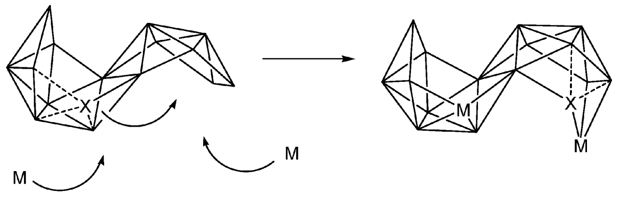


Fig. 2 Crystallographically determined molecular structure of $[(\eta^5\text{-}C_5Me_5)_2Ir_2B_{16}H_{15}Cl]$ **3**,[12] with $\{C_5Me_5\}$ C-methyl groups omitted for simplicity. The overall cluster assembly has two hydrogen atoms fewer, and thence two cluster electrons fewer, than that of compound **2** (Fig. 1), and the two clusters in **3** are fused with three boron atoms, B(2), B(7) and B(11), in common, rather than just two as in **1** and **2**. In general, loss of an electron pair from a macropolyhedral species can result either in the increase of intimacy of inter-cluster fusion (as here), or in a contraction of one of the individual subclusters one step along the *arachno*–*nido*–*closo* sequence.[9] Selected interatomic distances (Å) are as follows: Ir(6) to B(1) 2.211(6), to B(2) 2.132(7), to B(5) 2.215(7), to B(10) 2.156(7) and to B(11) 2.149(6); from Ir(7′) to B(2) 2.259(7), to B(3′) 2.196(6), to B(7) 2.309(6) and to B(8′) 2.204(7); with other interboron distances in the range 1.692(13)–1.864(12), except for B(10)–B(11), B(9)–B(10) and B(7)–B(8), all long at 1.907(9), 1.918(11) and 1.961(10) respectively; Ir(6)–C(aryl) ranges from 2.229(6) to 2.248(6) and Ir(7′)–C(aryl) ranges from 2.212(6) to 2.291(6).

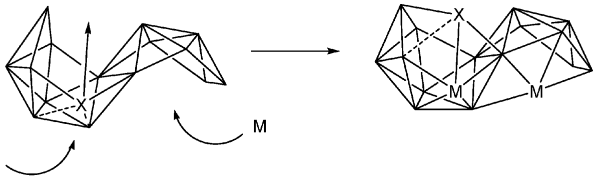
analyses.[12] In both cases, obtainable crystals were very small, and required synchrotron-generated X-rays for sufficient diffraction intensities for crystallographic analyses. Species **2** was additionally characterised by NMR spectroscopy.[13] In this preliminary work, it has not yet been possible to separate compound **3** sufficiently from other components to provide a definitive NMR spectrum, although we were able to isolate small crystalline fragments suitable for the X-ray work. The all-atom molecular structure derived from the diffraction analysis was self-consistent and chemically logical. Both species have previously unrecognised macropolyhedral metallaborane configurations (schematics **III** and **IV**).

Of the two new species, compound **2**, with two *nido* $\{MB_9\}$ ten-vertex units joined with a common $\{B_2\}$ edge, has the recognised overall eighteen-vertex + *nido* ten-vertex : *nido* ten-vertex, configuration of *anti*-$B_{18}H_{22}$ (schematic **I**) that is known to have some stability. This stability may constitute a driving force for the rearrangement. It is clear from the $\{B_{16}\}$ configuration of compound **2** (schematic **V**) that the geometry **II** of the starting $B_{16}H_{20}$ species **1** is broken. The geometrical difference resides essentially in the breaking of three inter-boron connectivities (hatched lines in **II A**) and a movement of one $\{BH(\textit{exo})\}$ centre (designated X in the diagram) from the original *nido* $\{B_{10}\}$ subcluster of $B_{16}H_{20}$ to add to the framework of the original *nido* $\{B_8\}$ subcluster (schematic **III A**).



II A **III A**

A disruption of the starting $B_{16}H_{20}$ skeleton **II** is also observed in compound **3**, of which the metallaborane assembly consists of a *nido* eleven-vertex $\{MB_{10}\}$ sub-cluster and a *nido* ten-vertex sub-cluster conjoined with a common $\{B_3\}$ triangular face (schematic **IV**). The configuration of the starting $B_{16}H_{20}$ substrate is now again disrupted (compare schematics **II** and **VI**). Although a *nido* eight-vertex unit (right-hand side of diagram **VI**) is readily apparent, the original arrangement of the *nido* decaboranyl $\{B_{10}\}$ unit of $B_{16}H_{20}$ is significantly altered, and it has a new 'remote *arachno*' ten-vertex $\{B_{10}\}$ shape. The geometrical change resides in the movement of the same boron atom (designated X) as in the formation of compound **2**, with, now, the breaking of two inter-boron connectivities (hatched lines in **II B**), and a relatively simple movement of the boron vertex within the original $\{B_{10}\}$ sub-unit (schematic **IV A**). In each case (compare schematics **II A** and **II B**) it appears that it is the same boron centre X that moves, with some



II B **IV A**

connections retained (solid lines to X) and some broken (hatched lines to X).

In both of the new compounds **2** and **3** this disruption of the *nido* ten-vertex component of starting **1** is unexpected, because $\{MB_{10}\}$ cluster formation from single-cluster *nido*-$B_{10}H_{14}$ itself has been generally presumed to involve no rearrangement of the $\{MB_{10}\}$ unit. Further, the (few) previously reported $B_{16}H_{20}$ reactions have left the basic $\{B_{16}\}$ framework **II** intact.[11] In this regard, however, it is recently reported that the $\{B_{10}\}$ cluster of *nido*-$B_{10}H_{14}$ itself may undergo rearrangement in its reactions with two-electron donor ligands L that give species [6,9-L_2-*arachno*-$B_{10}H_{12}$],[14] and there are also indications that reactions of $[PtCl_2(PMe_2Ph)_2]$ with halogenated *nido* decaboranes $[B_{10}H_{14-n}X_n]$ to give platinaboranes $[(PMe_2Ph)_2PtB_{10}H_{12-n}X_n]$ similarly also involve *nido*-decaboranyl cluster rearrangement,[15] perhaps *via* an off-open-face metal-centre attack on the *nido* ten-vertex cluster as in schematics **II A** and **II B**. There is also evidence that deprotonated *nido*-$B_{10}H_{14}$ itself undergoes a scrambling among all its boron sites.[16] Deprotonation of $B_{16}H_{20}$ **1** to give the $[B_{16}H_{19}]^-$ anion preferentially occurs from a hydrogen bridge on the smaller, *nido* eight-vertex, subcluster,[11] which may suggest that the first metal centre also attacks here; this would be the right-hand metal centre in schematic structures **II A** and **II B** (we thank a referee for drawing our attention to this point). In this mechanistic context, the incidence of chlorine substituents on **2** and **3** in this present report may imply a redox process rather than a direct displacement of halide by a real or incipient $\{B_{16}H_{19}\}^-$ anion. Also in this context it is interesting that, in some related reactions with the $B_{18}H_{22}$ isomers that yield $\{MB_{17}\}$ products, it is the equivalent vertex, *i.e.* the one next to the point of conjunction, in the $B_{18}H_{22}$ skeletons that is lost completely (hatched connectivities in **I** and **II** above).[7,8] This is also the position that appears to be most susceptible to nucleophilic attack under redox conditions with the formation of ligand species $LB_{18}H_{22}$,[17] and may therefore indicate an activation of this site by the adjacent inter-cluster conjunction.

Preliminary partial separations allied with NMR spectroscopy indicate that this present reaction between $B_{16}H_{20}$ and $[\{(IrCl_2(\eta^5\text{-}C_5Me_5)\}_2]$ engenders additional macropolyhedral species of unprecedented constitution. At the time of writing, these last have proved more difficult to isolate cleanly and thence characterise, but we hope to be able to report the elucidation of these products in due course, as well as to report on other reaction systems involving the sparsely examined $B_{16}H_{20}$ species **1**. Overall, the quite different chemistry (*i.e.* cluster rearrangement) compared to that of the only other (and much more comprehensively examined) macropolyhedral clusters, the $B_{18}H_{22}$ isomers, is remarkable.

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$Z = 4$, synchrotron wiggler-generated radiation as above, $\lambda = 0.6932$ Å, $\mu = 8.472$ mm^{-1}, $T = 150(2)$ K, $R_1 = 0.0409$ for 7769 reflections with $I > 2\sigma(I)$, and $wR_2 = 0.1028$ for all 8826 unique reflections; CCDC 286253. (For crystallographic data in CIF or other electronic format see DOI: 10.1039/b611734a) Methods and programs were standard:; G. M. Sheldrick, *SHELX97*, University of Göttingen, Germany, 1997; G. M. Sheldrick, *SHELXTL Manual*, Bruker AXS Inc., Madison, WI, USA, 1994 and 1998; L. J. Farrugia, ORTEP-3, *J. Appl. Crystallogr.*, 1997, **30**, 565.

13 NMR data. Compound **2**, $CDCl_3$, 294–299 K, ordered as tentative assignment $\delta(^{11}B)$/ppm [$\delta(^{1}H)$/ppm of directly bound H atoms]: BH(8) +24.6 [+5.31], BH(10′) +14.9 [+4.70], BH(8′) +14.9 [+4.24], BH(5) +13.0 [+4.35], B(7) +9.0 [*conjunto* position, no directly bound H(*exo*)], BH(10) +8.1 [+3.99], BH(3) +5.8 [+4.10], B(6) +0.9 [*conjunto* position, no directly bound H(*exo*)], BH(3′) −2.1 [+2.67], BH(1) −4.0 [+3.27], B(2′) −8.0 [bound to Cl(*exo*)], BH(1′) −9.2 [+2.67], BH(9′) −9.2 [+2.20], BH(2) −23.0 [+1.09], BH(4) −23.0 [+0.39], BH(4′) −42.1 [+0.63]; with $\delta(^{1}H)$/ppm for (μ-H) (9′,10′) −0.55, (μ-H) (8′,9′) −2.77, (μ-H) (5,6) −3.16, (μ-H) (9,10) −9.76, (μ-H) (8,9) −10.50 and (μ-H) (7,7′) −14.69; assignments by $\{^{11}B–^{11}B\}$ and $^{1}H–\{^{11}B\}$ correlation work. Compound **3** crystallised from a solution that was too weak and impure to provide unambiguous NMR data.

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