

# Macropolyhedral boron-containing cluster chemistry: nineteen-vertex $[(PPh_3)NiS_2B_{16}H_{12}(PPh_3)]$ and eighteen-vertex $S_2B_{16}H_{14}(PPh_3)$

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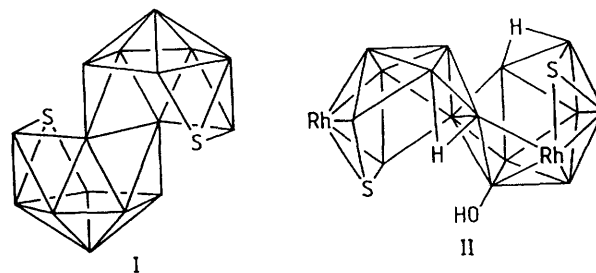
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Reaction of  $S_2B_{16}H_{16}$  and  $[NiBr_2(PPh_3)_2]$  in the presence of base has yielded nineteen-vertex  $[(PPh_3)NiS_2B_{16}H_{12}(PPh_3)]$ , of which the basic cluster structure consists of a *nido*-shaped nine-vertex  $\{1-NiB_8\}$  subcluster and a *closo*-shaped twelve-vertex  $\{1,4-NiSB_{10}\}$  subcluster fused with the  $\{1',3',4':1,2,3\}$   $\{NiB_2\}$  triangular face in common; a related co-product is non-metallated eighteen-vertex  $S_2B_{16}H_{14}(PPh_3)$ , of which the basic cluster structure consists of a *nido*-shaped  $\{B_8\}$  eight-vertex subcluster and a *nido*-shaped  $\{SB_9\}$  eleven-vertex subcluster fused with the  $\{3',8':6,7\}$  diboron edge in common.

There is current interest in larger molecular species derived from the linking together of polyhedral boron-containing single clusters.<sup>1</sup> We have currently renewed our interest in larger molecules derived from polyhedral boron-containing single clusters that are fused together with two or more cluster atoms held in common, the so-called 'macropolyhedral' boron-containing cluster compounds. In this area we have recently reported on macropolyhedral heteroboranes in which the heteroatom is carbon,<sup>2</sup> nitrogen,<sup>3</sup> oxygen<sup>4</sup> or sulfur.<sup>5,6</sup> Heteroborane chemistry, particularly dicarbaborane chemistry,<sup>7</sup> is a well represented area of polyhedral boron-containing cluster chemistry. A rich and interesting well examined subsection of this field is metallaheteroborane chemistry, particularly that of the metalladecaboranes,<sup>8</sup> although metallathaboranes are now receiving increasing attention.<sup>9</sup> It is therefore of interest to examine for any metallaheteroborane chemistry derived from macropolyhedral heteroboranes, particularly by the use of transition-element centres of which the bonding flexibility could result in hitherto unrecognised structural features. We have very recently noted<sup>10</sup> that the reaction of the macropolyhedral dithiaborane  $S_2B_{16}H_{16}$  **1** (structure I)<sup>6</sup> with  $[\{Rh(C_5Me_5)Cl_2\}_2]$  generates, amongst other compounds, two isomers of formulation  $[(C_5Me_5)_2Rh_2S_2B_{15}H_{14}(OH)]$  **2a** and **2b** (of these, schematic cluster structure II depicts the *anti* isomer). In these, substantial rearrangements of the starting thiaborane cluster have occurred, and there is a concomitant loss of a cluster boron atom. Here we now report results from a preliminary investigation of the reaction of  $S_2B_{16}H_{16}$  with the nickel complex  $[NiBr_2(PPh_3)_2]$ . This also exhibits metal-atom incorporation, but now involving retention of all sixteen boron atoms and an interesting structural rearrangement that is quite different from those<sup>10</sup> reported in the rhodathiaborane system.

The complex  $[NiBr_2(PPh_3)_2]$  (966 mg, 1300  $\mu$ mol),  $S_2B_{16}H_{16}$  (329 mg, 1300  $\mu$ mol), and *N,N,N',N'*-tetramethylnaphthalene-1,8-diamine (tmnd) (557 mg, 2600  $\mu$ mol) in toluene (30 cm<sup>3</sup>) were stirred at reflux for 18 h. Removal of solvent, followed by repeated thin-layer chromatography (TLC) (silica gel G, Fluka type GF<sub>254</sub>; eluent  $CH_2Cl_2$ ) thence revealed several coloured components. Repeated thin-layer and column chromatography, and repeated recrystallisation,

have so far rendered only two of these, compounds **3** and **4**, in sufficiently pure form for characterisation. Orange **3**, of TLC  $R_f$  0.06 using benzene-hexane (1:3 v/v) as eluent, was identified by single-crystal X-ray diffraction analysis†

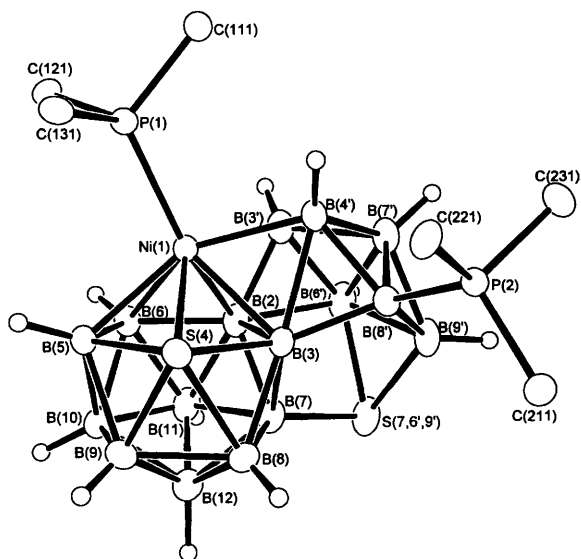


† Crystals of compound **3** were grown by diffusion of hexane into a solution in  $CH_2Cl_2$ , and of **4** by slow evaporation of a solution in  $CHCl_3$ . All measurements were made on a Siemens SMART CCD area-detector diffractometer at 160 K using Mo-K $\alpha$  radiation ( $\lambda = 0.71073$  Å). The structures were solved using SHELXS 86,<sup>11</sup> **3** by heavy-atom methods and **4** by direct methods. They were refined by full-matrix least squares (against all the  $F^2$  data) using SHELXL 93.<sup>12</sup> The non-hydrogen atoms were refined with anisotropic displacement parameters. The asymmetric unit of **3** also contained disordered  $CH_2Cl_2$  solvent. It was not possible to resolve the disorder and so the highest peaks in the difference map were refined as carbon atoms of partial occupancy with isotropic displacement parameters. In both cases the hydrogen atoms associated with the phosphine ligand were constrained to idealised positions, whereas those associated with the cluster were located on Fourier-difference maps and freely refined.

Compound **3**:  $C_{36}H_{43}B_{16}NiP_2S_2 \cdot CH_2Cl_2$ , crystal dimensions  $0.42 \times 0.28 \times 0.10$  mm, monoclinic, space group  $P2_1/c$ ,  $a = 11.4623(8)$ ,  $b = 17.2012(13)$ ,  $c = 23.858(2)$  Å,  $\beta = 92.972(2)^\circ$ ,  $Z = 4$ ,  $U = 4697.7(6)$  Å<sup>3</sup>,  $D_c = 1.229$  g cm<sup>-3</sup>. 24 327 Reflections were collected to  $\theta = 25.0^\circ$ ; 8276 unique reflections were used in calculations after Lorentz, polarisation and absorption corrections ( $\mu = 0.604$  mm<sup>-1</sup>; based on repeated and symmetry-equivalent reflections, transmission factors 0.732–0.80). Final  $wR_2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum(F_o^2)^2\}^{1/2} = 0.1288$  for all data, conventional  $R = 0.0448$  on  $F$  values of 6761 reflections having  $F_o^2 > 2.0\sigma(F_o^2)$ , goodness of fit = 1.157 on all  $F^2$  values with 615 refined parameters. Maximum and minimum residual electron density 0.87 and  $-0.49$  e Å<sup>-3</sup>.

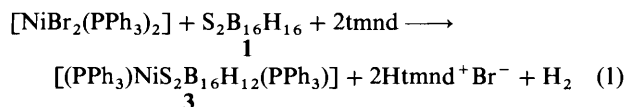
Compound **4**:  $C_{18}H_{29}B_{16}PS_2$ ,  $M_r = 513.46$ , crystal dimensions  $0.42 \times 0.40 \times 0.08$  mm, monoclinic, space group  $P2_1/n$ ,  $a = 9.5511(10)$ ,  $b = 17.790(2)$ ,  $c = 15.955(2)$  Å,  $\beta = 100.806(2)^\circ$ ,  $Z = 4$ ,  $U = 2662.9(5)$  Å<sup>3</sup>,  $D_c = 1.28$  g cm<sup>-3</sup>. 16 481 Reflections were collected to  $\theta = 28.53^\circ$ ; 6126 unique reflections were used in calculations after Lorentz, polarisation and absorption corrections ( $\mu = 0.271$  mm<sup>-1</sup>; transmission factors 0.713–0.899). Final  $wR_2 = 0.1339$  for all data, conventional  $R = 0.0486$  on  $F$  values of 4578 reflections with  $F_o^2 > 2.0\sigma(F_o^2)$ , goodness of fit = 1.082 on all  $F^2$  values with 390 refined parameters. Maximum and minimum residual electron density 0.40 and  $-0.43$  e Å<sup>-3</sup>.

Atomic coordinates, thermal parameters, and bond lengths and angles have been deposited at the Cambridge Crystallographic Data Centre (CCDC). See Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1996, Issue 1. Any request to the CCDC for this material should quote the full literature citation and the reference number 186/284.



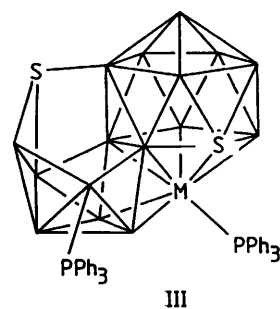
**Fig. 1** An ORTEX<sup>13</sup> diagram of [(PPh<sub>3</sub>)NiS<sub>2</sub>B<sub>16</sub>H<sub>12</sub>(PPh<sub>3</sub>)] **3**. Ellipses are drawn at the 30% probability level. For clarity all phenyl atoms other than the *ipso*-carbons have been omitted, and hydrogen atoms are drawn as circles with an arbitrarily small radius. Selected interatomic distances (Å): Ni(1)–B(2) 1.970(2), Ni(1)–B(4') 2.098(2), Ni(1)–B(3') 2.130(3), Ni(1)–B(3) 2.137(2), Ni(1)–B(5) 2.144(2), Ni(1)–B(6) 2.148(2), Ni(1)–P(1) 2.2273(6), Ni(1)–S(4) 2.2535(6), S(4)–B(3) 1.968(2), S(4)–B(9) 1.996(3), S(4)–B(8) 2.052(3), S(4)–B(5) 2.065(3), S(7,6',9')–B(7) 1.874(3), S(7,6',9')–B(9') 1.895(3), S(7,6',9')–B(6') 2.027(3), B(2)–B(3) 1.945(3), B(2)–B(6') 2.027(4), B(2)–B(3) 1.945(3), B(3)–B(8') 1.737(3) and B(6')–B(9') 1.960(4)

(Fig. 1) and additionally characterised by NMR spectroscopy ‡ as of formulation [(PPh<sub>3</sub>)NiS<sub>2</sub>B<sub>16</sub>H<sub>12</sub>(PPh<sub>3</sub>)] (15 mg, 26 μmol, 2%). A simple stoichiometric equation (1) can be written for its formation from the starting materials, although, in view of the small yield and other products, the reaction pathway could be more complex than this may imply.

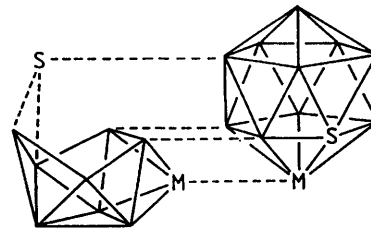


The cluster structure of compound **3** (schematic **III**) is based on a *closo* twelve-vertex {NiSB<sub>10</sub>} unit (structure **IIIB**) and a *nido* nine-vertex {NiB<sub>8</sub>} unit (**IIIA**) fused with a closed triangular {NiB<sub>2</sub>} face in common. In addition, there is an unusual intercluster cross-link in which a sulfur atom bridges *endo* from two boron atoms on the open face of the *nido* nine-vertex subcluster and *exo* to a boron atom on the *closo* twelve-vertex subcluster. The basic fusion mode, involving a common three-atom deltahedral unit, is of interest because most known macropolyhedral species, as typified by the binary boranes B<sub>12</sub>H<sub>16</sub>,<sup>14</sup> B<sub>13</sub>H<sub>19</sub>,<sup>15</sup> B<sub>14</sub>H<sub>18</sub>,<sup>16</sup> B<sub>14</sub>H<sub>20</sub>,<sup>17</sup> B<sub>16</sub>H<sub>20</sub>,<sup>18</sup> and the two isomers of B<sub>18</sub>H<sub>22</sub>,<sup>19,20</sup> exhibit an intercluster fusion mode that has two atoms in common. The positioning of the metal atom in compound **3** is somewhat reminiscent of the 'wedge' feature in species such as [(PEt<sub>3</sub>)<sub>2</sub>CoFe(Me<sub>2</sub>C<sub>2</sub>B<sub>4</sub>H<sub>4</sub>)<sub>2</sub>], [(thf)<sub>2</sub>Fe<sub>2</sub>(Me<sub>2</sub>C<sub>2</sub>B<sub>4</sub>H<sub>4</sub>)<sub>2</sub>] (thf = tetrahydrofuran) and [(tmen)Mn<sub>2</sub>{B<sub>4</sub>H<sub>4</sub>C<sub>2</sub>(SiMe<sub>3</sub>)<sub>2</sub>}<sub>2</sub>] (tmen = Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>-

‡ In CDCl<sub>3</sub> at 294–297 K: δ(<sup>11</sup>B) (relative to F<sub>3</sub>B·OEt<sub>2</sub>) [δ(<sup>1</sup>H) of directly bound *exo*-hydrogen atoms] +23.6 [+5.84 (quartet fine structure in <sup>1</sup>H-<sup>11</sup>B spectrum)], +19.3 [+4.43], +17.8 [+3.68], +6.1 [no *exo*-H], +2.3 [+2.87], -3.2 [+3.25], -4.2 [no *exo*-H], -6.5 [+2.55], -7.6 [no *exo*-H], -10.2 [+0.93], -12.6 [+2.00], -14.4 [+1.85], -15.6 [+1.70], -16.8 [+1.26], -18.0 [+1.60] and -35.7 [no *exo*-H; possible PPh<sub>3</sub> substituent site]. Additionally δ(<sup>31</sup>P) (relative to 85% H<sub>3</sub>PO<sub>4</sub>) (233 K) +18.0 (br) and +27.7 (sharp).

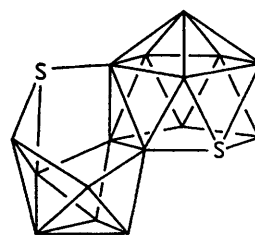


**III**

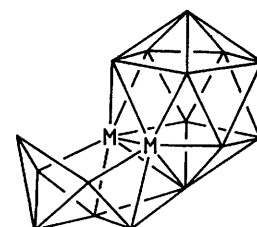


**IIIA**

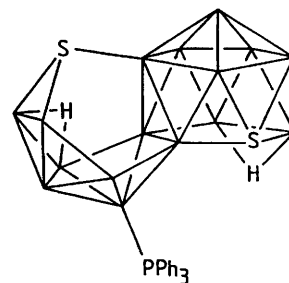
**IIIB**



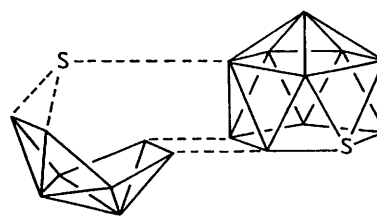
**IIIC**



**V**



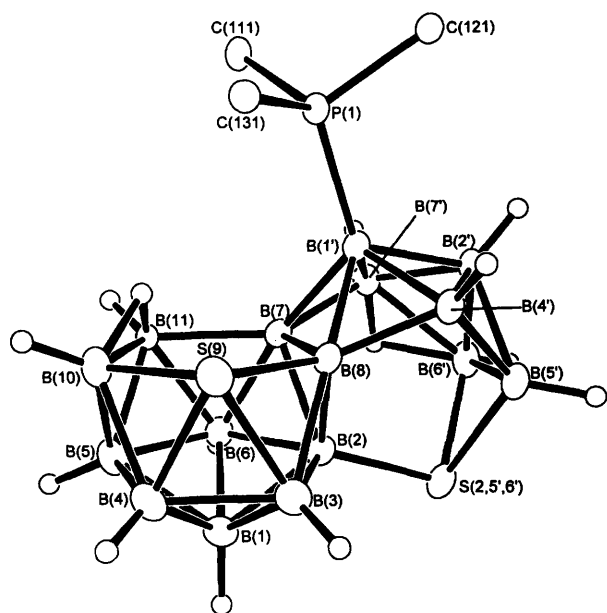
**IV**



**IVA**

**IVB**

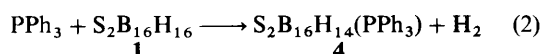
NMe<sub>2</sub>),<sup>21–23</sup> which are otherwise based on the fusion of two metallacarborane clusters with just one (metal) atom held in common. The basic cluster structure of the nickeladithiaborane **3** is however much more closely related to that of the *nido:closo* diplatinoiridaborane [(PMe<sub>2</sub>Ph)<sub>2</sub>Pt<sub>2</sub>B<sub>16</sub>H<sub>15</sub>(C<sub>6</sub>H<sub>4</sub>Me)(PMe<sub>2</sub>Ph)] (schematic cluster structure **V**),<sup>24</sup> although here the metal and boron occupancies of the sites in the common triangular unit are interchanged. There is also a close similarity to the configuration of the recently reported non-hetero metallaborane [(CO)<sub>2</sub>(PMe<sub>3</sub>)<sub>4</sub>Ir<sub>2</sub>B<sub>16</sub>H<sub>14</sub>].<sup>25</sup> Although an eighteen-atom contiguity of the {S<sub>2</sub>B<sub>16</sub>} framework is conserved in **3**, the final {S<sub>2</sub>B<sub>16</sub>} connectivity **IIIc** in this product does not have a



**Fig. 2** An ORTEX  $^{13}\text{C}$  diagram of  $\text{S}_2\text{B}_{16}\text{H}_{14}(\text{PPh}_3)$  **4**. Details as in Fig. 1. Selected interatomic distances (Å): B(3)–S(9) 2.044(3), B(4)–S(9) 2.011(3), B(8)–S(9) 1.945(3), S(9)–B(10) 1.977(3), B(2)–S(2,5',6') 1.886(3), B(5')–S(2,5',6') 1.936(3), B(6')–S(2,5',6') 1.963(3), B(7)–B(11) 1.839(3), B(10)–B(11) 1.851(4), B(7)–B(7') 1.790(4), B(8)–B(4') 1.724(4), B(5')–B(6') 1.899(4) and B(6')–B(7') 1.861(4)

straightforward relationship to that in the  $\text{S}_2\text{B}_{16}\text{H}_{16}$  starting substrate **1** (structure I).

It does, however, have a closer resemblance to a second product isolated from this reaction, the non-metallated dithiaborane compound **4**. Amber **4**, of TLC  $R_f$  0.85 using  $\text{CH}_2\text{Cl}_2$ –hexane (3:2 v/v) as eluent, was identified by single-crystal X-ray diffraction analysis (Fig. 2) to be of formulation  $\text{S}_2\text{B}_{16}\text{H}_{14}(\text{PPh}_3)$  (4 mg, 26  $\mu\text{mol}$ , 2%). It was additionally characterised by NMR spectroscopy. It could derive from  $\text{PPh}_3$  originating from  $[\text{NiBr}_2(\text{PPh}_3)_2]$ , as summarised in equation (2). This cluster configuration of compound **4**



(schematic IV) also has the intercluster sulfur link, but now with the presently more common  $^{14-20}$  two-atom intercluster fusion mode. It is related to the  $\{\text{S}_2\text{B}_{16}\}$  fragment of **3** (structure IIIc) by the making of one interboron connectivity in a square-to-diamond process, and the breaking of one interboron connectivity, but again it is not clear how it may derive from the  $\text{S}_2\text{B}_{16}\text{H}_{16}$  structure I. It could be that the other reaction products exemplify steps along the necessary reaction coordinate. In view of this and the several interesting structural features of compounds **3** and **4**, which suggest a rich general macropolyhedral metallaheteroborane chemistry, there is merit in characterising other products from this reaction, and we are currently attempting to purify and identify them.

## Acknowledgements

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§ In  $\text{CDCl}_3$  at 294–297 K:  $\delta(^{11}\text{B})$  [ $\delta(^1\text{H})$  of directly bound *exo*-hydrogen atoms] + 14.7 [+4.08], +10.1 [no *exo*-H], +1.4 [no *exo*-H], +0.6 [+2.67], –3.0 [+2.94], –4.5 [+2.02], –5.3 [+2.49], –6.7 [no *exo*-H; doublet,  $^1J(^{31}\text{P}-^{11}\text{B})$  ca. 160 Hz], –7.4 [no *exo*-H], –9.6 [no *exo*-H], –19.0 [+1.52], –20.0 [+2.14], –24.9 [+1.92], –27.0 [+1.21], –29.9 [+1.02], –30.9 [+0.66], –42.2 [+0.99].  $\delta(^1\text{H})$  –0.88 and –2.33 (both  $\mu\text{-H}$ );  $\delta(^{31}\text{P})$  (at 233 K) –4.9 (br).

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