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Macropolyhedral boron-containing cluster chemistry. $[PtMe_2(PMe_2Ph)_2]$ as a cluster metallating agent. Isolation and characterisation of nineteen-vertex $[(PMe_2Ph)HPt\text{-}\eta^4\text{-}syn\text{-}B_{18}H_{19}(PMe_2Ph)]$ and eighteen-vertex $[(PMe_2Ph)_2PtS_2B_{15}H_{14}(NHCOMe]$[1]

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Abstract

$[PtMe_2(PMe_2Ph)_2]$ reacts quantitatively with $[nido\text{-}B_{10}H_{14}]$, $[arachno\text{-}4\text{-}SB_8H_{12}]$ and $[nido\text{-}7\text{-}SB_{10}H_{12}]$ to give $[(PMe_2Ph)_2PtB_{10}H_{12}]$, $[(PMe_2Ph)_2PtSB_8H_{10}]$ and $[(PMe_2Ph)_2PtSB_{10}H_{10}]$. With $[syn\text{-}B_{18}H_{22}]$ it gives nineteen-vertex $[(PMe_2Ph)HPt\text{-}\eta^4\text{-}syn\text{-}B_{18}H_{19}(PMe_2Ph)]$[1] and only smaller amounts of $[(PMe_2Ph)_2Pt\text{-}\eta^4\text{-}syn\text{-}B_{18}H_{20}]$, whereas with $[(anti)\text{-}9,9'\text{-}S_2B_{16}H_{16}]$ a complex product mixture is generated from which eighteen-vertex $[(PMe_2Ph)_2PtS_2B_{15}H_{14}(NHCOMe)]$[1] is isolatable after treatment with MeCN in air.

Keywords: Macropolyhedral; Metallaboranes; Platinum

The horizons of boron-containing cluster chemistry are extended considerably by the recognition that the simple single-cluster borane and heteroborane building blocks can be fused with common edges or faces to generate extended contiguous 'macropolyhedral' cluster species [1]. An important sub-discipline of boron-containing single-cluster chemistry is that of the metallaheteroboranes, which has hitherto been dominated by the carbametallaboranes [2]. However, there is now increasing interest and activity in other metallaheteroboranes, particularly thiametallaboranes [3–10]. Recent reports on a variety of thiametallaboranes describe $\{MSB_8\}$, [3] $\{MSB_9\}$, [3–6] $\{MSB_{10}\}$, [7] $\{MS_2B_6\}$, [8] $\{MS_2B_7\}$, [9,10] $\{MS_2B_8\}$, [6] $\{MS_2B_9\}$, [6] and $\{M_2S_2B_7\}$ [9] single-cluster species. It is of interest to extend metallaheteroborane chemistry into the macropolyhedral area. Here the macropolyhedral dithiaborane substrate $[(anti)\text{-}9,9'\text{-}S_2B_{16}H_{16}]$ (schematic cluster structure **III**) [11] is a convenient entry into this field, but reactions of transition-element halide complexes in the presence of base have so far given considerable cluster rearrangements, [12] and we sought a milder cluster metallating reagent to help explore the field more systematically. We now report preliminary results from the reaction between $[S_2B_{16}H_{16}]$ and the organoplatinum species $[PtMe_2(PMe_2Ph)_2]$ to yield the macropolyhedral dithiaplatinaborane $[(PMe_2Ph)_2PtS_2B_{15}H_{14}(NHCOMe)]$ (schematic cluster structure **IV**). In general we have found that $[PtMe_2(PMe_2Ph)_2]$ is a useful reagent for the platination of many polyhedral

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[1] 4,9-bis(dimethylphenylphosphine)-9-(hydrido)-*nido*-decaborano-(5′,6′:7,8)-*nido*-9-platinaundecaborane (compound **2**) and 10,10-bis(-dimethylphenylphosphine)-7-(acetamid-N-yl)-*nido*-9′-thiadecaborano-(5′,6′:6,7)-*nido*-9,10-thiaplatinadecaborane (compound **4**).

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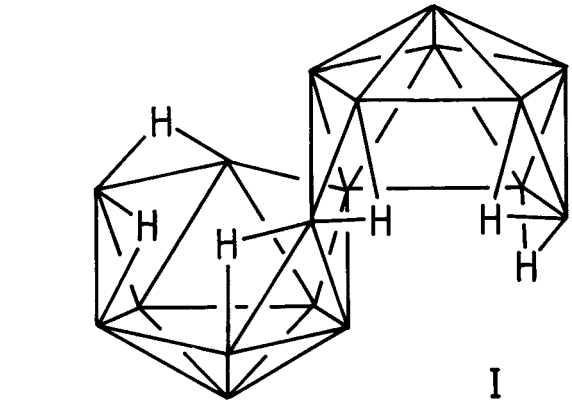
boron-containing cluster species. A similar elimination of methane has previously been employed for metallaborane synthesis using [AuMe(PPh$_3$)] as a reagent [13]. As part of this work, we also find that a convenient new alternative to the classical Grignard route for the preparation of [PtMe$_2$(PMe$_2$Ph)$_2$] is to use the reaction of methyllithium in toluene solution, which is commercially available[2]



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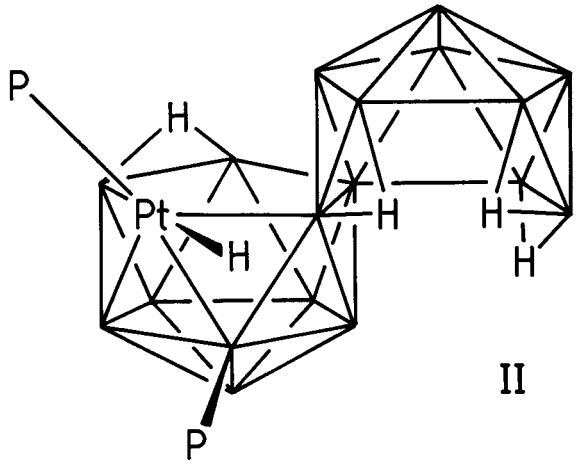

II

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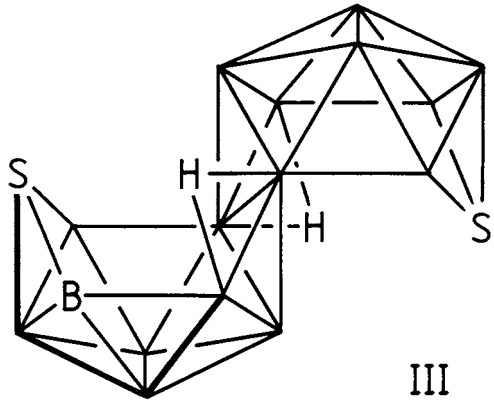
Many boron-containing cluster compounds with open-face bridging and/or *endo* hydrogen atoms are Brønsted acids. With single-cluster species in this category we find that [PtMe$_2$(PMe$_2$Ph)$_2$] is often an excellent metallaborane synthon under mild conditions. For example, it reacts quantitatively with [*nido*-$B_{10}H_{14}$], [*arachno*-4-SB_8H_{12}] and [*nido*-7-$SB_{10}H_{12}$] in neutral solvents at 20–50°C to give recognised species of known type, *viz*. *nido*-type [(PMe$_2$Ph)$_2$Pt$B_{10}H_{12}$], [14] *arachno*-type [(PMe$_2$Ph)$_2$PtSB_8H_{10}] [15] and *closo*-type [(PMe$_2$Ph)$_2$Pt$SB_{10}H_{10}$] [7], respectively, *via* methane loss. This reaction is effectively a simple replacement of two acidic bridging hydrogen atoms by the {Pt(PMe$_2$Ph)$_2$} moiety. We intend to report more fully on the generality of this reaction in due course. In an extension of this reaction to the well-known macropolyhedral substrate *syn*-$B_{18}H_{22}$ (compound **1**, schematic structure **I**), by contrast, the reaction is not such a simple replacement of two bridging hydrogen atoms by the {Pt(PMe$_2$Ph)$_2$} moiety to give known [16] [(PMe$_2$Ph)$_2$Pt-η^4-*syn*-$B_{18}H_{20}$]. Instead, in C_6D_6 solution at room temperature on a reaction scale of *ca*. 45 μmol, the new nineteen-vertex cluster compound [(PMe$_2$Ph)HPt-η^4-*syn*-$B_{18}H_{19}$(PMe$_2$Ph)] (compound **2**, schematic cluster structure **II**) is the predominant product (45%). Orange air-stable compound **2** is characterised by NMR spectroscopy[3] and by single-crystal X-ray diffraction analysis (Fig. 1)[4]. A much smaller quantity (13%) of the otherwise expected isomer [(PMe$_2$Ph)$_2$Pt-η^4-*syn*-$B_{18}H_{20}$] [16] is formed. The different course of reaction undergone by the fused-cluster macropolyhedral *nido*-decaborano-*nido*-decaborane [$B_{18}H_{22}$], compared to that of the single-cluster *nido*-decaborane [$B_{10}H_{14}$], is of interest. A differential reactivity has recently been noted also for reactions with alkyl isocyanides [11,17]

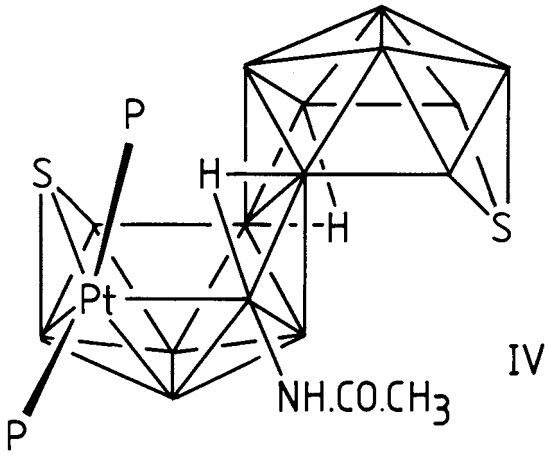
[2] Preparation of [PtMe$_2$(PMe$_2$Ph)$_2$]: A suspension of [PtCl$_2$(PMe$_2$Ph$_2$)] in dry toluene (12 ml) is cooled to −78°C and a solution of methyllithium in diethylether (Aldrich, 1.55 M, 2.84 ml; 4.40 mmol MeLi) is added. The mixture is stirred at −78°C for 2 h and then water (10 ml) added (still at −78°C) to give a pale brown precipitate. Additional toluene (15 ml) is added, the mixture filtered at room temperature and the layers separated. The toluene layer is dried with $MgSO_4$ and the solvents evaporated under reduced pressure. Dissolution of the oily residue in minimum diethyl ether, addition of pentane, and storage at ca. +4°C overnight gives white crystals of [PtMe$_2$(PMe$_2$Ph)$_2$] (600–650 mg; ca. 80%).

[3] Crystallographic results for [(PMe$_2$Ph)HPt-η^4-*syn*-$B_{18}H_{19}$(PMe$_2$Ph)] (compound **2**). Crystals were obtained from dichloromethane/hexane. All measurements were made on a Stoe STADI 4 diffractometer using graphite-monochromated Mo–K_α radiation ($\lambda = 0.71073$ Å) and ω-θ scans, with 3838 reflections collected up to 50° in 2θ. An absorption correction was applied based on azimuthal Ψ scans (max. and min. transmission factors 0.1104 and 0.0639, respectively). The structure was solved by heavy-atom methods (SHELXS-86) [20] and was refined anisotropically by full-matrix least-squares analysis based on all unique F^2 (SHELXL-93) [21]. Phenyl rings were restrained to be of C_{2v} symmetry. Ligand hydrogen atoms were included with a riding model; cluster-associated hydrogen atoms were refined freely. The complex was found to have crystallised as a racemic twin and was accordingly refined as such. The final $wR(F_2)$ for 3363 unique reflections was 0.0732 with a conventional $R(F)$ of 0.0275 [for 3259 reflections with $I > 2.0\sigma(I)$] for 418 parameters. Crystal data: $C_{16}H_{42}B_{18}P_2Pt$. $M = 686.11$, $0.58 \times 0.45 \times 0.30$ mm orthorhombic, space group $P2_12_12_1$, $a = 8.398(3)$ Å, $b = 16.659(4)$ Å, $c = 22.197(6)$ Å, $U = 6601.8(16)$ Å^3, $Z = 4$, $D_c = 1.486$ Mg m^{-3}, $F(000) = 1344$, μ(Mo–K_α) = 4.631 mm^{-1}, $T = 210$ K.

[4] NMR data for [(PMe$_2$Ph)HPt-η^4-*syn*-$B_{18}H_{19}$(PMe$_2$Ph)] (compound **2**), $CDCl_3$ solution at 294–297 K, ordered as δ(^{11}B) [δ(^{1}H) for directly bound hydrogen]: *ca*. +12.8 {+3.49], *ca*. +12.8 [+3.58], *ca*. +10.4 [-], *ca*. +3.8 [+3.11], *ca*. +3.8 [+2.82], *ca*. +0.7 [+2.93], *ca*. +0.7 [+2.96], *ca*. +0.7 [+3.68], *ca*. −3.2 [+2.93], *ca*. −3.2 [+2.28], *ca*. −3.3 [+2.28], −5.7 [-], −7.8 [PMe$_2$Ph site, ^1J(^{31}P–^{11}B) *ca*. 130 Hz, ^1J(^{195}Pt–^{11}B) *ca*. 190 Hz], −15.1 (broad) [+2.34], −24.1 [−0.38], −27.6 [−0.06], −35.4 [+0.99], −40.9 [+0.16]; additionally δ(^{1}H)(μH) −0.07 and −0.72, −2.18 and −3.47, δ(^{1}H)(PtH) −2.50 [^1J(^{195}Pt–^{1}H) 519 Hz, ^2J(^{31}P–^{1}H) 31 Hz]; δ(^{31}P)(223 K) −6.3 (sharper) [^1J(^{195}Pt–^{31}P) 3170 Hz] and −11.9 (broader) [^1J(^{31}P–^{11}B) *ca*. 130 Hz].



III



IV

With the eighteen-vertex macropolyhedral dithiaborane [(*anti*)-9,9′-$S_2B_{16}H_{16}$] (compound **3**, schematic cluster structure **III**) the reaction is more complex. Reaction between [$S_2B_{16}H_{16}$] and [$PtMe_2(PMe_2Ph)_2$] in CH_2Cl_2 for 18 h at room temperature, followed by chromatography on silica in air using MeCN as liquid phase, revealed several coloured reaction products in low yield. Of these the only product that we have so far been able to purify (by repeated chromatography) in sufficient quantity for assessment (*ca.* 4%; reaction scale *ca.* 200 μmol) is the purple air-stable eighteen-vertex dithiaplatinaborane [$(PMe_2Ph)_2$-$PtS_2B_{15}H_{14}$(NHCOMe)] (compound **4**, schematic cluster structure **IV**). This is characterised by single-crystal X-ray diffraction analysis (Fig. 2)[5] and NMR spectroscopy[6]. The cluster unit of this new compound has the eighteen-vertex *nido*-decaborano-*nido*-decaborane macropolyhedral structure of the starting substrate [$S_2B_{16}H_{16}$] (schematic cluster structure **III**) except that it has a {$Pt(PMe_2Ph)_2$} moiety as a cluster constituent at the 10-position instead of a {BH} unit (schematic cluster structure **IV**), and that it has gained an acetamido group at the B(5) position.

Compound **4** has several features of interest that suggest interesting future macropolyhedral chemistries. In contrast to compound **2**, it arises from an effective boron-vertex replacement to give an eighteen-vertex cluster, rather than metal-vertex addition to give a nineteen-vertex product. Further, the observed cluster-atom disposition in compound **4** suggests it does not arise from simple open-face metal-vertex addition followed by boron-vertex elimination. Rather, the effective displacement of the boron vertex B(10) (vertex B in schematic **III**) by the platinum centre (schematic **IV**) suggests an off-open-face attack in the {S(9)B(4)B(1)B(5)} region (heavier lines in structure **III**). The site of the acetamido substituent on the *nido*-decaborano-*nido*-decaborane eighteen-vertex unit is adjacent to the cluster fusion linkage, equivalent to that of the phosphine ligand in compound **2**. It is also the same as the ligand site in [(ligand)$B_{18}H_{20}$] species that can be formed from [*anti*-$B_{18}H_{22}$] with two-electron ligands in the presence of oxidizing agents [17,18]. These common sites of attack suggest a quite specific activation of this boron position on this type of cluster. In order to form the acetamido substituent at this position in compound **4**, ligand attack by MeCN solvent has presumably occurred, followed by hydrolysis either on silica or in air. There also seems to be a driving force to retain the *nido*-decaborano-*nido*-decaborane eighteen-vertex macropolyhedral shape. This therefore may have a particular stability, as noted elsewhere in carbaborane [18] and in azaborane [19] macropolyhedral systems. We currently continue our investigations of the reactions of [$S_2B_{16}H_{16}$] with other transition-element systems, as well as attempting to isolate additional products from its reaction with [$PtMe_2(PMe_2Ph)_2$] reported here.

[5] Crystallographic results for [$(PMe_2Ph)_2PtS_2B_{15}H_{14}$(NHCOMe)] (compound 4): Crystals were obtained from dichloromethane/hexane. Data collection was as for compound **2**, but for 12167 reflections, $2\theta_{max} = 50°$, with max. and min. transmission factors 0.6917 and 0.5101, respectively. Structure solution and refinement were similar to compound **2**. The final $wR(F^2)$ for 5830 unique reflections was 0.0654 with a conventional $R(F)$ of 0.0264 [for 4736 reflections with $I > 2.0\sigma(I)$] for 422 parameters. Crystal data: $C_{18}H_{40}B_{15}NOP_2PtS_2$. M = 769.81, $0.61 \times 0.53 \times 0.38$ mm, monoclinic, space group $I2/a$, $a = 24.626(3)$ Å, $b = 9.6266(15)$ Å, $c = 28.261(4)$ Å, $\beta = 99.802(9)°$, $U = 6601.8(16)$ Å^3, $Z = 8$, $D_c = 1.549$ Mg m^{-3}, $F(000) = 3024$, $\mu(Mo-K_\alpha) = 4.492$ mm^{-1}, $T = 220$ K.

[6] NMR data for [$(PMe_2Ph)_2PtS_2B_{15}H_{14}$(NHCOMe)] (compound **4**), $CDCl_3$ solution at 294–297 K, ordered as δ(11B) [δ(1H) for directly bound hydrogen]: +26.7 [+4.96], +9.6 [+3.89], +5.2 [+3.36], −3.7 [+4.49, $^nJ(^{195}Pt-^1H)$ ca. 100 Hz, $^nJ(^{31}P-^1H)$ ca. 20 Hz (doublet)], −5.4 [-], −8.3 [-], −10.8 [2.23], −10.8 [−1.67], −11.9 [-], −12.1 [+1.11], −16.0 [+1.35], −17.2 [+1.24], −25.7 [+1.08], −29.1 [+0.69], −34.0 [+1.64]; additionally $\delta(^1H)(\mu H)$ +0.07 and −1.67, $\delta(^1H)$(NH) +5.80, $\delta(^1H)(COCH_3)$ +2.21; $\delta(^{31}P)$(223 K) −5.3 [$^1J(^{195}Pt-^{31}P)$ 2930 Hz] and −7.2 [$^1J(^{195}Pt-^{31}P)$ 3423 Hz], $^2J(^{31}P-^{31}P)$ 27 Hz.

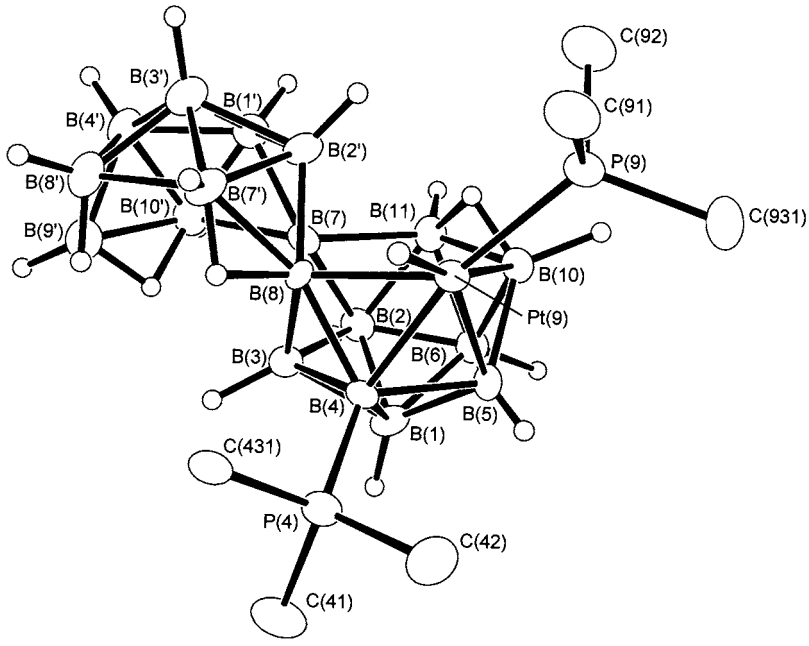


Fig. 1. ORTEX [22] drawing of $[(PMe_2Ph)HPt\text{-}\eta^4\text{-}syn\text{-}B_{18}H_{19}(PMe_2Ph)]$ (compound **2**) with P-organyl atoms, except the *ipso* carbon ones, omitted for clarity. For convenience of presentation, structure **II** and this drawing show different enantiomers. The compound differs from the previously reported conventional isomer by the interchange of a hydride unit and a phosphine ligand between the B(4) and Pt(9) positions. Selected interatomic dimensions (in pm) are Pt(9)–B(4) 218.6(7), Pt(9)–B(5) 222.2(9), Pt(9)–B(8) 223.9(8), Pt(9)–B(10) 235.4(9) and Pt(9)–P(9) 228.2(2). There is a close intercluster platinum–hydrogen contact, Pt(9)–H(2′), of 295(13), the associated Pt(9)–B(2′) distance being 293(1). The P(9)–Pt(9)–H(9) angle is 80(5)°. The common boron–boron edge, B(7)–B(8), is 184.8(11) and there are characteristically 'long' *nido* ten-vertex 'gunwale' open-face interboron distances B(7)–B(11), B(7′)–B(8′) and B(7)–B(10′) of 199.6(12) 195.6(12) and 200.10(12), respectively.

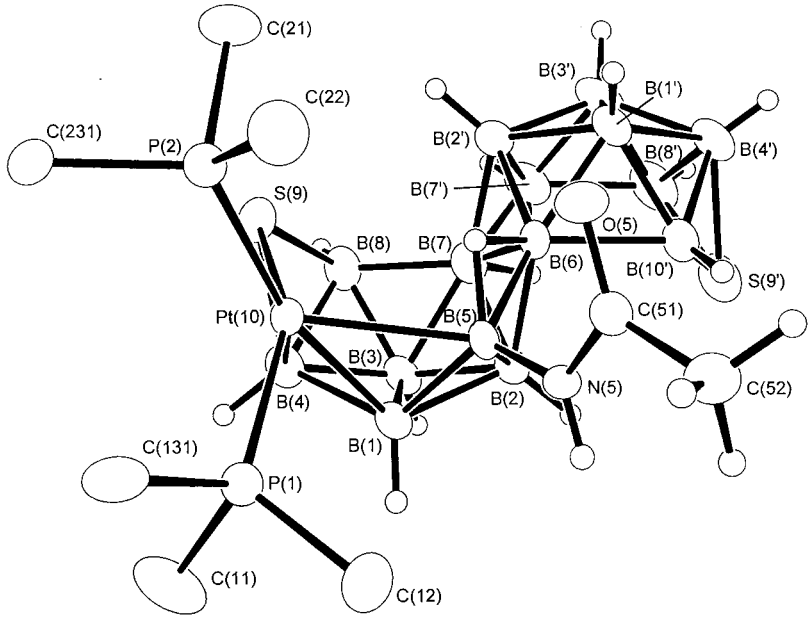


Fig. 2. ORTEX [22] drawing of $[(PMe_2Ph)_2PtS_2B_{15}H_{14}(NHCOMe)]$ (compound **4**), with P-organyl atoms, except the *ipso* carbon ones, omitted for clarity. Selected interatomic dimensions (in pm) are Pt(10)–S(9) 232.2(1), Pt(10)–B(8) 223.2(5), Pt(10)–B(4) 238.9(5), Pt(10)–B(5) 239.1(4), Pt(10)–P(1) 227.9(10) and Pt(10)–P(2) 231.1(1). The P(1)–Pt(10)–P(2) angle is 97.33(4)°. The common boron–boron edge, B(6)–B(7), is 178.4(6). No long 'gunwale' open-face interboron distances (contrast compound **2**) are now evident, with B(7)–B(8), B(7′)–B(8′) and B(6)–B(10′) at 179.2(7), 185.8(9) and 188.3(6), respectively. Sulphur–boron distances are: S(9)–B(4) 196.5(6), S(9)–B(8) 188.6(5), S(9′)–B(4′) 195.3(6), S(9′)–B(8′) 188.7(8) and S(9′)–B(10′) 186.3(5).

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References

[1] See, for example, (and references therein): T. Jelínek, J.D. Kennedy, B, Štíbr, M. Thornton-Pett, Angew. Chem., Int. Edn. Engl. 33 (1994) 1599; L. Barton, J. Bould, J.D. Kennedy, N.P. Rath, J. Chem. Soc. Dalton Trans. (1996) 3145; J.D. Kennedy, in: W. Siebert (Ed.), Advances in Boron Chemistry, R. Soc. Chem., Cambridge, UK, 1997, pp. 451–462.

[2] A.K. Saxena, N.S. Hosmane, Chem. Rev. 93 (1993) 1081; R.N. Grimes, in: G. Wilkinson, F.G.A. Stone, E. Abel (Eds.), Comprehensive Organometallic Chemistry, Pergamon, Oxford, UK, 1982, pp. 459–542.

[3] M. Bown, X.L.R. Fontaine, N.N. Greenwood, J.D. Kennedy, Z. Anorg. Allgem. Chem. 602 (1991) 17.

[4] G. Ferguson, M.C. Jennings, A.J. Lough, S. Coughlan, T.R. Spalding, J.D. Kennedy, X.L.R. Fontaine, B. Štíbr. J. Chem. Soc. Chem. Commun. (1990) 891.

[5] K. Nestor, X.L.R. Fontaine, N.N. Greenwood, J.D. Kennedy, M. Thornton-Pett, J. Chem. Soc. Dalton Trans. (1991) 2657; S. Coughlan, T.R. Spalding, G. Ferguson, J. Gallagher, A.J. Lough, X.L.R. Fontaine, J.D. Kennedy, J. Chem. Soc. Dalton Trans. (1992) 2865; G. Ferguson, A.J. Lough, S. Coughlan, T.R. Spalding, Acta Crystallogr. Sect. C, 48 (1992) 440; M. Murphy, T.R. Spalding, G. Ferguson, J. Gallagher, Acta Crystallogr. Sect. C, 48 (1992) 638; K.J. Adams, T.D. McGrath, A.J. Welch, Acta Crystallogr. Sect. C, 51 (1995) 401.

[6] R. Macías, J. Holub, J.D. Kennedy, B. Štíbr, M. Thornton-Pett, J. Chem. Soc. Chem. Commun. (1994) 2265; R. Macías, J. Holub, W. Clegg, M. Thornton-Pett, B. Štíbr, J.D. Kennedy, J. Chem. Soc. Dalton Trans. (1997) 149; Y.-H. Kim, R. Greatrex, J.D. Kennedy, Collect. Czech. Chem. Commun. 62 (1997) 1289.

[7] R. Macías, Synthesis, Structure and NMR Studies of some Novel Metallaboranes, Thesis, University of Leeds, 1997; G. Ferguson, G.F. Gallagher, Yi Wei, D. O'Connell, J.C. Patterson, T.R. Spalding, J.D. Kennedy, R. Macías, B. Štíbr, J. Chem. Soc. Dalton Trans. (1996) 3323; W.R. Hertler, F. Klanberg, E.L. Muetterties, Inorg. Chem. 6 (1967) 1696.

[8] K. Mazhighi, P.J. Carroll, L.G. Sneddon, Inorg. Chem. 31 (1992) 3197; C.-H. Kang, S.-J. Kun, J.-J. Ko, K.-B. Lee, S.-O. Kang, Bull. Korean Chem. Soc. 14 (1993) 537; M. Murphy, T.R. Spalding, J.D. Kennedy, M. Thornton-Pett, K.M.A. Malik, M.B. Hursthouse, J. Holub, J. Chem. Soc. Chem. Commun. (1994) 2403; G. Ferguson. D.E. McCarthy, T.R. Spalding, J.D. Kennedy, Acta Crystallogr. Sect. C, 52 (1996) 548.

[9] M.P. Murphy, T.R. Spalding, C. Cowey, J.D. Kennedy, M. Thornton-Pettand, J. Holub, J. Organomet. Chem. 550 (1998) 151.

[10] J.H. Jones, X.L.R. Fontaine, N.N. Greenwood, J.D. Kennedy, M. Thornton-Pett, B. Štíbr, H. Langhoff, J. Organomet. Chem. 445 (1993) C15.

[11] T.Jelínek, B. Štíbr, J.D. Kennedy, J. Chem. Soc. Chem. Commun. (1994) 1415; P. Kaur, T. Jelínek, B. Štíbr, J.D. Kennedy, M. Thornton-Pett, Structure of $S_2B_{16}H_{16}$ and the $[S_2B_{16}H_{15}]^-$ anion (in preparation).

[12] P. Kaur, J.D. Kennedy, M. Thornton-Pett, T. Jelínek, B. Štíbr, J. Chem. Soc. Dalton Trans. (1996) 1775; P. Kaur, M. Thornton-Pett, W. Clegg, J.D. Kennedy, J. Chem. Soc. Dalton Trans. (1996) 4155.

[13] A.J. Wynd, S.E. Robins, D.A. Welch, A.J. Welch, J. Chem. Soc. Chem. Commun. (1985) 819; A.J. Wynd, A.J. Welch, J. Chem. Soc. Chem. Commun. (1987) 1174; A.J. Wynd, A.J. McLennan, D. Reed, A.J. Welch, J. Chem. Soc. Dalton Trans. (1987) 2761.

[14] J.D. Kennedy, B. Wrackmeyer, J. Magn. Reson. 38 (1980) 529.

[15] J.H. Jones, New Heteroborane Cluster Compounds of Platinum Metals, Thesis, University of Leeds, 1991; see also Faridoon, O. Ni Dhubhghaill, T.R. Spalding, G. Ferguson, B. Kaitner, X.L.R. Fontaine, J.D. Kennedy, J. Chem. Soc. Dalton Trans. (1989) 1657; K. Baše, V. Gregor, S. Heřmánek, Chem. Ind. (London) (1979) 743; T.K. Hilty, D.A. Thompson, W.M. Butler, R.W. Rudolph, Inorg. Chem. 18 (1979) 2642.

[16] Y.M. Cheek, N.N. Greenwood, J.D. Kennedy, W.S. McDonald, J. Chem. Soc. Chem. Commun. (1982) 80.

[17] T. Jelínek, B. Štíbr, J.D. Kennedy, M. Thornton-Pett, J. Chem. Soc. Chem. Commun. (1994) 1999.

[18] T. Jelínek, J.D. Kennedy, B. Štíbr, M. Thornton-Pett, J. Chem. Soc. Chem. Commun. (1995) 2407.

[19] T. Jelínek, J.D. Kennedy, B. Štíbr, J. Chem. Soc. Chem. Commun. (1994) 677.

[20] G.M. Sheldrick, Acta Crystallogr. Sect. A 46 (1990) 467.

[21] G.M. Sheldrick, J. Appl. Cryst. (to be submitted).

[22] P. McArdle, J. Appl. Cryst. 28 (1995) 65.