

Note

# Partial incorporation of a cyclopentadienyl ligand into a molybdaborane to form a molybdacarbaborane

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## Abstract

Reaction of the molybdaborane *arachno*-2-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta$ <sup>5</sup>: $\eta$ <sup>1</sup>-C<sub>5</sub>H<sub>4</sub>)B<sub>4</sub>H<sub>7</sub>] (**I**) with NEt<sub>3</sub> in toluene at 120 °C for 7 days gives a 90% yield of the molybdacarbaborane *nido*-1-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta$ <sup>3</sup>: $\eta$ <sup>2</sup>-C<sub>3</sub>H<sub>3</sub>)C<sub>2</sub>B<sub>3</sub>H<sub>5</sub>] (**II**). Two of the carbon atoms in the substituted cyclopentadienyl ring in **I** are incorporated into the metallacarbaborane cluster **II**. The carbaborane {C<sub>2</sub>B<sub>3</sub>H<sub>5</sub>} fragment in **II** is attached to an allylic {C<sub>3</sub>H<sub>3</sub>} group and can be thought of as a new non-planar {C<sub>5</sub>B<sub>3</sub>H<sub>8</sub>} ligand providing seven electrons to the molybdenum atom. Reaction of **I** with KH in thf at 20 °C gives the anion [*nido*-1-Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta$ <sup>3</sup>: $\eta$ <sup>2</sup>-C<sub>3</sub>H<sub>3</sub>)C<sub>2</sub>B<sub>3</sub>H<sub>4</sub>]<sup>-</sup> (**III**) via deprotonation of a B–H–B bridging proton.

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The reaction of Lewis bases such as amines and phosphines with polyhedral borane clusters has been widely studied for many decades. Symmetrical [1,2] or unsymmetrical cleavage [3] of the borane cluster can occur to give simple Lewis acid–base adducts or ionic salts, respectively. Alternatively, polyhedral boranes can react with Lewis bases to form borane adducts with displacement of molecular hydrogen, without cleavage of the cluster framework [4,5]. In contrast to polyhedral boranes, relatively little work has been carried out on the reaction of Lewis bases with metallaboranes [6–10]. Macías and co-workers report that the reaction of the iridaborane *arachno*-1-[Ir( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)(H)B<sub>4</sub>H<sub>9</sub>] with a range of phosphines and amines (excess) leads to {BH<sub>3</sub>} or {2H} abstraction and the formation of the metallaboranes *arachno*-1-Ir( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)(H)-(L)B<sub>3</sub>H<sub>7</sub> and *nido*-1-[Ir( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)(H)B<sub>4</sub>H<sub>7</sub>], respectively (L = Lewis base). In addition, reaction of *arachno*-1-[Ir( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)(H)<sub>2</sub>B<sub>3</sub>H<sub>7</sub>] with phosphines and amines leads

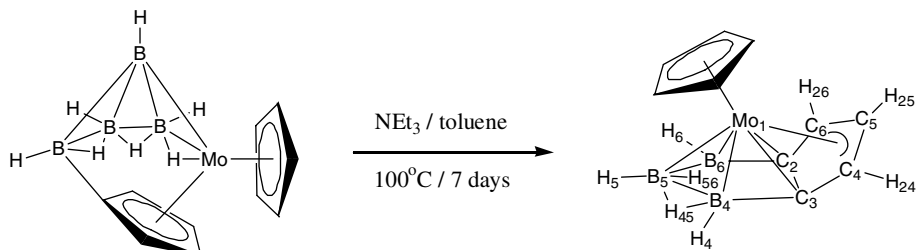
to formation of 1-[Ir( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)(H)<sub>2</sub>B<sub>2</sub>H<sub>4</sub>(L)] [11,12]. Shi-moi, Kawano and co-workers have reported the reaction of *nido*-[Ru<sub>2</sub>( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>( $\mu$ -H)<sub>2</sub>(B<sub>3</sub>H<sub>7</sub>)] with tertiary phosphines to give [Ru<sub>2</sub>( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>( $\mu$ -H)( $\mu$ - $\eta$ <sup>4</sup>-B<sub>2</sub>H<sub>5</sub>)(PR<sub>3</sub>)] (PR<sub>3</sub> = PMe<sub>3</sub>, PMe<sub>2</sub>Ph) via loss of {BH<sub>3</sub>} from the cluster [13].

We have been interested in exploring the formation and chemistry of metallaboranes of the early to middle transition metals [14–18]. In particular, we have been able to synthesise the molybdaborane *arachno*-2-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta$ <sup>5</sup>: $\eta$ <sup>1</sup>-C<sub>5</sub>H<sub>4</sub>)B<sub>4</sub>H<sub>7</sub>] (**I**) in gram quantities and this has permitted a study of its chemistry [19]. Here we report the novel reaction of **I** with the tertiary amine NEt<sub>3</sub>.

Reaction of **I** with NEt<sub>3</sub> in toluene at 120 °C for 7 days gives a 90% yield of the molybdacarbaborane *nido*-1-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta$ <sup>3</sup>: $\eta$ <sup>2</sup>-C<sub>3</sub>H<sub>3</sub>)C<sub>2</sub>B<sub>3</sub>H<sub>5</sub>] (**II**) (Scheme 1). Compound **II** is also formed in high yield from the reaction of **I** with the metal hydrides [W(PMe<sub>3</sub>)<sub>4</sub>H<sub>4</sub>], [W(PMe<sub>3</sub>)<sub>4</sub>H<sub>2</sub>Cl<sub>2</sub>], [Ta( $\eta$ -C<sub>5</sub>H<sub>5</sub>)<sub>2</sub>H<sub>3</sub>] and [Ru( $\eta$ -C<sub>5</sub>H<sub>5</sub>)(PMe<sub>3</sub>)<sub>3</sub>] at elevated temperature in toluene with concomitant formation of the respective metal borohydrides. Treatment of **I** with excess

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Scheme 1. Formation of *nido*-1-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta^3$ : $\eta^2$ -C<sub>3</sub>H<sub>3</sub>)C<sub>2</sub>B<sub>3</sub>H<sub>5</sub>] (**II**) from *arachno*-2-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta^5$ : $\eta^1$ -C<sub>5</sub>H<sub>4</sub>)B<sub>4</sub>H<sub>7</sub>] (**I**).

NMe<sub>3</sub> in toluene at room temperature gave an immediate reaction with formation of [Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)<sub>2</sub>H<sub>2</sub>] and [BH<sub>3</sub>·NMe<sub>3</sub>], as observed by <sup>1</sup>H and <sup>11</sup>B NMR spectroscopy. Treatment of **I** with trialkylphosphines does not give **II** but instead metallaborane phosphine adducts [20].

Compound **II** has been characterised by <sup>1</sup>H, <sup>11</sup>B and <sup>31</sup>P NMR spectroscopy and elemental analysis.<sup>1</sup> The line-narrowed <sup>11</sup>B NMR spectrum of **II** consists of two resonances, a doublet of triplets ( $\delta$  = 18.2 ppm) and a doublet of doublets ( $\delta$  = 4.1 ppm) in an integral ratio of 1:2. The <sup>1</sup>H NMR spectrum of **II** comprises a singlet ( $\delta$  = 4.30 ppm, 5H) assigned to the  $\eta$ -C<sub>5</sub>H<sub>5</sub> ligand, and a doublet of doublets ( $\delta$  = 5.01 ppm, 2H) and a triplet ( $\delta$  = 4.44 ppm, 1H) assigned to the bridging *exo*-cage C<sub>3</sub>H<sub>3</sub> fragment. In the <sup>1</sup>H{<sup>11</sup>B} NMR spectrum of **II** there is also a doublet of doublets ( $\delta$  = -2.59 ppm, 2H) assigned to the two equivalent B–B bridging hydrogen atoms, a multiplet ( $\delta$  = 3.70 ppm, 3H) assigned to the terminal B–H hydrogen atoms. Selective <sup>1</sup>H{<sup>11</sup>B} NMR experiments showed that the equivalent terminal B–H protons and the unique terminal B–H proton resonate at slightly different frequencies. Decoupling the unique B atom gave a triplet ( $\delta$  = 3.74 ppm) whilst decoupling the two equivalent B atoms gave a doublet ( $\delta$  = 3.70 ppm). The <sup>13</sup>C NMR spectrum consists of a doublet ( $\delta$  = -2.29 ppm) assigned to the  $\eta$ -C<sub>5</sub>H<sub>5</sub> ligand, and a doublet ( $\delta$  = 5.31 ppm) and doublet ( $\delta$  = 13.84 ppm) assigned to the allylic *exo*-cage C atoms. The quaternary C atoms were not observed probably because of considerable broadening due to coupling to the equivalent B atoms.

It appears that the Lewis base NEt<sub>3</sub> has removed a {BH<sub>3</sub>} unit from the borane cluster of **I** to form a metallocarbaborane in which two of the C atoms originally from the  $\eta^5$ : $\eta^1$ -C<sub>5</sub>H<sub>4</sub> ligand have been incorporated into the

cage. The C<sub>2</sub>(C<sub>3</sub>H<sub>3</sub>)B<sub>3</sub>H<sub>5</sub> carbaborane ligand in **II** carries an *exo*-cage allylic group. The C<sub>2</sub>(C<sub>3</sub>H<sub>3</sub>)B<sub>3</sub>H<sub>5</sub> carbaborane and allylic groups can be thought of as a non-planar {C<sub>5</sub>B<sub>3</sub>H<sub>8</sub>} ligand providing seven electrons to the molybdenum atom. Although the proposed structure for **II** is novel it shows close similarities to the previously reported *closo*-1-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta^2$ : $\eta^3$ -C<sub>3</sub>H<sub>3</sub>)C<sub>2</sub>B<sub>9</sub>H<sub>9</sub>], which has been crystallographically characterised [19]. The molybdenum–carbon–boron skeleton of **II** is exactly the same as in *closo*-1-[Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta^2$ : $\eta^3$ -C<sub>3</sub>H<sub>3</sub>)C<sub>2</sub>B<sub>9</sub>H<sub>9</sub>] if the boron atoms not directly bonded to the molybdenum atom are removed.

Pipal et al. reported the formation of the metallocarbaborane *closo*-1,7-[Co<sub>2</sub>( $\eta$ -C<sub>5</sub>H<sub>5</sub>)C<sub>2</sub>(C<sub>3</sub>H<sub>4</sub>)B<sub>3</sub>H<sub>3</sub>] from NaB<sub>5</sub>H<sub>8</sub>, NaC<sub>5</sub>H<sub>5</sub> and CoCl<sub>2</sub> at low temperatures in thf [21]. This metallocarbaborane cluster is a triple decker sandwich compound with a planar “C<sub>2</sub>B<sub>3</sub>H<sub>3</sub>” ligand bonded to an *exo*-cage bridging propylenyl group. The metallocarbaborane is formed via partial incorporation of a cyclopentadienyl group into the cluster. However, in contrast to the allylic {C<sub>3</sub>H<sub>3</sub>} fragment in **II**, the propylenyl {C<sub>3</sub>H<sub>4</sub>} bridging group is not interacting with the metal atoms.

Insertion of a cyclopentadienyl ligand into a metallaborane framework containing a group 6 metal has been reported previously for the formation of the 24-electron triple-decker sandwich complex [( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)Cr]<sub>2</sub>{ $\mu$ - $\eta^6$ - $\eta^6$ -( $\mu$ -1,2-C<sub>3</sub>H<sub>6</sub>-1,2-C<sub>2</sub>B<sub>4</sub>H<sub>4</sub>)} in low yield. The complex contains a metal-stabilised planar tetraborabenzene ligand [22].

Compound **II** is closely related to a large number of “*nido*-1-MC<sub>2</sub>B<sub>3</sub>” clusters that contain a planar “C<sub>2</sub>B<sub>3</sub>” ligand bonded to a metal centre. Examples include *nido*-[Fe( $\eta$ -C<sub>6</sub>Me<sub>6</sub>)C<sub>2</sub>B<sub>3</sub>H<sub>7</sub>] prepared by degradation of the 7-vertex cluster *closo*-[Fe( $\eta$ -C<sub>6</sub>Me<sub>6</sub>)C<sub>2</sub>B<sub>4</sub>H<sub>8</sub>] via loss of the apical {BH} fragment [23], *nido*-1-[Co( $\eta$ -C<sub>5</sub>H<sub>5</sub>)C<sub>2</sub>Me<sub>2</sub>B<sub>3</sub>H<sub>5</sub>] prepared similarly or from reaction of *nido*-2-[Co( $\eta$ -C<sub>5</sub>H<sub>5</sub>)B<sub>4</sub>H<sub>8</sub>] with but-2-yne [24], and [Fe(CO)<sub>3</sub>C<sub>2</sub>B<sub>3</sub>H<sub>7</sub>] formed by the reaction of [Fe(CO)<sub>5</sub>] with *closo*-[C<sub>2</sub>B<sub>4</sub>H<sub>8</sub>] [25]. There are also numerous *closo* clusters containing the planar ligand [26].

The NMR spectra of other “*nido*-1-MC<sub>2</sub>B<sub>3</sub>” metallocarbaboranes show similar shielding patterns to those observed for **II**. In the <sup>11</sup>B NMR spectrum of compound *nido*-1-Co( $\eta$ -C<sub>5</sub>H<sub>5</sub>)C<sub>2</sub>Me<sub>2</sub>B<sub>3</sub>H<sub>5</sub> the boron resonances appear close together at  $\delta$  = -2.7 ppm (2B) and -2.0 ppm (1B). This compares with  $\delta$  = 18.2 ppm (1B) and 4.1 ppm (2B) for **II**. In the <sup>1</sup>H NMR of the cobalt cluster the B–H terminal and B–B bridging hydrogen atoms appear at

<sup>1</sup> Analytical data for compound **II**. Found: C, 46.1; H, 5.1%. C<sub>10</sub>H<sub>13</sub>B<sub>3</sub>Mo Calc.: C, 45.9; H, 4.9. Selected NMR data: Compound **II**, solvent benzene-*d*<sub>6</sub>; Compound **III**, solvent thf-*d*<sub>8</sub>. <sup>1</sup>H NMR at 300 MHz, <sup>13</sup>C NMR at 75.5 MHz and <sup>11</sup>B NMR at 96.25 MHz. Compound **II**: <sup>11</sup>B [ $J$ (<sup>11</sup>B–<sup>1</sup>H)]  $\delta$  18.2 (d of t, [ $J$ (B–H) 147], [ $J$ (B–B) 36], 1B, B<sub>3</sub>), 4.1 (d of d, [ $J$ (B–H) 150], [ $J$ (B–B) 38], 2B, B<sub>4,6</sub>); <sup>13</sup>C{<sup>1</sup>H} [ $J$ (<sup>13</sup>C–<sup>1</sup>H)]  $\delta$  98.02 (d, (168) 1C, C<sub>5</sub>), 89.45 (d, (181), 2C, C<sub>4,6</sub>), 81.91 (d, (180), 5C, C<sub>5</sub>H<sub>5</sub>). C<sub>2</sub> and C<sub>3</sub> were not observed; <sup>1</sup>H{<sup>11</sup>B}  $\delta$  5.03 (d of d, 2H, H<sub>24,26</sub>), 4.44 (t, 1H, H<sub>25</sub>), 4.36 (s, 5H, C<sub>5</sub>H<sub>5</sub>), 3.70 (br m, 3H, H<sub>4,5,6</sub>), -2.60 (d of d, 2H, H<sub>45,56</sub>). Compound **III**: <sup>11</sup>B  $\delta$  11.9 (br s, 2B, B<sub>4,6</sub>), 13.4 (br s, 1B, B<sub>5</sub>); <sup>1</sup>H{<sup>11</sup>B}  $\delta$  4.86 (br d, 2H, H<sub>24,26</sub>), 4.44 (br t, 1H, H<sub>25</sub>), 4.35 (s, 5H, C<sub>5</sub>H<sub>5</sub>), 2.33 (br s, 3H, H<sub>4,5,6</sub>), -5.64 (s, 1H, H<sub>45</sub> or H<sub>56</sub>).

$\delta = 6.37$  ppm (3H) and  $-3.05$  ppm (2H), respectively. In compound **II** they appear at  $\delta = 3.70$  ppm (3H) and  $-2.60$  ppm, respectively. Other “*nido*-1-MC<sub>2</sub>B<sub>3</sub>” clusters also show similar chemical shifts for the three B–H terminal hydrogen atoms.

We tried to investigate some chemistry of **II**. Treatment of a solution of **II** in thf with KH gave [*nido*-1-Mo-( $\eta$ -C<sub>5</sub>H<sub>5</sub>)( $\eta^3$ : $\eta^2$ -C<sub>3</sub>H<sub>3</sub>)C<sub>2</sub>B<sub>3</sub>H<sub>4</sub>][K<sup>+</sup>] (**III**). The <sup>1</sup>H{<sup>11</sup>B} NMR spectrum of **III** showed that one of the B–H–B bridging hydrogen atoms had been lost. The remaining bridging hydrogen atom appears at much higher field ( $\delta = -5.64$  ppm) than the bridging hydrogens in the non-ionic compound **II**. The hydrogens associated with the [C<sub>3</sub>H<sub>3</sub>] allylic fragment in **III** appear in a 2:1 ratio as in the undeprotonated **II**. This can be explained if the lone B–H–B bridging hydrogen atom is rapidly exchanging between the two bridging sites. A similar fluxional process has been shown to occur in *nido*-[Me<sub>2</sub>C<sub>2</sub>B<sub>4</sub>H<sub>5</sub><sup>-</sup>] and several *nido*-[MC<sub>2</sub>B<sub>4</sub>H<sub>4</sub><sup>-</sup>] metal-laborane anions (M = two electron-donating metal fragment) [26–28]. The terminal B–H hydrogen atoms appear as a broad singlet at  $\delta = 2.33$  ppm. The <sup>11</sup>B NMR spectrum shows two broad resonances at close chemical shifts,  $\delta = 11.9$  (2B) and  $\delta = 13.4$  ppm (1B).

Refluxing compound **II** with various alkynes gave no reaction. Although small tetracarbon metallacarboranes are known there is no precedent for *nido*-1-MC<sub>2</sub>B<sub>3</sub> clusters to cage expand [29,30]. Treatment of **II** with PMe<sub>3</sub> or PEt<sub>3</sub> gave no reaction at 20 °C and only cage degradation and formation of [Mo( $\eta$ -C<sub>5</sub>H<sub>5</sub>)<sub>2</sub>H<sub>2</sub>] occurred at higher temperatures. Thus, although **II** has an unusual structure it behaves in a similar manner to other *nido*-1-MC<sub>2</sub>B<sub>3</sub> clusters.

## 1. Preparation of **II**

A solution of **I** (0.20 g, 0.72 mmol) and NEt<sub>3</sub> (0.5 ml, 5 mmol) in toluene (10 ml) was heated in a sealed Young's ampoule at 120 °C for 1 week. By then the orange-red solution had lightened to a brown-orange colour. Volatiles were removed in vacuo, pumping for at least 1 h to remove the NEt<sub>3</sub>. The residue was extracted with pentane (3 × 10 ml), filtered and concentrated to 15 ml. Cooling to  $-78$  °C for 3 days gave orange crystals of **II** which were filtered off, washed with cold pentane and pumped dry. Yield 0.17 g, 90%.

## 2. Preparation of **III**

An orange solution of **II** (10 mg) in thf-*d*<sub>8</sub> was treated with excess KH. Effervescence occurred and the solution

turned red. The solution was filtered and transferred to a 5-mm NMR tube and sealed under vacuum. Nmr spectroscopic analysis showed it to be pure **III**.

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