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- **Title:** 1,1-Hydroboration and Borane Adduct of Diphenyldiazomethane: A Prelude to FLP-N2 Chemistry?
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1,1-Hydroboration and Borane Adduct of Diphenyldiazomethane: A Prelude to FLP-N₂ Chemistry?

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Dedicated

Abstract: Diphenyldiazomethane reacts with HB(C₆F₅)₂ and B(C₆F₅)₃ resulting in 1,1-hydroboration and adduct formation, respectively. The hydroboration proceeds via a concerted reaction involving initial formation of the Lewis adduct Ph₂CN₂BH(C₆F₅)₂. The highly sensitive adduct Ph₂CN₂B(C₆F₅)₃, **5**, liberates N₂ and generates Ph₂CB(C₆F₅)₃. DFT computations reveal that formation of **5** from carbene, N₂ and borane is thermodynamically favourable, suggesting steric frustration could preclude carbene-borane adduct formation and effect FLP-N₂ capture.

Frustrated Lewis pair (FLP) chemistry has emerged as a strategy to both metal-free hydrogenations and the activation of a variety of small molecules.^[1] Immediately following the demonstration of the heterolytic activation of H₂ by combinations of Lewis acids and bases in 2006,^[2] the notion of metal-free reduction protocols garnered much attention.

Concurrent with the efforts described above, FLP strategies have been applied to activate small molecules other than H₂. Early efforts showed the unique reactivity of FLPs with olefins,^[3] alkynes,^[4] disulfides,^[5] N₂O^[6] and cyclopropanes,^[7] while more recent studies have extended this reactivity to include reactions with CO_2 ,^[8] CO,^[9] NO,^[10] SO_2 ^[11] and RNSO.^[12] Recent studies have creatively used these fundamentally new reactions in organic synthetic applications, and others have uncovered transition metal-based FLPs, with relevance to hydrogenase enzyme models and surface chemistry.^[13]

Notably absent from the list of small molecule substrates for FLPs is dinitrogen. Although this has been the subject of question and conjecture for some years, it is only recently that the groups of Szymczak^[14] and Simonneau^[15] reported interaction of boranes with metal-N₂ complexes (Scheme 1). The former demonstrated the prospect of protonation of the N₂ fragment and the latter explored related borylation and silylation of the N₂ bound between the metal and boron. Metal-free systems that capture N₂ are unknown however,

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binding of N₂ to Lewis acids has been the subject of a number of computational studies, and the species (N₂)BF₃was generated transiently via supersonic expansion at 600 torr and 170 K and characterized spectroscopically.^[16] Recent computational work on the compound Ph₃PNNPPh₃^[17] has described this species as involving two phosphine donors interacting with N2.^[18] This description has been controversial as this species is not derived from nor evolves N2. On the other hand, diazomethanes are isolable yet liberate N₂, thus acting as carbene precursors.^[19] In this regard, we note our previous report of insertion of diazomethanes into B-C bonds with liberation of N₂ (Scheme 1).^[20] Herein, the stericallyencumbered diazomethane, Ph₂CN₂ reacts with the boranes $HB(C_6F_5)_2$ and $B(C_6F_5)_3$ via adduct formation. In the former case, 1,1-hydroboration is seen, and the diazomethane adduct of $B(C_6F_5)_3$ foreshadows the possibility of FLP-N₂ chemistry.





Combination of Ph₂CN₂ and Piers borane, HB(C₆F₅)₂, results in a rapid reaction that affords a robust product **1**. The ¹H NMR spectrum of **1** showed a diagnostic resonance at 8.77 ppm, corresponding to a single NH proton. The corresponding ¹¹B NMR signal was seen at 35.3 ppm, while the ¹⁹F NMR spectrum showed six signals, attributable to inequivalent C₆F₅ rings. Collectively these data support the formulation of **1** as Ph₂CNNHB(C₆F₅)₂. This assignment was confirmed via a crystallographic study (Figure 1). The structural data reveal a pseudo trigonal planar geometry at B with N-B-C and C-B-C angles of 118.2(2)°, 121.1(1)° and 120.6(1)°. The C=N, N-N and N-B bond lengths are 1.287(2) Å, 1.394(2) Å and 1.385(2) Å, respectively. These data are consistent with considerable B-N π -bonding, which gives rise to a *trans*-diene type structure,

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inhibits rotation about the B-N bond, and renders the fluoroaryl rings inequivalent.



Figure 1. POV-ray depiction of molecular structure of **1** as determined by X-ray crystallography. H-atoms (except NH) are omitted for clarity. C: black, N: blue, B: yellow-green, F: pink.

It is noteworthy that conventional hydroborations give 1,2substitution.^[21] Although there are rare examples of 1,1hydroborations,^[22] the formation of 1 represents the first reported 1,1-hydroboration of an N-N bond. Mechanistically, two options were considered. In the first, initial 1,2hydroboration occurs, followed by proton migration. In the second, direct 1,1-hydroboration occurs. Computational work using the dispersion-corrected PBEh-3c^[23] (DSD-BLYP/def2-TZVPP//PBEh-3c+COSMO-RS) revealed a rather early transition state in which the B-H hydride exhibited a bond order of 0.54 to the B and 0.33 to the N. Localized orbitals show a 3-centre, 2-electron-bond for the B-H-N unit, inferring the participation of a low-lying orbital (π^*) on the N=N unit that accepts electron density from the B-H bond (Figure 2). The negative charge on the H atom is small (0.1 e more negative than H-atoms on carbon). Thus, the 1,1hydroboration affording 1 is best described as a H-atom migration from boron to the proximal nitrogen.



Figure 2. TS structure for H atom migration with a plot of the localized 3-centre, 2-electron MO which is characteristic of bond formation.

An orthogonal synthetic strategy was also developed involving the reaction of Ph_2CNNH_2 and $HB(C_6F_5)_2$ in a 1:1 stoichiometry. The reaction generates the Lewis acid-base adduct (Ph_2CNNH_2)HB(C_6F_5)₂ **2** in 62% isolated yield (Scheme 2). The ¹H NMR spectrum exhibits broad multiplets at 6.71-6.69 ppm and 3.89 ppm attributable to the NH₂ and BH fragments, respectively. The ¹¹B NMR spectrum shows a singlet at -12.1 ppm and ¹⁹F NMR signals appear at -134.5, -157.4 and -163.4 ppm, consistent with a 4-coordinate boron centre. X-ray data confirmed the formulation of **2** (Figure 3a) with pyramidal geometry at B and C=N, N-N and B-N bond distances of 1.287(3) Å, 1.453(3) Å and 1.610(4) Å, respectively. Gentle heating of **2** at 50 °C for 2 h resulted in the loss of H₂ affording **1** (Scheme 2). In a third synthetic route, **1** was prepared directly in 69% yield via reaction Ph₂CNNH₂ with 1.2 equivalents of HB(C₆F₅)₂ at room temperature.



Scheme 2. Synthetic routes to 1-4.



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In a similar fashion, the reaction of Ph_2CNNH_2 and $B(C_6F_5)_3$ afforded the corresponding adduct $(Ph_2CNNH_2)B(C_6F_5)_3$ **3** in 62% isolated yield (Scheme 2). Compound 3 exhibited a ¹H NMR singlet at 7.44 ppm, a ¹¹B NMR signal at -6.3 ppm, and ¹⁹F NMR peaks at -133.4, -155.4, and -162.7 ppm. Singlecrystal X-ray diffraction confirmed the formulation of 3 with C=N, N-N and N-B distances of 1.291(6) Å, 1.459(6) Å and 1.631(7) Å, respectively (Figure 3b). Compound 3, when heated at 110 °C for 20 h in a sealed vessel, generates a solution of compound 1 and C_6F_5H . Workup of this reaction afforded 1 in only 26% isolated yield. Interestingly, reaction of 3 with tBu₃P proceeds at room temperature over 22 h, and a new product 4 was isolated (Scheme 2). This species gives rise to a broad resonance in the ¹H NMR spectrum at 8.57 ppm and a doublet at 5.34 ppm (${}^{1}J_{H-P}$ = 456 Hz) in a 1:1 intensity ratio. These resonances are attributed to N- and P-bound protons, respectively. The ³¹P NMR spectrum shows a doublet at 58.2 ppm with a corresponding P-H coupling constant. A signal at -6.6 ppm was observed in the ¹¹B NMR spectrum and the ¹⁹F NMR spectrum of 4 showed three peaks at -133.3, -156.0 and -162.7 ppm. These data support the formulation of 4 as the salt [*t*Bu₃PH][Ph₂CN₂HB(C₆F₅)₃], which was confirmed crystallographically (Figure 3c). The C=N, N-N and N-B distances in the anion are 1.296(7) Å, 1.354(6) Å and 1.524(7) Å, respectively. The metric parameters in the cation were unexceptional.

Given the isolation of the adducts 2 and 3, the synthesis of a $B(C_6F_5)_3$ adduct of diphenyldiazomethane was undertaken. This reaction afforded a highly unstable product but performing the reaction at -78 °C permitted its characterization. The ¹¹B NMR spectrum of the product showed a resonance at 4.8 ppm and the ¹⁹F NMR spectrum showed resonances at -132.0, -154.7 and -163.4 ppm. The upfield ¹¹B NMR shift and the reduced meta-para gap of the fluorine resonances with respect to the free borane are consistent with the formation of a Lewis adduct between $B(C_6F_5)_3$ and Ph_2CN_2 . Infrared absorption data collected at 233 K showed bands at 1645 and 1516 cm⁻¹ attributable to N=N and C=N stretches. Crystals of the product proved remarkably sensitive and evolved gas (N₂) even at room temperature under Paratone-N oil. This sensitivity stands in contrast to Ph₃PNNPPh₃, which only evolved N₂ above 215 °C.^[18] Crystallographic data collected at 150 K confirmed that the product was indeed Ph₂CN₂B(C₆F₅)₃ 5. The C=N, N=N and B-N distances are 1.278(8) Å, 1.177(7) Å and 1.65(1) Å, respectively, while the N-N-B angle in 5 is 124.6(6)°. These metric data are consistent with C-N and N-N double bond character and the geometry about the N_2 -fragment is reminiscent of the M=N=N-B units seen in $M(R_2PCH_2CH_2PR_2)_2N_2B(C_6F_5)_3$ (R = Ph, M = Mo, W; R = Et, M = Fe).^[14-15] The B-N distance in 5 is significantly longer than those in these metal complexes (M = Mo 1.567(7) Å; W 1.570(6) Å; M = Fe 1.575(4) Å). At the same time, the N-N bond and the N-N-B angle in 5 are shorter/smaller than those in the metal complexes (M = Mo 1.196(5) Å, 141.4(5)°; W 1.212(6) Å, 140.3(4)°; Fe 1.186(3) Å, 137.0(3)°). The structural data for 5 are consistent with weak N-B donation. Nonetheless, it is

interesting that this interaction dramatically facilitates the ability of Ph_2CN_2 to evolve N_2 . Moreover, it appears that the steric demands of the arene rings on Ph_2CN_2 preclude the formal carbene insertion into B-C bonds that is readily seen with smaller diazomethanes.^[20] Upon warming the solution warm to room temperature, a ¹¹B NMR signal at -6.4 ppm and ¹⁹F resonances at -132.9, -155.8 and -162.9 ppm appear which is consistent with the formation of new adduct proposed to be $Ph_2C(B(C_6F_5)_3)$. While a ¹H-¹⁹F HOESY NMR data further support this proposal, efforts to isolate this species were unsuccessful (See SI).



Figure 4. POV-ray depiction of molecular structure of **5** as determined by X-ray crystallography. H-atoms are omitted for clarity. C: black, N: blue, B: yellow-green, F: pink.



Scheme 3. Degradation pathway for **5** showing optimized DFT structures. Relative free energies at the DSD-BLYP/def2-TZVPP//PBEh-3c+COSMO-RS[CH₂Cl₂] level are in kcal/mol.

DFT computational studies for 5 revealed a N-B donoracceptor interaction (bond order 0.6), an N-N double bond and a C-N bond order of 1.5. Consideration of the instability of 5 prompted computation of the pathway to loss of N_2 . Dissociation of 5 to a van der Waals complex is a low energy process (3.9 kcal/mol), while a transition state (TS) involving coordination of borane to the carbon of diphenyldiazomethane is computed to be thermally accessible at 19.4 kcal/mol above 5 (Scheme 3). From this TS, loss of N₂ affording the carbene adduct of $B(C_6F_5)_3$ is exergonic by about 53 kcal/mol. The B-C_{carbene} bonding in $Ph_2CB(C_6F_5)_3$ is computed to be 1.66 Å similar to that seen for the B-C bonds

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in $B(C_6F_5)_3$. Similarly, the bond order is computed to be 0.64 typical for B-C single bonds.

Computations also show that free carbene, free borane and N₂ are only 10.0 kcal/mol below the TS. However, in the present system formation of the carbene-borane adduct is the more exergonic reaction pathway. Nonetheless, this suggests that frustration of the carbene-borane interaction could provide an energetically favourable reaction pathway to the TS ultimately affording an analog of **5** and thus unveiling an FLP approach to N₂ capture.

In conclusion, we have reported the 1,1-hydroboration of Ph_2CN_2 with $HB(C_6F_5)_2$ to give 1, which proceeds via a concerted mechanism. In addition, reactions of Ph_2CNNH_2 with $HB(C_6F_5)_2$ or $B(C_6F_5)_3$ provide alternative routes to 1, as well as a related salt. The isolation of the first diazomethaneborane adduct, 5, is also reported. The isolation of this latter species raises the possibility that judicious choice of a Lewis acid and base could provide an avenue to metal-free capture of N₂. It is this ambitious objective that is the target of current efforts.

Keywords: diphenyldiazomethane • borane • 1,1-hydroboration • adduct • nitrogen •

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$\begin{array}{c} \text{1,1-hydroboration} \\ C_6F_5 \\ Ph \\ Ph \\ Ph \\ B(C_6F_5)_2 \\ Ph \\ H \\ B(C_6F_5)_3 \end{array} \begin{array}{c} Ph \\ Ph \\ H \\ B(C_6F_5)_3 \\ Ph \\ B(C_6F_5)_3 \\ Ph \\ Bh \\ Ph \\ B(C_6F_5)_3 \end{array}$

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N₂ the breach... Diphenyldiazomethane reacts with HB(C₆F₅)₂ and B(C₆F₅)₃ affording 1,1-hydroboration and adduct formation, respectively. The adduct Ph₂CN₂B(C₆F₅)₃ liberates N₂ and the diphenylcarbene-borane adduct. Computational studies suggest that N₂ capture is thermodynamically favourable, but for the exothermicity of carbene-borane adduct formation.

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