Phosphinoborane interception at magnesium by borane-assisted phosphine-borane dehydrogenation†

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Reactions of B(C6F5)3 with the β-diketiminato (BDI) alkaline-earth phosphidoborane complexes, 1a [[BDI] Ca(H2B·PPh2)] and 1b [[BDI]Mg(H2B·PPh2)] result in the formation of phosphinodiboronate complexes 4a [[BDI]Ca{η6-toluene}{H3B·PPh2·B(C6F5)3}] and 4b [[BDI]Mg{H3B·PPh2·B(C6F5)3}]. Calcium complex 4a is stable in aromatic solvents at room temperature and does not display well-defined onward reactivity at elevated temperatures. Magnesium complex 4b undergoes a room temperature transformation to provide the known hydridoborate derivative 3b [[BDI]Mg{HB(C6F5)3}] and an N,P,N'-ligated species, 5 [[HC{C(CH3)N(2,6-iPr-C6H3)}2(H2BPPh2)]Mg{H3B·PPh2·B(C6F5)3}] that results from interception of the putative phosphinoborane, H2B = PPh2, by the BDI ligand backbone following B(C6F5)3-mediated hydride abstraction. NMR spectroscopic investigations were supported by DFT calculations, which suggested a mechanism involving B(C6F5)3 migration and hydride abstraction within the coordination sphere of magnesium. Interception of H2B = PPh2 by B(C6F5)3 is proposed to stabilise this species, whilst activating it towards ligand-centred nucleophilic attack. The significant stabilisation energy calculated for the Ca-π(toluene) interaction in 4a accounts for the contrasting outcomes between the two Ae-elements. The crystal structures of compounds 4a and 5 are presented and discussed.

Introduction

Polyphosphinoboranes are an emerging class of solution-processable inorganic materials with contrasting properties to their formally isoelectronic all-carbon analogues. Whereas most polyolefins are highly flammable, polyphosphinoboranes, for example, display flame retardant properties1,2 and have seen use as lithographic resists.2–4 Although their synthesis, via the catalytic dehydrogenation-polymerisation of phosphine-borane complexes, was first achieved some 20 years ago, catalysts remain almost exclusively based on iron, ruthenium, rhodium or iridium.2–11 A metal-free stoichiometric approach was described by Manners, Scheer, and co-workers, who demonstrated the metal-free head-to-tail polymerisation of tert-butylphosphinoborane, which was generated in situ from the trimethylamine-stabilised monomer (Scheme 1a).12 More recently, Manners and co-workers have reported that treatment with stoichiometric quantities of cyclic alkyl amino carbene (CAACs) induces the dehydrogenative coupling of

Scheme 1 (a) Metal-free head-to-tail polymerisation of tert-butylphosphinoborane.12 (b) Cyclic alkyl amino carbene (CAAC)-mediated polymerisation of phosphine-boranes.13 (c) BCF-catalysed dehydropolymerisation of phenylphosphine-borane.14 Mm and Mw values determined by GPC relative to polystyrene standards and, in the case of bimodal distributions, refer to the polymeric fraction only.

† Electronic supplementary information (ESI) available: General synthetic experimental details, NMR spectra, X-ray diffraction analysis of compounds 4a and 5, details for the computational analysis and atomic coordinates of computed structures. CCDC 2024757 and 2024758. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/d0dt03415k

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phosphine-boranes to provide access to primary- and, previously unprecedented, secondary polyphosphinoboranes (Scheme 1b). Of particular relevance to the current work is the B(C₆F₅)₃ (BCF)-catalysed dehydropolymerisation of phosphine-borane and phenylphosphine-borane reported by Denis et al. in 2003 (Scheme 1c). This process was proposed to proceed via iterative borane transfer and dehydrocoupling steps involving a Bronsted-acidic phosphine-borane complex, (C₆F₅)₃B·PPhH₂, as the active species. The obtained polymers were, however, of modest molecular weight and accompanied by a significant fraction of low molecular-weight oligomeric species.

Our own interest in this topic is motivated by the use of alkaline-earth (Ae) elements as non-toxic and inexpensive catalysts for the synthesis of inorganic polymers. Following previous investigations into Ae-mediated catalytic and stoichiometric amine-borane dehydrogenation and dehydrocoupling, we recently reported a series of β-diketiminate (BDI) supported alkaline-earth (Ae) phosphidoborane complexes, which are prepared by exposing diphenylphosphine-borane to readily accessible BDI-Ae hydride, alkyl, or amide precursors (Scheme 2a). Attempts to achieve complete phosphine-borane dehydrogenation were unsuccessful, however, and the use of super-stoichiometric quantities of phosphine-borane resulted in BH₃ transfer to provide the phosphinodiboronate complex and uncomplexed diphenylphosphine (Scheme 2a). The preference for these systems to undergo BH₃ transfer rather than hydride-elimination was rationalised to be a consequence of the charge-dense Lewis acidic Ae-centre in conjunction with the soft phosphorus-centred Lewis base. In order to address this shortcoming, inspiration was drawn from the previously observed BCF-mediated hydride abstraction of Ae-amidoborane complexes (Scheme 2b). This transformation yielded hydridoborate derivatives 3a–c with concomitant formation of the cyclic diborazine, (Me₂NBH₂)₂. It was anticipated that analogous BCF addition to phosphidoborane complexes 1a and 1b would provide a similar outcome, thus yielding oligomerisation products of the unsaturated diphenylphosphinoborane monomer.

Results and discussion

Addition of an equimolar quantity of BCF to a d₈-toluene solution of the calcium complex 1a resulted in quantitative conversion to a new BDI-containing species, 4a (Scheme 3), whose γ-CH proton resonated at δ 4.85 ppm. Counter to expectation, the resultant ¹¹B NMR spectrum comprised two broadened signals at δ −11.0 and −31.2 ppm and no resonance corresponding to the anticipated [HB(C₆F₅)₃]⁻ anion was observed. Similarly, the ³¹P{¹H} NMR spectrum showed no resonances corresponding to oligophosphinoboranes; instead, a single broad resonance centred at δ −1.43 ppm was detected. Slow evaporation of a saturated toluene solution of the crude product at room temperature yielded single crystals, X-ray diffraction analysis of which provided the solid-state structure of the phosphinodiboronate complex, 4a (Fig. 1), in which the boron-bound hydrogens were located and refined without restraints. Solutions of compound 4a in d₈-toluene or C₆D₆ were stable at room temperature and attempts to induce further reactivity by heating to 60 °C resulted in partial decomposition to the homoleptic complex [(BDI)₂Ca]³₀ and other unidentified species.

The BDI-complexed calcium centre of 4a is further coordinated by threefold B-μ²-H-Ca binding of a [(C₆F₅)₃B·PPhH₂·BH₃]⁻ anion and its coordination sphere is completed by an asymmetric η⁶ interaction with the π-system of a neutral molecule of toluene. The toluene ligand is labile

![Scheme 2](https://i.imgur.com/5Q5Q5.png)

**Scheme 2** (a) Synthesis and reactivity of Ae-phosphidoborane complexes, 1a and 1b. BCF-promoted hydride abstraction of Ae-amidoborane complexes. Ar = 2,6-di-isopropylphenyl.
in solution such that when crystals of 4a were dissolved in C6D6, one equivalent of free toluene could be discerned in the resulting 1H NMR spectrum (Fig. S1†). The phosphinodiboronate anion displays near-perfect tetrahedral geometry at P1, a slightly flattened tetrahedron at B2 and essentially identical structural parameters to those of Lancaster and co-workers’ lithium salt, [(C6F5)3BH2·PPh2·BH3]Li(OEt2)3, which was prepared by an analogous route.31 The contrasting P1·B2 (1.9496 Å) and P1–B2 (2.1368 Å) bond lengths may be rationalised on simple steric grounds. Although toluene adducts of barium are well established, the comparable ability of magnesium and calcium centres to bind neutral arenes has only been established very recently.

Charge-separated [[BDI]Ae][WCA]− species (WCA = weakly coordinating anion: [B(C6F5)4]− or [Al(OC(CF3)3)4]−) coordinate arenes via 1,2,3 or 6 interactions to give complexes of the type [[BDI]Ae·arene][WCA]−.32–34 Compound 4a, however, the first crystallographic characterisation of an unsupported interaction between a formally charge neutral calcium centre and an arene. The accommodation of the toluene ligand at the pseudo-tetrahedral metal-centre (for a space-filling representation of the crystal structure, see ESI, Fig. S24†) is facilitated by the disposition of the calcium centre, which projects some 1.3821(18) Å out of the mean plane defined by the BDI ligand. Ca1 is located 2.6391(10) Å from the toluene centroid but is asymmetrically coordinated with Ca–C distances ranging from 2.843(2) Å (Ca1–C33) to 3.1365(19) Å (Ca1–C30). The average Ca–C distance of 2.979 Å is, thus, only slightly longer than the comparable measurement in the charge separated cationic component of [[BDI]Ca·C6H4][Al(OC(CF3)3)4]− (ca. 2.93 Å).3,3

Immediate assessment of an analogous reaction performed in C6D6 with the dimeric magnesium complex 4b was also indicative of the effectively instantaneously and quantitative generation of a phosphinodiboronate complex (4b, Scheme 4). The constitution of compound 4b was diagnosed by the emergence of a single new BDI ligand environment in its 1H NMR spectrum and the appearance of a broad signal centred at δ 0.7 ppm in the corresponding 31P{1H} NMR spectrum. In addition to the expected resonances at δ –10.4 and –36.7 ppm, however, a doublet signal at δ –22.0 ppm was observed to grow into the 11B NMR spectrum over the course of 16 hours. While this latter resonance, and the appearance of a further BDI-ligated species in the 1H NMR spectrum, was readily attributed to the formation of the hydridoborate derivative, [[BDI]Mg[HB(C6F5)3]2] (3b)28,29 this solution instability frustrated all attempts to isolate and structurally characterise compound 4b.

Although the production of 3b is indicative of the successful abstraction of hydride from the phosphinodiboronate anion of 4b, no signals corresponding to the expected oligomerisations of [Ph2P=BH2]2 could be detected.7 Rather, in addition to the doublet signal of [H(BC6F5)3]− at δ –22.0 ppm, the ultimate 11B NMR spectrum comprised three additional broad resonances at δ −10.2, −24.7 and −35.3 ppm and the 31P{1H} NMR spectrum a broad multiplet at δ −7.2 ppm and a singlet at δ −42.7 ppm. Similarly, two differentiated C6F5 environments were discriminated by 13C NMR spectroscopy, while the 1H NMR spectrum evidenced a further, less symmetrical BDI-coordinated species (5), whose quantitative evolution was simultaneous to that of 3b and took place over the course of 16 hours at room temperature, or two hours at 40 °C. Compound 5 was most clearly characterised by a broad pseudo-doublet at δ 3.05 ppm, which emerged with a similar ratio of 1H NMR signal intensities to those assigned to 3b. These spectroscopic features were eventually rationalised by X-ray diffraction analysis of single crystals of compound 5 (Fig. 2), obtained after removal of solvent and fractional crystallisation of the reaction products from a hexane/toluene.
solvent system. As for 4a, the hydrogen atoms attached to boron were located and refined freely.

Compound 5 consists of a mononuclear magnesium species ligated by a tripodal N₅P₅N₅-donor ligand and displays close μ-H-Mg contacts to the same \([\text{C}_6\text{F}_5\text{B}·\text{PPh}_2·\text{BH}_3]\) anion present in the X-ray crystal structure of 4a. The former anionic moiety is postulated to result from nucleophilic attack of the BDI γ-methine carbon centre upon the unsaturated phosphineborane monomer, \([\text{Ph}_2\text{P}≡\text{BH}_3]\), which is generated within the coordination sphere of magnesium by BCF-mediated hydride abstraction from \([\text{C}_6\text{F}_5\text{B}·\text{PPh}_2·\text{BH}_3]\) or \([\text{Ph}_2·\text{BH}_3]\). Although Weller and co-workers’ cationic rhodium complex \([\text{Cp}^*\text{Rh}·(\text{Bu}_2\text{P}·\text{BH}_2·\text{PMe}_3)·\text{BArF}_4]\) was similarly derived by interception of a phosphineborane monomer generated by in situ phosphine-borane dehydrogenation,\(^{35}\) compound 5 is, to the best of our knowledge, the first example of a phosphineborane trapped within the coordination sphere of a main-group metal. Other kinetically stabilised transition-metal phosphineboron complexes have been described by the groups of Scheer\(^{16,\text{b,}17}\) and Bourissou,\(^{28}\) but were prepared by non-dehydrogenative metathesis and ligand exchange routes. Harder’s calcium borylamine complex \([\text{BDI}·\text{Ca}·(\text{DippNBH}_2)]\) bears comparison to compound 5 as an Ae-coordinated dehydrogenation product of a group 13–15 complex.\(^{35}\)

Although examples of similar BDI γ-C–B bond formation are limited to a recent report by Jones and co-workers,\(^{39}\) the formation of 5 is clearly also reminiscent of the well documented reactivity of \([\text{BDI}·\text{Ac}]^+\) and \([\text{BDI}·\text{AlMe}]^+\) cations towards other unsaturated small molecules such as alkynes or \(\text{CO}_2.\)^\(^{29,40,41}\) Consistent with the formulation of the tripodal anion as a bis-imine unit, the N₁–C₂ (1.2950(19) Å) and N₂–C₄ (1.293(2) Å) bond lengths of compound 5 are significantly shorter than those observed in unperturbed BDI anions. The geometry about C₃ is also effectively tetrahedral, with X–C₃–X bond angles in the range of 110.5–119.⁴⁰ The B₁–P₁ bond (1.9791(18) Å) is significantly longer than those typical of B–P double bonds (1.763–1.913 Å),\(^{42,47}\) and even exceeds the dative \(\text{P}–\text{BH}_2\) bonds of secondary phosphine-boranes (1.91–1.94 Å), and is thus best described as an elongated single bond, consistent with the tetrahedral geometries at both phosphorus and boron.\(^{45,46,51}\) The C₃–B₁ bond (1.726(2) Å) is also elongated in comparison to the analogous γ-C–B bonds of Jones’ compounds (1.680(2)–1.697(3) Å).\(^{39}\) The \([\text{C}_6\text{F}_5\text{B}·\text{PPh}_2·\text{BH}_3]\) anion, however, remains geometrically similar to that of compound 4a.

Several potential mechanisms were assessed theoretically by density functional theory (DFT) studies (BP86-D3(BJ)-benzene/BS2/BP86/BS1) of the reaction between 1b and BCF that culminates in the formation of 3b and 5. Initial calculations addressed the direct abstraction of a hydride from the dimeric phosphidoborane, complex 1b (designated as pathway 1; see ESI, Fig. S27f). This process ensues by disruption of a B-
μ-H-Mg interaction and the formation of a B→μ-H-(BCF) intermediate via a free energy barrier of 26.8 kcal mol⁻¹. Although subsequent hydride abstraction, Ph₂P≡BH₂ elimination and interception steps were found to be kinetically facile and thermodynamically viable, this computed pathway (I) was discounted as it does not invoke the formation of the experimentally observed product, 4b.

Incorporation of 4b into the mechanism necessarily requires insertion of a BCF molecule into one of the Mg-P bonds of the dimeric starting complex 1b to form species A at +6.7 kcal mol⁻¹ (Fig. 3). Consideration of this requirement also raises a number of possibilities. Formation of a dative P→B bond significantly stabilises A to give intermediate B at −14.2 kcal mol⁻¹. Subsequent addition of a second molecule of BCF to B results in the cleavage of the Mg-P bond and yields two molecules of 4b at ΔGbnz = −15.5 kcal mol⁻¹. Alternatively, the second Mg-P bond in B can dissociate with the endergonic production of one molecule of 4b and one molecule of the monomeric phosphinoborane intermediate, C (ΔGbnz = +6.8 kcal mol⁻¹, left hand side of both Fig. 4a and b). The calculated molecular structure of 4b (see ESI, Fig. S31 and Table S3†) exhibits near co-planarity of the Mg centre with the BDI ligand, resulting in a sterically protected environment that obviates the possibility of a stable Mg-arene solvent interaction, of the type observed for the analogous calcium cation is loosely bound to a neutral BCF-stabilised phosphinoborane monomer (G) and a [HBC(F₅C₆)₃]⁻ anion via μ-P interactions. G can then dissociate from F to provide the observed product 3b (ΔGbnz(G + GE) = −1.5 kcal mol⁻¹), the DFT-optimised structure of which is in good agreement with the previously published X-ray crystal structure of the magnesium hydridoborate product.28

Alternatively, the direct abstraction of a hydride from D can proceed via pathway III (Fig. 4b) and TS(D→3b) at ΔGbnz = +7.2 kcal mol⁻¹. The B–H distances in TS(D→3b) clearly show near-complete hydride transfer to the flattened tetrahedral boron centre, whilst the unsaturated H₂B⁻ = PPh₂ fragment remains bound to magnesium. Minor adjustments to the [HBC(F₅C₆)₃]⁻ anion result in the endergonic elimination of free H₂B⁻ = PPh₂ to provide 3b at 23.9 kcal mol⁻¹ above 4b (ΔGbnz = +8.4 kcal mol⁻¹). Interestingly, H₂B⁻ = PPh₂ can also be readily intercepted by a free BCF molecule to generate G, ΔGbnz = −1.5 kcal mol⁻¹, such that, from this point onwards, the course of the reaction to produce compound 5 is completely common to both computed pathways II and III.

The DFT calculated Köhn–Sham frontier molecular orbitals of free H₂B⁻ = PPh₂ displayed significant contributions from the π- and π*–molecular orbitals of the P–B bond (see ESI, Table S4†). In contrast, formation of the BCF-complex G results in de-population of the π-orbital and elongation of the P–B bond by 0.08 Å to 1.96 Å. This bond length is comparable to P–B single bonds observed in crystallographically characterised Lewis base- and Lewis acid–base stabilised phosphinoboranes.12,14,36,37,52–54 Whilst the calculated existence of an ortho-F-B donor-acceptor interaction in G provides some degree of a “push–pull” stabilisation (F–B distance = 1.73 Å), the electron-withdrawing effect of the phosphorus-complexed BCF moiety enhances the electrophilicity at boron. The calculated HOMO of 4b (−5.17 eV) involves a relatively exposed π-system delocalised across the BDI-ligand backbone. NBO charge analysis shows that the γ-methine carbon of the BDI ligand exhibits a significant negative charge (−0.41) and can, hence, act as a nucleophile, attacking the electrophilic boron centre of the BCF-complex G via TS(G→H) at ΔGbnz = −0.9 kcal mol⁻¹ to give H at ΔGbnz = −14.7 kcal mol⁻¹. Although dissociation of BCF is computed to be endergonic by

Fig. 3 DFT computed free energies (BP86-D3(BJ)-benzene/BS2//BP86/BS1), in kcal mol⁻¹, for the reaction of 1b with BCF to form two molecules of 4b. Ar = 2,6-di-isopropylphenyl.
intramolecular formation of a P–Mg dative bond is thermodynamically favoured. As such, stepwise P–B cleavage and Mg–P bond formation is exergonic, affording compound 5 at ΔG_{bnz} = −24.3 kcal mol\(^{-1}\). The free energy barrier to 3b and 5 formation from 4b in pathway II is, therefore, computed to be 31.2 kcal mol\(^{-1}\) (via intermediates E and F), whilst pathway III (via 3b) is computed to be 23.9 kcal mol\(^{-1}\), the latter being 7.3 kcal mol\(^{-1}\) lower in free energy and most consistent with the reaction conditions.

Most known Ae-mediated catalytic processes have been shown to follow a trend of increasing reactivity on descending the group from magnesium to barium, as a result of increasing polarisability, electropositivity and ionic radius.\(^{55-57}\) In this context, the apparent inertness of compound 4a towards formation of calcium analogues of 3b and 5 is intriguing. To this

Fig. 4 DFT calculated free energy profile (BP86-D3(BJ)-benzene/BS2//BP86/BS1), in kcal mol\(^{-1}\), for the reaction of 4b with BCF by (a) pathway II, and (b) pathway III. Ar = 2,6-di-isopropylphenyl.
end, the mechanistic pathway for 4a formation and its onward reaction to the hypothetical calcium complexes 3c’ and 5’ was also calculated (see ESI, Fig. S29 and S30). Viable pathways for BCF-mediated hydride abstraction from the toluene-free intermediate 4a* displayed similarities to pathways II and III for magnesium. Intermediate 4a* was, however, located at −4.7 kcal mol⁻¹ with respect to 1a: some 26.7 kcal mol⁻¹ higher in energy than the crystallographically characterised η⁵-toluene complex 4a (ΔG° = −31.4 kcal mol⁻¹). This latter species, therefore, may be viewed as an energetic sink, which precludes onward reactivity analogous to that observed for its magnesium analogue 4b such that transformation of 4a to 3c’ + 5’ is both kinetically unrealistic with a suggested free energy barrier of 37.9 kcal mol⁻¹ and being almost thermoneutral.

Conclusion

In conclusion, addition of B(C₆F₅)₃ to the alkaline earth phosphido-borane complexes 1a and 1b initially results in P → B adduct formation to provide the monomeric phosphinodiboronate species, 4a and 4b. In the solid state, the calcium complex 4a features an η⁵-coordinated toluene molecule which, although labile in solution, was calculated to stabilise the compound towards onward reactivity. In contrast, arene solvent-coordination to the spectroscopically-detected magnesium analogue 4b was calculated to be endergonic, and this species transforms into the known [HB(C₆F₅)₃]⁻ derivative 3b and the N,P,N’-ligated species 5. Crystallographic characterisation of 5 revealed it to be the product of H₂B = PPh₂ interception by the nucleophilic BDI ligand backbone. Computational assessment of these processes suggests a mechanism involving BCF dissociation and subsequent hydride abstraction to generate the putative intermediate, H₂B = PPh₂. Key to the thermodynamic viability of the process are μ-F-Mg interactions; complexation of H₂B = PPh₂ by BCF was calculated to generate an electrophilic intermediate containing a P → B dative bond and a stabilising ortho-F-BH₃ interaction. These studies demonstrate the potential for cooperative main-group mediated reactivity in the dehydration of phosphine-borane adducts. Whilst non-innocence of the BDI ligand utilised herein is a clear obstacle towards the use of this system in catalytic phosphine-borane dehydropolymerisation, these results will inform future efforts to develop improved and inexpensive catalysts for the synthesis of useful inorganic polymers.

Conflicts of interest

There are no conflicts to declare.

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