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Authors: Anindya Swarnakar, Christian Hering-Junghans, Michael Ferguson, Robert McDonald, and Eric Rivard

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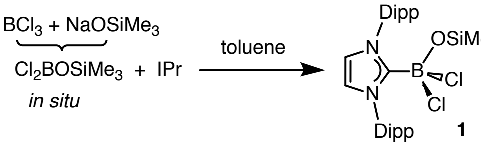
Oxoborane (RBO) Complexation and Concomitant Electrophilic Bond Activation Processes

Anindya K. Swarnakar, Christian Hering-Junghans, Michael J. Ferguson, Robert McDonald, and Eric Rivard*

Abstract: Donor-acceptor complexes of the oxoboranes, ClB=O and HOB=O, were synthesized and each feature short multiply bonded (B=O) linkages. The retention of high Lewis acidic character within these encapsulated monomeric oxoboranes was manifest by their ability to support C-F and Si-O bond activation/functionalization. The reported ClB=O complexes can be regarded as synthetic surrogates of the [BO]+ cation, an inorganic analogue of CO.

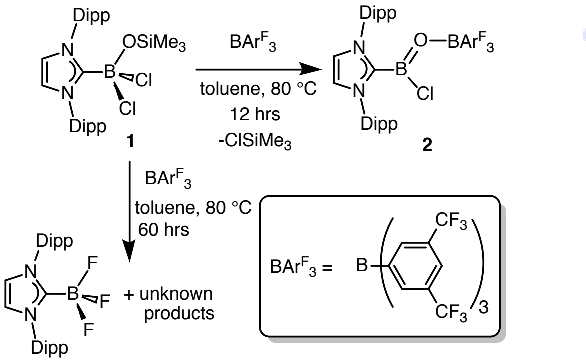
Monomeric oxoboranes (RBO) represent a fundamental building block of the synthetically useful boroxine $(RBO)_3$ compound class.[1] Due to their unsaturated nature and reactive polar B=O bonds, oxoboranes have only been identified within low temperature matrices or in the gas phase.[2] However encouraging work by Pachaly and West revealed the intermediacy of the bulky oxoborane $(2,4,6\text{-}{}^tBu_3C_6H_2)BO$ via trapping experiments.[3,4] This study was complemented by impressive work by Braunschweig and coworkers who used metal-boron complexation to stabilize triply bonded B≡O as a monodentate ligand.[5] Such breakthroughs challenge conventional bonding models and provide chemists with new reactive entities[6] for use in advanced material construction,[7] and for non-metal mediated small molecule activation/catalysis.[8] In this Communication a donor-acceptor protocol[9] is applied to isolate adducts of ClB=O and HOB=O. The resulting chlorooxoborane (ClB=O) adducts can act as electrophilic surrogates for $[B{\equiv}O]^+$ (an inorganic analogue of CO), opening the door to controllable C-F and Si-O bond activation processes.

The title chloroboroxane complexes were obtained via Lewis-acid mediated $ClSiMe_3$ elimination from $IPr{\bullet}Cl_2BOSiMe_3$ (**1**)[10,11] (Eqn. 1 and Scheme 1; IPr = $(HCNDipp)_2C$:; Dipp = $2,6\text{-}{}^iPr_2C_6H_3$). X-ray crystallography confirmed the formation of the dihalosilyl boryl precursor $IPr{\bullet}BCl_2OSiMe_3$ (**1**) that exhibits tetrahedral coordination at boron (Figure S22)[11] and a B-O single bond length of 1.393(2) Å.[4e]



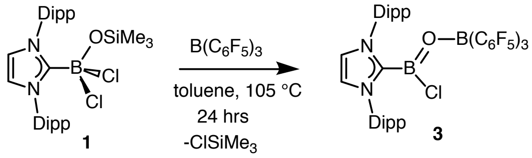
(1)

Compound **1** was heated to 100 °C in toluene for 12 hrs in an attempt to release Me_3SiCl and form a carbene-stabilized ClB=O unit, however no reaction transpired. Treatment of **1** with the Lewis acid BAr^F_3[12,13] (Ar^F = $3,5\text{-}(F_3C)_2C_6H_3$) followed by heating to 80 °C for 12 hrs afforded partial conversion of **1** (20 % by NMR spectroscopy) into the novel oxoborane donor-acceptor complex $IPr{\bullet}ClB{=}O{\bullet}BAr^F_3$ (**2**; Scheme 1) which was later identified by X-ray crystallography (Figure S23).[11] Prolonged heating of an equimolar mixture of **1** and BAr^F_3 in toluene for 60 hrs at 80 °C yielded $IPr{\bullet}BF_3$[14] as a carbene-containing product. This observation is in line with electrophilic boron-based activation of the $C(sp^3)$-F bonds in Ar^F, driven by the formation of strong B-F linkages in $IPr{\bullet}BF_3$ (*vide infra*).



Scheme 1. Reaction of **1** with BAr^F_3 leading to the formation of $IPr{\bullet}B(Cl)O{\bullet}BAr^F_3$ (**2**) and eventual C-F bond activation.

In order to mitigate degradative C-F activation within an oxoborane complex, BAr^F_3 was replaced with $B(C_6F_5)_3$ in the synthesis, as this latter borane contains less reactive $C(sp^2)$-F bonds.[15] Accordingly, stirring a toluene solution of $IPr{\bullet}BCl_2OSiMe_3$ (**1**) and $B(C_6F_5)_3$ at 105 °C for 24 hrs results in the formation of $IPr{\bullet}B(Cl){=}O{\bullet}B(C_6F_5)_3$ (**3**) as colorless crystals (Figure 1) in an isolated yield of 88 %; thus sacrificial C-F bond activation was effectively suppressed (Eqn. 2).



(2)

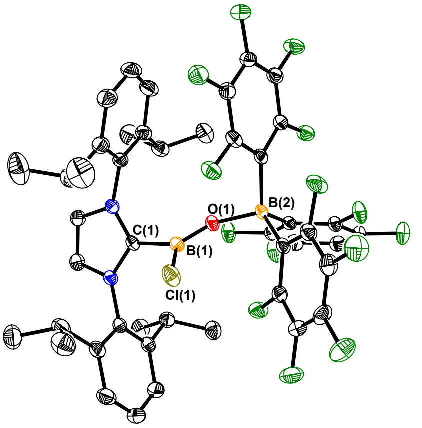


Figure 1. Molecular structure of $IPr{\bullet}B(Cl)O{\bullet}B(C_6F_5)_3$ (**3**) with thermal ellipsoids presented at a 30 % probability level. All H atoms have been omitted for clarity. Selected bond lengths (Å) and angles (deg): C(1)-B(1) 1.601(4), B(1)-O(1) 1.296(3), B(1)-Cl(1) 1.773(3), B(2)-O(1) 1.518(3); C(1)-B(1)-O(1) 117.3(2), B(1)-O(1)-B(2) 142.1(2), Cl-B(1)-O(1) 127.7(2), C(1)-B(1)-Cl 114.97(19).

A. K. Swarnakar, Dr. C. Hering-Junghans, Dr. M. J. Ferguson, Dr. R. McDonald, Prof. Dr. E. Rivard
Department of Chemistry, University of Alberta
11227 Saskatchewan Drive, Edmonton, Alberta, T6G 2G2 (Canada)
E-mail: erivard@ualberta.ca
Homepage: http://www.chem.ualberta.ca/~erivard/

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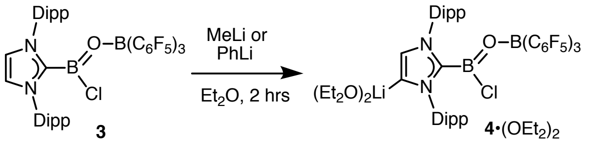
Two singlet resonances were found at 26.1 and -2.7 ppm in the ^{11}B NMR spectrum of **3**, consistent with the presence of 3- and 4-coordinate environments, respectively. A trigonal planar geometry exists about the oxoborane boron atom in **3** (B(1); Figure 1) as evidenced by a bond angle sum at B of 359.97(19)°. Most striking was the very short B-O length in **3** [1.296(3) Å], consistent with B=O π-bond character. A similar B-O bond distance [1.304(2) Å] was found in Cowley's nacnac $HC\{C(CH_3)N(C_6F_5)\}_2BO{\bullet}AlCl_3$,[4c] however the B=O linkage in **3** is substantial longer than the B-O triple bond length of 1.210(3) Å found in Braunschweig's *trans*-$PhS(Cy_3P)_2PtBO$.[5] A diagnostic ν(BO) IR band is present at 1646 cm^{-1} in **3** which is comparable to the ν(BO) vibration noted in Kinjo's 1,2,3,4-triazaborole-based oxoborane (1636 cm^{-1}).[4f]

Compounds **1** and **3** were investigated by DFT methods, and as anticipated, NBO analysis afforded a Wiberg Bond Index (WBI) for the central B-O linkage in **3** of 1.123, indicative of multiple bond character.[11] Substantial polarization of the σ and π components of this B-O bonding manifold toward O were also found (ca. 83 % of overall B-O bonding density located at oxygen). Natural Population Analysis (NPA) revealed a higher positive charge (and possible electrophilic character) at the oxoborane boron atom, Cl*B*O, in **3** (0.887 e) in relation to the boron center in $IPr{\bullet}Cl_2BOSiMe_3$ (**1**) (0.621 e). The LUMO of **3** has distinct B=O π*-character whereas the accompanying B=O π interaction in this ClB=O complex is energetically low lying (HOMO-12).[11]

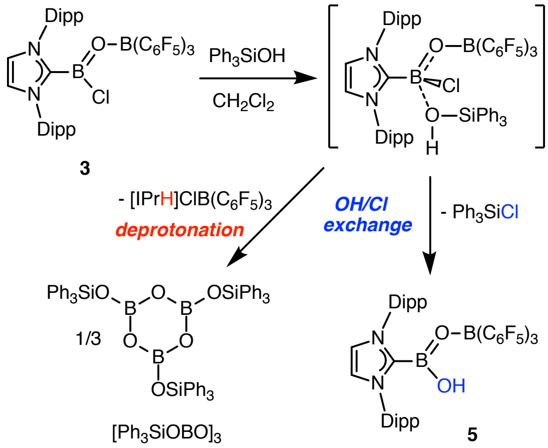
Motivated by the presence of a potentially functionalizable B-Cl bond in **3**, we attempted to synthesize the donor-acceptor complexes of parent oxoborane HBO and its organo counterparts RBO (R = Me or Ph). When $IPr{\bullet}B(Cl)O{\bullet}B(C_6F_5)_3$ (**3**) was combined with the hydride source $K[HB^sBu_3]$ no reaction occurred, despite heating to 80 °C in toluene. However reaction of **3** with MeLi (or PhLi) in Et_2O led to formation of the IPr-backbone deprotonated[16] product $[(Et_2O)_2Li][IPr{\bullet}B(Cl)O{\bullet}B(C_6F_5)_3]$ (**4•(OEt₂)₂**) (Eqn. 3; see Figure S24 for crystallographic characterization).[11]



(3)

We attempted to promote chloride migration in $IPr{\bullet}ClB{=}O{\bullet}B(C_6F_5)_3$ (**3**) to yield the oxoborylium salt $[IPr{\bullet}B{\equiv}O]^+[Cl\text{-}B(C_6F_5)_3]^-$, by heating **3** to 140 °C in xylenes for 3 days; this resulted in the complete conversion of **3** into the imidazolium salt $[IPrH]ClB(C_6F_5)_3$ (Figure S25).[11] One possible path for this transformation (reproduced many times under rigorously anhydrous conditions) is initial chloride abstraction by $B(C_6F_5)_3$ to yield a highly reactive oxoborylium adduct $[IPr{\bullet}B{\equiv}O]ClB(C_6F_5)_3$. This source of electrophilic $[B{\equiv}O]^+$ could then react with surface Si-OH groups of the glass vessel, liberating H$^+$ (trapped as IPrH$^+$) while concurrently borylating the glass.

In order to investigate the above borylation reaction in a homogenous system, Ph_3SiOH was combined with an equimolar amount of **3** at room temperature (Scheme 2). This reaction afforded a 1:1 mixture of the novel (hydroxy)oxoborane complex $IPr{\bullet}B(OH)O{\bullet}B(C_6F_5)_3$ (**5**) and the previously noted imidazolium borate salt $[IPrH]ClB(C_6F_5)_3$. To our surprise, the formation of Ph_3SiCl (via Cl/OH exchange) was confirmed by ^{1}H and ^{13}C{^{1}H} NMR, ruling out the direct hydrolysis of **3** to form **5**; the known boroxine[11] $[Ph_3SiOBO]_3$ was also recovered as a product (Scheme 2). By increasing the amount of Ph_3SiOH to 5 equiv., the amount of **5** formed increased two-fold. One preliminary mechanistic postulate is that compound **3** and Ph_3SiOH initially interacts to afford transient $[IPr{\bullet}B(HOSiPh_3)(Cl)O{\bullet}B(C_6F_5)_3]$ which can undergo Ph_3SiCl elimination/hydroxide-chloride metathesis to form $IPr{\bullet}B(OH)O{\bullet}B(C_6F_5)_3$ (**5**) (Scheme 2; right path). The same $HOSiPh_3$ interaction complex of ClB=O could undergo carbene-induced deprotonation of $HOSiPh_3$, followed by halide migration to $B(C_6F_5)_3$ to yield $[IPrH]ClB(C_6F_5)_3$ and transient $Ph_3SiO\text{-}B{=}O$, which then trimerizes to $[Ph_3SiOBO]_3$ (Scheme 2; left path). At high concentrations of Ph_3SiOH additional silanol-silanol hydrogen bonding (and a reduction of $HOSiPh_3$ Brønsted acidity) could lead to enhancement of Ph_3SiCl loss over carbene-induced deprotonation. Notably, the mild abstraction of OH$^-$ from a silanol by the encapsulated ClB=O unit in **3** suggests a high degree of electrophilicity is present, in line with our computational studies (*vide supra*).



Scheme 2. Reaction of **3** with Ph_3SiOH and the formation of $IPr{\bullet}B(OH)O{\bullet}B(C_6F_5)_3$ (**5**).

As shown in Figure 2, the core HOB=O unit in compound **5** possesses one shorter B(1)-O(2) bond [1.311(3) Å] and a longer B(1)-O(1) linkage involving the hydroxyl group [1.354(3) Å]; the remaining dative B-O bond between $B(C_6F_5)_3$ and the HOB=O unit is substantially elongated [1.504(3) Å]. Compound **5** is the first isolable complex of HOB=O; monomeric HOB=O was previously identified in the gas phase during the thermal conversion of orthoboric acid ($B(OH)_3$) to metaboric acid ($[HOBO]_3$).[17]

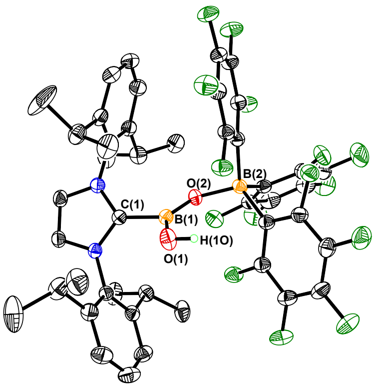
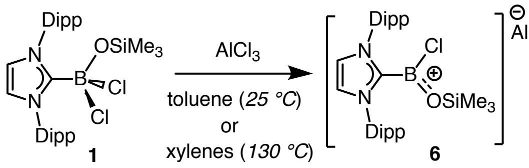


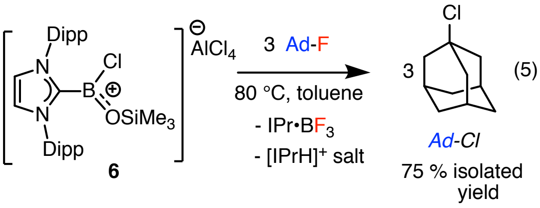
Figure 2. Molecular structure of $IPr{\bullet}B(OH)O{\bullet}B(C_6F_5)_3$ (**5**) with thermal ellipsoids presented at a 30 % probability level. All carbon-bound H atoms have been omitted for clarity. Selected bond lengths (Å) and angles (deg): C(1)-B(1) 1.636(3), B(1)-O(2) 1.311(3), B(1)-O(1) 1.354(3), O(2)-B(2) 1.504(3); C(1)-B(1)-O(1) 112.06(17), O(1)-B(1)-O(2) 128.97(19), B(1)-O(2)-B(2) 133.95(16).

We also attempted to form carbene-stabilized $[BO]^+$ salts $[IPr•BO•B(C_6F_5)_3]A^-$ (A^- = weakly coordinating anion) by treating **3** with either $NaBAr^F_4$ or AgOTf (OTf = OSO_2CF_3). While no reaction transpired at room temperature, heating $NaBAr^F_4$ with **3** at 80 °C led to C-F bond activation and the generation of $IPr•BF_3$.

The production of the ClB=O adduct **3** from $IPr•Cl_2BOSiMe_3$ (**1**) and $B(C_6F_5)_3$ (Eqn. 2) likely proceeds via the siloxyboronium cation $[IPr•BCl(OSiMe_3)]^+$.[18] The related tetrachloroaluminate salt $[IPr•BCl(OSiMe_3)]AlCl_4$ (**6**) was obtained in a quantitative yield by combining **1** and $AlCl_3$ (Eqn. 4). X-ray crystallography (Figure 3) showed the presence of a trigonal planar boron center in **6**, with B-O and B-Cl distances [1.310(4) Å and 1.758(3) Å, respectively] that match well those present within the ClB=O unit of **3**. NBO analysis[11] of the $[IPr•BCl(OSiMe_3)]^+$ cation in **6** afforded a WBI of 1.049 for the B-O bond, pointing towards possible double bond character. The computed NPA charge for the boron center in **6** (0.863 e) is also similar to that found within the ClB=O array in **3**.[19] Thus possible parallel C-F activation chemistry could be instigated by the electrophilic boron center in **6**.



(4)



(5)

$[IPr•BCl(OSiMe_3)]AlCl_4$ (**6**) is thermally stable with no sign of decomposition or $ClSiMe_3$ release found after heating in xylenes at 130 °C for 18 hrs. However compound **6** was still found to be an effective reagent for the C-F activation and functionalization (halogenation) of 1-fluoroadamantane AdF (Eqn. 5). When **6** was reacted with 3 equivalents of Ad-F at 100 °C in toluene for 16 hrs, the formation of 1-chloroadamantane (Ad-Cl) occurred (75 % isolated yield), along with the spectroscopic identification of $IPr•BF_3$ (60 %), an $[IPrH]^+$ salt (32 %) and a minor unknown carbene-containing species (<8 %) as co-products. As before, the formation of strong B-F bonds is likely spurring this process, and overall this transformation is a useful addition to the field of main group element instigated bond activation.[15], [20], [21]

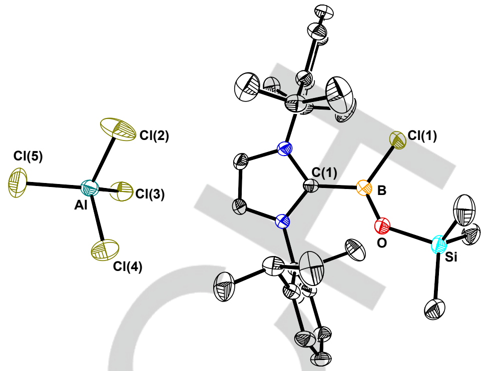


***Figure 3**. Molecular structure of $[IPr•BCl(OSiMe_3)]AlCl_4$ (**6**) with thermal ellipsoids presented at a 30 % probability level. All hydrogen atoms have been omitted for clarity. Selected bond lengths (Å) and angles (deg): C(1)-B 1.578(4), B-O 1.310 (4), B-Cl(1) 1.758 (3), O-Si 1.7023(19); C(1)-B-O 119.3(2), C(1)-B-Cl(1) 119.1(2), Cl(1)-B-O 121.7(2).*

In conclusion, we have synthesized the first coordination complexes of oxoboranes, ClB=O and HOB=O. The ClB=O adducts and their siloxyboronium counterparts can be viewed as synthetic sources of the electrophilic oxoborylium cation $[B{\equiv}O]^+$, an inorganic analogue of CO. Moreover initial studies show that these species can activate/functionalize alkane C-F and silanol Si-O bonds, showing a high degree of reactivity within these "trapped" oxoboranes. These promising initial studies provide ample motivation for us to explore $[B{\equiv}O]^+$ surrogates within the context of nonmetal-based catalysis,[15] and to pursue to the synthesis of related unsaturated main group species[6] featuring novel bonding motifs.

Acknowledgements

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Keywords: donor-acceptor system • oxoborane • boron • oxygen • bond activation

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[11] For full synthetic, crystallographic and computational details see the Supporting Information. CCDC 1543332-1543338 for compounds **1**-**6** and $[IPrH][ClB(C_6F_5)_3]$.

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[18] The formation of **3** from $IPr{\bullet}BCl_2OSiMe_3$ (**1**) could also proceed via initial coordination of $B(C_6F_5)_3$ to the oxygen atom in **1**, followed by intramolecular loss of $ClSiMe_3$.

[19] One can also view **3** as having a similar bonding environment as **6**, and thus both species can be regarded as electrophilic RO-substituted borocations.

[20] For related studies on main group element-based C-F activation, see: (a) T. Stahl, H. F. T. Klare, M. Oestreich, *ACS Catal*. **2013**, *3*, 1578. (b) C. Douvris, O. V. Ozerov, *Science* **2008**, *321*, 1188. (c) C. B. Caputo, D. W. Stephan, *Organometallics* **2012**, *31*, 27. (d) M. Alcarazo, C. Gomez, S. Holle, R. Goddard, *Angew. Chem.* **2010**,*122,* 5730; *Angew. Chem., Int. Ed.* **2010**, *49*, 5788. (e) M. Ahrens, G. Scholz, T. Braun, E. Kemnitz, *Angew. Chem*. **2013**,*125*, 5436; *Angew. Chem., Int. Ed*. **2013**, *52*, 5328.

[21] Attempts to induce $C(sp^2)$-F activation by heating **6** in fluorobenzene at 80 °C gave no reaction. For an early report concerning the activation of a $–CF_3$ group by an electron deficient boron center, see: T. Chivers, *Can. J. Chem.* **1970**, *48*, 3856.

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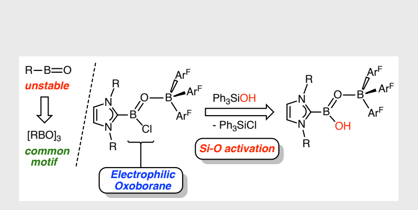
COMMUNICATION



Giving a HO-B=O molecule a home: In this Communication we use a general donor-acceptor stabilization protocol to isolate ClB=O and HOB=O complexes, and demonstrate the electrophilic activation of C-F and Si-O bonds.

*A. K. Swarnakar, C. Hering-Junghans, M. J. Ferguson, R. McDonald, E. Rivard**

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