

Supplementary Material

for the paper

9-H-9-Borafluorene Dimethyl Sulphide Adduct: Product of a Unique Ring- Contraction Reaction and a Useful Hydroboration Reagent.

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and Matthias Wagner***

Content:

1. Synthesis and NMR-spectroscopic characterisation of **2**(DMS), **4**, **5**, (Li(Et₂O))₂[**6**], **7**, *t*BuC(H)=C(H)BC₁₂H₈ and *t*BuC(H)₂-C(H)(BC₁₂H₈)₂; conversion of **7** into **2**(DMS).
2. Single crystal X-ray structure analyses of **2**(DMS), **4**, **5**, (Li(Et₂O))₂[**6**] and **7**.

1. Synthesis and NMR-spectroscopic characterisation of 2(DMS), 4, 5, (Li(Et₂O))₂[6], 7, *t*BuC(H)=C(H)BC₁₂H₈ and *t*BuC(H)₂-C(H)(BC₁₂H₈)₂; conversion of 7 into 2(DMS).

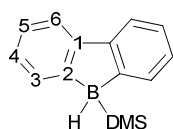
All reactions were carried out under an atmosphere of dry nitrogen using Schlenk techniques. Hexane, (deutero)benzene, Et₂O, THF and dimethyl sulphide (DMS) were dried over Na/benzophenone and freshly distilled prior to use; CDCl₃ was dried over CaH₂ and stored over molecular sieves (4 Å). NMR: Bruker AM250, Avance300 and Avance400; all NMR spectra were measured at 20 °C. Chemical shift values (¹H, ¹³C{¹H}) are reported in parts per million relative to SiMe₄ and were referenced to residual solvent signals. ¹¹B and ¹¹B{¹H} NMR spectra are referenced to external BF₃·Et₂O. Abbreviations: s = singlet, d = doublet, vt = virtual triplet, q = quartet, m = multiplet, n. o. = not observed. 9-Br-9-borofluorene (**3**),^{1S} 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (*i*PrO-Bpin)^{2S} and 2,2'-dilithiobiphenyl bis-tmeda adduct^{3S} were prepared according to literature procedures.

[1S] A. Hübner, H.-W. Lerner, M. Wagner and M. Bolte, *Acta. Cryst.*, 2010, **E66**, o444.

[2S] R. W. Hoffmann, R. Metternich and J. W. Lanz, *Liebigs Ann. Chem.*, 1987, 881-887.

[3S] T. Schaub and U. Radius, *Tetrahedron Lett.*, 2005, **46**, 8195-8197.

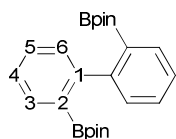
Synthesis of 2(DMS). Neat Et₃SiH (0.55 cm³, 0.40 g, 3.44 mmol) was added at r.t. via syringe to a slowly stirred solution of **3** (0.44 g, 1.81 mmol) in C₆H₆ (4.4 cm³). A colour change from yellow to almost colourless was observed about 10 min later. At this point, neat dry dimethyl sulphide (0.66 cm³, 0.56 g, 9.03 mmol) was added. The resulting clear solution was slowly evaporated to dryness under vacuum to obtain a crystalline off-white solid. Yield: 0.39 g (95 %).



¹H NMR: δ_H(400.1 MHz; C₆D₆) 1.10 (6 H, s, SCH₃), 3.75 (1 H, br, BH), 7.26 (2 H, vtd, ³J_{HH} = 7.2 Hz, ⁴J_{HH} = 1.1 Hz, H-4 or 5), 7.34 (2 H, vtd, ³J_{HH} = 7.4 Hz, ⁴J_{HH} = 1.3 Hz, H-4 or 5), 7.64 (2 H, dm, ³J_{HH} = 7.1 Hz, H-3 or 6), 7.79 (2 H, dm, ³J_{HH} = 7.5 Hz, H-3 or 6); ¹¹B NMR: δ_B(128.4 MHz; C₆D₆) -7.1 (d, ¹J_{BH} = 103 Hz); ¹¹B{¹H} NMR: δ_B(128.4 MHz; C₆D₆) -7.1 (*h*_{1/2} = 130 Hz); ¹³C{¹H}NMR: δ_C(100.6 MHz; C₆D₆) 19.0 (SCH₃), 120.0 (C-3 or 6), 126.6 (C-4 or

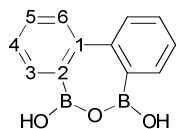
5), 127.9 (C-4 or 5), 132.0 (C-3 or 6), 150.8 (C-1), n.o. (C-2). *Note:* For full reproducibility, the concentrations of the reagents are of critical importance.

Synthesis of 4. Neat *i*PrO–Bpin (2.4 cm³, 2.2 g, 11.8 mmol) was slowly added with stirring at –78 °C to a suspension of 2,2'-dilithiobiphenyl bis-tmeda adduct (1.56 g, 3.92 mmol) in Et₂O (60 cm³). After the addition was complete, the mixture was stirred for another 30 min at –78 °C, slowly warmed to r.t. and stirred overnight. The reaction mixture was quenched with H₂O (20 cm³) and aqueous HCl (5%) was added until a pH of ~7 was achieved. The mixture was extracted with CHCl₃ (3 × 20 cm³) and the combined extracts were dried over anhydrous Na₂SO₄. After filtration, the filtrate was evaporated to dryness under vacuum. Colourless crystal plates were obtained by storing a saturated solution of **4** in petroleum ether at –40 °C. Yield: 0.96 g (60%).



¹H NMR: δ_{H} (300.0 MHz; CDCl₃) 1.09 (24 H, s, CH₃), 7.26 (2 H, dm, ³J_{HH} = 7.5 Hz, H-3 or 6), 7.29 (2 H, vtd, ³J_{HH} = 7.3 Hz, ⁴J_{HH} = 1.4 Hz, H-4 or 5), 7.38 (2 H, vtd, ³J_{HH} = 7.4 Hz, ⁴J_{HH} = 1.6 Hz, H-4 or 5), 7.67 (2 H, dm, ³J_{HH} = 7.3 Hz, H-3 or 6); ¹¹B{¹H} NMR: δ_{B} (96.3 MHz; CDCl₃) 31.3 (*h*_{1/2} = 350 Hz); ¹³C{¹H} NMR: δ_{C} (75.4 MHz; CDCl₃) 24.7 (CH₃), 83.3 (C(CH₃)₂), 125.8, 129.2, 129.3, 133.7 (C-3,4,5,6), 149.6 (C-1), n.o. (C-2); ESI-MS: *m/z* 407.3 ([M+H]⁺, 100%); Found: C, 70.80; H, 7.89. C₂₄H₃₂B₂O₄ [406.12] requires C, 70.98; H, 7.94.

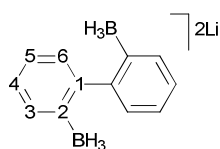
Synthesis of 5. Neat B(OMe)₃ (10.0 cm³, 9.2 g, 88.5 mmol) was added dropwise with stirring at –78 °C to a suspension of 2,2'-dilithiobiphenyl bis-tmeda adduct (7.86 g, 19.73 mmol) in Et₂O (150 cm³). After the addition was complete, the mixture was stirred for another 30 min at –78 °C, slowly warmed to r.t. and stirred overnight, whereupon a colourless precipitate formed. The precipitate was dissolved by the addition of dry THF (60 cm³), and stirring was continued for an additional 2 h. H₂O (75 cm³) was added, followed by aqueous HCl (5%; 65 cm³). The reaction mixture was vigorously stirred for another 4 h. The organic layer was removed using a separation funnel and the aqueous phase was extracted with CH₂Cl₂ (3 × 100 cm³). The combined organic phases were dried over anhydrous Na₂SO₄. After filtration, the filtrate was evaporated to dryness under vacuum. Colourless crystal needles of **5** were grown by recrystallisation from toluene/petroleum ether. Yield: 3.31 g (75%).



^1H NMR: δ_{H} (300.0 MHz; DMSO- d_6) 7.37 (2 H, vtd, $^3J_{\text{HH}} = 7.2$ Hz, $^4J_{\text{HH}} = 1.5$ Hz, H-4 or 5), 7.54 (2 H, vtd, $^3J_{\text{HH}} = 7.5$ Hz, $^4J_{\text{HH}} = 1.5$ Hz, H-4 or 5), 7.59 (2 H, dm, $^3J_{\text{HH}} = 7.9$ Hz, H-3 or 6), 7.72 (2 H, dm, $^3J_{\text{HH}} = 7.3$ Hz, H-3 or 6), 9.15 (s, 2 H, OH); $^{11}\text{B}\{^1\text{H}\}$ NMR: δ_{B} (96.3 MHz; DMSO- d_6) ca. 31 (very broad); $^{13}\text{C}\{^1\text{H}\}$ NMR: δ_{C} (75.4 MHz; DMSO- d_6) 126.4, 129.3, 130.5, 134.0 (C-3,4,5,6), 145.1 (C-1), n.o. (C-2); ESI-MS: m/z 222.6 ($[\text{M}-\text{H}]^-$, 30%); Found: C, 64.46; H, 4.64. $\text{C}_{12}\text{H}_{10}\text{B}_2\text{O}_3$ [223.82] requires C, 64.40; H, 4.50.

Note: ESI-MS spectra of **5** recorded on MeOH/H₂O solutions in the negative mode showed that under these conditions the compound tends to exchange terminal BOH for BOMe substituents and that also an intermolecular condensation takes place which leads to dimeric species.

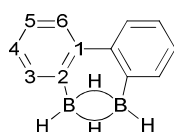
Synthesis of $(\text{Li}(\text{Et}_2\text{O}))_2[\mathbf{6}]$. A solution of **5** (0.46 g, 2.06 mmol) in Et₂O (50 cm³) was cooled to 0 °C. A solution (1 M) of Li[AlH₄] (6.88 cm³, 6.88 mmol) in Et₂O was added dropwise with stirring over a period of 30 min, whereupon a colourless precipitate formed (*Caution: vigorous hydrogen evolution occurs*). Stirring was continued at 0 °C for another 30 min and then at r.t. overnight. The reaction mixture was filtered, the insolubles were washed with Et₂O (15 cm³) and the combined ether phases were stored at -40 °C to obtain colourless crystal plates. Yield: 0.53 g (75%).



^1H NMR: δ_{H} (400.1 MHz; C₆D₆/THF) 1.49 (6 H, q, $^1J_{\text{HB}} = 77$ Hz, BH₃), 7.18 (2 H, vtd, $^3J_{\text{HH}} = 7.4$ Hz, $^4J_{\text{HH}} = 1.6$ Hz, H-4 or 5), 7.26 (2 H, vtd, $^3J_{\text{HH}} = 7.3$ Hz, $^4J_{\text{HH}} = 1.5$ Hz, H-4 or 5), 7.41 (dd, $^3J_{\text{HH}} = 7.3$ Hz, $^4J_{\text{HH}} = 1.5$ Hz, H-6), 8.07 (2 H, br, H-3); ^{11}B NMR: δ_{B} (128.4 MHz; C₆D₆/THF) -28.3 (q, $^1J_{\text{BH}} = 77$ Hz); $^{11}\text{B}\{^1\text{H}\}$ NMR δ_{B} (128.4 MHz, C₆D₆/THF) -28.3 ($h_{1/2} = 25$ Hz); $^{13}\text{C}\{^1\text{H}\}$ NMR: δ_{C} (100.6 MHz; C₆D₆/THF) 124.6, 125.4, 129.1, 138.0 (C-3,4,5,6), 153.7 (C-1), n.o. (C-2); ESI-MS: m/z 187.2 ($[\text{Li}[\mathbf{6}]]^-$, 100%); Found: C, 70.28; H, 9.89. $\text{C}_{12}\text{H}_{14}\text{B}_2\text{Li}_2$ [193.73] \times 2 C₄H₁₀O [74.12] requires C, 70.25; H, 10.02.

Note: The synthesis of $(\text{Li}(\text{Et}_2\text{O}))_2[\mathbf{6}]$ starting from **4** proceeds in an analogous manner.

Synthesis of 7. A solution of $(\text{Li}(\text{Et}_2\text{O}))_2[\mathbf{6}]$ (0.56 g, 1.64 mmol) in Et_2O (75 cm^3) was cooled to $-78 \text{ }^\circ\text{C}$. A solution of Me_3SiCl (2.10 cm^3 , 1.80 g, 16.5 mmol) in Et_2O (5 cm^3) was added under vigorous stirring over a period of 30 min. The reaction mixture was slowly warmed to r.t. and stirred overnight. After filtration, the insolubles were washed with Et_2O (5 cm^3) and the combined ether phases were evaporated to dryness under vacuum. Yield: 0.20 g (70%). Analytical pure colourless crystal needles were obtained by slow evaporation of a *n*-hexane solution.



^1H NMR: δ_{H} (300.0 MHz; C_6D_6) 1.30 (2 H, very broad, $\text{BH}_{\text{bridging}}$), 4.77 (2 H, q, $^1J_{\text{HB}} = 120 \text{ Hz}$, $\text{BH}_{\text{terminal}}$), 7.27 (2 H, vtd, $^3J_{\text{HH}} = 7.3 \text{ Hz}$, $^4J_{\text{HH}} = 1.1 \text{ Hz}$, H-4 or 5), 7.44 (2 H, ddd, $^3J_{\text{HH}} = 8.3 \text{ Hz}$, 7.3 Hz , $^4J_{\text{HH}} = 1.7 \text{ Hz}$, H-4 or 5), 7.85 (2 H, d, $^3J_{\text{HH}} = 7.3 \text{ Hz}$, H-3 or 6), 8.31 (2 H, dm, $^3J_{\text{HH}} = 8.3 \text{ Hz}$, H-3 or 6). ^{11}B NMR: δ_{B} (96.3 MHz; C_6D_6) 13.7 (d, $^1J_{\text{BH}} = 120 \text{ Hz}$). $^{11}\text{B}\{^1\text{H}\}$ NMR: δ_{B} (96.3 MHz; C_6D_6) 13.7 ($h_{1/2} = 80 \text{ Hz}$); $^{13}\text{C}\{^1\text{H}\}$ NMR: δ_{C} (75.4 MHz; C_6D_6) 125.0, 126.7, 131.0, 138.5 (C-3,4,5,6), 141.3 (C-1), n.o. (C-2).

Synthesis of $t\text{BuC}(\text{H})=\text{C}(\text{H})\text{BC}_{12}\text{H}_8$. A solution of $\mathbf{2}(\text{DMS})$ (46 mg, 0.20 mmol) in toluene (5 cm^3) was added dropwise with stirring at r.t. over a period of 2 h to neat $t\text{BuC}\equiv\text{CH}$ (1.5 cm^3 , 1.00 g, 12.3 mmol). The resulting green solution was evaporated to dryness in vacuo to obtain a green microcrystalline solid. NMR spectroscopy on this crude product revealed an essentially quantitative conversion to the DMS adduct of $t\text{BuC}(\text{H})=\text{C}(\text{H})\text{BC}_{12}\text{H}_8$. The adduct has to be stored for several days under a dynamic vacuum in order to completely remove the coordinated DMS.

NMR spectroscopic data of $t\text{BuC}(\text{H})=\text{C}(\text{H})\text{BC}_{12}\text{H}_8$ are given in ref.[9].

Synthesis of $t\text{BuC}(\text{H})_2-\text{C}(\text{H})(\text{BC}_{12}\text{H}_8)_2$. A solution of $t\text{BuC}\equiv\text{CH}$ (0.02 cm^3 , 13 mg, 0.16 mmol) in toluene (5 cm^3) was added dropwise with stirring at r.t. over a period of 1.5 h to a solution of $\mathbf{2}(\text{DMS})$ (70 mg, 0.31 mmol) in toluene (2 cm^3). The resulting yellow solution was evaporated to dryness in vacuo. The crude product was recrystallised from toluene to obtain single-crystalline $t\text{BuC}(\text{H})_2-\text{C}(\text{H})(\text{BC}_{12}\text{H}_8)_2$ (Yield: 75%).

NMR spectroscopic data of $t\text{BuC}(\text{H})_2-\text{C}(\text{H})(\text{BC}_{12}\text{H}_8)_2$ are given in ref.[9].

Conversion of 7 into 2(DMS). In an NMR tube, excess Me₂S in C₆D₆ was added at r.t. to a solution of **7** in C₆D₆. The tube was flame-sealed and investigated by NMR spectroscopy.

¹H NMR: δ_H(300.0 MHz, C₆D₆/DMS) 7.14 (2 H, vtd, ³J_{HH} = 7.3 Hz, ⁴J_{HH} = 1.2 Hz, H-4 or 5), 7.23 (2 H, vtd, ³J_{HH} = 7.4 Hz, ⁴J_{HH} = 1.4 Hz, H-4 or 5), 7.51 (2 H, dm, ³J_{HH} = 7.0 Hz, H-3 or 6), 7.67 (2 H, dm, ³J_{HH} = 7.5 Hz, H-3 or 6), n.o. (BH). ¹¹B NMR: δ_B(96.3 MHz; C₆D₆/DMS) -7.3 (d, ¹J_{BH} = 106 Hz, **2**(DMS)), -19.6 (q, ¹J_{BH} = 105 Hz, H₃B(DMS)). ¹¹B{¹H} NMR: δ_B(96.3 MHz; C₆D₆/DMS) -7.3 (*h*_{1/2} = 100 Hz, **2**(DMS)), -19.6 (*h*_{1/2} = 15 Hz, H₃B(DMS)).

Note: The ¹H NMR spectrum of **2**(DMS) depends on the amount of free DMS present in the C₆D₆ solution. The chemical shift values given here thus deviate from the values compiled above for a sample in neat C₆D₆. Authentic **2**(DMS), prepared from **3** / Et₃SiH and measured in C₆D₆/DMS, possesses the same proton shift values as given here.

2. Single crystal X-ray structure analyses of **2(DMS)**, **4**, **5**, **(Li(Et₂O))₂[6]** and **7**.

All crystals were measured on a STOE IPDS-II diffractometer with graphite-monochromated MoK α radiation. An absorption correction was applied to the data of **2(DMS)** using the MULABS^{4S} routine in PLATON^{5S}. The structures were solved by direct methods using the program SHELXS^{6S} and refined with full-matrix least-squares on F^2 using the program SHELXL97^{7S}. Hydrogen atoms bonded to C were placed on ideal positions and refined with fixed isotropic displacement parameters using a riding model.

The coordinates of the hydrogen atom bonded to B in **2(DMS)** were refined with a B–H distance restraint of 1.20(5) Å, whereas the hydrogen atoms bonded to B in **(Li(Et₂O))₂[6]** and **7** were freely refined. The hydrogen atoms bonded to O in **5** were freely refined. The crystal of **2(DMS)** was small and only weakly diffracting; the crystal of **(Li(Et₂O))₂[6]** was twinned with a contribution of 0.29(1) of the minor component. For these reasons, the $wR(F^2)$ values are elevated in both cases.

[4S] R. H. Blessing, *Acta Cryst.*, 1995, **A51**, 33-38. [5S] A. L. Spek, *Acta Cryst.*, 2009, **D65**, 148-155. [6S] G. M. Sheldrick, *Acta Cryst.*, 1990, **A46**, 467-473. [7S] G. M. Sheldrick, *SHELXL-97. A Program for the Refinement of Crystal Structures*. Universität Göttingen, Göttingen, 1997.

CCDC reference numbers: **2(DMS)** (CCDC 837677), **4** (CCDC 837678), **5** (CCDC 837679), **(Li(Et₂O))₂[6]** (CCDC 837680) and **7**(CCDC 837681).

Crystal data of 2(DMS). C₁₄H₁₅BS, $M = 226.13 \text{ g mol}^{-1}$, monoclinic, $a = 6.9507(13) \text{ \AA}$, $b = 16.302(3) \text{ \AA}$, $c = 22.288(4) \text{ \AA}$, $\beta = 91.155(14)^\circ$, $V = 2524.9(8) \text{ \AA}^3$, $T = 173(2) \text{ K}$, space group $P2_1/c$, $Z = 8$, $\mu(\text{Mo-K}\alpha) = 0.225 \text{ mm}^{-1}$, 16340 reflections measured, 4445 unique ($R_{\text{int}} = 0.1351$) which were used in all calculations. The final $wR(F^2)$ was 0.2382 (all data).

Crystal data of 7. C₁₂H₁₂B₂, $M = 177.84 \text{ g mol}^{-1}$, monoclinic, $a = 9.4074(12) \text{ \AA}$, $b = 14.8970(14) \text{ \AA}$, $c = 7.1428(10) \text{ \AA}$, $\beta = 99.524(11)^\circ$, $V = 987.2(2) \text{ \AA}^3$, $T = 173(2) \text{ K}$, space group $P2_1/c$, $Z = 4$, $\mu(\text{Mo-K}\alpha) = 0.064 \text{ mm}^{-1}$, 12161 reflections measured, 2012 unique ($R_{\text{int}} = 0.0688$) which were used in all calculations. The final $wR(F^2)$ was 0.1280 (all data).

Crystal data of 4. $C_{24}H_{32}B_2O_4$, $M = 406.12 \text{ g mol}^{-1}$, monoclinic, $a = 11.1036(5) \text{ \AA}$, $b = 11.2005(4) \text{ \AA}$, $c = 19.5494(10) \text{ \AA}$, $\beta = 105.072(4)^\circ$, $V = 2347.64(18) \text{ \AA}^3$, $T = 173(2) \text{ K}$, space group $P2_1/n$, $Z = 4$, $\mu(\text{Mo-K}\alpha) = 0.075 \text{ mm}^{-1}$, 34104 reflections measured, 4459 unique ($R_{\text{int}} = 0.0689$) which were used in all calculations. The final $wR(F^2)$ was 0.1568 (all data).

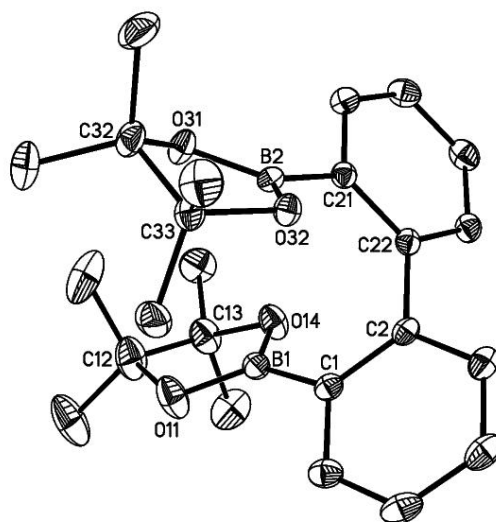


Figure S1. Molecular structure and numbering scheme of compound **4**; displacement ellipsoids are drawn at the 30% probability level; H atoms are omitted for clarity. Selected bond lengths [\AA], atom...atom distances [\AA], bond angles [$^\circ$] and dihedral angle [$^\circ$]: B(1)–O(11) = 1.358(3), B(1)–O(14) = 1.351(2), B(1)–C(1) = 1.565(3), B(2)–O(31) = 1.368(2), B(2)–O(32) = 1.363(2), B(2)–C(21) = 1.560(3), B(1)···B(2) = 3.433(3); O(11)–B(1)–O(14) = 113.2(2), O(31)–B(2)–O(32) = 113.4(2); Ar(C(1))//Ar(C(21)) = 62.3.

Crystal data of 5. $C_{12}H_{10}B_2O_3$, $M = 223.82 \text{ g mol}^{-1}$, monoclinic, $a = 10.130(2) \text{ \AA}$, $b = 7.2521(15) \text{ \AA}$, $c = 15.992(3) \text{ \AA}$, $\beta = 105.63(3)^\circ$, $V = 1131.4(4) \text{ \AA}^3$, $T = 173(2) \text{ K}$, space group $P2_1/c$, $Z = 4$, $\mu(\text{Mo-K}\alpha) = 0.090 \text{ mm}^{-1}$, 7543 reflections measured, 2114 unique ($R_{\text{int}} = 0.1062$) which were used in all calculations. The final $wR(F^2)$ was 0.1186 (all data).

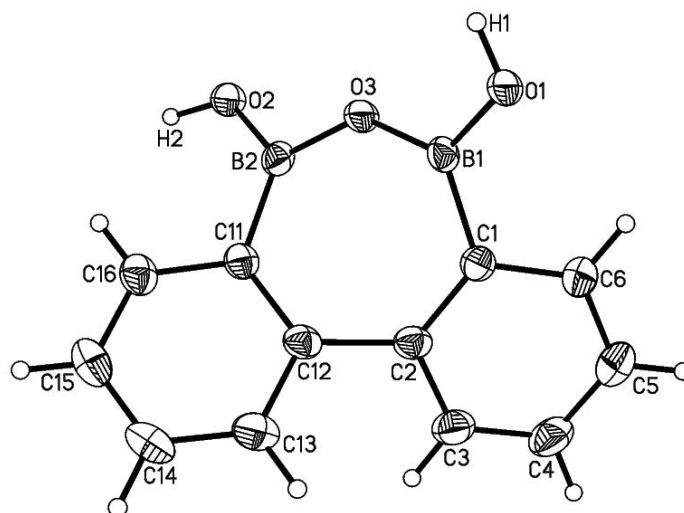


Figure S2. Molecular structure and numbering scheme of compound **5**; displacement ellipsoids are drawn at the 50% probability level. Selected bond lengths [\AA], bond angles [$^\circ$] and dihedral angle [$^\circ$]: $B(1)-O(1) = 1.365(2)$, $B(1)-O(3) = 1.365(2)$, $B(1)-C(1) = 1.565(3)$, $B(2)-O(2) = 1.363(2)$, $B(2)-O(3) = 1.366(2)$, $B(2)-C(11) = 1.563(3)$; $B(1)-O(3)-B(2) = 131.3(2)$, $O(3)-B(1)-C(1) = 122.5(2)$, $O(3)-B(2)-C(11) = 123.2(2)$; $\text{Ar}(C(1))//\text{Ar}(C(11)) = 38.2$.

Crystal data of Li₂[6]. C₂₀H₃₄B₂Li₂O₂, *M* = 341.97 g mol⁻¹, monoclinic, *a* = 11.229(3) Å, *b* = 12.210(2) Å, *c* = 15.985(4) Å, β = 92.907(19)°, *V* = 2188.8(9) Å³, *T* = 173(2) K, space group *C2/c*, *Z* = 4, μ(Mo-Kα) = 0.061 mm⁻¹, 6821 reflections measured, 1930 unique (*R*_{int} = 0.0731) which were used in all calculations. The final *wR*(*F*²) was 0.3036 (all data).

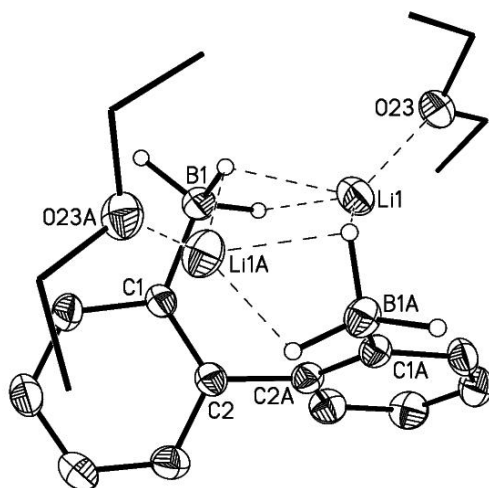


Figure S3. Molecular structure and numbering scheme of compound (Li(Et₂O))₂[6]; displacement ellipsoids are drawn at the 50% probability level; H atoms attached to carbon atoms are omitted for clarity. Selected bond lengths [Å], atom...atom distances [Å] and dihedral angle [°]: Li(1)–O(23) = 1.906(8), B(1)–C(1) = 1.620(6), Li(1)···B(1) = 2.414(10), Li(1)···B(1A) = 2.558(10), Li(1)···Li(1A) = 3.130(17); Ar(C(1))//Ar(C(1A)) = 68.1. Symmetry operations used to generate equivalent atoms: A: $-x+1, y, -z+1/2$.