Supporting Information

Valdecoxib vs Borazavaldecoxib: Isoxazole BN/CC Isosterism as a Case Study in Designing and Stabilizing Boron Heterocycles

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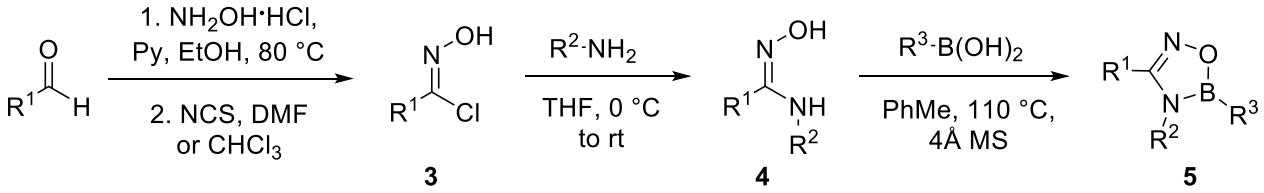
1 Synthetic chemistry

1.1 Experimental details and general information

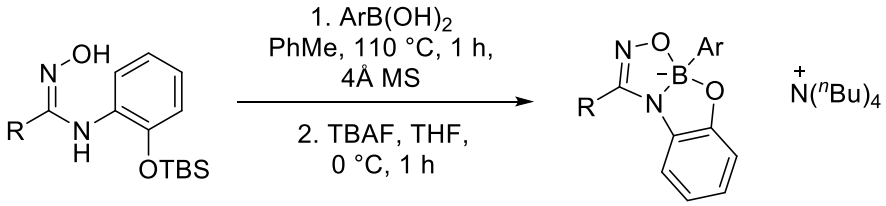
Unless otherwise stated, all reactions were performed under a nitrogen atmosphere using flame-dried glassware. THF, DMF and PhMe were obtained from a MBraun MB SPS* solvent system prior to use. Most of the reagents were purchased from SigmaAldrich and used as received. Arylboronic acids were purchased either from SigmaAldrich or Combi-Blocks. Thin layer chromatography (TLC) was performed on Merck Silica Gel 60 F_{254} plates and was visualized with UV light and $KMnO_4$ stain. NMR spectra were recorded on Varian INOVA-400 or INOVA-500. The residual solvent protons (^1H) of $CDCl_3$ (7.26 ppm), CD_3CN-d_3 (1.94 ppm), DMSO-d_6 (2.50 ppm), and the solvent carbons (^{13}C) of $CDCl_3$ (77.06 ppm), CD_3CN (1.32 and 118.26 ppm), DMSO-d_6 (39.52 ppm) were used as reference. ^1H NMR data is presented as follows: chemical shift in ppm (δ) downfield from tetramethylsilane (multiplicity, coupling constant, integration). The following abbreviations are used in reporting NMR data: s, singlet; br s, broad singlet; d, doublet; t, triplet; q, quartet; quin, quintet; sext, sextet; sept, septet; dd, doublet of doublets; ddd, doublet of doublet of doublets; td, triplet of doublets; dt, doublet of triplets; app q, apparent quartet; m, multiplet. The error of coupling constants from ^1H NMR spectra is estimated to be 0.3 Hz. The quaternary carbon bound to the boron atom is often missing due to the quadrupolar relaxation of boron. This effect was observed in each boron-containing compound. High-resolution mass spectra were recorded by the University of Alberta mass spectrometry services laboratory using either electron impact (EI) or electrospray ionization (ESI) techniques. Melting points (m. p.) were measured on a melting point apparatus and uncorrected. Infrared (IR) spectra were obtained using cast-film or solid technique with frequencies expressed in cm^{-1}.

1.2 Synthetic schemes

General synthetic scheme:

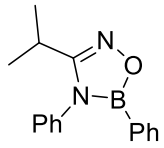


3 **4** **5**



4(m-n) **6**

Oxadiazaboroles and oxadiazaborates set:



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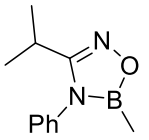

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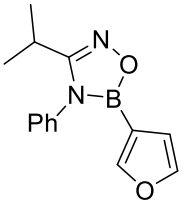

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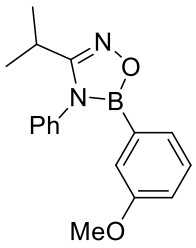

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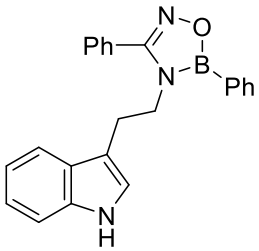

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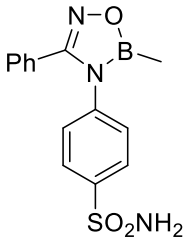

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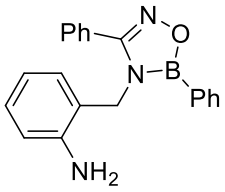

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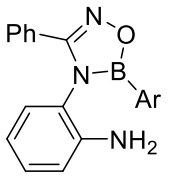
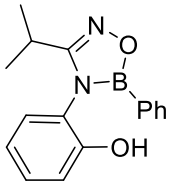


5g Ar = Ph
Me-5g Ar = *o*-tolyl



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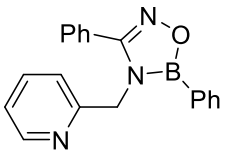

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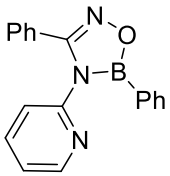

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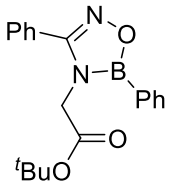

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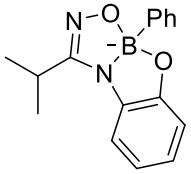

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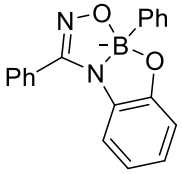

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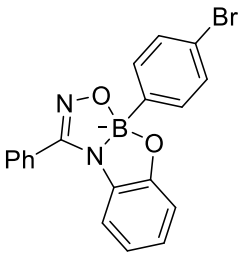

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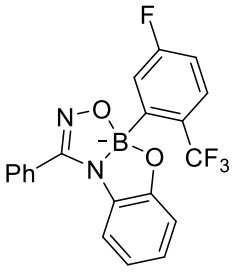

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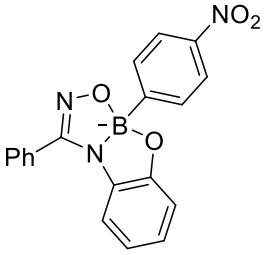
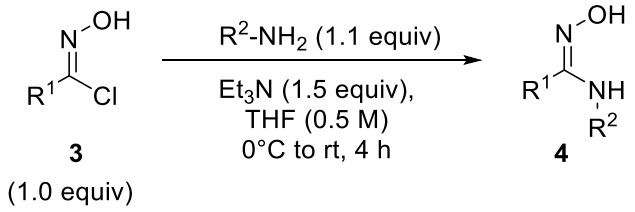


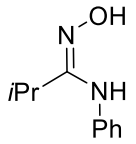
6e

1.3 Chemical synthesis and analytical data

1.3.1 Synthesis of Amidoximes

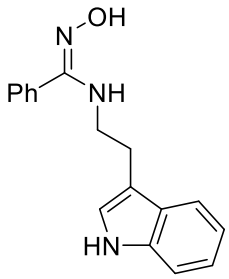


General procedure A: A solution of imidoyl chloride **3** (1.00 equiv) in THF (1.0 M) was added dropwise to a stirred mixture of amine (1.10 equiv) and Et_3N (1.50 equiv) in THF (0.5 M) at 0 °C. The resulting mixture was stirred and allowed to warm to room temperature. After 4 h, saturated $NaHCO_3$ was added and the mixture was extracted twice with EtOAc. The combined organic phases were washed with brine, dried ($MgSO_4$), filtered and concentrated *in vacuo*. The crude residue was purified as indicated below to provide the title amidoxime.

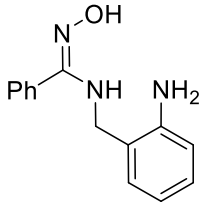


(*Z*)-*N*'-Hydroxy-*N*-phenylisobutyrimidamide (4a): Prepared from (*Z*)-*N*-hydroxyisobutyrimidoyl chloride[1] (**3a**) (1.03 g, 8.44 mmol, 1.00 equiv), aniline (0.85 mL, 9.28 mmol, 1.10 equiv) and Et_3N (1.77 mL, 12.7 mmol, 1.50 equiv) in THF (17.0 mL) following the general procedure **A**. The crude residue was purified by flash column chromatography (6:1 to 2:1 hexane/EtOAc) to yield the desired amidoxime as a white solid (455 mg, 30% yield); $\mathbf{R_f}$ = 0.17 (3:1, hexane/EtOAc); **mp** = 68 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 8.76 (br s, 1H), 7.37 – 7.29 (m, 2H), 7.20 – 7.13 (m, 1H), 7.13 – 7.06 (m, 2H), 7.03 (br s, 1H), 2.87 (sept, J = 6.8 Hz, 1H), 1.11 (d, J = 6.8 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 157.3, 139.1, 129.2, 124.9, 124.9, 27.2, 20.2; **IR** (cast film, cm^{-1}): 3363, 3218, 2972, 1651, 1596, 1498, 1394, 1286, 929, 700; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{10}H_{15}N_2O^+$: 179.1179, found: 179.1190.

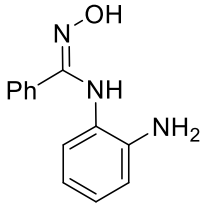
[1] Prepared according to the literature procedure: Dubrovskiy, A. V.; Larock, R. C. *Org. Lett.* **2010**, *12*, 1180-1183.



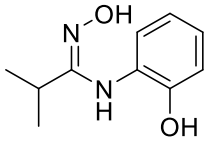
(*Z*)-*N*-(2-(1H-Indol-3-yl)ethyl)-*N′*-hydroxybenzimidamide (4e): Prepared from (*Z*)-*N*-hydroxy benz-imidoyl chloride[1] (**3b**) (0.40 g, 2.57 mmol, 1.00 equiv), tryptamine (0.45 g, 2.83 mmol, 1.10 equiv) and Et_3N (0.54 mL, 3.86 mmol, 1.50 equiv) in THF (5.0 mL) following the general procedure **A**. The crude residue was purified by flash column chromatography (2:1 hexane/EtOAc to pure EtOAc) to yield the desired amidoxime as a white solid (319 mg, 44% yield); $\mathbf{R_f}$ = 0.44 (1:1 hexane/EtOAc); **^{1}H NMR** (400 MHz, $CDCl_3$): δ (ppm) 7.97 (br s, 1H), 7.46 – 7.29 (m, 7H), 7.22 – 7.14 (m, 1H), 7.09 – 6.98 (m, 2H), 5.42 (br s, 1H), 3.35 (app q, *J* = 6.8 Hz, 2H), 2.88 (t, *J* = 7.0 Hz, 2H).



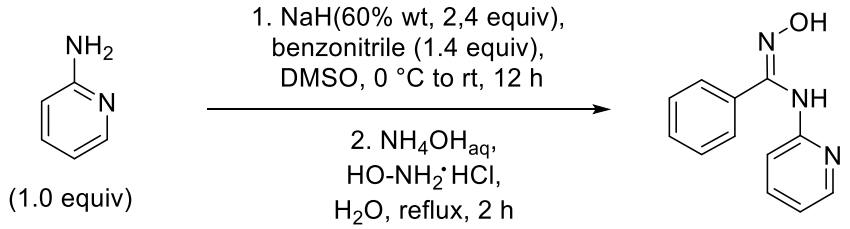
(*Z*)-*N*-(2-Aminobenzyl)-*N′*-hydroxybenzimidamide (4f): Prepared from (*Z*)-*N*-hydroxybenz-imidoyl chloride[1] (**3b**) (0.50 g, 3.21 mmol, 1.00 equiv), 2-(aminomethyl)aniline (0.43 g, 3.50 mmol, 1.10 equiv) and Et_3N (0.67 mL, 4.80 mmol, 1.50 equiv) in THF (6.5 mL) following the general procedure **A**. The crude residue was filtered through a short plug of silica gel (EtOAc 0.1% Et_3N). The volatiles were then evaporated to yield a crude solid, which was washed with Et_2O (trituration) to afford the desired amidoxime as a yellow solid (373 mg, 48% yield); $\mathbf{R_f}$ = 0.60 (EtOAc); **mp** = 112 °C; **^{1}H NMR** (498 MHz, DMSO-d_6): δ (ppm) 9.79 (s, 1H), 7.43 – 7.34 (m, 5H), 6.90 (td, *J* = 7.6, 1.6 Hz, 1H), 6.77 (dd, *J* = 7.5, 1.5 Hz, 1H), 6.56 (dd, *J* = 7.9, 1.2 Hz, 1H), 6.45 (td, *J* = 7.4, 1.2 Hz, 1H), 6.10 (t, *J* = 6.8 Hz, 1H), 4.86 (s, 2H), 3.98 (d, *J* = 6.8 Hz, 2H); **^{13}C NMR** (125 MHz, DMSO-d_6): δ (ppm) 155.0, 145.5, 132.5, 128.9, 128.11, 128.09, 127.3, 127.1, 123.7, 115.8, 114.5, 43.3; **IR** (solid, cm^{-1}): 3384, 3359, 3201, 3146, 2885, 1642, 1620, 1494, 1382, 1333, 1128, 964, 947, 923, 869, 778, 756, 704; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{14}H_{16}N_3O^+$: 242.1288, found: 242.1292.



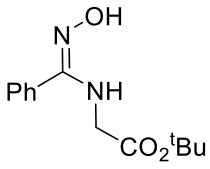
(*Z*)-*N*-(2-Aminophenyl)-*N'*-hydroxybenzimidamide (4g): Prepared from (*Z*)-*N*-hydroxybenzimidoyl chloride[1] (**3b**) (0.50 g, 3.21 mmol, 1.00 equiv), *o*-phenylenediamine (0.38 g, 3.50 mmol, 1.10 equiv) and Et_3N (0.67 mL, 4.80 mmol, 1.50 equiv) in THF (6.5 mL) following the general procedure **A**. The crude residue was filtered through a short plug of silica gel (EtOAc + 0.1% Et_3N). The volatiles were then evaporated to yield a crude brown solid, which was washed with Et_2O (trituration) to afford the desired amidoxime as a white solid (420 mg, 58% yield); $\mathbf{R_f}$ = 0.72 (EtOAc); **mp** = 168 °C; **^{1}H NMR** (498 MHz, DMSO-d_6): δ (ppm) 10.35 (s, 1H), 7.35 – 7.30 (m, 2H), 7.28 – 7.21 (m, 3H), 7.11 (s, 1H), 6.70 – 6.63 (m, 2H), 6.24 – 6.19 (m, 2H), 5.03 (s, 2H); **^{13}C NMR** (125 MHz, DMSO-d_6): δ (ppm) 151.4, 142.1, 132.9, 128.6, 127.9, 127.4, 126.6, 124.7, 124.0, 116.0, 115.0; **IR** (solid, cm^{-1}): 3416, 3330, 3192, 1646, 1612, 1505, 1390, 946, 907, 752, 696; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{13}H_{14}N_3O^+$: 228.1131, found: 228.1132.



(*Z*)-*N'*-Hydroxy-*N*-(2-hydroxyphenyl)isobutyrimidamide (4h): A solution of TBAF (1.0 M in THF, 3.0 mL, 2.0 equiv) was slowly added to a stirring solution of (*Z*)-*N*-(2-((*tert*-butyldimethylsilyl)oxy)phenyl)-*N'*-hydroxy-isobutyrimidamide (**4m**) (472 mg, 1.53 mmol, 1.00 equiv) in THF (7.0 mL, 0.2 M) at 0 °C. After 30 minutes the mixture was quenched with saturated NH_4Cl (20 mL) and extracted with DCM (3 × 25 mL). The combined organic phases were washed with water (25 mL) and brine (25 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The crude product was purified by flash column chromatography (1:1 to 1:2 hexane/EtOAc) to yield the desired amidoxime as yellow solid (200 mg, 69% yield); $\mathbf{R_f}$ = 0.23 (1:1 hexane/EtOAc); **mp** = 102 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.22 (ddd, *J* = 8.1, 7.4, 1.6 Hz, 1H), 7.11 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.02 (dd, *J* = 8.2, 1.4 Hz, 1H), 6.90 (td, *J* = 7.6, 1.4 Hz, 1H), 2.50 (sept, *J* = 6.9 Hz, 1H), 1.07 (d, *J* = 6.9 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 158.9, 153.0, 129.1, 128.8, 124.7, 120.5, 115.8, 27.6, 20.1; **IR** (solid, cm^{-1}): 3334, 3226, 2975, 1665, 1597, 1517, 1463, 1394, 1278, 1023, 912. 827, 744; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{10}H_{15}N_2O_2^+$: 195.1128, found: 195.113.

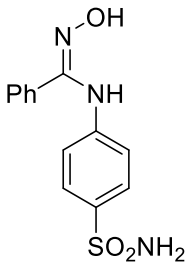


(*Z*)-*N'*-Hydroxy-*N*-(pyridin-2-yl)benzimidamide (4i): NaH (60% wt, 1.00 g, 25.0 mmol, 2.40 equiv) was carefully added to a stirred mixture of 2-aminopyridine (1.00 g, 10.6 mmol, 1.00 equiv) in DMSO (5.0 mL) at 0 °C. After stirring for 30 minutes, benzonitrile (1.50 mL, 14.8 mmol, 1.40 equiv) was slowly added to the mixture. After stirring for 12 hours at room temperature, the reaction mixture was quenched carefully at 0 °C with saturated NH_4Cl (30 mL) and extracted with Et_2O (3 × 40 mL). The combined organic phases were washed with water (30 mL) and brine (30 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The crude product was added to a mixture of hydroxylamine hydrochloride (3.70 g, 53.0 mmol, 5.00 equiv) and aqueous ammonium hydroxide (28% wt, 9.00 mL, 63.6 mmol, 6.00 equiv) in water (10 mL). After refluxing the suspension for 2 hours, the resulting solid was isolated by filtration on a Buchner funnel to furnish the desired amidoxime as pale yellow crystals (1.45 g, 67% yield).[2]

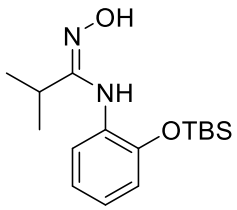


***tert*-Butyl (*Z*)-((hydroxyimino)(phenyl)methyl)glycinate (4k):** Prepared from (*Z*)-*N*-hydroxy benz-imidoyl chloride[1] (**3b**) (0.50 g, 3.21 mmol, 1.00 equiv), glycine *tert*-butyl ester hydrochloride (0.65 g, 3.86 mmol, 1.20 equiv) and Et_3N (1.30 mL, 9.60 mmol, 3.00 equiv) in THF (6.5 mL) following the general procedure **A**. The crude residue was purified by flash column chromatography (3:1 to 2:1 hexane/EtOAc) to yield the desired amidoxime as a yellow solid (460 mg, 57% yield); $\mathbf{R_f}$ = 0.50 (1:1 hexane/EtOAc); **mp** = 92 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.48 – 7.44 (m, 2H), 7.43 – 7.36 (m, 3H), 7.02 (br s, 1H), 5.75 (t, *J* = 5.5 Hz, 1H), 3.69 (d, *J* = 5.9 Hz, 2H), 1.42 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 169.9, 156.2, 131.3, 129.9, 128.7, 128.6, 82.2, 46.3, 28.1; **IR** (cast film, cm^{-1}): 3419, 3238, 2981, 1742, 1650, 1484, 1396, 1371, 1250, 1229, 1155, 911, 858, 776, 770, 705; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{13}H_{19}N_2O_3^+$: 251.1390, found: 251.1391.

[2] Gaber, A. -A.; Taib, L. *J. Chem. Sci.* **2016**, *128*, 745-752.



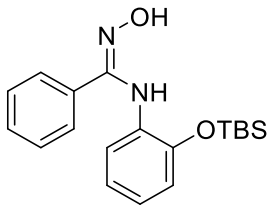
(*Z*)-*N'*-Hydroxy-*N*-(4-sulfamoylphenyl)benzimidamide (4l): Prepared from (*Z*)-*N*-hydroxy benzimidoyl chloride[1] (**3b**) (2.00 g, 12.9 mmol, 1.00 equiv), sulfanilamide (2.40 g, 14.1 mmol, 1.10 equiv) and Et_3N (2.67 mL, 19.3 mmol, 1.50 equiv) in THF (28.0 mL) following the general procedure **A**. The crude residue was purified by flash column chromatography (2:1 to 1:2 hexane/EtOAc) to yield the desired amidoxime as a yellow solid (0.70 g, 19% yield); $\mathbf{R}_f$ = 0.4 (1:2 hexane/EtOAc); **mp** = 180 °C; **^{1}H NMR** (400 MHz, DMSO-d_6): δ (ppm) 10.85 (s, 1H), 8.78 (s, 1H), 7.52 – 7.46 (m, 2H), 7.44 – 7.34 (m, 5H), 7.07 (s, 2H), 6.74 – 6.67 (m, 2H); **^{13}C NMR** (126 MHz, DMSO-d_6): δ (ppm) 147.9, 144.8, 135.0, 132.5, 129.3, 128.5, 127.4, 126.3, 117.8; **IR** (solid, cm^{-1}): 3318, 1625, 1594, 1499, 1382, 1353, 1310, 1149 1097, 931, 894., 836, 768, 695; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{13}H_{14}N_3O_3S^+$: 292.0750, found: 292.0754.



(*Z*)-*N*-(2-((*tert*-Butyldimethylsilyl)oxy)phenyl)-*N*'-hydroxyisobutyrimidamide (4m): Prepared from (*Z*)-*N*-hydroxyisobutyrimidoyl chloride[1] (**3a**) (30.0 mg, 0.25 mmol, 1.00 equiv), 2-((*tert*-butyldimethylsilyl)oxy)aniline[3] (0.11 g, 0.50 mmol, 2.00 equiv) and Et_3N (52.0 μL, 0.38 mmol, 1.50 equiv) in THF (0.5 mL) following the general procedure **A**. The crude residue was purified by flash column chromatography (15:1 to 5:1 hexane/EtOAc) to yield the desired amidoxime as a yellow oil (35 mg, 46% yield); $\mathbf{R}_f$ = 0.29 (5:1 hexane/EtOAc); **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 8.21 – 7.86 (m, 1H), 7.11 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.08 (br s, 1H), 6.99 (ddd, *J* = 8.0, 7.5, 1.7 Hz, 1H), 6.91 (td, *J* = 7.6, 1.5 Hz, 1H), 6.86 (dd, *J* = 8.0, 1.5 Hz, 1H), 2.85 (sept, *J* = 6.8 Hz, 1H), 1.11 (d, *J* = 6.8 Hz, 6H), 1.01 (s, 9H), 0.23 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 157.2, 148.8, 130.9, 124.8, 124.6, 121.5, 119.6, 27.6, 25.8, 20.4, 18.3, -4.2; **IR** (neat, cm^{-1}): 3259,

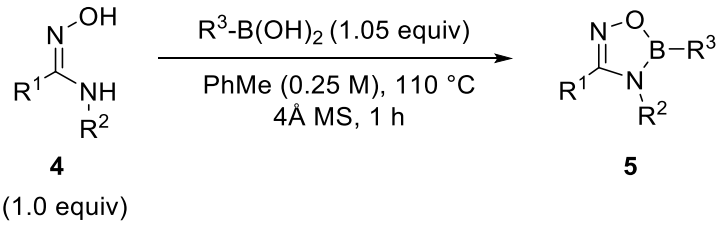
[3] Zhang, J. -W.; Cai, Q.; Gu, Q.; Shi, X. -X; You, S. -L. *Chem. Comm.* **2013**, *49*, 7750-7752.

2962, 2931, 2860, 1643, 1597, 1502, 1471, 1457, 1394, 1363, 1258, 953, 913, 881, 840, 783, 753; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{16}H_{29}N_2O_2Si^+$: 309,1993, found: 309.1992.

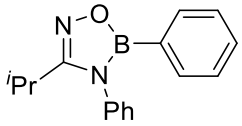


(*Z*)-*N*-(2-((*tert*-Butyldimethylsilyl)oxy)phenyl)-*N*'-hydroxybenzimidamide (4n): Prepared from (*Z*)-*N*-hydroxybenzimidoyl chloride[1] (**3b**) (0.63 g, 4.07 mmol, 1.00 equiv), 2-((*tert*-butyldimethyl silyl)oxy)aniline[3] (1.10 g, 4.48 mmol, 1.10 equiv) and Et_3N (1.08 mL, 7.72 mmol, 1.90 equiv) in THF (13.0 mL) following the general procedure **A**. The crude residue was purified by flash column chromatography (15:1 to 2:1 hexane/EtOAc) to yield the desired amidoxime (not completely pure: about 85 – 90% purity) as a yellow oil (648 mg, 46% yield); $\mathbf{R_f}$ = 0.2 (5:1 hexane/EtOAc); **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.53 (br s, 1H), 7.47 – 7.43 (m, 2H), 7.43 – 7.38 (m, 1H), 7.38 – 7.32 (m, 2H), 7.03 (br s, 1H), 6.81 (dd, *J* = 8.0, 1.5 Hz, 1H), 6.74 (ddd, *J* = 8.0, 7.4, 1.6 Hz, 1H), 6.54 (ddd, *J* = 8.0, 7.4, 1.5 Hz, 1H), 6.20 (dd, *J* = 8.1, 1.6 Hz, 1H), 1.07 (s, 9H), 0.31 (s, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 151.7, 145.2, 131.7, 131.4, 129.8, 128.6, 128.5, 121.8, 121.0, 120.1, 118.8, 25.9, 18.4, -4.0; **IR** (cast film, cm^{-1}): 3192, 2956, 2930, 2858, 1624, 1598, 1578, 1507, 1472, 1461, 1392, 1265, 1206, 1126, 1098, 954, 892, 839, 783, 758, 697, 677; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{19}H_{27}N_2O_2Si^+$: 343.1836, found: 343.1837.

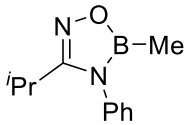
1.3.2 Oxadiazaboroles synthesis



General procedure B: A stirred mixture of amidoxime **4** (1.00 equiv), boronic acid (1.05 equiv) and molecular sieves 4Å (100 mg – 500 mg) in PhMe (0.25 M) was heated under reflux at 110 °C in a sealed vial for 1 h. The reaction mixture was allowed to cool down to room temperature and filtered through a short plug of silica gel (EtOAc). The volatiles were evaporated *in vacuo* to provide the title oxadiazaborole as a solid. When necessary the resulting solid was further purified by washing with Et_2O (trituration).

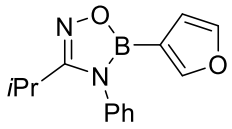


3-Isopropyl-4,5-diphenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (5a): Prepared from (*Z*)-*N*′-hydroxy-*N*-phenylisobutyrimidamide (**4a**) (50.0 mg, 0.28 mmol, 1.00 equiv) and phenylboronic acid (36.0 mg, 0.29 mmol, 1.05 equiv) in PhMe (1.1 mL) following the general procedure **B**. Isolated as a white solid (54 mg, 73% yield); **mp** = 81 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.52 – 7.43 (m, 4H), 7.40 – 7.34 (m, 1H), 7.29 – 7.21 (m, 5H), 2.73 (sept, *J* = 6.9 Hz, 1H), 1.22 (d, *J* = 6.9 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) δ 165.9, 137.3, 134.2, 130.9, 129.9, 128.4, 128.3, 128.0, 25.4, 20.7; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 31.9; **IR** (cast film, cm^{-1}): 2974, 1576, 1499, 1438, 1418, 1387, 1329, 1256, 1111, 772, 699; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{16}H_{18}BN_2O^+$: 265.1507, found: 265.1509.

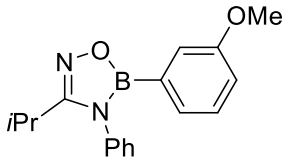


3-Isopropyl-4,5-diphenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (5b): Prepared from (*Z*)-*N*′-hydroxy-*N*-phenylisobutyrimidamide (**4a**) (50.0 mg, 0.28 mmol, 1.00 equiv) and methylboronic acid (18.0 mg, 0.29 mmol, 1.05 equiv) in PhMe (1.1 mL) following the general procedure **B**. Isolated as a white solid (47.0 mg, 83% yield); **mp** = 60 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm)

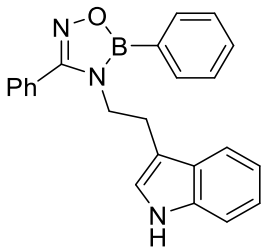
7.48 – 7.41 (m, 2H), 7.40 – 7.35 (m, 1H), 7.16 – 7.09 (m, 2H), 2.77 (sept, *J* = 6.9 Hz, 1H), 1.16 (d, *J* = 6.9 Hz, 6H), 0.52 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 164.9, 137.3, 129.7, 127.7, 127.2, 25.4, 20.4; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 35.0; **IR** (solid, cm^{-1}): 2979, 2966, 1568, 1497, 1387, 1380, 1363, 1331, 1273, 1200, 903, 769, 707; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{11}H_{16}BN_2O^+$: 203.1350, found: 203.1354.



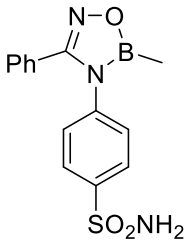
5-(Furan-3-yl)-3-isopropyl-4-phenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (5c): Prepared from (*Z*)-*N′*-hydroxy-*N*-phenylisobutyrimidamide (**4a**) (50.0 mg, 0.28 mmol, 1.00 equiv) and furan-3-ylboronic acid (33.0 mg, 0.29 mmol, 1.05 equiv) in PhMe (1.1 mL) following the general procedure **B**. Isolated as a white solid (63.0 mg, 89% yield); **mp** = 73 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.52 – 7.42 (m, 3H), 7.39 – 7.36 (m, 1H), 7.32 – 7.30 (m, 1H), 7.26 – 7.23 (m, 2H), 6.33 – 6.27 (m, 1H), 2.75 (sept, J = 6.9 Hz, 1H), 1.21 (d, J = 6.9 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 165.3, 149.9, 143.0, 137.0, 129.9, 128.4, 128.0, 112.7, 25.4, 20.6; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 31.0; **IR** (solid, cm^{-1}): 2967, 1571, 1503, 1404, 1391, 1326, 1256, 1168, 1151, 1070, 1020, 892, 878, 813, 777, 703, 670; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{14}H_{16}BN_2O_2^+$: 255.1299, found: 255.1299.



3-Isopropyl-5-(3-methoxyphenyl)-4-phenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (5d): Prepared from (*Z*)-*N′*-hydroxy-*N*-phenylisobutyrimidamide (**4a**) (50.0 mg, 0.28 mmol, 1.00 equiv) and (3-methoxyphenyl)boronic acid (45.0 mg, 0.29 mmol, 1.05 equiv) in PhMe (1.1 mL) following the general procedure **B**. Isolated as a white solid (79.0 mg, 95% yield); **mp** = 91 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) δ 7.51 – 7.44 (m, 1H), 7.43 – 7.29 (m, 4H), 7.20 – 7.12 (m, 2H), 6.93 – 6.84 (m, 1H), 6.72 (d, *J* = 8.3 Hz, 1H), 3.36 (s, 3H), 2.83 (sept, *J* = 6.9 Hz, 1H), 1.21 (d, *J* = 6.9 Hz, 6H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 165.5, 162.8, 138.2, 136.0, 132.1, 129.2, 127.5, 127.4, 120.5, 110.1, 54.6, 25.5, 20.6; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 32.5; **IR** (solid, cm^{-1}): 2981, 1600, 1574, 1489, 1432, 1378, 1247, 1027, 773, 761, 699; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{17}H_{20}BN_2O_2^+$: 295.1612, found: 295.1617.

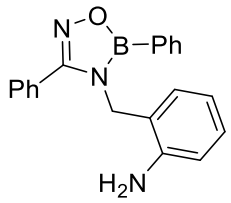


4-(2-(1H-Indol-3-yl)ethyl)-3,5-diphenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (5e): Prepared from (*Z*)-*N*-(2-(1H-indol-3-yl)ethyl)-*N′*-hydroxybenzimidamide (**4e**) (319 mg, 1.14 mmol, 1.00 equiv) and phenylboronic acid (146 mg, 1.12 mmol, 1.05 equiv) in PhMe (4.6 mL) following the general procedure **B**. Isolated as a grey solid (258 mg, 62% yield); **mp** = 190 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.90 (br s, 1H), 7.82 – 7.78 (m, 2H), 7.54 – 7.39 (m, 8H), 7.28 (dt, *J* = 8.2, 0.9 Hz, 1H), 7.14 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.03 – 7.00 (m, 1H), 6.97 (ddd, *J* = 7.9, 6.8, 1.0 Hz, 1H), 6.75 (d, *J* = 2.4 Hz, 1H), 4.05 – 3.93 (m, 2H), 2.93 – 2.84 (m, 2H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 162.6, 136.3, 133.7, 130.8, 130.4, 129.3, 128.9, 128.3, 127.2, 126.7, 122.3, 122.1, 119.6, 118.3, 111.9, 111.3, 43.8, 27.9; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 33.0; **IR** (cast film, cm^{-1}): 3219, 1560, 1401, 1300, 1234, 1112, 756, 723, 703; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{23}H_{21}BN_3O^+$: 366.1772, found: 366.1773.

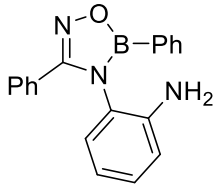


4-(5-Methyl-3-phenyl-1,2,4,5-oxadiazaborol-4(5H)-yl)benzenesulfonamide (borazavaldecoxib) (2): A stirred mixture of (*Z*)-*N′*-hydroxy-*N*-(4-sulfamoylphenyl)benzimidamide (**4l**) (300 mg, 1.03 mmol, 1.00 equiv), methylboronic acid (62.0 mg, 1.04 mmol, 1.01 equiv) and molecular sieves 4Å (300 mg) in PhMe (4.0 mL) was heated under reflux at 110 °C in a sealed vial for 1 h. The reaction mixture was allowed to cool down to room temperature. The volatiles were evaporated *in vacuo* to provide the title oxadiazaborole as a white solid (320 mg, 99% yield);); **mp** = 112 °C; **^{1}H NMR** (498 MHz, $CDCl_3$): δ (ppm) 7.88 (d, *J* = 8.6 Hz, 2H), 7.44 – 7.40 (m, 1H), 7.35 – 7.31 (m, 2H), 7.30 – 7.27 (m, 2H), 7.11 (d, *J* = 8.7 Hz, 2H), 4.80 (s, 2H), 0.74 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 159.3, 141.6, 140.4, 130.7, 129.0, 128.9, 128.0, 126.8, 125.6; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 35.6; **IR** (solid, cm^{-1}): 3321, 3251, 1597, 1587, 1547, 1502, 1398, 1376,

1334, 1286, 1267, 1188, 1176, 1098, 900, 889, 845, 773, 751, 700; **HRMS (EI)**: $(M)^+$ calcd for $C_{14}H_{14}BN_3O_3S^+$: 315.0849, found: 315.0830.

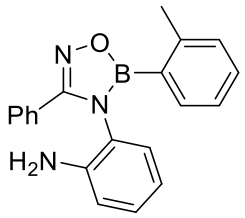


2-((3,5-Diphenyl-1,2,4,5-oxadiazaborol-4(5H)-yl)methyl)aniline (5f): Prepared from (*Z*)-*N*-(2-aminobenzyl)-*N*′-hydroxybenzimidamide (**4f**) (50.0 mg, 0.22 mmol, 1.00 equiv) and phenylboronic acid (28.0 mg, 0.23 mmol, 1.05 equiv) in PhMe (0.9 mL) following the general procedure **B**. Isolated as a white solid (56.0 mg, 78% yield); **mp** = 168 °C; **^{1}H NMR** (498 MHz, $CDCl_3$): δ (ppm) 7.75 – 7.69 (m, 2H), 7.49 – 7.42 (m, 4H), 7.41 – 7.34 (m, 4H), 7.16 – 7.11 (m, 1H), 6.99 (d, *J* = 7.6 Hz, 1H), 6.81 – 6.75 (m, 1H), 6.72 (dd, *J* = 7.9, 1.2 Hz, 1H), 4.71 (s, 2H), 3.43 (br s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ (ppm) 162.9, 142.9, 134.0, 131.1, 130.7, 129.1, 128.9, 128.51, 128.46, 126.3, 126.1, 122.7, 119.6, 116.3, 43.3; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 33.6; **IR** (cast film, cm^{-1}): 3361, 1639, 1494, 1461, 1397, 1291, 980, 772, 756, 700; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{20}H_{19}BN_3O^+$: 328.1616, found: 328.1625.

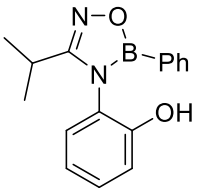


2-(3,5-Diphenyl-1,2,4,5-oxadiazaborol-4(5H)-yl)aniline (5g): Prepared from (*Z*)-*N*-(2-aminophenyl)-*N*′-hydroxybenzimidamide (**4g**) (50.0 mg, 0.22 mmol, 1.00 equiv) and phenylboronic acid (28.0 mg, 0.23 mmol, 1.05 equiv) in PhMe (0.9 mL) following the general procedure **B**. Isolated as a white solid (57.0 mg, 83% yield); **mp** = 177 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ (ppm) 7.62 (dd, *J* = 8.0, 1.4 Hz, 2H), 7.51 – 7.46 (m, 2H), 7.45 – 7.40 (m, 1H), 7.40 – 7.35 (m, 1H), 7.34 – 7.26 (m, 4H), 7.19 (ddd, *J* = 8.1, 7.3, 1.5 Hz, 1H), 6.96 (dd, *J* = 7.8, 1.5 Hz, 1H), 6.81 (dd, *J* = 8.0, 1.3 Hz, 1H), 6.72 (td, *J* = 7.6, 1.4 Hz, 1H), 3.76 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ (ppm) 161.4, 143.0, 134.1, 131.4, 130.5, 129.7, 129.4, 128.6, 128.6, 128.3, 126.0, 123.1, 119.2, 116.4; **^{11}B NMR** (128 MHz, $CDCl_3$): δ (ppm) 32.4; **IR** (cast film, cm^{-1}): 3440, 3368, 3344, 3056, 1625, 1602, 1502,

1442, 1394, 1372, 1312, 1263, 1129, 1072, 1029, 969, 862, 757, 693; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{19}H_{17}BN_3O^+$: 314.1459, found: 314.1469.

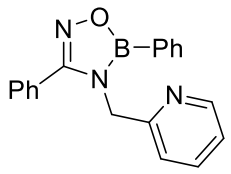


2-(3-Phenyl-5-(o-tolyl)-1,2,4,5-oxadiazaborol-4(5H)-yl)aniline (Me-5g): Prepared from (*Z*)-*N*-(2-amino- phenyl)-*N*′-hydroxybenzimidamide (**4g**) (50.0 mg, 0.22 mmol, 1.00 equiv) and phenylboronic acid (28.0 mg, 0.23 mmol, 1.05 equiv) in PhMe (0.9 mL) following the general procedure **B**. Isolated as a white solid (65.0 mg, 68% yield); **mp** = 140 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.48 – 7.44 (m, Hz, 2H), 7.42 – 7.35 (m, 1H), 7.33 – 7.27 (m, 3H), 7.23 – 7.20 (m, 2H), 7.16 – 7.10 (m, 1H), 7.00 (t, J = 7.4 Hz, 1H), 6.91 (dd, J = 7.9, 1.4 Hz, 1H), 6.73 (dd, J = 8.1, 1.3 Hz, 1H), 6.68 (td, J = 7.6, 1.3 Hz, 1H), 3.66 (br s, 2H), 2.61 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 160.7, 144.2, 142.6, 134.1, 130.6, 130.3, 130.1, 129.3, 128.5, 128.4, 126.0, 124.9, 123.2, 119.1, 116.3, 22.9; **^{11}B NMR** (128 MHz, $CDCl_3$): δ (ppm) 32.8; **IR** (cast film, cm^{-1}): 3381, 1621, 1504, 1363, 1316, 1130, 871, 754, 727, 695; **HRMS (EI)**: $(M)^+$ calcd for $C_{20}H_{18}BN_3O^+$: 327.15429, found: 327.15457.

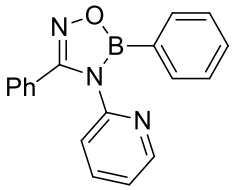


2-(3-Isopropyl-5-phenyl-1,2,4,5-oxadiazaborol-4(5H)-yl)phenol (5h) Prepared from (*Z*)-*N*'-hydroxy-*N*-(2-hydroxyphenyl)isobutyrimidamide (**4h**) (50.0 mg, 0.26 mmol, 1.00 equiv) and phenylboronic acid (35.0 mg, 0.29 mmol, 1.10 equiv) in PhMe (1.0 mL) following the general procedure **B**. Isolated as a white solid (49.0 mg, 67% yield); **mp** = 165 °C; **^{1}H NMR** (498 MHz, $CDCl_3$): δ (ppm) 7.52 – 7.46 (m, 2H), 7.42 – 7.34 (m, 2H), 7.28 – 7.23 (m, 2H), 7.17 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.12 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.03 (td, *J* = 7.6, 1.4 Hz, 1H), 5.54 (s, 1H), 2.65 (sept, *J* = 6.9 Hz, 1H), 1.22 (dd, *J* = 6.9, 5.4 Hz, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ (ppm) 166.4, 151.9, 134.0, 131.3, 130.3, 129.5, 128.2, 123.8, 121.6, 117.2, 25.6, 20.7, 20.3; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 32.2; **IR** (solid, cm^{-1}): 3116, 2975, 1603, 1563, 1520, 1406, 1390, 1329, 1296,

1264, 1112, 900, 756, 686; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{16}H_{18}BN_2O_2^+$: 281,1456, found: 281.1460, $(M-H)^-$ calcd for $C_{16}H_{16}BN_2O_2^-$: 279,1310, found: 279,1310.

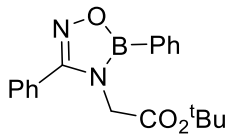


3,5-Diphenyl-4-(pyridin-2-ylmethyl)-4,5-dihydro-1,2,4,5-oxadiazaborole (5i): Prepared from (*Z*)-*N′*-hydroxy-*N*-(pyridin-2-ylmethyl)benzimidamide[4] (**4i**) (50.0 mg, 0.22 mmol, 1.00 equiv) and phenylboronic acid (28.0 mg, 0.23 mmol, 1.05 equiv) in PhMe (0.9 mL) following the general procedure **B**. Isolated as a white solid (54.0 mg, 78% yield); **mp** = 123 °C; **^{1}H NMR** (498 MHz, $CDCl_3$): δ (ppm) 8.57 (ddd, *J* = 4.9, 1.8, 0.9 Hz, 1H), 7.78 – 7.74 (m, 2H), 7.63 (td, *J* = 7.7, 1.8 Hz, 1H), 7.48 – 7.41 (m, 4H), 7.40 – 7.34 (m, 4H), 7.21 – 7.17 (m, 1H), 7.09 (dt, *J* = 7.8, 1.0 Hz, 1H), 5.02 (s, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ (ppm) 162.8, 157.9, 149.9, 137.2, 134.0, 131.0, 130.7, 129.3, 128.9, 128.4, 126.2, 122.6, 120.4, 48.8; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 33.3; **IR** (cast film, cm^{-1}): 1600, 1434, 1399, 1363, 1253, 977, 758, 700; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{19}H_{17}BN_3O^+$: 314.1459, found: 314.1465.

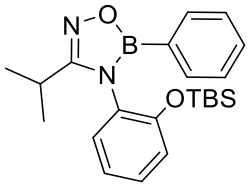


3,5-Diphenyl-4-(pyridin-2-yl)-4,5-dihydro-1,2,4,5-oxadiazaborole (5j): Prepared from (*Z*)-*N′*-hydroxy-*N*-(pyridin-2-yl)benzimidamide (**4j**) (500 mg, 2.34 mmol, 1.00 equiv) and phenylboronic acid (300 mg, 2.46 mmol, 1.05 equiv) in PhMe (10.0 mL) following the general procedure **B**. Isolated as a white solid (480 mg, 69% yield); **mp** = 178 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ (ppm) δ 8.60 – 8.51 (m, 1H), 7.68 (td, *J* = 7.7, 2.0 Hz, 1H), 7.65 – 7.60 (m, 2H), 7.45 – 7.27 (m, 9H), 7.03 (d, *J* = 7.9, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ (ppm) 160.4, 151.1, 149.7, 138.6, 134.4, 131.2, 130.3, 129.0, 128.6, 128.1, 126.3, 123.2, 122.4; **^{11}B NMR** (128 MHz, $CDCl_3$): δ (ppm) 33.0; **IR** (cast film, cm^{-1}): 3057, 3009, 1595, 1472, 1443, 1387, 1324, 1250, 1199, 972, 921, 765, 700; **HRMS (EI)**: $(M)^+$ calcd for $C_{18}H_{14}BN_3O^+$: 299.12299, found: 299.12108.

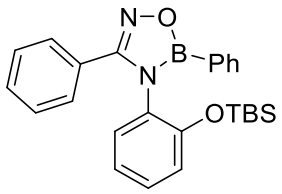
[4] Prepared according to the literature procedure: Zhang, F. –L.; Wang, Y. –F.; Chiba, S. *Org. Biomol. Chem.* **2013**, *11*, 6003-6007.



1-(3,5-Diphenyl-1,2,4,5-oxadiazaborol-4(5H)-yl)-3,3-dimethylbutan-2-one (5k): Prepared from *tert*-butyl (*Z*)-((hydroxyimino)(phenyl)methyl)glycinate (**4k**) (50.0 mg, 0.20 mmol, 1.00 equiv) and phenylboronic acid (26.0 mg, 0.21 mmol, 1.05 equiv) in PhMe (0.9 mL) following the general procedure **B**. Isolated as a yellow solid (57.0 mg, 85% yield); **mp** = 95 °C; **^{1}H NMR** (400 MHz, $CDCl_3$): δ (ppm) 7.81 – 7.76 (m, 2H), 7.58 – 7.42 (m, 8H), 4.24 (s, 2H), 1.39 (s, 9H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 168.4, 162.2, 133.8, 131.0, 130.8, 129.4, 129.0, 128.4, 126.1, 82.9, 46.1, 28.0; **^{11}B NMR** (128 MHz, $CDCl_3$): δ (ppm) 33.3; **IR** (solid, cm^{-1}): 3002, 2935, 1741, 1603, 1554, 1499, 1422, 1404, 1394, 1373, 1298, 1236, 1155, 1122, 974, 848, 746, 701; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{19}H_{22}BN_2O_3^+$: 337.1718, found: 337.1717.



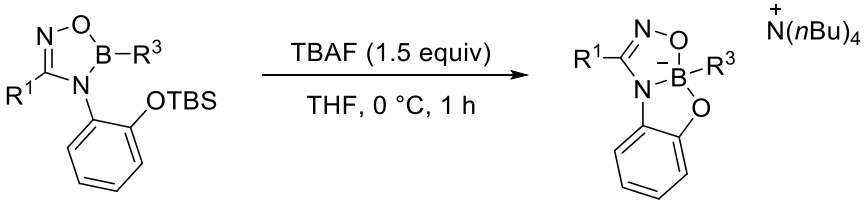
4-(2-((*tert*-Butyldimethylsilyl)oxy)phenyl)-3-isopropyl-5-phenyl-4,5-dihydro-1,2,4,5-oxadiaza borole (5m): Prepared from (*Z*)-*N*-(2-((*tert*-butyldimethylsilyl)oxy)phenyl)-*N*'-hydroxyiso-butyrimidamide (**4m**) (35.0 mg, 0.11 mmol, 1.00 equiv) and phenylboronic acid (14.0 mg, 0.12 mmol, 1.05 equiv) in PhMe (0.4 mL) following the general procedure **B**. Isolated as a yellow oil (34.0 mg, 76% yield); $\mathbf{R_f}$ = 0.50 (hexane/EtOAc 5:1); **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.47 (dd, *J* = 7.9, 1.4 Hz, 2H), 7.38 – 7.31 (m, 2H), 7.27 – 7.21 (m, 3H), 7.05 (td, *J* = 7.6, 1.3 Hz, 1H), 6.99 (dd, *J* = 8.2, 1.3 Hz, 1H), 2.67 (sept, *J* = 6.9 Hz, 1H), 1.27 (d, *J* = 6.9 Hz, 3H), 1.15 (d, *J* = 6.9 Hz, 3H), 0.77 (s, 9H), 0.17 (s, 3H), -0.11 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 166.2, 151.8, 134.0, 130.8, 130.2, 129.6, 128.4, 127.9, 121.8, 120.0, 25.6, 25.4, 21.2, 19.8, 18.0, -3.9, -4.8; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 32.3; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{22}H_{32}BN_2O_2Si^+$: 395.4045, found: 395.2327.



4-(2-((*tert*-Butyldimethylsilyl)oxy)phenyl)-3,5-diphenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (5n): Prepared from (*Z*)-*N*-(2-((*tert*-butyldimethylsilyl)oxy)phenyl)-*N*'-hydroxybenzimidamide (**4n**) (648 mg, 1.89 mmol, 1.00 equiv) and phenylboronic acid (254 mg, 2.08 mmol, 1.10 equiv) in PhMe (4.0 mL) following the general procedure **B**. Isolated as a yellow oil (805 mg, 99% yield); **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.56 (dd, *J* = 8.1, 1.5 Hz, 2H), 7.43 – 7.37 (m, 3H), 7.37 – 7.32 (m, 1H), 7.30 – 7.22 (m, 6H), 6.98 (td, *J* = 7.6, 1.3 Hz, 1H), 6.90 – 6.84 (m, 1H), 0.75 (s, 9H), 0.06 (s, 3H), -0.07 (s, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 161.1, 151.8, 134.1, 131.0, 130.1, 130.0, 129.5, 128.8, 128.6, 128.3, 128.0, 126.7, 121.7, 119.7, 25.5, 18.0, -4.1, -4.5; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 32.8; **IR** (cast film, cm^{-1}): 3057, 2954, 2930, 2858, 1601, 1506, 1456, 1371, 1288, 1254, 1115 1029, 914, 840, 823, 783, 697; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{25}H_{30}BN_2O_2Si^+$: 429.2164, found: 429.2169.

1.3.3 Synthesis of oxadiazaborate salts

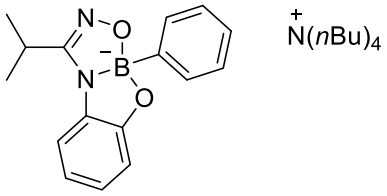
Stepwise protocol:



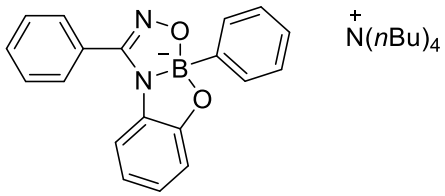
5
(1.0 equiv)

6

General procedure C: a solution of TBAF (1.00 M in THF, 1.50 equiv) was slowly added to a stirred mixture of oxadiazaborole **5** (1.00 equiv) in THF (0.2 M) at 0 °C. After stirring for one hour at that temperature, the reaction mixture was quenched with a 2.0 M solution of K_2CO_3 (20 mL) and extracted with DCM (3 × 25 mL). The combined organic phases were washed with saturated $NaHCO_3$ (30 mL), water (25 mL) and brine (25 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The crude product was washed (triturated) with Et_2O three times to provide the title borate as a solid.

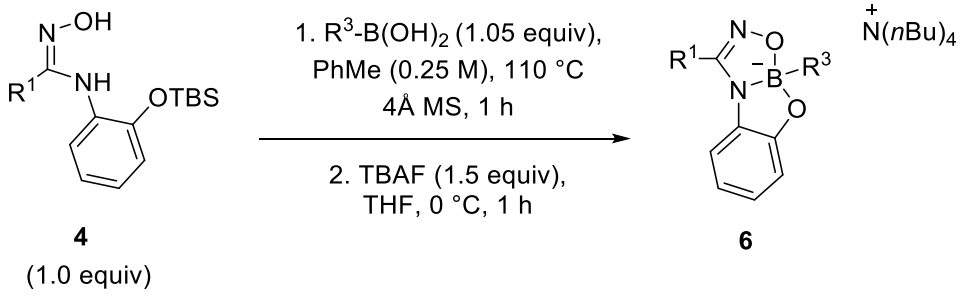


Tetrabutylammonium 3-isopropyl-10-phenyl-10H-benzo[4,5][1,3,2]oxazaborolo[3,2-d][1,2,4,5] oxadiazaborol-10-uide (6a): Prepared from 4-(2-((*tert*-butyldimethylsilyl)oxy)phenyl)-3-isopropyl-5-phenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (**5m**) (428 mg, 1.39 mmol, 1.00 equiv) and TBAF (1.00 M in THF, 2.09 mL, 2.09 mmol, 1.50 equiv) in THF (7.0 mL) following the general procedure **C**. Isolated as a gray solid (427 mg, 59% yield); **mp** = 162 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.58 – 7.52 (m, 2H), 7.14 – 7.08 (m, 2H), 7.06 – 7.00 (m, 1H), 6.99 – 6.92 (m, 1H), 6.84 – 6.76 (m, 1H), 6.70 – 6.64 (m, 1H), 6.56 – 6.48 (m, 1H), 3.02 – 2.94 (m, 8H), 2.67 (sept, *J* = 6.5 Hz, 1H), 1.44 – 1.35 (m, 11H), 1.27 (sext, *J* = 7.3 Hz, 8H), 1.16 (d, *J* = 6.8 Hz, 3H), 0.90 (t, *J* = 7.2 Hz, 12H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 165.1, 157.4, 139.1, 132.7, 126.8, 125.6, 123.0, 118.4, 116.0, 110.0, 58.6, 27.5, 24.1, 22.4, 19.7, 19.3, 13.8; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 14.8; **IR** (solid, cm^{-1}): 2962, 2873, 1575, 1474, 1431, 1383, 1356, 1256, 1188, 1152, 1020, 1000, 907, 877, 855, 748, 737, 707, 659; **HRMS (ESI-TOF)**: $(M^*)^-$ calcd for $C_{16}H_{16}BN_2O_2^-$: 279.1310, found: 279.1309.



Tetrabutylammonium 3,10-diphenyl-10H-benzo[4,5][1,3,2]oxazaborolo[3,2-d][1,2,4,5] oxa diaza- borol-10-uide (6b): Prepared from 4-(2-((*tert*-butyldimethylsilyl)oxy)phenyl)-3,5-diphenyl-4,5-dihydro-1,2,4,5-oxadiazaborole (**5n**) (805 mg, 1.88 mmol, 1.00 equiv) and TBAF (1.00 M in THF, 2.82 mL, 2.82 mmol, 1.50 equiv) in THF (9.4 mL) following the general procedure **C**. Isolated as a white solid (481 mg, 46% yield); **mp** = 145 °C; **^{1}H NMR** (500 MHz, CD_3CN): δ (ppm) 7.77 (d, *J* = 6.9 Hz, 2H), 7.50 – 7.35 (m, 5H), 7.16 (t, *J* = 7.4 Hz, 2H), 7.11 – 7.04 (m, 1H), 6.68 (t, *J* = 7.3 Hz, 2H), 6.62 (dd, *J* = 8.0, 1.4 Hz, 1H), 6.29 (td, *J* = 7.5, 1.4 Hz, 1H), 3.10 – 3.01 (m, 8H), 1.62 – 1.52 (m, 8H), 1.33 (sext, *J* = 7.4 Hz, 8H), 0.95 (t, *J* = 7.3 Hz, 12H); **^{13}C NMR** (126 MHz, CD_3CN): δ (ppm) 161.8, 158.1, 133.8, 132.8, 129.5, 129.2, 129.0, 127.5, 126.3, 123.5, 116.7, 110.8, 59.3, 24.3, 20.3, 13.8; **^{11}B NMR** (160 MHz, CD_3CN): δ (ppm) 14.9; **IR** (cast film, cm^{-1}): 3000, 2962, 2875, 1475, 1381, 1353, 1246, 1219, 1193, 1022, 961, 921, 876., 750, 703; **HRMS (ESI-TOF)**: $(M^*)^-$ calcd for $C_{19}H_{14}BN_2O_2^-$: 313.1154, found: 313.1155.

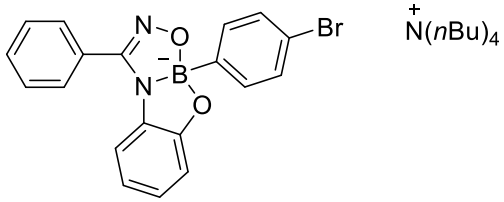
Two-step protocol:



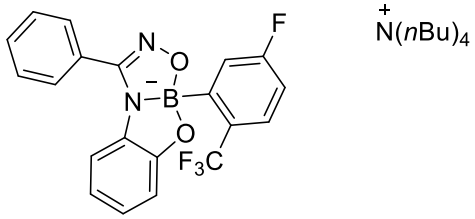
General procedure D: A stirred mixture of amidoxime **4** (1.00 equiv), boronic acid (1.05 equiv) and molecular sieves 4Å (100 mg – 500 mg) in PhMe (0.5 M) was heated under reflux at 110 °C in a sealed vial for 1 h. The reaction mixture was allowed to cool down to room temperature and filtered through a short plug of silica gel (EtOAc). The volatiles were evaporated *in vacuo* to provide the corresponding oxadiazaborole that was used directly for the next step without further purification.

A solution of TBAF (1.0 M in THF, 1.5 equiv) was slowly added to a stirred mixture of the oxadiazaborole (1.0 equiv as theoretical amount) in THF (0.2 M) at 0 °C. After stirring for one hour

at that temperature, the reaction mixture was quenched with a 2.0 M solution of K_2CO_3 (20 mL) and extracted with DCM (3 × 25 mL). The combined organic phases were washed with saturated $NaHCO_3$ (30 mL), water (25 mL) and brine (25 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The crude product was washed (triturated) with Et_2O three times to provide the title borate as a solid.

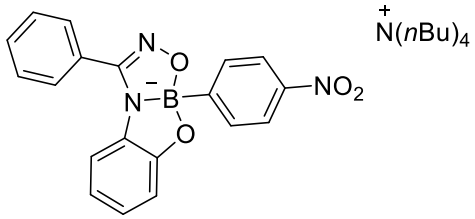


Tetrabutylammonium 10-(4-bromophenyl)-3-phenyl-10H-benzo[4,5][1,3,2]oxazaborolo[3,2-d][1,2,4,5]oxadiazaborol-10-uide (6c): Prepared from (*Z*)-*N*-(2-((*tert*-butyldimethylsilyl)oxy) phenyl)-*N*'-hydroxybenzimidamide (**4n**) (300 mg, 0.88 mmol, 1.00 equiv), (4-bromophenyl)boronic acid (193 mg, 0.96 mmol, 1.10 equiv) in PhMe (1.8 mL) and TBAF (1.00 M in THF, 1.31 mL, 1.31 mmol, 1.50 equiv as theoretical amount) in THF (4.4 mL) following the general procedure **D**. Isolated as a grey solid (350 mg, 63% yield); **mp** = 151 °C; **^{1}H NMR** (498 MHz, CD_3CN): δ (ppm) 7.77 (br s, 2H), 7.45 – 7.37 (m, 5H), 7.34 – 7.29 (m, 2H), 6.69 (td, *J* = 7.7, 1.3 Hz, 2H), 6.63 (dd, *J* = 7.9, 1.4 Hz, 1H), 6.30 (td, *J* = 7.5, 1.4 Hz, 1H), 3.08 – 3.03 (m, 8H), 1.60 – 1.53 (m, 8H), 1.33 (sext, *J* = 7.4 Hz, 8H), 0.95 (t, *J* = 7.3 Hz, 12H); **^{13}C NMR** (126 MHz, CD_3CN): δ (ppm) 161.9, 157.9, 140.6, 134.9, 133.6, 130.5, 129.7, 129.3, 129.0, 123.6, 120.1, 117.0, 110.8, 59.3, 24.3, 20.3, 13.8; **^{11}B NMR** (160 MHz, CD_3CN): δ (ppm) 14.8; **IR** (cast film, cm^{-1}): 2962, 2875, 1475, 1353, 1246, 1217, 1187, 1067, 960, 810, 751, 700; **HRMS (ESI-TOF)**: $(M^*)^-$ calcd for $C_{19}H_{13}BBrN_2O_2^-$: 391.0259, found: 391.0258.



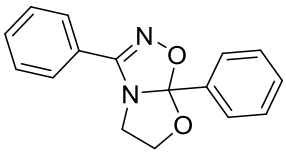
Tetrabutylammonium 10-(5-fluoro-2-(trifluoromethyl)phenyl)-3-phenyl-10H-benzo[4,5][1,3,2] oxazaborolo[3,2-d][1,2,4,5]oxadiazaborol-10-uide (6d): Prepared from (*Z*)-*N*-(2-((*tert*-butyldimethylsilyl)oxy) phenyl)-*N*′-hydroxybenzimidamide (**4n**) (100 mg, 0.29 mmol, 1.00 equiv), (5-fluoro-2-(trifluoromethyl)phenyl)boronic acid (67.0 mg, 0.32 mmol, 1.10 equiv) in PhMe (0.9

mL) and TBAF (1.00 M in THF, 0.44 mL, 0.44 mmol, 1.50 equiv as theoretical amount) in THF (1.5 mL) following the general procedure **D**. Isolated as a grey solid (91.0 mg, 49% yield); **mp** = 127 °C; **^{1}H NMR** (400 MHz, CD_3CN): δ (ppm) 7.90 – 7.85 (m, 2H), 7.64 – 7.58 (m, 1H), 7.52 – 7.39 (m, 4H), 6.98 – 6.87 (m, 1H), 6.76 (dd, *J* = 7.6, 1.4 Hz, 1H), 6.66 (td, *J* = 7.6, 1.4 Hz, 1H), 6.51 (ddd, *J* = 7.7, 1.3, 0.4 Hz, 1H), 6.32 (td, *J* = 7.6, 1.3 Hz, 1H), 3.10 – 3.02 (m, 8H), 1.58 (ddd, *J* = 11.8, 10.0, 6.2 Hz, 8H), 1.41 – 1.27 (m, 8H), 0.95 (t, *J* = 7.3 Hz, 12H); **^{13}C NMR** (126 MHz, CD_3CN): δ (ppm) 165.2 (d, *J* = 248.1 Hz), 161.7, 157.1, 140.3, 133.5, 129.8, 129.4, 129.1, 129.0, 128.4 (d, *J* = 30.5 Hz), 126.6 (d, *J* = 273.0 Hz), 123.5, 120.9 (d, *J* = 19.1 Hz), 117.8, 117.2, 112.9 (d, *J* = 22.7 Hz), 111.0, 59.3, 24.3, 20.3, 13.8; **^{19}F NMR** (376 MHz, CD_3CN): δ (ppm) -58.3, -113.2; **^{11}B NMR** (128 MHz, CD_3CN): δ (ppm) 15.2; **IR** (cast film, cm^{-1}): 2964, 2877, 1575, 1477, 1359, 1313, 1256, 1246, 1211, 1140, 1115, 1049, 1022, 1002, 888, 806, 751, 700; **HRMS (ESI-TOF)**: $(M^*)^-$ calcd for $C_{20}H_{12}BF_4N_2O_2^-$: 399.0933, found: 399.0931.

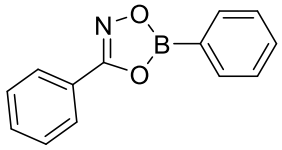


Tetrabutylammonium 10-(4-nitrophenyl)-3-phenyl-10H-benzo[4,5][1,3,2]oxazaborolo[3,2-d][1,2,4,5]oxadiazaborol-10-uide (6e): Prepared from (*Z*)-*N*-(2-((*tert*-butyldimethylsilyl)oxy) phenyl)-*N*'-hydroxybenzimidamide (**4n**) (120 mg, 0.35 mmol, 1.00 equiv), (4-nitrophenyl)boronic acid (65.0 mg, 0.39 mmol, 1.10 equiv) in PhMe (1.4 mL) and TBAF (1.00 M in THF, 0.53 mL, 0.53 mmol, 1.50 equiv as theoretical amount) in THF (1.8 mL) following the general procedure **D**. Isolated as a yellow solid (135 mg, 64% yield); **mp** = 156 °C; **^{1}H NMR** (498 MHz, CD_3CN): δ (ppm) 8.08 – 7.97 (m, 2H), 7.79 (d, *J* = 6.3 Hz, 2H), 7.71 – 7.65 (m, 2H), 7.46 – 7.36 (m, 3H), 6.74 – 6.69 (m, 2H), 6.65 (dd, *J* = 8.0, 1.4 Hz, 1H), 6.33 (td, *J* = 7.6, 1.4 Hz, 1H), 3.11 – 3.02 (m, 8H), 1.58 (ddd, *J* = 11.8, 10.0, 6.2 Hz, 8H), 1.33 (sext, *J* = 7.4 Hz, 8H), 0.95 (t, *J* = 7.3 Hz, 12H); **^{13}C NMR** (126 MHz, CD_3CN): δ (ppm) 162.0, 157.7, 147.5, 140.4, 133.4, 133.3, 129.8, 129.3, 129.1, 123.7, 122.6, 117.3, 111.0, 59.3, 24.3, 20.3, 13.8; **^{11}B NMR** (160 MHz, CD_3CN): δ (ppm) 14.5; **IR** (cast film, cm^{-1}): 2963, 2876, 1584, 1507, 1381, 1245, 1185, 1024, 961, 924, 847, 748, 701; **HRMS (ESI-TOF)**: $(M^*)^-$ calcd for $C_{19}H_{13}BN_3O_4^-$: 358.1005, found: 358.1003.

1.3.4 Additional compounds



3,7a-Diphenyl-5,6-dihydro-7aH-oxazolo[3,2-d][1,2,4]oxadiazole (7): Prepared from (*Z*)-*N*-hydroxybenz-imidoyl chloride[1] (**3b**) (0.49 g, 3.15 mmol, 1.00 equiv), 2-phenyl-2-oxazoline (0.50 mL, 3.78 mmol, 1.20 equiv) and Et_3N (0.66 mL, 4.73 mmol, 1.50 equiv) in THF (5.0 mL) following the general procedure **A**. The crude residue was purified by flash column chromatography (hexane/EtOAc). The resulting solid was washed with Et_2O (trituration) to yield the pure compound as a white crystalline solid which spectral data were in agreement with those previously reported in the literature;[5] **mp** = 94 °C; **^{1}H NMR** (500 MHz, $CDCl_3$): δ (ppm) 7.80 – 7.75 (m, 2H), 7.71 – 7.65 (m, 2H), 7.54 – 7.45 (m, 3H), 7.44 – 7.38 (m, 3H), 4.12 – 4.04 (m, 1H), 3.81 – 3.69 (m, 2H), 3.45 – 3.36 (m, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$): δ (ppm) 158.2, 137.6, 131.3, 129.6, 129.2, 128.4, 128.0, 126.6, 125.6, 124.4, 64.6, 49.6; **IR** solid, cm^{-1}): 3060, 3007, 2968, 2895, 1597, 1570, 1454, 1377, 1315, 1285, 1216, 1073, 1023, 1009, 972, 862, 771, 701; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{16}H_{15}N_2O_2^+$: 267.1128, found: 267.1131.



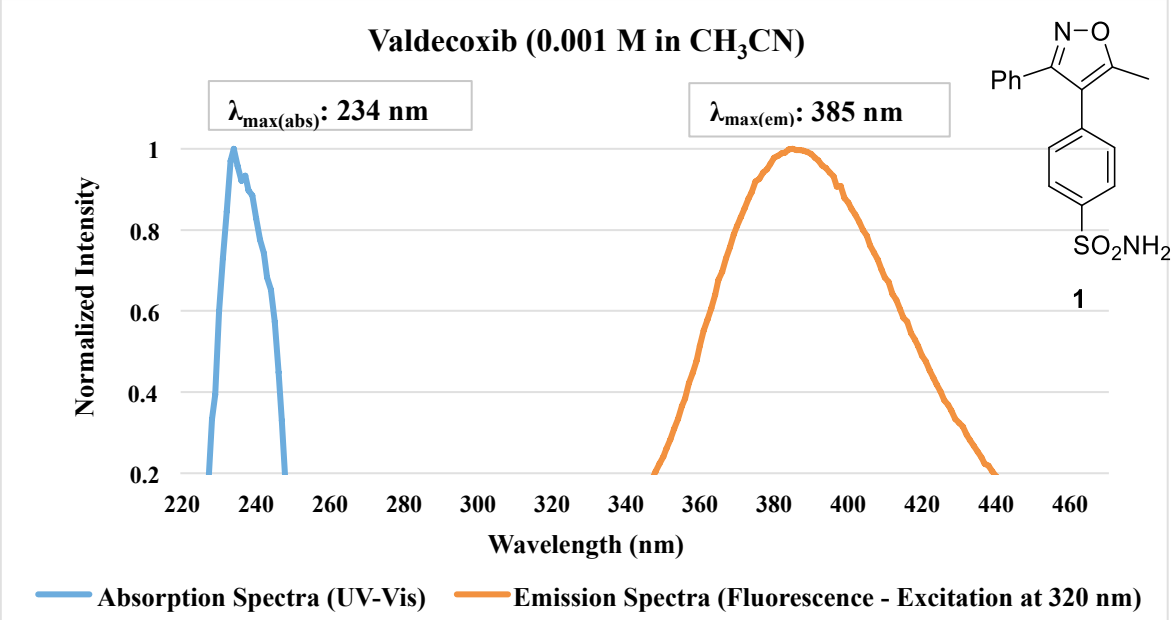
2,5-Diphenyl-1,3,4,2-dioxazaborole (8): Prepared from *N*-hydroxybenzamide (200 mg, 1.46 mmol, 1.00 equiv) and phenylboronic acid (187 mg, 1.53 mmol, 1.05 equiv) in PhMe (3.0 mL) following the general procedure **B**. Isolated as a white solid (126 mg, 39% yield); **mp** = 95 °C; **^{1}H NMR** (498 MHz, $CDCl_3$): δ (ppm) 8.07 – 8.01 (m, 4H), 7.63 – 7.47 (m, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ (ppm) 165.1, 135.2, 132.8, 132.1, 129.1, 128.5, 127.0, 124.3; **^{11}B NMR** (160 MHz, $CDCl_3$): δ (ppm) 33.2; **IR** (solid, cm^{-1}): 1603, 1410, 1383, 1364, 1353, 1088, 1028, 781, 699; **HRMS (ESI-TOF)**: $(M+H)^+$ calcd for $C_{13}H_{11}BNO_2^+$: 224.0877, found: 224.0883.

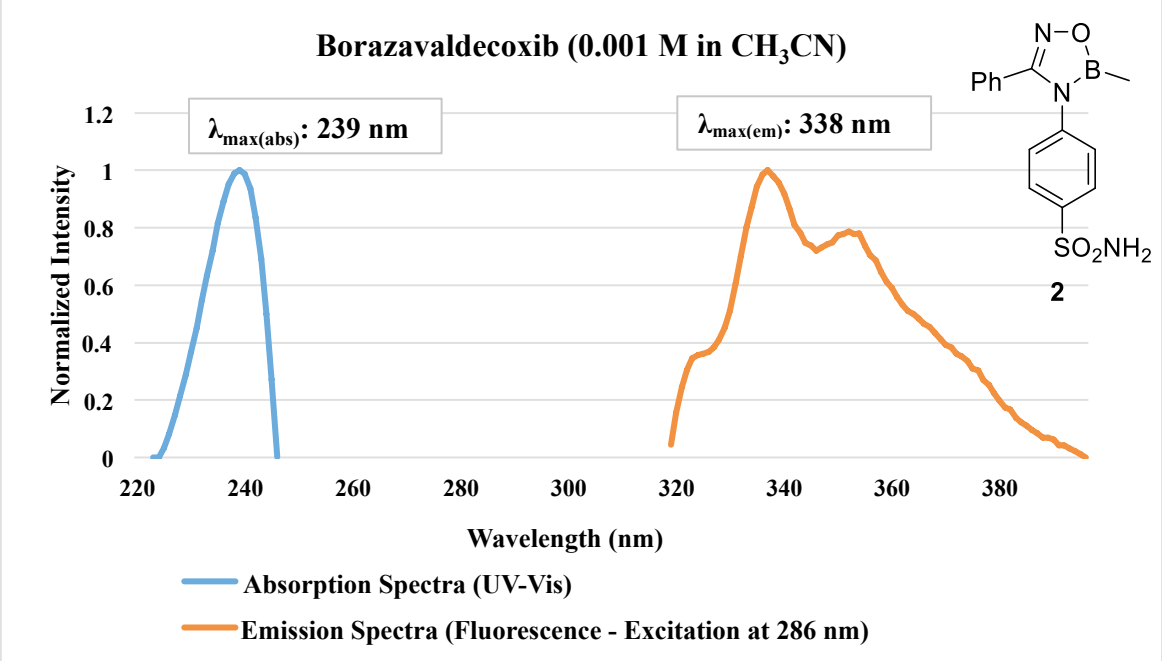
[5] (a) Kennewell, P. D.; Miller, D. J.; Scrowston, R. M.; Westwood, R. *J. Chem. Soc., Perkin Trans. 1*, **1994**, 3563-3566; (b) Miller, D. J.; Scrowston, R. M.; Kennewell, P. D.; Westwood, R. *Tetrahedron,* **1992**, *48*, 7703-7712; (c) Magosch, K. H.; Feinauer, R. *Angew. Chem. Int. Ed.* **1971**, *10*, 810.

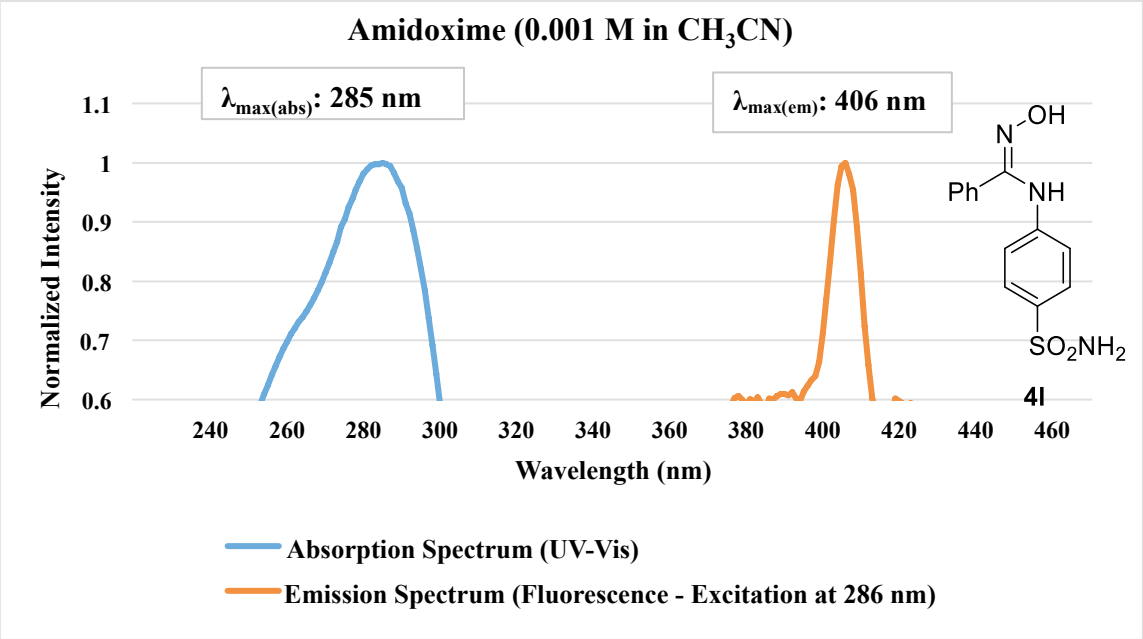
2 Spectroscopic comparison: Borazavaldecoxib *vs* Valdecoxib

2.1 UV absorption and fluorescence emission spectra

Physical properties	Valdecoxib 1	Borazavaldecoxib 2	Amidoxime 4l
$\lambda_{max(abs)}$ (nm)	234	239	285
$\lambda_{max(em)}$ (nm)	385	338	406
Stokes shift	151 nm, 66225 cm^{-1}	99 nm, 101010 cm^{-1}	121 nm, 82644 cm^{-1}
ε ($\times 10^4$ $M^{-1}cm^{-1}$)	1.95	1.93	1.85

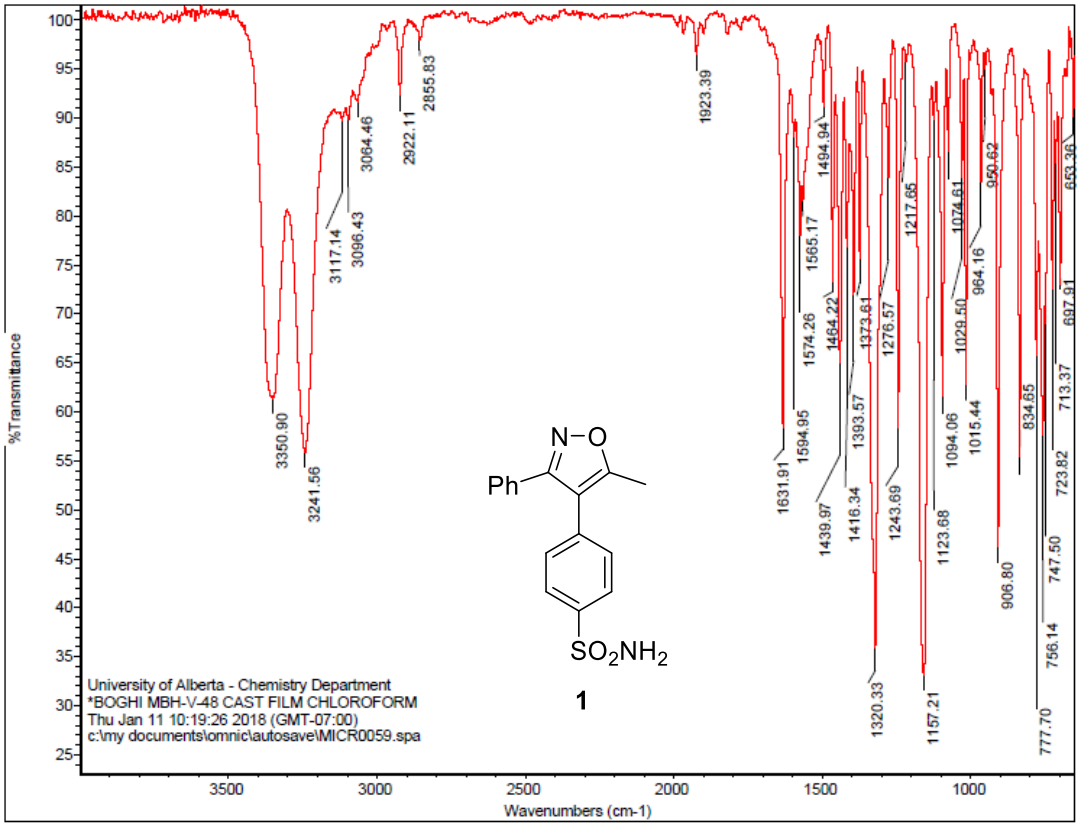


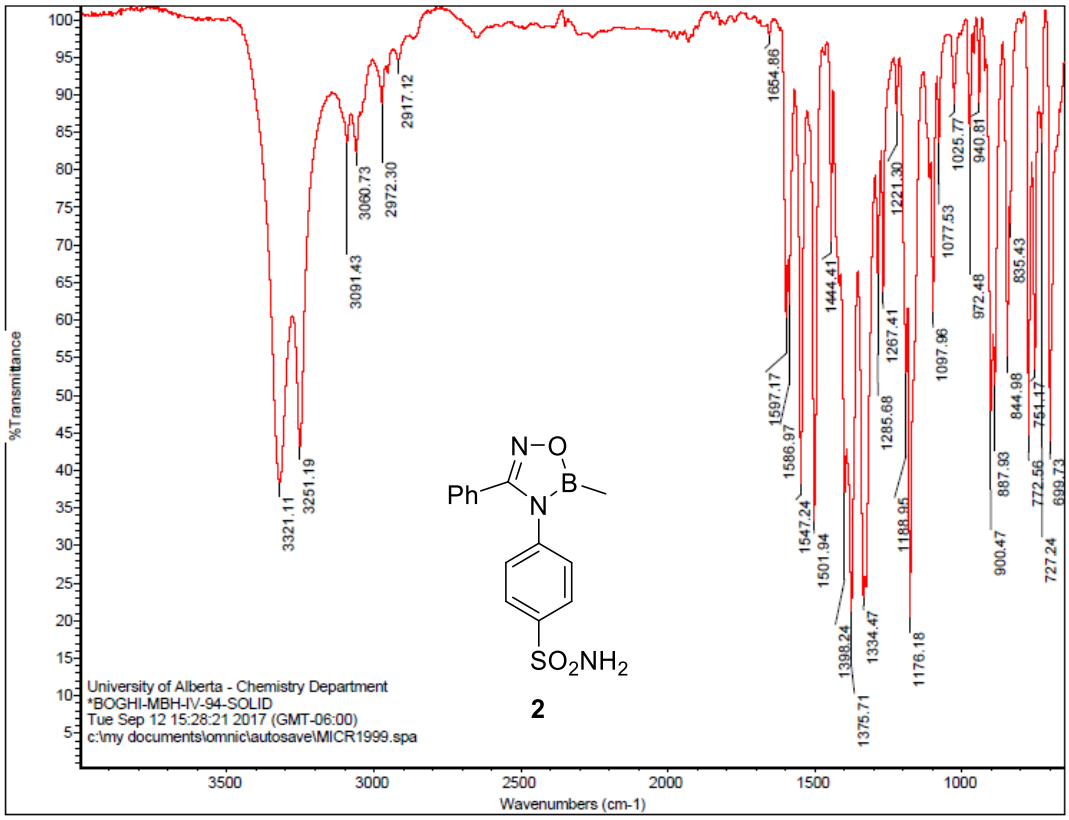




$\lambda_{max(am)}$ (UV) of tetrahedral borate **6a** = **290 nm**

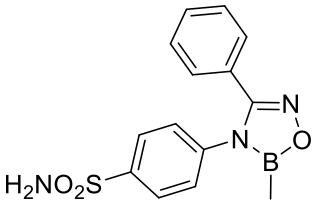
2.2 Infrared spectra



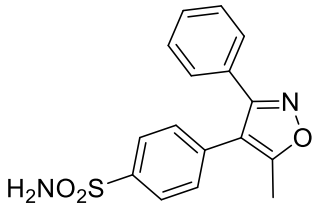


3 Structural comparisons

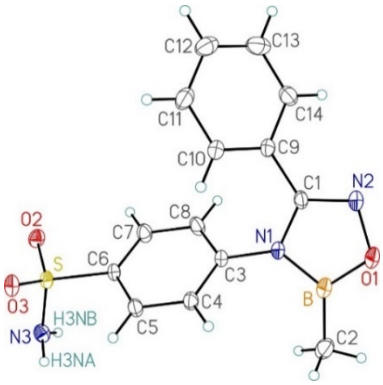
3.1 Borazavaldecoxib vs valdecoxib

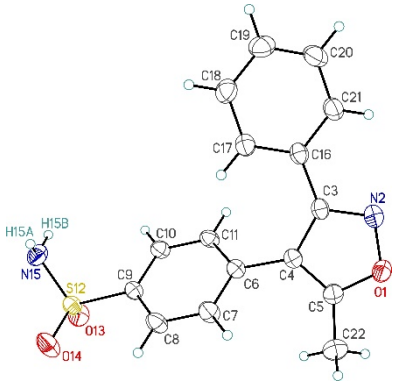


Borovaldecoxib (**2**)



Valdecoxib (**1**)





Selected Interatomic Distances (Å):

Borazavaldecoxib (2)			Valdecoxib (1)			
Atom 1	**Atom 2**	**Distance (Å)**	**Atom 1**	**Atom 2**	**Distance (Å)**	**Δ (Å)**
O1	N2	**1.443**	O1	N2	**1.404**	**0.039**
O1	B	**1.384**	O1	C5	**1.348**	**0.036**
N1	C1	**1.389**	C3	C4	**1.441**	**0.052**
N1	C3	**1.423**	C4	C6	**1.482**	**0.059**
N1	B	**1.433**	C4	C5	**1.347**	**0.086**
N2	C1	**1.292**	N2	C3	**1.317**	**0.025**
C1	C9	**1.480**	C3	C16	**1.479**	**0.001**
C2	B	**1.555**	C5	C22	**1.486**	**0.069**
Mean difference in bond lengths:						**0.046**

Selected Interatomic Angles (deg):

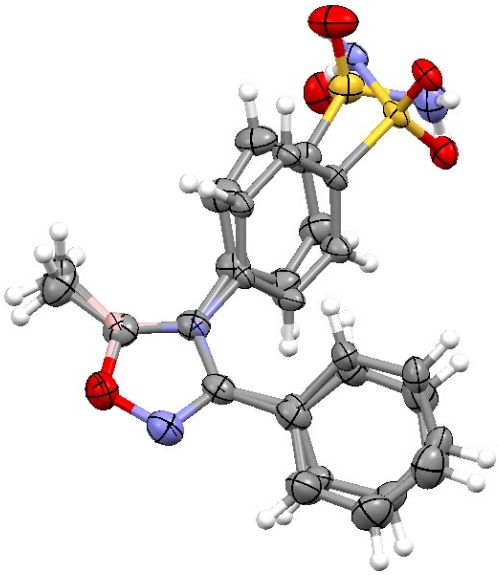
Borazavaldecoxib (2)				Valdecoxib (1)				
Atom 1	**Atom 2**	**Atom 3**	**Angle (dec)**	**Atom 1**	**Atom 2**	**Atom 3**	**Angle (dec)**	**Δ (dec)**
N2	O1	B	**109,2**	C5	O1	N2	**108,7**	**0,5**
C1	N1	C3	**124,5**	C3	C4	C6	**130,2**	**5,7**
C1	N1	B	**105,6**	C5	C4	C3	**104,0**	**1,6**

C3	N1	B	**129,9**	C5	C4	C6	**125,8**	**4,1**
O1	N2	C1	**105,7**	C3	N2	O1	**106,0**	**0,3**
N1	C1	N2	**113,8**	N2	C3	C4	**110,8**	**3,0**
N1	C1	C9	**125,0**	C4	C3	C16	**131,6**	**6,6**
N2	C1	C9	**121,1**	N2	C3	C16	**117,5**	**3,6**
N1	C3	C4	**118,9**	C11	C6	C4	**120,9**	**2,0**
N1	C3	C8	**120,4**	C7	C6	C4	**120,6**	**0,2**
C1	C9	C10	**121,0**	C21	C16	C3	**119,7**	**1,3**
C1	C9	C14	**119,3**	C17	C16	C3	**121,3**	**2,0**
O1	B	N1	**105,7**	C4	C5	O1	**110,5**	**4,8**
O1	B	C2	**124,6**	O1	C5	C22	**115,5**	**9,1**
N1	B	C2	**129,7**	C4	C5	C22	**133,9**	**4,2**
Mean difference in bond angles:								**3,3**

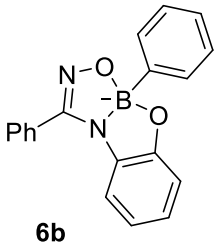
Torsional Angles (deg):

Borazavaldecoxib (2)				
Atom 1	**Atom 2**	**Atom 3**	**Atom 4**	**Angle**
B	O1	N2	C1	-1.0(3)
N2	O1	B	N1	1.4(3)
C1	N1	B	O1	-1.2(3)

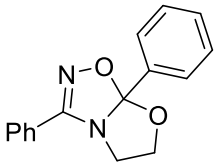
Overlaid picture of valdecoxib (**1**) and borazavaldecoxib (**2**) X-ray crystal structures:



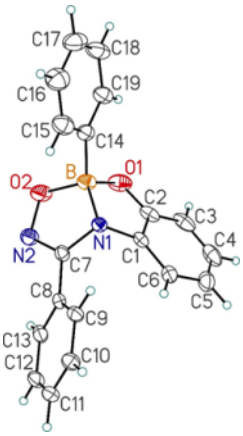
3.2 Tetrahedral oxadiazaborate 6b *vs* orthoamide 7

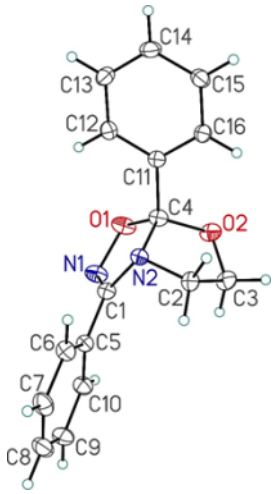


6b



7





Selected Interatomic Distances (Å):

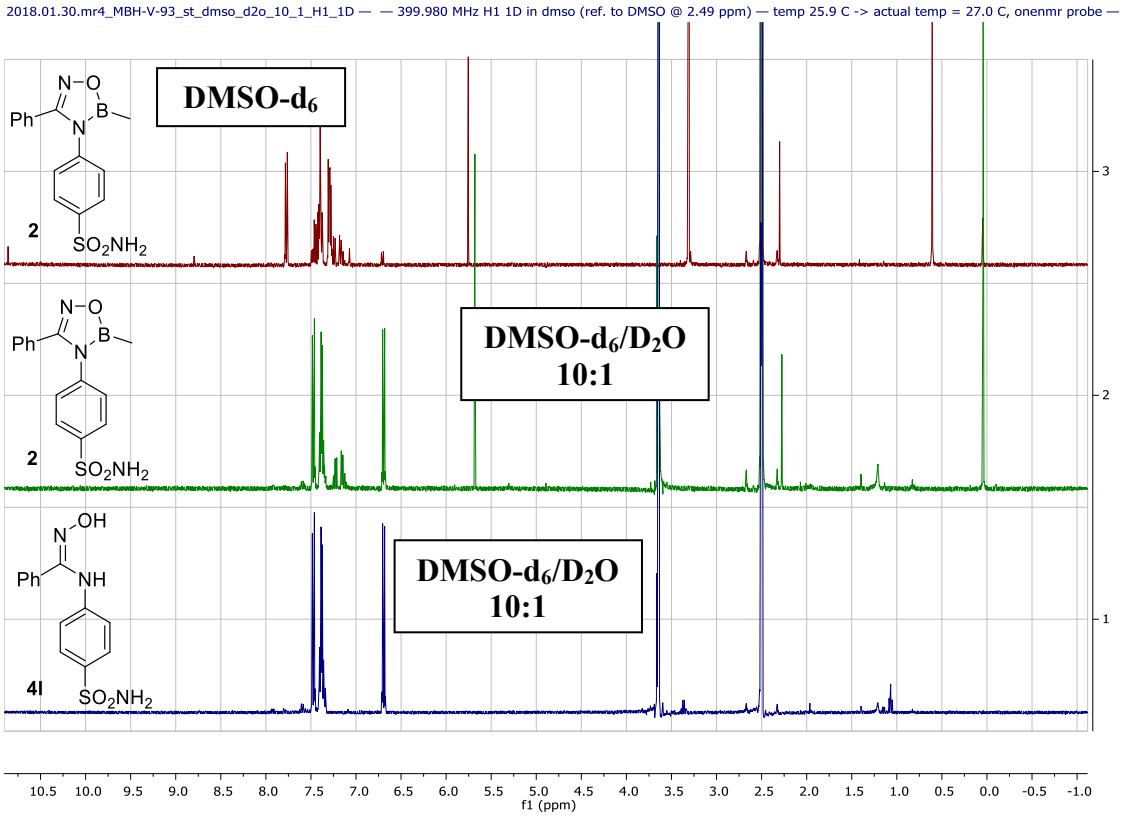
Oxadiazaborate (6b)			Carbon bicycle (7)			
Atom 1	**Atom 2**	**Distance (Å)**	**Atom 1**	**Atom 2**	**Distance (Å)**	**Δ (Å)**
O1	B	**1.514**	O2	C4	**1.4135**	**0.1005**
O2	B	**1.484**	O1	C4	**1.4226**	**0.0614**
N1	B	**1.559**	N2	C4	**1.4714**	**0.0876**
Mean difference in bond lengths:						**0.0832**

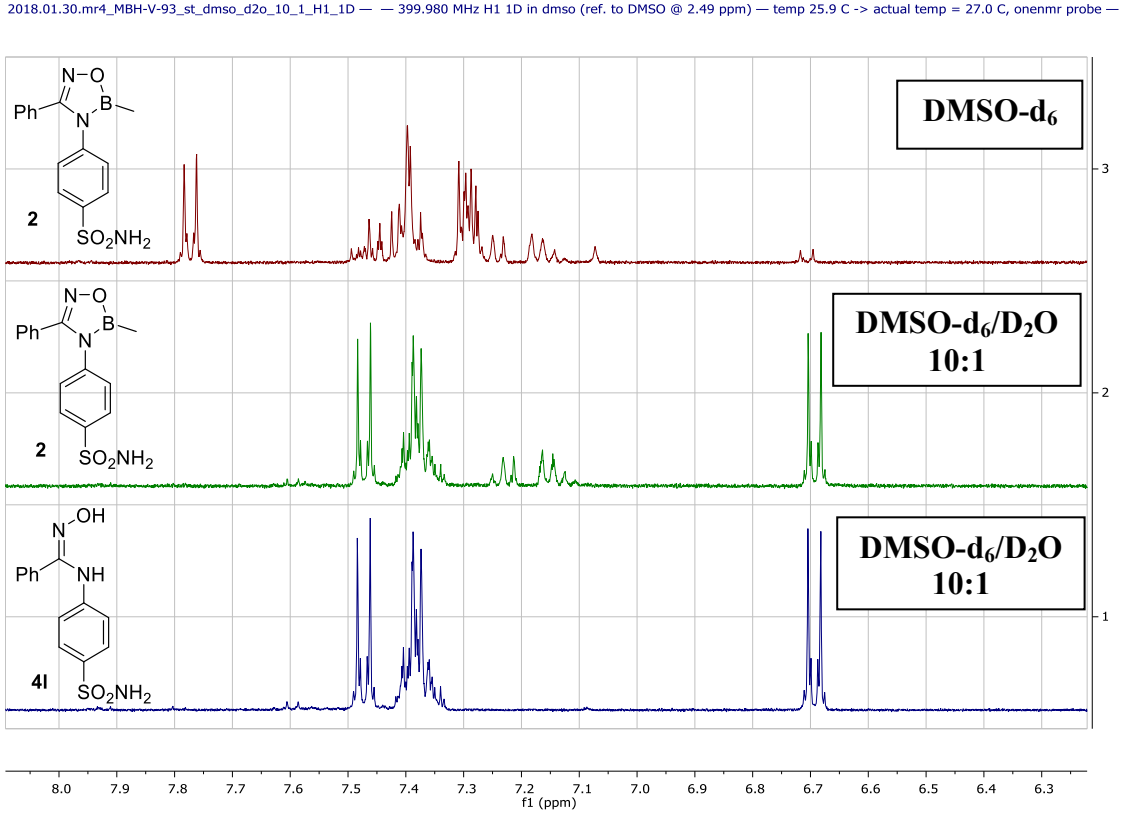
Selected Interatomic Angles (deg):

Oxadiazaborate (6b)				Carbon bicycle (7)				
Atom 1	**Atom 2**	**Atom 3**	**Angle (dec)**	**Atom 1**	**Atom 2**	**Atom 3**	**Angle (dec)**	**Δ (dec)**
O1	B	O2	**114.5**	O1	C4	O2	**112.1**	**2.4**
O1	B	N1	**103.0**	O2	C4	N2	**106.7**	**3.7**
O1	B	C14	**110.0**	O2	C4	C11	**109.9**	**0.1**
O2	B	N1	**101.5**	O1	C4	N2	**103.8**	**2.3**
O2	B	C14	**108.4**	O1	C4	C11	**109.7**	**1.3**
N1	B	C14	**119.5**	N2	C4	C11	**114.6**	**4.9**
Mean difference in bond angles:								**2.4**

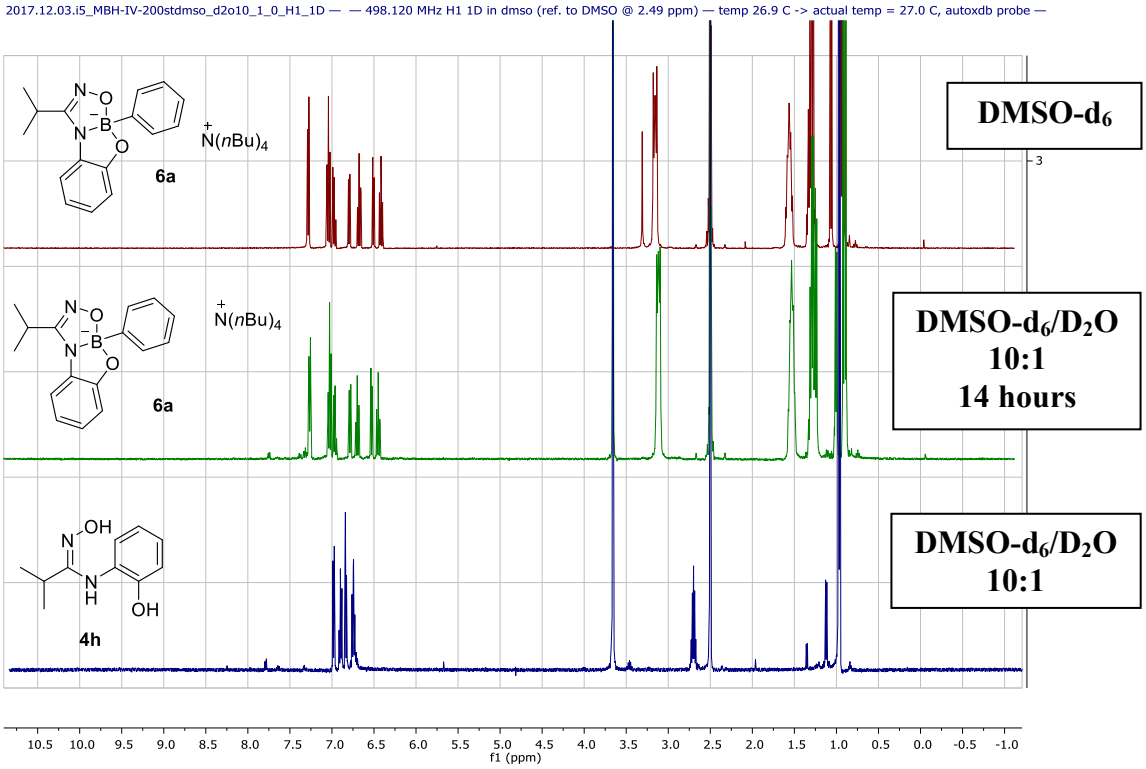
4 Hydrolytic studies (NMR)

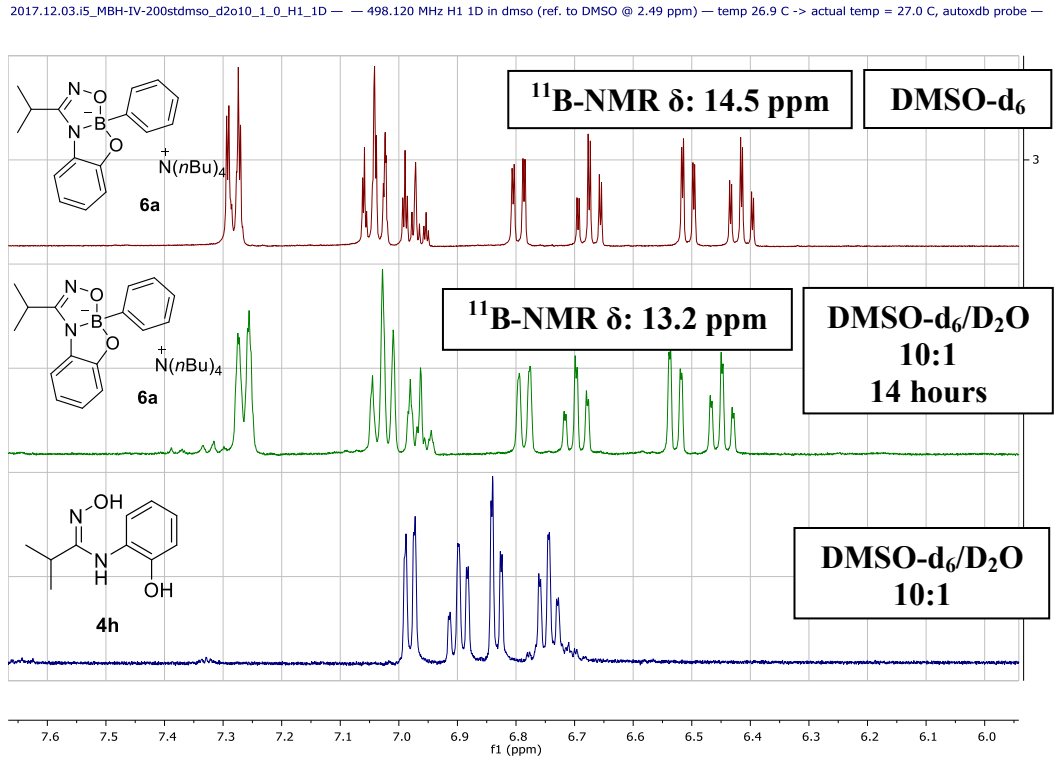
4.1 Borazavaldecoxib 2 in aqueous DMSO-d_6 (0.02 M, 20 °C)



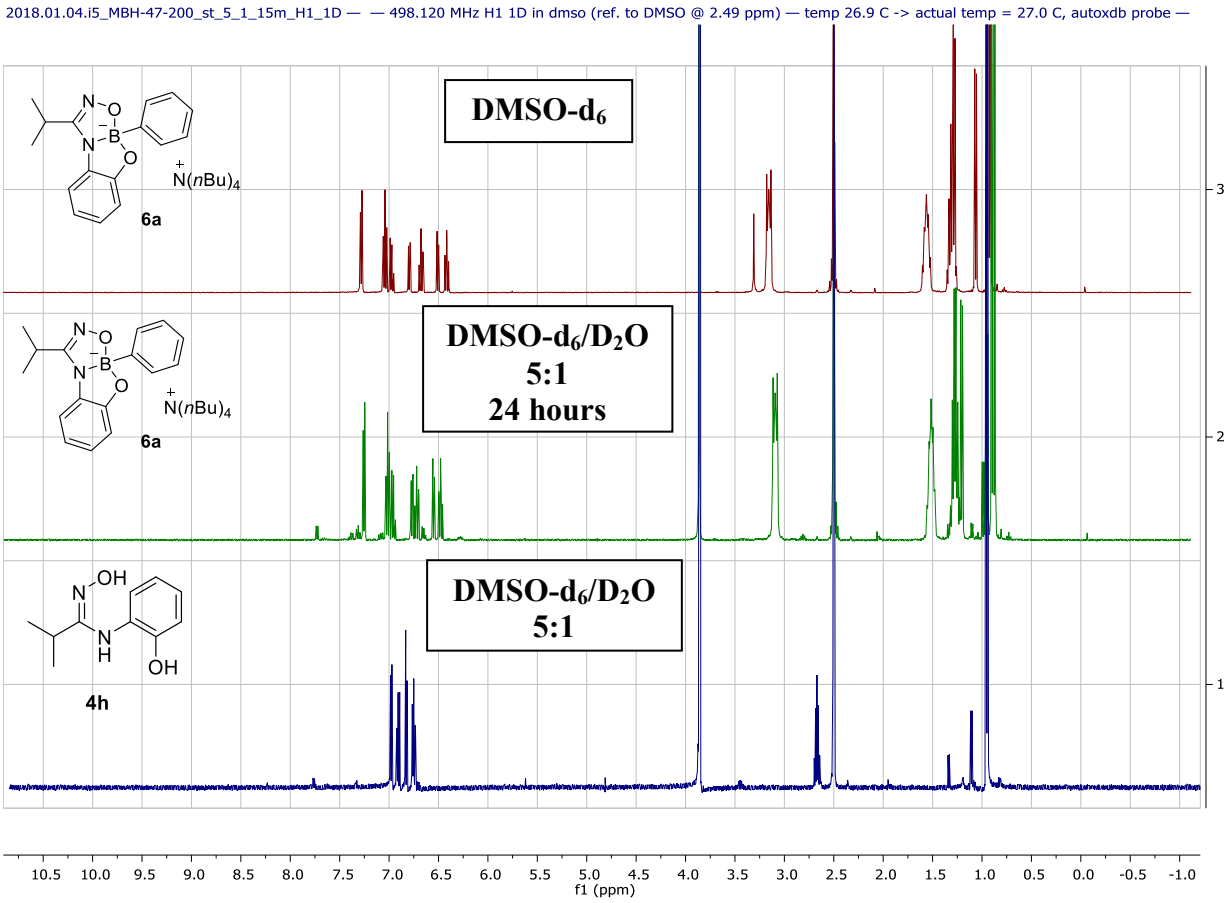


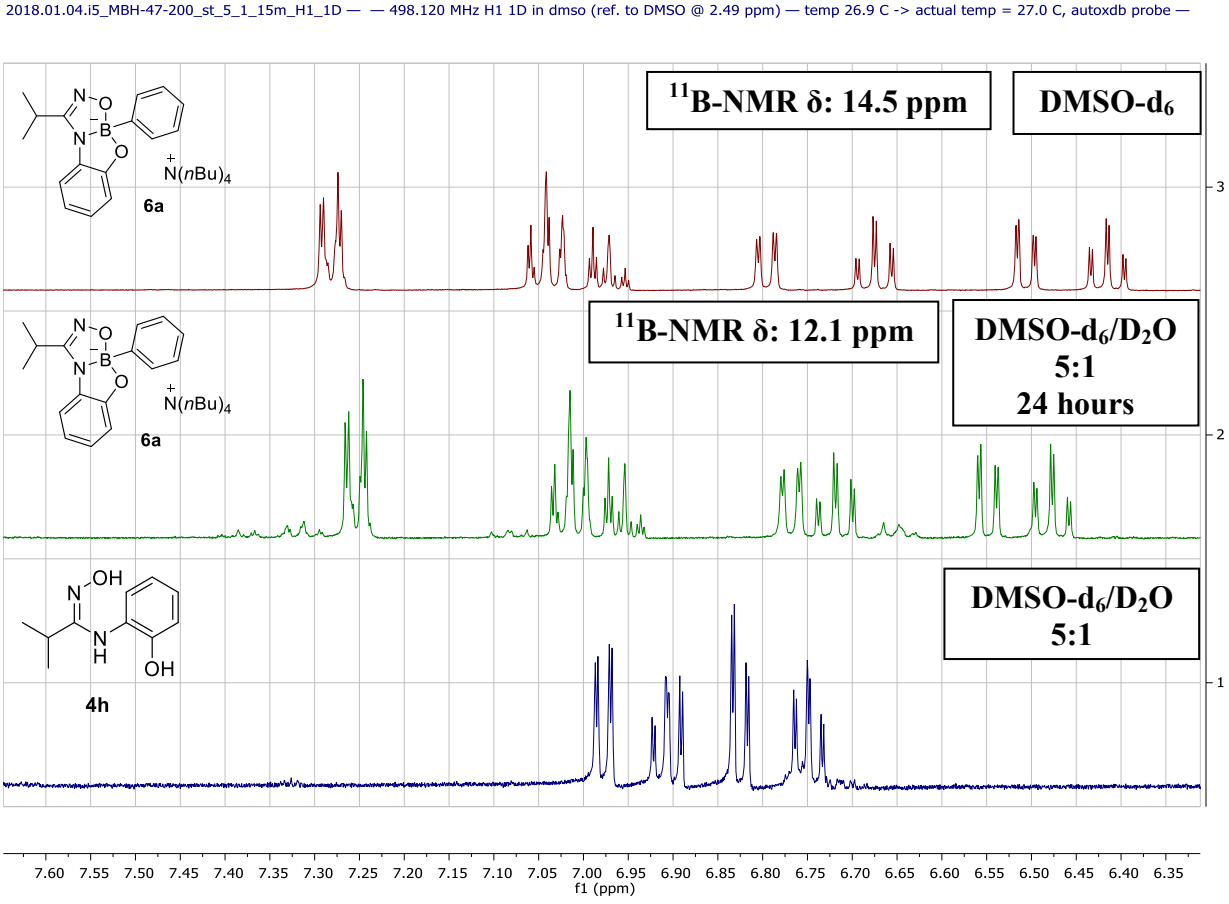
4.2 Tetrahedral borate 6a in aqueous DMSO-d_6 (0.02 M, 20 °C)[6]



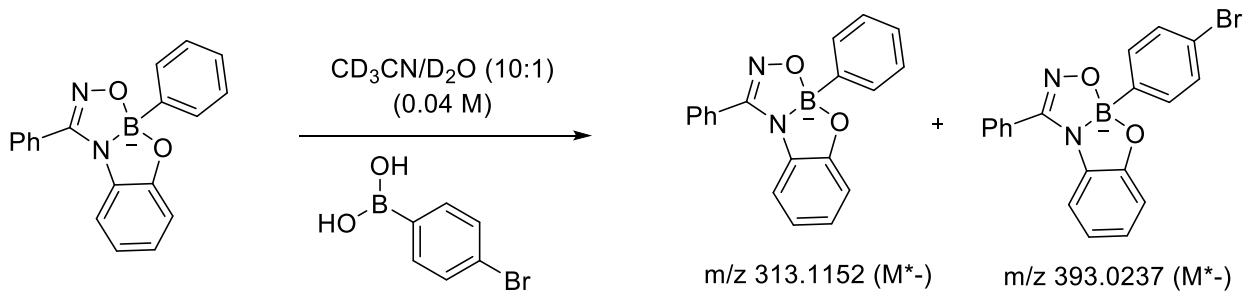


[6] When a higher content of water was present in the mixture, the oxadiazaborate **6a** was too insoluble for a proper analysis. In such conditions, a more complex mixture of products was observed by ^{1}H-NMR, suggesting the presence of different aggregates in equilibrium. However, even with a high content of water, the hydrolyzed product **4h** was clearly not observed in the mixture


2018.01.04.i5_MBH-47-200_st_5_1_15m_H1_1D — — 498.120 MHz H1 1D in dmso (ref. to DMSO @ 2.49 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
DMSO-d6
6a
DMSO-d6/D2O
5:1
24 hours
6a
DMSO-d6/D2O
5:1
4h
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


2018.01.04.i5_MBH-47-200_st_5_1_15m_H1_1D — — 498.120 MHz H1 1D in dmso (ref. to DMSO @ 2.49 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
11B-NMR δ: 14.5 ppm
DMSO-d6
6a
11B-NMR δ: 12.1 ppm
DMSO-d6/D2O
5:1
24 hours
6a
DMSO-d6/D2O
5:1
4h
7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75 6.70 6.65 6.60 6.55 6.50 6.45 6.40 6.35
f1 (ppm)

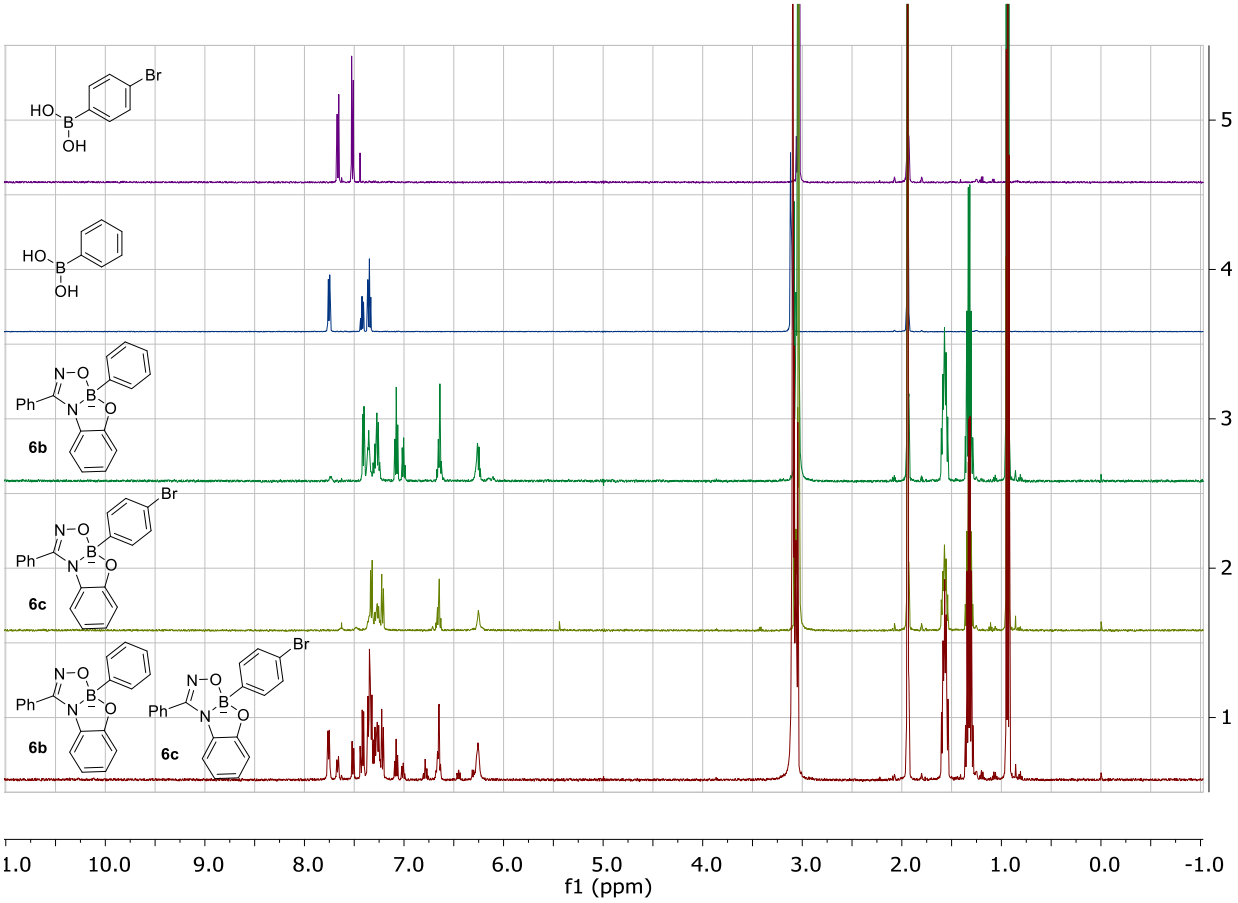
5 Exchange experiment of oxadiazaborate 6b in aqueous conditions

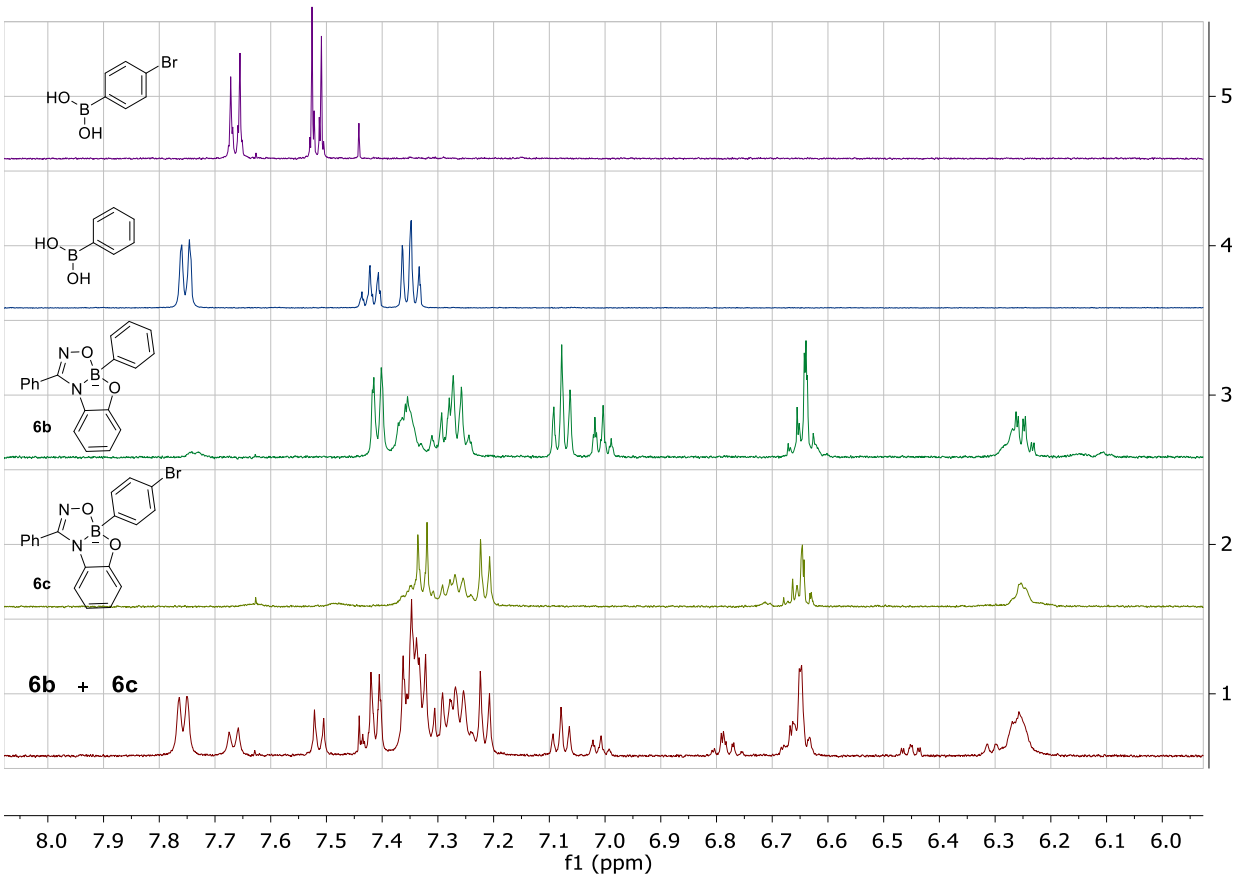


6b (1.0 eq) (1..0 eq) **6b** **6c**

A mixture of **6b** (5.0 mg, 0.009 mmol, 1.0 equiv) and 4-bromophenylboronic acid (1.8 mg, 0.009 mmol, 1.0 equiv) in CD_3CN/D_2O 10:1 (0.04M, 2.2 mL) was stirred at room temperature for 5 minutes before it was analyzed by ^{1}H-NMR and HRMS (-ESI). **6b** was found to have exchanged parts to give a mixture of **6b** and **6c**.

^{1}H-NMR (500 MHz, CD_3CN):





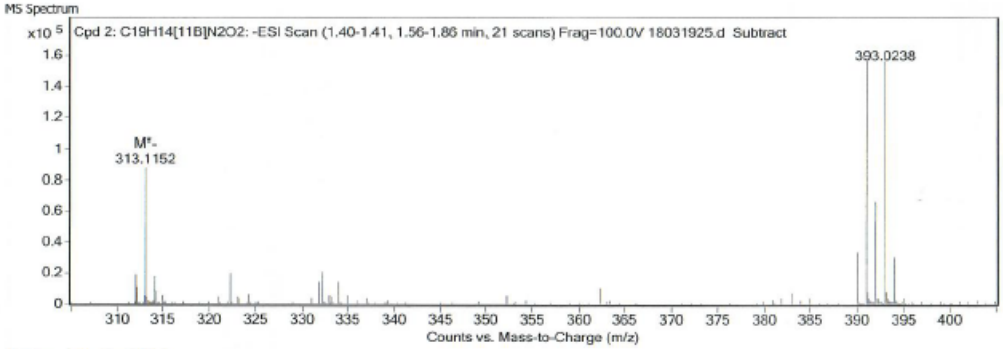
University of Alberta — **Qualitative Compound Report** — Department of Chemistry

Comment	M. Boghi, Hall	**Sample Name**	mbh-v 88
Data File	18031925.d	**Instrument Name**	caTOF6220
Position	-1	**Operator**	ami
Acq Method		**DA Method**	da ami low mass.m

Compound Table

Formula	Mass	Abund
C19H14[11B]N2O2	313.1146	87616

MS Spectrum



MS Spectrum Peak List

Ion Formula	Ion type	*Measured m/z*	*Calc m/z*	Diff(ppm)	z	Abund
C19 H14 [11B] N2 O2	M*-	313.1152	313.1154	-0.64	-1	87616

--- End Of Report ---

Mass Spectrometry Facility
+1-780-492-5577

Page 1 of 1

Printed at: 8:45 PM on:3/19/2018

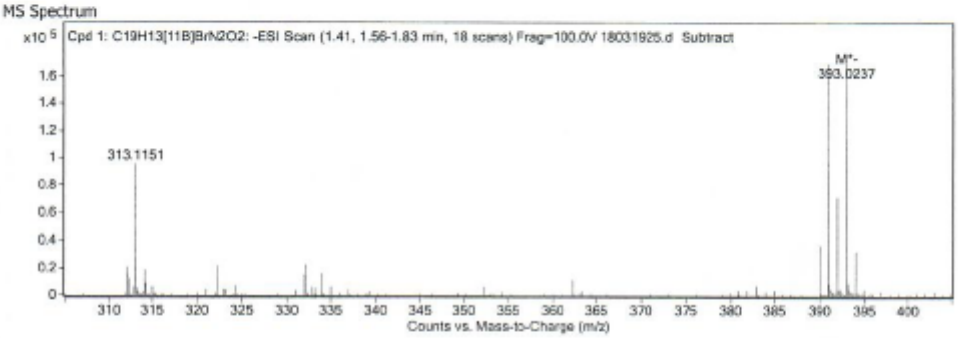
University of Alberta **Qualitative Compound Report** Department of Chemistry

Comment	M. Boghi, Hall	**Sample Name**	mbh-v 88
Data File	18031925.d	**Instrument Name**	oaTOF6220
Position	-1	**Operator**	ami
Acq Method		**DA Method**	da ami low mass.m

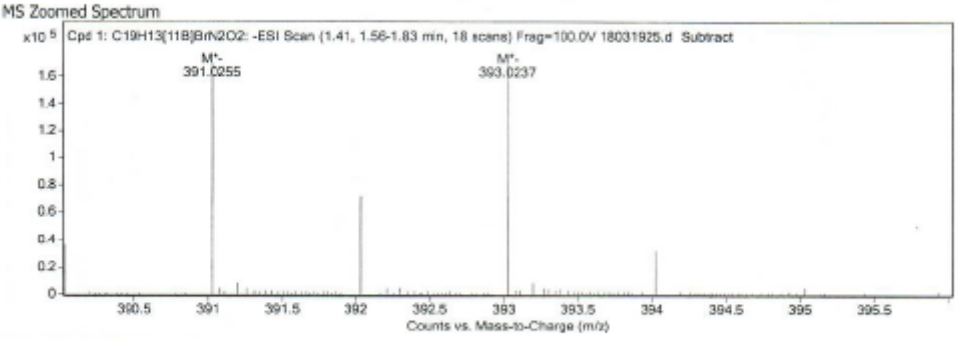
Compound Table

Formula	Mass	Abund
C19H13[11B]BrN2O2	391.0249	168775

MS Spectrum



MS Zoomed Spectrum



MS Spectrum Peak List

Ion Formula	Ion type	*Measured m/z*	*Calc m/z*	Diff(ppm)	z	Abund
C19 H13 [11B] Br N2 O2	M*-	391.0255	391.0259	-1.12	1	168775

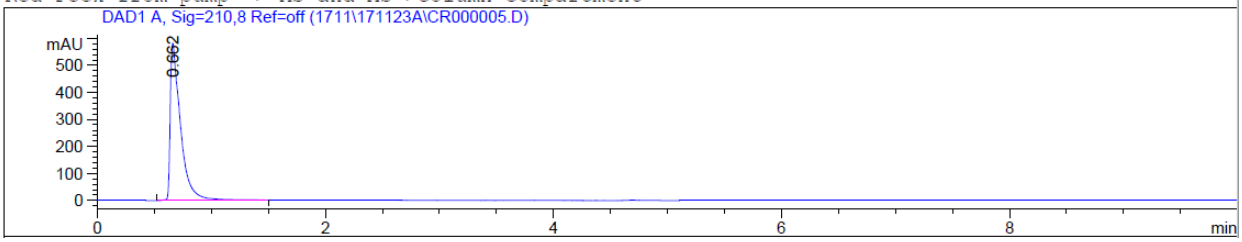
--- End Of Report ---

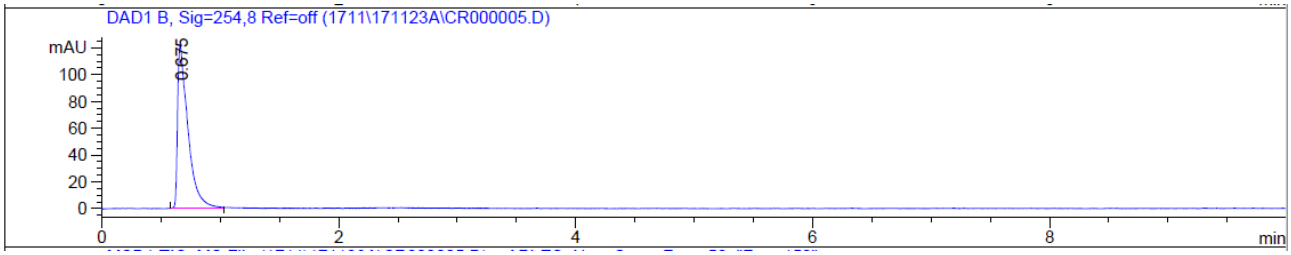
6 HPLC trace of oxadiazaborate 6a with ACN as mobile phase

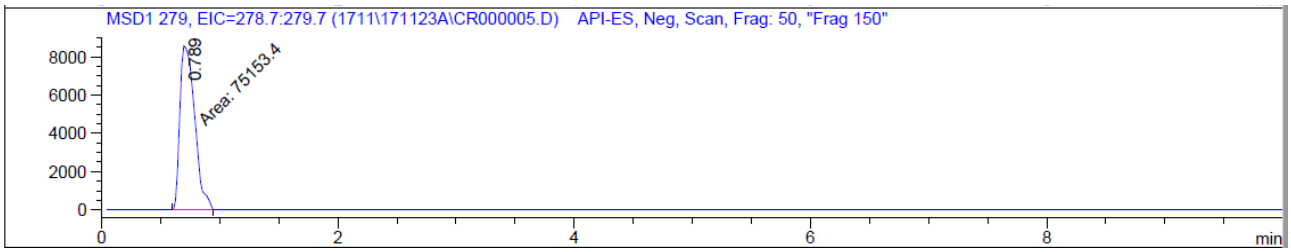
```
Data File C:\HPCHEM\1\DATA\1711\171123A\CR000005.D                    Sample Name: MBH_IV_172_ACN

    HILIC column  ACN. Flowinn.m Negative mode
    Sample dissolved in ACN

    ========================================================================
    Injection Date  : 11/23/2017 5:47:03 PM          Seq. Line :   5
    Sample Name     : MBH_IV_172_ACN                  Location : P1-D-01
    Acq. Operator   : Ed                                 Inj :   1
    Acq. Instrument : 1100LC                       Inj Volume : 1 µl
    Different Inj Volume from Sequence !     Actual Inj Volume : 2 µl
    Acq. Method     : C:\HPCHEM\1\METHODS\FLOWINN.M
    Last changed    : 11/15/2017 2:22:51 PM by Ed
    Analysis Method : C:\HPCHEM\1\METHODS\GEN_ANRR.M
    Last changed    : 2/2/2018 11:45:45 AM by Ed
                      (modified after loading)
    Agilent SB-C18 4.6*250mm, 5um; 0.6mL/min, 40'C
    M.P.A:0.1% AA/FA/TFA in H2O M.P.B:0.1% AA/FA/TFA in ACN
    Red Peek from pump -> AS and AS->column compartment
```



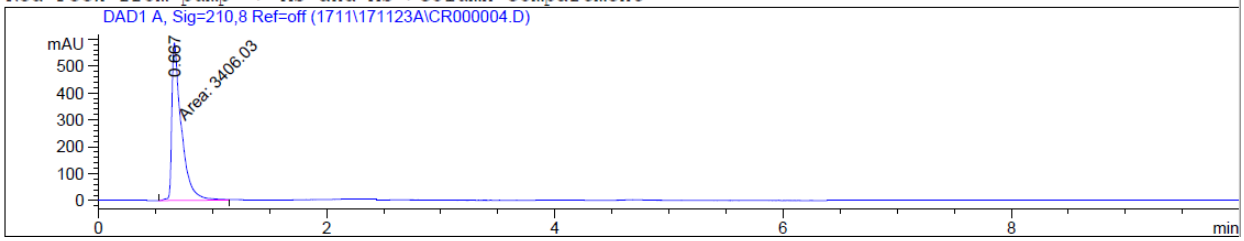


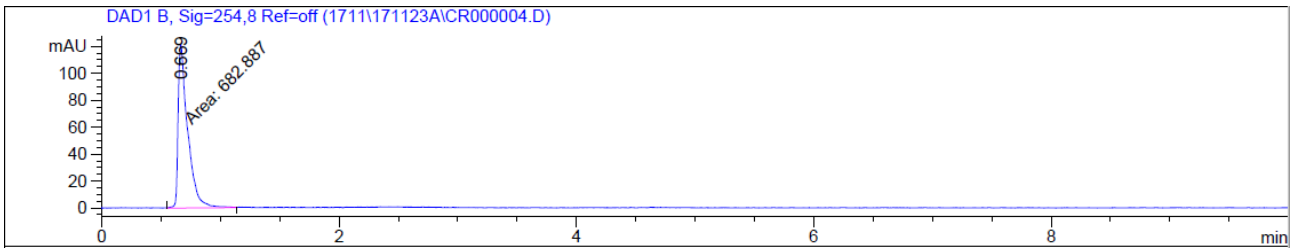


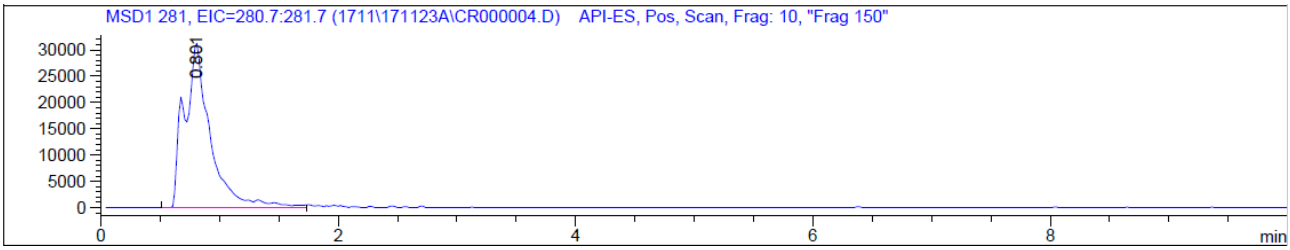
```
Data File C:\HPCHEM\1\DATA\1711\171123A\CR000004.D                Sample Name: MBH_IV_172_ACN

    HILIC column  ACN. Flowin.m Positive mode
    Sample dissolved in ACN

    ======================================================================
    Injection Date  : 11/23/2017 5:35:41 PM          Seq. Line :   4
    Sample Name     : MBH_IV_172_ACN                  Location : P1-D-01
    Acq. Operator   : Ed                                 Inj :   1
    Acq. Instrument : 1100LC                       Inj Volume : 1 µl
    Different Inj Volume from Sequence !     Actual Inj Volume : 2 µl
    Acq. Method     : C:\HPCHEM\1\METHODS\FLOWIN.M
    Last changed    : 11/22/2017 10:46:05 AM by Ed
    Analysis Method : C:\HPCHEM\1\METHODS\GEN_ANRR.M
    Last changed    : 2/1/2018 4:06:37 PM by Ed
                      (modified after loading)
    Agilent SB-C18 4.6*250mm, 5um; 0.6mL/min, 40'C
    M.P.A:0.1% AA/FA/TFA in H2O M.P.B:0.1% AA/FA/TFA in ACN
    Red Peek from pump -> AS and AS->column compartment
```







7 Biological assays

Assays were performed in the laboratory of the non-for-profit public organization Community for Antimicrobial Drug Discovery (CO-ADD), University of Queensland, Australia.

Antibacterial data collection

Inhibition of bacterial growth was determined measuring absorbance at 600 nm (OD600), using a Tecan M1000 Pro monochromator plate reader. The percentage of growth inhibition was calculated for each well, using the negative control (media only) and positive control (bacteria without inhibitors) on the same plate as references.

Antifungal data collection

Growth inhibition of C. albicans was determined measuring absorbance at 530 nm (OD530), while the growth inhibition of C. neoformans was determined measuring the difference in absorbance between 600 and 570 nm (OD600-570), after the addition of resazurin (0.001% final concentration) and incubation at 35 °C for additional 2 h. The absorbance was measured using a Biotek Synergy HTX plate reader. The percentage of growth inhibition was calculated for each well, using the negative control (media only) and positive control (bacteria without inhibitors) on the same plate as references.

Inhibition

Percentage growth inhibition of an individual sample is calculated based on Negative controls (media only) and Positive Controls (bacterial/fungal media without inhibitors). Please note negative inhibition values indicate that the growth rate (or OD600) is higher compared to the Negative Control (Bacteria/fungi only, set to 0% inhibition). The growth rates for all bacteria and fungi has a variation of -/+ 10%, which is within the reported normal distribution of bacterial/fungal growth. Any significant variation (or outliers/hits) is identified by the modified Z-Score, and actives are selected by a combination of inhibition value and Z-Score.

Z-Score

Z-Score analysis is done to investigate outliers or hits among the samples. The Z-Score is calculated based on the sample population using a modified Z-Score method which accounts for possible skewed sample population. The modified method uses median and MAD (median average derviation) instead of average and sd, and a scaling factor [Iglewicz, B. & Hoaglin, D. C. Volume 16: How to Detect and Handle Outliers. The ASQC Basic Reference in Quality Control: Statistical Techniques, 1993]: M(i) = 0.6745 * (x(i) - median(x))/MAD). M(i) values of > |2.5| (absolute) label outliers or hits.

Quality Control

All screening is performed as two replica (n=2), with both replicas on different assay plates, but from single plating and performed in a single screening experiment (microbial incubation). Each individual value is reported in the table (see ..1 and ..2). In addition, two values are used as quality controls for individual plates: Z'-Factor [1-(3 * (sd(NegCtrl)+sd(PosCtrl))/(average(PosCtrl)-average(NegCtrl)))] and Standard Antibiotic controls at different concentrations (>MIC and < MIC). The plate passes the quality control if Z'-Factor >0.4 and Standards are active and inactive at highest and lowest concentrations, respectively.

Antibiotic standards preparation and Quality control

Colistin and Vancomycin were used as positive bacterial inhibitor standards for Gram-negative and Gram-positive bacteria, respectively (see MIC values on following page). Fluconazole was used as a positive fungal inhibitor standard for C. albicans and C. neoformans. The antibiotics were provided in 4 concentrations, with 2 above and 2 below its MIC value, and plated into the first 8 wells of column 23 of the 384-well NBS plates. The quality control (QC) of the assays was determined by the antimicrobial controls and the Z'-factor (using positive and negative controls). Each plate was deemed to fulfill the quality criteria (pass QC), if the Z'-factor was above 0.4, and the antimicrobial standards showed full range of activity, with full growth inhibition at their highest concentration, and no growth inhibition at their lowest concentration.

Primary Screening Panel
Strain Susceptibility Profiles

CO-ADD Community for Open Antimicrobial Drug Dis

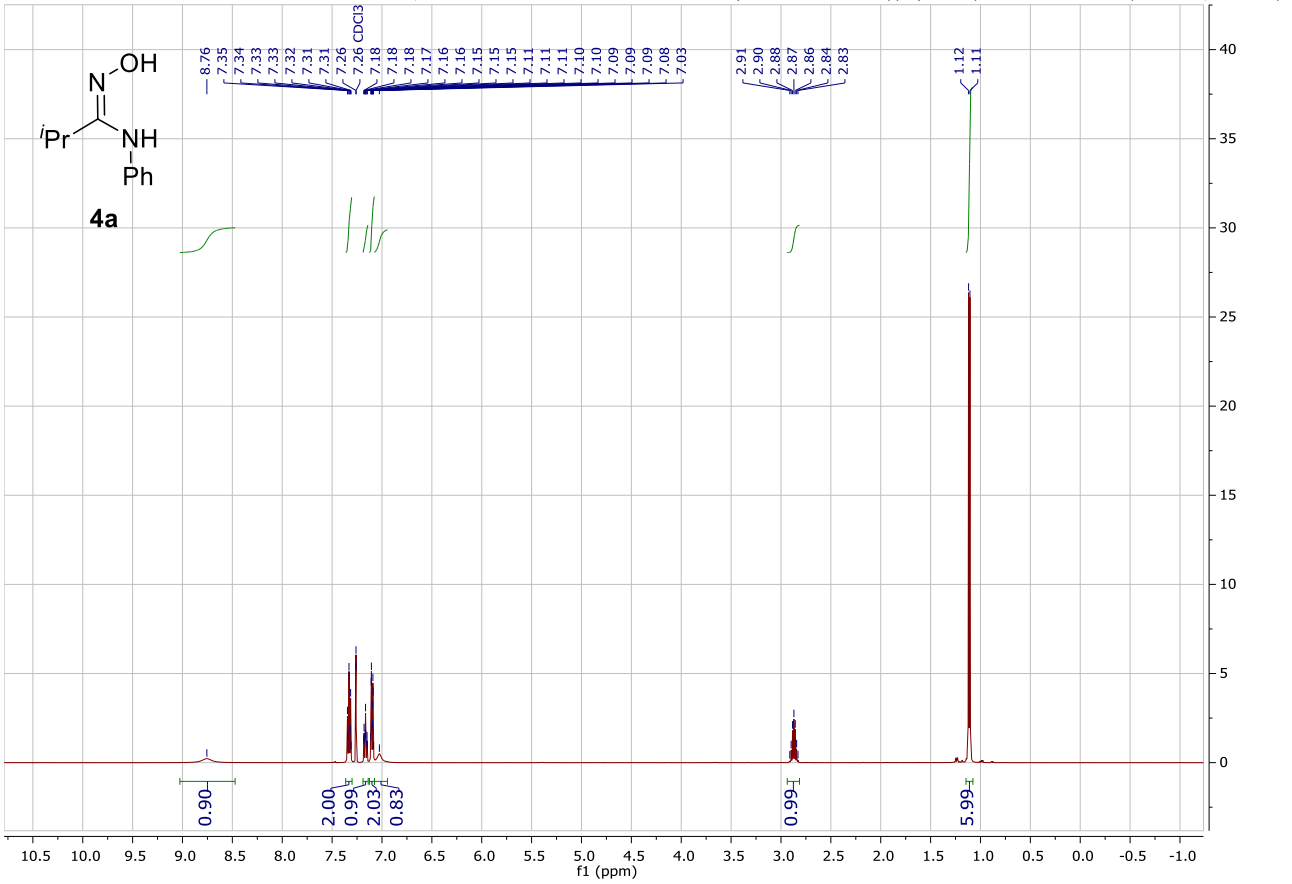
MIC [µg/mL] determined in BMD method; CA-MHB, Corning 3640 384-w NBS plates		GP_020_2 *Staphylococcus aureus* ATCC 43300 MRSA	GN_001_2 *Escherichia coli* ATCC 25922 FDA control	GN_003_2 *Klebsiella pneumoniae* ATCC 700603 ESBL	GN_034_2 *Acinetobacter baumannii* ATCC 19606 Type strain	GN_042_2 *Pseudomonas aeruginosa* ATCC 27853 QC control strain	FG_001_2 *Candida albicans* ATCC 90028 CLSI reference	FG_002_2 *Cryptococcus neoformans H99* ATCC 208821 Type strain
CompoundID	Compound type							
Amoxicillin	antibiotic	16	8	>32	>32	>32		
Ciprofloxacin	antibiotic	0.25	0.007	0.5	1	0.25		
Ampicillin	antibiotic	32	8	>32	>32	>32		
Colistin	antibiotic	>32	0.125	0.25	0.25	0.25		
Azithromycin	antibiotic	>32	4	32	32	32		
Dalbavancin	antibiotic	0.25	>32	>32	>32	>32		
Daptomycin	antibiotic	2	>32	>32	>32	>32		
Cefepime	antibiotic	16	0.125	2	16	2		
Cefotaxime	antibiotic	16	0.125	16	16	32		
Ceftazidime	antibiotic	>32	2	>32	32	4		
Gentamicin	antibiotic	>32	1	8	8	1		
Ceftriaxone	antibiotic	32	0.125	16	32	32		
Amikacin	antibiotic	16	2	1	16	4		
Linezolid	antibiotic	4	>32	>32	>32	>32		
Cephalothin	antibiotic	4	16	>32	>32	>32		
Meropenem	antibiotic	4	0.06	0.125	2	1		
Chloramphenicol	antibiotic	8	8	>32	>32	>32		
Oxacillin	antibiotic	4	>32	>32	>32	>32		
Tobramycin	antibiotic	>32	0.5	0.5	0.25	0.5		
Vancomycin	antibiotic	1	>32	>32	>32	>32		
Tetracycline	antibiotic	0.25	1	16	2	32		
Trimethoprim	antibiotic	1	0.5	2	16	>32		
Piperacillin	antibiotic		1	>32	≥32	16		
Aztreonam	antibiotic		0.06	≥32	16	16		
Ceftaroline	antibiotic	1	0.06					
Erythromycin	antibiotic		>32					
Streptomycin	antibiotic		8					
Oritavancin	antibiotic	≤0.016						
Telavancin	antibiotic	0.125						
Chloroeremomycin	antibiotic	0.25						
Amphotericin B	antifungal						1.56	1.56
Fluconazole	antifungal						0.125	8
5-Fluorocytosine	antifungal						0.06	0.03
Posaconazole	antifungal						≤0.06	≤0.02
Voriconazole	antifungal						0.002	0.03
Ketoconazole	antifungal						0.125	2
Itraconazole	antifungal						≤0.001	0.03
Caspofungin	antifungal						≤0.0009	8
Values are the mode of ≥6 independent biological replicates. All values are within the expected range as per CLSI guidelines								

Toxicity assessment for compounds for compounds 6c and 6e

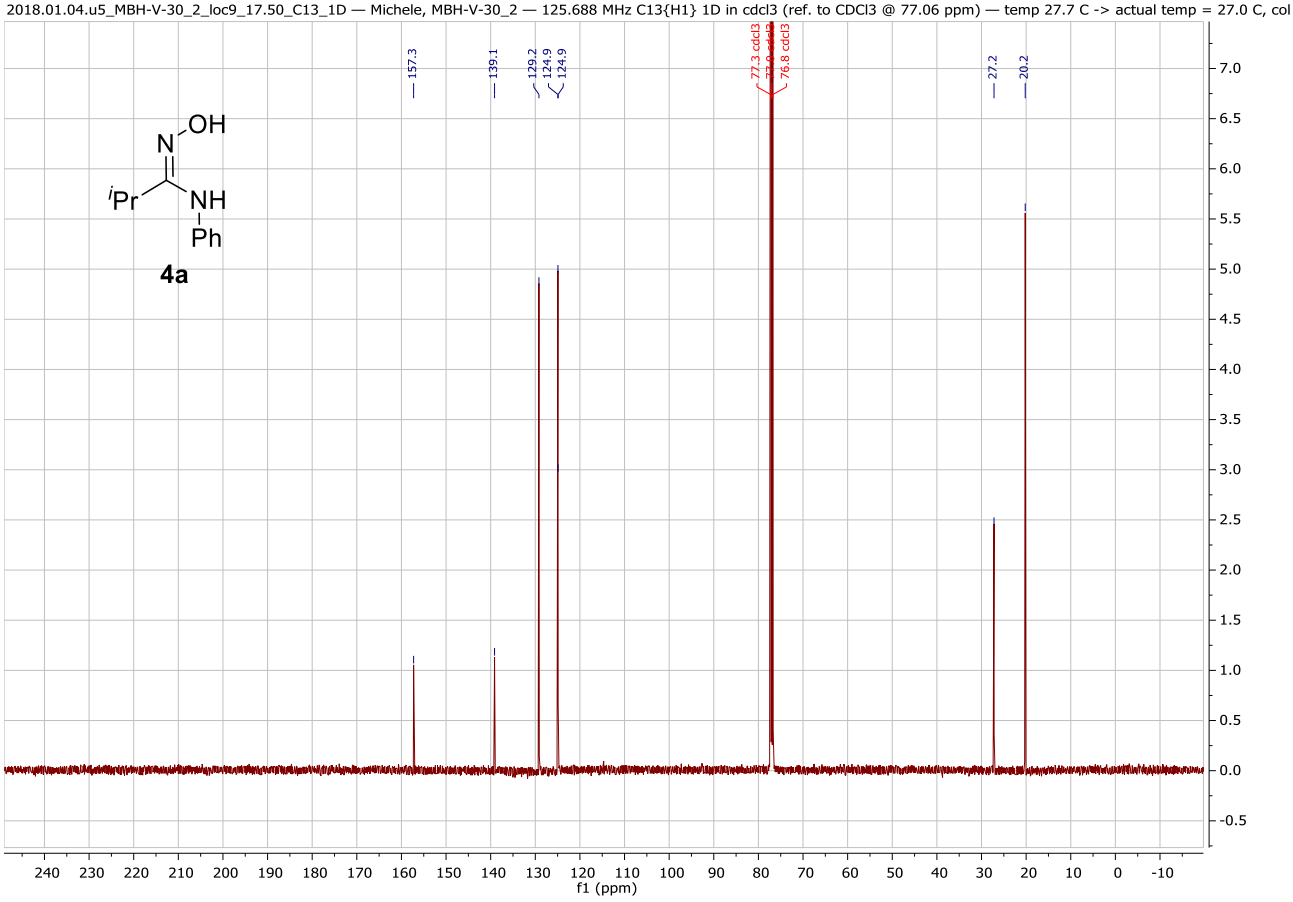
Growth inhibition of HEK293 cells was determined measuring fluorescence at ex:530/10 nm and em: 590/10 nm (F560/590), after the addition of resazurin (25 ug/mL final concentration) and incubation at 37 °C and 5% CO_2, for additional 3 h. The fluorescence was measured using a Tecan M1000 Pro monochromator plate reader. The percentage of growth inhibition was calculated for each well, using the Negative Control (media only) and Positive Control (cell culture without inhibitors) on the same plate as references. No toxicity was observed at 50 ug/mL.

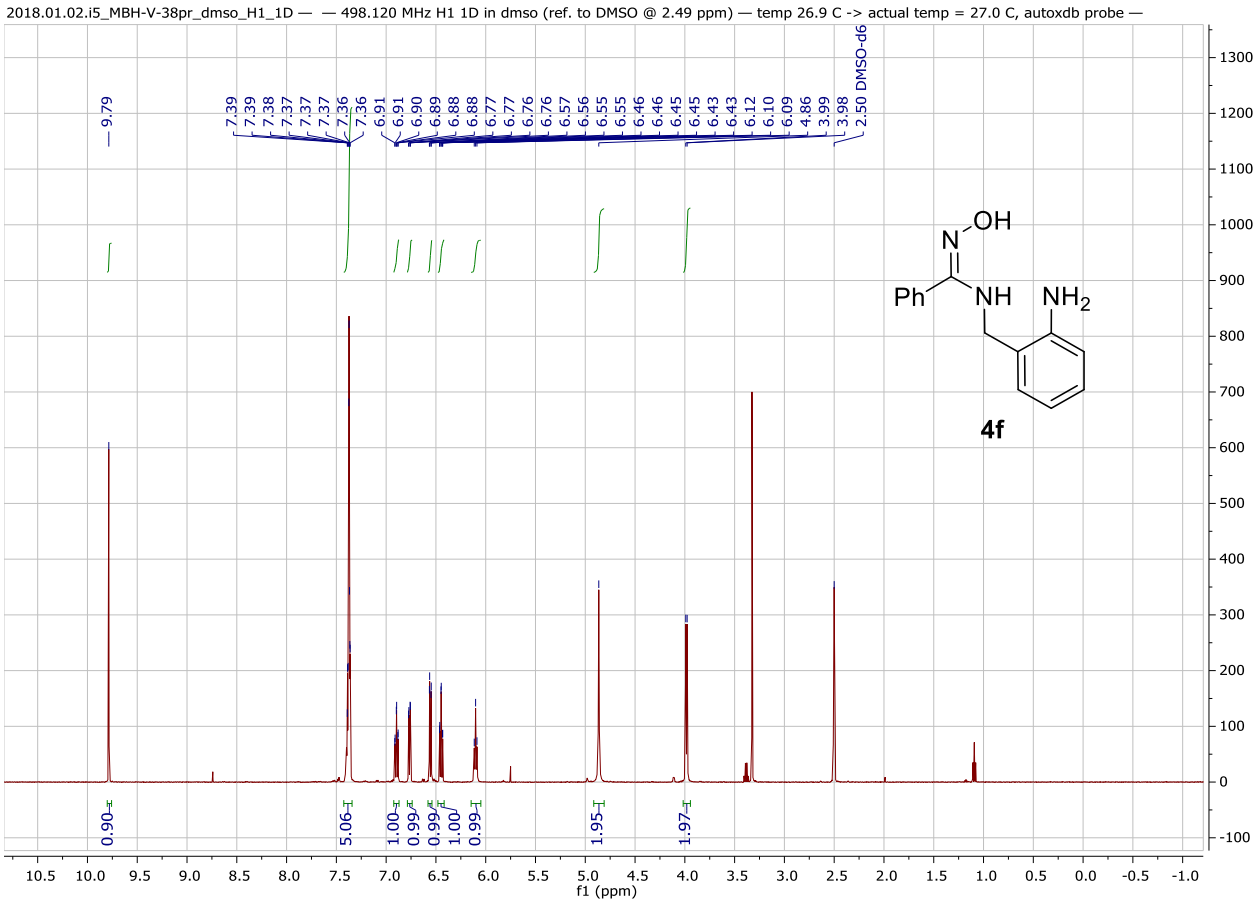
8 NMR spectra

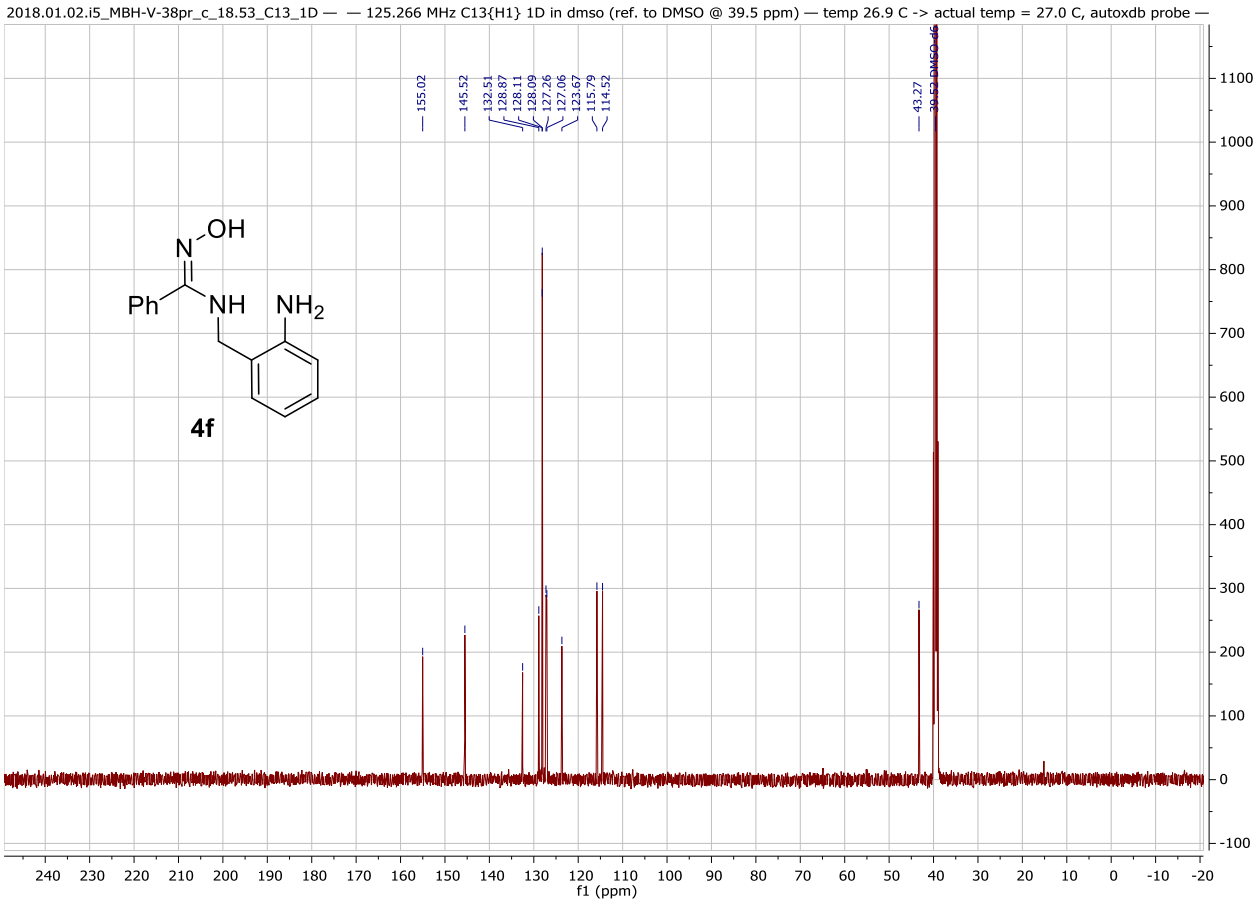
2018.01.04.u5_MBH-V-30_2_loc9_17.50_H1_1D — Michele, MBH-V-30_2 — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddual pr

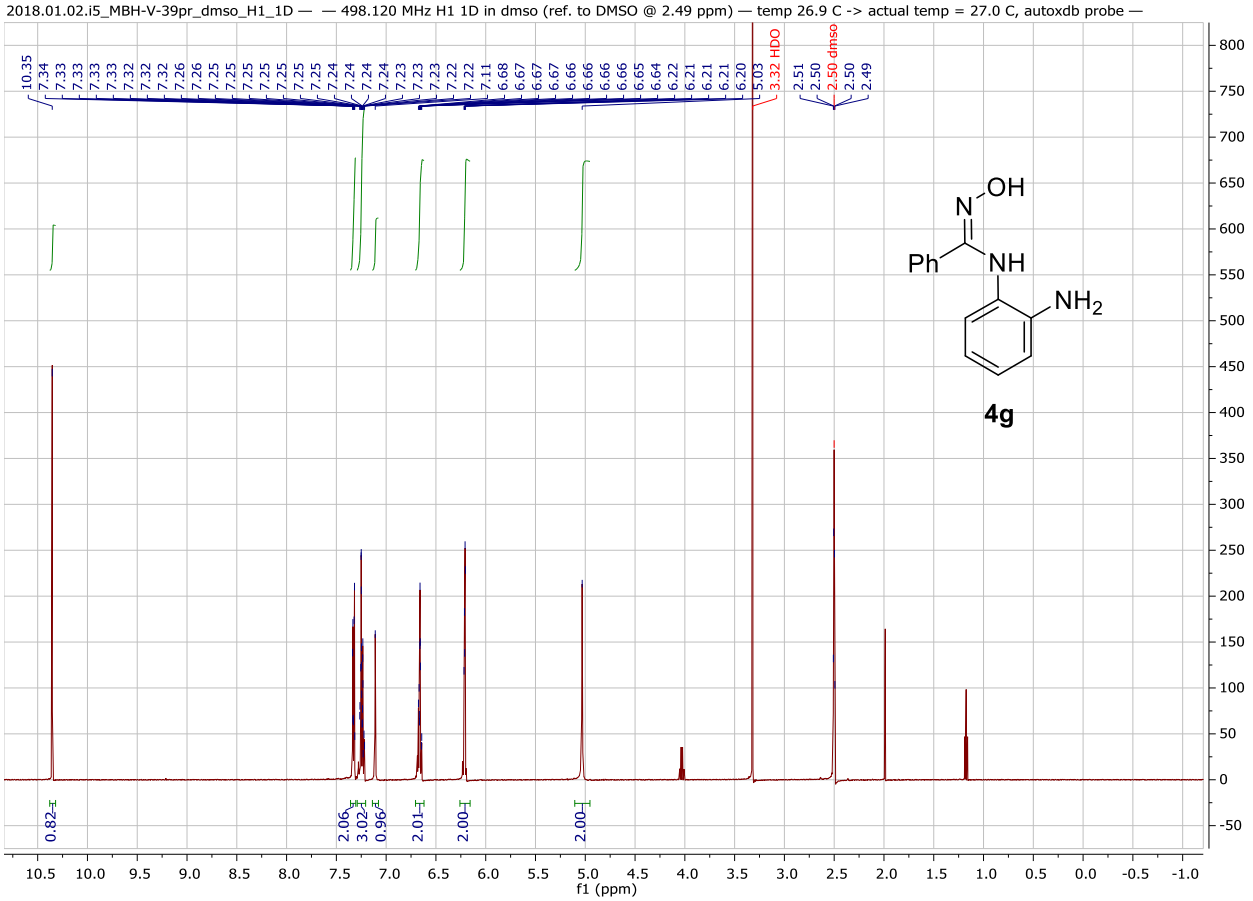


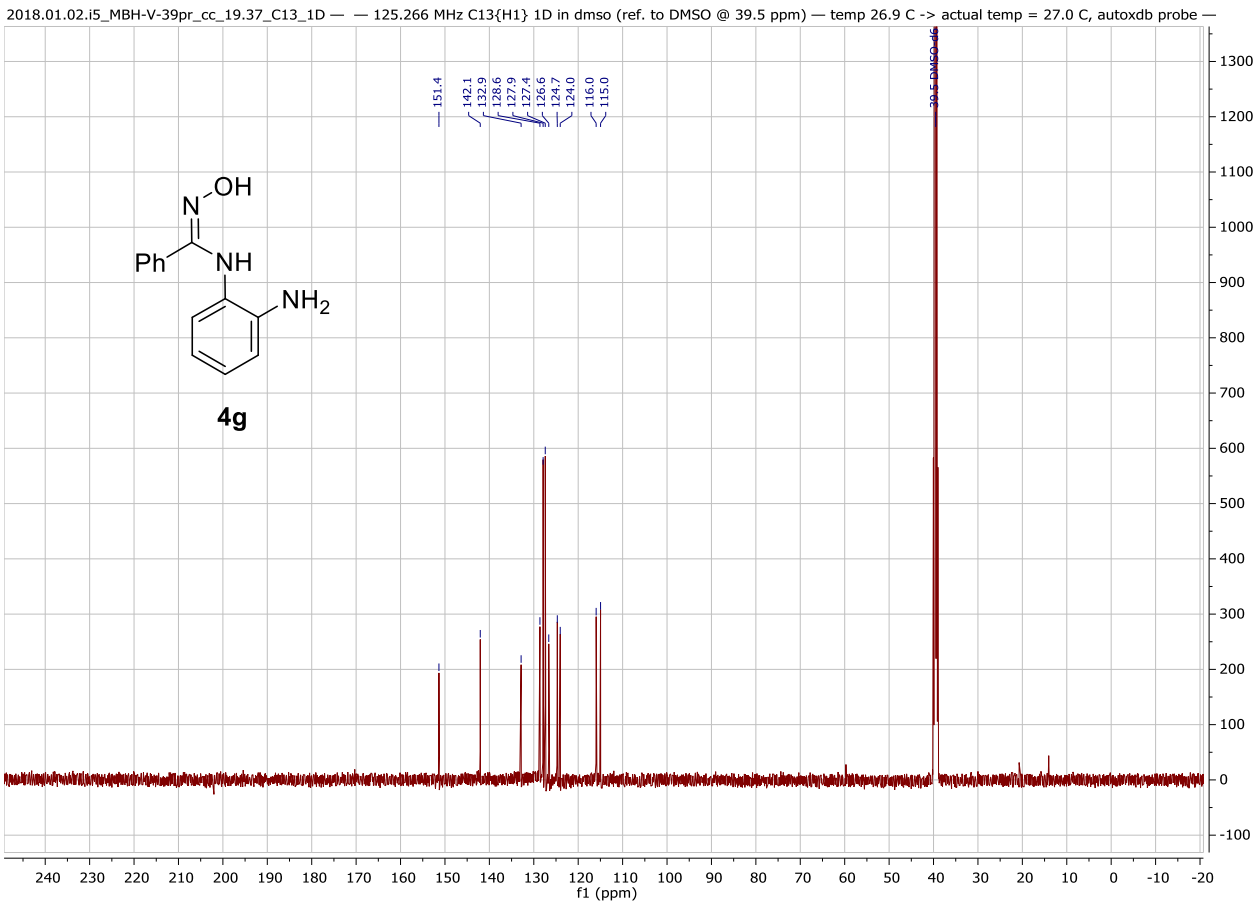
2018.01.04.u5_MBH-V-30_2_loc9_17.50_C13_1D — Michele, MBH-V-30_2 — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C, col

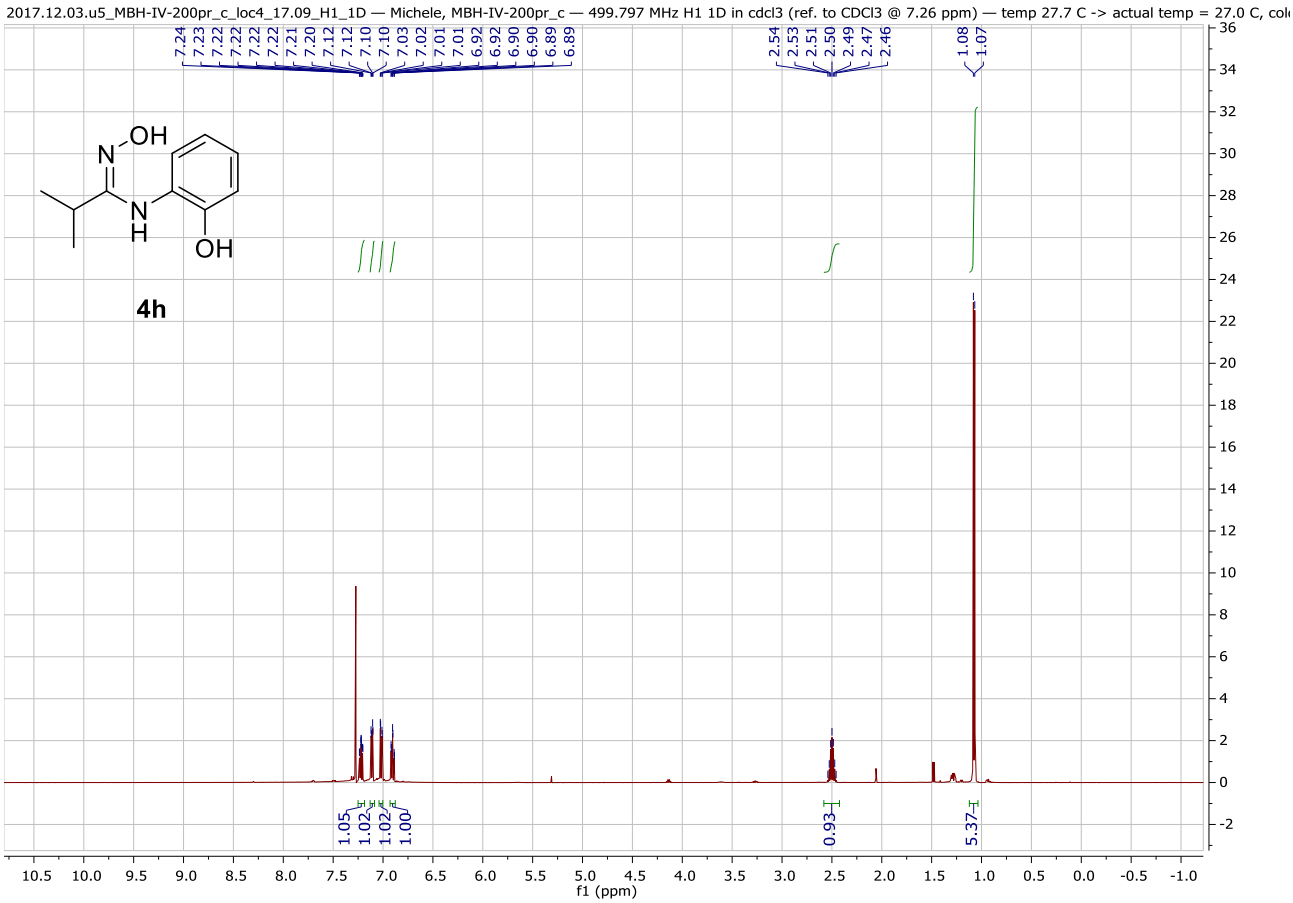


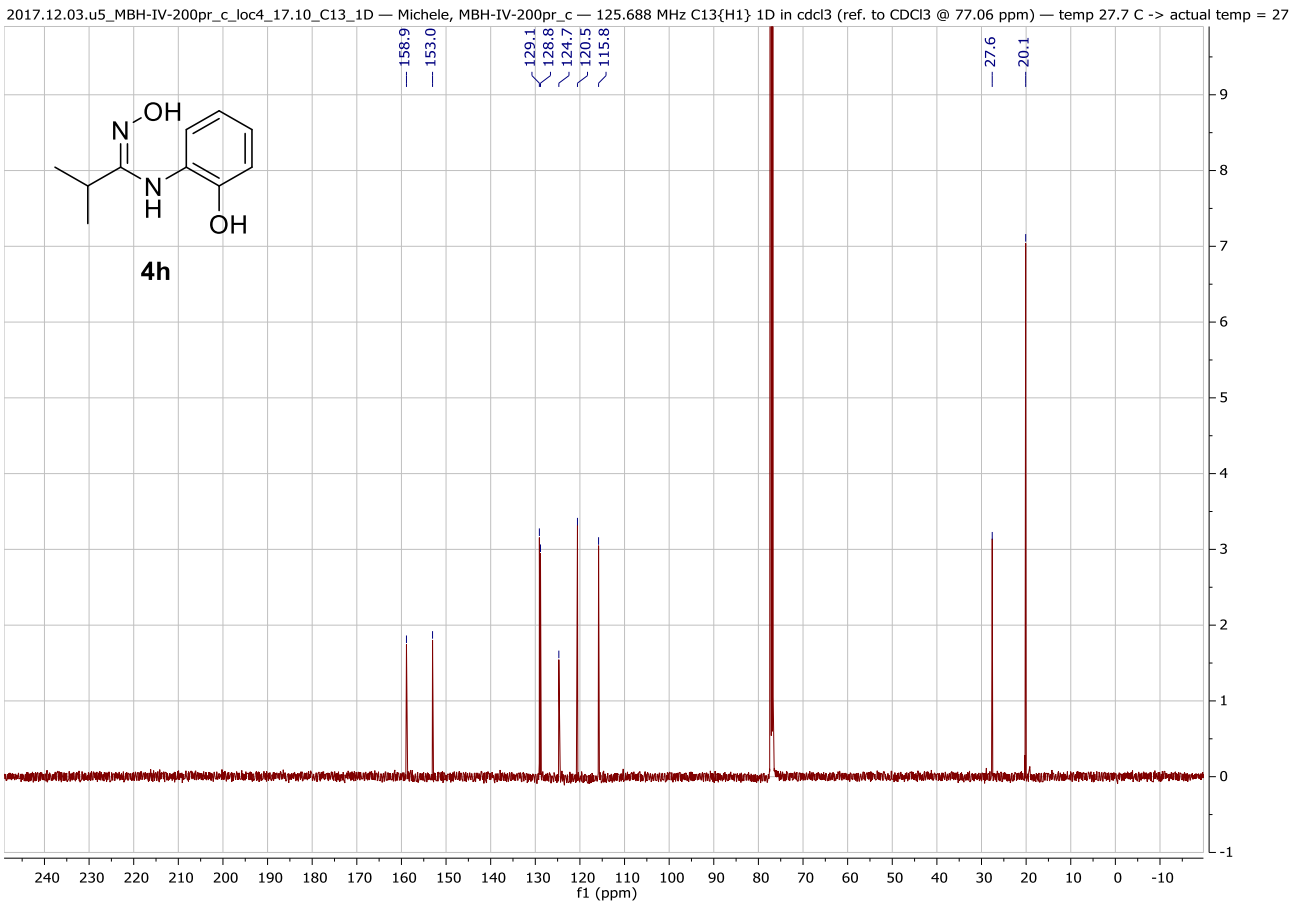

2018.01.02.i5_MBH-V-38pr_dmso_H1_1D — — 498.120 MHz H1 1D in dmso (ref. to DMSO @ 2.49 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
9.79
7.39
7.39
7.38
7.37
7.37
7.37
7.36
7.36
6.91
6.91
6.90
6.89
6.88
6.88
6.77
6.77
6.76
6.76
6.57
6.56
6.55
6.55
6.46
6.46
6.45
6.45
6.43
6.43
6.12
6.10
6.09
4.86
3.99
3.98
2.50 DMSO-d6
OH
N
Ph
NH
NH2
4f
0.90
5.06
1.00
0.99
0.99
1.00
0.99
1.95
1.97
f1 (ppm)

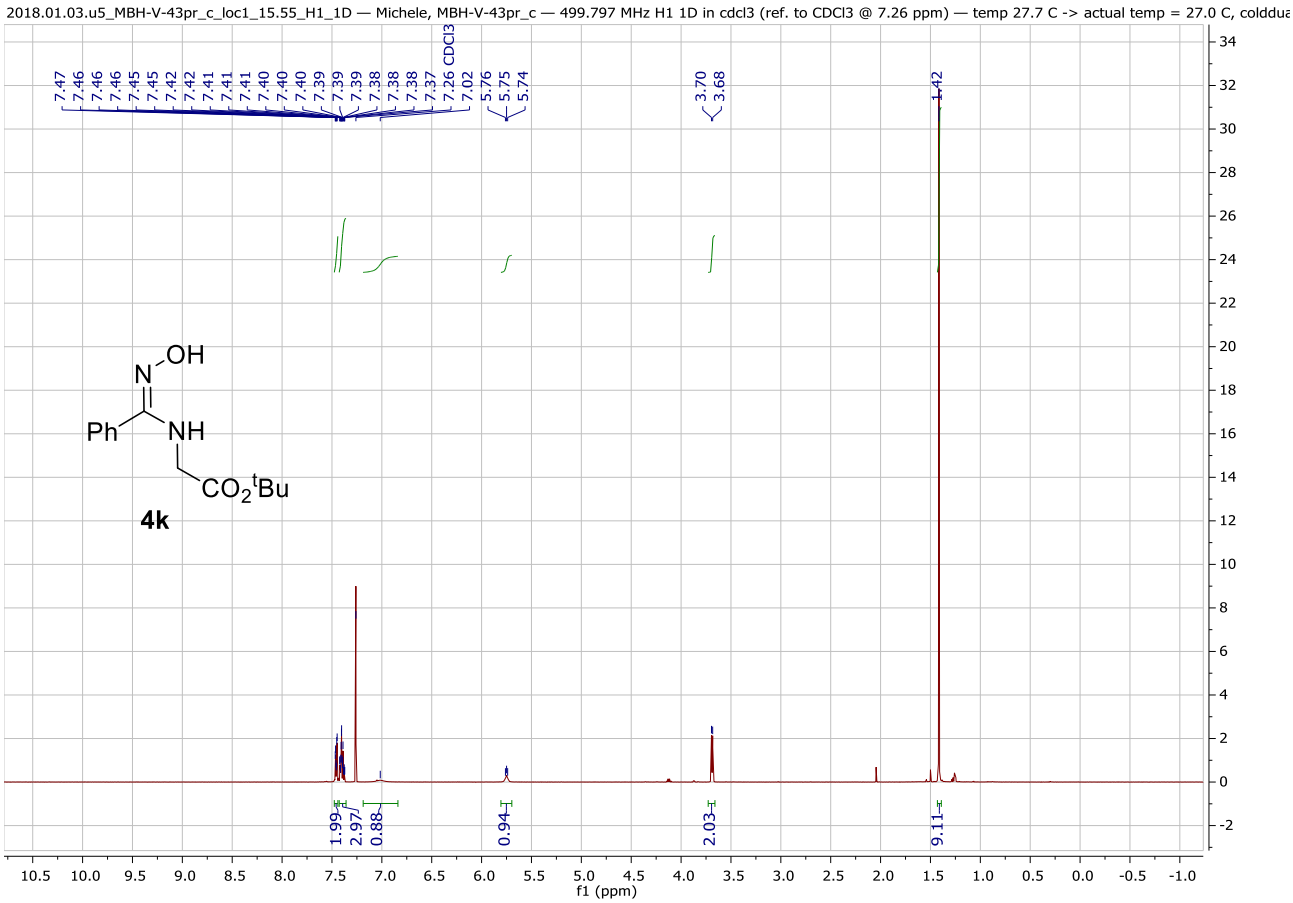

2018.01.02.i5_MBH-V-38pr_c_18.53_C13_1D — — 125.266 MHz C13{H1} 1D in dmso (ref. to DMSO @ 39.5 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
155.02
145.52
132.51
128.87
128.11
128.09
127.26
127.06
123.67
115.79
114.52
43.27
39.52 DMSO-d6
OH
N
Ph
NH
NH2
4f
f1 (ppm)

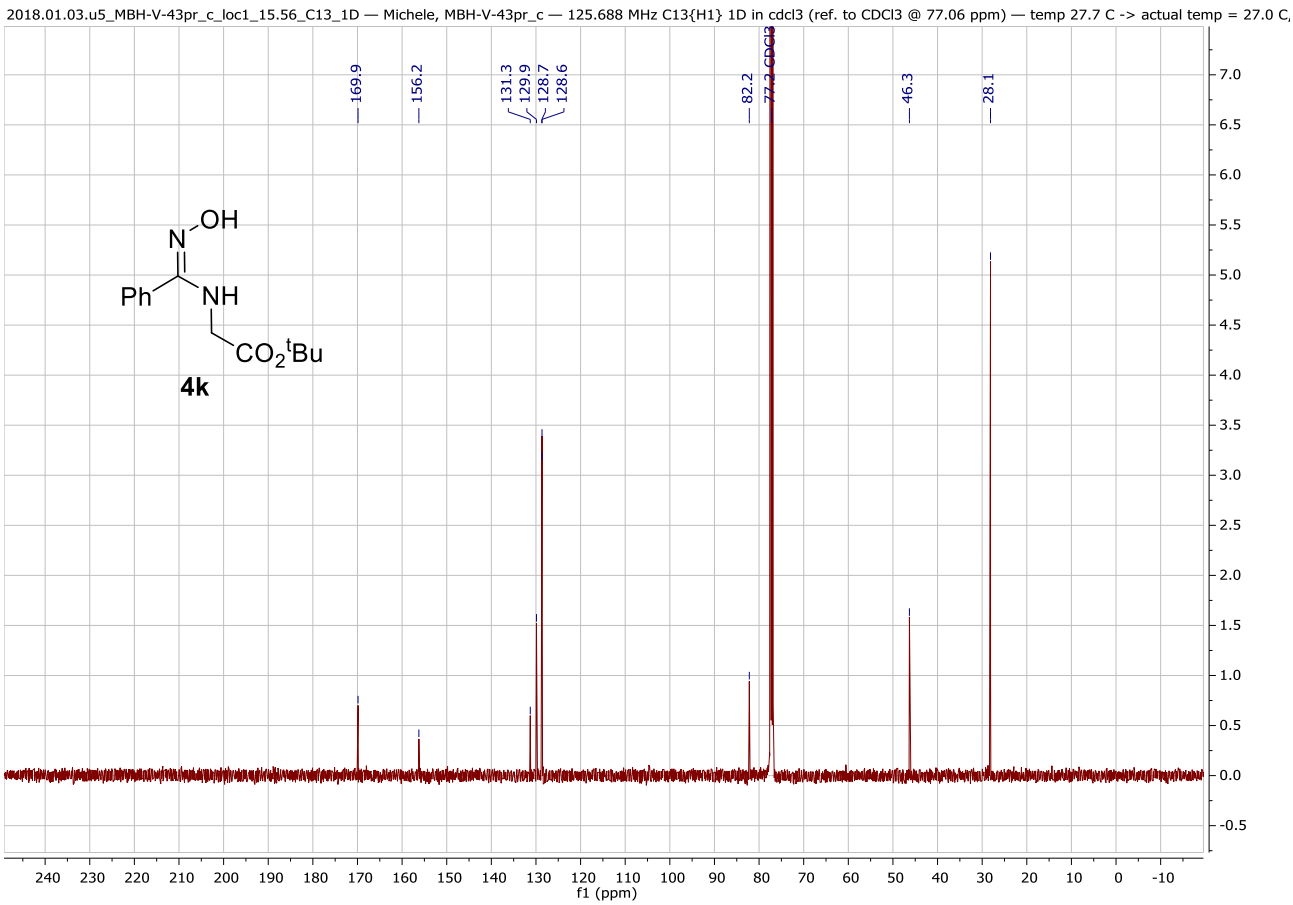

2018.01.02.i5_MBH-V-39pr_dmso_H1_1D — — 498.120 MHz H1 1D in dmso (ref. to DMSO @ 2.49 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
10.35
7.34
7.33
7.33
7.33
7.33
7.32
7.32
7.32
7.26
7.26
7.25
7.25
7.25
7.25
7.25
7.25
7.25
7.24
7.24
7.24
7.24
7.23
7.23
7.23
7.22
7.22
7.11
6.68
6.67
6.67
6.67
6.66
6.66
6.66
6.66
6.65
6.64
6.22
6.21
6.21
6.21
6.20
5.03
3.32 HDO
2.51
2.50
2.50 dmso
2.50
2.49
0.82
2.06
3.02
0.96
2.01
2.00
2.00
f1 (ppm)
OH
N
Ph
NH
NH2
4g

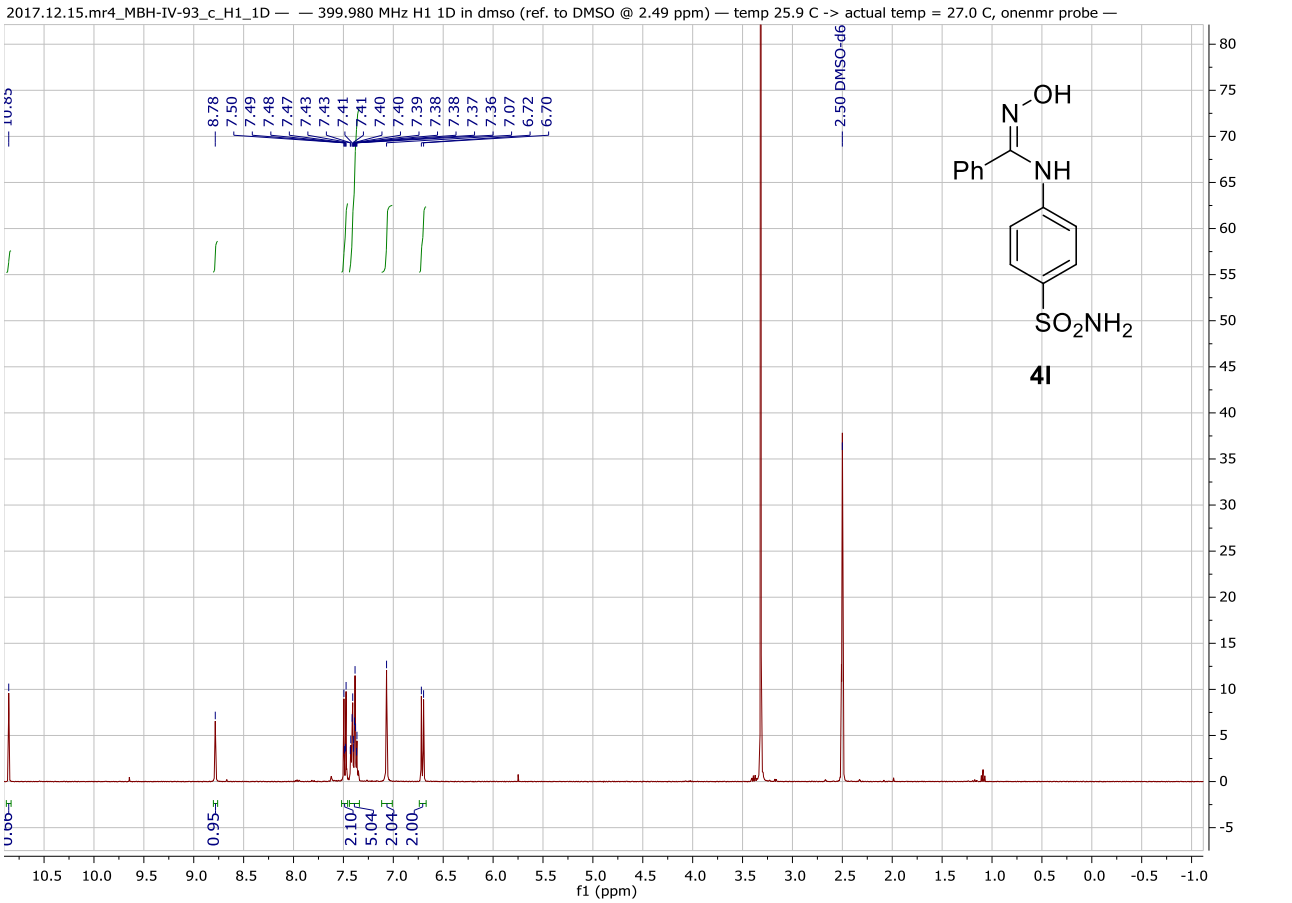

2018.01.02.i5_MBH-V-39pr_cc_19.37_C13_1D — — 125.266 MHz C13{H1} 1D in dmso (ref. to DMSO @ 39.5 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
151.4
142.1
132.9
128.6
127.9
127.4
126.6
124.7
124.0
116.0
115.0
39.5 DMSO-d6
f1 (ppm)
OH
N
Ph
NH
NH2
4g

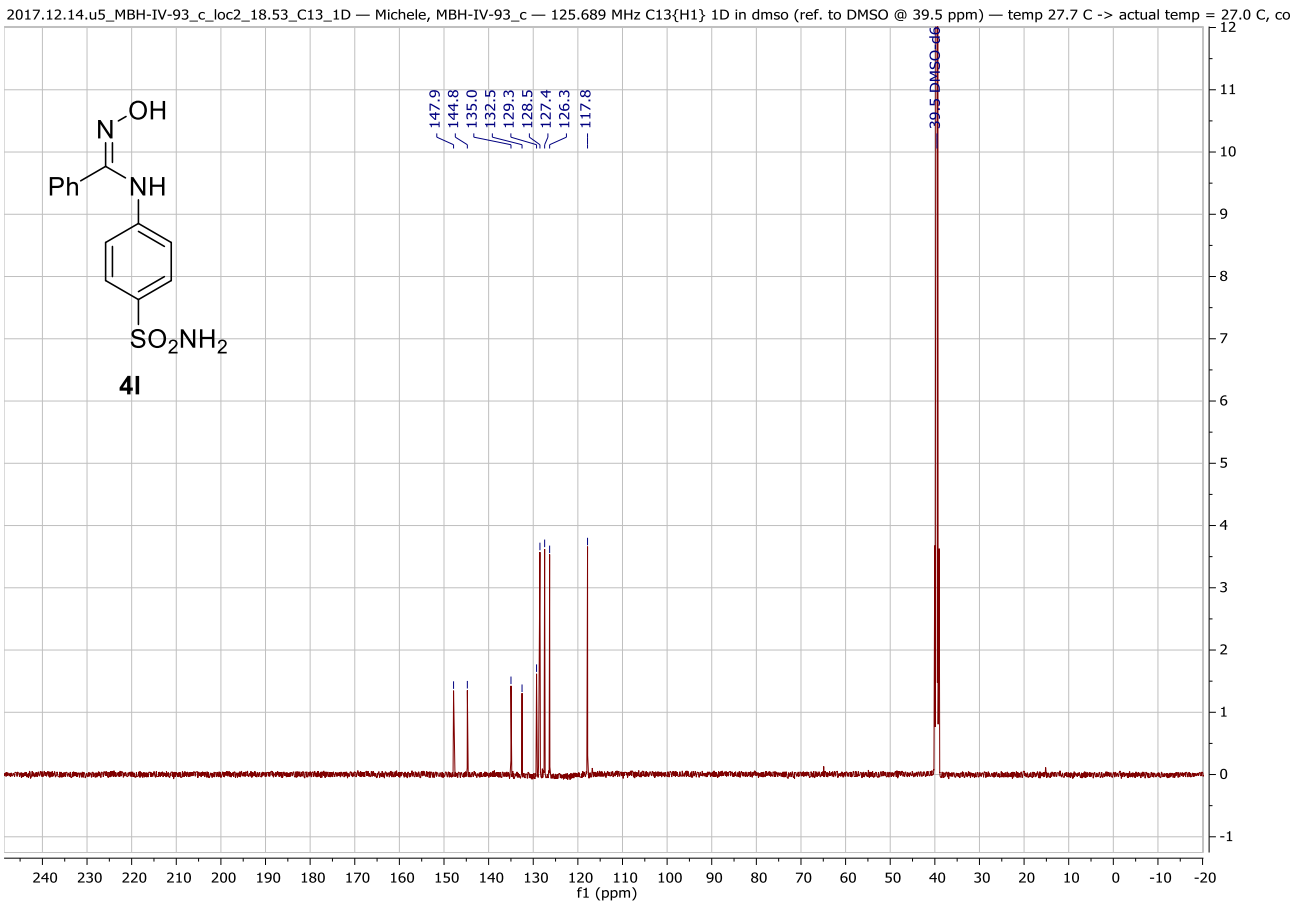

2017.12.03.u5_MBH-IV-200pr_c_loc4_17.09_H1_1D — Michele, MBH-IV-200pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, col
7.24 7.23 7.22 7.22 7.22 7.22 7.21 7.20 7.12 7.12 7.10 7.10 7.03 7.02 7.01 7.01 6.92 6.92 6.90 6.90 6.89 6.89
2.54 2.53 2.51 2.50 2.49 2.47 2.46
1.08 1.07
OH
N
N
H
OH
4h
1.05 1.02 1.02 1.00
0.93
5.37
f1 (ppm)

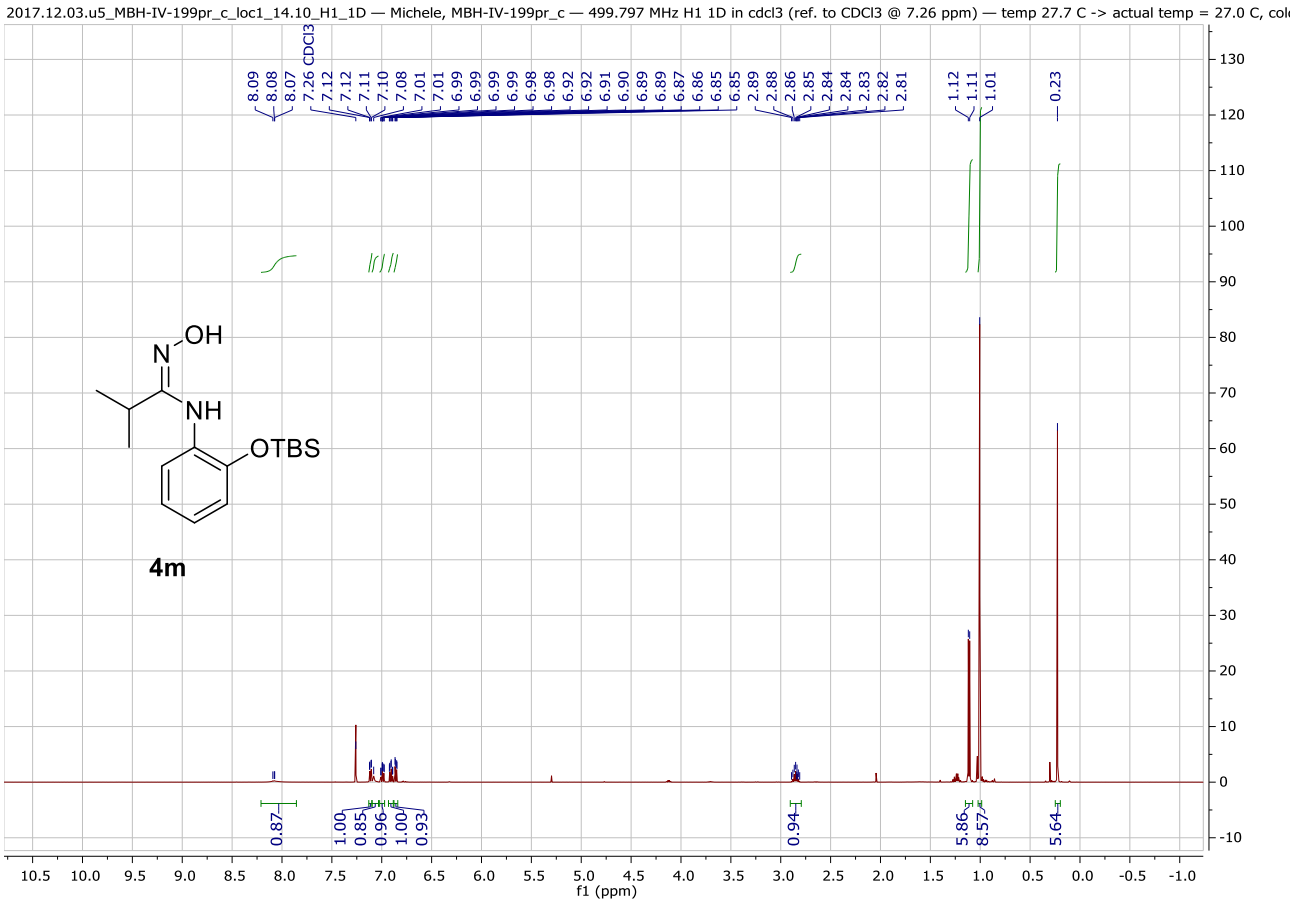

2017.12.03.u5_MBH-IV-200pr_c_loc4_17.10_C13_1D — Michele, MBH-IV-200pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27
158.9 153.0
129.1 128.8 124.7 120.5 115.8
27.6 20.1
OH
N
N
H
OH
4h
f1 (ppm)

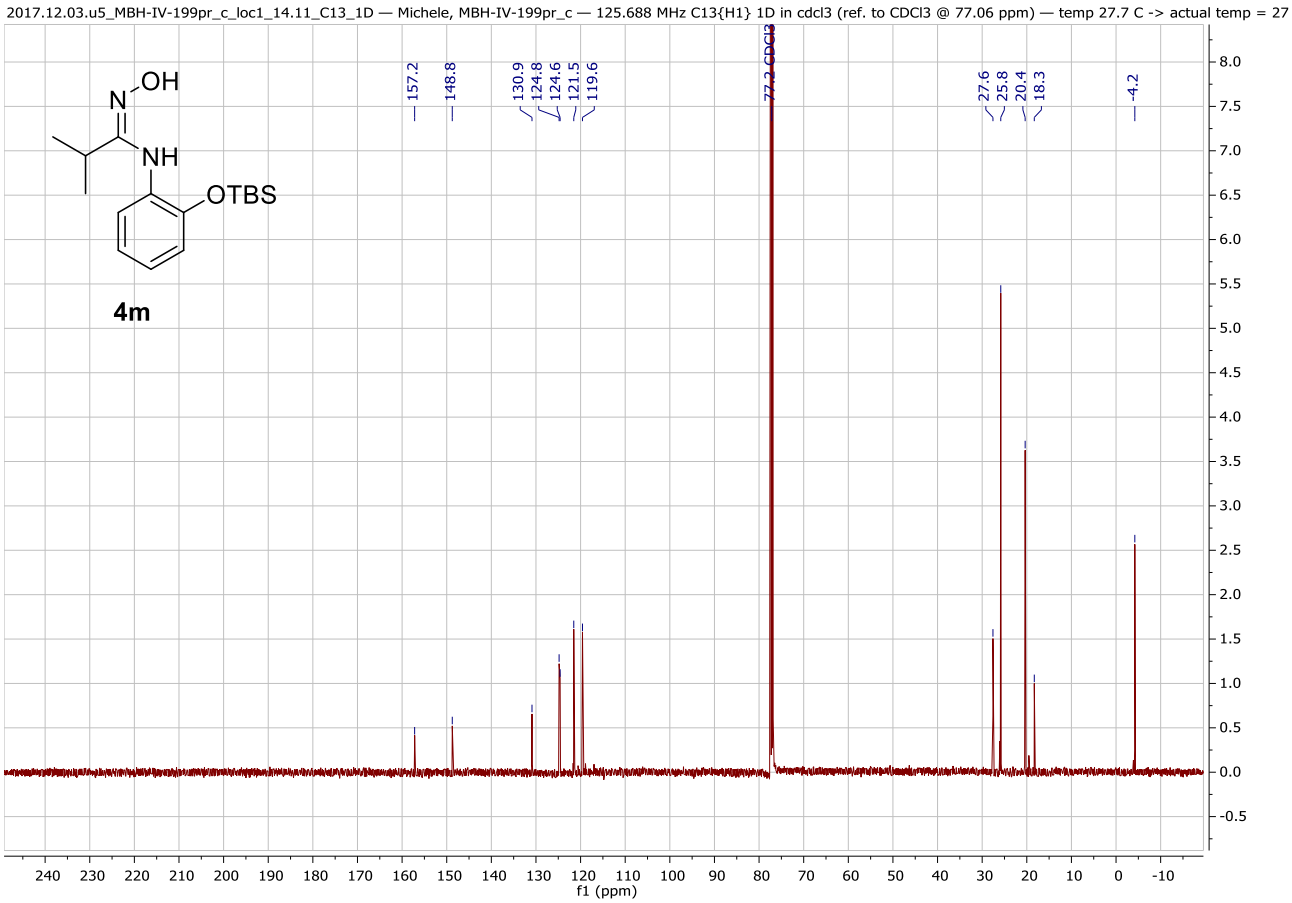

2018.01.03.u5_MBH-V-43pr_c_loc1_15.55_H1_1D — Michele, MBH-V-43pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddua
7.47 7.46 7.46 7.46 7.45 7.45 7.42 7.42 7.41 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.39 7.38 7.38 7.38 7.37 7.26 CDCl3 7.02
5.76 5.75 5.74
3.70 3.68
1.42
OH
N
Ph
NH
CO2tBu
4k
1.99 2.97 0.88 0.94 2.03 9.11
f1 (ppm)


2018.01.03.u5_MBH-V-43pr_c_loc1_15.56_C13_1D — Michele, MBH-V-43pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C,
169.9
156.2
131.3 129.9 128.7 128.6
82.2
77.2 CDCl3
46.3
28.1
OH
N
Ph
NH
CO2tBu
4k
f1 (ppm)

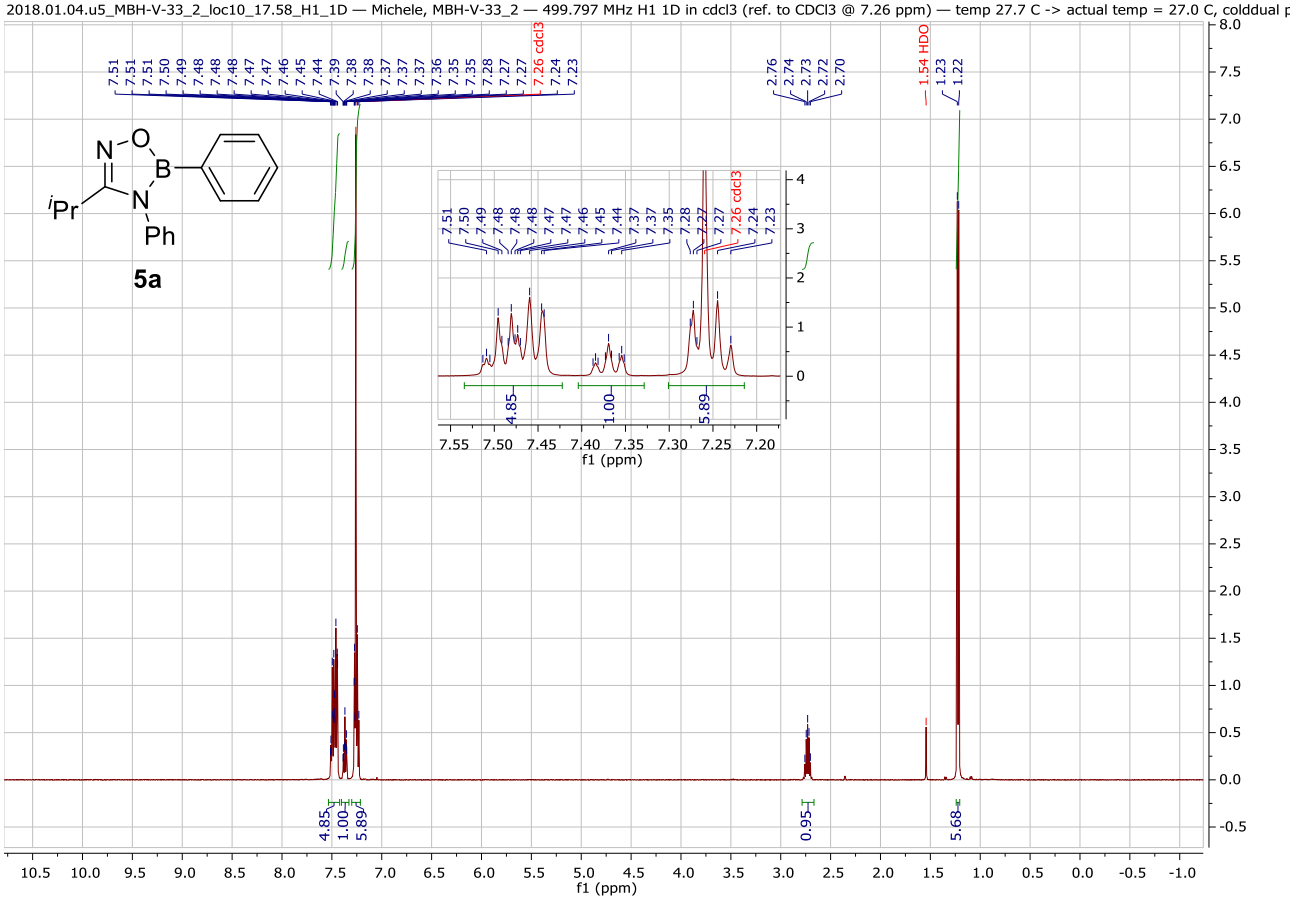

2017.12.15.mr4_MBH-IV-93_c_H1_1D — — 399.980 MHz H1 1D in dmso (ref. to DMSO @ 2.49 ppm) — temp 25.9 C -> actual temp = 27.0 C, onenmr probe —
10.85
8.78
7.50
7.49
7.48
7.47
7.43
7.43
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.37
7.36
7.07
6.72
6.70
2.50 DMSO-d6
OH
N
Ph
NH
SO2NH2
4l
0.95
2.10
5.04
2.04
2.00
f1 (ppm)


2017.12.14.u5_MBH-IV-93_c_loc2_18.53_C13_1D — Michele, MBH-IV-93_c — 125.689 MHz C13{H1} 1D in dmso (ref. to DMSO @ 39.5 ppm) — temp 27.7 C -> actual temp = 27.0 C, co
147.9
144.8
135.0
132.5
129.3
128.5
127.4
126.3
117.8
39.5 DMSO-d6
OH
N
Ph
NH
SO2NH2
4l
f1 (ppm)

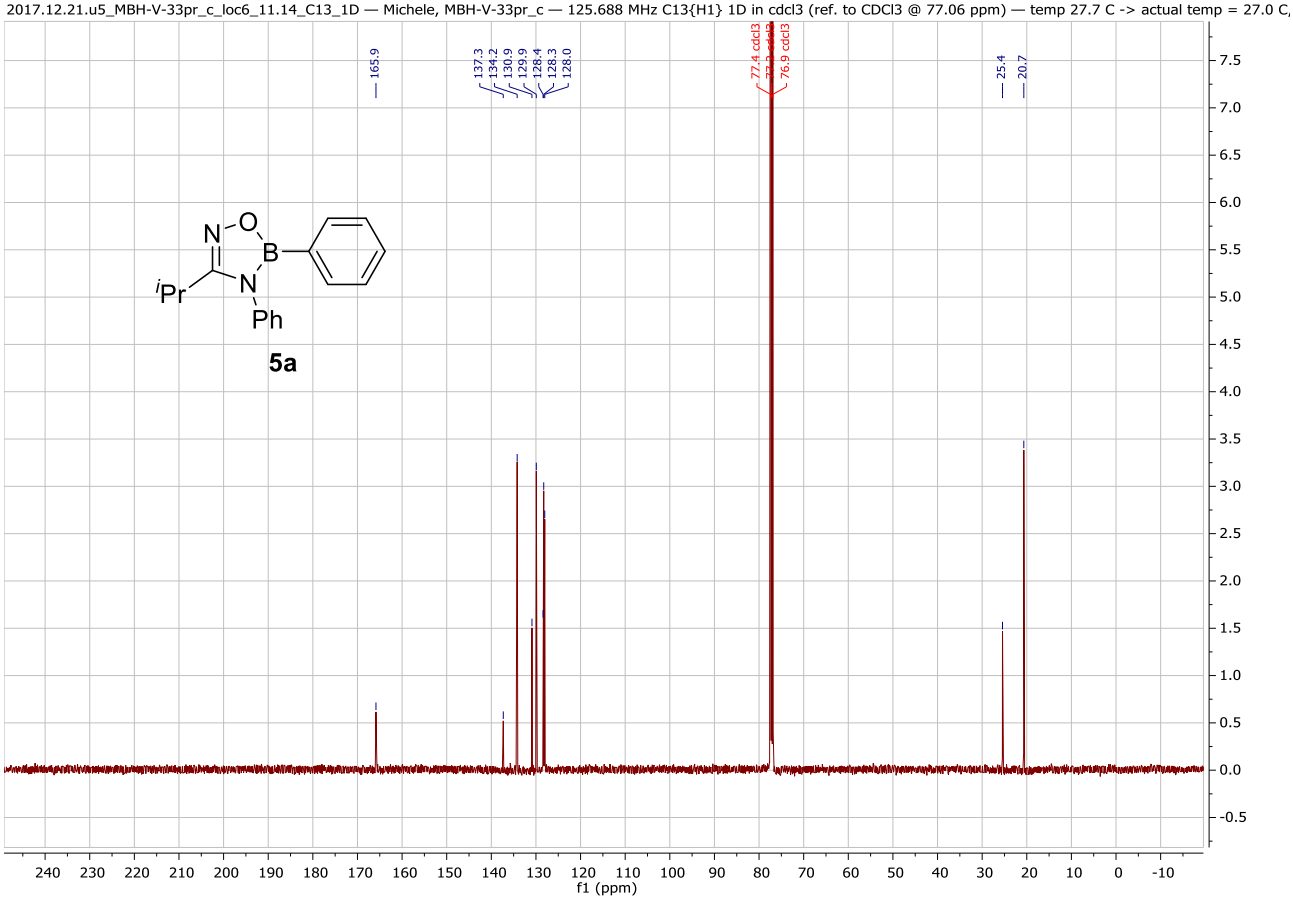

2017.12.03.u5_MBH-IV-199pr_c_loc1_14.10_H1_1D — Michele, MBH-IV-199pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, col
CDCl3
8.09 8.08 8.07 7.26 7.12 7.12 7.11 7.10 7.08 7.01 7.01 6.99 6.99 6.99 6.99 6.98 6.98 6.92 6.92 6.91 6.90 6.89 6.89 6.87 6.86 6.85 6.85 2.89 2.88 2.86 2.85 2.84 2.84 2.83 2.82 2.81 1.12 1.11 1.01 0.23
OH
N
NH
OTBS
4m
0.87 1.00 0.85 0.96 1.00 0.93 0.94 5.86 8.57 5.64
f1 (ppm)

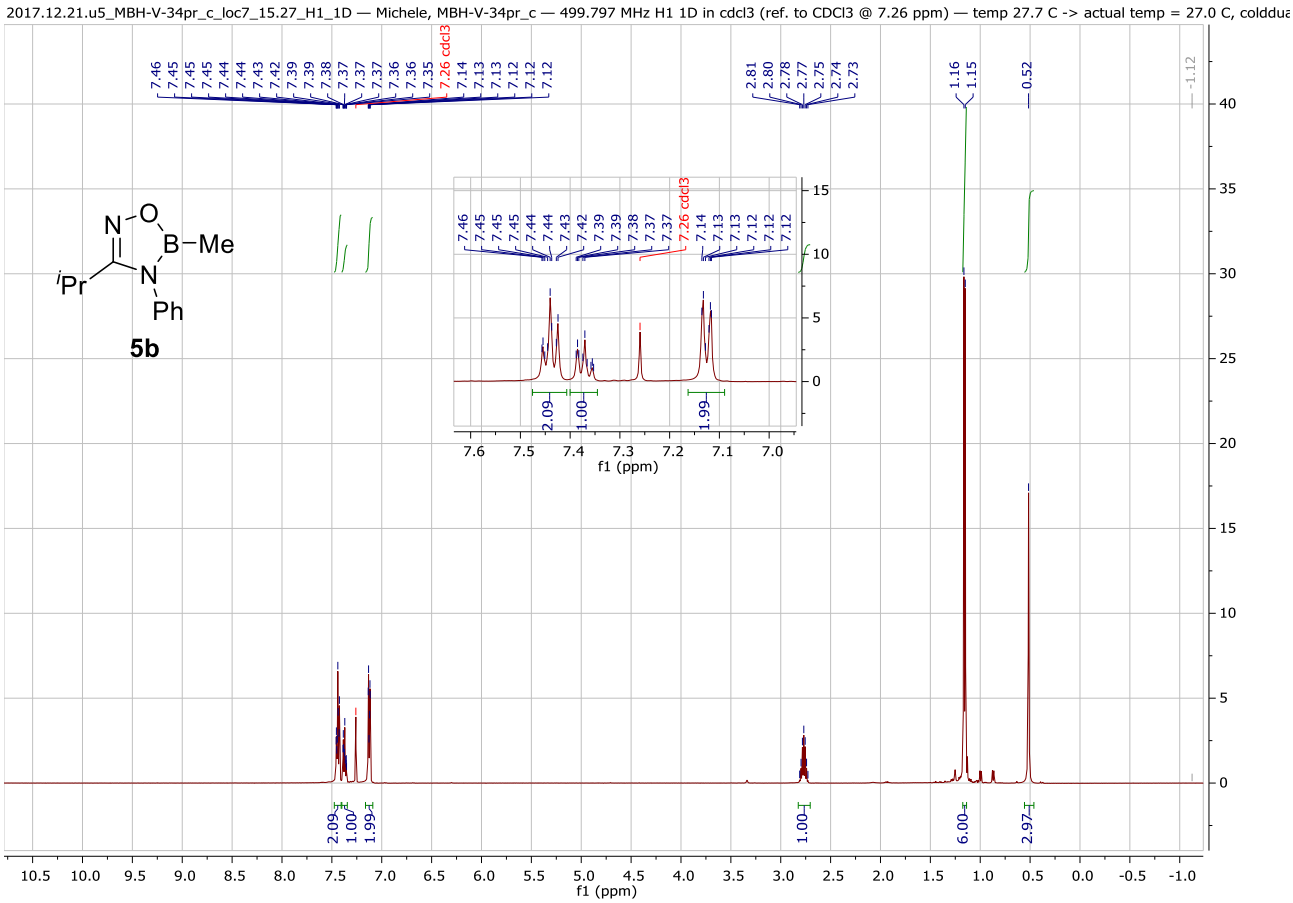

2017.12.03.u5_MBH-IV-199pr_c_loc1_14.11_C13_1D — Michele, MBH-IV-199pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27
OH
N
NH
OTBS
4m
157.2 148.8 130.9 124.8 124.6 121.5 119.6 77.2 CDCl3 27.6 25.8 20.4 18.3 -4.2
f1 (ppm)

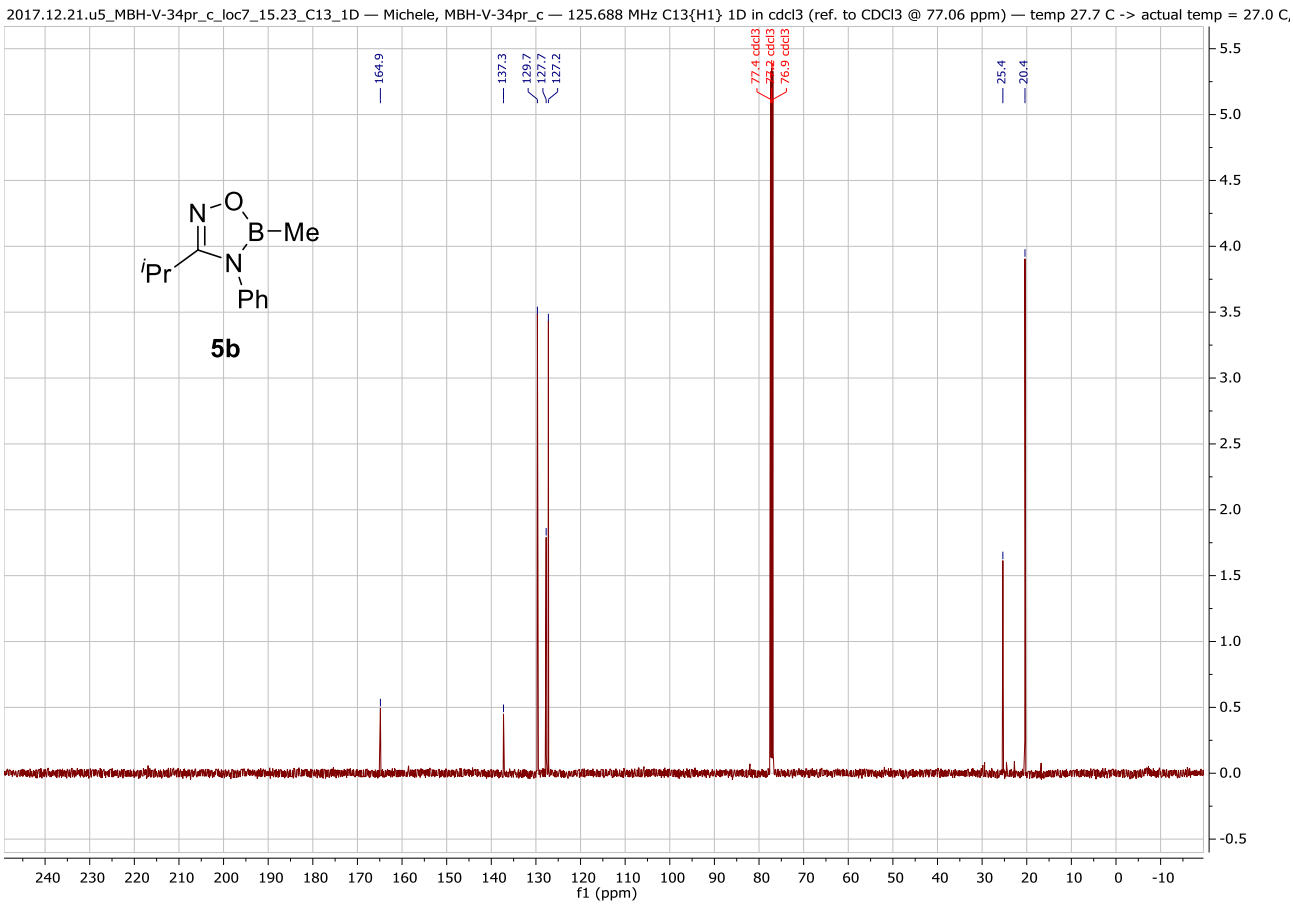
2018.01.04.u5_MBH-V-33_2_loc10_17.58_H1_1D — Michele, MBH-V-33_2 — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddual p

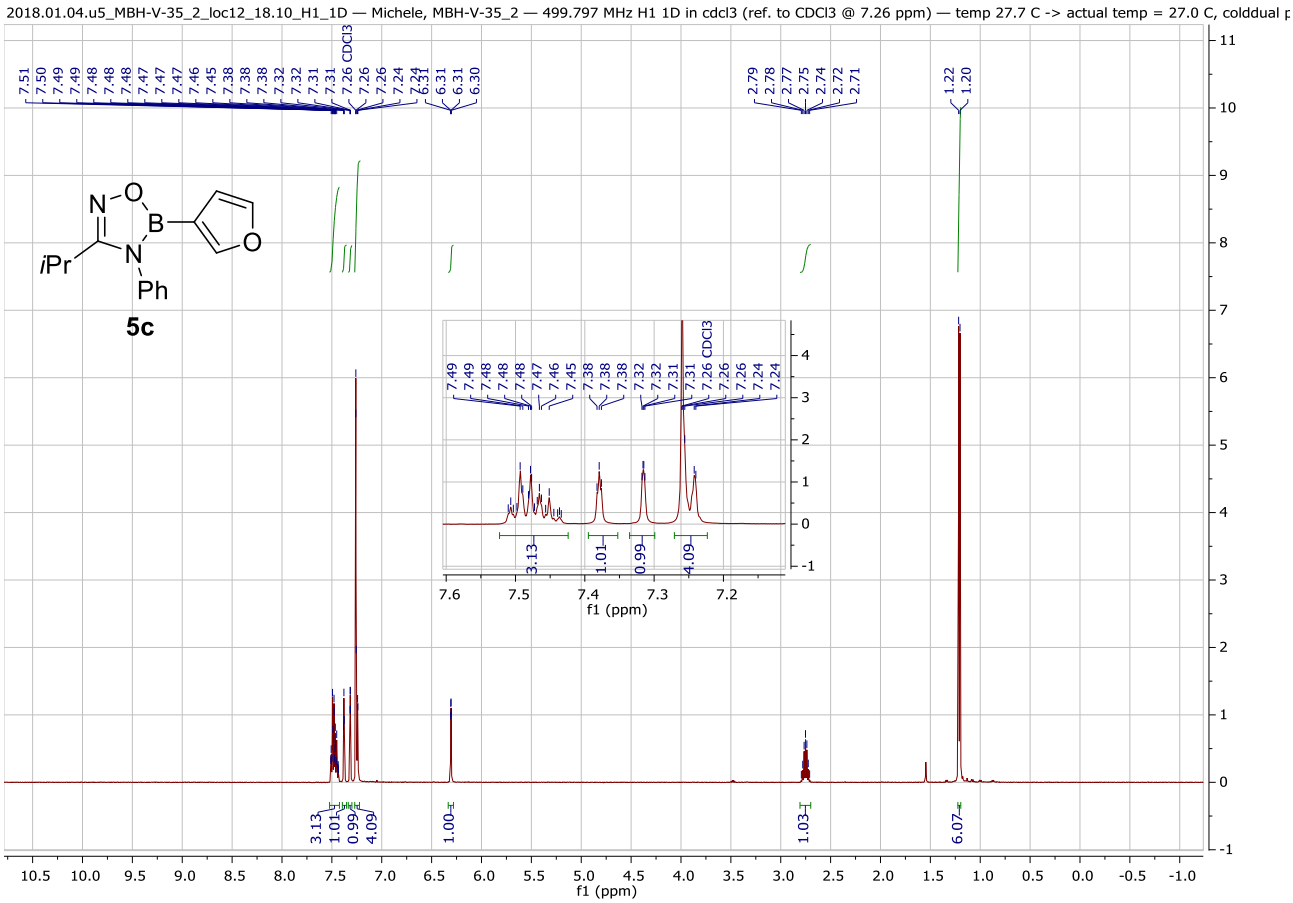


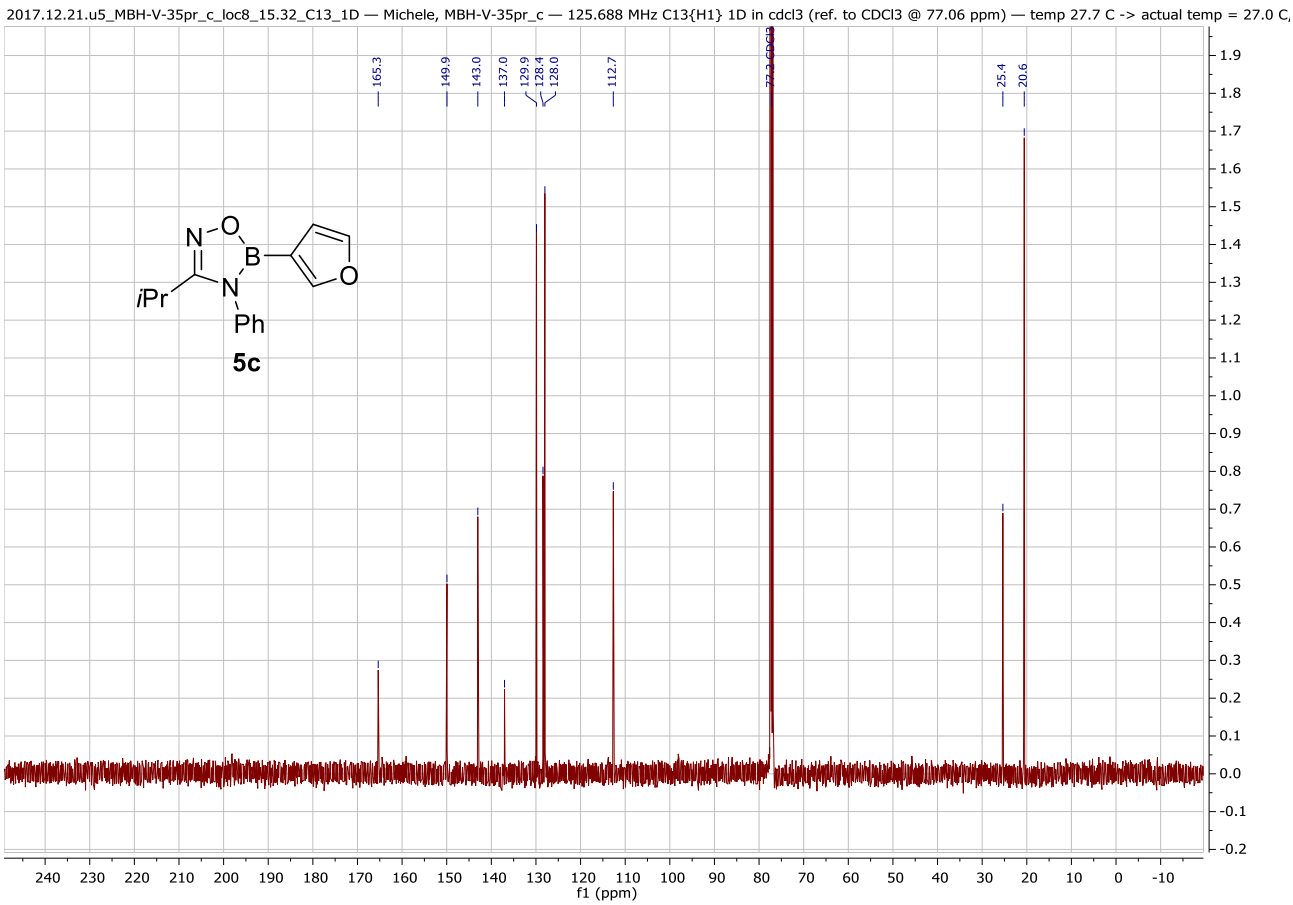
2017.12.21.u5_MBH-V-33pr_c_loc6_11.14_C13_1D — Michele, MBH-V-33pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C,



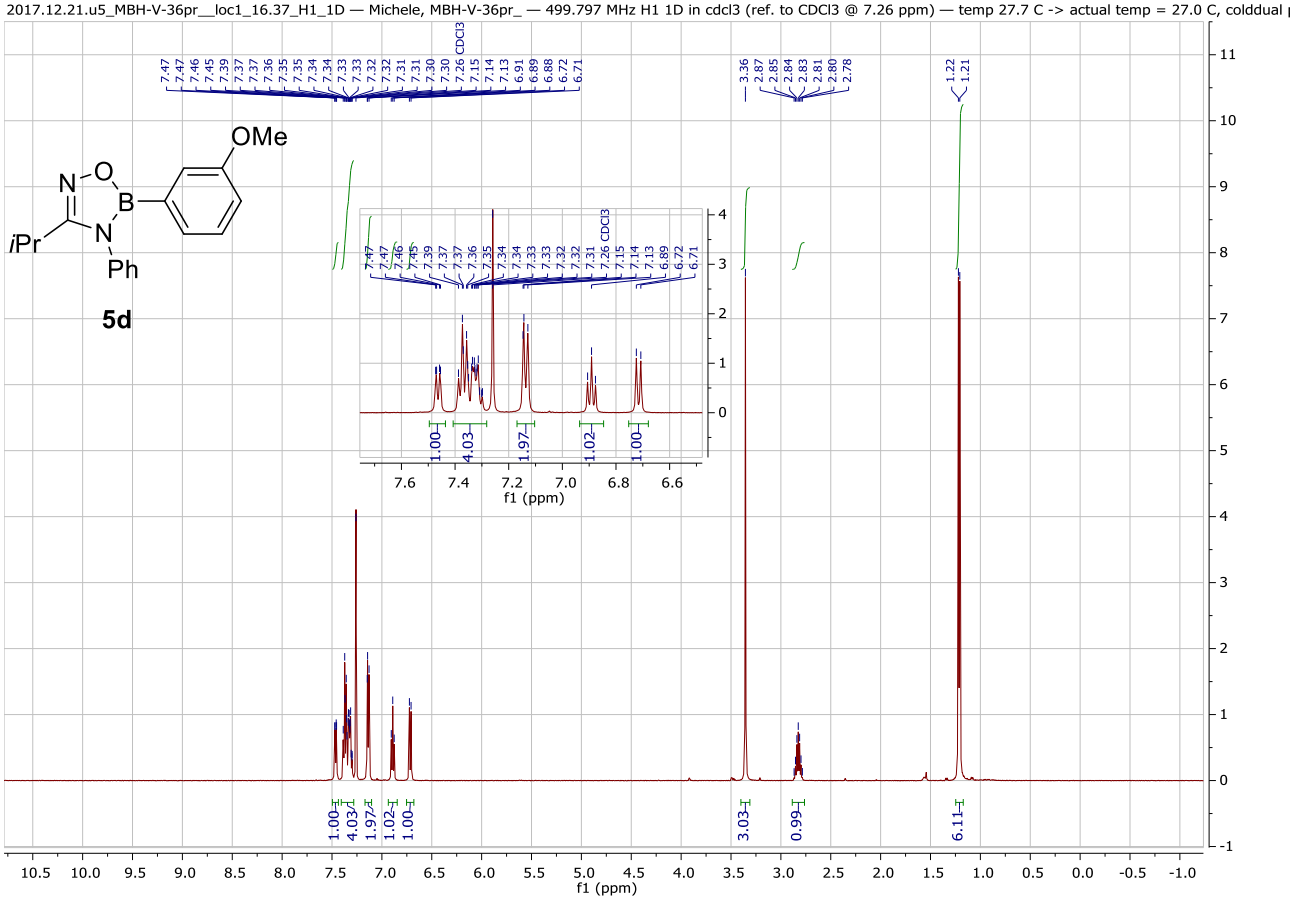

2017.12.21.u5_MBH-V-34pr_c_loc7_15.27_H1_1D — Michele, MBH-V-34pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddua
7.46 7.45 7.45 7.45 7.44 7.44 7.43 7.42 7.39 7.39 7.38 7.37 7.37 7.37 7.36 7.36 7.35 7.26 cdcl3 7.14 7.13 7.13 7.12 7.12 7.12
2.81 2.80 2.78 2.77 2.75 2.74 2.73
1.16 1.15
0.52
-1.12
N O B–Me
iPr N Ph
5b
2.09 1.00 1.99 1.00 6.00 2.97
f1 (ppm)


2017.12.21.u5_MBH-V-34pr_c_loc7_15.23_C13_1D — Michele, MBH-V-34pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C,
164.9
137.3
129.7
127.7
127.2
77.4 cdcl3
77.2 cdcl3
76.9 cdcl3
25.4
20.4
N O B–Me
iPr N Ph
5b
f1 (ppm)

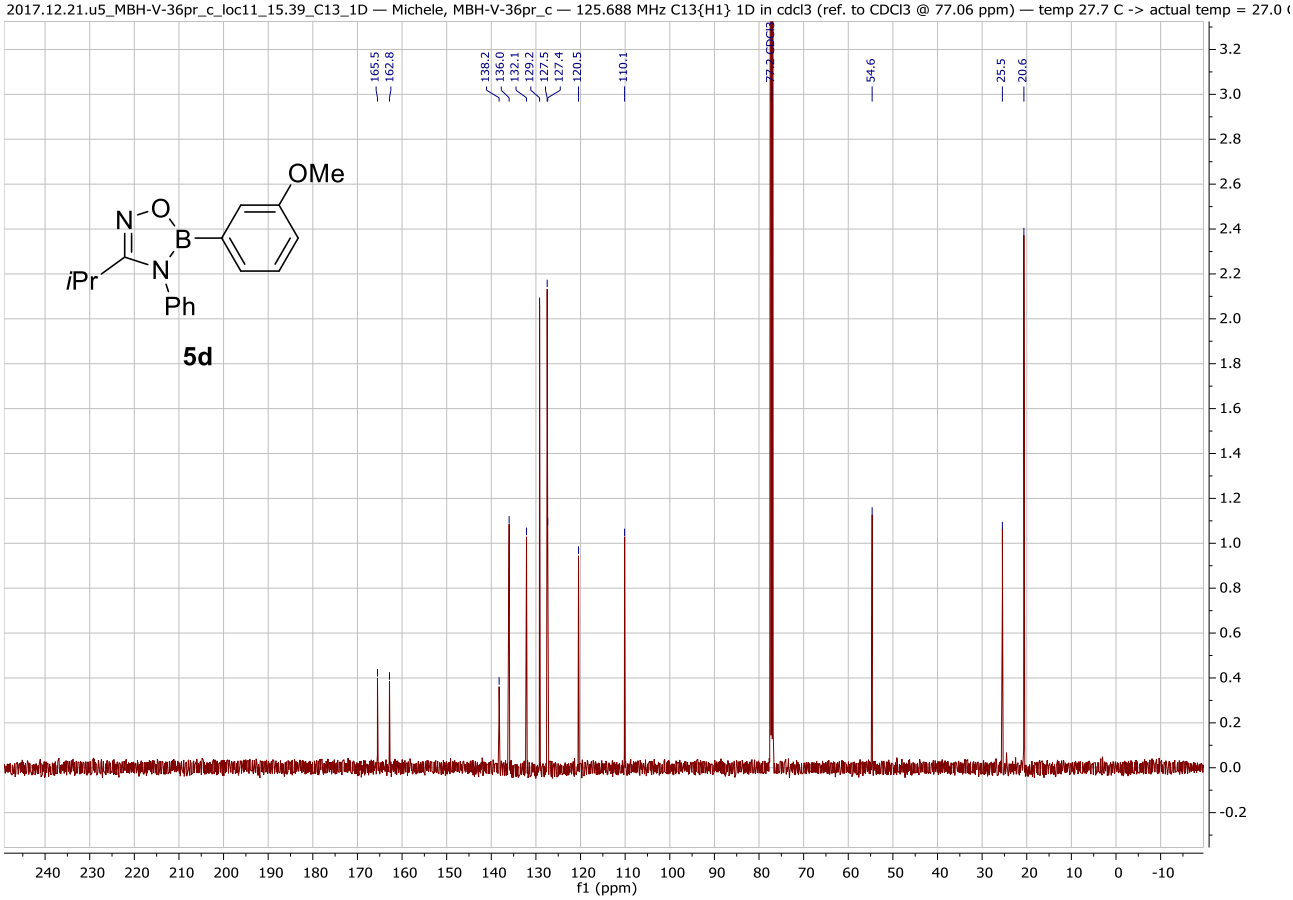

2018.01.04.u5_MBH-V-35_2_loc12_18.10_H1_1D — Michele, MBH-V-35_2 — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddual p
7.51 7.50 7.49 7.49 7.48 7.48 7.48 7.47 7.47 7.47 7.46 7.45 7.38 7.38 7.38 7.32 7.32 7.31 7.31 7.26 CDCl3 7.26 7.26 7.24 6.31 6.31 6.30
2.79 2.78 2.77 2.75 2.74 2.72 2.71
1.22 1.20
iPr
Ph
5c
3.13 1.01 0.99 4.09 1.00 1.03 6.07
f1 (ppm)

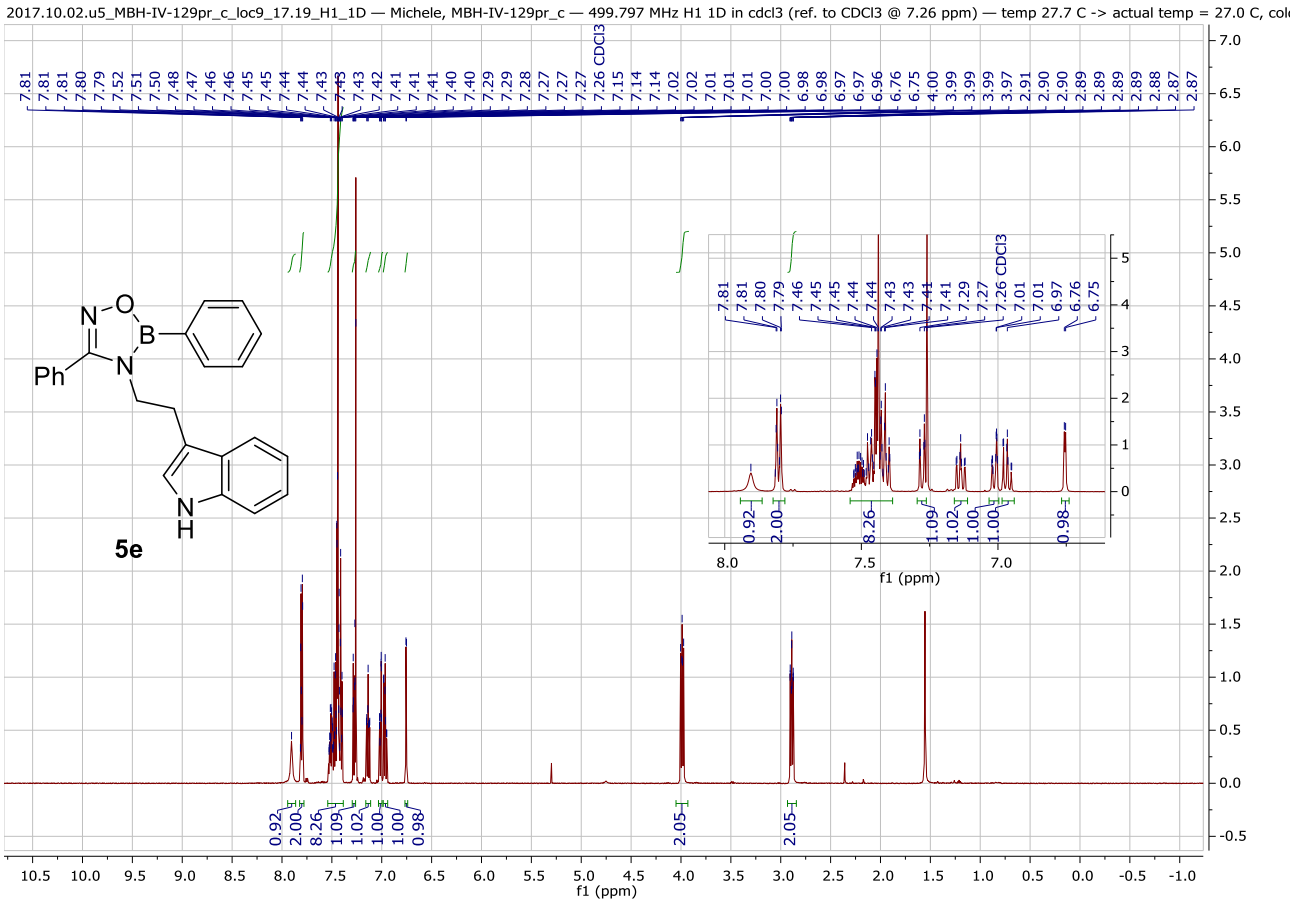

2017.12.21.u5_MBH-V-35pr_c_loc8_15.32_C13_1D — Michele, MBH-V-35pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C,
165.3 149.9 143.0 137.0 129.9 128.4 128.0 112.7 77.3 CDCl3 25.4 20.6
iPr
Ph
5c
f1 (ppm)

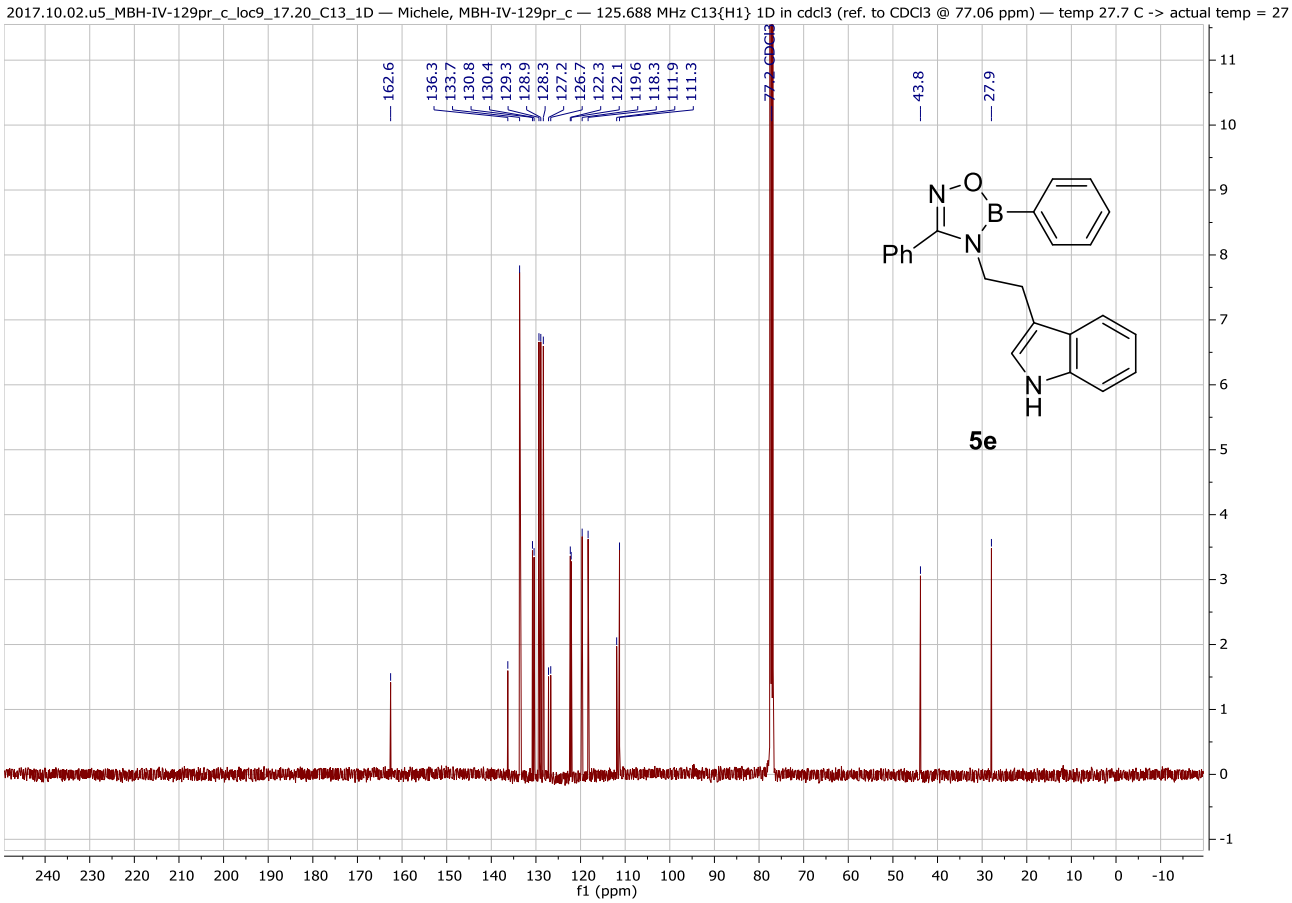
2017.12.21.u5_MBH-V-36pr__loc1_16.37_H1_1D — Michele, MBH-V-36pr_ — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddual

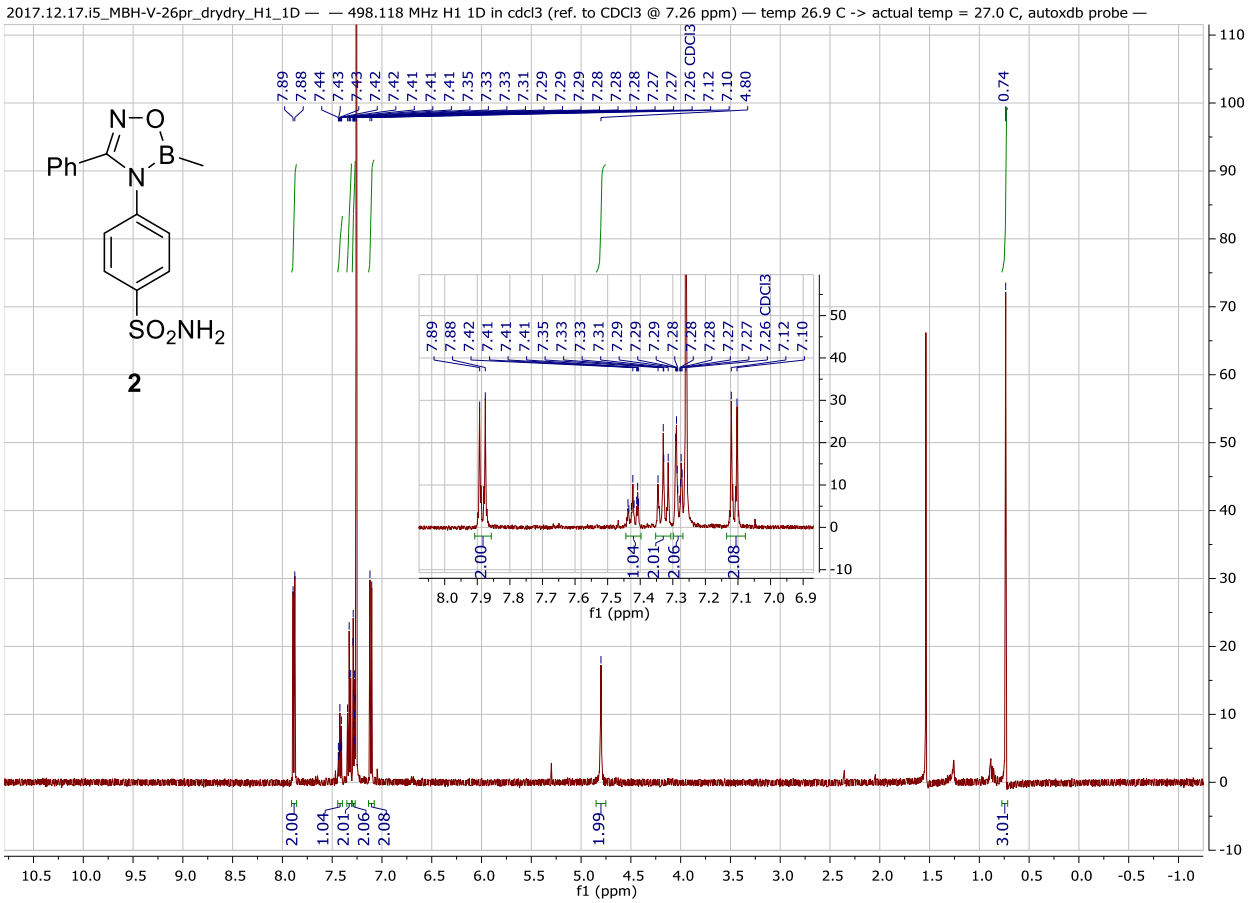


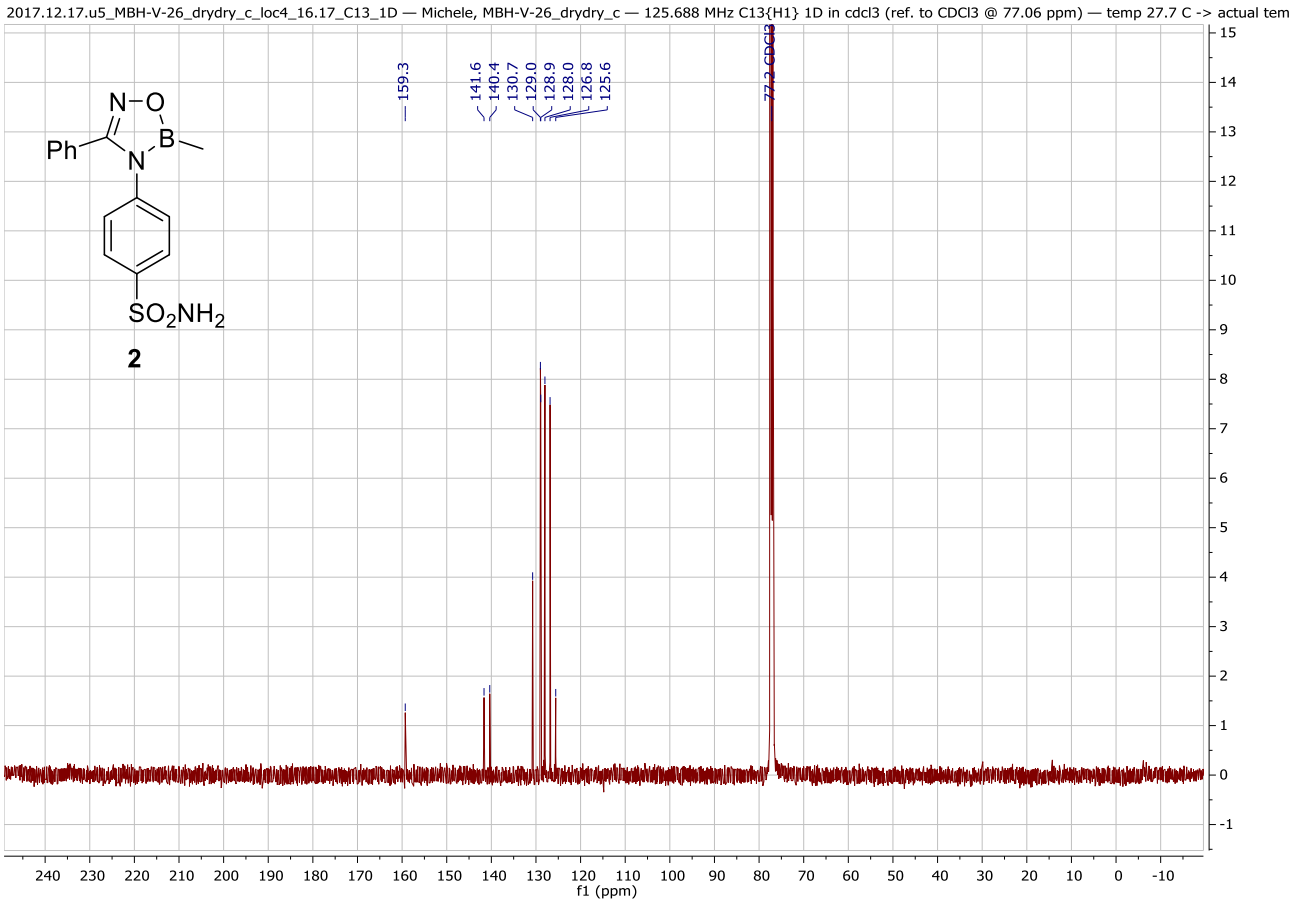
2017.12.21.u5_MBH-V-36pr_c_loc11_15.39_C13_1D — Michele, MBH-V-36pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C

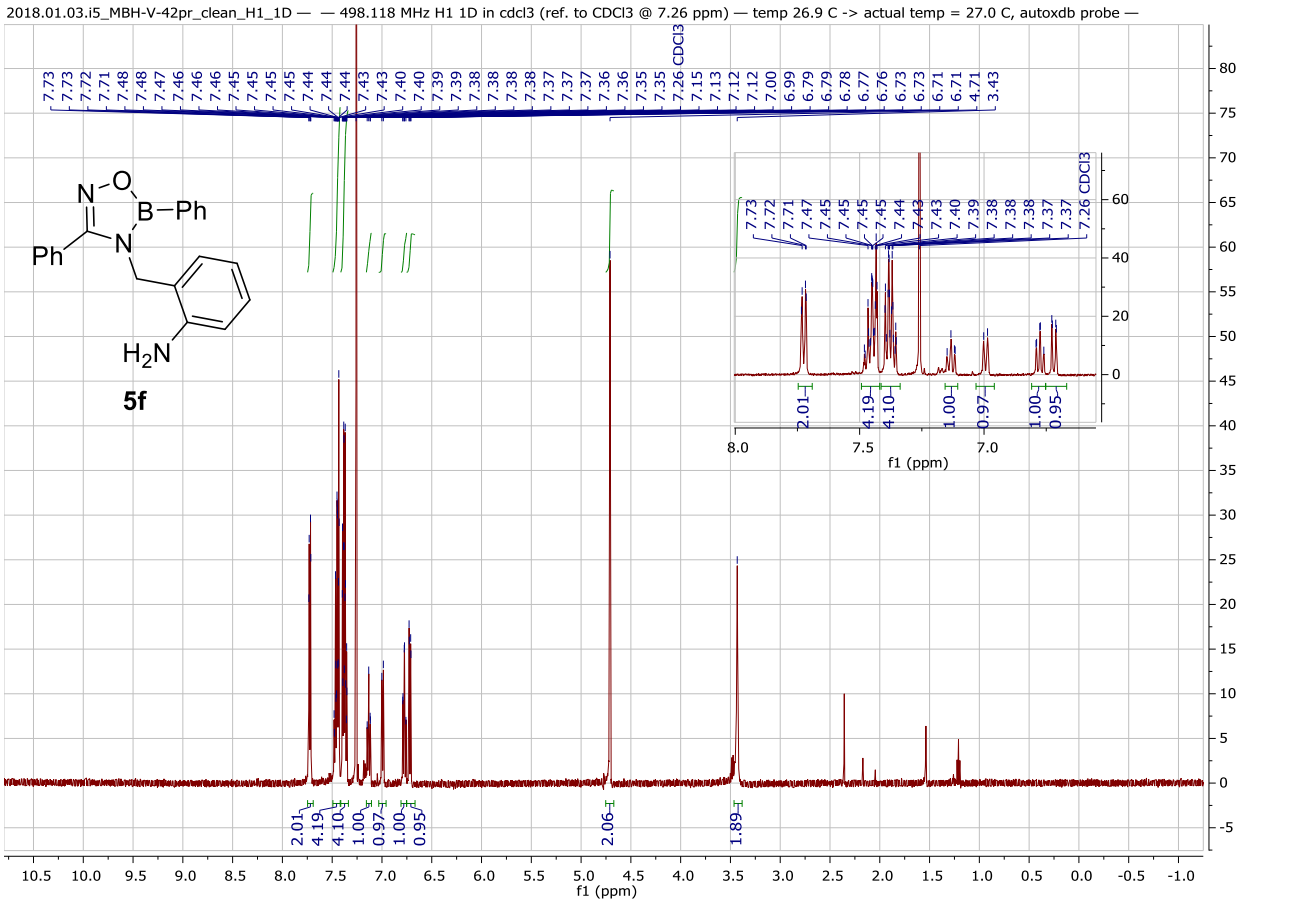


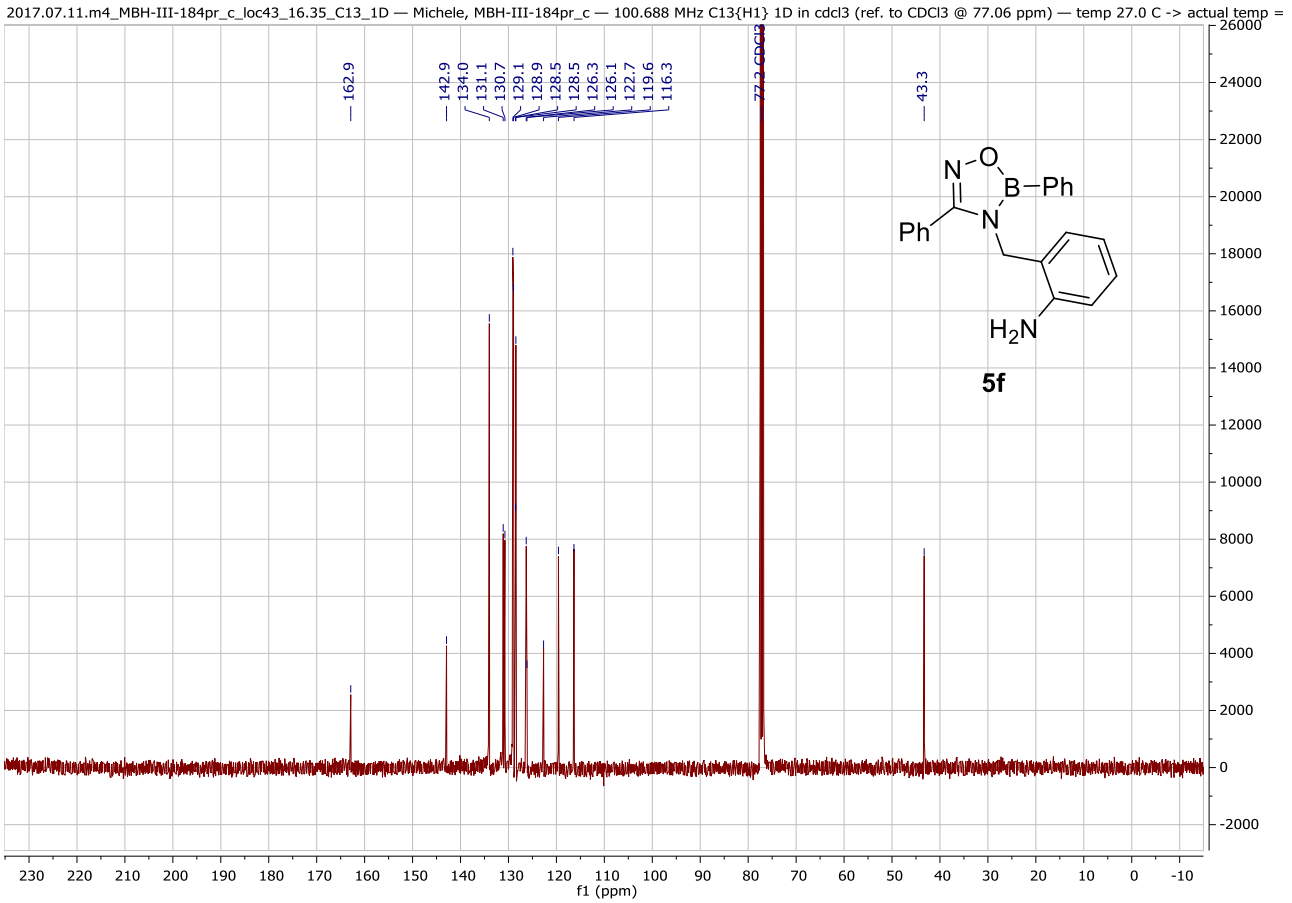

2017.10.02.u5_MBH-IV-129pr_c_loc9_17.19_H1_1D — Michele, MBH-IV-129pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, col
7.81 7.81 7.81 7.80 7.79 7.52 7.51 7.50 7.48 7.47 7.46 7.46 7.45 7.45 7.44 7.44 7.43 7.43 7.43 7.42 7.41 7.41 7.41 7.40 7.40 7.29 7.29 7.28 7.27 7.27 7.27 7.26 CDCl3 7.15 7.14 7.14 7.02 7.02 7.01 7.01 7.00 7.00 6.98 6.98 6.97 6.97 6.96 6.76 6.75 4.00 3.99 3.99 3.99 3.97 2.91 2.90 2.90 2.89 2.89 2.89 2.88 2.87 2.87
0.92 2.00 8.26 1.09 1.02 1.00 1.00 0.98 2.05 2.05
f1 (ppm)
5e

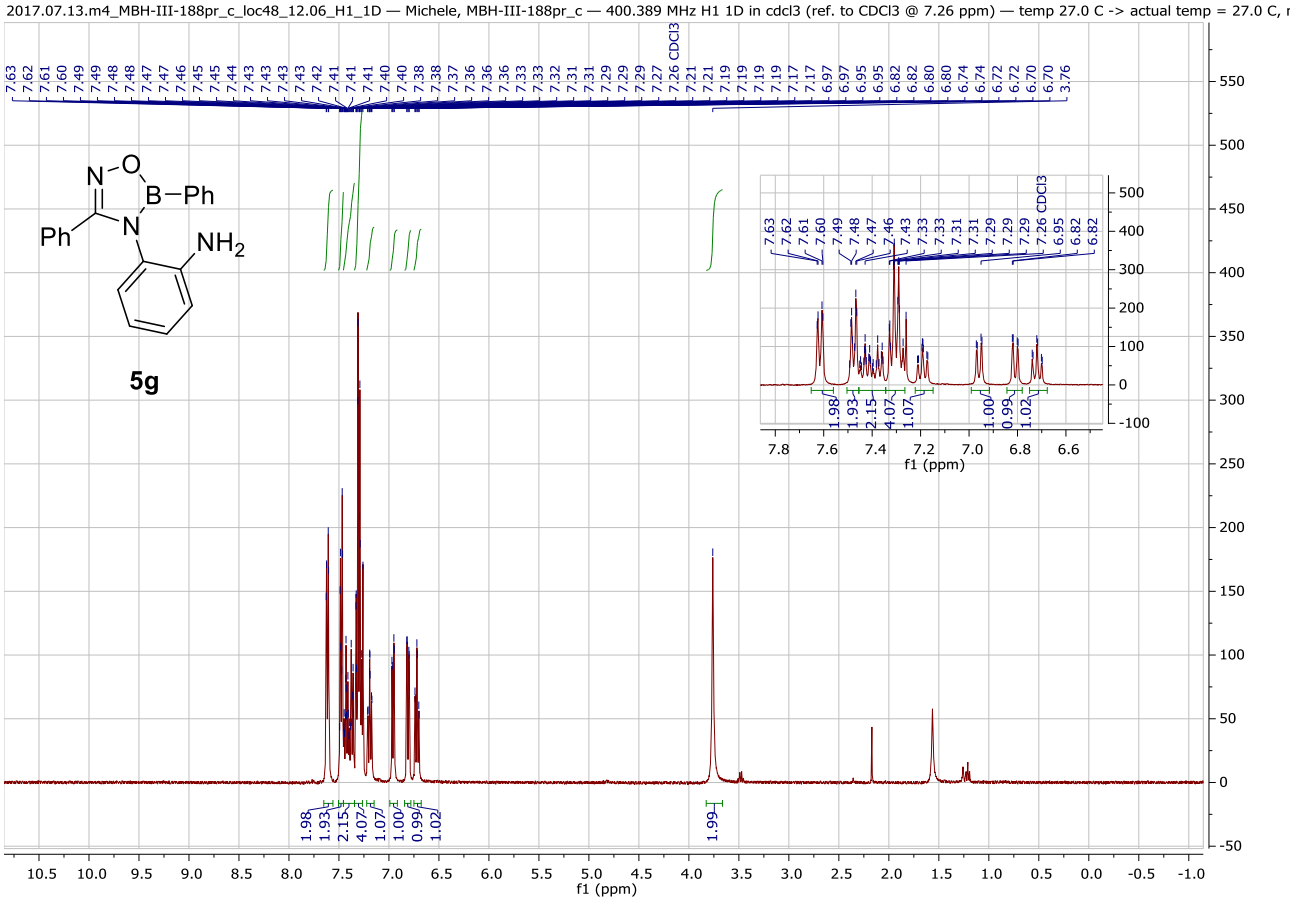

2017.10.02.u5_MBH-IV-129pr_c_loc9_17.20_C13_1D — Michele, MBH-IV-129pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27
162.6 136.3 133.7 130.8 130.4 129.3 128.9 128.3 127.2 126.7 122.3 122.1 119.6 118.3 111.9 111.3 77.2 CDCl3 43.8 27.9
f1 (ppm)
5e

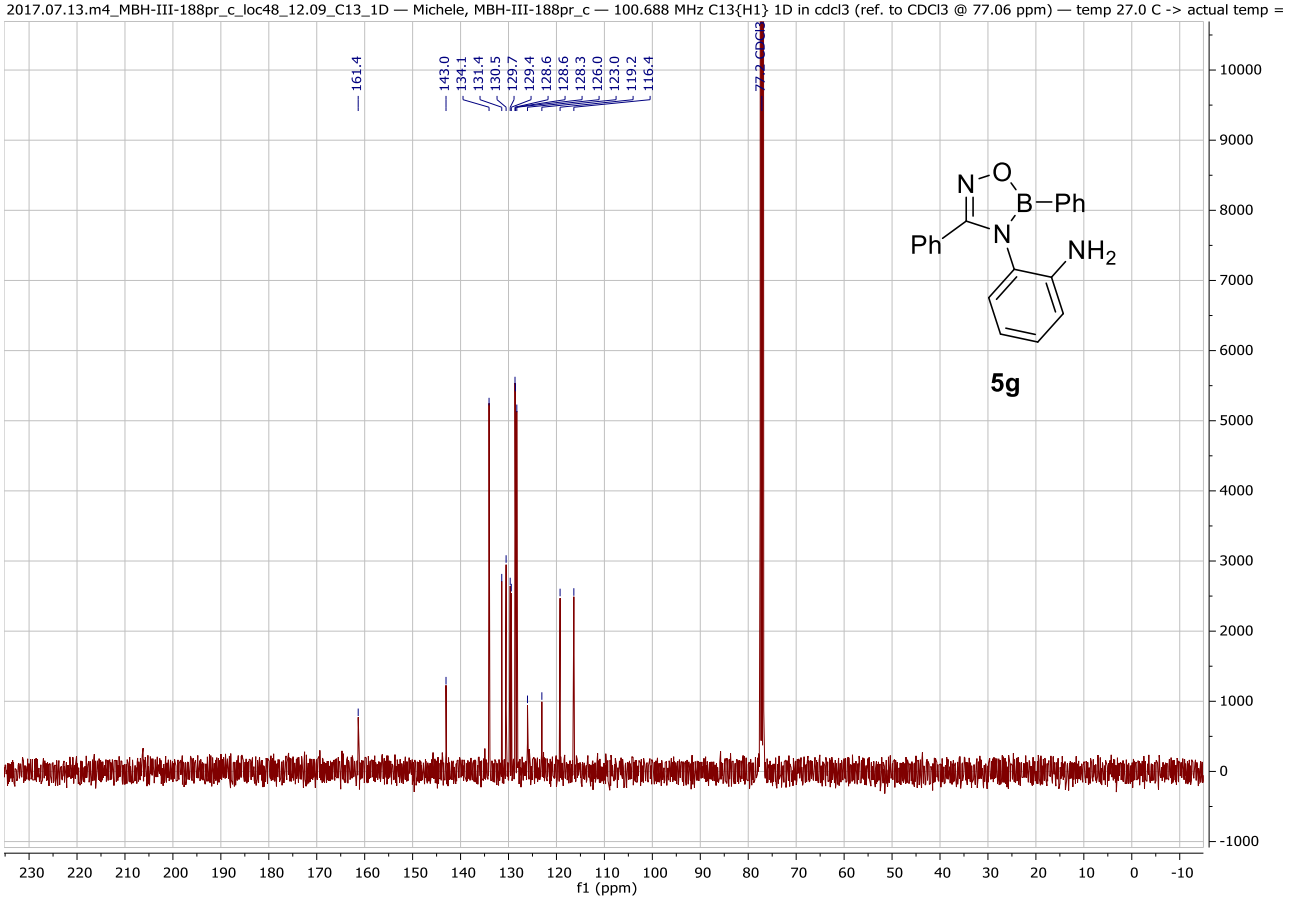

2017.12.17.i5_MBH-V-26pr_drydry_H1_1D — — 498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
Ph
N
O
B
N
SO2NH2
2
f1 (ppm)

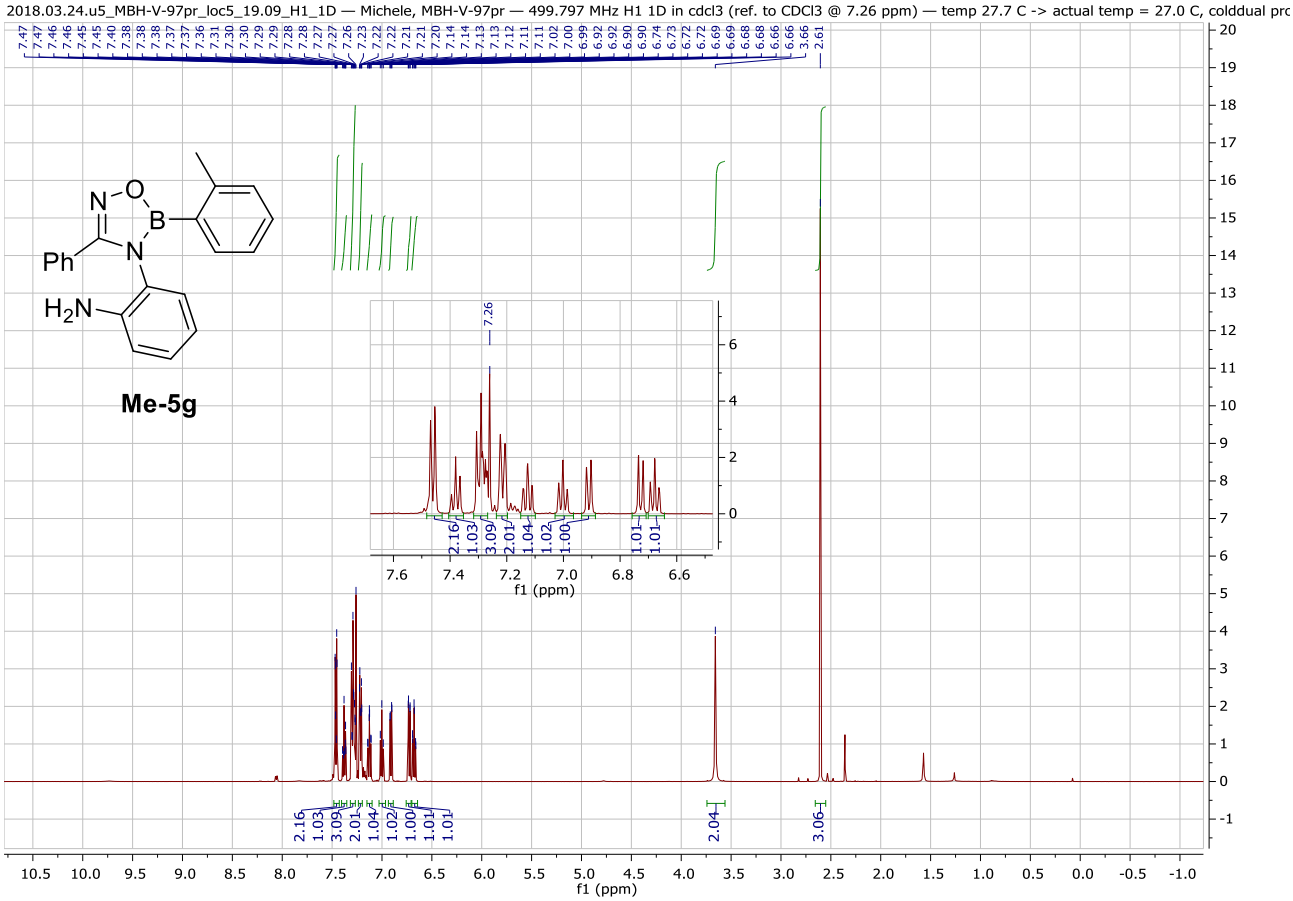

2017.12.17.u5_MBH-V-26_drydry_c_loc4_16.17_C13_1D — Michele, MBH-V-26_drydry_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual tem
Ph
N
O
B
N
SO2NH2
2
f1 (ppm)

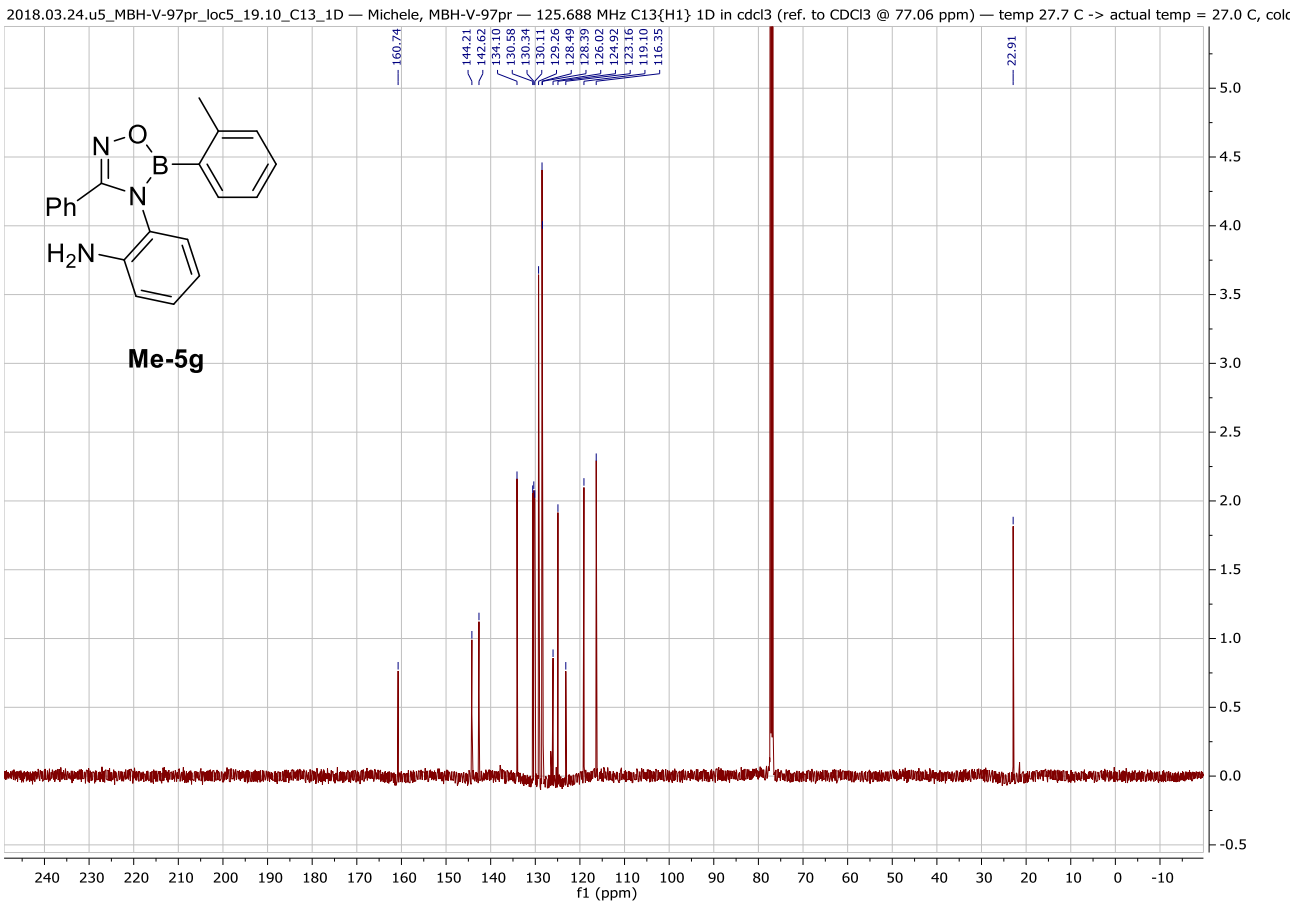

2018.01.03.i5_MBH-V-42pr_clean_H1_1D — — 498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
CDCl3
f1 (ppm)
5f

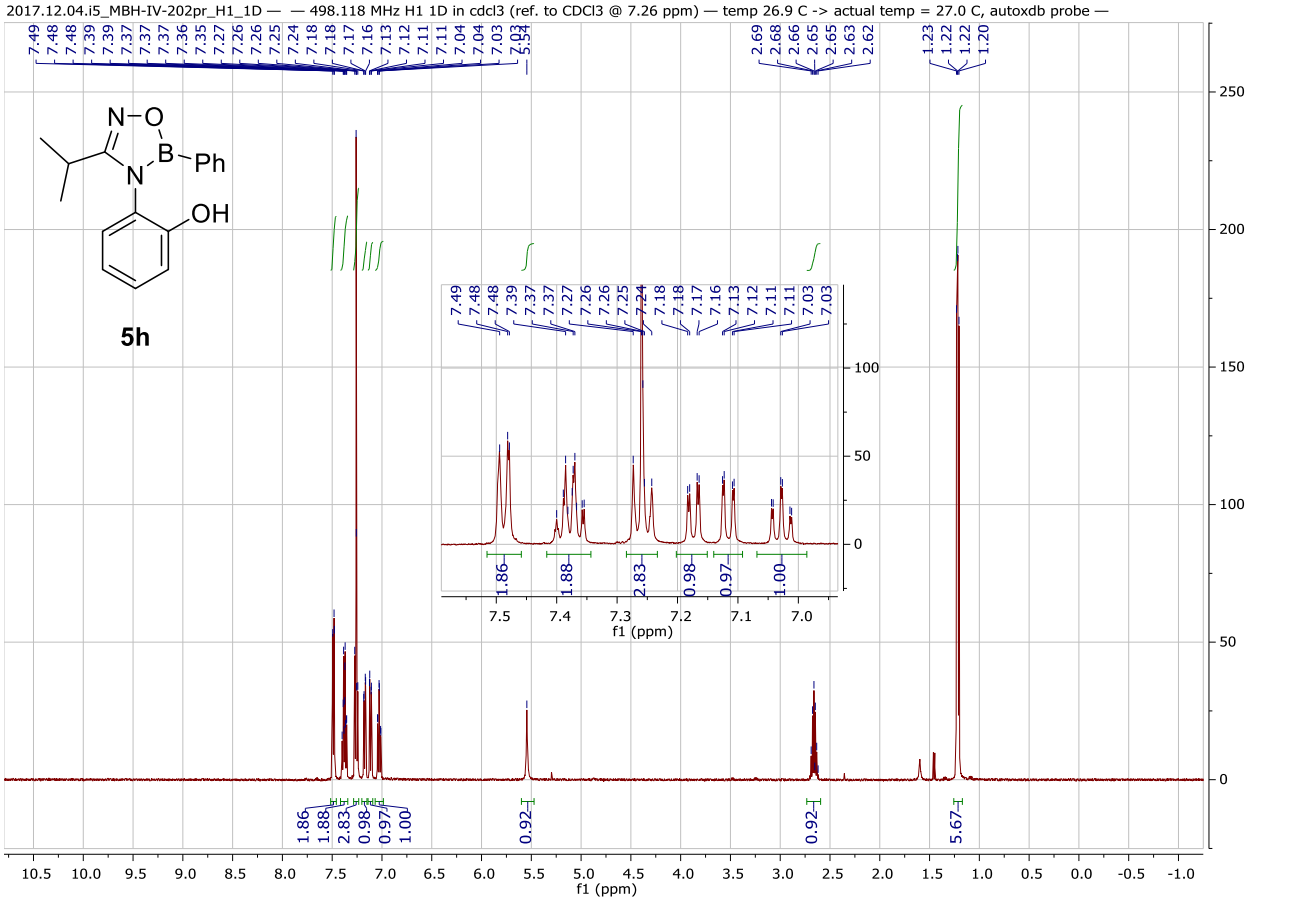

2017.07.11.m4_MBH-III-184pr_c_loc43_16.35_C13_1D — Michele, MBH-III-184pr_c — 100.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.0 C -> actual temp =
CDCl3
f1 (ppm)
5f

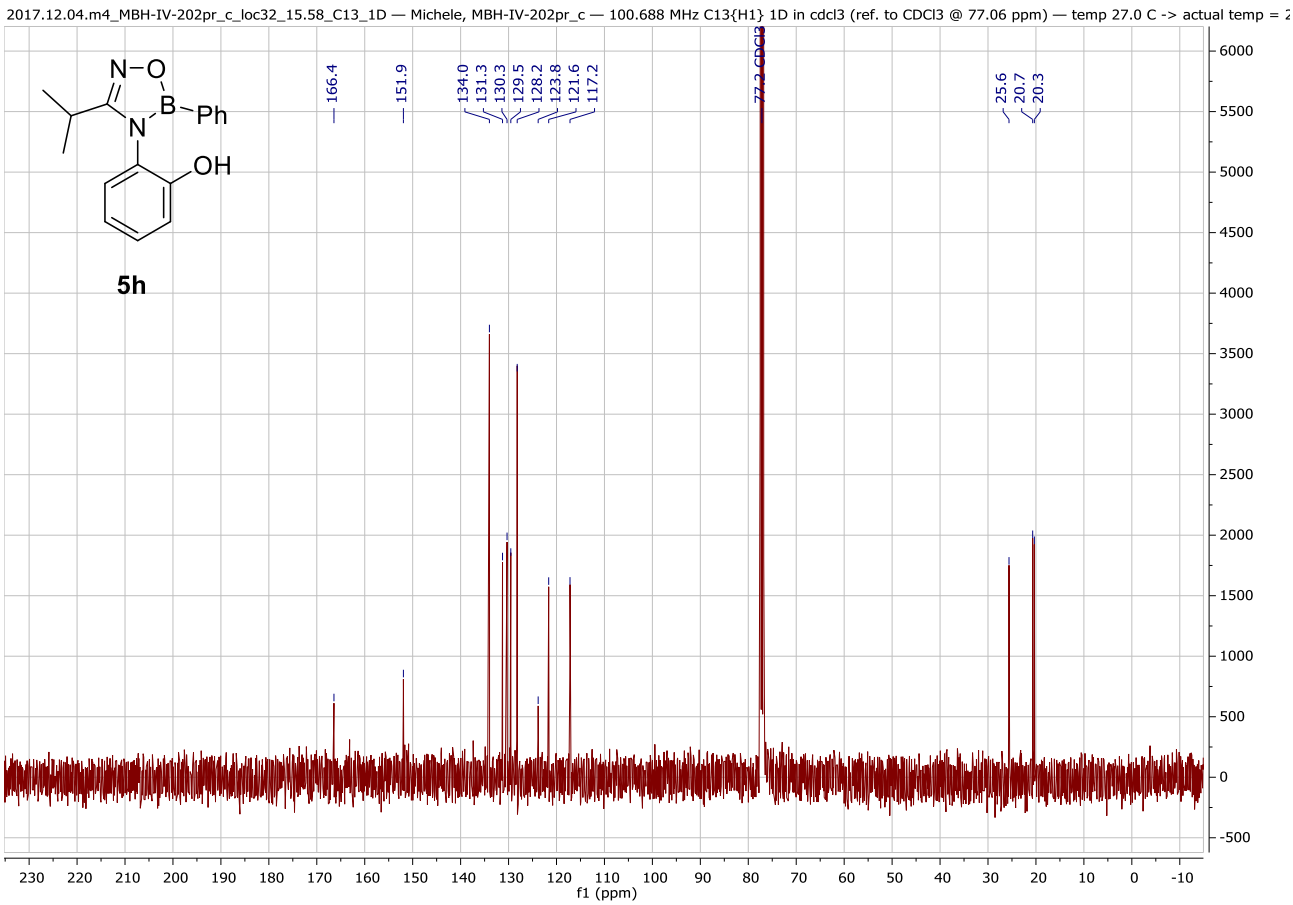

2017.07.13.m4_MBH-III-188pr_c_loc48_12.06_H1_1D — Michele, MBH-III-188pr_c — 400.389 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.0 C -> actual temp = 27.0 C, r
5g
f1 (ppm)

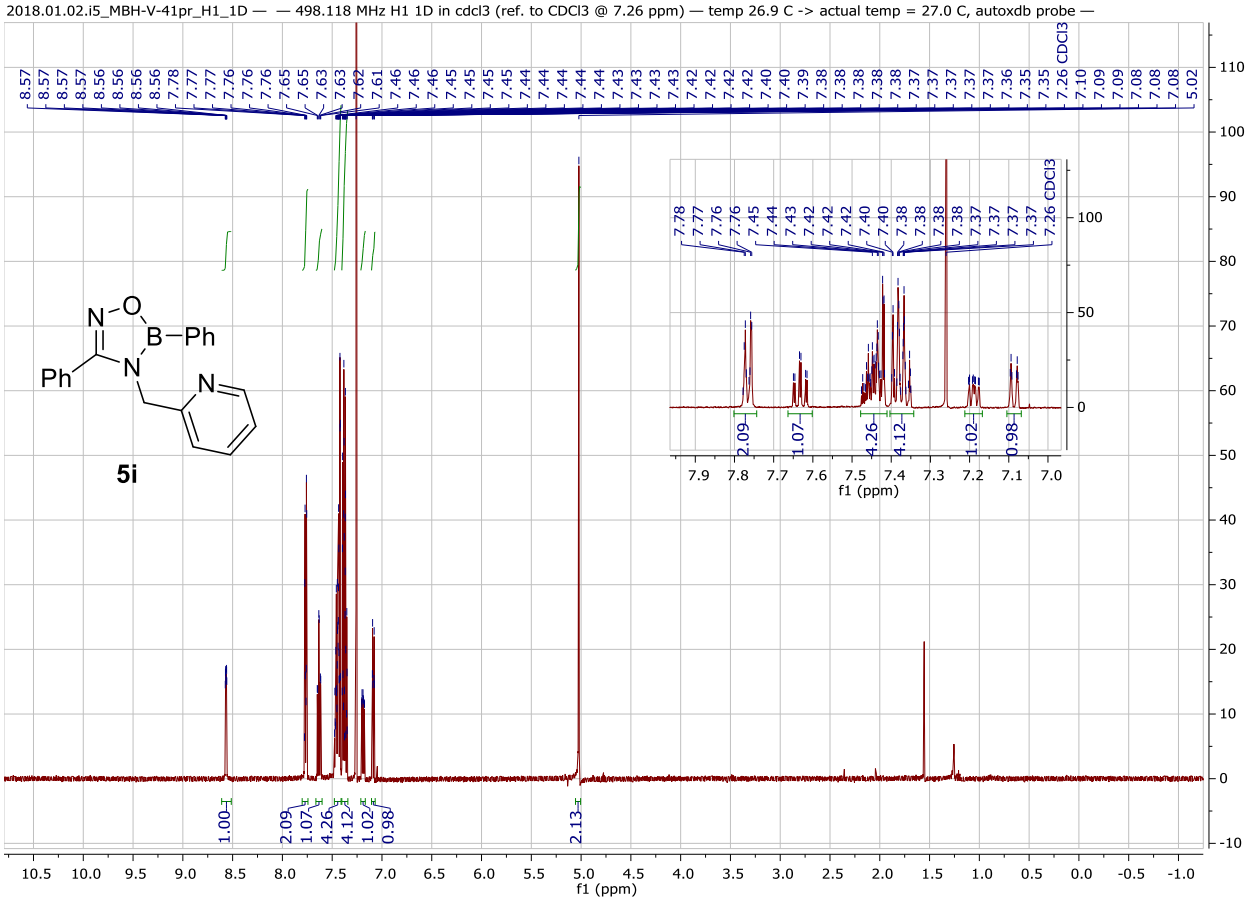

2017.07.13.m4_MBH-III-188pr_c_loc48_12.09_C13_1D — Michele, MBH-III-188pr_c — 100.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.0 C -> actual temp =
161.4
143.0
134.1
131.4
130.5
129.7
129.4
128.6
128.6
128.3
126.0
123.0
119.2
116.4
5g
f1 (ppm)

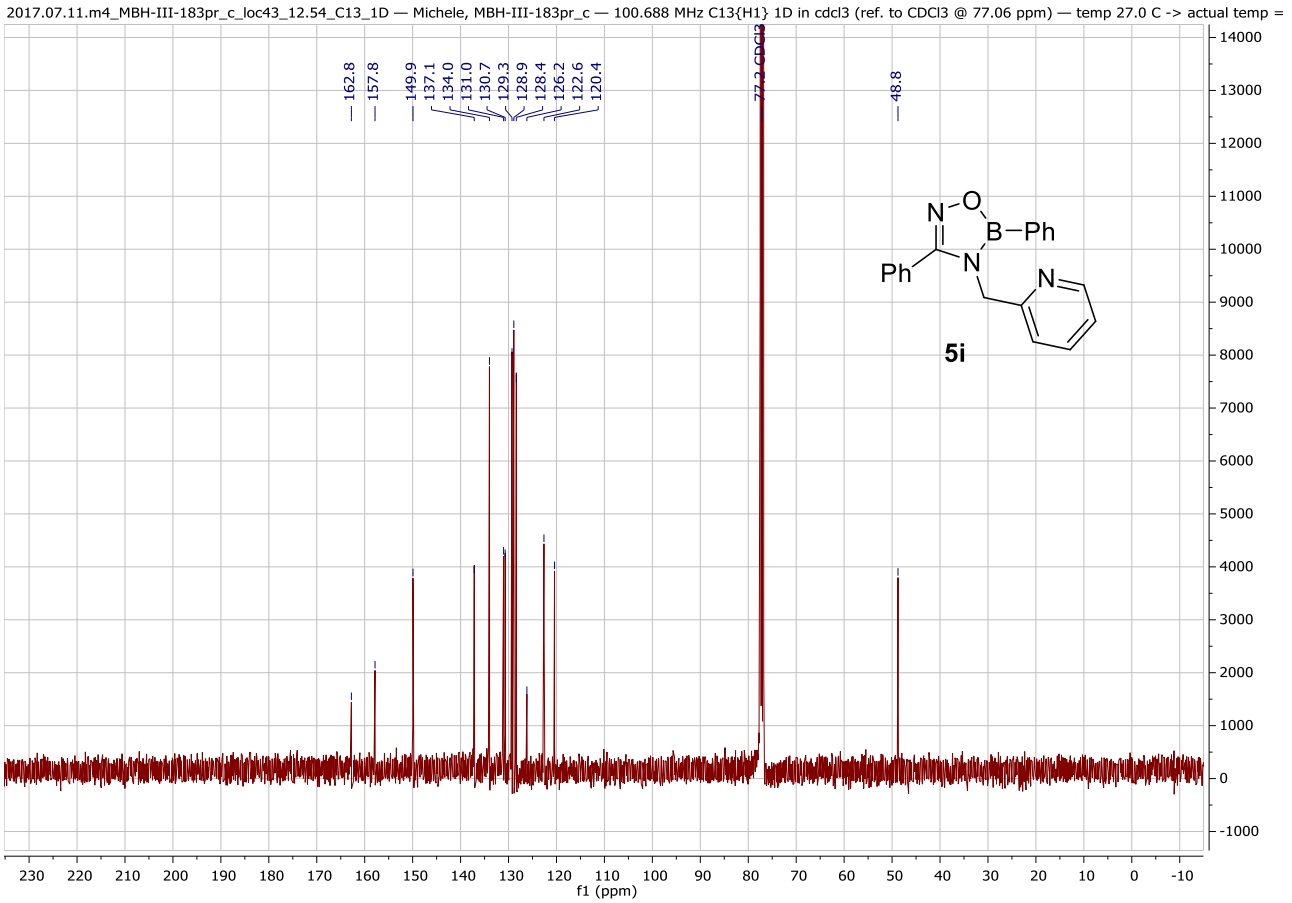

2018.03.24.u5_MBH-V-97pr_loc5_19.09_H1_1D — Michele, MBH-V-97pr — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddual prc
Me-5g
f1 (ppm)

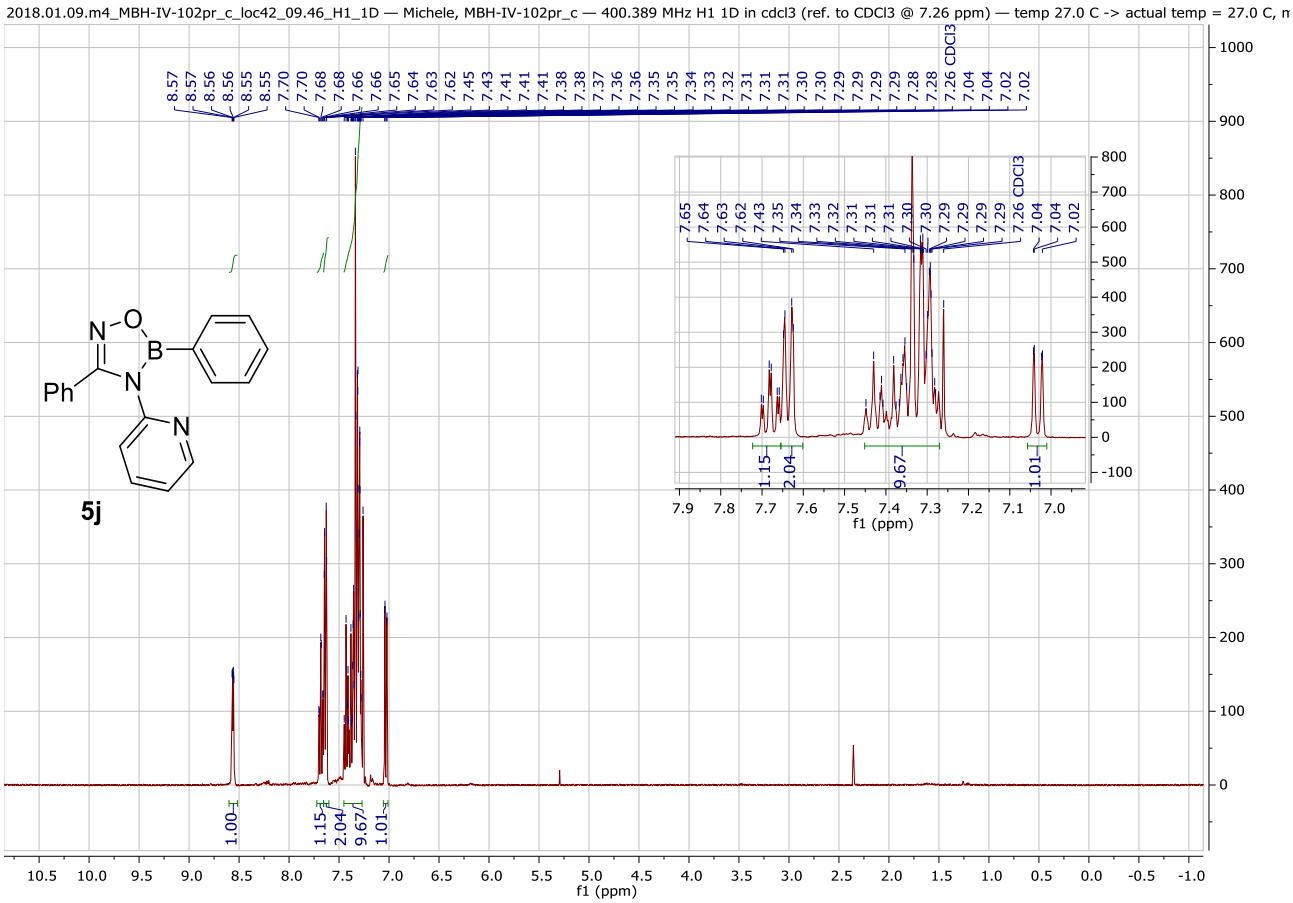

2018.03.24.u5_MBH-V-97pr_loc5_19.10_C13_1D — Michele, MBH-V-97pr — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C, colc
Me-5g
f1 (ppm)

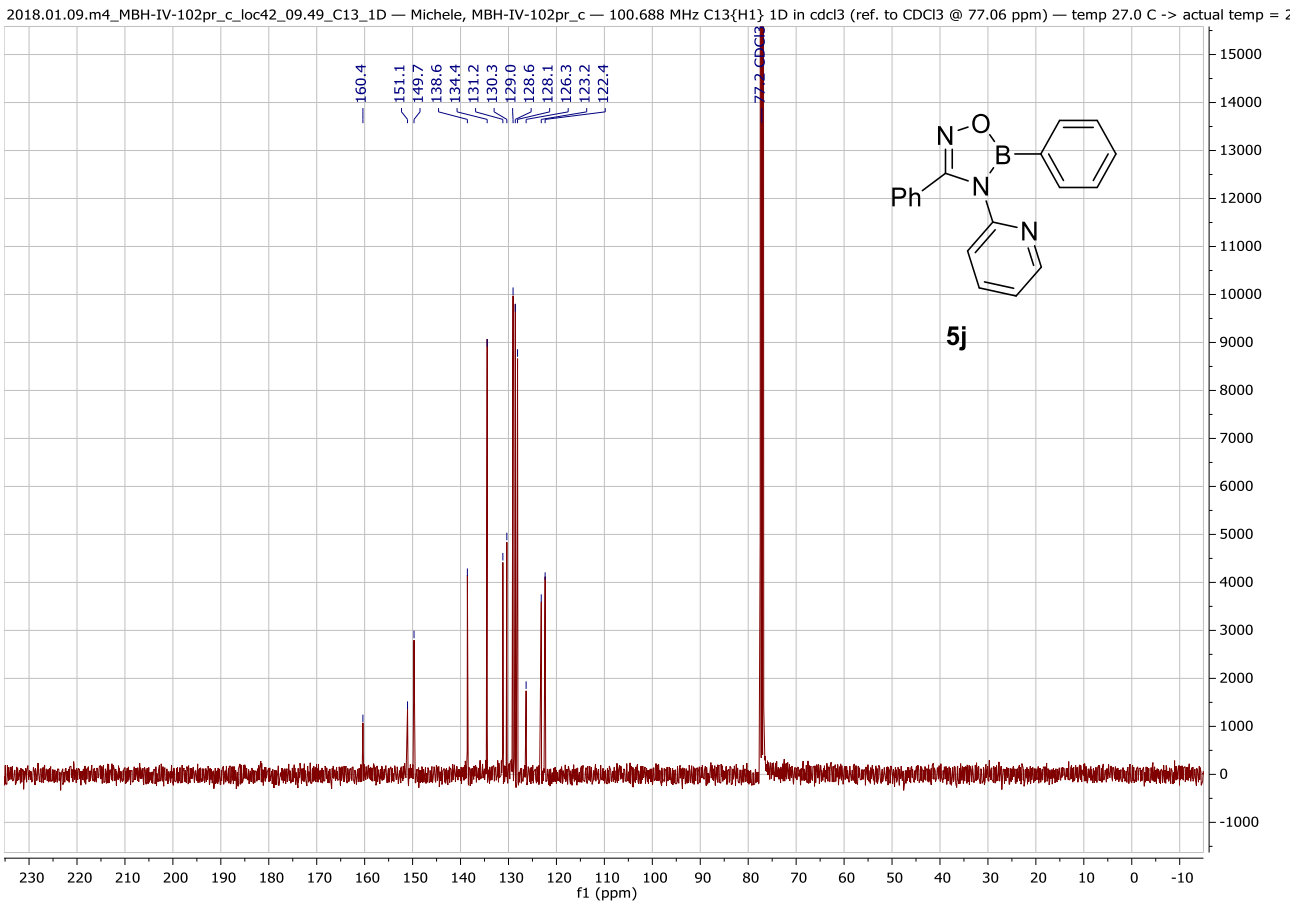

2017.12.04.i5_MBH-IV-202pr_H1_1D — — 498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
5h
f1 (ppm)

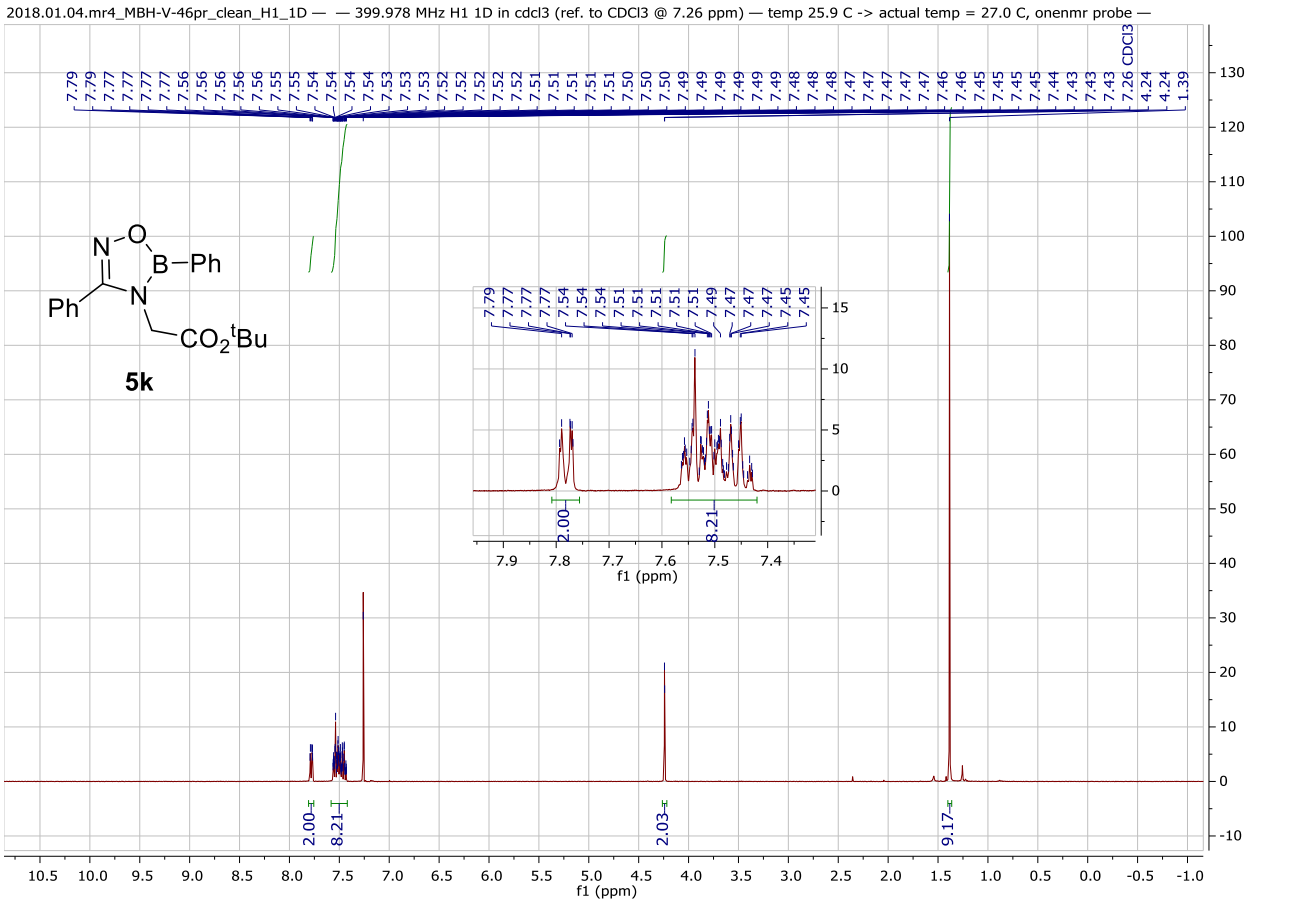

2017.12.04.m4_MBH-IV-202pr_c_loc32_15.58_C13_1D — Michele, MBH-IV-202pr_c — 100.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.0 C -> actual temp = 2
5h
f1 (ppm)

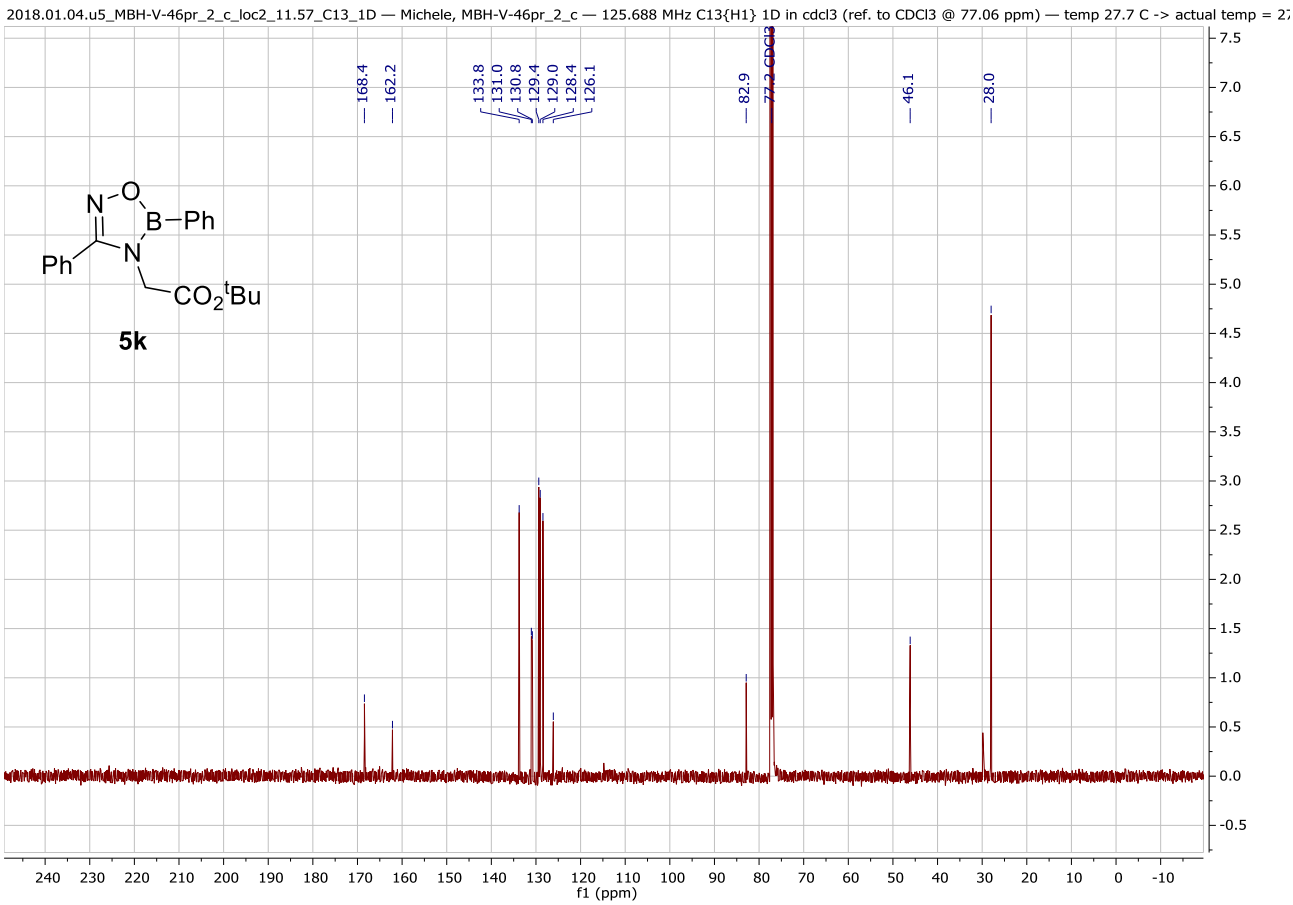

2018.01.02.i5_MBH-V-41pr_H1_1D — — 498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
5i
f1 (ppm)

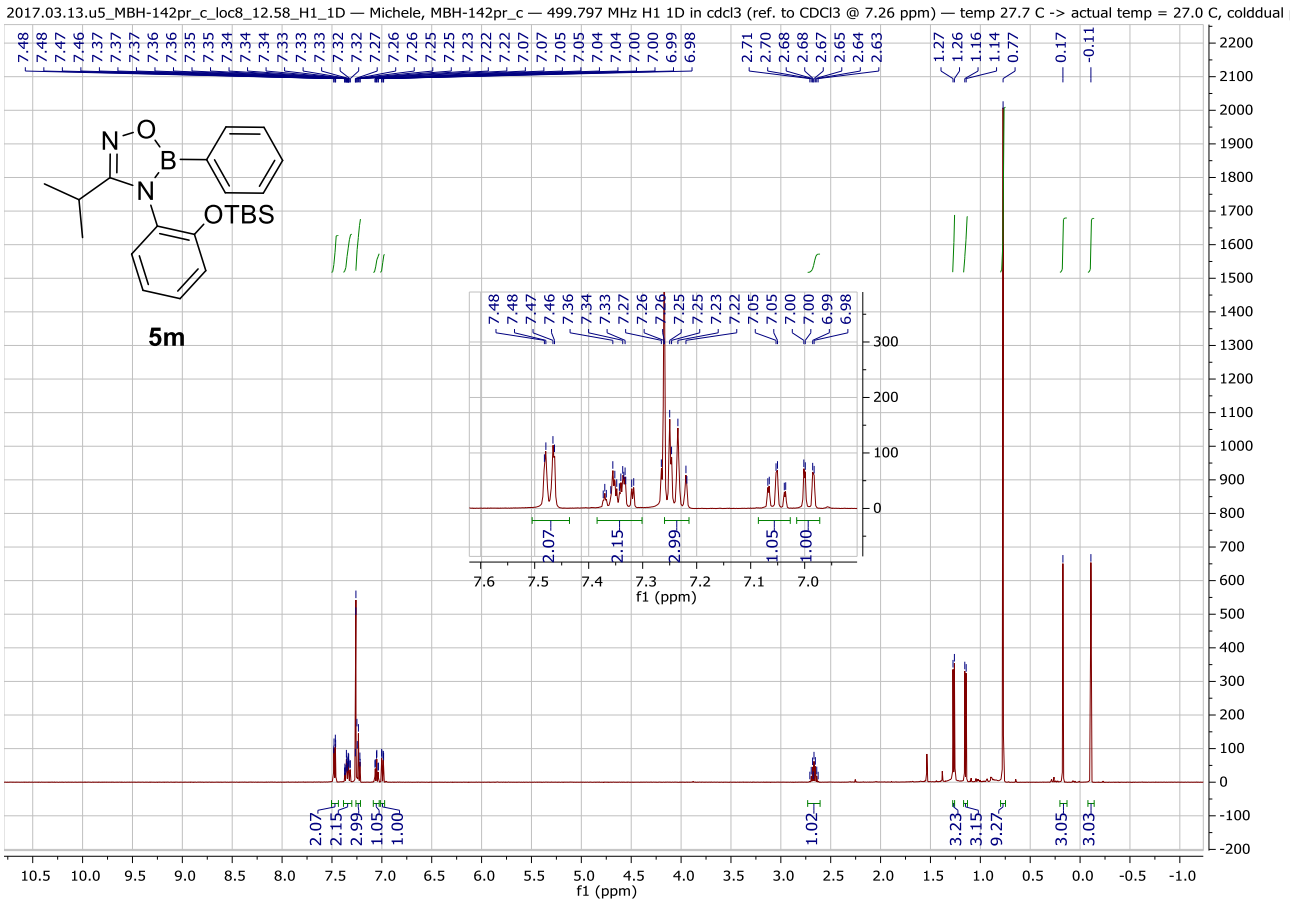

2017.07.11.m4_MBH-III-183pr_c_loc43_12.54_C13_1D — Michele, MBH-III-183pr_c — 100.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.0 C -> actual temp =
5i
f1 (ppm)

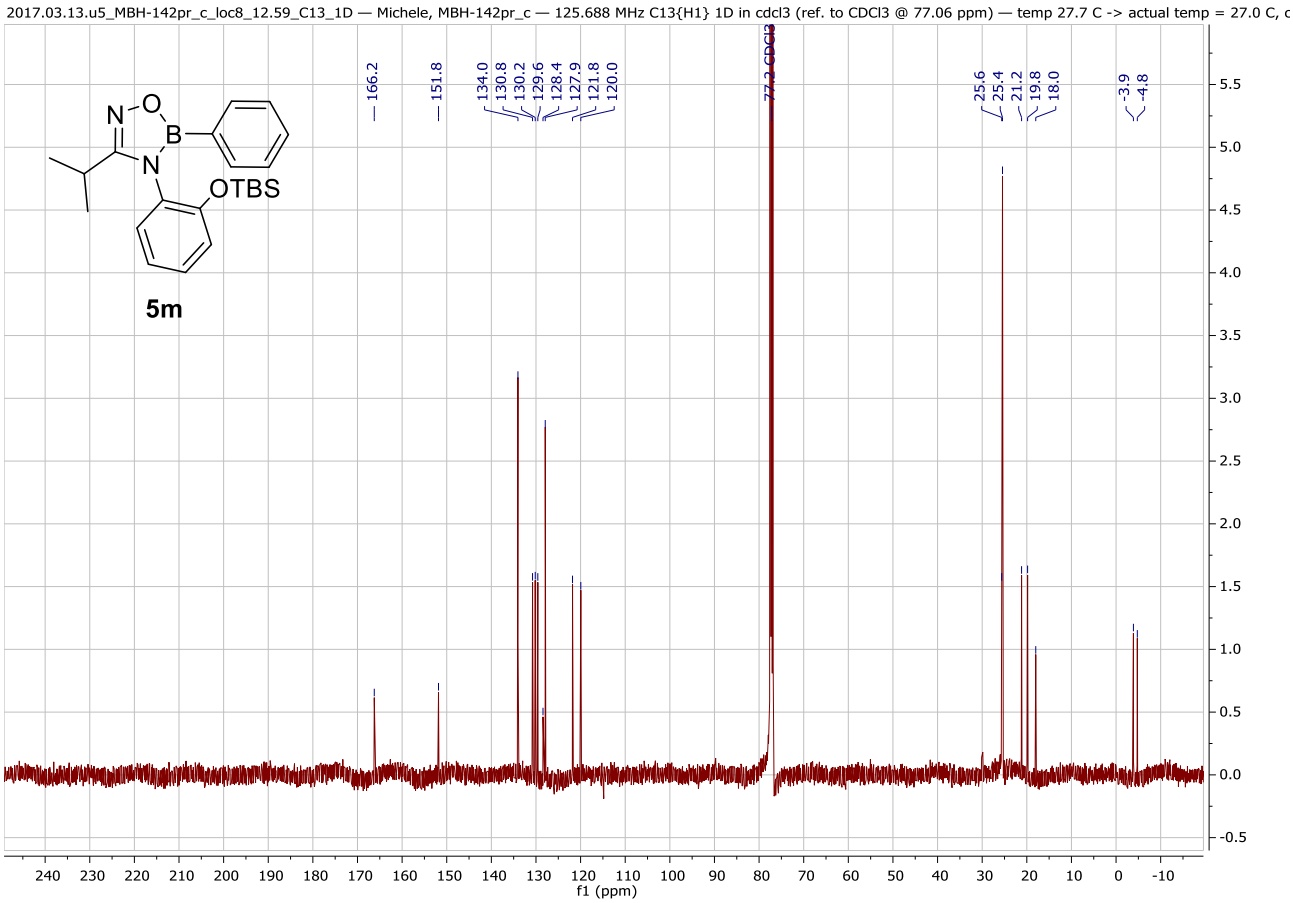

2018.01.09.m4_MBH-IV-102pr_c_loc42_09.46_H1_1D — Michele, MBH-IV-102pr_c — 400.389 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.0 C -> actual temp = 27.0 C, n
8.57 8.57 8.56 8.56 8.55 8.55 7.70 7.68 7.68 7.66 7.66 7.65 7.64 7.63 7.62 7.45 7.43 7.41 7.41 7.41 7.38 7.38 7.37 7.36 7.36 7.35 7.35 7.34 7.33 7.32 7.31 7.31 7.31 7.30 7.30 7.29 7.29 7.29 7.28 7.28 7.26 CDCl3 7.04 7.04 7.02 7.02
7.65 7.64 7.63 7.62 7.43 7.35 7.34 7.33 7.32 7.31 7.31 7.31 7.30 7.29 7.29 7.29 7.29 7.26 CDCl3 7.04 7.04 7.02
1.15 2.04 9.67 1.01
1.00 1.15 2.04 9.67 1.01
f1 (ppm)
5j

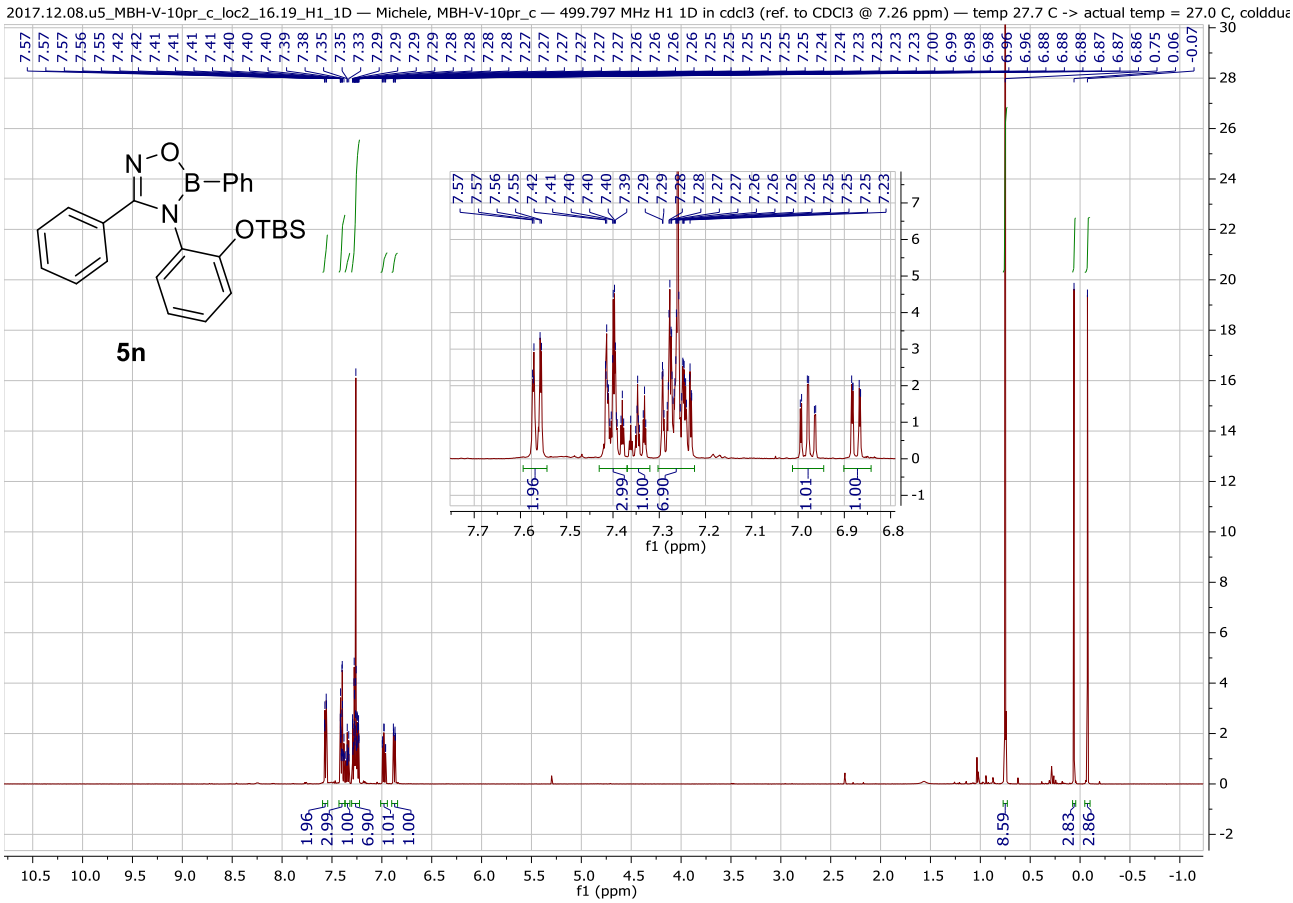

2018.01.09.m4_MBH-IV-102pr_c_loc42_09.49_C13_1D — Michele, MBH-IV-102pr_c — 100.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.0 C -> actual temp = 2
160.4 151.1 149.7 138.6 134.4 131.2 130.3 129.0 128.6 128.1 126.3 123.2 122.4
77.2 CDCl3
f1 (ppm)
5j

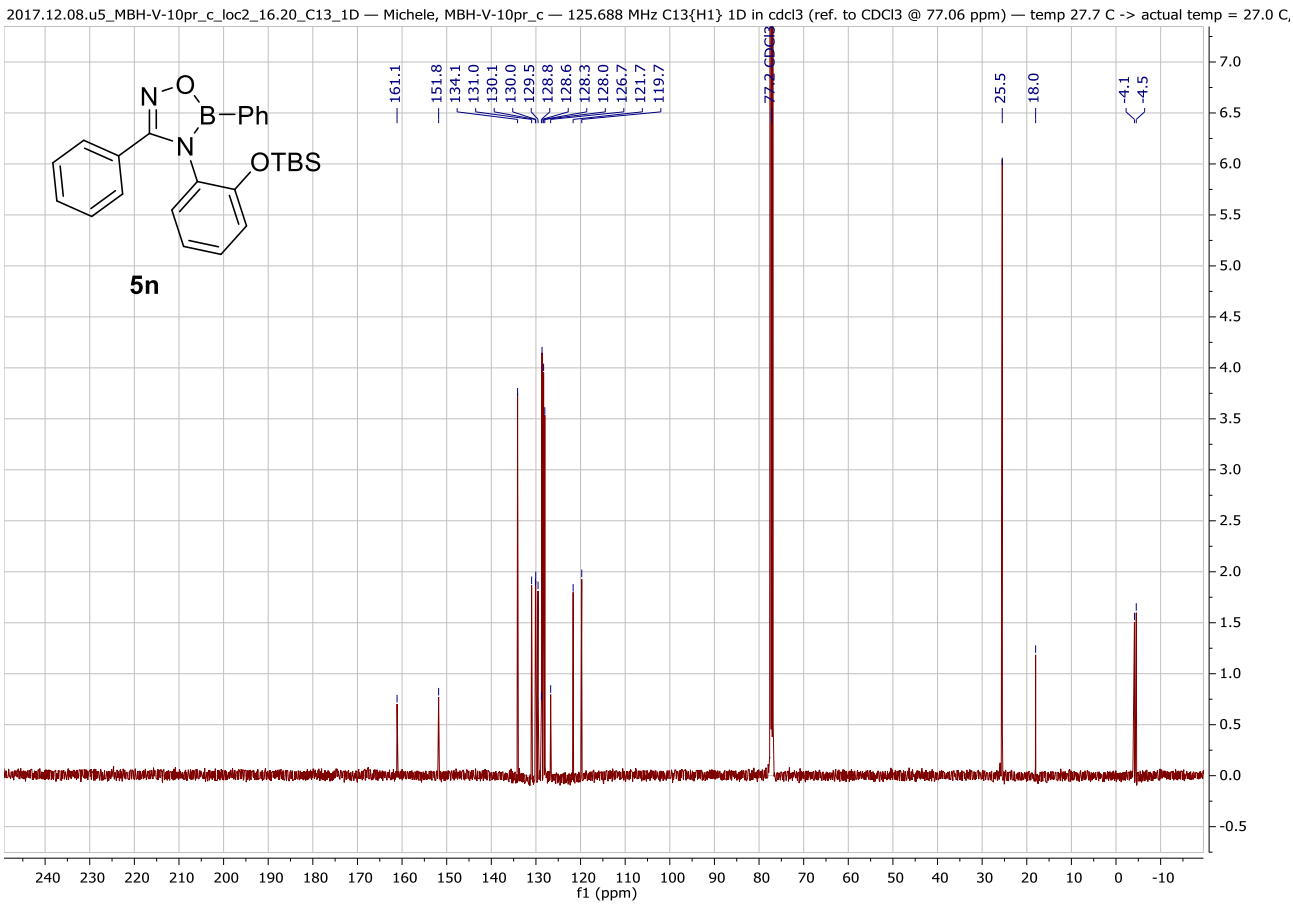

2018.01.04.mr4_MBH-V-46pr_clean_H1_1D — — 399.978 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 25.9 C -> actual temp = 27.0 C, onenmr probe —
5k
f1 (ppm)


2018.01.04.u5_MBH-V-46pr_2_c_loc2_11.57_C13_1D — Michele, MBH-V-46pr_2_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27
5k
f1 (ppm)

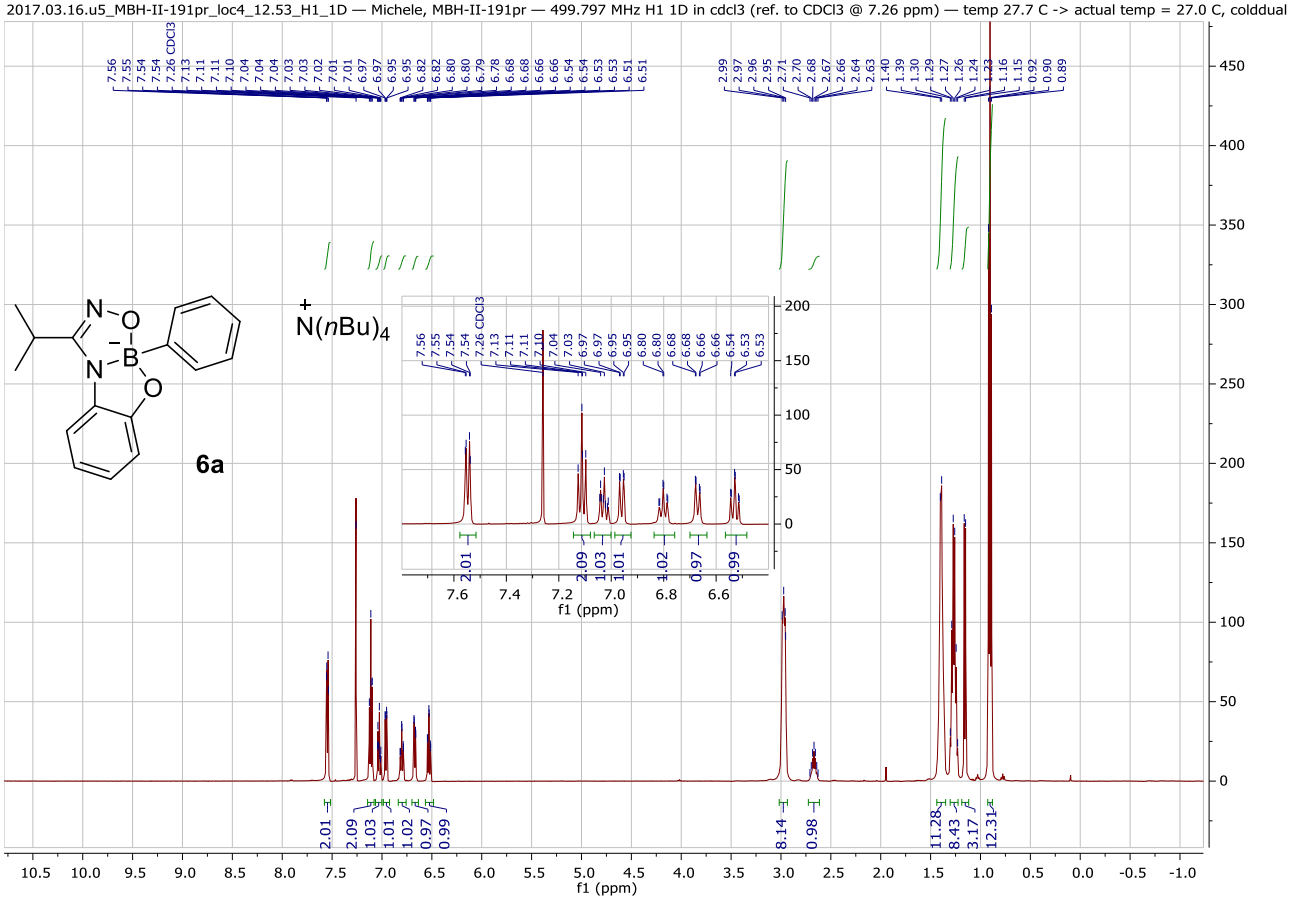

2017.03.13.u5_MBH-142pr_c_loc8_12.58_H1_1D — Michele, MBH-142pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddual
OTBS
5m
f1 (ppm)


2017.03.13.u5_MBH-142pr_c_loc8_12.59_C13_1D — Michele, MBH-142pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C, c
OTBS
5m
f1 (ppm)

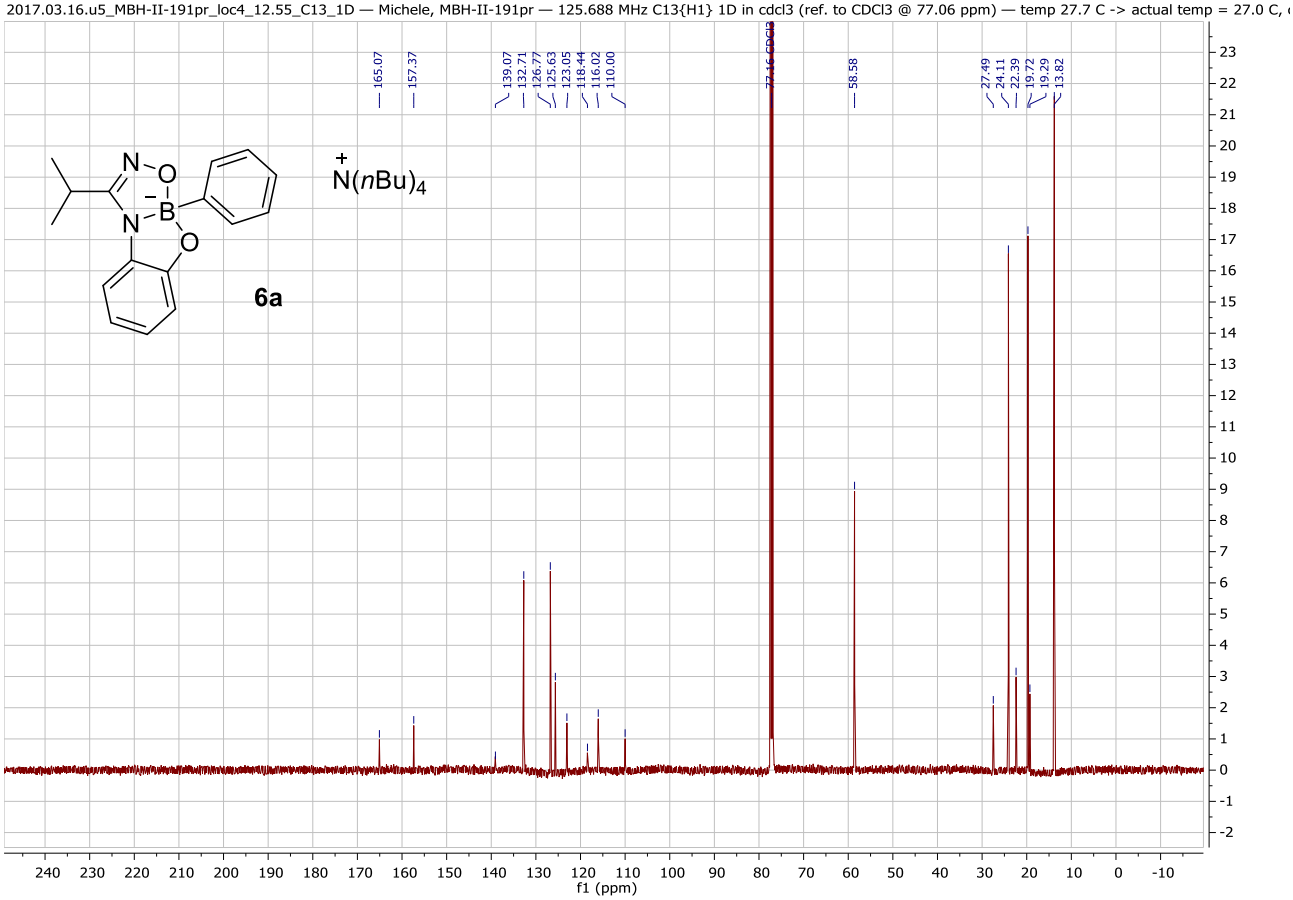

2017.12.08.u5_MBH-V-10pr_c_loc2_16.19_H1_1D — Michele, MBH-V-10pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddua
N
O
B—Ph
N
OTBS
5n
f1 (ppm)

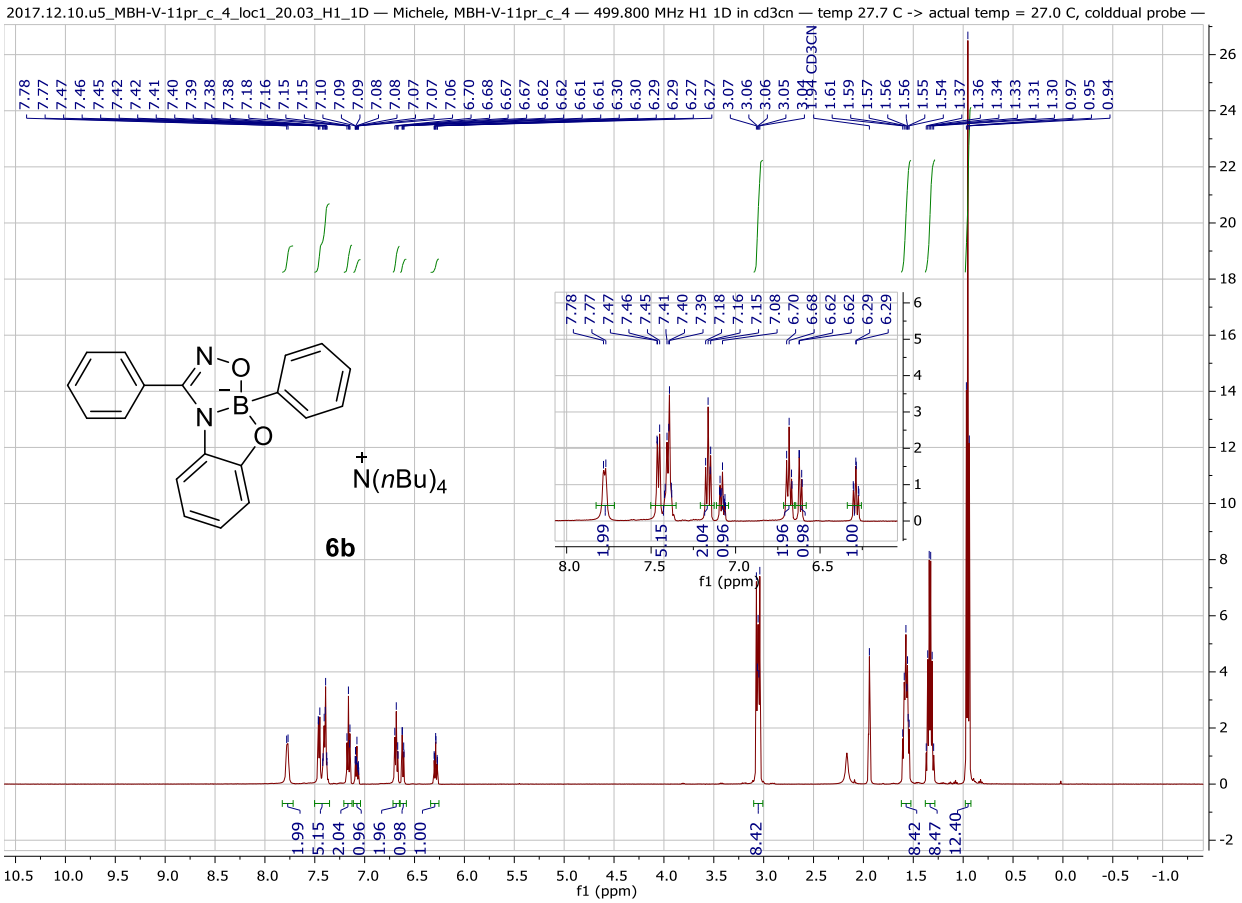

2017.12.08.u5_MBH-V-10pr_c_loc2_16.20_C13_1D — Michele, MBH-V-10pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C,
N
O
B—Ph
N
OTBS
5n
161.1
151.8
134.1
131.0
130.1
130.0
129.5
128.8
128.6
128.3
128.0
126.7
121.7
119.7
77.2 CDCl3
25.5
18.0
-4.1
-4.5
f1 (ppm)

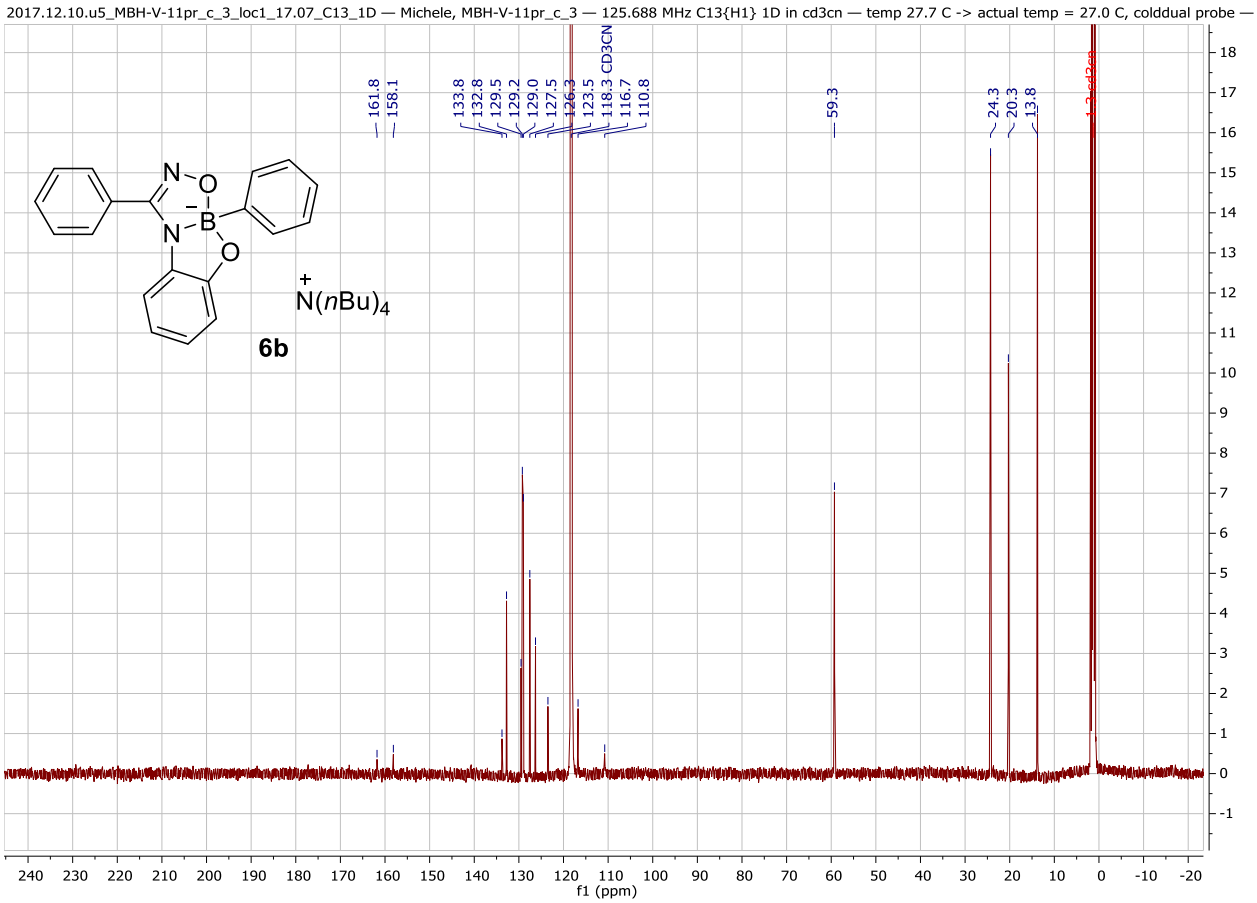
2017.03.16.u5_MBH-II-191pr_loc4_12.53_H1_1D — Michele, MBH-II-191pr — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, colddual

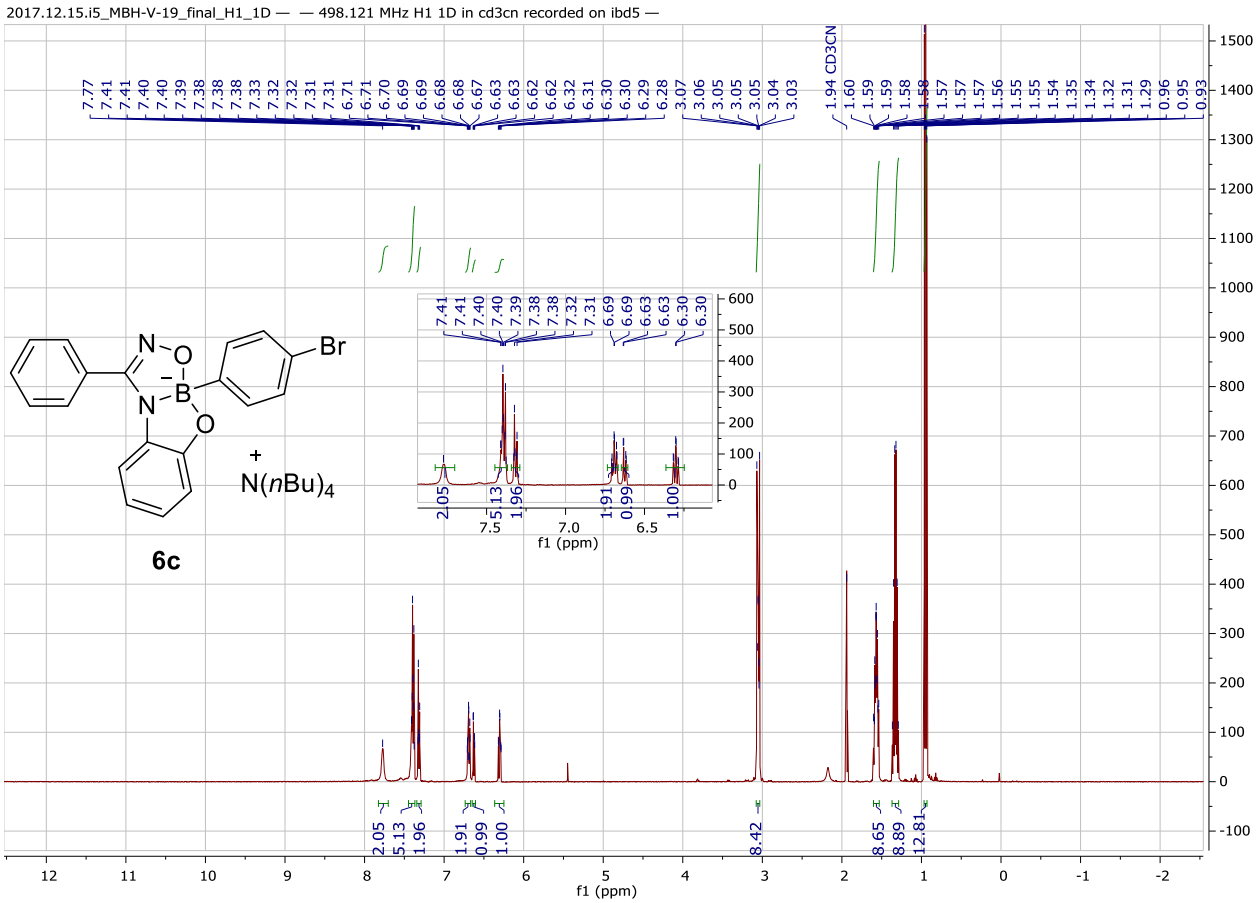


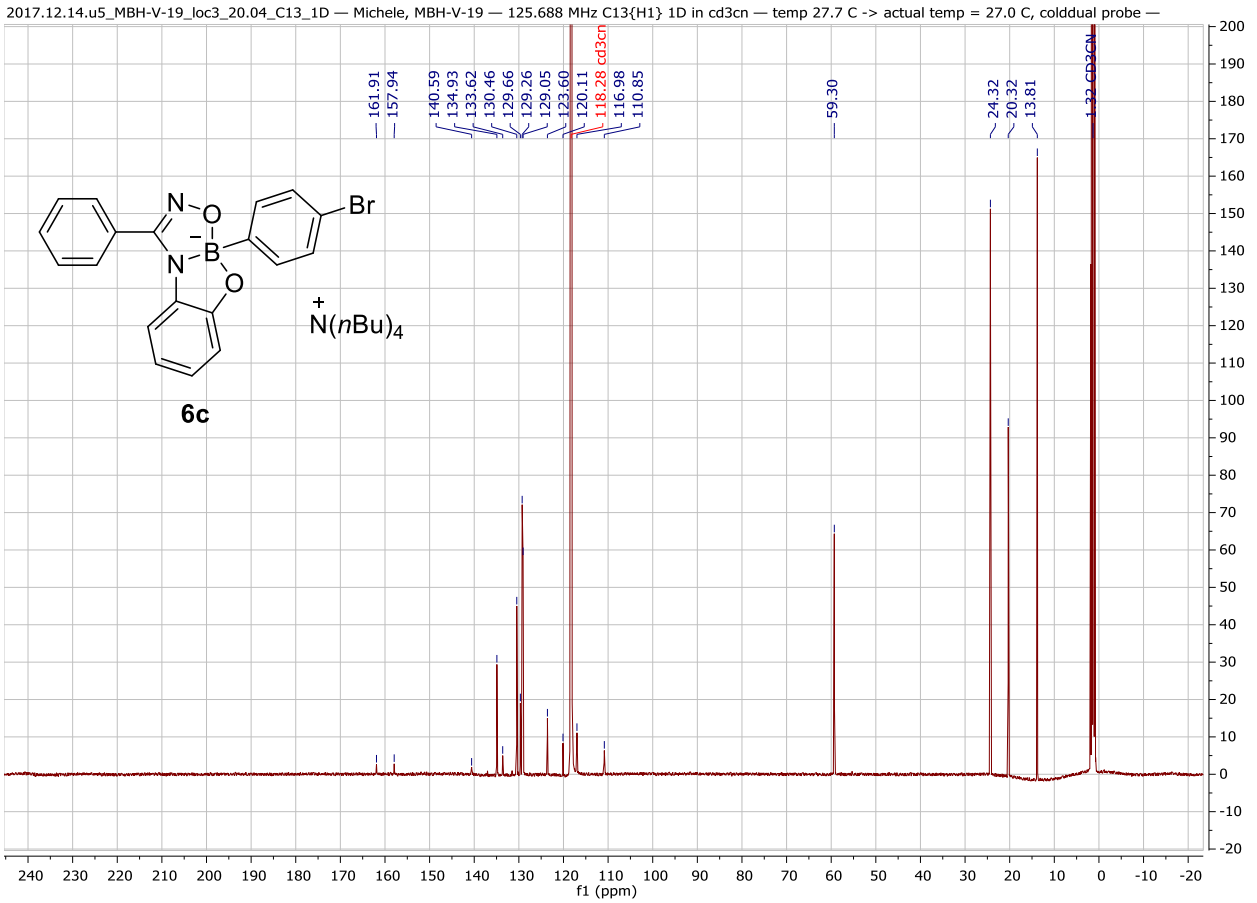
2017.03.16.u5_MBH-II-191pr_loc4_12.55_C13_1D — Michele, MBH-II-191pr — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27.0 C,

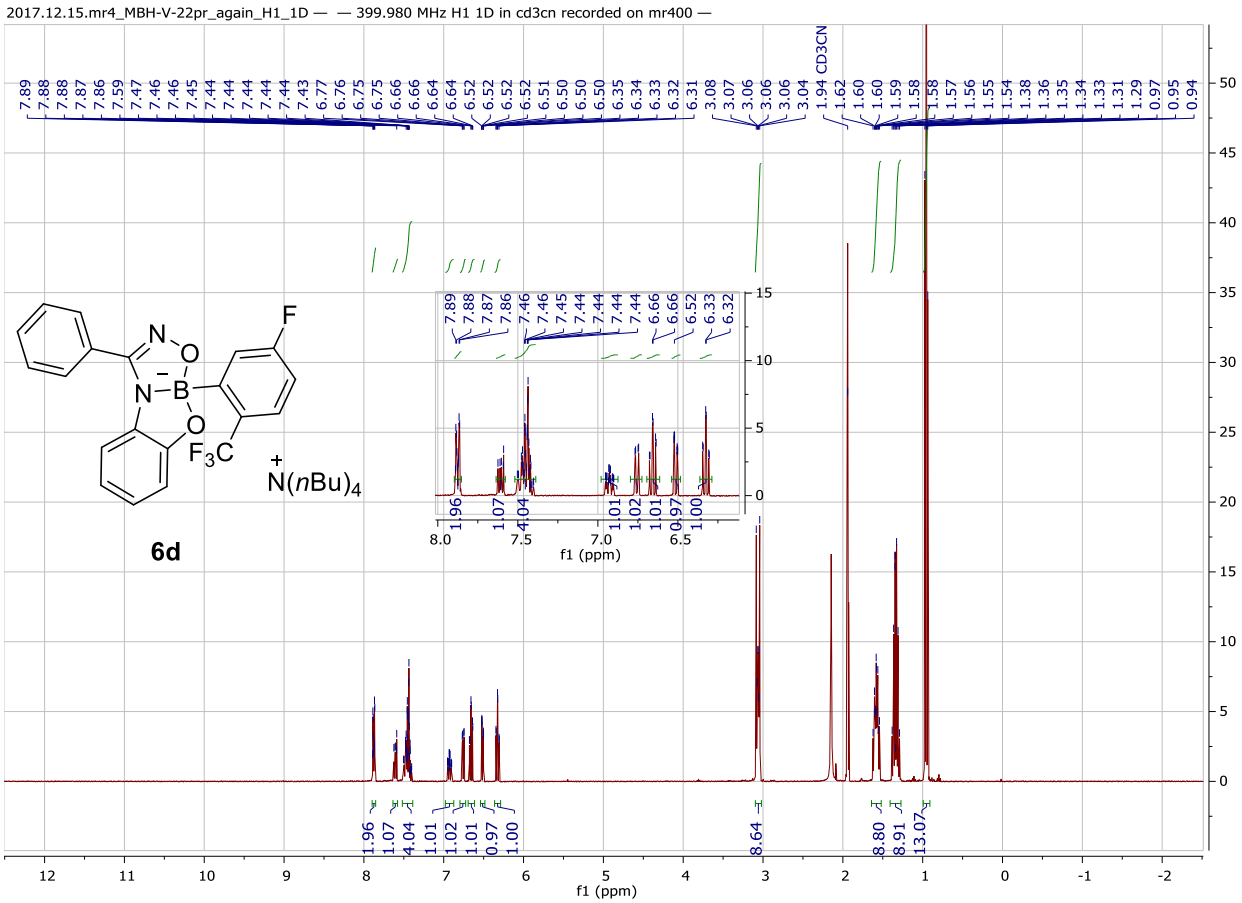


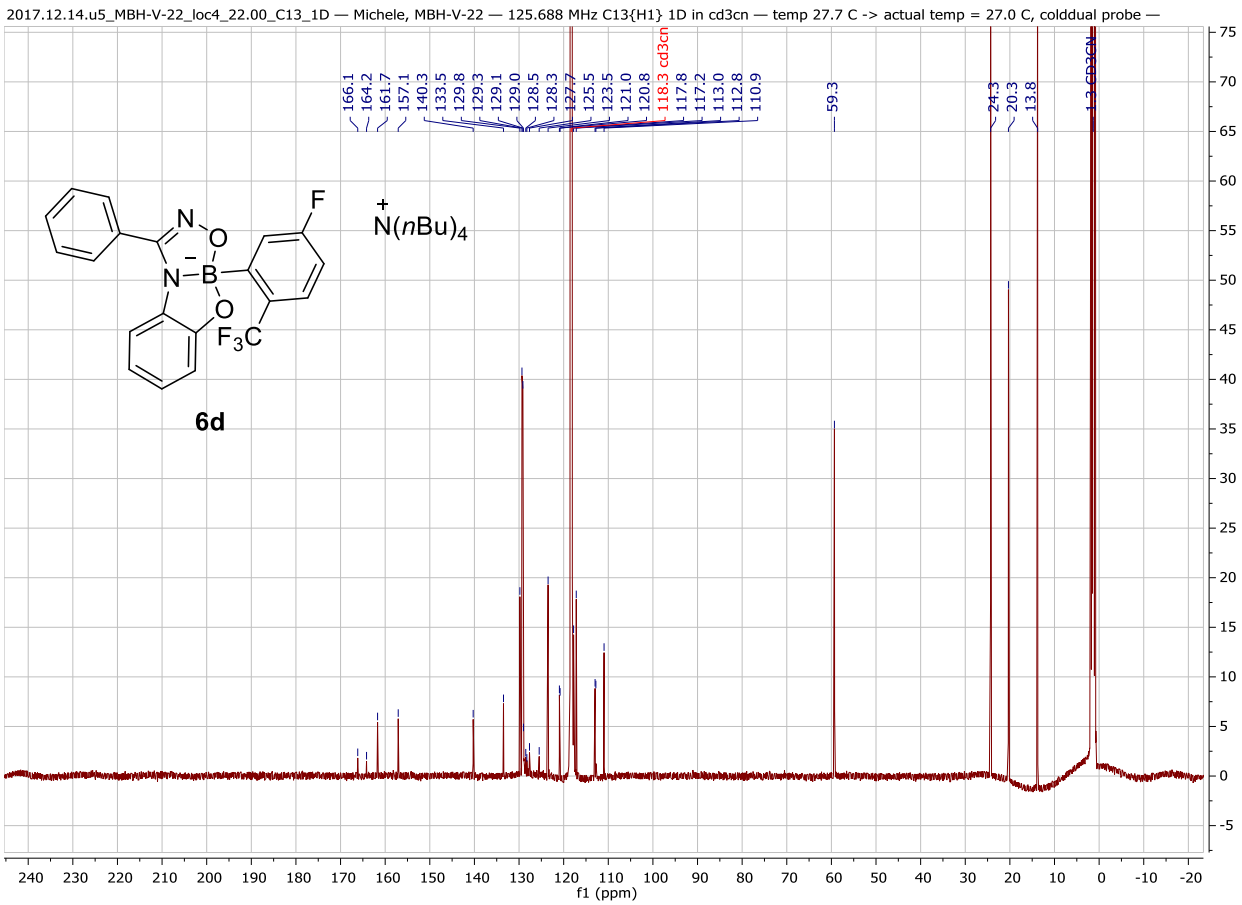

2017.12.10.u5_MBH-V-11pr_c_4_loc1_20.03_H1_1D — Michele, MBH-V-11pr_c_4 — 499.800 MHz H1 1D in cd3cn — temp 27.7 C -> actual temp = 27.0 C, colddual probe —
7.78 7.77 7.47 7.46 7.45 7.42 7.42 7.41 7.40 7.39 7.38 7.38 7.18 7.16 7.15 7.15 7.10 7.09 7.09 7.08 7.08 7.07 7.07 7.06 6.70 6.68 6.67 6.67 6.62 6.62 6.61 6.61 6.30 6.30 6.29 6.29 6.27 6.27 3.07 3.06 3.06 3.05 3.04 CD3CN 1.61 1.59 1.57 1.56 1.56 1.55 1.54 1.37 1.36 1.34 1.33 1.31 1.30 0.97 0.95 0.94
N(nBu)4
6b
1.99 5.15 2.04 0.96 1.96 0.98 1.00 8.42 8.42 8.47 12.40
f1 (ppm)

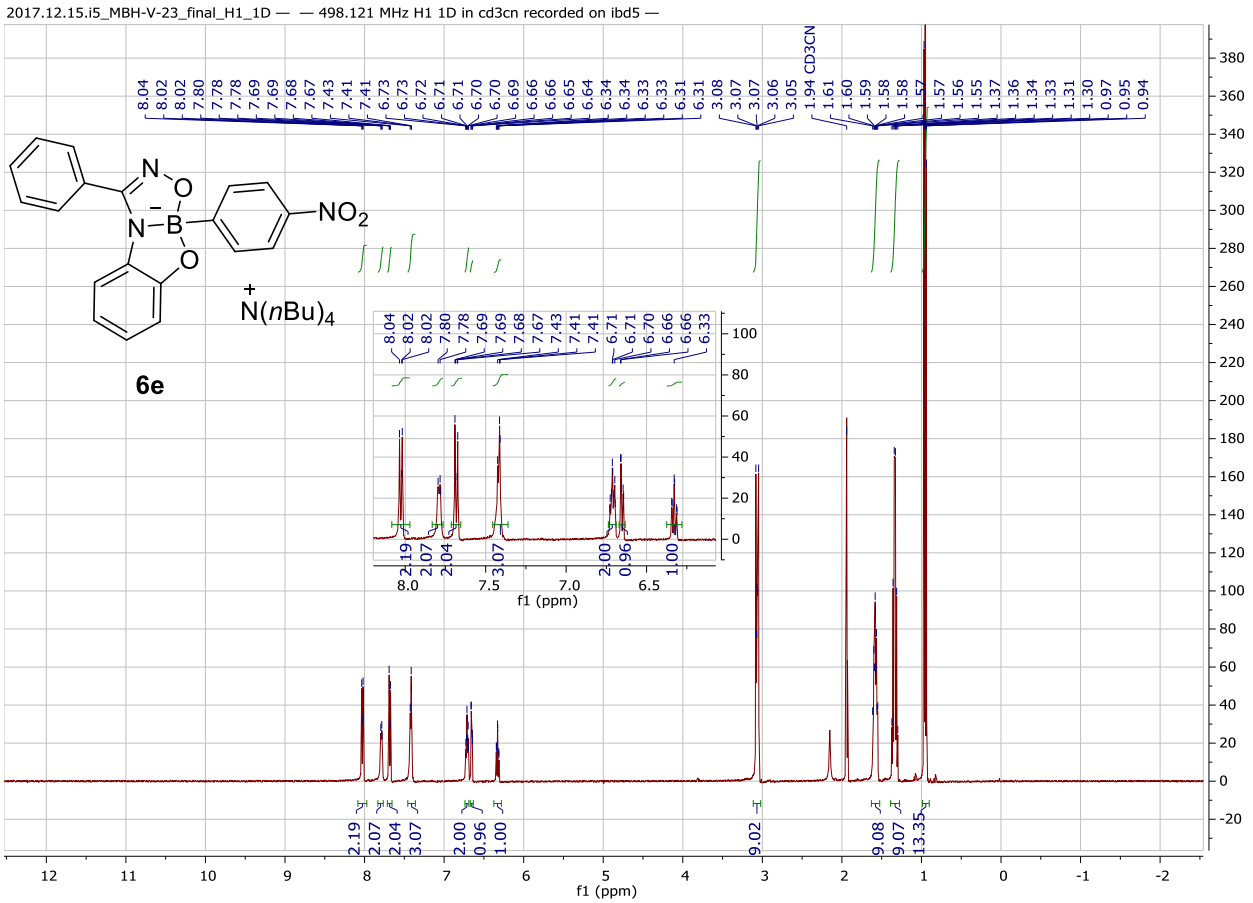

2017.12.10.u5_MBH-V-11pr_c_3_loc1_17.07_C13_1D — Michele, MBH-V-11pr_c_3 — 125.688 MHz C13{H1} 1D in cd3cn — temp 27.7 C -> actual temp = 27.0 C, colddual probe —
161.8 158.1 133.8 132.8 129.5 129.2 129.0 127.5 126.3 123.5 118.3 CD3CN 116.7 110.8 59.3 24.3 20.3 13.8
N(nBu)4
6b
f1 (ppm)

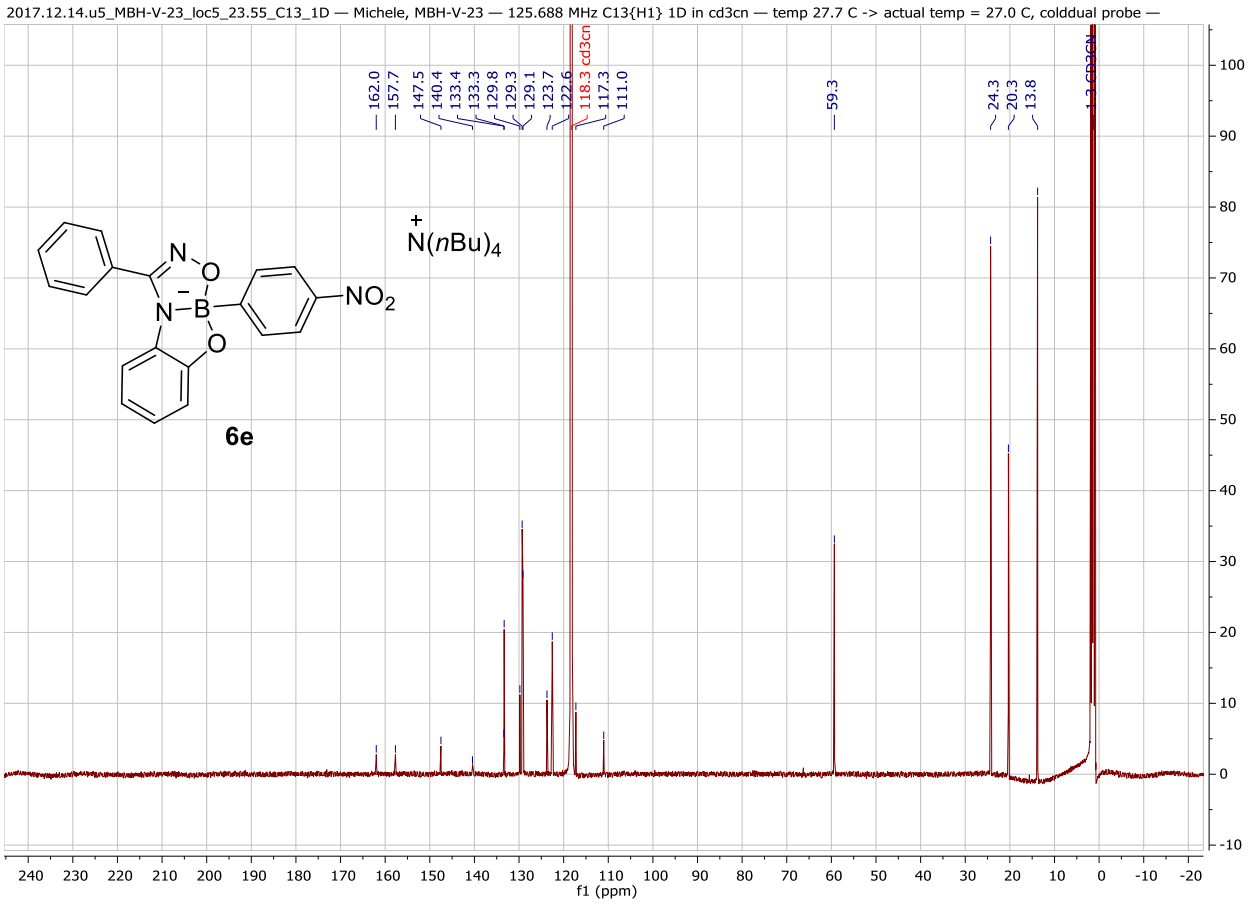

2017.12.15.i5_MBH-V-19_final_H1_1D — — 498.121 MHz H1 1D in cd3cn recorded on ibd5 —
Br
N(nBu)4
6c
f1 (ppm)

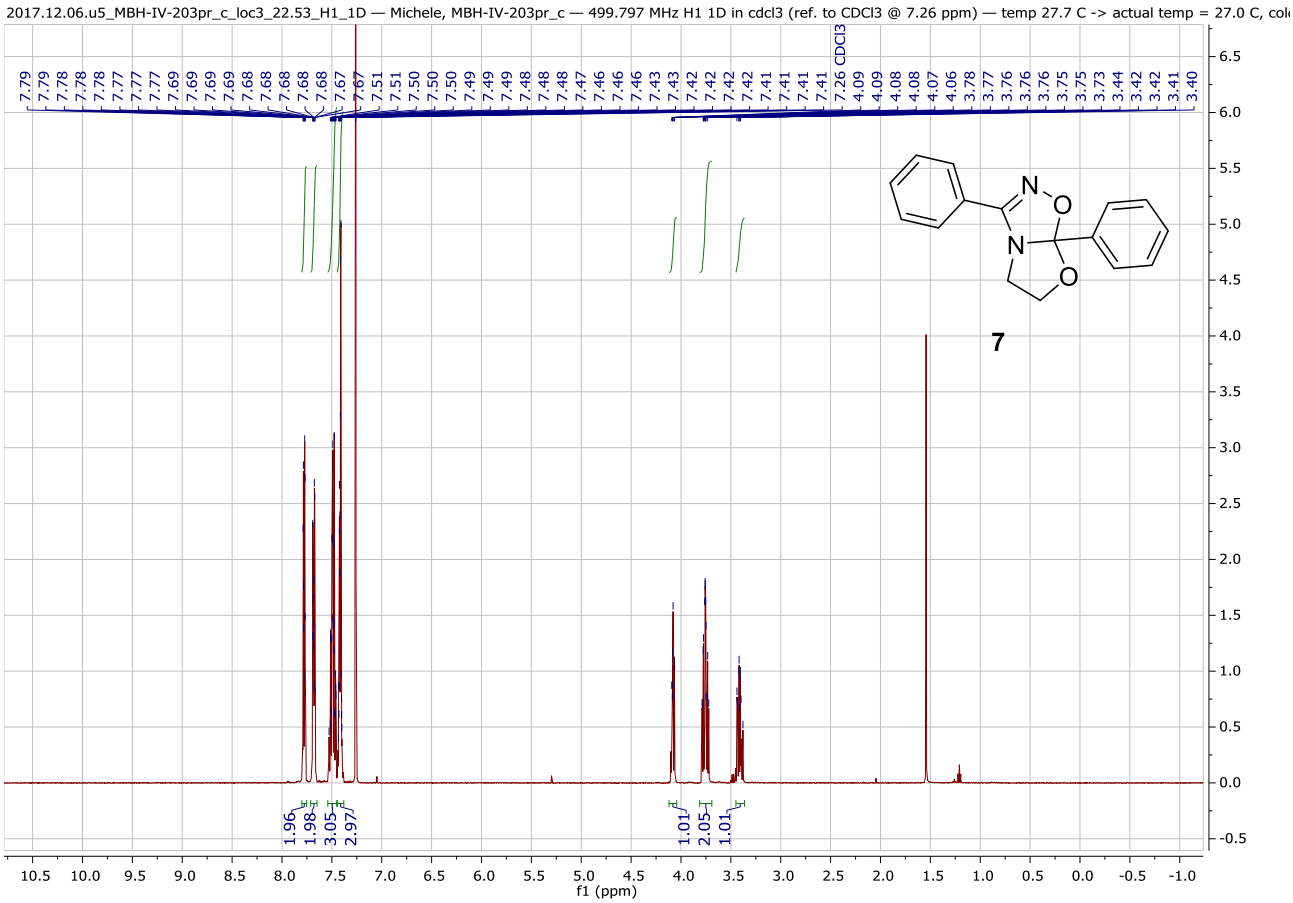

2017.12.14.u5_MBH-V-19_loc3_20.04_C13_1D — Michele, MBH-V-19 — 125.688 MHz C13{H1} 1D in cd3cn — temp 27.7 C -> actual temp = 27.0 C, colddual probe —
Br
N(nBu)4
6c
f1 (ppm)

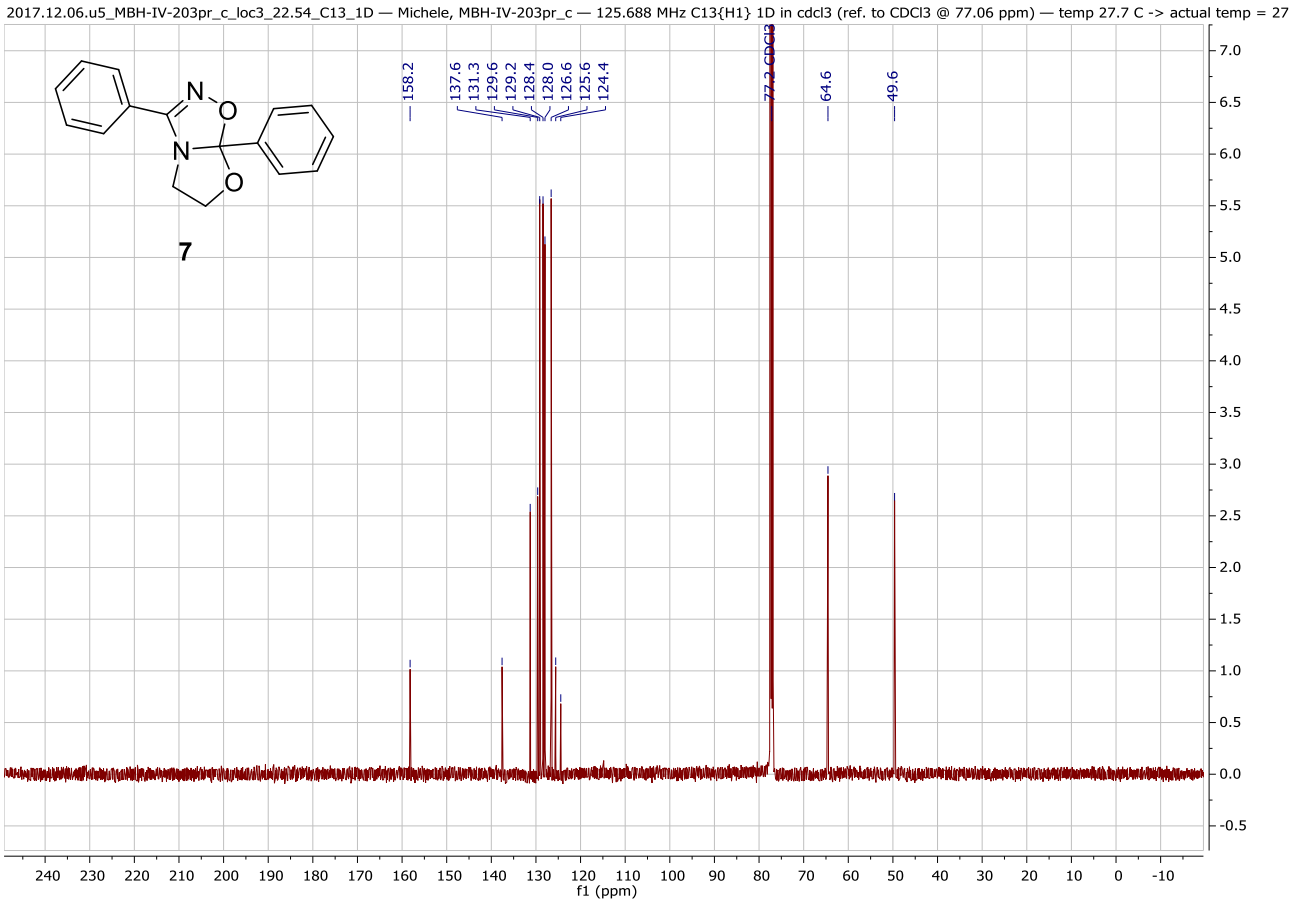

2017.12.15.mr4_MBH-V-22pr_again_H1_1D — — 399.980 MHz H1 1D in cd3cn recorded on mr400 —
6d
f1 (ppm)

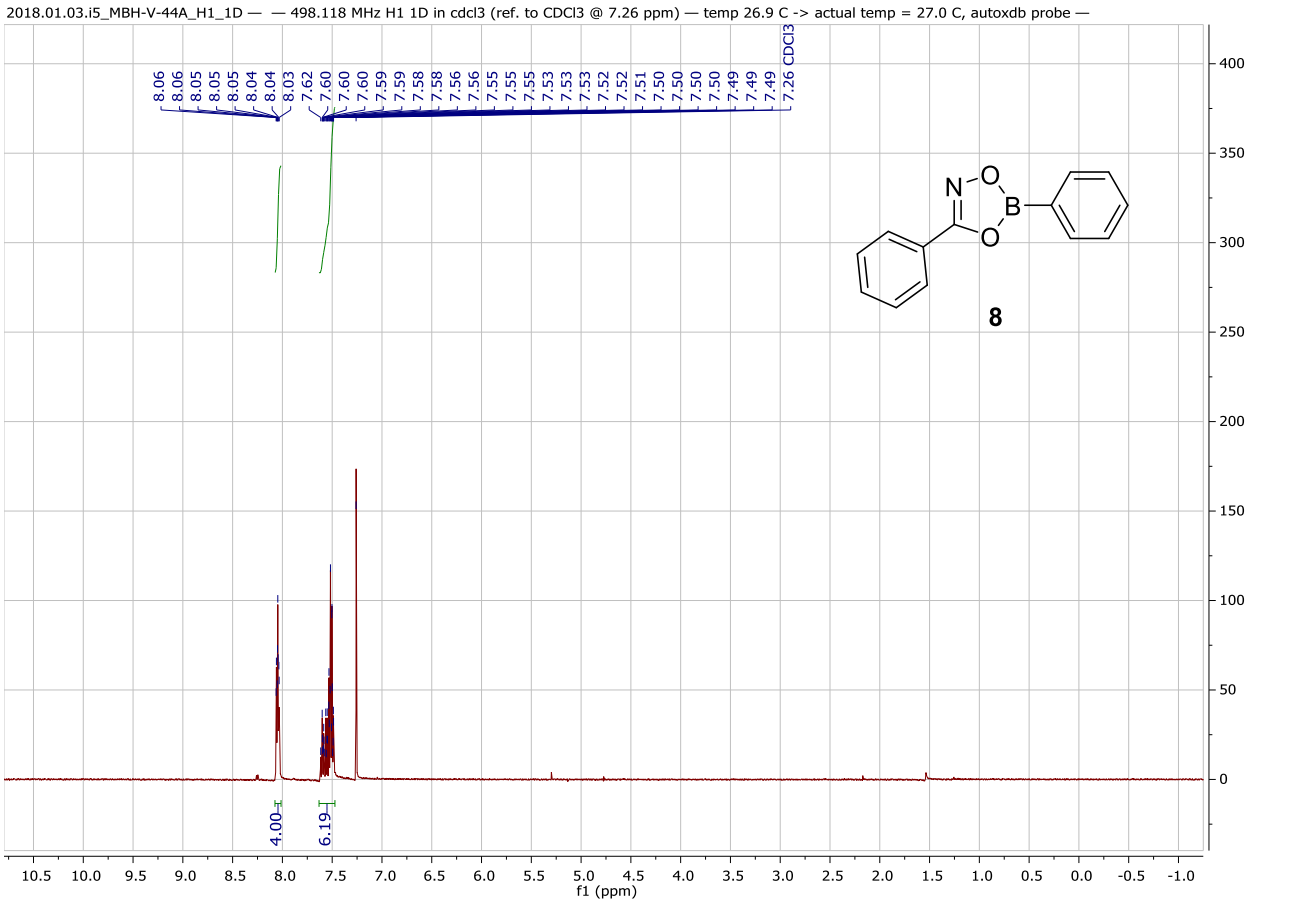

2017.12.14.u5_MBH-V-22_loc4_22.00_C13_1D — Michele, MBH-V-22 — 125.688 MHz C13{H1} 1D in cd3cn — temp 27.7 C -> actual temp = 27.0 C, colddual probe —
6d
f1 (ppm)

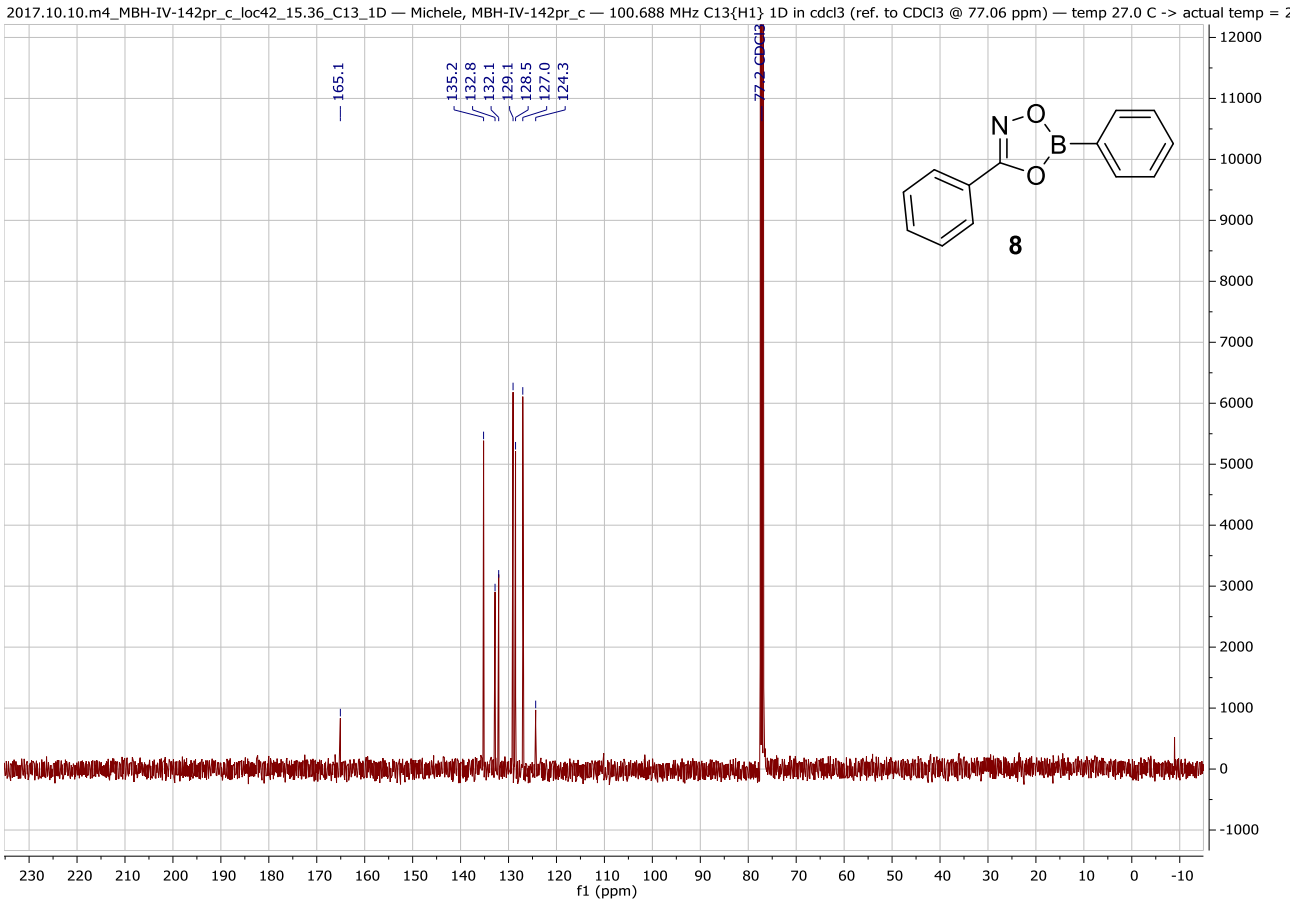

2017.12.15.i5_MBH-V-23_final_H1_1D — — 498.121 MHz H1 1D in cd3cn recorded on ibd5 —
NO2
N(nBu)4
6e
f1 (ppm)


2017.12.14.u5_MBH-V-23_loc5_23.55_C13_1D — Michele, MBH-V-23 — 125.688 MHz C13{H1} 1D in cd3cn — temp 27.7 C -> actual temp = 27.0 C, colddual probe —
NO2
N(nBu)4
6e
f1 (ppm)


2017.12.06.u5_MBH-IV-203pr_c_loc3_22.53_H1_1D — Michele, MBH-IV-203pr_c — 499.797 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 27.7 C -> actual temp = 27.0 C, col
CDCl3
7
1.96
1.98
3.05
2.97
1.01
2.05
1.01
f1 (ppm)


2017.12.06.u5_MBH-IV-203pr_c_loc3_22.54_C13_1D — Michele, MBH-IV-203pr_c — 125.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.7 C -> actual temp = 27
158.2
137.6
131.3
129.6
129.2
128.4
128.0
126.6
125.6
124.4
77.2 CDCl3
64.6
49.6
7
f1 (ppm)


2018.01.03.i5_MBH-V-44A_H1_1D — — 498.118 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm) — temp 26.9 C -> actual temp = 27.0 C, autoxdb probe —
8.06 8.06 8.05 8.05 8.05 8.04 8.04 8.03 7.62 7.60 7.60 7.60 7.59 7.59 7.58 7.58 7.56 7.56 7.55 7.55 7.55 7.53 7.53 7.53 7.52 7.52 7.51 7.50 7.50 7.50 7.50 7.49 7.49 7.49 7.26 CDCl3
4.00
6.19
f1 (ppm)
8


2017.10.10.m4_MBH-IV-142pr_c_loc42_15.36_C13_1D — Michele, MBH-IV-142pr_c — 100.688 MHz C13{H1} 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm) — temp 27.0 C -> actual temp = 2
165.1
135.2 132.8 132.1 129.1 128.5 127.0 124.3
77.2 CDCl3
f1 (ppm)
8

9 X-ray crystallographic reports

STRUCTURE REPORT (2)

XCL Code: DGH1703 **Date:** 3 October 2017

Compound: 4-(2-Methyl-4-phenyl-1,3,5,2-oxadiazaborol-3(2*H*)-yl)benzenesulfonamide•0.5*n*-hexane

Formula: $C_{17}H_{21}BN_3O_3S$ ($C_{14}H_{14}BN_3O_3S$•$0.5C_6H_{14}$)

Supervisor: D. G. Hall **Crystallographer:** R. McDonald

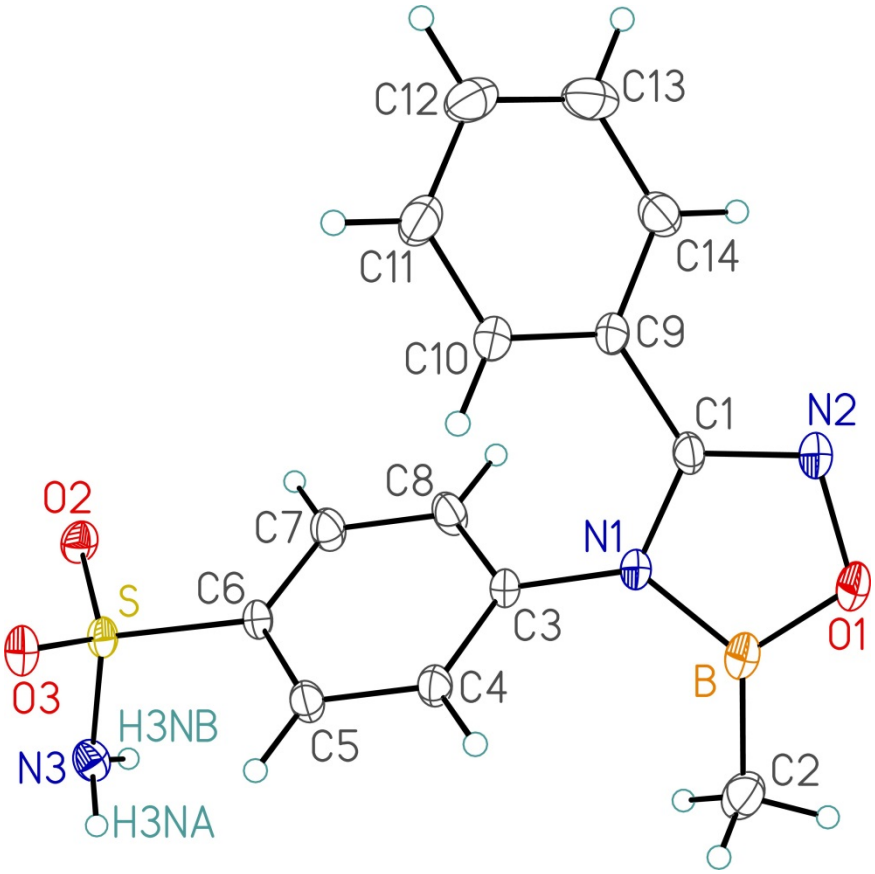
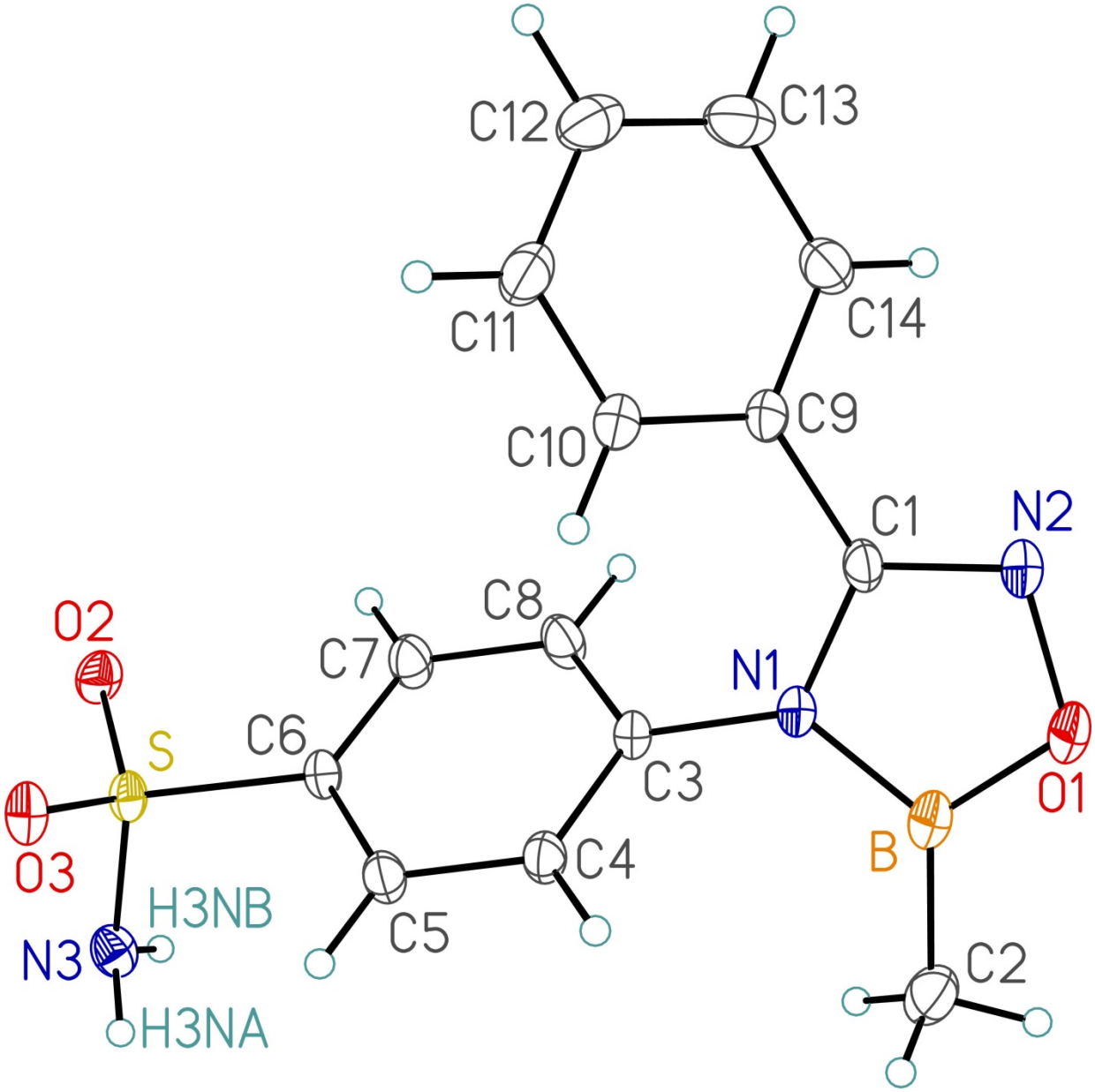
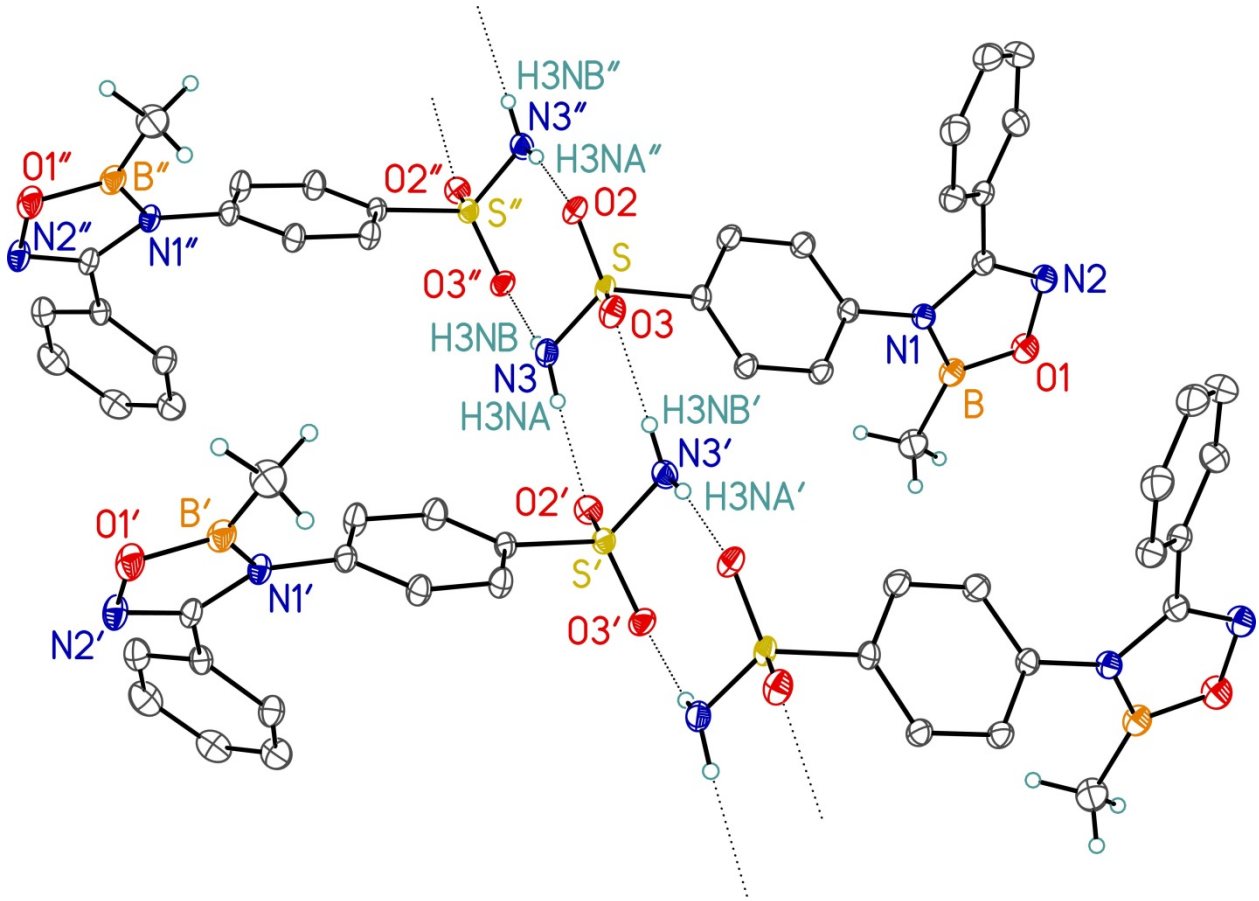


Figure Legends

Figure 1. Perspective view of the 4-(2-methyl-4-phenyl-1,3,5,2-oxadiazaborol-3(2*H*)-yl)benzenesulfonamide molecule showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Illustration of hydrogen-bonded interactions between adjacent molecules within the crystal lattice. Primed atoms are related to unprimed ones via the crystallographic symmetry operation ($1–x$, $^{1}/_{2}+y$, $^{1}/_{2}–z$). Douple-primed atoms are related to unprimed ones via the crystallographic symmetry operation ($1–x$, $^{-1}/_{2}+y$, $^{1}/_{2}–z$). The chain propagates in a direction parallel to the crystal unit cell's *b* axis.


C12
C13
C11
C14
C10
C9
C1
N2
C8
C7
N1
O2
S
C6
C3
O1
O3
C4
B
H3NB
C5
N3
C2
H3NA


H3NB″
N3″
H3NA″
O1″
B″
O2″
S″
O2
N2″
N1″
O3″
S
N2
H3NB
O3
N1
N3
O1
H3NA
B
H3NB′
N3′
O1′
B′
O2′
H3NA′
S′
N1′
O3′
N2′

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data

formula	$C_{17}H_{21}BN_3O_3S$
formula weight	358.24
crystal dimensions (mm)	0.28 × 0.13 × 0.12
crystal system	monoclinic
space group	$P2_1/c$ (No. 14)
unit cell parameters[a]	
a (Å)	12.7713 (2)
b (Å)	7.93520 (14)
c (Å)	17.9462 (3)
β (deg)	94.7903 (7)
V (Å^3)	1812.36 (5)
Z	4
ρ_{calcd} (g cm^{-3})	1.313
μ (mm^{-1})	1.762

B. Data Collection and Refinement Conditions

diffractometer	Bruker D8/APEX II CCD[b]
radiation (λ [Å])	Cu Kα (1.54178) (microfocus source)
temperature (°C)	–100
scan type	ω and ϕ scans (1.0°) (5 s exposures)
data collection 2θ limit (deg)	147.77
total data collected	12464 (-15 ≤ h ≤ 15, -9 ≤ k ≤ 9, -22 ≤ l ≤ 22)
independent reflections	3667 (R_{int} = 0.0312)
number of observed reflections (*NO*)	3375 [$F_o^2 \geq 2\sigma(F_o^2)$]
structure solution method	intrinsic phasing (*SHELXT-2014*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	1.0000–0.6717

data/restraints/parameters	3667 / 9[e] / 227
goodness-of-fit (S)[f] [all data]	1.080
final R indices[g]	
R_1 [$F_o^2 \geq 2\sigma(F_o^2)$]	0.0553
wR_2 [all data]	0.1704
largest difference peak and hole	0.918 and –0.492 e Å^{-3}

[a]Obtained from least-squares refinement of 9862 reflections with $6.94° < 2\theta < 147.52°$.

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[c]Sheldrick, G. M. *Acta Crystallogr.* **2015**, *A71*, 3–8.

[d]Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[e]Distances within the inversion-disordered solvent *n*-hexane molecule were constrained during refinement: d(C1S–C2S) = d(C2S–C3S) = d(C3S–C4S) = d(C4S–C5S) = d(C5S–C6S) = 1.52(1) Å; d(C1S···C3S) = d(C2S···C4S) = d(C3S···C5S) = d(C4S···C6S) = 2.52(1) Å.

[f]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0946P)^2 + 1.7529P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[g]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

(a) atoms of 4-(2-methyl-4-phenyl-1,3,5,2-oxadiazaborol-3(2H)-yl)benzenesulfonamide

Atom	*x*	*y*	*z*	U_{eq}, Å^2
S	0.43007(4)	0.21218(7)	0.27000(3)	0.0262(2)*
O1	-0.01279(15)	0.3062(3)	-0.08233(9)	0.0397(4)*
O2	0.41776(13)	0.0542(2)	0.30713(9)	0.0330(4)*
O3	0.42576(13)	0.3645(2)	0.31286(9)	0.0337(4)*
N1	0.08786(15)	0.2745(2)	0.02552(10)	0.0280(4)*
N2	-0.07993(17)	0.2721(3)	-0.02333(11)	0.0355(5)*
N3	0.54288(16)	0.2092(3)	0.23615(12)	0.0321(5)*
C1	-0.01772(18)	0.2549(3)	0.03673(12)	0.0275(5)*
C2	0.1864(2)	0.3463(4)	-0.09836(14)	0.0451(6)*
C3	0.17176(17)	0.2550(3)	0.08221(12)	0.0255(4)*
C4	0.24025(18)	0.3894(3)	0.09790(12)	0.0297(5)*
C5	0.32049(18)	0.3744(3)	0.15460(13)	0.0300(5)*
C6	0.33051(17)	0.2258(3)	0.19547(12)	0.0250(4)*
C7	0.26400(19)	0.0905(3)	0.17935(14)	0.0338(5)*
C8	0.18426(19)	0.1049(3)	0.12190(14)	0.0347(5)*
C9	-0.05960(18)	0.2271(3)	0.11019(12)	0.0282(5)*
C10	-0.02020(19)	0.3162(3)	0.17327(13)	0.0334(5)*
C11	-0.0628(2)	0.2933(4)	0.24094(14)	0.0408(6)*
C12	-0.1449(2)	0.1823(4)	0.24625(16)	0.0444(7)*
C13	-0.1854(2)	0.0945(4)	0.18412(18)	0.0473(7)*
C14	-0.14317(19)	0.1159(3)	0.11542(15)	0.0379(6)*
B	0.0907(2)	0.3104(4)	-0.05254(15)	0.0342(6)*
H3NA	0.559(3)	0.303(5)	0.217(2)	0.050(10)
H3NB	0.555(3)	0.124(4)	0.2121(19)	0.043(8)

(b) solvent n-hexane atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C1S[a]	0.5451(17)	0.3895(19)	0.0095(14)	0.219(5)
C2S[a]	0.5997(19)	0.2286(19)	-0.0088(16)	0.219(5)
C3S[a]	0.5457(16)	0.0739(18)	0.0182(12)	0.219(5)
C4S[a]	0.5537(14)	-0.084(2)	-0.0216(14)	0.219(5)
C5S[a]	0.4547(18)	-0.197(2)	-0.0242(14)	0.219(5)
C6S[a]	0.3840(17)	-0.144(3)	0.0362(13)	0.219(5)

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$. [a]Refined with an occupancy factor of 0.5 and a common isotropic displacement parameter.

Table 3. Selected Interatomic Distances (Å)

(a) within 4-(2-methyl-4-phenyl-1,3,5,2-oxadiazaborol-3(2H)-yl)benzenesulfonamide

Atom1	Atom2	Distance	Atom1	Atom2	Distance
S	O2	1.4345(17)	C3	C4	1.393(3)
S	O3	1.4361(17)	C3	C8	1.390(3)
S	N3	1.610(2)	C4	C5	1.388(3)
S	C6	1.770(2)	C5	C6	1.389(3)
O1	N2	1.443(3)	C6	C7	1.384(3)
O1	B	1.384(3)	C7	C8	1.392(3)
N1	C1	1.389(3)	C9	C10	1.393(3)
N1	C3	1.423(3)	C9	C14	1.394(3)
N1	B	1.433(3)	C10	C11	1.383(3)
N2	C1	1.292(3)	C11	C12	1.379(4)
C1	C9	1.480(3)	C12	C13	1.379(5)
C2	B	1.555(4)	C13	C14	1.397(4)

(b) within the solvent n-hexane molecule

Atom1	Atom2	Distance	Atom1	Atom2	Distance
C1S	C2S	1.505(10)[a]	C4S	C5S	1.548(9)[a]
C2S	C3S	1.507(10)[a]	C5S	C6S	1.528(10)[a]
C3S	C4S	1.450(9)[a]			

[a]Distances were constrained to a target value of 1.52(1) Å during refinement.

Table 4. Selected Interatomic Angles (deg)

(a) within 4-(2-methyl-4-phenyl-1,3,5,2-oxadiazaborol-3(2H)-yl)benzenesulfonamide

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
O2	S	O3	118.42(10)	N1	C3	C4	118.9(2)
O2	S	N3	107.38(11)	N1	C3	C8	120.4(2)
O2	S	C6	107.74(10)	C4	C3	C8	120.6(2)
O3	S	N3	106.86(11)	C3	C4	C5	119.7(2)
O3	S	C6	107.23(10)	C4	C5	C6	119.4(2)
N3	S	C6	108.96(11)	S	C6	C5	118.66(16)
N2	O1	B	109.15(18)	S	C6	C7	120.01(17)
C1	N1	C3	124.51(19)	C5	C6	C7	121.3(2)
C1	N1	B	105.55(19)	C6	C7	C8	119.3(2)
C3	N1	B	129.9(2)	C3	C8	C7	119.7(2)
O1	N2	C1	105.69(19)	C1	C9	C10	121.0(2)
N1	C1	N2	113.8(2)	C1	C9	C14	119.3(2)
N1	C1	C9	125.0(2)	C10	C9	C14	119.6(2)
N2	C1	C9	121.1(2)	C9	C10	C11	120.3(2)
				C10	C11	C12	120.1(3)
				C11	C12	C13	120.2(3)
				C12	C13	C14	120.4(3)
				C9	C14	C13	119.4(2)
				O1	B	N1	105.7(2)
				O1	B	C2	124.6(2)
				N1	B	C2	129.7(2)

(b) within the solvent n-hexane molecule

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C1S	C2S	C3S	112.8(10)[a]	C3S	C4S	C5S	115.2(10)[a]
C2S	C3S	C4S	119.5(11)[a]	C4S	C5S	C6S	110.4(8)[a]

[a]Angle contains distances constrained during refinement: d(C1S–C2S) = d(C2S–C3S) = d(C3S–C4S) = d(C4S–C5S) = d(C5S–C6S) = 1.52(1) Å; d(C1S···C3S) = d(C2S···C4S) = d(C3S···C5S) = d(C4S···C6S) = 2.52(1) Å.

Table 5. Hydrogen-Bonded Interactions

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	∠D–H···A (deg)
N3–H3NA···O2[a]	0.85(4)	2.07(4)	2.901(3)	167(3)
N3–H3NB···O3[b]	0.82(3)	2.12(4)	2.911(3)	159(3)

[a]At 1–*x*, $^{1}/_{2}$+*y*, $^{1}/_{2}$–*z*.

[b]At 1–*x*, $^{-1}/_{2}$+*y*, $^{1}/_{2}$–*z*.

Table 6. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
O2	S	C6	C5	174.75(18)	N1	C1	C9	C14	-141.4(2)
O2	S	C6	C7	-4.7(2)	N2	C1	C9	C10	-134.4(3)
O3	S	C6	C5	46.3(2)	N2	C1	C9	C14	42.4(3)
O3	S	C6	C7	-133.2(2)	N1	C3	C4	C5	178.0(2)
N3	S	C6	C5	-69.0(2)	C8	C3	C4	C5	-1.2(4)
N3	S	C6	C7	111.5(2)	N1	C3	C8	C7	-177.2(2)
B	O1	N2	C1	-1.0(3)	C4	C3	C8	C7	2.0(4)
N2	O1	B	N1	1.4(3)	C3	C4	C5	C6	-0.7(4)
N2	O1	B	C2	-179.1(3)	C4	C5	C6	S	-177.50(18)
C3	N1	C1	N2	-177.4(2)	C4	C5	C6	C7	2.0(4)
C3	N1	C1	C9	6.2(4)	S	C6	C7	C8	178.24(19)
B	N1	C1	N2	0.6(3)	C5	C6	C7	C8	-1.2(4)
B	N1	C1	C9	-175.8(2)	C6	C7	C8	C3	-0.8(4)
C1	N1	C3	C4	-122.1(2)	C1	C9	C10	C11	177.6(2)
C1	N1	C3	C8	57.1(3)	C14	C9	C10	C11	0.8(4)
B	N1	C3	C4	60.4(3)	C1	C9	C14	C13	-177.6(2)
B	N1	C3	C8	-120.4(3)	C10	C9	C14	C13	-0.7(4)
C1	N1	B	O1	-1.2(3)	C9	C10	C11	C12	-0.2(4)
C1	N1	B	C2	179.3(3)	C10	C11	C12	C13	-0.4(4)
C3	N1	B	O1	176.7(2)	C11	C12	C13	C14	0.5(4)
C3	N1	B	C2	-2.8(5)	C12	C13	C14	C9	0.1(4)
O1	N2	C1	N1	0.2(3)	C1S	C2S	C3S	C4S	151(2)[a]
O1	N2	C1	C9	176.8(2)	C2S	C3S	C4S	C5S	-145(2)[a]
N1	C1	C9	C10	41.7(4)	C3S	C4S	C5S	C6S	-18(3)[a]

[a]Angle contains distances constrained during refinement: d(C1S–C2S) = d(C2S–C3S) = d(C3S–C4S) = d(C4S–C5S) = d(C5S–C6S) = 1.52(1) Å; d(C1S···C3S) = d(C2S···C4S) = d(C3S···C5S) = d(C4S···C6S) = 2.52(1) Å.

Table 7. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S	0.0268(3)	0.0272(3)	0.0230(3)	0.00082(18)	-0.0063(2)	0.00362(19)
O1	0.0417(10)	0.0556(12)	0.0206(8)	0.0011(7)	-0.0055(7)	-0.0017(8)
O2	0.0374(9)	0.0312(9)	0.0294(8)	0.0072(7)	-0.0033(7)	0.0050(7)
O3	0.0392(9)	0.0328(9)	0.0273(8)	-0.0056(7)	-0.0072(7)	0.0037(7)
N1	0.0283(10)	0.0343(10)	0.0204(9)	-0.0002(7)	-0.0041(7)	-0.0030(7)
N2	0.0341(11)	0.0464(12)	0.0245(10)	0.0012(8)	-0.0070(8)	-0.0044(9)
N3	0.0280(10)	0.0302(11)	0.0368(11)	0.0014(9)	-0.0043(8)	0.0036(8)
C1	0.0288(11)	0.0289(11)	0.0235(10)	-0.0012(8)	-0.0063(8)	-0.0036(9)
C2	0.0468(15)	0.0587(18)	0.0301(12)	0.0051(12)	0.0049(11)	0.0024(13)
C3	0.0250(10)	0.0306(11)	0.0200(10)	0.0002(8)	-0.0036(8)	-0.0006(8)
C4	0.0326(11)	0.0287(11)	0.0266(11)	0.0036(9)	-0.0056(9)	-0.0029(9)
C5	0.0310(11)	0.0276(11)	0.0298(11)	0.0026(9)	-0.0075(9)	-0.0039(9)
C6	0.0231(10)	0.0284(11)	0.0224(10)	0.0001(8)	-0.0049(8)	0.0017(8)
C7	0.0366(12)	0.0283(12)	0.0344(12)	0.0058(9)	-0.0091(9)	-0.0032(9)
C8	0.0342(12)	0.0289(12)	0.0385(13)	0.0033(10)	-0.0108(10)	-0.0077(9)
C9	0.0271(11)	0.0324(12)	0.0242(11)	0.0041(8)	-0.0040(8)	0.0005(9)
C10	0.0347(12)	0.0395(13)	0.0251(11)	0.0018(9)	-0.0021(9)	-0.0035(10)
C11	0.0445(15)	0.0516(16)	0.0263(12)	0.0039(10)	0.0023(10)	0.0060(12)
C12	0.0442(15)	0.0511(16)	0.0394(14)	0.0139(12)	0.0126(11)	0.0080(12)
C13	0.0376(13)	0.0468(16)	0.0588(17)	0.0128(13)	0.0117(12)	-0.0032(12)
C14	0.0313(12)	0.0397(14)	0.0419(13)	0.0015(11)	-0.0023(10)	-0.0050(10)
B	0.0396(14)	0.0388(14)	0.0230(12)	-0.0004(10)	-0.0035(10)	0.0010(11)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 8. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H2A	0.243989	0.269599	-0.082187	0.068
H2B	0.209471	0.463070	-0.090120	0.068
H2C	0.165945	0.328814	-0.151658	0.068
H4	0.232061	0.490849	0.069919	0.036
H5	0.368110	0.464795	0.165341	0.036
H7	0.272682	-0.011013	0.207206	0.041
H8	0.138612	0.012519	0.109866	0.042
H10	0.036185	0.393152	0.169802	0.040
H11	-0.035394	0.354153	0.283818	0.049
H12	-0.173674	0.166368	0.292872	0.053
H13	-0.242281	0.018809	0.188058	0.057
H14	-0.171179	0.055361	0.072641	0.046
H1SA[a]	0.583199	0.485719	-0.009215	0.263
H1SB[a]	0.473067	0.388454	-0.014162	0.263
H1SC[a]	0.543306	0.399166	0.063827	0.263
H2SA[a]	0.672805	0.231665	0.014284	0.263
H2SB[a]	0.602590	0.220957	-0.063677	0.263
H3SA[a]	0.573117	0.054226	0.070698	0.263
H3SB[a]	0.470122	0.100797	0.018720	0.263
H4SA[a]	0.613898	-0.148380	0.002140	0.263
H4SB[a]	0.569218	-0.058755	-0.073602	0.263
H5SA[a]	0.415423	-0.187870	-0.074003	0.263
H5SB[a]	0.476071	-0.316254	-0.016488	0.263
H6SA[a]	0.321767	-0.216869	0.033916	0.263
H6SB[a]	0.422707	-0.154960	0.085507	0.263
H6SC[a]	0.362160	-0.026790	0.028088	0.263

[a]Included with an occupancy factor of 0.5.

STRUCTURE REPORT (6a)

XCL Code: DGH1702 **Date:** 20 March 2017

Compound: [^{n}Bu$_4$N][(*N*'-(hydroxy-κ*O*)-*N*-{2-(hydroxy-κ*O*)phenyl}-2-methylpropanimid-amidato-κ*N*)(phenyl)borate]

Formula: $C_{32}H_{52}BN_3O_2$

Supervisor: D. G. Hall **Crystallographer:** R. McDonald

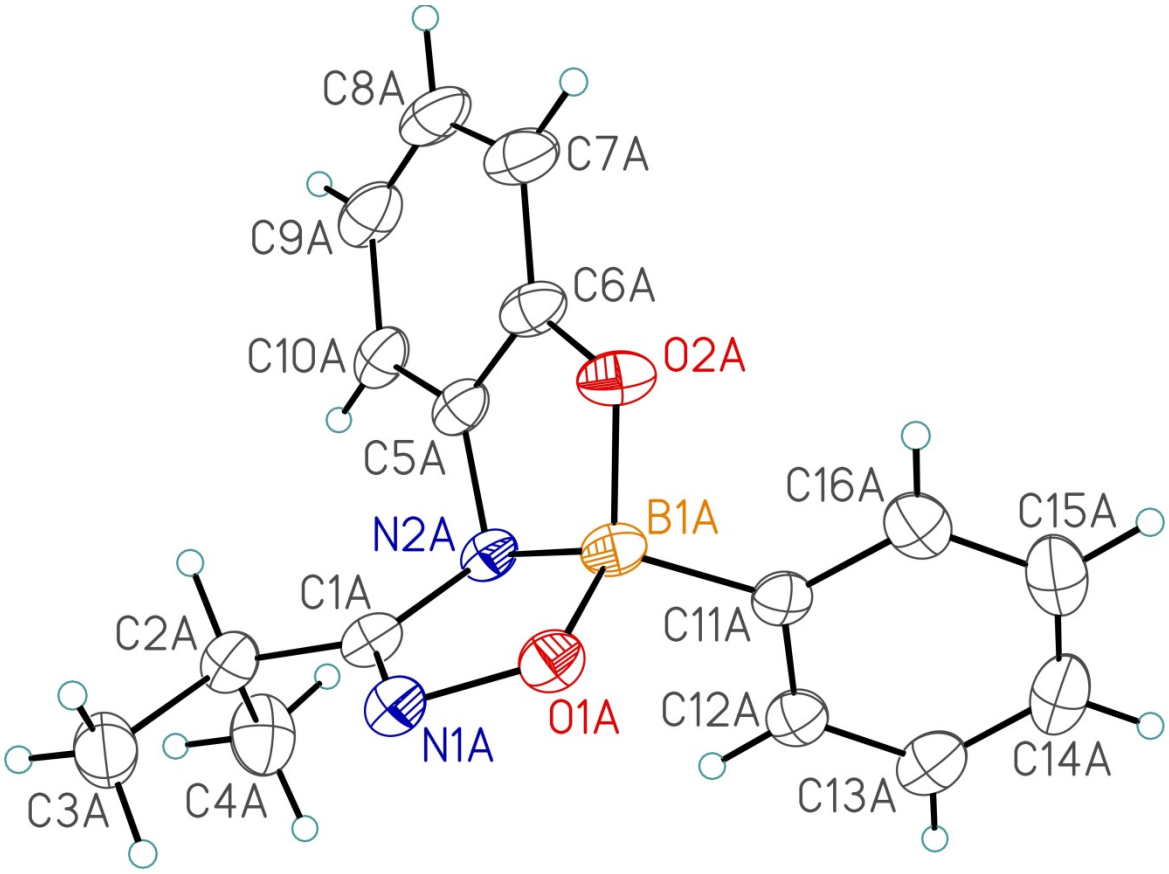


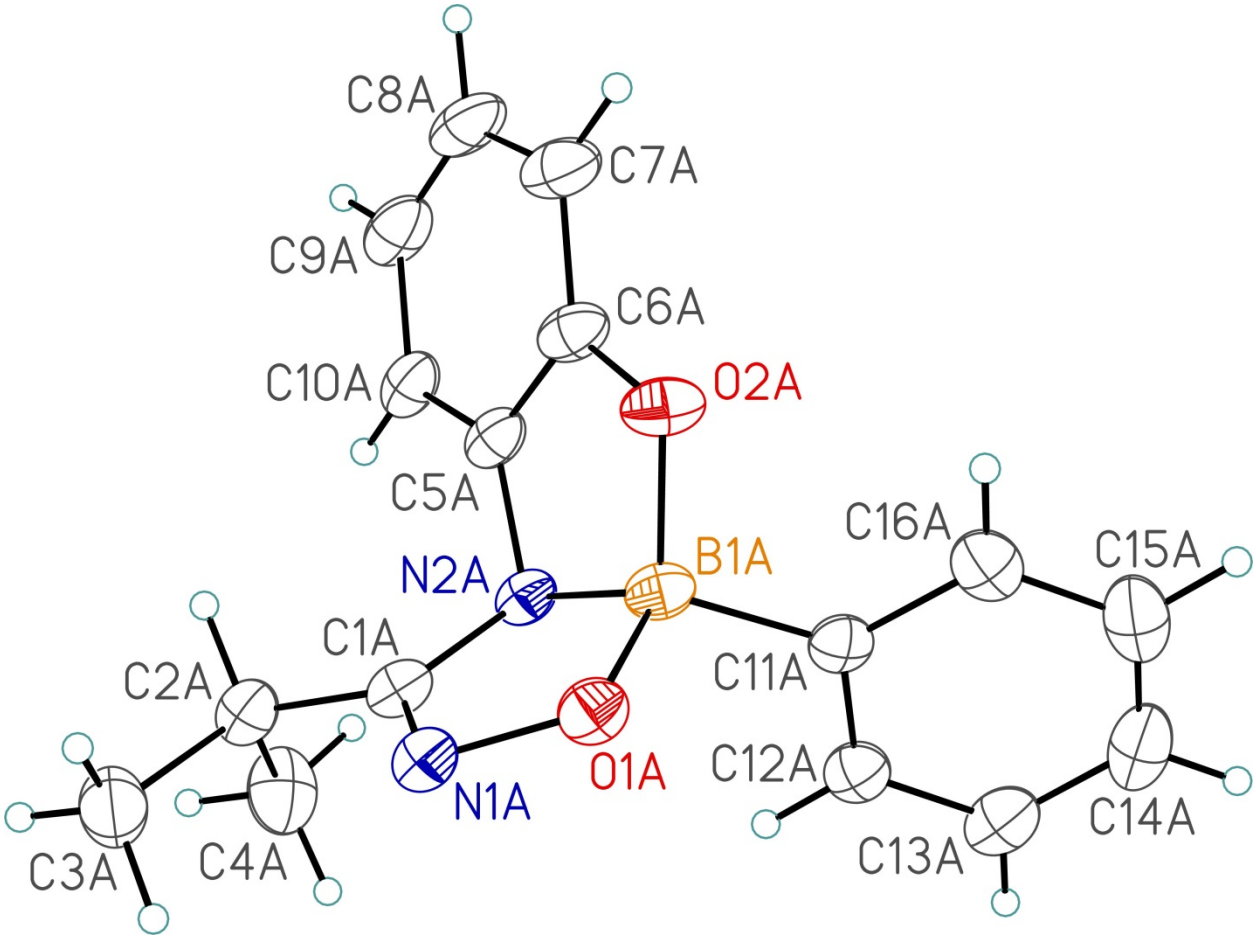
Figure Legends

Figure 1. Perspective view of one of the two crystallographically-independent [(*N*'-(hydroxy-κ*O*)-*N*-{2-(hydroxy-κ*O*)phenyl}-2-methylpropanimidamidato-κ*N*)(phenyl)borate]$^{+}$ ions (moiety A) showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Alternate view of moiety A.

Fugure 3. View of the second crystallographically-independent [(*N*'-(hydroxy-κ*O*)-*N*-{2-(hydroxy-κ*O*)phenyl}-2-methylpropanimidamidato-κ*N*)(phenyl)borate]$^{+}$ ion (moiety B).

Figure 4. Alternate view of moiety B.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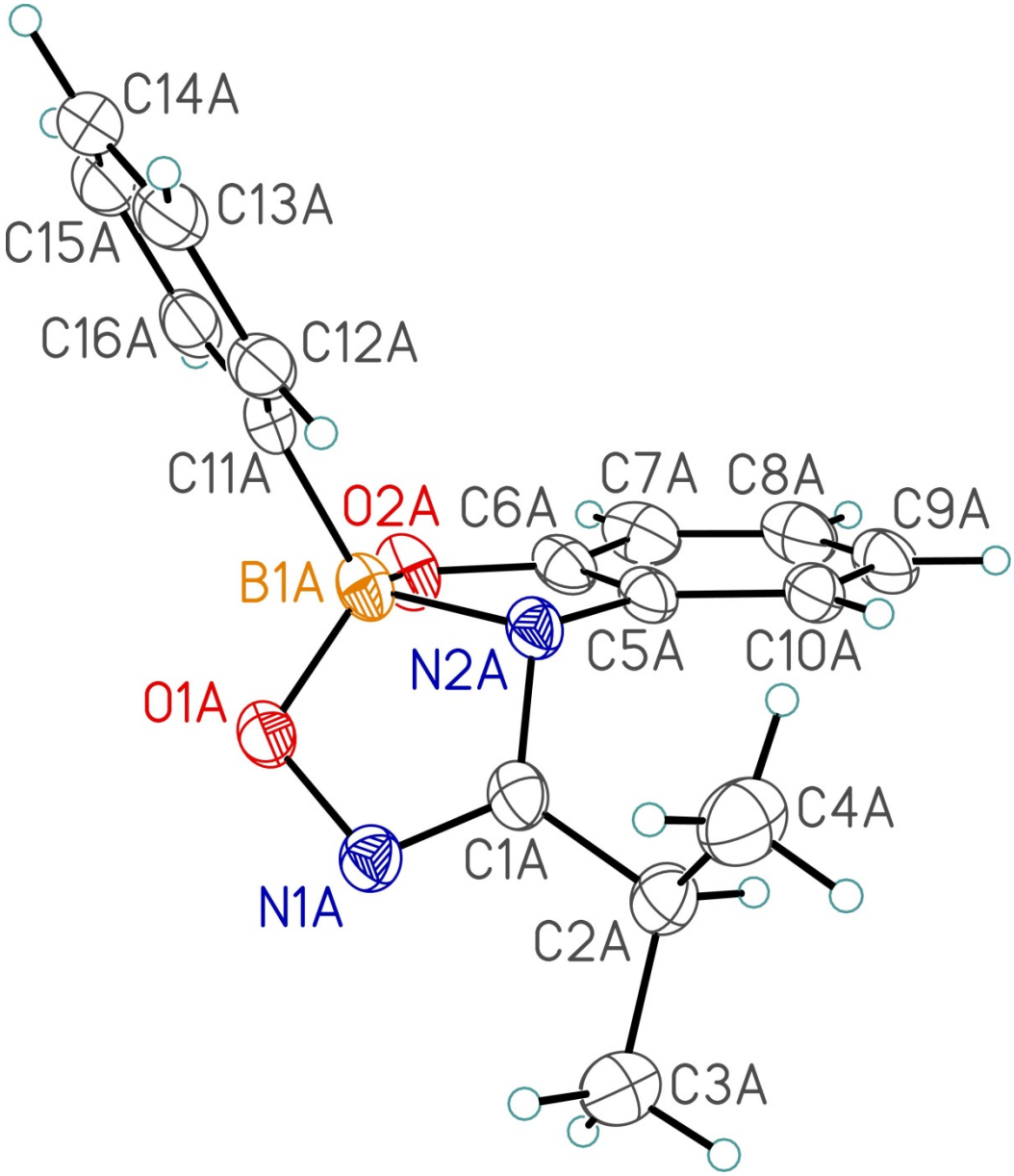
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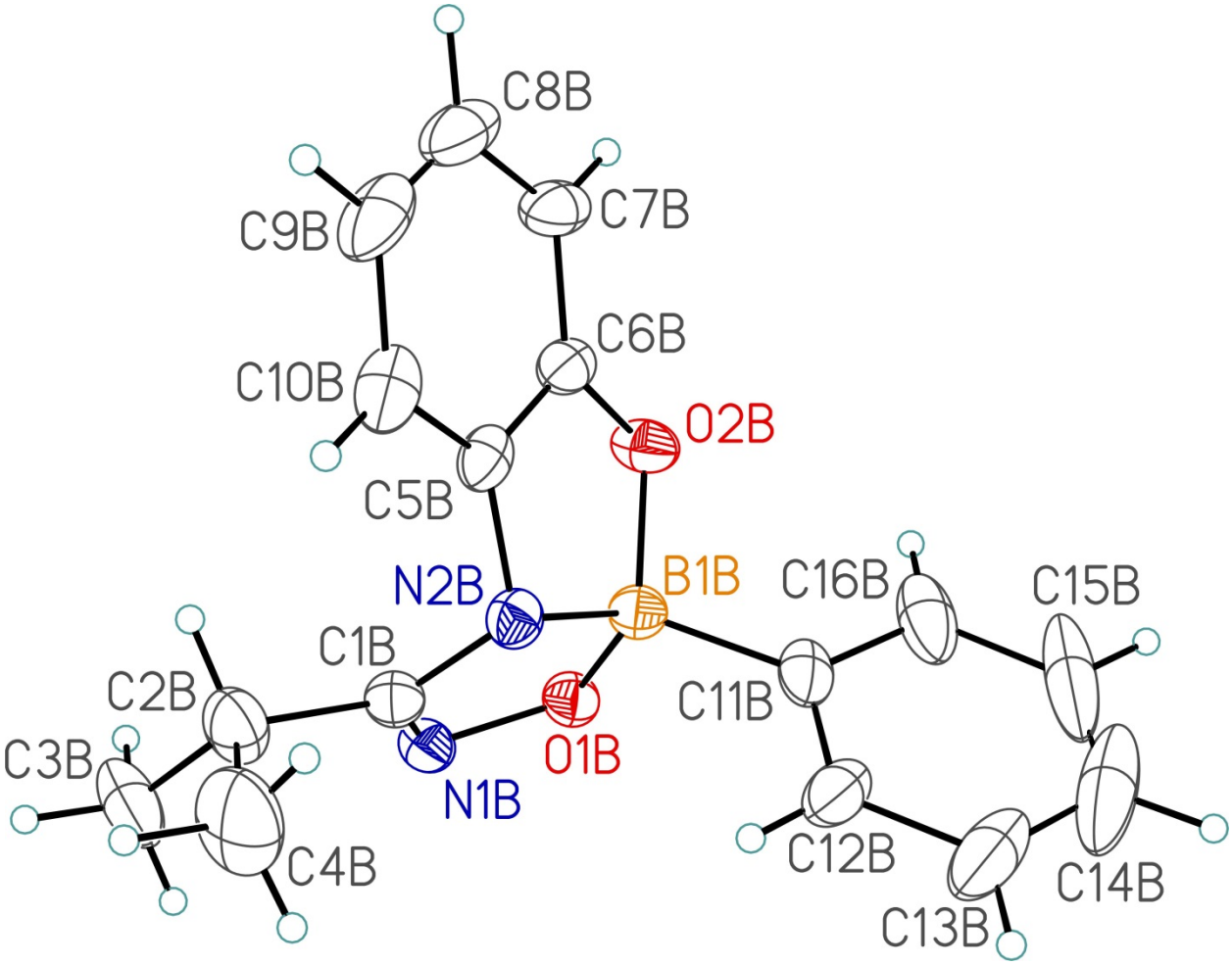
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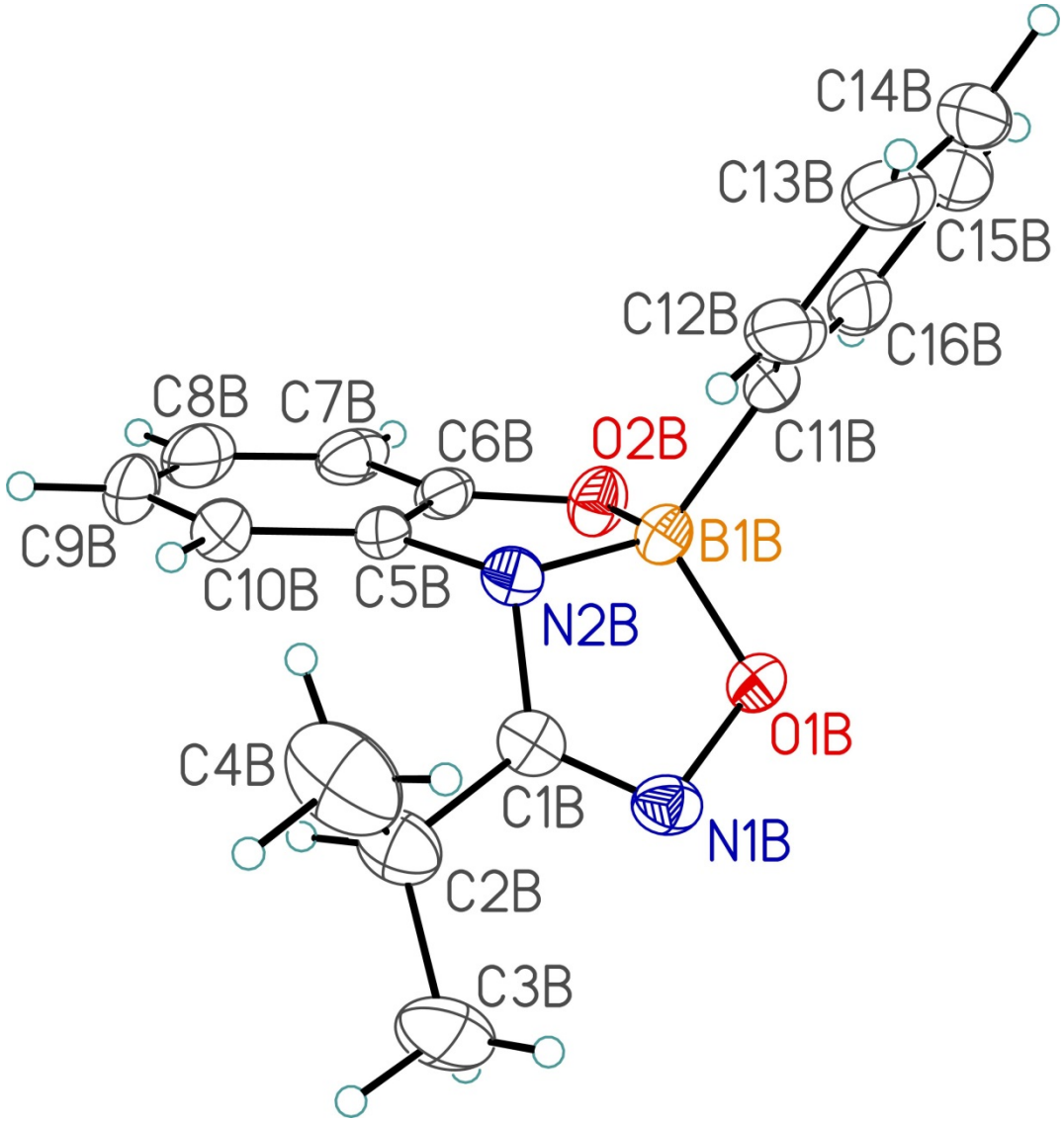
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C10B
C5B
N2B
O1B
C4B
C1B
N1B
C2B
C3B

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data

formula	$C_{32}H_{52}BN_3O_2$
formula weight	521.57
crystal dimensions (mm)	0.32 × 0.31 × 0.04
crystal system	orthorhombic
space group	*Pbca* (No. 61)
unit cell parameters[a]	
a (Å)	17.9063 (5)
b (Å)	21.5998 (6)
c (Å)	33.6902 (9)
V (Å^3)	13030.4 (6)
Z	16
ρ_{calcd} (g cm^{-3})	1.063
μ (mm^{-1})	0.501

B. Data Collection and Refinement Conditions

diffractometer	Bruker D8/APEX II CCD[b]
radiation (λ [Å])	Cu Kα (1.54178) (microfocus source)
temperature (°C)	–100
scan type	ω and ϕ scans (1.0°) (5-10 s exposures)
data collection 2θ limit (deg)	137.66
total data collected	77128 (-21 ≤ *h* ≤ 21, -25 ≤ *k* ≤ 22, -40 ≤ *l* ≤ 40)
independent reflections	11938 (R_{int} = 0.1059)
number of observed reflections (*NO*)	7068 [$F_o^2 \geq 2\sigma(F_o^2)$]
structure solution method	direct methods/dual space (*SHELXD*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	1.0000–0.6961
data/restraints/parameters	11938 / 0 / 686

extinction coefficient $(x)^e$	0.00008(3)
goodness-of-fit $(S)^f$ [all data]	1.028
final R indices[g]	
R_1 [$F_o^2 \geq 2\sigma(F_o^2)$]	0.0740
wR_2 [all data]	0.2436
largest difference peak and hole	0.798 and –0.795 e Å^{-3}

[a]Obtained from least-squares refinement of 9888 reflections with $6.92° < 2\theta < 134.34°$.

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[c]Schneider, T. R.; Sheldrick, G. M. *Acta Crystallogr.* **2002**, *D58*, 1772-1779.

[d]Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[e]$F_c^* = kF_c[1 + x\{0.001F_c^2\lambda^3/\sin(2\theta)\}]^{-1/4}$ where k is the overall scale factor.

[f]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.1225P)^2 + 5.1297P]^{-1}$ where $P = [\text{Max}(F_o^2, 0) + 2F_c^2]/3$).

[g]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

(a) atoms of the [(N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}-2-methylpropanimidamidato-κN)(phenyl)borate]$^+$ ion, moiety A

Atom	*x*	*y*	*z*	U_{eq}, Å^2
O1A	0.15445(11)	0.24171(10)	0.42198(6)	0.0469(5)*
O2A	0.29400(12)	0.25462(11)	0.41236(6)	0.0550(6)*
N1A	0.12854(14)	0.18018(12)	0.41457(7)	0.0457(6)*
N2A	0.23906(13)	0.16827(11)	0.44712(7)	0.0407(6)*
C1A	0.17702(17)	0.14204(15)	0.42802(9)	0.0445(7)*
C2A	0.16605(19)	0.07363(16)	0.42450(10)	0.0527(8)*
C3A	0.0989(2)	0.05639(18)	0.39973(12)	0.0697(10)*
C4A	0.1604(3)	0.04350(19)	0.46553(11)	0.0776(12)*
C5A	0.31144(17)	0.15348(15)	0.43220(8)	0.0440(7)*
C6A	0.34047(17)	0.20495(17)	0.41248(9)	0.0491(8)*
C7A	0.4106(2)	0.2025(2)	0.39499(10)	0.0641(10)*
C8A	0.4500(2)	0.1464(2)	0.39763(11)	0.0684(12)*
C9A	0.4212(2)	0.0963(2)	0.41691(11)	0.0651(11)*
C10A	0.35134(18)	0.09896(16)	0.43481(9)	0.0516(8)*
C11A	0.23372(15)	0.28432(14)	0.47816(9)	0.0421(7)*
C12A	0.19976(17)	0.26690(16)	0.51394(9)	0.0488(8)*
C13A	0.1998(2)	0.30519(19)	0.54730(10)	0.0611(10)*
C14A	0.2343(2)	0.36198(19)	0.54569(12)	0.0674(11)*
C15A	0.2690(2)	0.37994(18)	0.51148(13)	0.0674(11)*
C16A	0.26870(18)	0.34170(16)	0.47823(11)	0.0546(8)*
B1A	0.22948(19)	0.23896(17)	0.44020(10)	0.0439(8)*

(b) atoms of the [(N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}-2-methylpropanimidamidato-κN)(phenyl)borate]$^+$ ion, moiety B

Atom	*x*	*y*	*z*	U_{eq}, Å^2
O1B	0.41142(12)	0.31765(10)	0.16464(6)	0.0471(5)*
O2B	0.37990(13)	0.28783(10)	0.23488(6)	0.0536(6)*
N1B	0.44108(15)	0.37867(13)	0.16001(8)	0.0497(7)*
N2B	0.35087(13)	0.38750(12)	0.20748(7)	0.0440(6)*
C1B	0.40766(18)	0.41485(15)	0.18442(9)	0.0472(7)*
C2B	0.4287(2)	0.48127(17)	0.18804(13)	0.0681(10)*
C3B	0.4954(3)	0.4987(2)	0.16189(15)	0.0948(15)*
C4B	0.3614(3)	0.5233(2)	0.18009(19)	0.115(2)*
C5B	0.35865(16)	0.38993(15)	0.24983(9)	0.0458(7)*
C6B	0.37591(16)	0.33116(16)	0.26372(9)	0.0472(8)*
C7B	0.38900(19)	0.3210(2)	0.30358(10)	0.0635(10)*
C8B	0.3836(2)	0.3712(3)	0.32949(12)	0.0830(14)*
C9B	0.3656(2)	0.4280(3)	0.31609(13)	0.0842(15)*

Table 2. Atomic Coordinates and Displacement Parameters (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å2
C10B	0.3527(2)	0.43878(19)	0.27570(12)	0.0656(10)*
C11B	0.2785(2)	0.28607(18)	0.18122(9)	0.0579(10)*
C12B	0.2233(2)	0.3197(2)	0.16181(10)	0.0775(13)*
C13B	0.1588(3)	0.2920(4)	0.14770(13)	0.123(3)*
C14B	0.1477(4)	0.2293(6)	0.1531(2)	0.173(5)*
C15B	0.2010(5)	0.1961(4)	0.1710(2)	0.152(4)*
C16B	0.2657(3)	0.2230(2)	0.18577(13)	0.0911(16)*
B1B	0.3541(2)	0.31871(18)	0.19618(10)	0.0452(8)*

(c) tetra-n-butylammonium ion atoms

Atom	*x*	*y*	*z*	U_{eq}, Å2
N2	0.44384(12)	0.28416(11)	0.04319(6)	0.0374(5)*
C21	0.45268(16)	0.24800(14)	0.08193(8)	0.0398(7)*
C22	0.39830(18)	0.19555(16)	0.08844(9)	0.0499(8)*
C23	0.42020(19)	0.16019(16)	0.12605(10)	0.0567(9)*
C24	0.3750(3)	0.1020(2)	0.13144(13)	0.0844(14)*
C25	0.50738(15)	0.33062(14)	0.04369(8)	0.0397(7)*
C26	0.51384(17)	0.37241(15)	0.00764(9)	0.0465(7)*
C27	0.57596(17)	0.41944(15)	0.01467(10)	0.0508(8)*
C28	0.58810(18)	0.46219(16)	-0.02046(11)	0.0593(9)*
C29	0.36848(15)	0.31505(15)	0.04030(9)	0.0446(7)*
C30	0.35230(17)	0.36730(16)	0.06894(10)	0.0510(8)*
C31	0.2719(2)	0.3885(2)	0.06302(12)	0.0721(12)*
C32	0.2536(3)	0.4479(2)	0.08536(14)	0.0904(15)*
C33	0.44893(17)	0.24175(15)	0.00740(8)	0.0460(7)*
C34	0.5171(2)	0.20035(17)	0.00573(10)	0.0567(9)*
C35	0.5302(3)	0.1731(2)	-0.03554(12)	0.0801(12)*
C36	0.4731(3)	0.1325(3)	-0.04942(18)	0.137(3)*
N3	0.12880(13)	0.27778(12)	0.29338(7)	0.0402(6)*
C37	0.12129(16)	0.30842(14)	0.33390(8)	0.0408(7)*
C38	0.17959(18)	0.35675(15)	0.34380(9)	0.0479(8)*
C39	0.16048(18)	0.38732(15)	0.38349(9)	0.0482(8)*
C40	0.2119(2)	0.44060(17)	0.39329(11)	0.0651(10)*
C41	0.13243(16)	0.32501(14)	0.26012(8)	0.0433(7)*
C42	0.06738(18)	0.36980(17)	0.25824(10)	0.0536(8)*
C43	0.0771(2)	0.41510(19)	0.22383(11)	0.0677(11)*
C44	0.1427(2)	0.4574(2)	0.22791(16)	0.0941(16)*
C45	0.20114(16)	0.24118(15)	0.29024(9)	0.0444(7)*
C46	0.2098(2)	0.18686(19)	0.31779(12)	0.0699(11)*
C47	0.2800(2)	0.1508(2)	0.30751(16)	0.0860(14)*

Table 2. Atomic Coordinates and Displacement Parameters (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å2

C48	0.2857(4)	0.0919(3)	0.3199(3)	0.187(4)*
C49	0.06031(17)	0.23661(16)	0.28945(9)	0.0496(8)*
C50	0.0537(2)	0.20252(18)	0.24990(11)	0.0649(10)*
C51	-0.0096(2)	0.1546(2)	0.25102(16)	0.0833(14)*
C52	0.0080(4)	0.1002(3)	0.27362(18)	0.125(2)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

(a) within the [(N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}-2-methylpropanimidamidato-κN)(phenyl)borate]$^+$ ions

Moiety A			*Moiety B*		
Atom1	Atom2	Distance	Atom1	Atom2	Distance
O1A	N1A	1.430(3)	O1B	N1B	1.430(3)
O1A	B1A	1.478(4)	O1B	B1B	1.477(4)
O2A	C6A	1.358(4)	O2B	C6B	1.351(4)
O2A	B1A	1.526(4)	O2B	B1B	1.536(4)
N1A	C1A	1.280(4)	N1B	C1B	1.283(4)
N2A	C1A	1.403(4)	N2B	C1B	1.409(4)
N2A	C5A	1.426(4)	N2B	C5B	1.435(4)
N2A	B1A	1.554(4)	N2B	B1B	1.535(4)
C1A	C2A	1.495(5)	C1B	C2B	1.488(5)
C2A	C3A	1.511(5)	C2B	C3B	1.530(6)
C2A	C4A	1.531(5)	C2B	C4B	1.533(6)
C5A	C6A	1.395(5)	C5B	C6B	1.388(5)
C5A	C10A	1.380(4)	C5B	C10B	1.373(4)
C6A	C7A	1.388(4)	C6B	C7B	1.381(4)
C7A	C8A	1.405(5)	C7B	C8B	1.395(6)
C8A	C9A	1.363(6)	C8B	C9B	1.347(7)
C9A	C10A	1.390(5)	C9B	C10B	1.399(6)
C11A	C12A	1.402(4)	C11B	C12B	1.391(6)
C11A	C16A	1.389(4)	C11B	C16B	1.389(5)
C11A	B1A	1.613(5)	C11B	B1B	1.608(5)
C12A	C13A	1.395(5)	C12B	C13B	1.384(6)
C13A	C14A	1.374(5)	C13B	C14B	1.381(11)
C14A	C15A	1.365(6)	C14B	C15B	1.337(13)
C15A	C16A	1.392(5)	C15B	C16B	1.388(8)

(b) within the tetra-n-butylammonium ions

Atom1	Atom2	Distance	Atom1	Atom2	Distance
N2	C21	1.529(3)	C29	C30	1.513(4)
N2	C25	1.517(3)	C30	C31	1.523(4)
N2	C29	1.509(3)	C31	C32	1.523(6)
N2	C33	1.517(3)	C33	C34	1.514(4)
C21	C22	1.510(4)	C34	C35	1.528(5)
C22	C23	1.531(4)	C35	C36	1.426(6)
C23	C24	1.506(5)	N3	C37	1.523(3)
C25	C26	1.518(4)	N3	C41	1.517(4)
C26	C27	1.525(4)	N3	C45	1.521(4)
C27	C28	1.517(4)	N3	C49	1.521(4)

Table 3. Selected Interatomic Distances (continued)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
C37	C38	1.514(4)	C45	C46	1.504(5)
C38	C39	1.530(4)	C46	C47	1.519(5)
C39	C40	1.510(4)	C47	C48	1.342(7)
C41	C42	1.515(4)	C49	C50	1.527(4)
C42	C43	1.527(5)	C50	C51	1.534(5)
C43	C44	1.495(6)	C51	C52	1.435(7)

Table 4. Selected Interatomic Angles (deg)

(a) within the [(N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}-2-methylpropanimidamidato-κN)(phenyl)borate]$^+$ ions

Moiety A				*Moiety B*			
Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
N1A	O1A	B1A	109.3(2)	N1B	O1B	B1B	108.8(2)
C6A	O2A	B1A	106.7(2)	C6B	O2B	B1B	107.1(2)
O1A	N1A	C1A	108.4(2)	O1B	N1B	C1B	108.6(2)
C1A	N2A	C5A	117.9(2)	C1B	N2B	C5B	117.6(2)
C1A	N2A	B1A	103.9(2)	C1B	N2B	B1B	104.0(2)
C5A	N2A	B1A	105.5(2)	C5B	N2B	B1B	106.2(2)
N1A	C1A	N2A	116.1(3)	N1B	C1B	N2B	115.8(3)
N1A	C1A	C2A	121.3(3)	N1B	C1B	C2B	121.4(3)
N2A	C1A	C2A	122.6(3)	N2B	C1B	C2B	122.8(3)
C1A	C2A	C3A	113.1(3)	C1B	C2B	C3B	112.8(3)
C1A	C2A	C4A	110.9(3)	C1B	C2B	C4B	111.0(3)
C3A	C2A	C4A	110.0(3)	C3B	C2B	C4B	111.5(4)
N2A	C5A	C6A	109.1(3)	N2B	C5B	C6B	108.9(3)
N2A	C5A	C10A	129.7(3)	N2B	C5B	C10B	130.7(3)
C6A	C5A	C10A	121.1(3)	C6B	C5B	C10B	120.4(3)
O2A	C6A	C5A	113.7(3)	O2B	C6B	C5B	113.8(3)
O2A	C6A	C7A	125.7(3)	O2B	C6B	C7B	125.5(3)
C5A	C6A	C7A	120.5(3)	C5B	C6B	C7B	120.8(3)
C6A	C7A	C8A	117.4(4)	C6B	C7B	C8B	118.2(4)
C7A	C8A	C9A	121.7(3)	C7B	C8B	C9B	121.0(4)
C8A	C9A	C10A	120.9(3)	C8B	C9B	C10B	121.1(4)
C5A	C10A	C9A	118.3(4)	C5B	C10B	C9B	118.5(4)
C12A	C11A	C16A	115.7(3)	C12B	C11B	C16B	116.5(4)
C12A	C11A	B1A	119.9(3)	C12B	C11B	B1B	121.1(3)
C16A	C11A	B1A	124.4(3)	C16B	C11B	B1B	122.3(4)
C11A	C12A	C13A	122.2(3)	C11B	C12B	C13B	121.9(5)
C12A	C13A	C14A	119.9(4)	C12B	C13B	C14B	119.9(7)
C13A	C14A	C15A	119.4(4)	C13B	C14B	C15B	118.9(6)
C14A	C15A	C16A	120.6(4)	C14B	C15B	C16B	122.1(8)
C11A	C16A	C15A	122.2(4)	C11B	C16B	C15B	120.6(6)
O1A	B1A	O2A	115.1(3)	O1B	B1B	O2B	113.3(3)
O1A	B1A	N2A	101.7(3)	O1B	B1B	N2B	102.7(3)
O1A	B1A	C11A	110.3(2)	O1B	B1B	C11B	110.6(3)
O2A	B1A	N2A	103.1(2)	O2B	B1B	N2B	102.8(2)
O2A	B1A	C11A	108.5(3)	O2B	B1B	C11B	109.2(3)
N2A	B1A	C11A	118.2(3)	N2B	B1B	C11B	118.1(3)

Table 4. Selected Interatomic Angles (continued)

(b) within the tetra-n-butylammonium ions

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C21	N2	C25	104.5(2)	C37	N3	C41	111.9(2)
C21	N2	C29	111.9(2)	C37	N3	C45	111.3(2)
C21	N2	C33	111.3(2)	C37	N3	C49	105.1(2)
C25	N2	C29	112.3(2)	C41	N3	C45	105.2(2)
C25	N2	C33	111.3(2)	C41	N3	C49	111.3(2)
C29	N2	C33	105.6(2)	C45	N3	C49	112.1(2)
N2	C21	C22	116.1(2)	N3	C37	C38	115.9(2)
C21	C22	C23	109.2(2)	C37	C38	C39	109.6(2)
C22	C23	C24	112.3(3)	C38	C39	C40	112.6(3)
N2	C25	C26	116.2(2)	N3	C41	C42	115.3(2)
C25	C26	C27	109.1(2)	C41	C42	C43	110.7(3)
C26	C27	C28	112.9(3)	C42	C43	C44	114.3(3)
N2	C29	C30	117.4(2)	N3	C45	C46	116.8(2)
C29	C30	C31	108.8(3)	C45	C46	C47	110.2(3)
C30	C31	C32	113.1(4)	C46	C47	C48	118.6(4)
N2	C33	C34	115.8(2)	N3	C49	C50	114.9(2)
C33	C34	C35	112.7(3)	C49	C50	C51	111.2(3)
C34	C35	C36	115.1(4)	C50	C51	C52	113.8(4)

Table 5. Torsional Angles (deg)

(a) within the [(N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}-2-methylpropanimidamidato-κN)(phenyl)borate]$^+$ ions

Moiety A					*Moiety B*				
Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
B1A	O1A	N1A	C1A	3.4(3)	B1B	O1B	N1B	C1B	-0.8(3)
N1A	O1A	B1A	O2A	103.9(3)	N1B	O1B	B1B	O2B	-107.0(3)
N1A	O1A	B1A	N2A	-6.8(3)	N1B	O1B	B1B	N2B	3.1(3)
N1A	O1A	B1A	C11A	-133.0(2)	N1B	O1B	B1B	C11B	130.0(3)
B1A	O2A	C6A	C5A	8.7(4)	B1B	O2B	C6B	C5B	-6.6(3)
B1A	O2A	C6A	C7A	-171.7(3)	B1B	O2B	C6B	C7B	174.9(3)
C6A	O2A	B1A	O1A	-122.7(3)	C6B	O2B	B1B	O1B	120.7(3)
C6A	O2A	B1A	N2A	-12.9(3)	C6B	O2B	B1B	N2B	10.7(3)
C6A	O2A	B1A	C11A	113.2(3)	C6B	O2B	B1B	C11B	-115.5(3)
O1A	N1A	C1A	N2A	2.3(3)	O1B	N1B	C1B	N2B	-2.3(4)
O1A	N1A	C1A	C2A	-179.6(2)	O1B	N1B	C1B	C2B	177.2(3)
C5A	N2A	C1A	N1A	-122.8(3)	C5B	N2B	C1B	N1B	121.2(3)
C5A	N2A	C1A	C2A	59.1(4)	C5B	N2B	C1B	C2B	-58.3(4)
B1A	N2A	C1A	N1A	-6.6(3)	B1B	N2B	C1B	N1B	4.2(4)
B1A	N2A	C1A	C2A	175.4(3)	B1B	N2B	C1B	C2B	-175.2(3)
C1A	N2A	C5A	C6A	107.1(3)	C1B	N2B	C5B	C6B	-108.0(3)
C1A	N2A	C5A	C10A	-72.5(4)	C1B	N2B	C5B	C10B	71.0(4)
B1A	N2A	C5A	C6A	-8.3(3)	B1B	N2B	C5B	C6B	7.8(3)
B1A	N2A	C5A	C10A	172.1(3)	B1B	N2B	C5B	C10B	-173.2(3)
C1A	N2A	B1A	O1A	7.6(3)	C1B	N2B	B1B	O1B	-4.2(3)
C1A	N2A	B1A	O2A	-111.9(2)	C1B	N2B	B1B	O2B	113.7(3)
C1A	N2A	B1A	C11A	128.5(3)	C1B	N2B	B1B	C11B	-126.1(3)
C5A	N2A	B1A	O1A	132.2(2)	C5B	N2B	B1B	O1B	-128.8(2)
C5A	N2A	B1A	O2A	12.7(3)	C5B	N2B	B1B	O2B	-11.0(3)
C5A	N2A	B1A	C11A	-106.9(3)	C5B	N2B	B1B	C11B	109.2(3)
N1A	C1A	C2A	C3A	7.0(4)	N1B	C1B	C2B	C3B	-3.3(5)
N1A	C1A	C2A	C4A	-117.1(3)	N1B	C1B	C2B	C4B	122.6(4)
N2A	C1A	C2A	C3A	-175.1(3)	N2B	C1B	C2B	C3B	176.1(3)
N2A	C1A	C2A	C4A	60.9(4)	N2B	C1B	C2B	C4B	-58.0(5)
N2A	C5A	C6A	O2A	-0.1(4)	N2B	C5B	C6B	O2B	-0.8(3)
N2A	C5A	C6A	C7A	-179.7(3)	N2B	C5B	C6B	C7B	177.7(3)
C10A	C5A	C6A	O2A	179.5(3)	C10B	C5B	C6B	O2B	-179.9(3)
C10A	C5A	C6A	C7A	-0.1(5)	C10B	C5B	C6B	C7B	-1.4(5)
N2A	C5A	C10A	C9A	178.9(3)	N2B	C5B	C10B	C9B	-178.0(3)
C6A	C5A	C10A	C9A	-0.6(5)	C6B	C5B	C10B	C9B	0.9(5)
O2A	C6A	C7A	C8A	-178.8(3)	O2B	C6B	C7B	C8B	179.1(3)
C5A	C6A	C7A	C8A	0.7(5)	C5B	C6B	C7B	C8B	0.7(5)

Table 5. Torsional Angles (continued)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C6A	C7A	C8A	C9A	-0.7(5)	C6B	C7B	C8B	C9B	0.4(6)
C7A	C8A	C9A	C10A	0.0(6)	C7B	C8B	C9B	C10B	-0.9(6)
C8A	C9A	C10A	C5A	0.7(5)	C8B	C9B	C10B	C5B	0.2(6)
C16A	C11A	C12A	C13A	1.1(4)	C16B	C11B	C12B	C13B	0.0(5)
B1A	C11A	C12A	C13A	-178.1(3)	B1B	C11B	C12B	C13B	178.1(3)
C12A	C11A	C16A	C15A	-1.0(4)	C12B	C11B	C16B	C15B	0.6(5)
B1A	C11A	C16A	C15A	178.1(3)	B1B	C11B	C16B	C15B	-177.4(4)
C12A	C11A	B1A	O1A	75.8(3)	C12B	C11B	B1B	O1B	-88.7(4)
C12A	C11A	B1A	O2A	-157.3(3)	C12B	C11B	B1B	O2B	146.0(3)
C12A	C11A	B1A	N2A	-40.5(4)	C12B	C11B	B1B	N2B	29.2(4)
C16A	C11A	B1A	O1A	-103.3(3)	C16B	C11B	B1B	O1B	89.3(4)
C16A	C11A	B1A	O2A	23.6(4)	C16B	C11B	B1B	O2B	-36.0(4)
C16A	C11A	B1A	N2A	140.3(3)	C16B	C11B	B1B	N2B	-152.9(3)
C11A	C12A	C13A	C14A	-0.2(5)	C11B	C12B	C13B	C14B	0.7(7)
C12A	C13A	C14A	C15A	-0.8(5)	C12B	C13B	C14B	C15B	-2.0(10)
C13A	C14A	C15A	C16A	0.9(5)	C13B	C14B	C15B	C16B	2.7(11)
C14A	C15A	C16A	C11A	0.1(5)	C14B	C15B	C16B	C11B	-2.0(9)

(b) within the tetra-n-butylammonium ions

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C25	N2	C21	C22	-177.0(3)	C33	C34	C35	C36	-64.0(6)
C29	N2	C21	C22	61.3(3)					
C33	N2	C21	C22	-56.7(3)					
C21	N2	C25	C26	178.4(2)					
C29	N2	C25	C26	-60.1(3)					
C33	N2	C25	C26	58.1(3)					
C21	N2	C29	C30	67.5(3)					
C25	N2	C29	C30	-49.7(3)					
C33	N2	C29	C30	-171.2(3)					
C21	N2	C33	C34	-52.4(3)					
C25	N2	C33	C34	63.7(3)					
C29	N2	C33	C34	-174.1(3)					
N2	C21	C22	C23	173.9(3)					
C21	C22	C23	C24	-172.1(3)					
N2	C25	C26	C27	175.2(2)					
C25	C26	C27	C28	178.3(3)					
N2	C29	C30	C31	-175.9(3)					
C29	C30	C31	C32	-170.3(3)					
N2	C33	C34	C35	-163.7(3)					

C41	N3	C37	C38	-53.7(3)
C45	N3	C37	C38	63.6(3)
C49	N3	C37	C38	-174.7(3)
C37	N3	C41	C42	-56.1(3)
C45	N3	C41	C42	-177.1(3)
C49	N3	C41	C42	61.2(3)
C37	N3	C45	C46	63.7(4)
C41	N3	C45	C46	-174.9(3)
C49	N3	C45	C46	-53.8(4)
C37	N3	C49	C50	177.6(3)
C41	N3	C49	C50	56.2(4)
C45	N3	C49	C50	-61.3(4)
N3	C37	C38	C39	174.8(3)
C37	C38	C39	C40	-174.2(3)
N3	C41	C42	C43	179.9(3)
C41	C42	C43	C44	-64.8(5)
N3	C45	C46	C47	173.0(3)
C45	C46	C47	C48	-159.9(6)
N3	C49	C50	C51	171.0(3)
C49	C50	C51	C52	-74.3(5)

Table 6. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1A	0.0440(12)	0.0484(13)	0.0483(12)	0.0023(9)	-0.0024(9)	0.0091(9)
O2A	0.0474(13)	0.0668(16)	0.0508(13)	0.0155(11)	0.0124(10)	0.0083(11)
N1A	0.0465(15)	0.0473(16)	0.0435(14)	-0.0030(11)	0.0026(11)	0.0057(12)
N2A	0.0386(13)	0.0471(15)	0.0363(12)	-0.0018(10)	0.0013(10)	0.0098(11)
C1A	0.0445(17)	0.051(2)	0.0382(16)	-0.0003(13)	0.0060(13)	0.0100(14)
C2A	0.056(2)	0.051(2)	0.0508(19)	-0.0061(14)	0.0023(15)	0.0060(15)
C3A	0.070(3)	0.064(3)	0.075(3)	-0.0128(19)	-0.007(2)	-0.0022(19)
C4A	0.108(3)	0.059(3)	0.065(2)	0.0073(19)	-0.006(2)	-0.009(2)
C5A	0.0422(16)	0.057(2)	0.0328(14)	-0.0057(13)	-0.0048(12)	0.0124(14)
C6A	0.0406(17)	0.071(2)	0.0355(15)	-0.0019(14)	-0.0004(12)	0.0104(15)
C7A	0.048(2)	0.102(3)	0.0425(19)	-0.0067(18)	0.0050(15)	0.0036(19)
C8A	0.0394(19)	0.111(4)	0.055(2)	-0.023(2)	0.0013(16)	0.020(2)
C9A	0.046(2)	0.086(3)	0.063(2)	-0.021(2)	-0.0094(17)	0.0269(19)
C10A	0.0497(18)	0.060(2)	0.0450(17)	-0.0127(15)	-0.0082(14)	0.0166(15)
C11A	0.0340(15)	0.0427(18)	0.0495(17)	0.0044(13)	-0.0029(13)	0.0108(12)
C12A	0.0427(17)	0.057(2)	0.0465(17)	0.0005(14)	-0.0048(14)	0.0051(14)
C13A	0.055(2)	0.081(3)	0.0481(19)	-0.0058(17)	-0.0085(16)	0.0172(19)
C14A	0.070(3)	0.063(3)	0.070(3)	-0.0191(19)	-0.030(2)	0.021(2)
C15A	0.059(2)	0.051(2)	0.092(3)	-0.006(2)	-0.030(2)	0.0064(17)
C16A	0.0433(18)	0.052(2)	0.069(2)	0.0079(17)	-0.0123(16)	0.0048(15)
B1A	0.0382(18)	0.051(2)	0.0427(18)	0.0064(15)	0.0038(14)	0.0090(15)
O1B	0.0508(12)	0.0517(13)	0.0390(11)	-0.0016(9)	0.0074(9)	-0.0046(10)
O2B	0.0715(15)	0.0499(14)	0.0393(11)	0.0027(10)	0.0039(10)	0.0059(11)
N1B	0.0484(15)	0.0550(18)	0.0459(15)	0.0098(12)	-0.0006(12)	-0.0067(12)
N2B	0.0394(14)	0.0464(16)	0.0463(14)	-0.0008(11)	-0.0008(11)	-0.0016(11)
C1B	0.0471(18)	0.0462(19)	0.0482(18)	0.0061(14)	-0.0060(14)	0.0014(14)
C2B	0.069(2)	0.050(2)	0.086(3)	0.0115(19)	-0.004(2)	-0.0030(18)
C3B	0.109(4)	0.065(3)	0.110(4)	0.021(3)	0.022(3)	-0.028(3)
C4B	0.112(4)	0.057(3)	0.176(6)	0.021(3)	-0.027(4)	0.011(3)
C5B	0.0336(15)	0.057(2)	0.0467(17)	-0.0134(14)	0.0058(13)	-0.0063(14)
C6B	0.0373(16)	0.067(2)	0.0371(15)	-0.0040(14)	0.0052(12)	-0.0064(14)
C7B	0.048(2)	0.103(3)	0.0397(18)	0.0049(18)	0.0033(15)	-0.0095(19)
C8B	0.063(3)	0.143(5)	0.043(2)	-0.019(3)	0.0033(18)	-0.026(3)
C9B	0.073(3)	0.117(4)	0.063(3)	-0.050(3)	0.017(2)	-0.025(3)
C10B	0.051(2)	0.071(3)	0.075(3)	-0.028(2)	0.0151(18)	-0.0090(18)
C11B	0.063(2)	0.072(3)	0.0394(17)	-0.0152(16)	0.0207(16)	-0.0271(18)
C12B	0.055(2)	0.135(4)	0.043(2)	-0.003(2)	-0.0018(17)	-0.031(2)
C13B	0.065(3)	0.249(8)	0.055(3)	-0.029(4)	0.003(2)	-0.058(4)
C14B	0.123(6)	0.298(13)	0.097(5)	-0.106(7)	0.060(4)	-0.142(8)
C15B	0.170(7)	0.156(7)	0.129(6)	-0.084(5)	0.096(5)	-0.126(6)

Table 6. Anisotropic Displacement Parameters (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C16B	0.111(4)	0.086(3)	0.076(3)	-0.032(2)	0.048(3)	-0.051(3)
B1B	0.049(2)	0.051(2)	0.0362(17)	-0.0019(15)	0.0038(15)	-0.0023(16)
N2	0.0315(12)	0.0472(15)	0.0334(12)	0.0024(10)	-0.0045(9)	-0.0033(10)
C21	0.0365(15)	0.0516(19)	0.0312(14)	0.0042(12)	-0.0031(11)	-0.0003(13)
C22	0.0439(18)	0.063(2)	0.0432(17)	0.0081(14)	-0.0041(13)	-0.0093(15)
C23	0.053(2)	0.064(2)	0.0522(19)	0.0157(16)	-0.0049(15)	-0.0058(16)
C24	0.097(3)	0.081(3)	0.076(3)	0.029(2)	-0.012(2)	-0.026(2)
C25	0.0311(14)	0.0491(18)	0.0388(15)	0.0013(12)	-0.0058(11)	-0.0018(12)
C26	0.0399(17)	0.054(2)	0.0453(17)	0.0058(14)	-0.0006(13)	0.0000(14)
C27	0.0351(16)	0.055(2)	0.063(2)	0.0084(16)	-0.0001(14)	-0.0004(13)
C28	0.0433(18)	0.060(2)	0.075(2)	0.0188(18)	0.0058(17)	0.0034(16)
C29	0.0299(15)	0.064(2)	0.0398(16)	0.0077(14)	-0.0068(12)	0.0003(13)
C30	0.0384(17)	0.067(2)	0.0475(18)	0.0057(15)	-0.0028(14)	0.0089(15)
C31	0.050(2)	0.106(3)	0.061(2)	0.012(2)	0.0036(17)	0.025(2)
C32	0.077(3)	0.096(4)	0.098(3)	0.016(3)	0.029(2)	0.036(3)
C33	0.0483(18)	0.058(2)	0.0320(15)	-0.0047(13)	-0.0068(13)	-0.0070(14)
C34	0.058(2)	0.066(2)	0.0459(18)	-0.0075(16)	0.0005(15)	0.0041(17)
C35	0.086(3)	0.096(3)	0.058(2)	-0.018(2)	0.001(2)	0.005(2)
C36	0.098(4)	0.181(6)	0.132(5)	-0.081(5)	-0.002(4)	-0.011(4)
N3	0.0343(13)	0.0475(15)	0.0389(13)	-0.0028(10)	0.0057(10)	-0.0049(10)
C37	0.0396(16)	0.0481(18)	0.0346(15)	-0.0009(12)	0.0066(12)	-0.0001(13)
C38	0.0462(17)	0.054(2)	0.0432(17)	-0.0069(14)	0.0052(13)	-0.0059(14)
C39	0.0524(19)	0.050(2)	0.0425(17)	-0.0022(13)	0.0035(14)	-0.0015(14)
C40	0.083(3)	0.058(2)	0.055(2)	-0.0139(17)	0.0085(18)	-0.0165(19)
C41	0.0398(16)	0.054(2)	0.0363(15)	0.0031(13)	0.0043(12)	-0.0065(13)
C42	0.0384(17)	0.068(2)	0.054(2)	0.0086(16)	0.0013(14)	-0.0004(15)
C43	0.052(2)	0.085(3)	0.067(2)	0.023(2)	0.0013(17)	0.0081(19)
C44	0.070(3)	0.081(3)	0.132(4)	0.049(3)	0.006(3)	0.002(2)
C45	0.0376(16)	0.051(2)	0.0447(16)	-0.0050(13)	0.0088(13)	0.0011(13)
C46	0.058(2)	0.072(3)	0.080(3)	0.019(2)	0.0235(19)	0.0138(18)
C47	0.064(3)	0.064(3)	0.130(4)	0.015(3)	0.017(3)	0.017(2)
C48	0.118(5)	0.078(5)	0.366(13)	-0.046(6)	0.084(7)	0.007(4)
C49	0.0401(17)	0.053(2)	0.0561(19)	-0.0073(15)	0.0079(14)	-0.0144(14)
C50	0.055(2)	0.071(3)	0.069(2)	-0.0186(19)	0.0031(18)	-0.0143(18)
C51	0.056(2)	0.063(3)	0.131(4)	-0.028(3)	0.001(2)	-0.014(2)
C52	0.136(5)	0.091(4)	0.146(5)	-0.038(4)	0.039(4)	-0.040(4)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H2A	0.2112	0.0562	0.4111	0.063
H3A	0.0945	0.0112	0.3985	0.084
H3B	0.0537	0.0738	0.4118	0.084
H3C	0.1048	0.0730	0.3729	0.084
H4A	0.1533	-0.0012	0.4625	0.093
H4B	0.2065	0.0513	0.4804	0.093
H4C	0.1179	0.0611	0.4799	0.093
H7A	0.4311	0.2373	0.3817	0.077
H8A	0.4979	0.1433	0.3857	0.082
H9A	0.4493	0.0590	0.4181	0.078
H10A	0.3316	0.0642	0.4485	0.062
H12A	0.1759	0.2277	0.5155	0.059
H13A	0.1761	0.2920	0.5711	0.073
H14A	0.2341	0.3885	0.5682	0.081
H15A	0.2935	0.4189	0.5104	0.081
H16A	0.2933	0.3553	0.4548	0.066
H2B	0.4442	0.4883	0.2162	0.082
H3D	0.5068	0.5427	0.1654	0.114
H3E	0.5388	0.4738	0.1695	0.114
H3F	0.4831	0.4907	0.1340	0.114
H4D	0.3766	0.5667	0.1826	0.138
H4E	0.3427	0.5159	0.1532	0.138
H4F	0.3219	0.5143	0.1994	0.138
H7B	0.4014	0.2809	0.3131	0.076
H8B	0.3927	0.3652	0.3570	0.100
H9B	0.3615	0.4613	0.3344	0.101
H10B	0.3402	0.4790	0.2664	0.079
H12B	0.2299	0.3630	0.1581	0.093
H13B	0.1223	0.3161	0.1343	0.147
H14B	0.1030	0.2101	0.1443	0.207
H15B	0.1943	0.1527	0.1738	0.182
H16B	0.3016	0.1981	0.1991	0.109
H21A	0.5039	0.2309	0.0828	0.048
H21B	0.4477	0.2774	0.1043	0.048
H22A	0.3991	0.1673	0.0653	0.060
H22B	0.3471	0.2122	0.0913	0.060
H23A	0.4738	0.1493	0.1247	0.068
H23B	0.4129	0.1874	0.1494	0.068
H24A	0.3908	0.0809	0.1558	0.101
H24B	0.3219	0.1126	0.1334	0.101

Table 7. Derived Parameters for Hydrogen Atoms (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H24C	0.3828	0.0746	0.1086	0.101
H25A	0.5549	0.3076	0.0466	0.048
H25B	0.5016	0.3571	0.0675	0.048
H26A	0.4659	0.3942	0.0031	0.056
H26B	0.5253	0.3473	-0.0162	0.056
H27A	0.5635	0.4446	0.0383	0.061
H27B	0.6230	0.3971	0.0203	0.061
H28A	0.6285	0.4913	-0.0144	0.071
H28B	0.6016	0.4376	-0.0438	0.071
H28C	0.5421	0.4852	-0.0259	0.071
H29A	0.3297	0.2829	0.0440	0.054
H29B	0.3628	0.3313	0.0130	0.054
H30A	0.3869	0.4022	0.0640	0.061
H30B	0.3595	0.3529	0.0966	0.061
H31A	0.2630	0.3952	0.0343	0.086
H31B	0.2378	0.3553	0.0721	0.086
H32A	0.2015	0.4593	0.0805	0.109
H32B	0.2613	0.4414	0.1139	0.109
H32C	0.2864	0.4813	0.0761	0.109
H33A	0.4038	0.2152	0.0069	0.055
H33B	0.4481	0.2676	-0.0169	0.055
H34A	0.5109	0.1661	0.0250	0.068
H34B	0.5616	0.2245	0.0137	0.068
H35A	0.5354	0.2076	-0.0547	0.096
H35B	0.5781	0.1502	-0.0353	0.096
H36A	0.4862	0.1173	-0.0759	0.164
H36B	0.4684	0.0973	-0.0312	0.164
H36C	0.4256	0.1549	-0.0507	0.164
H37A	0.0714	0.3281	0.3355	0.049
H37B	0.1232	0.2757	0.3545	0.049
H38A	0.1811	0.3885	0.3226	0.057
H38B	0.2294	0.3371	0.3455	0.057
H39A	0.1084	0.4026	0.3826	0.058
H39B	0.1637	0.3560	0.4049	0.058
H40A	0.1975	0.4586	0.4189	0.078
H40B	0.2635	0.4256	0.3948	0.078
H40C	0.2081	0.4722	0.3725	0.078
H41A	0.1791	0.3492	0.2631	0.052
H41B	0.1353	0.3026	0.2345	0.052
H42A	0.0641	0.3931	0.2835	0.064

Table 7. Derived Parameters for Hydrogen Atoms (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H42B	0.0203	0.3464	0.2548	0.064
H43A	0.0825	0.3912	0.1989	0.081
H43B	0.0313	0.4405	0.2215	0.081
H44A	0.1452	0.4849	0.2048	0.113
H44B	0.1373	0.4822	0.2521	0.113
H44C	0.1886	0.4329	0.2295	0.113
H45A	0.2058	0.2259	0.2626	0.053
H45B	0.2432	0.2700	0.2950	0.053
H46A	0.1657	0.1595	0.3155	0.084
H46B	0.2129	0.2017	0.3455	0.084
H47A	0.2851	0.1508	0.2783	0.103
H47B	0.3232	0.1739	0.3183	0.103
H48A	0.3336	0.0747	0.3112	0.225
H48B	0.2830	0.0907	0.3490	0.225
H48C	0.2448	0.0675	0.3087	0.225
H49A	0.0152	0.2624	0.2932	0.060
H49B	0.0612	0.2056	0.3111	0.060
H50A	0.1014	0.1814	0.2439	0.078
H50B	0.0438	0.2328	0.2285	0.078
H51A	-0.0547	0.1743	0.2625	0.100
H51B	-0.0217	0.1422	0.2235	0.100
H52A	-0.0347	0.0718	0.2731	0.149
H52B	0.0517	0.0798	0.2621	0.149
H52C	0.0188	0.1120	0.3011	0.149

STRUCTURE REPORT (6b)

XCL Code: DGH1707 **Date:** 21 December 2017

Compound: Tetra-*n*-butylammonium {*N'*-(hydroxy-κ*O*)-*N*-{2-(hydroxy-κ*O*)phenyl}benzene-carboximidamidato-κ*N*}(phenyl)borate

Formula: $C_{35}H_{50}BN_3O_2$

Supervisor: D. G. Hall **Crystallographer:** R. McDonald

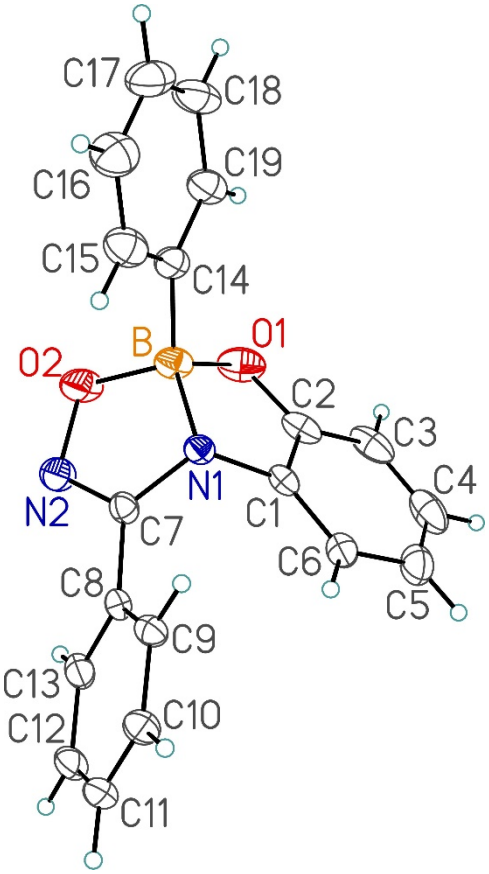
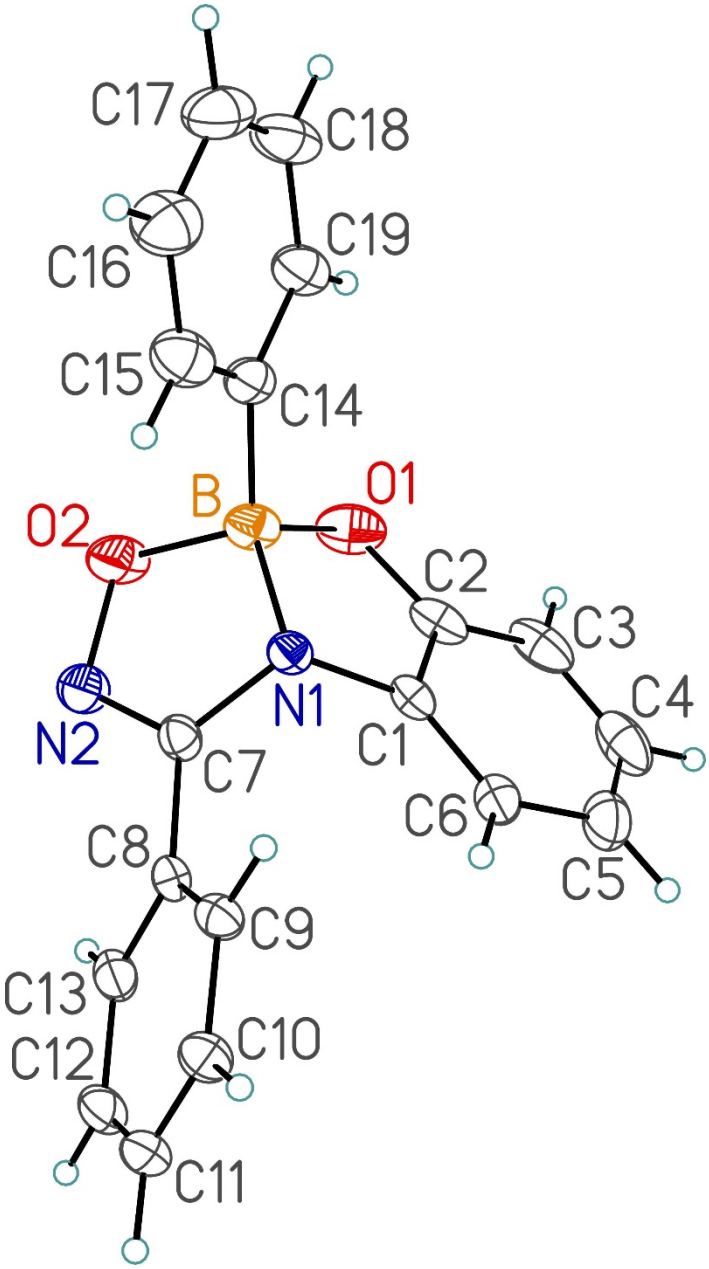
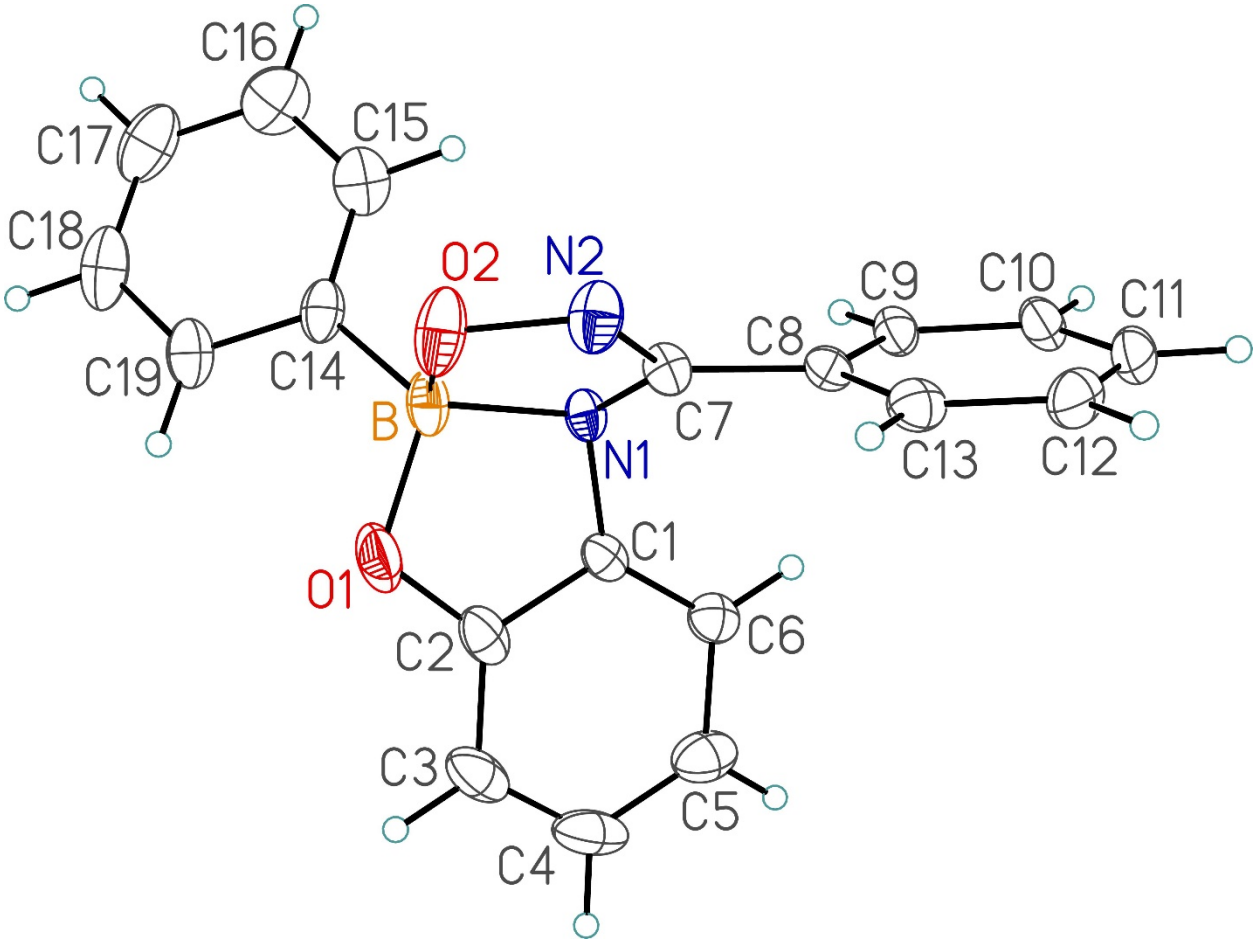


Figure Legends

Figure 1. Perspective view of the {*N'*-(hydroxy-κ*O*)-*N*-{2-(hydroxy-κ*O*)phenyl}benzene-carboximidamidato-κ*N*}(phenyl)borate ion showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Alternate view of the {*N'*-(hydroxy-κ*O*)-*N*-{2-(hydroxy-κ*O*)phenyl}benzene-carboximidamidato-κ*N*}(phenyl)borate ion.


C17
C18
C16
C19
C15
C14
B
O1
O2
C2
C3
N1
C4
N2
C7
C1
C6
C8
C5
C9
C13
C10
C12
C11


C16
C15
C17
C18
O2
N2
C9
C10
C11
C8
C19
C14
B
C7
N1
C13
C12
C1
O1
C2
C6
C3
C5
C4

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data	
formula	$C_{35}H_{50}BN_3O_2$
formula weight	555.59
crystal dimensions (mm)	0.37 × 0.23 × 0.06
crystal system	monoclinic
space group	$P2_1/c$ (No. 14)
unit cell parameters[a]	
a (Å)	9.7871(3)
b (Å)	17.5209(4)
c (Å)	19.0833(5)
β (deg)	94.1963(16)
V (Å^3)	3263.61(15)
Z	4
ρ_{calcd} (g cm^{-3})	1.131
μ (mm^{-1})	0.533
B. Data Collection and Refinement Conditions	
diffractometer	Bruker D8/APEX II CCD[b]
radiation (λ [Å])	Cu Kα (1.54178) (microfocus source)
temperature (°C)	–100
scan type	ω scans (1.0°) (5-10 s exposures)[c]
data collection 2θ limit (deg)	147.97
total data collected	90976 (-12 ≤ h ≤ 12, -21 ≤ k ≤ 21, -23 ≤ l ≤ 23)
independent reflections	6562 (R_{int} = 0.0893
number of observed reflections (*NO*)	5673 [$F_o^2 \geq 2\sigma(F_o^2)$]
structure solution method	direct methods/dual space (*SHELXD*[d])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[e])
absorption correction method	multi-scan (*TWINABS*)
range of transmission factors	0.9194–0.7139

data/restraints/parameters	6562 / 0 / 371
goodness-of-fit (S)[f] [all data]	1.051
final R indices[g]	
R_1 [$F_o^2 \geq 2\sigma(F_o^2)$]	0.0624
wR_2 [all data]	0.1793
largest difference peak and hole	0.695 and –0.275 e Å^{-3}

[a]Obtained from least-squares refinement of 9892 reflections with 6.86° < 2θ < 146.32°.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker. The crystal used for data collection was found to display non-merohedral twinning. Both components of the twin were indexed with the program *CELL_NOW* (Bruker AXS Inc., Madison, WI, 2004). The second twin component can be related to the first component by 180º rotation about the [0.15 0 1] axis in real space and about the [0 0 1] axis in reciprocal space. Integrated intensities for the reflections from the two components were written into a *SHELXL-2014* HKLF 5 reflection file with the data integration program *SAINT* (version 8.38A), using all reflection data (exactly overlapped, partially overlapped and non-overlapped). The refined value of the twin fraction (*SHELXL-2014* BASF parameter) was 0.2962(16).

[c]Data were collected with the detector set at three different positions. Low-angle (detector 2θ = –33º) and medium-angle (detector 2θ = 75º) data frames were collected using a scan time of 5 s, and high-angle (detector 2θ = 117º) frames using a scan time of 10 s.

[d]Schneider, T. R.; Sheldrick, G. M. *Acta Crystallogr.* **2002**, *D58*, 1772-1779.

[e]Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[f]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0962P)^2 + 1.1326P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[g]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

(a) atoms of the {N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}benzenecarboximidamidato-κN}(phenyl)borate ion

Atom	*x*	*y*	*z*	U_{eq}, Å^2
O1	0.11338(17)	0.40487(14)	0.34547(11)	0.0602(5)*
O2	0.2284(2)	0.52211(11)	0.30080(11)	0.0616(6)*
N1	0.34931(17)	0.40647(10)	0.31800(10)	0.0332(4)*
N2	0.3624(2)	0.53620(12)	0.33174(12)	0.0500(5)*
C1	0.3263(2)	0.35108(12)	0.37082(11)	0.0334(4)*
C2	0.1875(2)	0.35359(16)	0.38526(13)	0.0456(6)*
C3	0.1385(3)	0.30799(19)	0.43598(14)	0.0596(8)*
C4	0.2284(4)	0.25794(19)	0.47188(15)	0.0675(10)*
C5	0.3645(4)	0.25397(16)	0.45766(15)	0.0586(7)*
C6	0.4154(3)	0.30152(13)	0.40632(13)	0.0420(5)*
C7	0.4223(2)	0.47184(12)	0.34151(12)	0.0349(5)*
C8	0.5642(2)	0.46924(12)	0.37318(11)	0.0332(4)*
C9	0.6585(2)	0.41968(13)	0.34721(12)	0.0370(5)*
C10	0.7941(2)	0.41953(15)	0.37466(14)	0.0464(6)*
C11	0.8354(3)	0.46764(16)	0.42870(14)	0.0517(7)*
C12	0.7418(3)	0.51623(16)	0.45632(14)	0.0528(7)*
C13	0.6060(3)	0.51762(14)	0.42875(12)	0.0424(5)*
C14	0.1426(2)	0.42200(13)	0.21401(14)	0.0438(5)*
C15	0.2229(3)	0.43284(17)	0.15807(16)	0.0596(7)*
C16	0.1712(4)	0.4251(2)	0.08869(18)	0.0721(9)*
C17	0.0364(4)	0.4071(2)	0.07361(18)	0.0741(10)*
C18	-0.0464(3)	0.39507(19)	0.12757(19)	0.0698(9)*
C19	0.0069(3)	0.40183(16)	0.19726(16)	0.0524(6)*
B	0.2053(3)	0.43871(17)	0.29312(16)	0.0439(6)*

(b) tetra-n-butylammonium ion atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
N3	0.1346(2)	0.14983(10)	0.23890(10)	0.0385(4)*
C20	0.0495(3)	0.18481(13)	0.29379(12)	0.0418(5)*
C21	-0.0659(3)	0.13570(16)	0.31794(15)	0.0510(6)*
C22	-0.1568(3)	0.18209(19)	0.36327(19)	0.0656(8)*
C23	-0.2755(4)	0.1373(2)	0.3880(2)	0.0782(10)*
C24	0.2398(2)	0.20954(13)	0.22193(13)	0.0405(5)*
C25	0.3360(3)	0.18709(18)	0.16618(15)	0.0538(7)*
C26	0.4455(3)	0.2473(2)	0.15780(18)	0.0657(8)*
C27	0.5565(4)	0.2465(3)	0.2159(2)	0.0853(12)*
C28	0.0454(3)	0.12743(15)	0.17344(13)	0.0455(6)*
C29	-0.0437(3)	0.18961(18)	0.13967(14)	0.0539(7)*

Table 2. Atomic Coordinates and Displacement Parameters (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C30	-0.1278(3)	0.1562(2)	0.07671(15)	0.0660(9)*
C31	-0.2203(4)	0.2144(3)	0.03875(18)	0.0920(13)*
C32	0.2035(3)	0.07685(14)	0.26450(16)	0.0519(6)*
C33	0.2931(4)	0.0815(2)	0.33208(18)	0.0695(9)*
C34	0.3711(4)	0.0030(2)	0.3409(2)	0.0784(10)*
C35	0.4293(4)	-0.0074(3)	0.4133(2)	0.1040(15)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

(a) within the {N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}benzenecarboximidamidato-κN}-(phenyl)borate ion

Atom1	Atom2	Distance	Atom1	Atom2	Distance
O1	C2	1.353(3)	C7	C8	1.474(3)
O1	B	1.514(4)	C8	C9	1.385(3)
O2	N2	1.420(3)	C8	C13	1.395(3)
O2	B	1.484(4)	C9	C10	1.390(3)
N1	C1	1.429(3)	C10	C11	1.370(4)
N1	C7	1.406(3)	C11	C12	1.383(4)
N1	B	1.559(3)	C12	C13	1.394(4)
N2	C7	1.278(3)	C14	C15	1.384(4)
C1	C2	1.406(3)	C14	C19	1.389(4)
C1	C6	1.374(3)	C14	B	1.614(4)
C2	C3	1.369(4)	C15	C16	1.389(4)
C3	C4	1.387(5)	C16	C17	1.366(5)
C4	C5	1.380(5)	C17	C18	1.372(5)
C5	C6	1.405(4)	C18	C19	1.397(4)

(b) within the tetra-n-butylammonium ion

Atom1	Atom2	Distance	Atom1	Atom2	Distance
N3	C20	1.515(3)	C25	C26	1.521(4)
N3	C24	1.519(3)	C26	C27	1.494(5)
N3	C28	1.522(3)	C28	C29	1.510(4)
N3	C32	1.510(3)	C29	C30	1.523(4)
C20	C21	1.518(3)	C30	C31	1.513(5)
C21	C22	1.521(4)	C32	C33	1.508(4)
C22	C23	1.506(4)	C33	C34	1.575(5)
C24	C25	1.523(4)	C34	C35	1.468(5)

Table 4. Selected Interatomic Angles (deg)

(a) within the {N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}benzenecarboximidamidato-κN}-(phenyl)borate ion

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C2	O1	B		N2	C7	C8	119.7(2)
	108.07(18)			C7	C8	C9	
N2	O2	B			120.36(19)		
	109.98(17)			C7	C8	C13	120.5(2)
C1	N1	C7		C9	C8	C13	119.1(2)
	115.53(17)			C8	C9	C10	120.6(2)
C1	N1	B		C9	C10	C11	120.3(3)
	105.97(18)			C10	C11	C12	119.9(2)
C7	N1	B		C11	C12	C13	120.5(2)
	103.21(18)			C8	C13	C12	119.7(2)
O2	N2	C7		C15	C14	C19	116.4(3)
	107.84(19)			C15	C14	B	119.8(2)
N1	C1	C2	108.8(2)	C19	C14	B	123.6(3)
N1	C1	C6	130.5(2)	C14	C15	C16	122.3(3)
C2	C1	C6	120.7(2)	C15	C16	C17	120.1(3)
O1	C2	C1	113.4(2)	C16	C17	C18	119.4(3)
O1	C2	C3	125.7(2)	C17	C18	C19	120.1(3)
C1	C2	C3	120.9(3)	C14	C19	C18	121.6(3)
C2	C3	C4	118.4(3)	O1	B	O2	114.5(2)
C3	C4	C5	121.4(3)	O1	B	N1	103.0(2)
C4	C5	C6	120.3(3)	O1	B	C14	110.0(2)
C1	C6	C5	118.3(3)	O2	B	N1	
N1	C7	N2	117.1(2)		101.46(19)		
N1	C7	C8		O2	B	C14	108.4(2)
	123.15(18)			N1	B	C14	119.5(2)

(b) within the tetra-n-butylammonium ion

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C20	N3	C24		N3	C20	C21	
	106.52(17)				116.08(19)		
C20	N3	C28		C20	C21	C22	110.4(2)
	111.11(18)			C21	C22	C23	113.3(3)
C20	N3	C32	112.0(2)				
C24	N3	C28					
	111.09(19)						
C24	N3	C32					
	111.01(19)						
C28	N3	C32					
	105.24(18)						

N3	C24	C25	115.7(2)
C24	C25	C26	111.7(2)
C25	C26	C27	113.3(3)
N3	C28	C29	116.3(2)
C28	C29	C30	108.7(2)
C29	C30	C31	112.9(3)
N3	C32	C33	116.7(2)
C32	C33	C34	106.9(3)
C33	C34	C35	111.1(4)

Table 5. Torsional Angles (deg)

(a) within the {N'-(hydroxy-κO)-N-{2-(hydroxy-κO)phenyl}benzenecarboximidamidato-κN}-(phenyl)borate ion

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
B	O1	C2	C1	4.7(3)	O1	C2	C3	C4	179.6(2)
B	O1	C2	C3	-176.3(2)	C1	C2	C3	C4	-1.4(4)
C2	O1	B	O2	-117.4(2)	C2	C3	C4	C5	0.4(4)
C2	O1	B	N1	-8.2(3)	C3	C4	C5	C6	0.5(4)
C2	O1	B	C14	120.3(2)	C4	C5	C6	C1	-0.5(4)
B	O2	N2	C7	1.7(3)	N1	C7	C8	C9	38.6(3)
N2	O2	B	O1	105.4(2)	N1	C7	C8	C13	-142.7(2)
N2	O2	B	N1	-4.7(3)	N2	C7	C8	C9	-138.4(2)
N2	O2	B	C14	-131.4(2)	N2	C7	C8	C13	40.3(3)
C7	N1	C1	C2	106.9(2)	C7	C8	C9	C10	176.9(2)
C7	N1	C1	C6	-72.0(3)	C13	C8	C9	C10	-1.8(3)
B	N1	C1	C2	-6.7(2)	C7	C8	C13	C12	-177.7(2)
B	N1	C1	C6	174.4(2)	C9	C8	C13	C12	1.1(3)
C1	N1	C7	N2	-120.8(2)	C8	C9	C10	C11	1.1(4)
C1	N1	C7	C8	62.1(3)	C9	C10	C11	C12	0.4(4)
B	N1	C7	N2	-5.6(3)	C10	C11	C12	C13	-1.1(4)
B	N1	C7	C8	177.3(2)	C11	C12	C13	C8	0.4(4)
C1	N1	B	O1	8.9(2)	C19	C14	C15	C16	0.8(4)
C1	N1	B	O2	127.6(2)	B	C14	C15	C16	-174.9(3)
C1	N1	B	C14	-113.4(2)	C15	C14	C19	C18	-1.8(4)
C7	N1	B	O1	-112.9(2)	B	C14	C19	C18	173.7(3)
C7	N1	B	O2	5.8(2)	C15	C14	B	O1	-166.1(2)
C7	N1	B	C14	124.8(2)	C15	C14	B	O2	68.1(3)
O2	N2	C7	N1	2.7(3)	C15	C14	B	N1	-47.3(4)
O2	N2	C7	C8	179.9(2)	C19	C14	B	O1	18.6(3)
N1	C1	C2	O1	1.5(3)	C19	C14	B	O2	-107.3(3)
N1	C1	C2	C3	-177.7(2)	C19	C14	B	N1	137.3(3)
C6	C1	C2	O1	-179.5(2)	C14	C15	C16	C17	0.9(5)
C6	C1	C2	C3	1.4(3)	C15	C16	C17	C18	-1.6(5)
N1	C1	C6	C5	178.4(2)	C16	C17	C18	C19	0.6(5)
C2	C1	C6	C5	-0.5(3)	C17	C18	C19	C14	1.2(5)

(b) within the tetra-n-butylammonium ion

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C24	N3	C20	C21	178.6(2)	C20	N3	C24	C25	-178.8(2)
C28	N3	C20	C21	57.5(3)	C28	N3	C24	C25	-57.7(3)
C32	N3	C20	C21	-59.9(3)	C32	N3	C24	C25	59.1(3)

C20	N3	C28	C29	54.5(3)
C24	N3	C28	C29	-63.9(3)

Table 5. Torsional Angles (continued)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C32	N3	C28	C29	175.9(2)	N3	C24	C25	C26	-174.7(2)
C20	N3	C32	C33	-55.7(3)	C24	C25	C26	C27	75.8(4)
C24	N3	C32	C33	63.2(3)	N3	C28	C29	C30	-178.4(2)
C28	N3	C32	C33	-176.6(3)	C28	C29	C30	C31	-179.6(3)
N3	C20	C21	C22	-170.6(2)	N3	C32	C33	C34	-170.9(3)
C20	C21	C22	C23	178.8(3)	C32	C33	C34	C35	-163.5(3)

Table 6. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0270(8)	0.0899(15)	0.0639(12)	-0.0100(11)	0.0047(8)	0.0075(9)
O2	0.0508(11)	0.0455(10)	0.0846(14)	-0.0187(10)	-0.0225(10)	0.0214(9)
N1	0.0257(8)	0.0313(9)	0.0422(9)	-0.0036(7)	0.0007(7)	0.0034(7)
N2	0.0470(12)	0.0371(11)	0.0640(13)	-0.0069(9)	-0.0093(10)	0.0074(9)
C1	0.0322(10)	0.0334(10)	0.0349(10)	-0.0093(8)	0.0052(8)	-0.0075(8)
C2	0.0349(12)	0.0579(15)	0.0445(12)	-0.0208(11)	0.0053(10)	-0.0114(11)
C3	0.0507(15)	0.082(2)	0.0477(14)	-0.0217(14)	0.0159(13)	-0.0320(15)
C4	0.090(2)	0.072(2)	0.0423(13)	-0.0061(13)	0.0193(15)	-0.0452(19)
C5	0.079(2)	0.0477(15)	0.0485(14)	0.0064(12)	0.0030(14)	-0.0132(14)
C6	0.0443(13)	0.0374(12)	0.0442(12)	-0.0017(10)	0.0036(10)	-0.0053(10)
C7	0.0320(10)	0.0297(10)	0.0431(11)	-0.0029(9)	0.0034(9)	-0.0002(8)
C8	0.0326(10)	0.0297(10)	0.0374(10)	0.0035(8)	0.0034(9)	-0.0071(8)
C9	0.0307(10)	0.0396(12)	0.0405(11)	-0.0009(9)	0.0018(9)	-0.0057(9)
C10	0.0292(11)	0.0523(14)	0.0575(14)	0.0073(11)	0.0032(10)	-0.0062(10)
C11	0.0391(13)	0.0547(15)	0.0592(15)	0.0135(12)	-0.0117(12)	-0.0165(12)
C12	0.0631(17)	0.0486(14)	0.0441(13)	0.0011(11)	-0.0131(12)	-0.0225(13)
C13	0.0495(13)	0.0361(11)	0.0417(11)	-0.0007(9)	0.0042(10)	-0.0097(10)
C14	0.0369(12)	0.0353(11)	0.0574(14)	-0.0053(10)	-0.0080(11)	0.0106(9)
C15	0.0534(16)	0.0595(17)	0.0654(17)	-0.0132(14)	0.0004(14)	0.0024(14)
C16	0.084(2)	0.074(2)	0.0586(17)	-0.0126(16)	0.0038(17)	0.0036(19)
C17	0.091(3)	0.067(2)	0.0599(18)	-0.0171(15)	-0.0248(18)	0.0194(18)
C18	0.0533(17)	0.0662(19)	0.085(2)	-0.0238(17)	-0.0255(17)	0.0131(15)
C19	0.0393(13)	0.0489(14)	0.0674(16)	-0.0125(13)	-0.0059(12)	0.0089(11)
B	0.0281(12)	0.0478(15)	0.0552(15)	-0.0102(12)	-0.0019(11)	0.0121(11)
N3	0.0392(10)	0.0293(9)	0.0467(10)	-0.0091(8)	0.0018(9)	-0.0046(8)
C20	0.0427(12)	0.0389(12)	0.0439(12)	-0.0109(10)	0.0035(10)	-0.0051(10)
C21	0.0476(14)	0.0466(14)	0.0592(15)	-0.0071(12)	0.0072(12)	-0.0091(11)
C22	0.0519(16)	0.0666(19)	0.081(2)	-0.0214(16)	0.0209(15)	-0.0168(14)
C23	0.0577(18)	0.074(2)	0.106(3)	-0.014(2)	0.030(2)	-0.0130(17)
C24	0.0366(12)	0.0320(11)	0.0524(13)	-0.0063(10)	0.0004(10)	-0.0060(9)
C25	0.0463(14)	0.0641(17)	0.0517(14)	-0.0113(12)	0.0069(12)	-0.0079(13)
C26	0.0514(17)	0.074(2)	0.0739(19)	0.0056(16)	0.0168(15)	-0.0048(15)
C27	0.066(2)	0.115(3)	0.077(2)	-0.036(2)	0.0161(18)	-0.037(2)
C28	0.0404(12)	0.0457(13)	0.0505(13)	-0.0174(11)	0.0037(11)	-0.0080(11)
C29	0.0448(14)	0.0678(18)	0.0480(14)	-0.0140(13)	-0.0031(11)	0.0034(13)
C30	0.0569(17)	0.096(2)	0.0447(14)	-0.0015(15)	-0.0010(13)	-0.0292(17)
C31	0.060(2)	0.163(4)	0.0509(17)	-0.004(2)	-0.0100(15)	0.004(2)
C32	0.0478(14)	0.0338(12)	0.0738(17)	-0.0003(12)	0.0015(13)	-0.0010(11)
C33	0.0652(19)	0.076(2)	0.0669(18)	0.0111(16)	-0.0007(16)	0.0142(17)

Table 6. Anisotropic Displacement Parameters (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C34	0.063(2)	0.092(3)	0.080(2)	0.014(2)	0.0026(18)	-0.0046(19)
C35	0.072(2)	0.146(4)	0.095(3)	0.043(3)	0.012(2)	0.013(3)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H3	0.045174	0.310549	0.446357	0.072
H4	0.195633	0.225710	0.506998	0.081
H5	0.423961	0.218953	0.482680	0.070
H6	0.509071	0.299452	0.396402	0.050
H9	0.630362	0.385499	0.310301	0.044
H10	0.858237	0.385928	0.355886	0.056
H11	0.928238	0.467627	0.447165	0.062
H12	0.770318	0.548890	0.494353	0.063
H13	0.542224	0.551341	0.447693	0.051
H15	0.316546	0.446064	0.167455	0.072
H16	0.229699	0.432212	0.051653	0.086
H17	0.000276	0.403011	0.026180	0.089
H18	-0.140038	0.382127	0.117534	0.084
H19	-0.051182	0.392392	0.234082	0.063
H20A	0.009563	0.233079	0.274690	0.050
H20B	0.111408	0.197938	0.335400	0.050
H21A	-0.027036	0.091732	0.345218	0.061
H21B	-0.121309	0.115646	0.276499	0.061
H22A	-0.100646	0.201419	0.404764	0.079
H22B	-0.192751	0.226770	0.336031	0.079
H23A	-0.330015	0.170078	0.416778	0.094
H23B	-0.332987	0.118944	0.347196	0.094
H23C	-0.240814	0.093574	0.415980	0.094
H24A	0.295860	0.222070	0.265724	0.049
H24B	0.190559	0.256543	0.206164	0.049
H25A	0.380528	0.137905	0.179390	0.065
H25B	0.282221	0.179860	0.120670	0.065
H26A	0.486771	0.238928	0.112590	0.079
H26B	0.401829	0.298306	0.155910	0.079
H27A	0.623676	0.286371	0.207585	0.102
H27B	0.516825	0.256011	0.260747	0.102
H27C	0.601828	0.196591	0.217396	0.102
H28A	0.105846	0.107969	0.138075	0.055
H28B	-0.014619	0.084787	0.185890	0.055
H29A	0.014226	0.231855	0.124246	0.065
H29B	-0.105334	0.210165	0.173973	0.065
H30A	-0.064875	0.134869	0.043411	0.079
H30B	-0.184473	0.113778	0.092869	0.079
H31A	-0.271843	0.189936	-0.001118	0.110
H31B	-0.284356	0.234896	0.071129	0.110

Table 7. Derived Parameters for Hydrogen Atoms (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H31C	-0.164713	0.255995	0.021652	0.110
H32A	0.131444	0.038274	0.270592	0.062
H32B	0.260188	0.057962	0.227247	0.062
H33A	0.236530	0.090273	0.372253	0.083
H33B	0.359301	0.124001	0.330010	0.083
H34A	0.306801	-0.039239	0.328247	0.094
H34B	0.445517	0.001343	0.308446	0.094
H35A	0.477170	-0.056500	0.417358	0.125
H35B	0.494136	0.033968	0.425610	0.125
H35C	0.355569	-0.006570	0.445390	0.125

STRUCTURE REPORT (7)

XCL Code: DGH1706 **Date:** 21 December 2017

Compound: 3,7a-Diphenyl-5,6-dihydro[1,3]oxazolo[3,2-*d*][1,2,4]oxadiazole

Formula: $C_{16}H_{14}N_2O_2$

Supervisor: D. G. Hall **Crystallographer:** R. McDonald

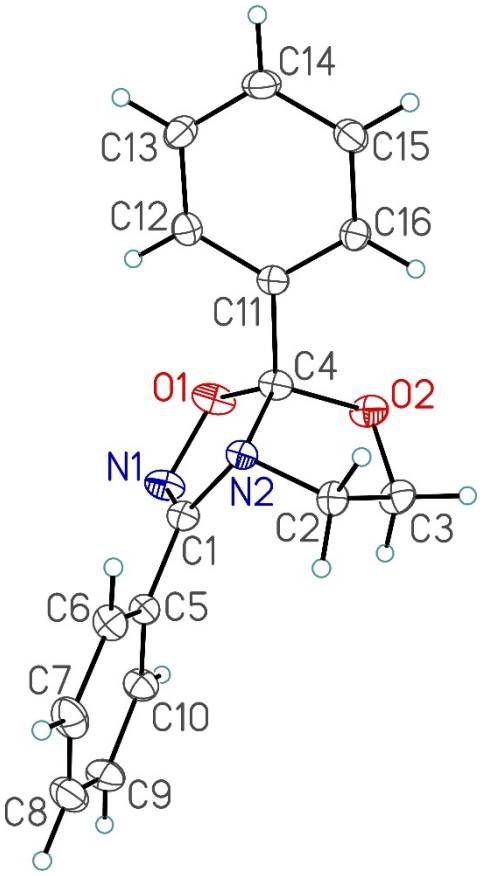
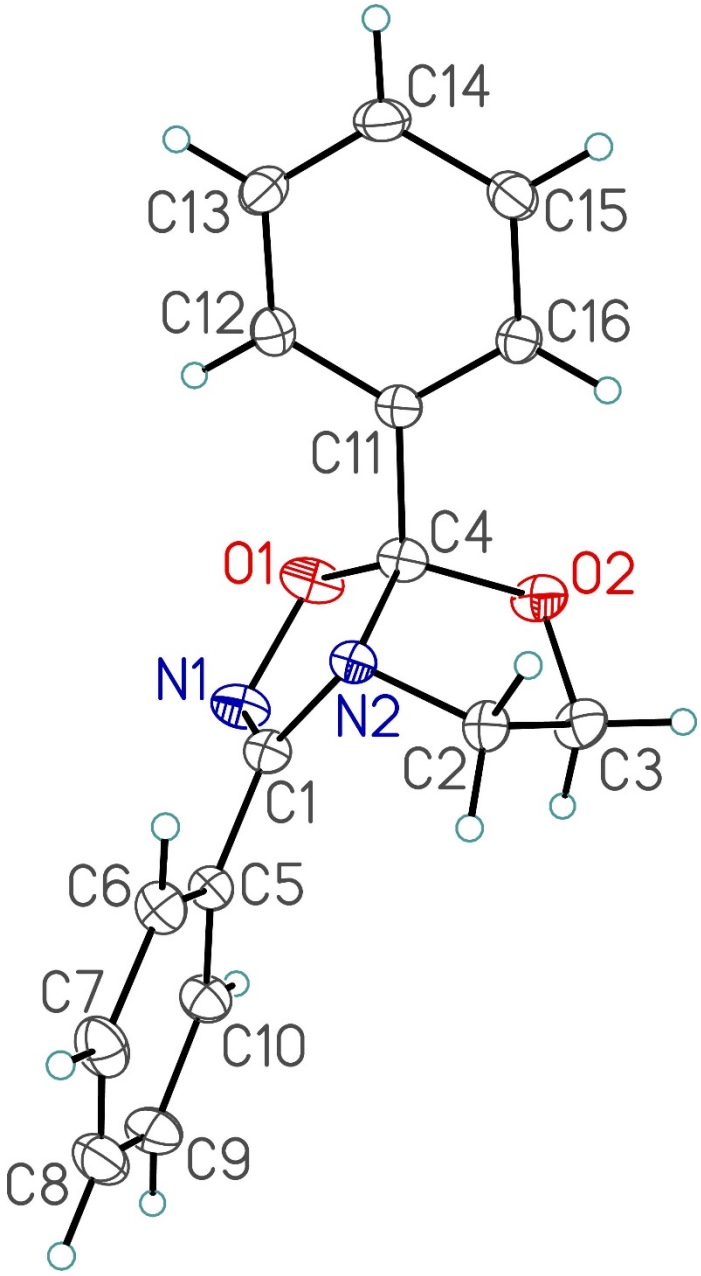


Figure Legends

Figure 1. Perspective view of the 3,7a-diphenyl-5,6-dihydro[1,3]oxazolo[3,2-*d*][1,2,4]oxadiazole molecule showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 30% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

Figure 2. Alternate view of the molecule.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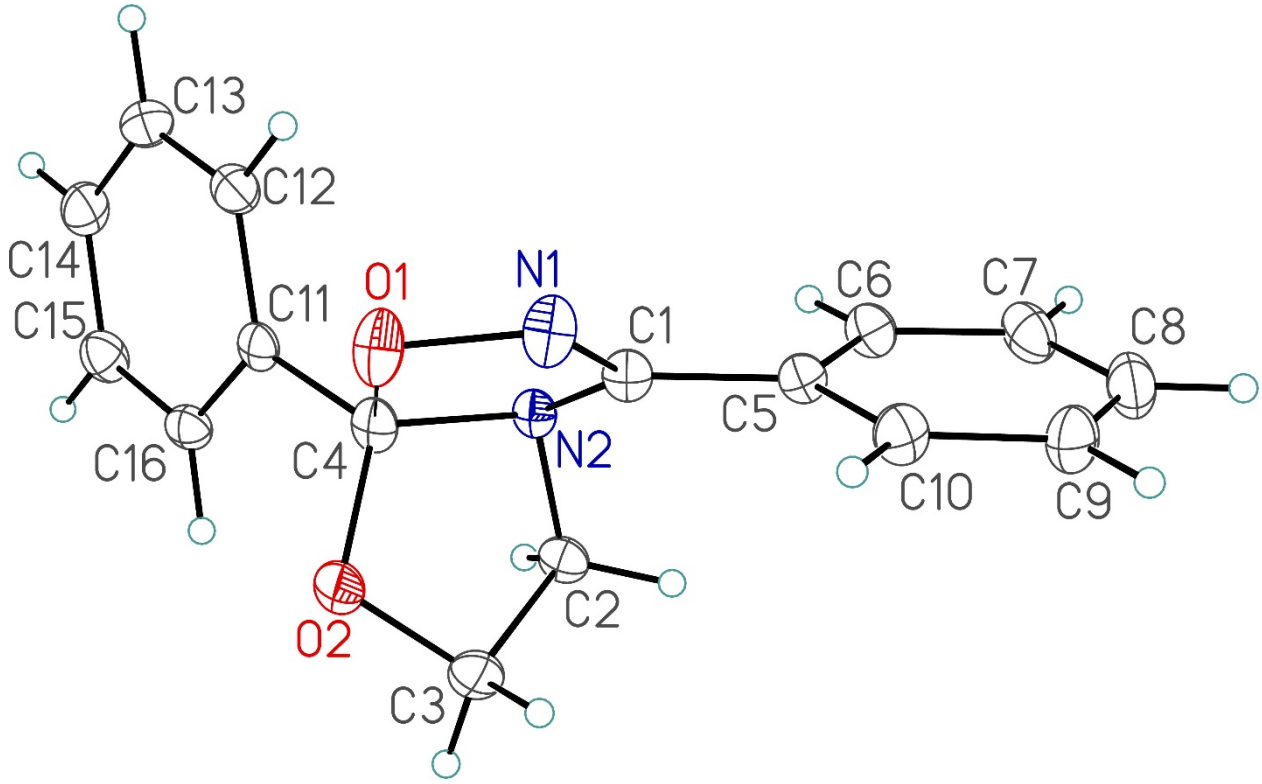
C15
C12
C16
C11
C4
O1
O2
N1
N2
C2
C3
C1
C6
C5
C7
C10
C8
C9


C13
C12
C14
C15
C11
O1
N1
C1
C6
C7
C8
C5
C16
C4
N2
C10
C9
C2
O2
C3

List of Tables

Table 1. Crystallographic Experimental Details

A. Crystal Data

formula	$C_{16}H_{14}N_2O_2$
formula weight	266.29
crystal dimensions (mm)	0.73 × 0.55 × 0.12
crystal system	monoclinic
space group	$P2_1/c$ (No. 14)
unit cell parameters[a]	
a (Å)	8.9000(2)
b (Å)	9.5626(3)
c (Å)	15.9864(5)
β (deg)	98.4476(10)
V (Å^3)	1345.80(7)
Z	4
ρ_{calcd} (g cm^{-3})	1.314
μ (mm^{-1})	0.714

B. Data Collection and Refinement Conditions

diffractometer	Bruker D8/APEX II CCD[b]
radiation (λ [Å])	Cu Kα (1.54178) (microfocus source)
temperature (°C)	–100
scan type	ω and ϕ scans (1.0°) (5 s exposures)
data collection 2θ limit (deg)	147.57
total data collected	9199 (-11 ≤ h ≤ 11, -11 ≤ k ≤ 11, -16 ≤ l ≤ 19)
independent reflections	2621 (R_{int} = 0.0354)
number of observed reflections (*NO*)	2545 [$F_o^2 \geq 2\sigma(F_o^2)$]
structure solution method	direct methods/dual space (*SHELXD*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–2014*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	1.0000–0.5852
data/restraints/parameters	2621 / 0 / 181

goodness-of-fit (S)[e] [all data]	1.034
final R indices[f]	
R_1 [$F_o^2 \geq 2\sigma(F_o^2)$]	0.0403
wR_2 [all data]	0.1064
largest difference peak and hole	0.267 and –0.232 e Å^{-3}

[a]Obtained from least-squares refinement of 9708 reflections with $10.82° < 2\theta < 147.58°$.

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[c]Schneider, T. R.; Sheldrick, G. M. *Acta Crystallogr.* **2002**, *D58*, 1772-1779.

[d]Sheldrick, G. M. *Acta Crystallogr.* **2015**, *C71*, 3–8.

[e]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0597P)^2 + 0.3398P]^{-1}$ where $P = [\text{Max}(F_o^2, 0) + 2F_c^2]/3$).

[f]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	*x*	*y*	*z*	U_{eq}, Å^2
O1	0.23178(10)	0.35084(9)	0.37277(6)	0.0404(2)*
O2	0.08866(10)	0.39212(8)	0.23928(6)	0.0389(2)*
N1	0.13377(12)	0.33608(11)	0.43632(7)	0.0375(3)*
N2	0.03611(10)	0.20586(8)	0.32277(6)	0.0249(2)*
C1	0.02483(12)	0.25612(11)	0.40362(7)	0.0279(2)*
C2	-0.09659(13)	0.22720(11)	0.25674(7)	0.0302(3)*
C3	-0.07449(14)	0.37863(12)	0.23196(9)	0.0375(3)*
C4	0.15893(12)	0.29029(10)	0.29612(8)	0.0293(3)*
C5	-0.10125(12)	0.21773(11)	0.44928(7)	0.0284(2)*
C6	-0.16039(13)	0.08234(12)	0.44351(8)	0.0342(3)*
C7	-0.27817(15)	0.04789(15)	0.48778(9)	0.0438(3)*
C8	-0.33918(16)	0.14800(17)	0.53552(9)	0.0482(3)*
C9	-0.28170(16)	0.28271(16)	0.54041(9)	0.0459(3)*
C10	-0.16162(15)	0.31789(13)	0.49809(8)	0.0367(3)*
C11	0.27337(12)	0.20786(10)	0.25485(7)	0.0269(2)*
C12	0.39077(13)	0.13679(12)	0.30465(8)	0.0346(3)*
C13	0.48641(14)	0.05010(14)	0.26710(9)	0.0422(3)*
C14	0.46677(15)	0.03379(13)	0.18035(9)	0.0414(3)*
C15	0.35207(15)	0.10624(13)	0.13047(8)	0.0376(3)*
C16	0.25573(13)	0.19323(11)	0.16755(8)	0.0313(3)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
O1	N1	1.4399(13)	C5	C10	1.3921(16)
O1	C4	1.4226(14)	C6	C7	1.3878(17)
O2	C3	1.4453(15)	C7	C8	1.384(2)
O2	C4	1.4135(14)	C8	C9	1.384(2)
N1	C1	1.2842(15)	C9	C10	1.3876(18)
N2	C1	1.3963(14)	C11	C12	1.3939(16)
N2	C2	1.4780(14)	C11	C16	1.3883(17)
N2	C4	1.4714(13)	C12	C13	1.3865(18)
C1	C5	1.4722(15)	C13	C14	1.381(2)
C2	C3	1.5217(15)	C14	C15	1.3852(18)
C4	C11	1.5137(14)	C15	C16	1.3885(16)
C5	C6	1.3955(16)			

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
N1	O1	C4	108.73(8)	C1	C5	C6	120.40(10)
C3	O2	C4	109.57(8)	C1	C5	C10	119.43(10)
O1	N1	C1	105.70(9)	C6	C5	C10	120.17(11)
C1	N2	C2	117.06(9)	C5	C6	C7	119.49(12)
C1	N2	C4	103.69(8)	C6	C7	C8	120.26(12)
C2	N2	C4	105.49(8)	C7	C8	C9	120.22(12)
N1	C1	N2	115.41(10)	C8	C9	C10	120.19(12)
N1	C1	C5	122.11(10)	C5	C10	C9	119.65(12)
N2	C1	C5	122.47(9)	C4	C11	C12	120.06(10)
N2	C2	C3	101.36(9)	C4	C11	C16	120.44(10)
O2	C3	C2	103.33(9)	C12	C11	C16	119.31(10)
O1	C4	O2	112.08(8)	C11	C12	C13	120.04(12)
O1	C4	N2	103.76(9)	C12	C13	C14	120.48(12)
O1	C4	C11	109.67(9)	C13	C14	C15	119.70(11)
O2	C4	N2	106.66(9)	C14	C15	C16	120.20(12)
O2	C4	C11	109.94(9)	C11	C16	C15	120.25(11)
N2	C4	C11	114.62(8)				

Table 5. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle	Atom1	Atom2	Atom3	Atom4	Angle
C4	O1	N1	C1	8.70(13)	N2	C1	C5	C10	-141.86(11)
N1	O1	C4	O2	99.42(10)	N2	C2	C3	O2	35.27(12)
N1	O1	C4	N2	-15.29(11)	O1	C4	C11	C12	34.62(13)
N1	O1	C4	C11	-138.18(9)	O1	C4	C11	C16	-150.45(10)
C4	O2	C3	C2	-23.89(13)	O2	C4	C11	C12	158.29(10)
C3	O2	C4	O1	-110.48(10)	O2	C4	C11	C16	-26.78(13)
C3	O2	C4	N2	2.43(12)	N2	C4	C11	C12	-81.59(13)
C3	O2	C4	C11	127.26(10)	N2	C4	C11	C16	93.34(12)
O1	N1	C1	N2	2.34(14)	C1	C5	C6	C7	179.32(12)
O1	N1	C1	C5	-179.02(10)	C10	C5	C6	C7	-0.96(18)
C2	N2	C1	N1	-127.50(11)	C1	C5	C10	C9	179.20(12)
C2	N2	C1	C5	53.87(13)	C6	C5	C10	C9	-0.52(19)
C4	N2	C1	N1	-11.82(13)	C5	C6	C7	C8	1.7(2)
C4	N2	C1	C5	169.54(10)	C6	C7	C8	C9	-0.9(2)
C1	N2	C2	C3	80.34(11)	C7	C8	C9	C10	-0.6(2)
C4	N2	C2	C3	-34.34(10)	C8	C9	C10	C5	1.3(2)
C1	N2	C4	O1	15.80(10)	C4	C11	C12	C13	173.54(10)
C1	N2	C4	O2	-102.71(10)	C16	C11	C12	C13	-1.44(17)
C1	N2	C4	C11	135.37(10)	C4	C11	C16	C15	-173.55(10)
C2	N2	C4	O1	139.40(9)	C12	C11	C16	C15	1.41(16)
C2	N2	C4	O2	20.90(10)	C11	C12	C13	C14	0.26(19)
C2	N2	C4	C11	-101.03(11)	C12	C13	C14	C15	1.0(2)
N1	C1	C5	C6	-140.68(12)	C13	C14	C15	C16	-1.0(2)
N1	C1	C5	C10	39.59(17)	C14	C15	C16	C11	-0.20(18)
N2	C1	C5	C6	37.86(16)					

Table 6. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0400(5)	0.0360(5)	0.0485(6)	-0.0193(4)	0.0176(4)	-0.0172(4)
O2	0.0420(5)	0.0154(4)	0.0627(6)	0.0089(3)	0.0194(4)	0.0029(3)
N1	0.0388(5)	0.0336(5)	0.0426(6)	-0.0157(4)	0.0142(4)	-0.0096(4)
N2	0.0296(4)	0.0153(4)	0.0310(5)	-0.0032(3)	0.0081(4)	-0.0033(3)
C1	0.0335(5)	0.0178(5)	0.0332(6)	-0.0050(4)	0.0074(4)	-0.0013(4)
C2	0.0363(6)	0.0198(5)	0.0342(6)	0.0003(4)	0.0037(4)	-0.0022(4)
C3	0.0402(6)	0.0205(5)	0.0522(8)	0.0044(5)	0.0086(5)	0.0030(4)
C4	0.0350(6)	0.0143(5)	0.0405(7)	-0.0034(4)	0.0118(5)	-0.0052(4)
C5	0.0314(5)	0.0256(5)	0.0287(6)	-0.0012(4)	0.0057(4)	-0.0007(4)
C6	0.0391(6)	0.0282(6)	0.0358(6)	-0.0011(4)	0.0068(5)	-0.0050(4)
C7	0.0457(7)	0.0447(7)	0.0410(8)	0.0062(5)	0.0064(5)	-0.0152(6)
C8	0.0400(7)	0.0678(10)	0.0390(8)	0.0062(6)	0.0131(5)	-0.0070(6)
C9	0.0457(7)	0.0549(8)	0.0403(8)	-0.0042(6)	0.0175(6)	0.0055(6)
C10	0.0428(6)	0.0317(6)	0.0371(7)	-0.0051(5)	0.0111(5)	0.0007(5)
C11	0.0306(5)	0.0162(5)	0.0355(6)	0.0011(4)	0.0099(4)	-0.0042(4)
C12	0.0374(6)	0.0343(6)	0.0326(6)	0.0042(4)	0.0068(5)	-0.0002(5)
C13	0.0364(6)	0.0425(7)	0.0483(8)	0.0104(5)	0.0086(5)	0.0110(5)
C14	0.0421(7)	0.0356(6)	0.0506(8)	0.0035(5)	0.0208(6)	0.0099(5)
C15	0.0468(7)	0.0347(6)	0.0335(7)	0.0009(5)	0.0134(5)	0.0027(5)
C16	0.0357(6)	0.0235(5)	0.0352(6)	0.0040(4)	0.0069(4)	0.0016(4)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H2A	-0.193260	0.214356	0.279527	0.036
H2B	-0.094190	0.163167	0.208292	0.036
H3A	-0.123656	0.397082	0.173343	0.045
H3B	-0.116429	0.443717	0.270787	0.045
H6	-0.120324	0.014272	0.409546	0.041
H7	-0.317074	-0.044770	0.485297	0.053
H8	-0.420772	0.124168	0.565029	0.058
H9	-0.324588	0.351295	0.572826	0.055
H10	-0.120822	0.409850	0.502397	0.044
H12	0.405260	0.147769	0.364351	0.042
H13	0.566019	0.001561	0.301294	0.051
H14	0.531634	-0.026847	0.154991	0.050
H15	0.339279	0.096376	0.070710	0.045
H16	0.177444	0.242926	0.133055	0.038