

# An Improved System for the Palladium-Catalyzed Borylation of Aryl Halides with Pinacol Borane

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## Experimental Section

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**General Considerations.** All reactions were stirred with the aid of a magnetic stirrer and carried out under an argon atmosphere. 1,4-Dioxane (anhydrous), triethylamine ( $\geq 99.5\%$ ) and pinacol borane (97%) were purchased from Aldrich Chemical Co. in SureSeal® bottles. Commercially available materials were used without further purification unless otherwise noted. SPhos (**1**) and aryl halides were purchased from Aldrich Chemical Co. Liquid aryl halides were purified by passage through a pad of basic alumina prior to use.  $\text{PdCl}_2(\text{CH}_3\text{CN})_2$  was purchased from Strem Chemicals, Inc. and stored in a benchtop desiccator.

All new compounds were characterized by  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, IR spectroscopy, melting points (for solids) and, in some cases, elemental analysis. Known compounds were characterized by  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR and melting points (for solids) and compared to their literature values.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian Mercury 300. Infrared spectra were recorded on an ASI Applied Systems ReactIR 1000 (neat samples were placed directly on the DiComp probe). Elemental analyses were performed by Atlantic Microlabs Inc., Norcross, GA. All  $^1\text{H}$  NMR experiments are reported in  $\delta$  units, parts per million (ppm) downfield of TMS and were measured relative to the signals for the residual benzene (7.16 ppm), chloroform (7.26 ppm), dimethylsulfoxide

(2.50 ppm) or methanol (3.31 ppm). All  $^{13}\text{C}$  NMR spectra were reported in ppm relative to residual chloroform (77 ppm), dimethylsulfoxide (39.5 ppm) or methanol (49 ppm) and were obtained with  $^1\text{H}$  decoupling. Melting points were obtained on a Mel-Temp capillary melting point apparatus and are uncorrected. Gas chromatographic analyses were performed on Hewlett-Packard 6890 gas chromatography instrument with a FID detector using 25 m x 0.20 mm capillary column with cross-linked methyl siloxane as a stationary phase.

The yields in table 2 refer to isolated yields (average of two runs) of compounds estimated to be  $\geq 95\%$  pure as determined by  $^1\text{H}$  NMR and GC analysis and/or combustion analysis.

## **I. Experimental for the Borylation of Aryl Halides.**

### **General Procedure A: Pd-Catalyzed Borylation of Aryl Iodides and Bromides.**

An oven-dried resealable Schlenk tube possessing a Teflon screw valve was charged with  $\text{PdCl}_2(\text{CH}_3\text{CN})_2$  (0.25%-2.0%) and SPhos (1.0-8.0%). The Schlenk tube was capped with a rubber septum and then evacuated and backfilled with argon (this sequence was carried out a total of two times). 1,4-Dioxane (0.30 mL) was added via syringe, through the septum, followed by the addition of the aryl halide (0.50 mmol),  $\text{NEt}_3$  (0.209 mL, 152 mg, 1.50 mmol) and pinacol borane (0.109 mL, 96.1 mg, 0.75 mmol) in a like manner (aryl halides that were solids were added with the other solid reagents). The septum was then replaced with a Teflon screw valve and the Schlenk tube was sealed. The reaction mixture was heated to 110 °C (*These reactions are conducted in a sealed tube at a temperature higher than the boiling point of 1,4-dioxane and  $\text{NEt}_3$ . For larger scale reactions, the appropriate safety precautions should be undertaken including the use of a blast shield*) until the aryl halide had been completely consumed as determined by gas chromatography and the reaction mixture was then allowed to cool to room temperature. The reaction solution was filtered through a thin pad of celite (eluting with ethyl acetate) and the eluent was concentrated under reduced pressure. The crude material so obtained was purified via flash chromatography on silica gel.

### **General Procedure B: Pd-Catalyzed Borylation of Aryl Chlorides.**

An oven-dried resealable Schlenk tube possessing a Teflon screw valve was charged with PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (3.0-4.0%) and SPhos (12.0-16.0%). The Schlenk tube was capped with a rubber septum and then evacuated and backfilled with argon (this sequence was carried out a total of two times). NEt<sub>3</sub> (0.500 mL) was added via syringe, through the septum, followed by the addition of the aryl chloride (0.50 mmol) and pinacol borane (0.109 mL, 96.1 mg, 0.75 mmol) in a like manner (aryl halides that were solids were added with the other solid reagents). The septum was then replaced with a Teflon screw valve and the Schlenk tube was sealed. The reaction mixture was heated to 110 °C (*These reactions are conducted in a sealed tube at a temperature higher than the boiling point of 1,4-dioxane and NEt<sub>3</sub>. For larger scale reactions, the appropriate safety precautions should be undertaken (i.e. the use of a blast shield)*) until the aryl halide had been completely consumed as determined by gas chromatography and was then allowed to cool to room temperature. The reaction solution was filtered through a thin pad of celite (eluting with ethyl acetate) and the eluent was concentrated under reduced pressure. The crude material so obtained was purified via flash chromatography on silica gel.

**2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 1).<sup>1</sup>**

Following general procedure A, a mixture of 4-iodoanisole (127 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (1.3 mg, 0.0050 mmol) and SPhos (8.2 mg, 0.020 mmol) was heated in 1,4-dioxane for 30 min. Flash column chromatography (5% EtOAc/Hexanes) yielded the title compound in 110 mg (94% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.75 (d, J = 9 Hz, 2H), 6.90 (d, J = 9 Hz, 2H), 3.83 (s, 3H), 1.34 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 162.1, 136.4, 113.2, 102.7, 83.4, 55.0, 24.8. <sup>1</sup>H NMR spectrum included.

**2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 2).<sup>1</sup>**

Following general procedure A on a larger scale, a mixture of 4-iodoanisole (234 mg, 1.00 mmol), pinacol borane (0.218 mL, 192 mg, 1.50 mmol), NEt<sub>3</sub> (0.418 mL, 354 mg, 3.00 mmol), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (0.26 mg, 0.0010 mmol) and SPhos (1.6 mg, 0.0040 mmol) was heated in 1,4-dioxane for 30 min. Flash column chromatography (5% EtOAc/Hexanes) yielded the title compound in 212 mg (91 % yield) as a colorless oil.

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<sup>1</sup> Zhu, W.; Ma, D. *Org. Lett.* **2006**, 8, 261.

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.75 (d, J = 9 Hz, 2H), 6.90 (d, J = 9 Hz, 2H), 3.83 (s, 3H), 1.34 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 162.1, 136.4, 113.2, 102.7, 83.4, 55.0, 24.8. <sup>1</sup>H NMR spectrum included.

**2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 3).<sup>1</sup>**

Following general procedure A, a mixture of 4-bromoanisole (62.5 μL, 93.5 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (1.3 mg, 0.0050 mmol) and SPhos (8.2 mg, 0.020 mmol) was heated in 1,4-dioxane for 1 h. Flash column chromatography (5% EtOAc/Hexanes) yielded the title compound in 113 mg (97% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.75 (d, J = 9 Hz, 2H), 6.90 (d, J = 9 Hz, 2H), 3.83 (s, 3H), 1.34 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 162.1, 136.4, 113.2, 102.7, 83.4, 55.0, 24.8. <sup>1</sup>H NMR spectrum included.

**2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 4).<sup>1</sup>**

Following general procedure B, a mixture of 4-chloroanisole (61.2 μL, 71.3 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub> (0.500 mL), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (3.9 mg, 0.015 mmol) and SPhos (24.6 mg, 0.060 mmol) was heated for 24 h. Flash column chromatography (5% EtOAc/Hexanes) yielded the title compound in 112 mg (96% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.75 (d, J = 9 Hz, 2H), 6.90 (d, J = 9 Hz, 2H), 3.83 (s, 3H), 1.34 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 162.1, 136.4, 113.2, 102.7, 83.4, 55.0, 24.8. <sup>1</sup>H NMR spectrum included.

***N,N*-dimethyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)aniline (Table 1, Entry 5).<sup>2</sup>**

Following general procedure A, a mixture of 4-bromo-*N,N*-dimethylaniline (100.0 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (1.3 mg, 0.0050 mmol) and SPhos (8.2 mg, 0.020 mmol) was heated in 1,4-dioxane for 3 h. Recrystallization (Hexanes) yielded the title compound in 105 mg (85% yield) as a white solid, mp 116-117 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.71 (d, J = 8 Hz, 2H), 6.70 (d, J = 8 Hz, 2H), 3.00 (s, 6H), 1.34 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 152.5, 136.1, 111.2, 83.1, 40.1, 24.8 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

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<sup>2</sup> Broutin, P.-E.; Cerna, I.; Campaniello, M.; Leroux, F.; Colobert, F. *Org. Lett.* **2004**, *6*, 4419.

**2-(4-butylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 6).<sup>3</sup>**

Following general procedure A, a mixture of 4-bromo-*n*-butylbenzene (106.5 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (1.3 mg, 0.0050 mmol) and SPhos (8.2 mg, 0.020 mmol) was heated in 1,4-dioxane for 4 h. Flash column chromatography (2.5% EtOAc/Hexanes) yielded the title compound in 109 mg (84% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.75 (d, J = 8 Hz, 2H), 7.22 (d, J = 8 Hz, 2H), 2.64 (t, J = 8 Hz, 2H), 1.62 (p, J = 8 Hz, 2H), 1.37 (sex, J = 8 Hz, 2H), 1.36 (s, 12H), 0.94 (t, J = 8 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 146.3, 134.8, 127.9, 83.5, 35.8, 33.5, 24.8, 22.3, 13.9 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**2-(4-butylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 7).<sup>3</sup>**

Following general procedure B, a mixture of 4-*n*-butylchlorobenzene (82.0 μL, 84.4 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub> (0.50 mL), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (3.9 mg, 0.015 mmol) and SPhos (24.6 mg, 0.060 mmol) was heated in 1,4-dioxane for 24 h. Flash column chromatography (2.5% EtOAc/Hexanes) yielded the title compound in 81 mg (62% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.75 (d, J = 8 Hz, 2H), 7.22 (d, J = 8 Hz, 2H), 2.64 (t, J = 8 Hz, 2H), 1.62 (p, J = 8 Hz, 2H), 1.37 (sex, J = 8 Hz, 2H), 1.36 (s, 12H), 0.94 (t, J = 8 Hz, 3H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 146.3, 134.8, 127.9, 83.5, 35.8, 33.5, 24.8, 22.3, 13.9 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**phenyl(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)methanone (Table 1, Entry 8).<sup>4</sup>**

Following general procedure A, a mixture of 4-bromobenzophenone (130 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (1.3 mg, 0.0050 mmol) and SPhos (8.2 mg, 0.020 mmol) was heated in 1,4-dioxane for 5 h. Flash column chromatography (10% EtOAc/Hexanes) yielded the title compound in 108 mg (70% yield) as a yellow solid, mp 96-97 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.91 (d, J = 8 Hz, 2H), 7.79 (dd, J = 1,8 Hz, 2H), 7.76 (d, J = 8 Hz, 2H), 7.59 (dt, J = 1,8 Hz, 1H), 7.48 (t, J = 8 Hz, 2H), 1.37 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 196.9, 139.7, 137.4, 134.5, 132.5, 130.1, 129.0, 128.3, 84.2, 24.9 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

<sup>3</sup> Laza, C.; Duñach, E. *Adv. Synth. Catal.* **2003**, *345*, 580.

<sup>4</sup> Fürstner, A.; Seidel, G. *Org. Lett.* **2002**, *4*, 541.

**3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzotrile (Table 1, Entry 9).<sup>5</sup>**

Following general procedure A, a mixture of 3-bromobenzotrile (91 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (1.3 mg, 0.0050 mmol) and SPhos (8.2 mg, 0.020 mmol) was heated in 1,4-dioxane for 3 h. Flash column chromatography (5% EtOAc/Hexanes) yielded the title compound in 65 mg (57% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 8.08 (s, 1H), 7.99 (d, J = 7 Hz, 1H), 7.71 (d, J = 7 Hz, 1H), 7.46 (t, J = 7 Hz, 1H), 1.34 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 138.7, 138.4, 134.4, 128.4, 118.8, 112.0, 84.4, 24.8 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**2-(2-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 10).<sup>2</sup>**

Following general procedure A, a mixture of 2-bromoanisole (62.3 μL, 93.5 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (2.6 mg, 0.010 mmol) and SPhos (16.4 mg, 0.040 mmol) was heated in 1,4-dioxane for 4 h. Flash column chromatography (5% EtOAc/Hexanes) yielded the title compound in 104 mg (89% yield) as a white solid, mp 77-78 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.67 (dd, J = 7,2 Hz, 1H), 7.39 (dt, J = 8,2 Hz, 1H), 6.94 (dt, J = 7,2 Hz, 1H), 6.85 (d, J = 8 Hz, 1H), 3.83 (s, 3H), 1.35 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 164.8, 137.4, 133.2, 120.9, 111.1, 84.1, 56.5, 25.5 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**2-(2-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 11).<sup>2</sup>**

Following general procedure B, a mixture of 2-chloroanisole (62.3 μL, 93.5 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub> (0.50 mL), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (5.2 mg, 0.020 mmol) and SPhos (32.8 mg, 0.080 mmol) was heated for 24 h. Flash column chromatography (5.0% EtOAc/Hexanes) yielded the title compound in 60 mg (51% yield) as a white solid, mp 76-77 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.67 (dd, J = 7,2 Hz, 1H), 7.39 (dt, J = 8,2 Hz, 1H), 6.94 (dt, J = 7,2 Hz, 1H), 6.85 (d, J = 8 Hz, 1H), 3.83 (s, 3H), 1.35 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 164.8, 137.4, 133.2, 120.9, 111.1, 84.1, 56.5, 25.5 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**2-mesityl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 12).<sup>6</sup>**

Following general procedure A, a mixture of 2-bromomesitylene (76.5 μL, 99.5 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (2.6 mg, 0.010 mmol) and SPhos (16.4 mg, 0.040

<sup>5</sup> Zhu, L.; Duquette, J.; Zhang, M. *J. Org. Chem.* **2003**, *68*, 3729.

<sup>6</sup> Ishiyama, T.; Murata, M.; Miyaura, N. *J. Org. Chem.* **1995**, *60*, 7508.

mmol) was heated in 1,4-dioxane for 4 h. Flash column chromatography (2.5% EtOAc/Hexanes) yielded the title compound in 111 mg (90% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 6.82 (s, 2H), 2.42 (s, 6H), 2.29 (s, 3H), 1.41 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 142.0, 138.8, 127.4, 83.3, 24.9, 22.1, 21.2 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**(E)-4,4,5,5-tetramethyl-2-styryl-1,3,2-dioxaborolane (Table 1, Entry 13).**<sup>7</sup> Following general procedure A, a mixture of β-bromostyrene (64.1 μL, 91.5 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (2.6 mg, 0.010 mmol) and SPhos (16.4 mg, 0.040 mmol) was heated in 1,4-dioxane for 4 h. Flash column chromatography (2.5% EtOAc/Hexanes) yielded the title compound in 79 mg (69% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.49 (d, J = 8 Hz, 2H), 7.41 (d, J = 18 Hz, 1H), 7.29-7.36 (m, 3H), 6.18 (d, J = 18 Hz, 1H), 1.33 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 149.5, 137.4, 128.9, 128.5, 127.0, 126.5, 83.3, 24.8. <sup>1</sup>H NMR spectrum included.

**1-acetyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (Table 1, Entry 14).** Following general procedure A, a mixture of *N*-acetyl-5-bromoindole (119 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (2.6 mg, 0.010 mmol) and SPhos (16.4 mg, 0.040 mmol) was heated in 1,4-dioxane for 4 h. Flash column chromatography (15% EtOAc/Hexanes) yielded the title compound in 138 mg (97% yield) as a yellow oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 8.41 (d, J = 7 Hz, 1H), 8.06 (s, 1H), 7.80 (d, J = 7 Hz, 1H), 7.40 (d, J = 2 Hz, 1H), 6.63 (d, J = 2 Hz, 1H), 2.63 (s, 3H), 1.37 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 168.7, 137.4, 131.4, 129.8, 125.2, 115.8, 109.4, 83.7, 24.9, 24.1 (No C-B Signal). IR (neat, cm<sup>-1</sup>): 3149, 3109, 2978, 1712, 1610, 1539, 1471, 1430, 1352, 1231, 1145. Anal. Calcd. for C<sub>16</sub>H<sub>20</sub>BNO<sub>3</sub>: C, 67.39; H, 7.07. Found C, 67.15; H, 7.08. <sup>1</sup>H and <sup>13</sup>C NMR spectrum included.

**3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(triisopropylsilyl)-1H-pyrrole (Table 1, Entry 15).**<sup>8</sup> Following general procedure A, a mixture of 3-bromo-1-(triisopropyl-silanyl)-1H-pyrrole<sup>9</sup> (156 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub>, PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (2.6 mg, 0.010 mmol) and SPhos (16.4 mg, 0.040 mmol) was heated in

<sup>7</sup> Pereira, S.; Srebnik, M. *Organometallics* **1995**, *14*, 3127.

<sup>8</sup> Billingsley, K. L.; Buchwald, S. L. *J. Am. Chem. Soc.* **2007**, *129*, 3358-3366.

<sup>9</sup> Alzare, A.; Guzman, A.; Ruiz, A.; Velards, E.; Muchowski, J. *J. Org. Chem.* **1992**, *57*, 1653-1656.

1,4-dioxane with stirring for 4 h. Flash column chromatography (15% EtOAc/Hexanes) yielded the title compound in 119 mg (74% yield) as a light yellow solid, m.p. 59 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.24 (dd, J = 2,1 Hz, 1H), 6.81 (dd, J = 3,2 Hz, 1H) 6.63 (dd, J = 3,1 Hz, 1H), 7.00 (dd, J = 7,1 Hz, 1H), 1.46 (sept, J = 7 Hz, 3H), 1.33 (s, 12H), 1.09 (d, J = 7 Hz, 18H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 133.6, 124.9, 115.6, 110.0, 82.6, 24.8, 17.7, 11.6. <sup>1</sup>H NMR spectrum included.

***N,N*-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)aniline (Table 1, Entry 16).**<sup>2</sup> Following general procedure B, a mixture of 3-chloro-*N,N*-dimethylaniline (77.8 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub> (0.50 mL), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (3.9 mg, 0.015 mmol) and SPhos (24.6 mg, 0.060 mmol) was heated with stirring for 24 h. Flash column chromatography (10% EtOAc/Hexanes) yielded the title compound in 99 mg (80% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.27 (t, J = 8 Hz, 1H), 7.19-7.22 (m, 2H), 6.87 (dd, J = 8,3 Hz, 1H), 2.97 (s, 6H), 1.35 (s, 12 H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 150.1, 128.5, 123.2, 118.6, 115.8, 83.6, 40.8, 24.8 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**2-(2,5-dimethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 17).**<sup>10</sup> Following general procedure B, a mixture of 2-chloro-*p*-xylene (67.0 μL, 70.3 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub> (0.50 mL), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (3.9 mg, 0.015 mmol) and SPhos (24.6 mg, 0.060 mmol) was heated with stirring for 24 h. Flash column chromatography (2.5% EtOAc/Hexanes) yielded the title compound in 101 mg (87% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.60 (s, 1H), 7.15 (d, J = 8 Hz, 1H), 7.08 (d, J = 8 Hz, 1H), 2.52 (s, 3H), 2.32 (s, 3H), 1.36 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 141.7, 136.3, 133.9, 131.5, 129.8, 83.3, 24.8, 21.7, 20.8 (No C-B Signal). <sup>1</sup>H NMR spectrum included.

**4,4,5,5-tetramethyl-2-(thiophen-3-yl)-1,3,2-dioxaborolane (Table 1, Entry 18).**<sup>11</sup> Following general procedure B, a mixture of 3-chlorothiophene (59.2 mg, 46.4 μL, 0.50 mmol), pinacol borane, NEt<sub>3</sub> (0.50 mL), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (3.9 mg, 0.015 mmol) and SPhos (24.6 mg, 0.060 mmol) was heated with stirring for 24 h. Flash column

<sup>10</sup> Thompson, A. L. S.; Kabalka, G. W.; Akula, M. R.; Huffman, J. W. *Synthesis* **2005**, *4*, 547.

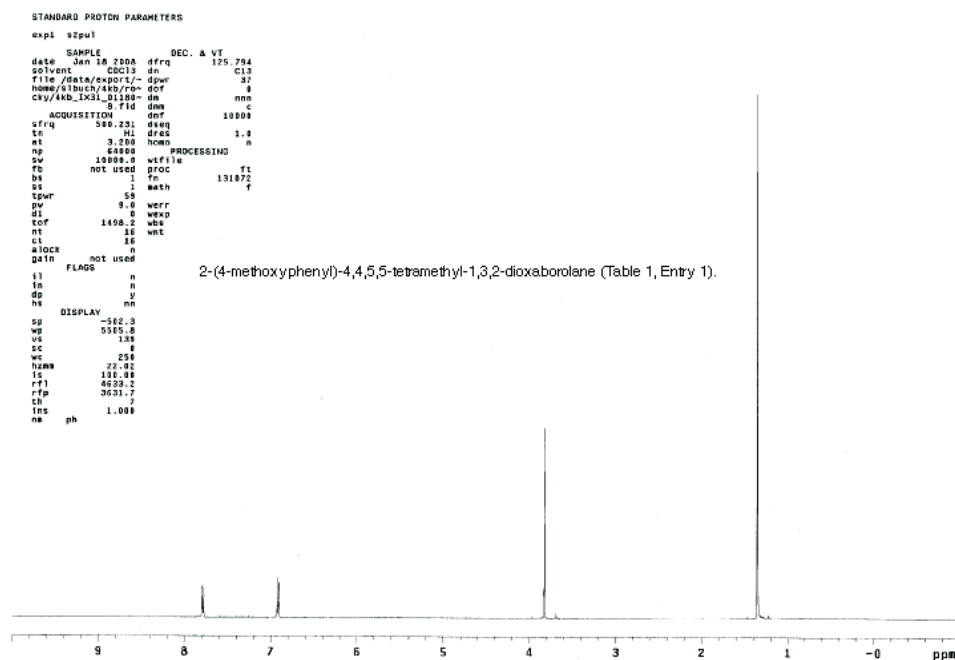
<sup>11</sup> Koolmeister, T.; Södergren, M.; Scobie, M. *Tetrahedron Lett.* **2002**, *43*, 5965.



chromatography (2.5% EtOAc/Hexanes) yielded the title compound in 62 mg (59% yield) as brown solid, mp 55-56 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 7.92 (dt, J = 1,3 Hz, 1H), 7.41 (dt, J = 1,5 Hz, 1H), 7.34 (dt, J = 3,5 Hz, 1H), 1.33 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 131.2, 132., 126.1, 103.8, 84.4, 25.6. <sup>1</sup>H NMR spectrum included.

**2-cyclopentenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 19).<sup>12</sup>**

Following general procedure B, a mixture of 1-chlorocyclopentene (49.6 μL, 51.3 mg, 0.50 mmol), pinacol borane, NEt<sub>3</sub> (0.50 mL), PdCl<sub>2</sub>(CH<sub>3</sub>CN)<sub>2</sub> (3.9 mg, 0.015 mmol) and SPhos (24.6 mg, 0.060 mmol) was heated with stirring for 24 h. Flash column chromatography (2.5% EtOAc/Hexanes) yielded the title compound in 71 mg (73% yield) as a colorless oil. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ: 6.54 (t, J = 2 Hz, 1H), 2.37-2.41 (m, 4H), 1.83 (pent, J = 7 Hz, 2H), 1.28 (s, 12H). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ: 147.6, 83.0, 34.7, 34.5, 24.8, 23.9. <sup>1</sup>H NMR spectrum included.



<sup>12</sup> Takagi, J.; Takahashi, K.; Ishiyama, T.; Miyaura, N. *J. Am. Chem. Soc.* **2002**, *124*, 8001.

## STANDARD PROTON PARAMETERS

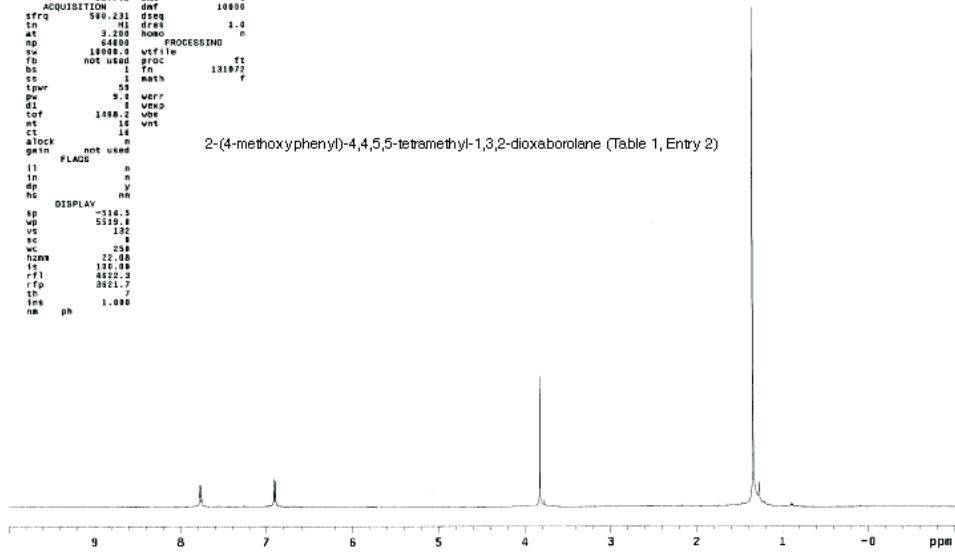
expt szpul

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solvent CDCl3  dm  C13
file /data/export/- spwr 37
home/sibuch/482/rp- dof 8
cxy/4kb_EXB7C_8303- dm  nnn
          SE. F1d  dm  c
ACQUISITION    der  10000
sfrq 500.231  dseq
ln 41  dres  1.0
at 3.200  homo  n
np 64000  PROCESSING
sw 18000.0  vtfile  ft
fb not used  proc  131872
bs 1  fn
cs 1  math  f
tpr 53
pw 9.3  verr
d1 8  wesp
tof 1498.2  vbs
nt 16  vnt
ct 16
clock n
data not used
          FLAGS
ll n
ln n
sp y
hg nn
          DISPLAY
sp -514.5
wp 5519.8
vc 102
ec 8
wc 258
hzms 72.08
ls 105.08
rfi 4622.3
rfp 3621.7
th
ims ph 1.090

```

2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 2)



## STANDARD PROTON PARAMETERS

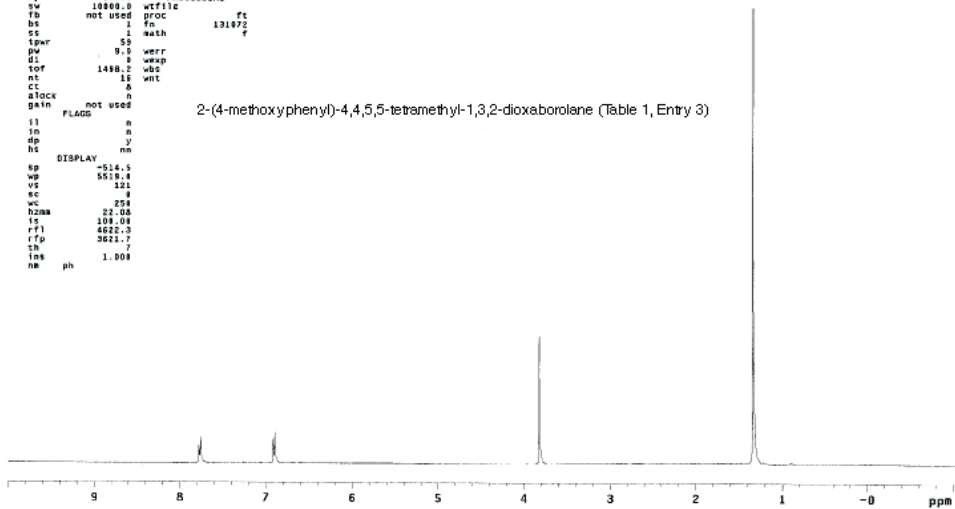
expt szpul

```

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solvent CDCl3  dm  C13
file /data/export/- spwr 37
home/sibuch/482/rp- dof 8
cxy/4kb_EXB_81080- dm  nnn
          SE. F1d  dm  c
ACQUISITION    der  10000
sfrq 500.231  dseq
ln 41  dres  1.0
at 3.200  homo  n
np 64000  PROCESSING
sw 18000.0  vtfile  ft
fb not used  proc  131872
bs 1  fn
cs 1  math  f
tpr 53
pw 9.3  verr
d1 8  wesp
tof 1498.2  vbs
nt 16  vnt
ct 16
clock n
data not used
          FLAGS
ll n
ln n
sp y
hg nn
          DISPLAY
sp -514.5
wp 5519.8
vc 102
ec 8
wc 258
hzms 72.08
ls 105.08
rfi 4622.3
rfp 3621.7
th
ims ph 1.009

```

2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 3)



## STANDARD PROTON PARAMETERS

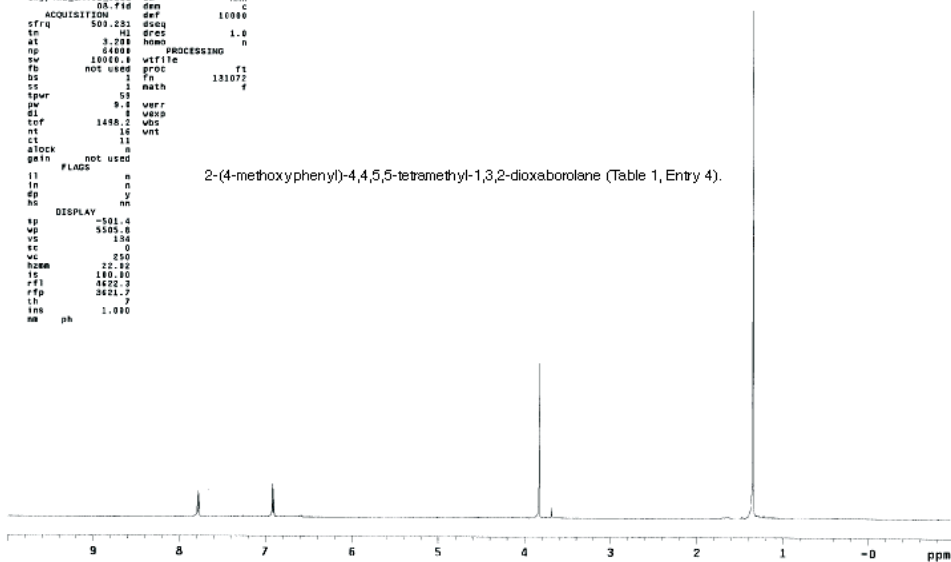
expt szpu1

```

SAMPLE          DEC. & VT
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file /data/export/~ dpwr 37
home/sibach/4kb/ra- dof 0
cxy/4kb_2k488_1118- dm  nnn
                        def  c
ACQUISITION    05.118  dm  10000
cfrq 500.251  dseq  c
in  H1  drcz  1.0
at  3.288  homo  n
np  64000  PROCESSING n
sw  10000.0 wfile  ft
fb  not used  proc  131072
cs  1  bath  f
spwr 53
pw  9.4  verr  c
el  0  werr  c
tof  1498.2  wbs  c
nt  16  wnt  c
ct  11
clock n
gain  not used
FLAGS n
in  n
sp  y
ns  nn
DISPLAY nn
sp  -501.4
wp  5585.8
vc  130
sc  0
uc  250
hzmm 22.02
rs  180.00
rff  4672.3
rfp  3621.7
lh  c
lms 1.000
ms  ph

```

2-(4-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 4).



## STANDARD PROTON PARAMETERS

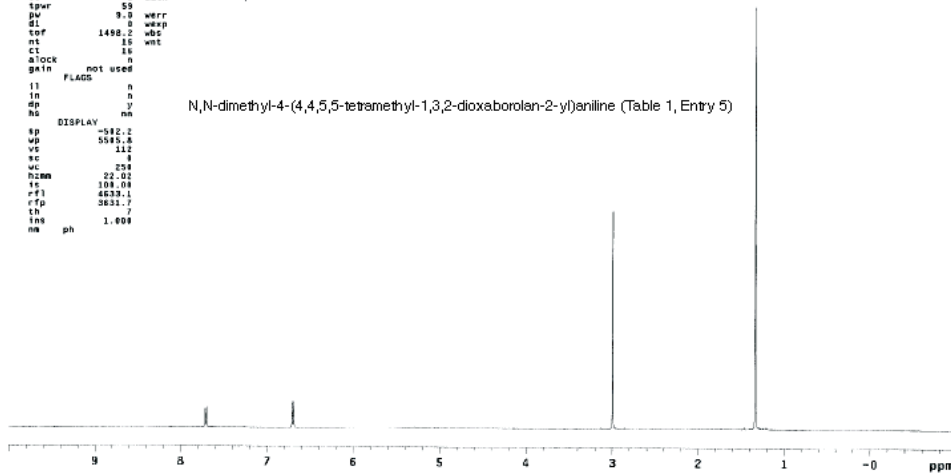
expt szpu1

```

SAMPLE          DEC. & VT
date Jan 22 2008 dfrq 125.784
solvent CDCl3  dm  C13
file /data/export/~ dpwr 37
home/sibach/4kb/ra- dof 0
cxy/4kb_2k51_01159- dm  nnn
                        def  c
ACQUISITION    0.115  dm  10000
cfrq 500.251  dseq  c
in  H1  drcz  1.0
at  3.200  homo  n
np  64000  PROCESSING n
sw  10000.0 wfile  ft
fb  not used  proc  131072
cs  1  bath  f
spwr 53
pw  9.0  verr  c
el  0  werr  c
tof  1498.2  wbs  c
nt  16  wnt  c
ct  11
clock n
gain  not used
FLAGS n
in  n
sp  y
ns  nn
DISPLAY nn
sp  -502.2
wp  5585.8
vc  112
sc  0
uc  250
hzmm 22.02
rs  180.00
rff  4673.1
rfp  3631.7
lh  c
lms 1.000
ms  ph

```

N,N-dimethyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)aniline (Table 1, Entry 5)



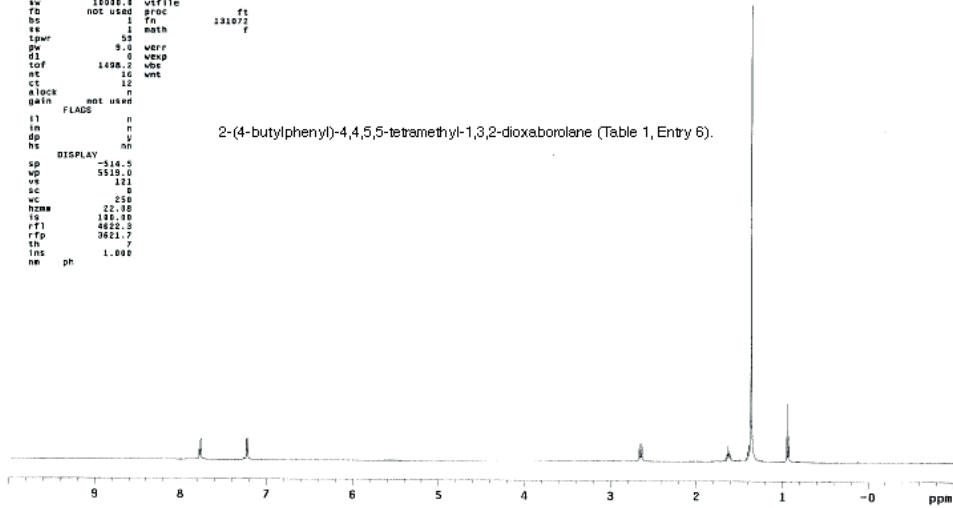
## STANDARD PROTON PARAMETERS

```

exp1 s1pu1
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date Jan 22 2008 dfrq 125.794
solvent CDCl3 dn C13
file /data/export/- dpwr 37
name/s1buck/4kb/r0- dot 0
ckv/4kb_2K56_01028- da nnn
c
ACQUISITION    dms c
sfrq 500.231 dseq def 10000
in H1 drec 1.0
at 3.200 homo n
sp 64000 PROCESSING
sw 10000.0 vrf1ic ft
fs not used ploc 131072
ss 1 math f
tpr 50
pv 9.0 verr
si 0 verr
tof 1498.2 wbs
nt 16 wnt
ct 12
a lock n
gain not used
FLAGS
il n
in n
sp y
ns DISPLAY nn
sp -514.5
wp 5529.0
vt 121
sc 0
wc 258
hzmm 22.98
ts 130.08
rfl 4622.3
rfd 3621.7
th
ims ph 1.000
nm

```

2-(4-butylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 6).



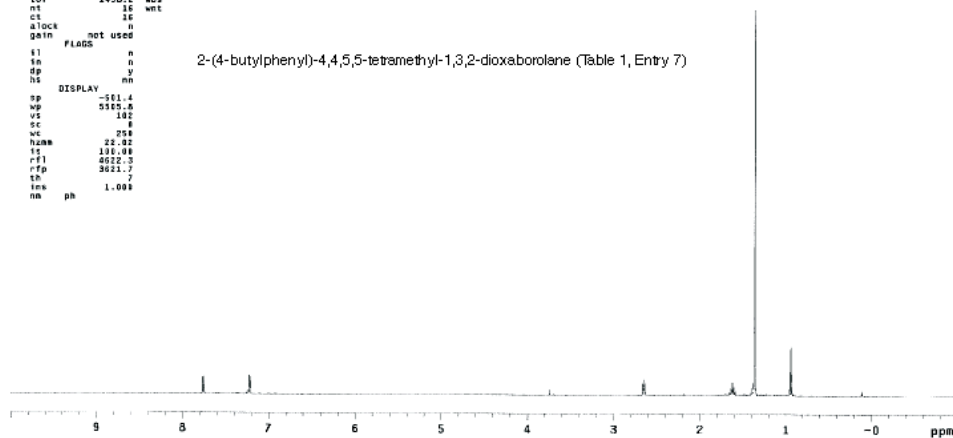
## STANDARD PROTON PARAMETERS

```

exp1 s2pu1
SAMPLE          DEC. & VT
date Jan 18 2008 dfrq 125.794
solvent CDCl3 dn C13
file /data/export/- dpwr 37
name/s1buck/4kb/r0- dot 0
ckv/4kb_2K45_01109- da nnn
c
ACQUISITION    dms c
sfrq 500.231 dseq def 10000
in H1 drec 1.0
at 3.200 homo n
sp 64000 PROCESSING
sw 10000.0 vrf1ic ft
fs not used ploc 131072
ss 1 math f
tpr 50
pv 9.0 verr
si 0 verr
tof 1498.2 wbs
nt 16 wnt
ct 12
a lock n
gain not used
FLAGS
il n
in n
sp y
ns DISPLAY nn
sp -501.4
wp 5529.0
vt 102
sc 0
wc 258
hzmm 22.02
ts 130.08
rfl 4622.3
rfd 3621.7
th
ims ph 1.000
nm

```

2-(4-butylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 7)

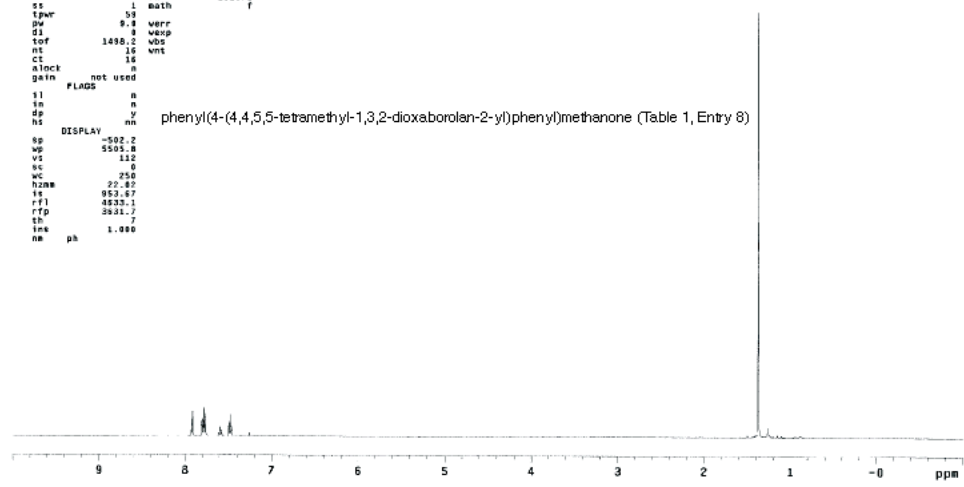


STANDARD PROTON PARAMETERS

```

exp1 szpu1
SAMPLE DEC. & VT
date Jan 22 2005 dfrq 125.794
solvent CDCl3 dn CL3
file /data/export/- dpwr 37
home/sibuch/4kb/no- dot 8
chx/4k_5K5_0222- de nnn
0.716 dm 10000 C
ACQUISITION det 131072
sfrq 500.231 dsdq 1.4
in H3 dres n
at 3.201 homo n
sp 64000 drc PROCESSING
sw 10000.0 vefile ft
fb not used proc 131072
ss 1 fn
ss 1 math f
tpr 59
pw 9.8 verr
q1 9 vesp
tof 1498.2 vbs
et 16 vnt
alock 16 n
data not used
FLAGS n
in n
sp n
ns nn
DISPLAY
sp -502.2
wp 5505.8
vs 112
sc 0
vc 250
hzmm 27.22
is 180.10
rf1 4622.3
rfp 3621.7
tn 7
ins ph 1.000
nm
  
```

phenyl(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)methanone (Table 1, Entry 8)

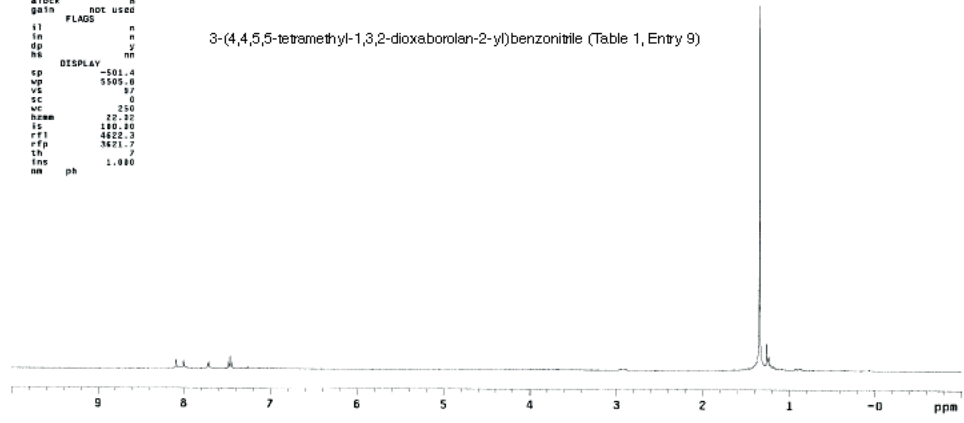


STANDARD PROTON PARAMETERS

```

exp1 szpu1
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solvent CDCl3 dn CL3
file /data/export/- dpwr 37
home/sibuch/4kb/no- dot 8
chx/4k_5K5_0222- de nnn
0.716 dm 10000 C
ACQUISITION det 131072
sfrq 500.231 dsdq 1.4
in H3 dres n
at 3.201 homo n
sp 64000 drc PROCESSING
sw 10000.0 vefile ft
fb not used proc 131072
ss 1 fn
ss 1 math f
tpr 59
pw 9.8 verr
q1 9 vesp
tof 1498.2 vbs
et 16 vnt
alock 16 n
data not used
FLAGS n
in n
sp n
ns nn
DISPLAY
sp -501.4
wp 5505.8
vs 97
sc 0
vc 250
hzmm 27.22
is 180.10
rf1 4622.3
rfp 3621.7
tn 7
ins ph 1.000
nm
  
```

3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzoinitrile (Table 1, Entry 9)



## STANDARD PROTON PARAMETERS

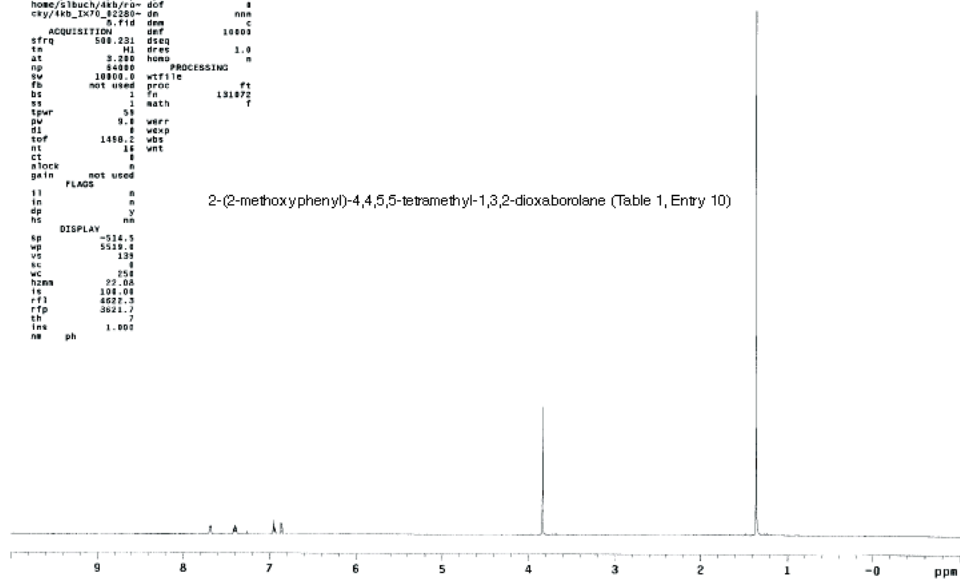
expt1 s2p1

```

SAMPLE          DEC. A VT
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solvent  CDCl3  dn      C13
file /data/export/~dpwr  37
home/sibuch/443/ro- dot  8
cxy/4h_2x48_83030- dn  nnn
          5. f1d  dmz  18003
ACQUISITION    dnt
f1rq  509.231  dseq  1.0
in    H1  dfrz  1.0
at    3.230  homo  n
np    24008  PROCESSING
sw    10950.0  wtf1ic
fb    not used  proc  ft
bs    1  fa  131872
ss    1  math  f
tspur  59
pw    9.8  verr
sl    8  vbrp
topf  1489.2  vbs
ct    16  vnt
atock  n
gwin  not used
FLAGS  n
in    n
ex    y
ns    nn
DISPLAY
sp    -514.5
wp    5519.0
vr    132
vc    0
wc    250
hzmm  32.88
fs    180.80
rf1   4822.3
rfp   3921.7
th    1.000
ns    ph  1.000

```

2-(2-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 10)



## STANDARD PROTON PARAMETERS

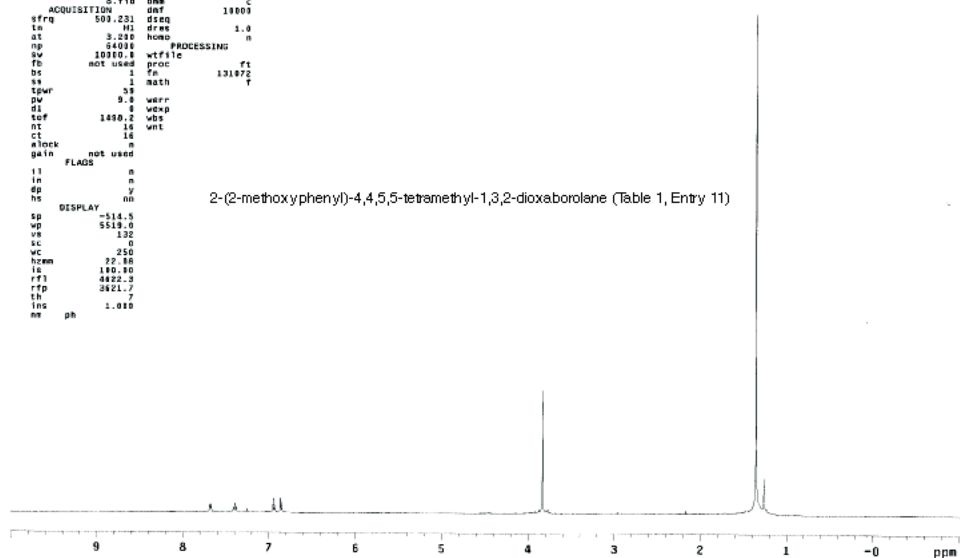
expt1 s2p1

```

SAMPLE          DEC. A VT
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solvent  CDCl3  dn      C13
file /data/export/~dpwr  37
home/sibuch/443/ro- dot  8
cxy/4h_2x48_83030- dn  nnn
          5. f1d  dmz  18003
ACQUISITION    dnt
f1rq  509.231  dseq  1.0
in    H1  dfrz  1.0
at    3.230  homo  n
np    24008  PROCESSING
sw    10950.0  wtf1ic
fb    not used  proc  ft
bs    1  fa  131872
ss    1  math  f
tspur  59
pw    9.8  verr
sl    8  vbrp
topf  1489.2  vbs
ct    16  vnt
atock  n
gwin  not used
FLAGS  n
in    n
ex    y
ns    nn
DISPLAY
sp    -514.5
wp    5519.0
vr    132
vc    0
wc    250
hzmm  32.88
fs    180.80
rf1   4822.3
rfp   3921.7
th    1.000
ns    ph  1.000

```

2-(2-methoxyphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 11)

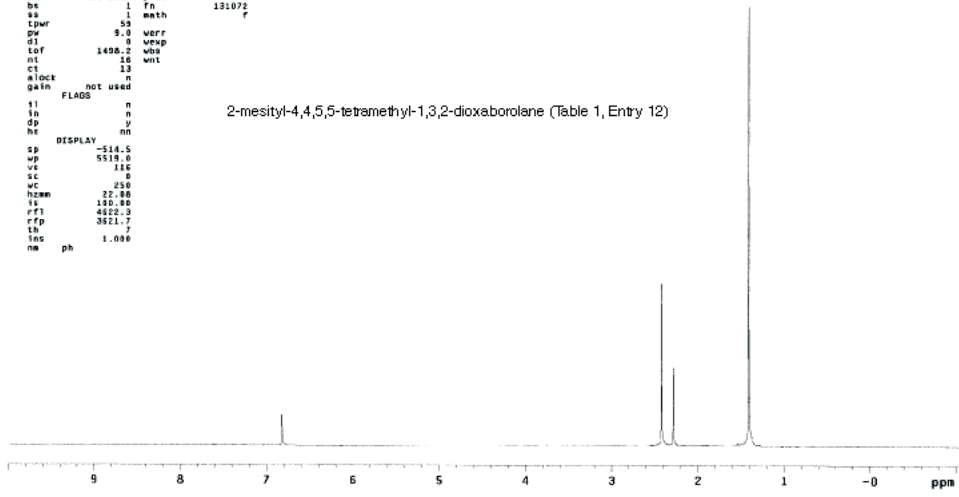


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STANDARD PROTON PARAMETERS
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solvent CDCl3 dn C13
file /data/export/- dpwr 37
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cky/4kb_2x6_01051- dw nnn
0. f10 dm c
ACQUISITION det 18000
xfrq 500.231 dseq
in H1 drcd 1.0
at 3.208 homo n
np 64008 PROCESSING
sw 10000.0 wtitle
fn not used proc fc
bs 1 fn 131072
ss 1 math f
tpr 53
pw 9.0 verr
sl 0 wesp
tof 1498.2 vbs
nt 16 vnt
ct 13
clock n
gain not used
FLAGS
il n
in n
sp y
hs nn
DISPLAY
sp -518.5
wp 5519.0
vs 116
sc 0
uc 250
hzmm 22.02
ts 180.00
rf1 4622.3
rfp 3621.7
th 7
ins ph 1.000
nm

```

2-mesityl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 12)

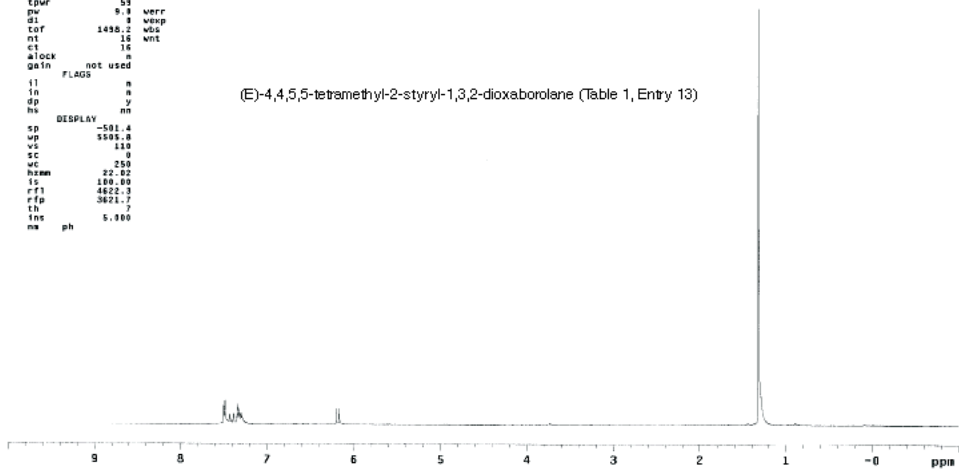


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STANDARD PROTON PARAMETERS
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solvent CDCl3 dn C13
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home/sibuch/465/ro- dof 0
cky/4kb_2x6_01051- dw nnn
0. f10 dm c
ACQUISITION det 18000
xfrq 500.231 dseq
in H1 drcd 1.0
at 3.208 homo n
np 64008 PROCESSING
sw 10000.0 wtitle
fn not used proc fc
bs 1 fn 131072
ss 1 math f
tpr 53
pw 9.0 verr
sl 0 wesp
tof 1498.2 vbs
nt 16 vnt
ct 13
clock n
gain not used
FLAGS
il n
in n
sp y
hs nn
DISPLAY
sp -501.4
wp 5505.8
vs 110
sc 0
uc 250
hzmm 22.02
ts 180.00
rf1 4622.3
rfp 3621.7
th 7
ins ph 5.900
nm

```

(E)-4,4,5,5-tetramethyl-2-styryl-1,3,2-dioxaborolane (Table 1, Entry 13)

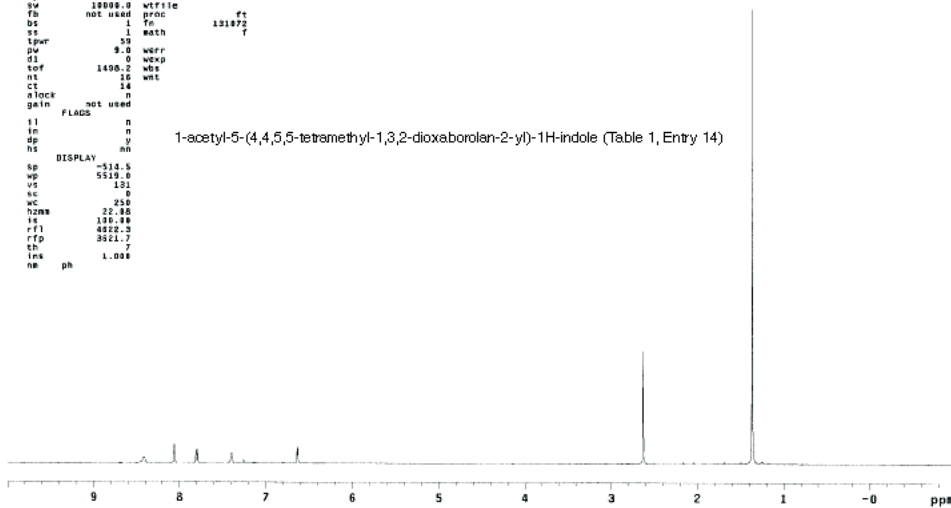


STANDARD PROTON PARAMETERS

```

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solvent CDCl3 dn C13
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home/s1bch/4kb/ro- got 8
exp/4kb_incl_0129- de nmh
ACQUISITION dnm 10000
sfrq 500.231 dseq 1.0
tn 81 dres
at 3.200 homo n
np 64000 PROCESSING
sw 10000.0 wtf1le ft
fb not used proc 131872
bs 1 te
ss 1 math f
tqwr 50
pw 9.0 wefr
dl 0 wexp
tof 1406.2 wnt
nt 16 wnt
ct 16 wnt
alock n
gain not used
FLAGS
fl n
in n
dp y
hs nh
DISPLAY
sp -514.5
wp 5519.0
vs 101
sc 0
wc 250
hzmm 22.10
rs 131.88
rf1 4522.3
rfp 3521.7
th 7
ins ph 1.000
nb
  
```

1-acetyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (Table 1, Entry 14)

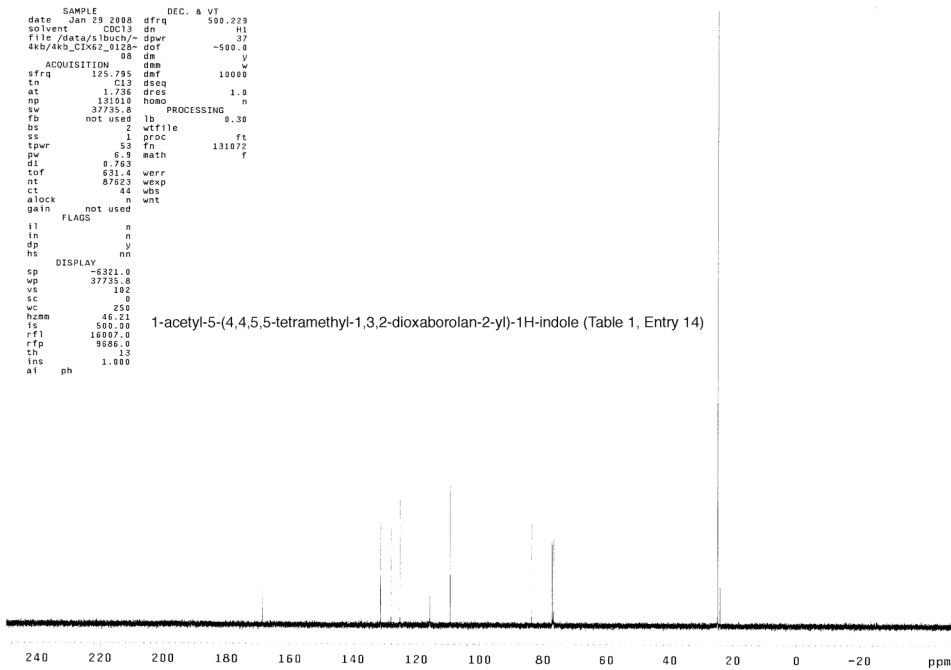


STANDARD CARBON PARAMETERS

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solvent CDCl3 dn H1
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4kb/4kb_incl_0129- got -500.0
ACQUISITION dnm 10000
sfrq 125.795 dmf 10000
tn 512 dseq
at 1.736 dres 1.0
np 131010 homo n
sw 37735.8 PROCESSING
fb not used lb 0.30
bs 1 wtf1le ft
ss 1 proc 131072
tqwr 53 fin
pw 6.3 math f
dl 0.763
tof 621.4 wefr
nt 87623 wexp
ct 44 wnt
alock n
gain not used
FLAGS
fl n
in n
dp y
hs nh
DISPLAY
sp -5321.0
wp 37735.8
vs 102
sc 0
wc 250
hzmm 46.21
rs 500.00
rf1 16807.0
rfp 9886.0
th 13
ins ph 1.000
al
  
```

1-acetyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (Table 1, Entry 14)





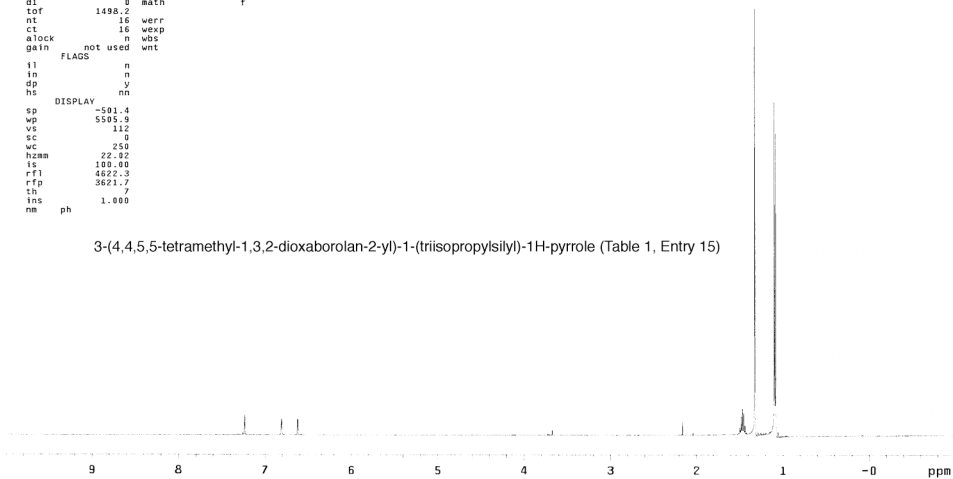
## STANDARD PROTON PARAMETERS

```

expl s2pu1
SAMPLE
date Mar 3 2008 dfrq 125.794
solvent CDCl3 dn C13
file /data/export/ dpar 37
ACQUISITION exp dof 0
sfrq 500.231 dm nnn
tn 101 dm c
at 3.200 def 10000
np 64000 dseq 1.0
sw 10000.0 dres
fb not used homo n
bs 1 PROCESSING
ss 1 wfile ft
tpr 50 proc
pw 9.0 fn 131872
dl 0 math f
tof 1498.2
nt 16 verr
ct 16 vesp
alock n vbs
gain not used vnt
FLAGS
il n
in n
dp y
hs nm
DISPLAY
sp -591.4
wp 5595.9
vs 112
sc 0
wc 250
hzmm 22.02
ls 100.00
rf1 4622.3
rfp 3621.7
tn 7
ins ph 1.000
nm

```

3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1-(trisopropylsilyl)-1H-pyrrole (Table 1, Entry 15)



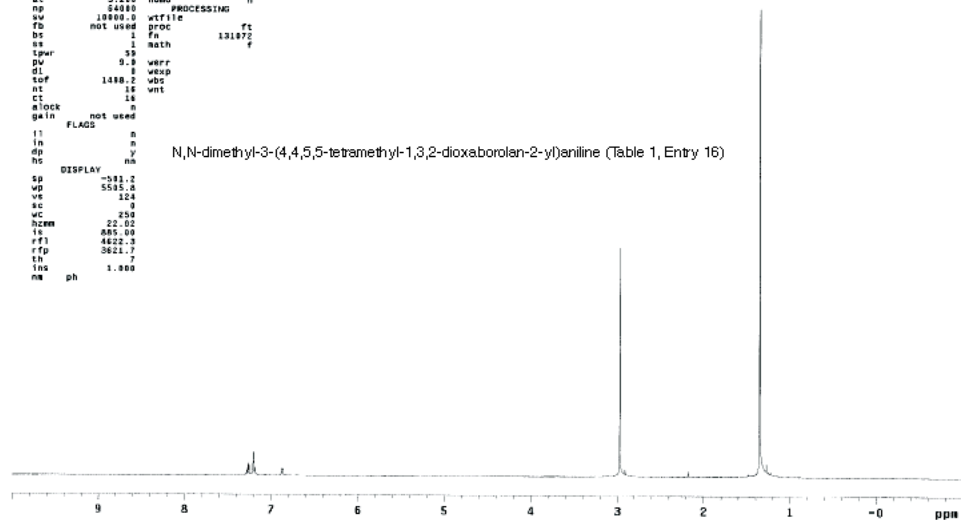
## STANDARD PROTON PARAMETERS

```

expl s2pu1
SAMPLE
date Mar 3 2008 dfrq 125.794
solvent CDCl3 dn C13
file /data/export/ dpar 37
home/Touch/462/10- dof 3
cty/150_1506_13830- dn nnn
ACQUISITION 8.fid dm c
sfrq 500.231 dseq 10000
tn 101 dm c
at 3.200 def 10000
np 64000 dseq 1.0
sw 10000.0 dres
fb not used homo n
bs 1 PROCESSING
ss 1 wfile ft
tpr 50 proc
pw 9.0 fn 131872
dl 0 math f
tof 1498.2
nt 16 verr
ct 16 vesp
alock n vbs
gain not used vnt
FLAGS
il n
in n
dp y
hs nm
DISPLAY
sp -581.2
wp 5535.8
vs 104
sc 0
wc 250
hzmm 22.02
ls 885.00
rf1 4622.3
rfp 3621.7
tn 7
ins ph 1.000
nm

```

N,N-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)aniline (Table 1, Entry 16)



## STANDARD PROTON PARAMETERS

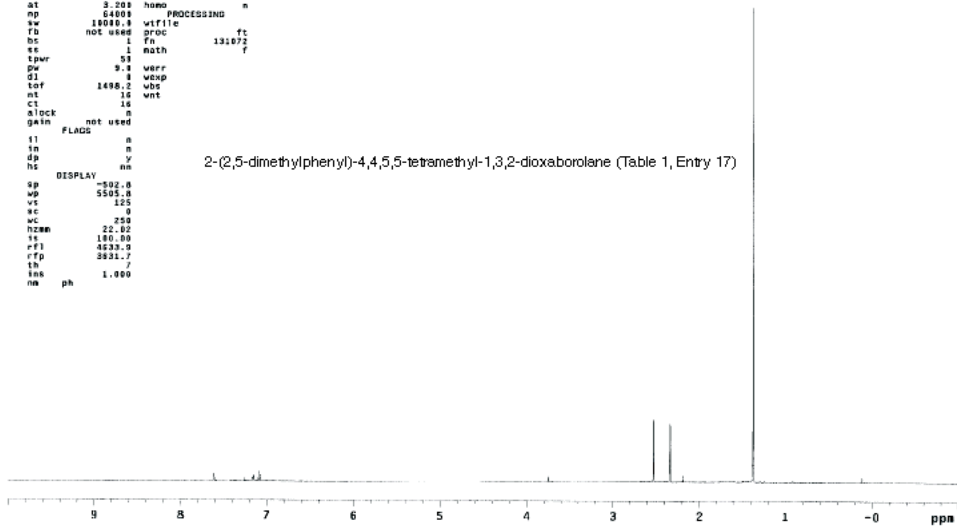
expt1 s2pu1

```

SAMPLE          DEC. & VT
date    Jan 18 2008  dffrq  125.794
solvent  CDCl3    da      C13
file    /data/export/- dpar   37
home    /touch/4k/ro-  dof   0
ckyy/4k_2K4_01181-  da      nnn
          0.fid  dms      C
ACQUISITION  0.fid  dmf      1008
          508.231 dseq
          101    drag      1.0
          3.200 homo      n
          64000 PROCESSING
          10000.0 wtf file
          not used proc      fc
          1    fo      131072
          1    math      f
          58
          9.4 verr
          8    wesp
          1498.2 vbs
          16    vnt
          16
          n
          not used
          FLAGS
          n
          y
          nn
          DISPLAY
          -502.8
          5503.8
          125
          0
          250
          22.00
          180.00
          4533.9
          3621.7
          10
          1.000
          ph

```

2-(2,5-dimethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 17)



## STANDARD PROTON PARAMETERS

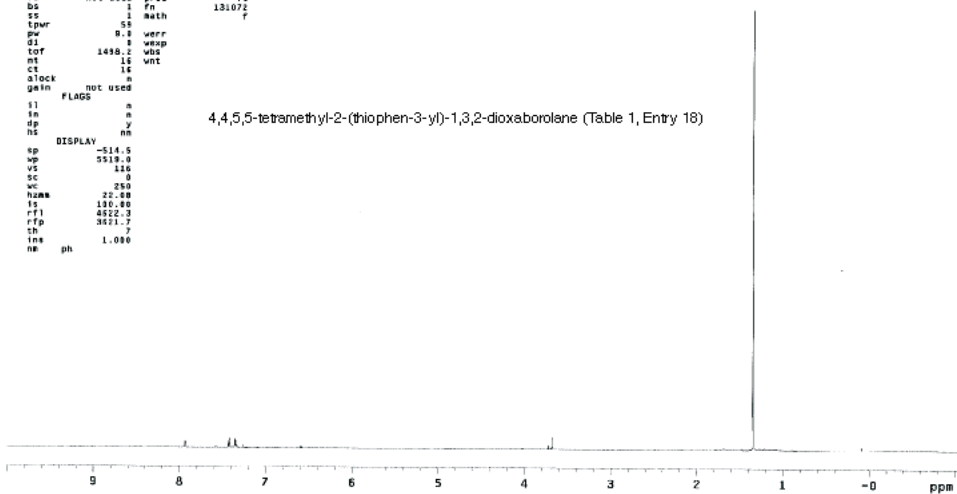
expt1 s2pu1

```

SAMPLE          DEC. & VT
date    Jan 18 2008  dffrq  125.794
solvent  CDCl3    da      C13
file    /data/export/- dpar   37
home    /touch/4k/ro-  dof   0
ckyy/4k_2K4_01181-  da      nnn
          0.fid  dms      C
ACQUISITION  0.fid  dmf      10080
          508.231 dseq
          101    drag      1.0
          3.200 homo      n
          64000 PROCESSING
          10000.0 wtf file
          not used proc      fc
          1    fo      131072
          1    math      f
          58
          8.8 verr
          8    wesp
          1498.2 vbs
          16    vnt
          16
          n
          not used
          FLAGS
          n
          y
          nn
          DISPLAY
          -514.5
          5519.0
          125
          0
          250
          22.00
          180.00
          4522.3
          3621.7
          10
          1.000
          ph

```

4,4,5,5-tetramethyl-2-(thiophen-3-yl)-1,3,2-dioxaborolane (Table 1, Entry 18)



## STANDARD PROTON PARAMETERS

exp1 s2pu1

SAMPLE		DEC. & VT	
date	Mar 6 2008	dfrq	125.872
solvent	CDCl3	dn	C13
file		dpuw	30
ACQUISITION			
sfrq	499.746	dm	0
tn	H1	dmm	nnn
at	3.001	daf	w
np	63059	dseq	10000
pw	13504.2	dres	1.0
fb	not used	homo	n
bs	1	DEC2	0
tpwr	53	dfrq2	0
pw	8.6	dn2	1
d1	2.008	dpuw2	0
tof	1519.5	dof2	0
nt	16	dac	n
ct	16	dmm2	c
atlock	n	daf3	200
gain	not used	dseq2	1.0
flags	n	homo2	n
il	n	DEC3	0
in	n	dfrq3	0
dp	y	dn3	1
hs	DISPLAY	dpuw3	0
sp	-502.2	dof3	n
wb	5493.6	dm3	c
vs	0	dms	200
sc	0	dms3	1.0
wc	1376.51	homo3	n
hzmm	21.97	dres3	1.0
is	1376.51	homo3	n
rf1	0	PROCESSING	
th	7	proc	ft
ins	1.000	fn	262144
al	cdc ph	math	f
		werr	
		wexp	
		wds	
		wnt	wft

2-cyclopentyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (Table 1, Entry 19)

