Supporting Information
for

Iridium/N-heterocyclic carbene-catalyzed C–H borylation of arenes by diisopropylaminoborane

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Experimental procedures, data for optimization studies and copies of 1H and 13C NMR spectra

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I. General information

$^1$H NMR and $^{13}$C NMR spectra were recorded on a JEOL ECS-400 spectrometer in CDCl$_3$ or C$_6$D$_6$ with tetrachloroethane as the internal standard. Data are reported as follows: chemical shift in ppm ($\delta$), multiplicity (s = singlet, d = doublet, t = triplet, and m = multiplet), coupling constant (Hz), and integration. Infrared spectra (IR) were obtained using a JASCO FT/IR-4200 spectrometer; absorptions are reported in reciprocal centimeters with the following relative intensities: s (strong), m (medium), or w (weak). Mass spectra and high resolution mass spectra (HRMS) were obtained on a JEOL JMS-700 spectrometer. Analytical gas chromatography (GC) was carried out on a Shimazu GC-2014 gas chromatograph, equipped with a flame ionization detector. Melting points were determined using a Yamato melting point apparatus. Column chromatography was performed with SiO$_2$ (silicycle SilicaFlash F60 (230–400 mesh)).

II. Materials

[Ir(OMe)(cod)$_2$] (TCI), ICy·HCl (TCI) and NaOt-Bu (TCI) were used as received. Methylcyclohexane was purified by distillation prior to use. N-methylindole (TCI), benzo[b]thiophene (TCI), 5-chloro3-methylbenzo[b]thiophene (TCI), 2,3-benzofuran (TCI), thiophene (TCI), 2-methylthiophene (TCI), 2-methoxythiophene (TCI), 2-methylfurran (TCI) and N-methylpyrrole (TCI) were obtained from commercial suppliers and used as received. All arenes (TCI) and naphthalene (Aldrich) were used as received. The other N-methylindoles used in this study were synthesized by the reaction of the corresponding indole with MeI according to the literature procedure.$^1$

III. Synthesis of starring material

Diisopropylaminoborane (1g). [CAS: 22092-92-8]

Diisopropylaminoborane was prepared as described in literatures.$^2$

To a stirred solution of diisopropylamine (28.2 mL, 200 mmol, 1.0 equiv) in THF (70 mL), H$_2$SO$_4$ (5.4 mL, 100 mmol, 0.5 equiv) were added at 0 °C. A white precipitate appeared immediately. After

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$^1$ Greulich, T. W.; Daniliuc, C. G.; Studer, A. Org. Lett. 2015, 17, 254.

$^2$ a) Marciasini, L.; Richy, N.; Vautier, M.; Pucheault, M. Chem. Commun. 2012, 448, 1553.

b) Marciasini, L.; Richy, N.; Vautier, M.; Pucheault, M. Adv. Synth. Catal. 2013, 6, 1083.
the mixture was stirred at 0 °C for 30 min, NaBH₄ (8.2 g, 220 mmol, 1.1 equiv) was carefully added. The mixture was allowed to warm to room temperature and stirred for 4 h. The crude mixture was concentrated under vacuum and the residue was taken with toluene (100 mL), washed with water (4 × 100 mL). The organic phase was dried using Na₂SO₄ and concentrated under reduced pressure to give an amine-borane complex as a colorless oil. The resulting amine-borane complex was then refluxed at 195 °C for 9 h, and the diisopropylaminoborane was distilled under N₂ to give 17.2 g (76% yield).

IV. Optimization studies

IV-I. Optimization studies for heteroarenes

The effect of the ligand was initially examined using 2 (0.50 mmol), 1g (1.0 mmol), [Ir(OMe)(cod)]₂ (0.050 mmol), ligand (0.10 mmol) and base (0.20 mmol) in methylcyclohexane (1.0 mL) at 140 °C, 15 h (Table S1). Under these conditions, ICy·HCl was found to be an optimal ligand with a borylated product 2-B being formed in 33% (Entry 17).

Table S1. Effect of ligands.

| Entry | Ligand   | Base     | NMR yield [%] | 2-Isomer/3-Isomer | Recovered 2 [%] |
|-------|----------|----------|---------------|--------------------|-----------------|
| 1     | dtbpy    | none     | trace         | -                  | 85              |
| 2     | dppe     | none     | 2             | 100/0              | 48              |
| 3     | dpff     | none     | 11            | 91/9               | 65              |
| 4     | Xantphos | none     | 18            | 56/44              | 59              |
| 5     | DPEPhos  | none     | 6             | 85/15              | 69              |
| 6     | PPh₃     | none     | 21            | 57/43              | 66              |
| 7     | PCy₃     | none     | 3             | >99/1              | 71              |
| 8     | P(OPh)₃ | none     | 11            | 45/55              | 82              |
| 9     | P(C₆F₅)₃ | none     | 19            | 21/79              | 64              |
| 10    | Cy-JohnPhos | none | 11            | 64/36              | 72              |
| 11    | JohnPhos | none     | 6             | 50/50              | 87              |
| 12    | DavePhos | none     | 15            | 67/33              | 71              |
| 13    | XPhos    | none     | 21            | 71/29              | 61              |
| 14    | SPhos    | none     | 15            | 67/33              | 69              |
| 15    | IrHCl    | NaO²Bu   | 3             | >99/1              | 72              |
| 16    | IMesHCl  | NaO²Bu   | 5             | >99/1              | 65              |
| 17    | ICyHCl   | NaO²Bu   | 33            | 88/12              | 53              |
| 18    | IBuHCl   | NaO²Bu   | 0             | -                  | 89              |

The effect of the temperature was then examined using 2 (0.50 mmol), 1g (1.0 mmol), [Ir(OMe)(cod)]₂ (0.050 mmol), ICy·HCl (0.10 mmol) and NaO²Bu (0.20 mmol) in methylcyclohexane (1.0 mL) for 15 h (Table S2). Under these conditions, 110 °C was found to be an
optimal temperature with a borylated product 2-B being formed in 58% (Entry 5).

The effect of the amount of 1g was then examined using 2 (0.50 mmol), 1g, [Ir(OMe)(cod)]$_2$ (0.050 mmol), ICy·HCl (0.10 mmol) and NaO-Bu (0.20 mmol) in methylcyclohexane (1.0 mL) at 110 °C, 15 h (Table S3). Under these conditions, 2 was found to be an optimal amount of 1g with a borylated product 2-B being formed in 58% (Entry 4).

The effect of the amount of ICy·HCl was then examined using 2 (0.50 mmol), 1g (1.0 mmol), [Ir(OMe)(cod)]$_2$ (0.050 mmol), ICy·HCl and NaO-Bu in methylcyclohexane (1.0 mL) at 110 °C, 15 h (Table S4). Under these conditions, 20 mol % was found to be an optimal amount of ICy·HCl with a borylated product 2-B being formed in 58% (Entry 3).
The effect of the amount of \([\text{Ir(OMe)cod]}_2\) was then examined using \(\mathbf{2}\) (0.50 mmol), \(\mathbf{1g}\) (1.0 mmol), \([\text{Ir(OMe)cod]}_2\) (x mmol), ICy-HCl and NaO-Bu in methycyclohexane (1.0 mL) at 110 °C for 15 h (Table S5). Under these conditions, 10 mol % was found to be an optimal amount of \([\text{Ir(OMe)cod]}_2\) with a borylated product \(\mathbf{2-B}\) being formed in 58% (Entry 4).

| Entry | x [mol %] | NMR yield [%] | 2-Isomer/3-Isomer | Recovered \(\mathbf{2}\) [%] |
|-------|-----------|---------------|-------------------|---------------------|
| 1     | 10        | 24            | 83/17             | 59                  |
| 2     | 15        | 31            | 94/6              | 54                  |
| 3     | 20        | 58            | 95/5              | 44                  |
| 4     | 25        | 61            | 95/5              | 33                  |
| 5     | 30        | 58            | 93/7              | 46                  |
| 6     | 35        | 61            | 92/8              | 42                  |
| 7     | 40        | 57            | 91/9              | 51                  |

The effect of the amount of \([\text{Ir(OMe)cod]}_2\) was also examined using \(\mathbf{10}\) (0.50 mmol), \(\mathbf{1g}\) (1.0 mmol), \([\text{Ir(OMe)cod]}_2\), ICy-HCl and NaOr-Bu in methycyclohexane (1.0 mL) at 110 °C for 4 h (Table S6). Under these conditions, we were able to reduce the catalyst loading to 10 mol % without any loss of the yield of the product (Entry 4). This result indicate that benzo[b]thiophene is more reactive than \(N\)-methylindole toward this borylation.
The effect of the solvent was then examined using 2 (0.50 mmol), 1g (1.0 mmol), [Ir(OMe)(cod)]₂ (0.050 mmol), ICy·HCl (0.10 mmol) and NaOt-Bu (0.20 mmol) in solvent (1.0 mL) at 110 °C, 15 h (Table S7). Under these conditions, methylcyclohexane was found to be an optimal solvent with a borylated product 2-B being formed in 58% (Entry 1).

The effect of H₂ scavengers was then examined using 2 (0.50 mmol), 1g (1.0 mmol), [Ir(OMe)(cod)]₂ (0.050 mmol), ICy·HCl (0.10 mmol), NaOt-Bu (0.20 mmol) and H₂ scavenger (0.50 mmol) in methylcyclohexane (1.0 mL) at 110 °C, 15 h (Table S8). However, addition of a hydrogen scavenger did not improve the yield of 2-B under these conditions (Entry 9).
The effect of the reaction time was then examined using 2 (0.50 mmol), 1g (1.0 mmol), [Ir(OMe)(cod)]_2 (0.050 mmol), ICy·HCl (0.10 mmol) and NaO'Bu (0.20 mmol) in methylcyclohexane (1.0 mL) at 110 °C (Table S9). Under these conditions, 4 h were found to be an optimal reaction time with a borylated product 2-B being formed in 72% (Entry 3).

Table S9. Effect of reaction time.

| Entry | t [h] | NMR yield [%] | 2-Isomer/3-Isomer | Recovered 2 [%] |
|-------|-------|---------------|--------------------|-----------------|
| 1     | 1     | 37            | >99/1              | 68              |
| 2     | 3     | 57            | 98/2               | 49              |
| 3     | 4     | 72            | 99/1               | 40              |
| 4     | 5     | 68            | 96/4               | 47              |
| 5     | 6     | 69            | 94/6               | 42              |
| 6     | 9     | 60            | 98/2               | 37              |
| 7     | 12    | 57            | 98/2               | 39              |
| 8     | 15    | 58            | 95/5               | 44              |
| 9     | 24    | 50            | 92/8               | 58              |
| 10    | 48    | 42            | 90/10              | 52              |

a: H_2 scavenger was 2 equiv.
b: The reaction was conducted two neck flask in refluxing solvent.

IV-II. Optimization studies for arenes
The effect of the ligand was initially examined using 2 (0.50 mmol), [Ir(OMe)(cod)]_2 (0.050 mmol), ligand (0.10 mmol) and NaO'Bu (0.20 mmol) in benzene (1.0 mL) at 110 °C, 18 h (Table S10). Under these conditions, ICy·HCl was found to be an optimal ligand with a borylated product 17-B being formed in 53% (Entry 1).
The effect of the temperature was then examined using 2 (0.50 mmol), [Ir(OMe)(cod)]₂ (0.050 mmol), ICy·HCl (0.10 mmol) and NaOt-Bu (0.20 mmol) in benzene (1.0 mL) for 15 h (Table S11). Under these conditions, 110 °C was found to be an optimal temperature with a borylated product 17-B being formed in 47% (Entry 4).

The effect of the reaction time was then examined using 2 (0.50 mmol), [Ir(OMe)(cod)]₂ (0.050 mmol), ICy·HCl (0.10 mmol) and NaOt-Bu (0.20 mmol) in benzene (1.0 mL) at 110 °C (Table S12). Under these conditions, 18 h was found to be an optimal reaction time with a borylated product 17-B being formed in 53% (Entry 7).
The effect of the amount of ICy·HCl was initially examined using 2 (0.50 mmol), \([\text{Ir(OMe)(cod)}]_2\) (0.050 mmol), ICy·HCl and NaO\textsubscript{t}-Bu in benzene (1.0 mL) at 110 °C for 18 h (Table S13). Under these conditions, 20 mol % was found to be an optimal amount of ICy·HCl with a borylated product 17-B being formed in 53% (Entry 1).

### IV. Typical procedure

**Method A: Procedure for the Ir-catalyzed borylation of heteroarenes using 1g.**

In a glovebox filled with nitrogen, \([\text{Ir(OMe)(cod)}]_2\) (33.1 mg, 0.050 mmol, 0.10 equiv), ICy·HCl (26.2 mg, 0.10 mmol, 0.20 equiv), NaO\textsubscript{t}-Bu (19.2 mg, 0.20 mmol, 0.40 equiv) and methylcyclohexane (1.0 mL) were added to a 10 mL-sample vial with a Teflon-sealed screwcap, and stirred for 5 min at room temperature. A heteroarene (0.50 mmol, 1.0 equiv) and 1g (113.1 mg, 2.0 equiv) were added, and then the cap was screwed on seal the vial. The vial was stirred at 110 °C for 4 h. The reaction mixture was cooled to room temperature. Pinacol (236 mg, 2.0 mmol) in THF (2.0...
mL) was added and the reaction mixture was stirred under N₂ at room temperature for 1.5 h. The crude mixture was filtered through a pad of Celite and eluted with EtOAc. The filtrate was concentrated in vacuo and sampled for analysis by ¹H NMR spectroscopy using 1,2-dichloroethane as an internal standard. The residue was purified by flash column chromatography over silica gel eluting with hexane/EtOAc. Product-containing fractions were concentrated in vacuo to give a pure borylated product.

Method B: Procedure for the Ir-catalyzed borylation of arenes using 1g.

In a glovebox, [Ir(OMe)(cod)]₂ (33.1 mg, 0.050 mmol, 0.10 equiv), ICy·HCl (26.2 mg, 0.10 mmol, 0.20 equiv), NaOt-Bu (19.2 mg, 0.20 mmol, 0.40 equiv) and benzene (1.0 mL) were added to a 10 mL-sample vial with a Teflon-sealed screwcap, and stirred for 5 min at room temperature. 1g (113.1 mg, 1.0 mmol, 2.0 equiv) was added, and then the cap was screwed on to seal the vial. The vial was stirred at 110 °C for 18 h. The reaction mixture was cooled to room temperature. Pinacol (236 mg, 2.0 mmol, 4.0 equiv) in THF (2.0 mL) was added and the reaction mixture was stirred for 1.5 h at room temperature under N₂. The crude mixture was filtered through a pad of Celite and eluted with EtOAc. The filtrate was concentrated in vacuo and sampled for analysis by ¹H NMR spectroscopy using 1,2-dichloroethane as an internal standard. The residue was purified by flash column chromatography over silica gel eluting with hexane/EtOAc. Product-containing fractions were concentrated in vacuo to give a pure borylated product.

A procedure for the gram scale synthesis of 2-borylated 10.

In a glovebox, [Ir(OMe)(cod)]₂ (91.0 mg, 0.138 mmol, 0.025 equiv), ICy·HCl (73.8 mg, 0.275 mmol, 0.050 equiv), NaOt-Bu (52.8 mg, 0.55 mmol, 0.10 equiv) and methylcyclohexane (11.0 mL) were added to a 190 mL-sample vial with a Teflon-sealed screwcap, and stirred for 5 min at room temperature. Compounds 10 (1.00 g, 5.50 mmol, 1.0 equiv) and 1g (1.24g, 11.0 mmol, 2.0 equiv) were added, and then the cap was screwed on to seal the vial. The vial was stirred at 110 °C for 24 h. The reaction mixture was cooled to room temperature. Pinacol (2.57 g, 22.0 mmol, 4.0 equiv) in THF (16 mL) was added and the reaction mixture was stirred under N₂ at room temperature for 1.5 h. The crude mixture was filtered through a pad of Celite and eluted with EtOAc. The filtrate was concentrated in vacuo and sampled for analysis ¹H NMR spectroscopy using 1,2-dichloroethane as an internal standard. The residue was purified by flash column chromatography over silica gel eluting with hexane/EtOAc (40/1) solution. Product-containing fractions were concentrated in vacuo to give 10-B as a white solid (1.25 g, 74%).
V. Spectroscopic Data

1-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (2-B). [CAS: 596819-10-2]

Method A was used. Rf 0.14 (Hexane/EtOAc =20/1). White solid (83 mg, 65%).

$^1$H NMR (C$_6$D$_6$, 399.78 MHz): δ 1.12 (s, 12H), 3.69 (s, 3H), 7.13 (d, $J = 7.8$ Hz, 2H), 7.27 (td, $J = 0.9$, 7.8 Hz, 1H), 7.57 (s, 1H), 7.68-7.70 (m, 1H).

$^{13}$C NMR (C$_6$D$_6$, 100.53 MHz): δ 24.9, 32.1, 83.6, 110.1, 115.6, 119.9, 122.2, 123.6, 128.8, 140.9.

HRMS (EI): Calcd for C$_{15}$H$_{20}$BNO$_2$ 257.1587, Found 257.1585.

$^1$H NMR spectroscopic data was in agreement with the reported value.

5-Methoxy-1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (4-B).

[CAS: 1256360-41-4]

Method A was used. Rf 0.057 (Hexane/EtOAc = 40/1). White solid (69 mg, 48%).

$^1$H NMR (C$_6$D$_6$, 399.78 MHz): δ 1.13 (s, 12H), 3.45 (s, 3H), 3.68 (s, 3H), 6.99 (d, $J = 9.2$ Hz, 1H), 7.08 (d, $J = 2.6$ Hz, 1H), 7.19 (d, $J = 2.6$ Hz, 1H), 7.56 (s, 1H).

$^{13}$C NMR (C$_6$D$_6$, 100.53 MHz): δ 24.9, 30.1, 32.2, 55.2, 83.6, 102.4, 110.9, 114.9, 136.53, 154.9.

HRMS (EI): Calcd for C$_{16}$H$_{22}$BNO$_3$ 287.1693, Found 287.1695.

$^1$H NMR spectroscopic data was in agreement with the reported value.

5-Fluoro-1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (5-B).

[CAS: 1683582-67-3]

Method A was used. Rf 0.085 (Hexane/EtOAc = 40/1). White solid (103 mg, 75%).

$^1$H NMR (C$_6$D$_6$, 399.78 MHz): δ 1.10 (s, 12H), 3.57 (s, 3H), 6.79 (dd, $J = 4.1$, 9.2 Hz, 1H), 7.00 (dt, $J = 2.3$, 9.2 Hz, 1H), 7.28 (dd, $J = 2.3$, 9.6 Hz, 1H), 7.36 (s, 1H).

$^3$ Furukawa, T.; Tobisu, M.; Chatani, N. Chem. Commun. 2015, 51, 6508.
13C NMR (C<sub>6</sub>D<sub>6</sub>, 100.53 MHz): δ 24.8, 32.2, 83.7, 106.3 (d, J = 23 Hz), 110.8 (d, J = 9.5 Hz), 112.2 (d, J = 27 Hz), 115.1 (d, J = 4.8 Hz), 128.7 (d, J = 9.5 Hz), 137.5, 158.5 (d, J = 234 Hz).

HRMS (EI): Calcd for C<sub>15</sub>H<sub>20</sub>BFNO<sub>2</sub> 276.1568, Found 276.1570.

1H NMR spectroscopic data was in agreement with the reported value.<sup>3</sup>

5-Chloro-1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (6-B).

![Structure of 6-B](image)

Method A was used. After purification by flush column chromatography over silica gel, a mixture of borylated product 6B and 6 were obtained (6-B: 66%, 6: 22%). GC/MS analysis revealed the existence of 6-B and 6; 6-B had an m/z of 291 (M<sup>+</sup>), and 6 had an m/z of 165 (M<sup>+</sup>). The identity and ratio of 6 and 6B was determined by the 1H NMR spectrum of the mixture. The resonances specific to each compound are as follows: 1H NMR (C<sub>6</sub>D<sub>6</sub>, 399.78 MHz): δ 0.454 (s, 3H, 6), 3.52 (s, 3H, 6-B).

![Structure of 6](image)

R<sub>f</sub> 0.086 (Hexane/EtOAc = 40/1). White solid as a 3:1 mixture of 6-B and 6 (114 mg). Mp = 111 °C.

1H NMR (C<sub>6</sub>D<sub>6</sub>, 399.78 MHz): δ 1.10 (s, 12H), 3.52 (s, 3H), 6.76 (d, 1H, J = 8.8 Hz), 7.23 (dd, J = 2.0, 8.7 Hz, 1H), 7.33 (s, 1H), 7.60 (d, J = 1.9 Hz, 1H).

13C NMR (C<sub>6</sub>D<sub>6</sub>, 100.53 MHz): δ 24.8, 32.1, 83.8, 111.1, 114.8, 121.4, 123.9, 125.7, 129.5, 139.0.

IR (ATR): 2977 w, 2927 w, 2361 m, 2339 w, 1735 w, 1649 w, 1558 w, 1526 m, 1438 w, 1361 s, 1306 s, 1264 m, 1208 w, 1137 s, 1106 m, 1077 m, 1030 m, 974 w, 949 w, 866 m, 849 s, 805 m, 780 w, 732 w, 692 w, 671 m.

MS m/z (% relative intensity): 293 (32), 292 (24), 291 (M<sup>+</sup>), 290 (25), 218 (12), 209 (18), 208 (17), 207 (10), 206 (31), 205 (12), 193 (12), 192 (21), 191 (35), 190 (22).

HRMS (EI): Calcd for C<sub>15</sub>H<sub>19</sub>BClNO<sub>2</sub> 291.1197, Found 291.1204.

5-Bromo-1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (7-B) [CAS: 1192037-87-8] and 1-methyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (7-B'). [CAS: 837392-62-8]

![Structure of 7](image)

Method A was used. After purification by flush column chromatography over silica gel, a mixture of two borylated products and 7 was obtained (7-B: 50%, 7-B': 6%, 7: 20%). GC/MS analysis revealed the existence of two borylated products and 7; 7-B had an m/z of 335 (M<sup>+</sup>), 7-B' had an m/z of 257
(M⁺), and 7 had an m/z of 209 (M⁺). The identity and ratio of each of these was determined by the 1H NMR spectrum of the mixture. The resonances specific to each isomer are as follows: 1H NMR (C₆D₆, 399.78 MHz): δ 2.74 (s, 3H, 7), 3.50 (s, 3H, 7-B), 3.69 (s, 3H, 7-B').

MS m/z (% relative intensity) 7-B: 338 (16), 337 (99), 336 (39), 335 (M⁺, 100), 334 (23), 255 (15), 253 (15), 252 (25), 251 (11), 250 (22), 237 (24), 236 (24), 235 (27), 234 (14), 183 (11), 156 (10).

7-B': 258 (18), 257 (M⁺, 100), 256 (25), 184 (21), 175 (21), 172 (31), 158 (15), 157 (36), 156 (25).

HRMS (EI) 7-B: Calcd for C₁₅H₁₉BBrNO₂ 335.0692, Found 335.0689.

7-B': Calcd for C₁₅H₂₀BNO₂ 257.1587, Found 257.1583.

1H NMR spectroscopic data was in agreement with the reported value.

1,4-Dimethyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-indole (8-B).

Method A was used. Rf 0.14 (Hexane/EtOAc = 40/1). White solid (69 mg, 51%). Mp = 151 °C.

1H NMR (C₆D₆, 399.78 MHz): δ 1.14 (s, 12H), 2.51 (s, 3H), 3.71 (s, 3H), 6.97 (d, J = 7.4 Hz, 1H), 7.04 (d, J = 8.2 Hz, 1H), 7.25 (t, J = 8.2 Hz, 1H), 7.64 (s, 1H).

13C NMR (C₆D₆, 100.53 MHz): δ 18.8, 24.9, 32.3, 83.6, 107.9, 114.3, 120.1, 124.0, 128.8, 131.4, 140.8.

IR (ATR): 2975 w, 2921 w, 2361 w, 1606 w, 1580 w, 1522 m, 1496 w, 1467 w, 1383 m, 1349 w, 1293 m, 1258 m, 1239 m, 1216 w, 1139 m, 1111 w, 1070 m, 964 w, 858 m, 827 w, 805 w, 770 m, 739 m, 688 m, 670 w.

MS m/z (% relative intensity): 272 (18), 271 (M⁺, 100), 270 (25), 198 (16), 189 (29), 188 (11), 172 (10), 171 (29), 170 (24).

HRMS (EI): Calcd for C₁₆H₂₂BNO₂ 271.1744, Found 271.17430.

2-(Benzo[b]thiophen-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolan (9-B). [CAS: 376584-76-8]

Method A was used. Rf 0.086 (Hexane/EtOAc = 40/1). White solid (122 mg, 94%).

1H NMR (C₆D₆, 399.78 MHz): δ 1.10 (s, 12H), 7.01-7.09 (m, 2H), 7.54-7.58 (m, 2H), 8.06 (s, 1H).

1H NMR (CDCl₃, 399.78 MHz): δ 1.38 (s, 12H), 7.35-7.39 (m, 2H), 7.85-7.92 (m, 3H).

13C NMR (C₆D₆, 100.53 MHz): δ 24.8, 84.3, 124.4, 124.7, 122.9, 125.6, 135.3, 141.0, 144.4.

HRMS (EI) A: Calcd for C₁₄H₁₂BO₂S 260.1042, Found 260.1040.

Stadlwieser, J. F.; Dambaur, M. E. Helv. Chim. Acta. 2006, 89, 936.
$^1$H NMR spectroscopic data was in agreement with the reported value.$^5$

2-(5-Chloro-3-methylbenzo[b]thiophen-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (10-B).

[CAS: 1809298-96-1]

\[ \text{Cl} \]

Method A was used. $R_f$ 0.22 (Hexane/EtOAc = 40/1). White solid (140 mg, 91%).

$^1$H NMR (C$_6$D$_6$, 399.78 MHz): $\delta$ 1.07 (s, 12H), 2.51 (s, 3H), 7.04 (dd, $J = 1.8, 8.7$ Hz, 1H), 7.18 (d, $J = 8.7$ Hz, 1H), 7.63 (d, $J = 1.8$ Hz, 1H).

$^{13}$C NMR (C$_6$D$_6$, 100.53 MHz): $\delta$ 14.0, 24.8, 84.1, 122.7, 124.0, 126.1, 130.5, 141.9, 142.9, 143.4.

HRMS (EI): Calcd for C$_{15}$H$_{18}$BClO$_2$S 308.0809, Found 308.0811.

$^1$H NMR spectroscopic data was in agreement with the reported value.$^5$

2-(Benzofuran-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (11-B). [CAS: 402503-13-3]

\[ \text{O} \]

Method A was used. $R_f$ 0.057 (Hexane/EtOAc = 40/1). White solid (79 mg, 65%).

$^1$H NMR (C$_6$D$_6$, 399.78 MHz): $\delta$ 1.08 (s, 12H), 6.98-7.08 (m, 2H), 7.34-7.36 (m, 1H), 7.40-7.42 (m, 1H), 7.48 (d, $J = 0.92$ Hz, 1H).

$^1$H NMR (CDCl$_3$, 399.78 MHz): $\delta$ 1.39 (s, 12H), 7.23 (t, $J = 7.8$ Hz, 1H), 7.34 (td, 0.9, $J = 8.2$ Hz, 1H), 7.40 (s, 1H), 7.57 (d, $J = 8.2$ Hz, 1H), 7.63 (1H, $J = 7.8$ Hz, 1H).

$^{13}$C NMR (C$_6$D$_6$, 100.53 MHz): $\delta$ 24.8, 84.4, 112.1, 120.1, 122.2, 123.0, 126.3, 158.3. One carbon peak is overlapped with solvent peaks.

HRMS (EI): Calcd for C$_{14}$H$_{17}$BO$_3$ 244.1271, Found 244.1276.

$^1$H NMR spectroscopic data was in agreement with the reported value.$^5$

1-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-pyrrole (12-B).

[CAS: 850567-47-4]

\[ \text{Me} \]

Method B was used except that the reaction was conducted in N-methyl pyrrole (1.0 mL).

$R_f$ 0.14 (Hexane/EtOAc = 40/1). White solid (52 mg, 50%).

$^1$H NMR (C$_6$D$_6$, 399.78 MHz): $\delta$ 1.11 (s, 12H), 3.52 (s, 3H), 6.30 (dd, $J = 1.4, 2.3$ Hz, 1H), 7.22 (t, $J$

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$^5$ Furukawa, T.; Tobisu, M.; Chatani, N. *J. Am. Chem. Soc.* **2015**, *137*, 12211.
= 1.8 Hz, 1H), 7.33 (dd, J = 1.4, 2.3 Hz, 1H).

$^{13}$C NMR (CD$_6$D, 100.53 MHz): δ 24.9, 36.3, 83.0, 109.2, 123.4. One carbon peak is overlapped with solvent peaks.

HRMS (EI): Calcd for C$_{11}$H$_{18}$BNO$_2$ 207.1431, Found 207.1431.

$^1$H NMR spectroscopic data was in agreement with the reported value.$^3$

4,4,5,5-Tetramethyl-2-(thiophen-2-yl)-1,3,2-dioxaborolane (13-B) and 2,5-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)thiophene (13-2B).

Method A was used. The product was obtained as a mixture of mono and diborylated thiophenes. It was possible to purify two products by flush column chromatography over silica gel.

4,4,5,5-Tetramethyl-2-(thiophen-2-yl)-1,3,2-dioxaborolane (13-B). [CAS: 193978-23-3]

![Structure of 13-B](image)

R$_f$ 0.22 (Hexane/EtOAc = 40/1). White solid (41 mg, 39%).

$^1$H NMR (CD$_6$D, 399.78 MHz): δ 1.08 (s, 12H), 6.89 (m, 1H), 7.18 (dd, J = 0.92, 4.6 Hz, 1H), 7.88-7.89 (m, 1H).

$^{13}$C NMR (CD$_6$D, 100.53 MHz): δ 24.8, 84.0, 128.5, 132.8, 137.7.

HRMS (EI): Calcd for C$_{10}$H$_{15}$BO$_2$S 210.0886, Found 210.0889.

$^1$H NMR spectroscopic data was in agreement with the reported value.$^6$

2,5-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)thiophene (13-2B). [CAS: 175361-81-6]

![Structure of 13-2B](image)

R$_f$ 0.14 (Hexane/EtOAc = 40/1). White solid (54 mg, 32%).

$^1$H NMR (CD$_6$D, 399.78 MHz): δ 1.03 (s, 24H), 7.97 (s, 2H).

$^1$H NMR (CDCl$_3$, 399.78 MHz): δ 1.34 (s, 24H), 7.66 (s, 2H).

$^{13}$C NMR (CD$_6$D, 100.53 MHz): δ 24.8, 84.1, 138.6.

HRMS (EI): Calcd for C$_{16}$H$_{26}$B$_2$O$_4$S 336.1738, Found 336.1738.

$^1$H NMR spectroscopic data was in agreement with the reported value.$^7$

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$^6$ Boller, T. M.; Murphy, J. M.; Hapke, M.; Ishiyama, T.; Miyaura, N.; Hartwig, J. F. J. Am. Chem. Soc. 2005, 127, 14263.

$^7$ Guerrand, H. D. S.; Marciasini, L. D.; Jousseame, M.; Valtier. M.; Pucheault, M. Chem. Eur. J. 2014, 20, 5573.
4,4,5,5-Tetramethyl-2-(5-methylthiophen-2-yl)-1,3,2-dioxaborolane (14-B). [CAS: 476004-80-5]

![Structural formula]

Method A was used. R_f 0.14 (Hexane/EtOAc = 40/1). Colorless oil (108 mg, 96%).

^1_H NMR (C_6D_6, 399.78 MHz): δ 1.09 (s, 12H), 2.11 (s, 3H), 6.62 (d, J = 3.3 Hz, 1H), 7.8 (d, J = 3.5 Hz, 1H).

^13_C NMR (C_6D_6, 100.53 MHz): δ 15.1, 24.9, 83.9, 127.5, 138.4, 147.8.

HRMS (EI): Calcd for C_{11}H_{17}BO_{3}S 208.1271, Found 208.1272.

^1_H NMR spectroscopic data was in agreement with the reported value.\(^5\)

2-(5-Methoxythiophen-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (15-B).

[CAS: 596819-12-4]

![Structural formula]

Method A was used. R_f 0.14 (Hexane/EtOAc = 40/1). Colorless oil (109 mg, 91%).

^1_H NMR (C_6D_6, 399.78 MHz): δ 1.09 (s, 12H), 3.24 (s, 3H), 6.07 (d, J = 4.0 Hz, 1H), 7.62 (d, J = 3.9 Hz, 1H).

^1_H NMR (CDCl_3, 399.78 MHz): δ 1.32 (s, 12H), 3.92 (s, 3H), 6.30 (d, J = 3.8 Hz, 1H), 7.33 (d, J = 3.8 Hz, 1H).

^13_C NMR (C_6D_6, 100.53 MHz): δ 24.9, 59.7, 83.8, 106.4, 137.2, 173.5.

HRMS (EI): Calcd for C_{16}H_{26}BO_{2}S 240.0991, Found 240.0994.

^1_H NMR spectroscopic data was in agreement with the reported value.\(^5\)

4,4,5,5-Tetramethyl-2-(5-methylfuran-2-yl)-1,3,2-dioxaborolane (16-B). [CAS: 338998-93-9]

![Structural formula]

Method A was used. R_f 0.028 (Hexane/EtOAc = 40/1). Colorless oil (71 mg, 68%).

^1_H NMR (C_6D_6, 399.78 MHz): δ 1.09 (s, 12H), 1.99 (s, 3H), 5.81 (d, J = 2.3 Hz, 1H), 7.22 (d, J = 3.2 Hz, 1H).

^13_C NMR (C_6D_6, 100.53 MHz): δ 13.6, 24.8, 83.7, 107.2, 125.4, 157.6.

HRMS (EI): Calcd for C_{11}H_{17}BO_{3} 208.1271, Found 208.1270.

^1_H NMR spectroscopic data was in agreement with the reported value.\(^8\)

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\(^5\) Hatanaka, T.; Ohki, Y.; Tatsumi, K. *Chem. Asian. J.* 2010, 5, 1657.
4,4,5,5-Tetramethyl-2-phenyl-1,3,2-dioxaborolane (17-B). [CAS: 24388-23-6]

Method B was used. Rf 0.20 (Hexane/EtOAc = 40/1). White solid (49 mg, 48%).

$^1$H NMR ($C_6D_6$, 399.78 MHz): δ 1.11 (s, 12H), 7.21-7.22 (m, 3H), 8.15-8.17 (m, 2H).

$^1$H NMR (CDCl$_3$, 399.78 MHz): δ 1.35 (s, 12H), 7.34-7.38 (m, 2H), 7.44-7.48 (m, 1H), 7.78-7.82 (m, 1H).

$^{13}$C NMR (CDCl$_3$, 100.53 MHz): δ 25.0, 83.9, 127.8, 131.4, 134.9.

HRMS (EI): Calcd for $C_{12}H_{17}BO_2$ 204.1322, Found 204.1321.

Borylation of toluene (Entry 2, Table 3). [CAS: 253342-48-2] and [CAS: 195062-57-8]

Method B was followed except that the reaction was conducted in toluene (1.0 mL). After purification by flash column chromatography over silica gel eluting with hexane/AcOEt = 20/1, a mixture of two isomers was obtained. GC/MS analysis revealed the two isomers of the borylated products had an m/z of 218 (M$^+$). The identity and ratio of each of the two isomers were determined by comparing the $^1$H NMR spectrum of the product mixture with those reported in the literature. The resonances specific to each isomer are as follows: $^1$H NMR (CDCl$_3$, 399.78 MHz): 7.60-7.64 ppm (m, 2H, meta isomer, H$_a$ and H$_b$), 7.70 ppm (d, $J = 7.8$ Hz, 2H, para isomer, H$_c$).

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9 Ishiyama, T.; Takagi, J.; Ishida, K.; Miyaura, N.; Hartwig, J. F. J. Am. Chem. Soc. 2002, 124, 390.
Borylation of anisole (Entry 3, Table 3). [CAS: 325142-84-5] and [CAS: 171364-79-7]

Method B was followed except that the reaction was conducted in anisole (1.0 mL). After purification by flash column chromatography over silica gel eluting with hexane/AcOEt = 20/1, a mixture of two isomers was obtained. GC/MS analysis revealed the two isomers of the borylated products had an m/z of 232 (M⁺). The identity and ratio of each of the two isomers were determined by comparing the ¹H NMR spectrum of the product mixture with those reported in the literature. The resonances specific to each isomer are as follows: ¹H NMR (CDCl₃, 399.78 MHz): 7.01 ppm (ddd, J = 0.8, 2.8, 8.0 Hz, 1H, meta isomer, Hₐ); 7.75 ppm (d, J = 8.2 Hz, 2H, para isomer, Hₐ).
Borylation of trifluoromethylbenzene (Entry 4, Table 3).
[CAS: 325142-82-3] and [CAS: 214360-65-3]

Method B was followed except that the reaction was conducted in trifluoromethylbenzene (1.0 mL). After purification by flash column chromatography over silica gel eluting with hexane/AcOEt = 40/1, a mixture of two isomers was obtained. GC/MS analysis revealed the two isomers of the borylated products had an m/z of 272 (M⁺). The identity and ratio of each of the two isomers were determined by comparing the ¹H NMR spectrum of the product mixture with those reported in the literature. The resonances specific to each isomer are as follows: ¹H NMR (CD₆, 399.78 MHz): 7.96 (d, J = 8.2 Hz, 1H, para isomer, H_a), 8.46 (s, 1H, meta isomer, H_b).
2-(3,5-Dichlorophenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (21-B). [CAS: 68716-51-8]

Method B was followed except that the reaction was conducted in 1,3-dichlorobenzene (1.0 mL). White solid (42 mg, 31%).

\(^1\)H NMR (CDCl\textsubscript{3}, 399.78 MHz): \(\delta\) 1.34 (s, 12H), 7.43 (t, \(J = 2.2\) Hz, 1H), 7.64 (d, \(J = 2.3\) Hz, 2H).

\(^13\)C NMR (CDCl\textsubscript{3}, 100.53 MHz): \(\delta\) 25.0, 84.7, 131.2, 132.9, 134.9.

HRMS (EI): Calcd for C\textsubscript{12}H\textsubscript{15}BCl\textsubscript{2}O\textsubscript{2} 272.0542, Found 272.0540.

4,4,5,5-Tetramethyl-2-(naphthalen-2-yl)-1,3,2-dioxaborolane (22-B). [CAS: 256652-04-7]

In a glovebox, [Ir(OMe)(cod)]\textsubscript{2} (33.1 mg, 0.050 mmol, 0.10 equiv), ICy·HCl (26.2 mg, 0.10 mmol, 0.20 equiv), NaOt-Bu (19.2 mg, 0.20 mmol, 0.40 equiv) and methylcyclohexane (1.0 mL) were added to a 10 mL-sample vial with Teflon-sealed screwcap, and stirred for 5 min at room
temperature. A naphthalene (384.1 mg, 3.0 mmol, 6.0 equiv) and 1g (113.1 mg, 1.0 mmol, 2.0 equiv) were then added, and the cap was applied to seal the vial. The vial was stirred at 110 °C for 4 h. After the reaction mixture was cooled to room temperature, pinacol (236 mg, 2.0 mmol) in THF (2.0 mL) was added and stirred for 1.5 h at room temperature under N₂. The crude mixture was filtered through a pad of Celite eluting with AcOEt. The filtrate was concentrated in vacuo and analyzed by ¹H NMR using 1,2-dichloroethane as an internal standard. The crude mixture was concentrated under reduced pressure, and purified by flash column chromatography over silica gel eluting with hexane/AcOEt (40/1) solution. The filtrate was concentrated in vacuo to give a pure borylated product as a white solid (63.5 mg, 50%).

Rᵣ 0.17 (Hexane/EtOAc = 40/1). White solid (64 mg, 50%).

¹H NMR (C₆D₆, 399.78 MHz): δ 1.16 (s, 12H), 7.19-7.24 (m, 2H), 7.6 (d, J = 8.2 Hz, 1H), 7.69 (t, J = 18.3, 18.3 Hz, 2H), 8.23 (d, J = 7.3 Hz, 1H), 8.75 (s, 1H).

¹H NMR (CDCl₃, 399.78 MHz): δ 1.40 (s, 12H), 7.47-7.53 (m, 2H), 7.82-7.83 (m, 3H), 7.89 (d, J = 7.8 Hz, 1H), 8.37 (s, 1H).

¹³C NMR (CDCl₃, 100.53 MHz): δ 25.0, 84.0, 125.9, 127.0, 127.8, 128.7, 130.5, 132.9, 135.1, 136.3. One carbon peak is overlapped with solvent peaks.

HRMS (EI): Calcd for C₁₆H₁₉BO₂ 254.1478, Found 254.1482.

¹H NMR spectroscopic data was in agreement with the reported value.¹⁰

2-(5-Chloro-3-methylbenzo[b]thiophen-2-yl)-5,5-dimethyl-1,3,2-dioxaborinane (10-Bnep).

Method A was followed except that after the reaction mixture was cooled to room temperature, the neopentyl glycol (208 mg, 2.0 mmol) in THF (2.0 mL) was added and stirred for 1.5 h at room temperature under N₂.

Rᵣ 0.085 (Hexane/EtOAc = 40/1). White Solid (130 mg, 88%). Mp = 119 °C.

¹H NMR (C₆D₆, 399.78 MHz): δ 0.53 (s, 6H), 2.50 (s, 3H), 3.32 (s, 4H), 7.08 (dd, J = 1.8, 7.8 Hz, 1H), 7.27 (d, J = 7.3 Hz, 1H), 7.69 (d, J = 2.3 Hz, 1H).

¹³C NMR (C₆D₆, 100.53 MHz): δ 13.7, 21.5, 31.5, 72.2, 122.6, 124.0, 125.7, 130.3, 141.4, 141.6, 143.5.

IR (ATR): 2964 w, 2936 w, 1895 w, 1580 w, 1525 m, 1475 w, 1438 w, 1415 m, 1375 w, 1341 m, 1290 s, 1272 s, 1244 s, 1149 w, 1117 s, 1074 m, 1028 w, 977 w, 933 w, 916 w, 894 w, 865 w, 850 m, 809 s, 729 w, 697 w, 669 m.

MS m/z (% relative intensity): 296 (38), 295 (27), 294 (M⁺, 100), 293 (30), 260 (12), 259 (67), 258 (21), 208 (15), 207 (14), 181 (14), 173 (15).

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¹⁰ Kinuta, H.; Tobisu, M.; Chatani, N. J. Am. Chem. Soc. 2015, 137, 1593.
HRMS (EI): Calcd for C_{14}H_{16}BClO_{2}S 294.0653, Found 294.0653.

2-(5-Chloro-3-methylbenzo[b]thiophen-2-yl)-4,4,6-trimethyl-1,3,2-dioxaborinane (10-Bmep).

Method A was followed except that after the reaction mixture was cooled to room temperature, the 2-methylpentane-2,4-diol (236 mg, 2.0 mmol) in THF (2.0 mL) was added and stirred under N\textsubscript{2} at room temperature for 1.5 h.

R\textsubscript{f} 0.23 (Hexane/EtOAc = 40/1). Colorless oil (134 mg, 87%).

\textsuperscript{1}H NMR (C\textsubscript{6}D\textsubscript{6}, 399.78 MHz): \( \delta \) 1.00 (s, 3H), 1.06 (d, \( J = 6.4 \) Hz, 3H), 1.12-1.14 (m, 5H), 2.57 (s, 3H), 3.87-3.95 (m, 1H), 7.08 (dd, \( J = 1.8, 8.5 \) Hz, 1H), 7.28 (d, \( J = 8.4 \) Hz, 1H), 7.70 (d, \( J = 1.8 \) Hz, 1H).

\textsuperscript{13}C NMR (C\textsubscript{6}D\textsubscript{6}, 100.53 MHz): \( \delta \) 13.6, 23.0, 28.0, 31.1, 45.7, 65.5, 71.7, 122.5, 123.9, 125.6, 130.3, 141.1, 141.3, 143.5.

IR (ATR): 2973 \textit{w}, 2914 \textit{w}, 2360 \textit{w}, 2340 \textit{w}, 1737 w, 1581 w, 1554 w, 1523 w, 1440 w, 1396 m, 1379 w, 1344 m, 1319 w, 1286 s, 1265 s, 1243 s, 1206 m, 1160 m, 1109 m, 1077 m, 1059 w, 1027 w, 980 w, 963 w, 937 w, 901 w, 864 w, 851 w, 823 w, 799 m, 768 m, 730 w, 692 w, 972 m.

MS m/z (% relative intensity): 310 (38), 309 (26), 308 (M\textsuperscript{+}, 100), 307 (24), 254 (13), 252 (35), 251 (32), 237 (16), 225 (11), 211 (18), 210 (42), 209 (57), 208 (97), 207 (24), 182 (11), 181 (18), 173 (26), 83 (21), 55 (10), 43 (26).

HRMS (EI): Calcd for C_{15}H_{18}BClO_{2}S 308.0809, Found 308.0804.

2-(5-Chloro-3-methylbenzo[b]thiophen-2-yl)-2,3-dihydro-1H-naphtho[1,8-de][1,3,2]diazaborinane (10-Bdan).

Method A was followed except that after the reaction mixture was cooled to room temperature, the 1,8-naphthalenediamine (316 mg, 2.0 mmol) in THF (2.0 mL) was added and stirred for 1.5 h at room temperature under N\textsubscript{2}.

R\textsubscript{f} 0.29 (Hexane/EtOAc = 20/1). White solid (131 mg, 75\%). Mp = 173 \textdegree C.

\textsuperscript{1}H NMR (C\textsubscript{6}D\textsubscript{6}, 399.78 MHz): \( \delta \) 1.97 (s, 3H), 5.32 (s, 2H), 5.92 (dd, \( J = 0.92, 7.3 \) Hz, 2H), 7.02-7.15 (m, 5H), 7.33 (d, \( J = 8.2 \) Hz, 1H). 7.65 (d, \( J = 1.8 \) Hz, 1H).

\textsuperscript{13}C NMR (C\textsubscript{6}D\textsubscript{6}, 100.53 MHz): \( \delta \) 13.9, 106.8, 118.8, 120.6, 122.2, 123.8, 125.5, 130.9, 136.9, 137.0,
IR (ATR): 3428 w, 3415 w, 3049 w, 2360 w, 1734 w, 1627 w, 1596 s, 1554 w, 1528 m, 1497 m, 1437 w, 1405 m, 1371 m, 1337 m, 1281 w, 1195 m, 1164 m, 1099 m, 1074 m, 1035 w, 935 w, 859 m, 814 m, 798 m, 751 s, 660 s.

MS m/z (% relative intensity): 350 (42), 349 (32), 348 (M+, 100), 347 (29), 174 (15), 173 (14), 166 (38), 165 (21).

HRMS (EI): Calcd for C_{19}H_{14}BClN_{2}S348.0659, Found 348.0662.

**Borylation of other substrates.**

\[ \text{1g (2.0 equiv)} \]

\[ \text{[Ir(OMe)(cod)]_2 (10 mol\%)} \]

\[ \text{ICy·HCl (20 mol\%)} \]

\[ \text{NaO\'Bu (40 mol\%)} \]

\[ \text{methylcyclohexane} \]

\[ 110 \degree \text{C, 4 h} \]

\[ \text{pinacol} \]

\[ \text{THF} \]

\[ \text{rt, 1.5 h} \]

\[ \text{45\%} \]

\[ \text{pinB} \]

\[ \text{40\%} \]

\[ \text{pinB} \]

\[ \text{11\%} \]

8\% by NMR

36\% by NMR
2-B Meindole Bpin F12 in CDCl₃
144.402
141.046
135.287
128.298
128.060
127.812
125.648
124.723
124.399
122.892
84.382
24.848

9-B

TI-4-205 F10-13_13C
S38
11-B
in CDCl3
13-2B
in CDCl₃
X: parts per Million: Proton

MeO

15-B

7.629
7.619
7.160
6.076
6.066
3.238
1.091
12.20

2.93
1.01
1.00
17-B
in C₆D₆
$X : \text{parts per Million : Carbon}^{13}$

![Chemical Structure](image)

- $134.882$
- $131.393$
- $127.846$
- $83.910$
- $77.475$
- $77.160$
- $76.836$
- $25.015$
X: parts per Million: Proton

7.652
7.646
7.438
7.432
7.427
7.262

1.340
0.000

Cl-

21-B

Cl-

O

O

S60

T4-dichloro F8-15
