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# Catalytic borylation of *o*-xylene and heteroarenes via C–H activation

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**Abstract**

Iridium and rhodium complexes catalyze the borylation of xylene and different heteroarenes using pinacolborane via C–H activation. Various five-membered heterocycles such as thiophene, pyrrole, thionaphthene, and indole derivatives yield the borylated products in moderate to good yields. In general, the reactions proceed with high selectivity to give borylation *ortho* to the heteroatom.


*Keywords:* Boronates; Borylation; C–H activation; Iridium; Rhodium

## 1. Introduction

The versatility of organoboron compounds in organic chemistry renders them attractive targets for synthesis. For example, palladium- and nickel-catalyzed cross-coupling reactions of boronic acids with aryl halides have become the most important method for the synthesis of biaryls (Suzuki reaction) [1]. In addition to their role in cross-coupling reactions, boronic acids and esters are used for the preparation of amines [2], alcohols [3], and olefins [4]. Traditionally, arylboronic acids are prepared by reacting aryl Grignard [5] or lithium [6] reagents with trialkylborates, followed by hydrolytic work up. More recently, Miyaura and coworkers [7] and Masuda and coworkers [8] have synthesized arylboronates by Pd-catalyzed coupling of (di)borane reagents and arenes substituted by a leaving group. Clearly, from an environmental point of view, the selective borylation of arenes as depicted in Scheme 1 is a more attractive goal. While the catalytic functionalization of inert C–H bonds remains one of the major challenges in synthetic organic chemistry [9], recently important progress has been made in the direct borylation of C–H bonds.

Theoretical estimation of B–H and B–C bond enthalpies gave conviction in organoborane synthesis via direct borylation of unsubstituted hydrocarbons [10]. Fundamental studies on hydrocarbon activation by $Cp^*M(PMe_3)(H)_2$ (M: Ir, Rh) were described by Bergman [11] and Jones [12]. The direct borylation of non-activated C–H bonds was first described using alkanes. Initial stoichiometric reactions developed by Bergmann [13] were soon followed up by catalytic protocols reported by Hartwig and coworkers [14]. Hartwig also developed a photochemical borylation of non-activated hydrocarbons using catalytic amounts of metal complexes [15]. In 1999, the first thermally induced metal-catalyzed reaction of a borane and an arene has been reported by Smith and coworkers [16]. Recently, Hartwig and coworkers have described the borylation of halogenated arenes even at room temperature [17]. Most reports on catalytic borylations use the substrate as solvent [18]. Nevertheless, in some cases cyclohexane [19] or octane [20] served as an inert solvent for borylation of arenes. Due to the importance of heteroarenes as part of pharmaceuticals and agrochemicals [21] we became interested in the direct borylation of heteroarenes. When we started our investigations mid of 2002 the catalytic borylation of heteroaromatic substrates has been known just for 2,6-dimethylpyridine [16b], 2,6-dichloropyridine [22] and 1-triisopropylsilyl-pyrrole as substrates [19]. Parallel to our work, Miyaura and coworkers have described the borylation of different heteroarenes using diboranes in the presence of $[Ir(COD)Cl]_2$ and 4,4′-di-*t*-butyl-2,2′-bipyridine at 80–100 °C to give the corresponding heteroarylboronates [20].

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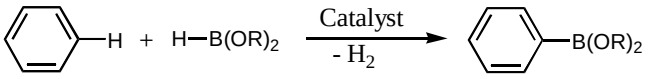



Scheme 1. Borylation of benzene.

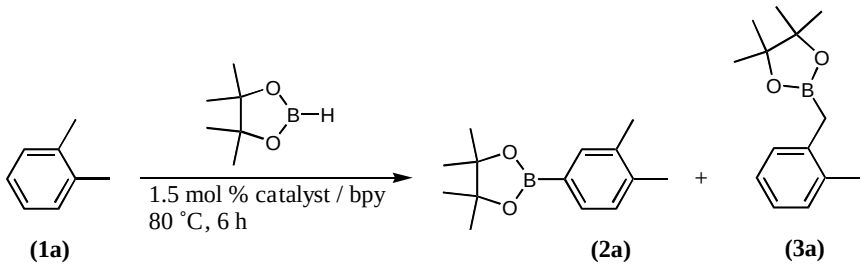
## 2. Results and discussion

In order to become familiar with the methodology we started our investigation on the influence of the metal precursors and additional P- and N-donor ligands using the reaction of *o*-xylene with pinacolborane at different reaction conditions (Scheme 2). As catalyst precursors, we chose 15 commercially available metal complexes selected from the transition metals Ir, Rh, Ru and Pd. Product yields were determined by GC analysis of the crude reaction mixtures. Selected results from approximately 100 experiments are summarized in Table 1. Among the various complexes $[Ir(COD)Cl]_2$ and Rh(COD)acac gave the best results at 80 °C.

In general, all catalysts containing TMEDA, sparteine, $PPh_3$, or $PCy_3$ were found to be ineffective in the model reaction. In agreement with previous literature selective borylation of $sp^2$ C–H bonds was observed in the presence of Ir complexes and 2,2′-bipyridine (bpy). No activation of the position 3 occurred, the 4-borylated isomer being the major product for steric reasons. In no case, double borylated products were observed. On the other hand, benzylic activation of *o*-xylene became the main reaction pathway for Rh-catalysts. Shimada et al. found a similar strong preference for benzylic functionalization of toluene by Rh-catalysts [18b]. Surprisingly, we found that $Cp^*RhCl_2$ resulted in a predominant arene activation reaction. Best yields of borylated products were obtained using Rh(COD)acac in the presence of 1 eq. of bpy giving 67% 4,4,5,5-tetramethyl-2-(*o*-tolylmethyl)-1,3,2-dioxaborolane (**3a**) and $[Ir(COD)Cl]_2$ in the presence of 8 eq. of bpy, resulting in 69% 4,4,5,5-tetramethyl-2-(3,4-dimethylphenyl)-1,3,2-dioxaborolane (**2a**).

The combination of ruthenium(II)acetylacetonate, dichloro(*p*-cymene)ruthenium(II) and dichloro(1,5-cyclooctadiene)ruthenium(II), respectively, with bpy did not catalyze the borylation reaction at all.

In analogy to the functionalization of *o*-xylene we attempted the coupling of pinacolborane and several heterocycles catalyzed by $[Ir(COD)Cl]_2$/2bpy at 80 °C (Table 2). Here, we studied initially the reaction of several *N*-heterocycles. Typically, 2 mmol pinacolborane were reacted with an excess of 60 mmol *N*-heteroarene in the presence of 0.015 mmol $[Ir(COD)Cl]_2$ (1.5 mol% Ir) and 0.03 mmol (1.5 mol%) bpy. While pyrrole gave the corresponding *o*-borylated product in 42% yield, *N*-methylpyrrole gave only 3% of the desired product. Indoles proved to be more reactive leading to the *o*-borylated products in



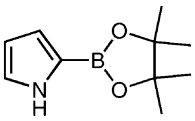
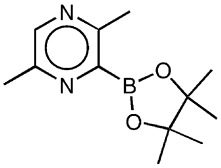
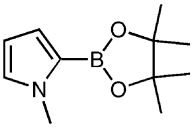
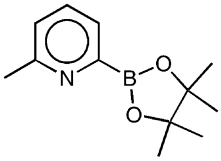
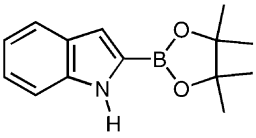
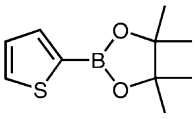
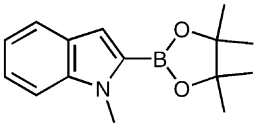
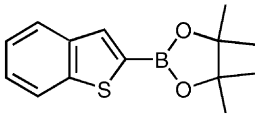
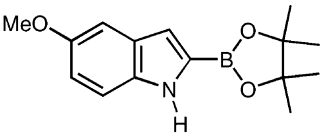
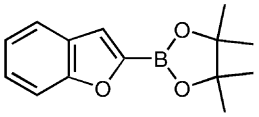

Scheme 2. Borylation of *o*-xylene.

Table 1
Influence of several catalysts and ligands on the borylation of *o*-xylene

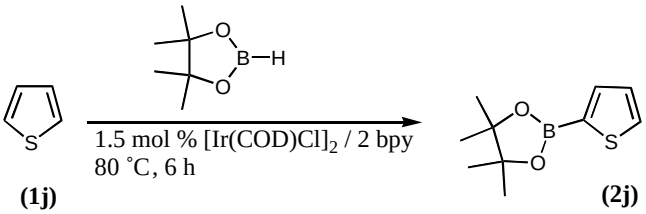
| Entry | Catalyst | Ligand | Conversion (%)[a] | Yield **2a** (%)[a] | Yield **3a** (%)[a] |
|---|---|---|---|---|---|
| 1 | $[Ir(COD)Cl]_2$ | 2bpy | 100 | 67 | 1 |
| 2 | $[Ir(COD)Cl]_2$ | $4PCy_3$ | 86 | 0 | 0 |
| 3 | $[Ir(COD)Cl]_2$ | $4PPh_3$ | 96 | 0 | 0 |
| 4 | $[Ir(COD)Cl]_2$ | 2sparteine | 100 | 3 | 1 |
| 5 | $[Ir(COD)Cl]_2$ | 2TMEDA | 55 | 6 | 0 |
| 6 | $[Ir(COD)Cl]_2$ | 8bpy | 100 | 69 | 0 |
| 7 | $[Cp^*RhCl_2]_2$ | 2bpy | 100 | 28 | 2 |
| 8 | $[Rh(COD)Cl]_2$ | 2bpy | 100 | 4 | 59 |
| 9 | Rh(COD)acac | 1bpy | 100 | 3 | 67 |
| 10 | $Rh_2(OAc)_4$ | 2bpy | 100 | 3 | 39 |
| 11 | [PindophosRh(COD)][$BF_4$][b] | – | 27 | 0 | 0 |
| 12 | [PindophosRh(COD)][$BF_4$][b] | 1bpy | 72 | 0 | 0 |
| 13 | $[Rh(COD)_2][BF_4]$ | – | 11 | 0 | 0 |
| 14 | $[Rh(COD)_2][BF_4]$ | 1bpy | 65 | 0 | 16 |

[a] GC yield and conversion based on pinacolborane.
[b] Pindophos: 2,3-*O*,*N*-bis(diphenylphosphino)-1-(4-indolyloxy)-2-hydroxy-3-isopropylamino propane.

Table 2
Borylation of different heterocycles

| Entry | Educt **1** | Product **2** | Yield **2b–o** (%)[a] | Entry | Educt **1** | Product **2** | Yield **2b–o** (%)[a] |
|---|---|---|---|---|---|---|---|
| 1 | **1b** |  | 42 | 6 | **1h** |  | 0 |
| 2 | **1c** |  | 3 | 7 | **1i** |  | 1 |
| 3 | **1d** |  | 89 | 8 | **1j** |  | 88 |
| 4 | **1e** |  | 45 | 9 | **1k** |  | 61 |
| 5 | **1f** |  | 53 | 10 | **1l** |  | 4 |

[a] GC yield based on pinacolborane.




**(1j)** **(2j)**

Scheme 3. Borylation of thiophene.

45–89% (Table 2, entries 3–5). Other *N*-heterocycles such as pyrazine or pyridine did not react to an appreciable amount. The reaction of pinacolborane with thiophene (Scheme 3) and benzothiophene resulted in 88 and 61% of the monoborylated products **2j** and **2k**, respectively (Table 2, entries 8 and 9). In all reactions, the borylation occurred at position 2 regioselectively (>95%). Only in case of *N*-methylindole, significant amounts of another isomer were observed. Here, besides the 2-borylated product (45%) the 3-substituted isomer is obtained in 36% yield.

## 3. Conclusions

We have shown for the first time that selective catalytic borylation of substituted indoles can be achieved. Previous catalytic borylation of other heteroarenes used more expensive diboranes as borylation reagent. Our results demonstrate that similar reactions can be performed with various heteroarenes using less expensive pinacolborane in the presence of a reduced catalyst amount. In general, the catalytic borylation using Ir catalysts proceeds with high selectivity for position 2 of electron-rich *N*- and *S*-heteroarenes.

## 4. Experimental

All experiments were carried out under an argon atmosphere. $^{1}H$ and $^{13}C$ NMR spectra were recorded in $CDCl_3$ solutions using a Bruker ARX 400 FT Spectrometer ($^{1}H$: 400.1 MHz, $^{13}C$: 100.6 MHz). Chemical shifts are given in ppm and refer to residual solvent as internal standard. Gas chromatography was performed on a Hewlett Packard HP6890 chromatograph with a HP5 column. The products were purified on silica gel 60, 230–400 mesh.

A flask containing 2 mmol pinacolborane, 1.5 mol% catalyst, and 1.5 mol% ligand was charged with 60 mmol arene or heteroarene. The solution was stirred at 80 °C for 6 h. The solvent was then removed under vacuum at room temperature and the residue was chromatographed over silica gel, eluting with $CH_2Cl_2$, to yield the analytically pure product.

*4.1. 4,4,5,5-Tetramethyl-2-(3,4-dimethylphenyl)-1,3,2-dioxaborolane (**2a**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.25 (s, 12H), 2.18 (s, 3H), 2.19 (s, 3H), 7.05–7.07 (d, $J = 7.3$ Hz, 1H), 7.46–7.48 (d, $J = 7.3$ Hz, 1H), 7.5 (s, 1H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 19.9, 20.5, 25.3, 84.0, 129.6, 132.9, 136.3, 136.4, 140.6. Anal. Calcd. for $C_{14}H_{21}BO_2$: C, 72.44; H, 9.12; B, 4.66; O, 13.79. Found: C, 72.65; H, 9.05; B, 4.49; O, 13.62.

*4.2. 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-pyrrole (**2b**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.35 (s, 12H), 6.33 (dt, $J = 2.4$, 3.4 Hz, 1H), 6.88 (ddd, $J = 1.2$, 2.4, 3.6 Hz, 1H), 7.03 (dt, $J = 1.4$, 2.6 Hz, 1H), 8.80 (br, 1H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 25.2, 84.0, 110.1, 120.4, 123.1; IR ($\nu$): 746, 1142, 1559, 3354 cm$^{-1}$. Anal. Calcd. for $C_{10}H_{16}BNO_2$: C, 62.22; H, 8.35; N, 7.26. Found: C, 62.01; H, 8.14; N, 7.04.

*4.3. 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-indole (**2d**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.38 (s, 12H), 7.11 (m, 2H), 7.26 (m, 1H), 7.39 (dd, $J = 0.8$, 8.3 Hz, 1H), 7.68 (dd, $J = 0.8$, 7.9 Hz, 1H), 8.58 (br, 1H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 25.4, 84.7, 111.8, 114.4, 120.3, 122.2, 124.2, 128.8, 138.7; IR ($\nu$): 706, 852, 1137, 1539, 3415 cm$^{-1}$. Anal. Calcd. for $C_{14}H_{18}BNO_2$: C, 69.17; H, 7.46; N, 5.76. Found: C, 69.24; H, 7.32; N, 5.71.

*4.4. 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-N-methylindole (**2e**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.35 (s, 12H), 3.95 (s, 3H), 7.07 (m, 1H), 7.14 (s, 1H), 7.25 (m, 1H), 7.33 (d, $J = 7.8$ Hz, 1H), 7.65 (d, $J = 7.9$ Hz, 1H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 25.3, 32.7, 84.2, 101.4, 110.2, 114.7, 119.8, 122.1, 123.7, 128.4, 140.6; IR ($\nu$): 689, 735, 1140, 1524 cm$^{-1}$. Anal. Calcd. for $C_{15}H_{20}BNO_2$: C, 70.06; H, 7.84; N, 5.45. Found: C, 69.85; H, 7.73; N, 5.66.

*4.5. 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-5-methoxyindole (**2f**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.37 (s, 12H), 3.86 (s, 3H), 6.93 (dd, $J = 2.4$, 8.9 Hz, 1H), 7.05 (d, $J = 1.2$ Hz, 1H), 7.10 (d, $J = 2.4$ Hz, 1H), 7.29 (d, $J = 8.9$ Hz, 1H), 8.52 (br, 1H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 25.4, 53.9, 84.5, 102.5, 112.4, 113.8, 115.3, 129.0, 134.1, 154.6; IR ($\nu$): 835, 1138, 1120, 1537, 3377 cm$^{-1}$. Anal. Calcd. for $C_{15}H_{20}BNO_3$: C, 65.96; H, 7.38; N, 5.13. Found: C, 65.90; H, 7.12; N, 5.30.

*4.6. 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-thiophene (**2j**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.39 (s, 12H), 7.23 (dd, $J = 3.6$, 4.8 Hz, 1H), 7.68 (dd, $J = 0.9$, 4.6 Hz, 1H), 7.70 (dd, $J = 0.9$, 3.4 Hz, 1H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 25.2, 84.5, 128.6, 132.8, 137.6; IR ($\nu$): 724, 856, 1142, 1521 cm$^{-1}$. Anal. Calcd. for $C_{10}H_{15}BO_2S$: C, 57.17; H, 7.20; S, 15.26. Found: C, 57.42; H, 6.96; S, 15.45.

*4.7. 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-benzothiophene (**2k**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.39 (s, 12H), 7.37 (m, 2H), 7.89 (m, 3H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 25.4, 84.9, 123.0, 124.5, 124.8, 125.7, 134.9, 140.9, 144.1; IR (KBr) $\nu$: 664, 752, 1137, 1523 cm$^{-1}$. Anal. Calcd. for $C_{14}H_{17}BO_2S$: C, 64.63; H, 6.59; S, 12.33. Found: C, 64.89; H, 6.69; S, 12.12.

*4.8. 4,4,5,5-Tetramethyl-2-(o-tolylmethyl)-1,3,2-dioxaborolane (**3a**)*

$^1$H NMR ($CDCl_3$, 25 °C, 400.1 MHz) $\delta$: 1.15 (s, 12H), 2.18 (s, 2H), 2.19 (s, 3H), 7.02 (m, 4H); $^{13}$C NMR ($CDCl_3$, 25 °C, 100.6 MHz) $\delta$: 20.5, 25.2, 83.8, 125.6, 126.3, 129.9, 130.2, 136.3, 137.9. Anal. Calcd. for $C_{14}H_{21}BO_2$: C, 72.44; H, 9.12; B, 4.66; O, 13.79. Found: C, 72.56; H, 9.10; B, 4.56; O, 13.65.

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