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Rhodium-catalyzed regioselective arylation of phenylazoles and related compounds with arylboron reagents via C–H bond cleavage

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ABSTRACT

The rhodium-catalyzed direct *ortho*-arylation reactions of phenylazoles using arylboron reagents such as tetraarylborates were examined. Ethyl chloroacetate and potassium fluoride were found to effectively act as a hydrogen acceptor and a promoter, respectively, to afford selective formation of the corresponding mono- or diarylated products with good yields. In addition, azobenzene as well as 2-phenylpyridine also underwent the direct arylation under similar conditions.

1. Introduction

Transition-metal-catalyzed cross-coupling of arylmetal reagents (metal = Mg, Zn, B, Sn, Si, etc.) with aryl halides are now recognized to be one of the most useful methods for constructing biaryls [1], whose skeletons are found in a wide range of important compounds including natural products and organic functional materials [2–4]. Particularly, the palladium-catalyzed coupling of non-toxic, readily available arylboron reagents with aryl halides (Suzuki-Miyaura coupling) is very often employed [1,5,6].

Obviously, the direct arylation of arenes with arylboron reagents, in which prehalogenation of the arene coupling partners is not required, may provide a straightforward and environmentally benign method for biaryl synthesis [7–14]. As an example for the direct reaction, we have reported the multiple phenylation of benzonitrile using sodium tetraphenylborate under rhodium catalysis (Scheme 1) [15,16]. In this reaction, benzophenone imine, generated initially through nucleophilic phenylation on the C–N triple bond of benzonitrile, is considered to undergo the subsequent coordination-assisted *ortho*-phenylation via C–H bond cleavage to form multiply phenylated products, as depicted in Scheme 2. According to this reaction scheme, however, 1 equivalent of benzophenone imine is consumed as a hydrogen acceptor for the C–H phenylation to form (diphenylmethyl)amine (Ph_2CHNH_2) as a byproduct. Actually, formation of a significant amount of the amine was observed in the reaction.

To suppress the undesired consumption of the imine intermediate, addition of ethyl α-chloroacetate as a hydrogen acceptor was examined. α-Chloroacetyl compounds have been employed as reoxidants in the palladium-catalyzed oxidative coupling reactions of organometallic reagents with alkenes [17,18]. As a model substrate, cyclohexanecarbonitrile was used, because the product mixture would be simpler than that with benzonitrile. Thus, the aliphatic nitrile was treated with sodium tetraphenylborate using $[RhCl(cod)]_2$ as a catalyst in *o*-xylene in the presence or absence of $ClCH_2CO_2Et$, and (biphenyl-2-yl) cyclohexyl ketone was obtained as a major product after hydrolysis with aq. HCl (Scheme 3) [19]. Expectedly, the addition of the hydrogen acceptor increased the yield of the diphenylated product from 14% to 53% without forming any amines. While efficiency of the reaction could not be further improved, the catalyst system using the hydrogen acceptor was found to be applicable to the direct arylation of other substrates such as phenylazoles and related compounds. The findings are described herein.

2. Results and discussion

In an initial attempt, 1-methyl-2-phenylimidazole (**1**) (0.5 mmol) was treated with sodium tetraphenylborate (**2a**) (0.5 mmol) in the presence of $[RhCl(cod)]_2$ (cod = 1,5-cyclooctadiene) (0.005 mmol) and $ClCH_2CO_2Et$ (0.5 mmol) in *o*-xylene at 120 °C for 6 h under nitrogen. As a result, the *ortho*-phenylated

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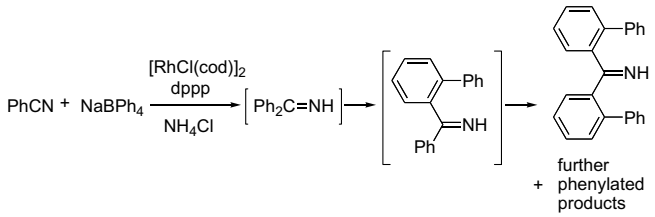
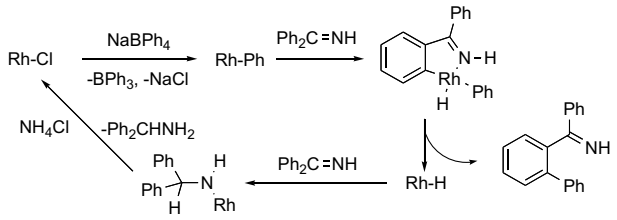
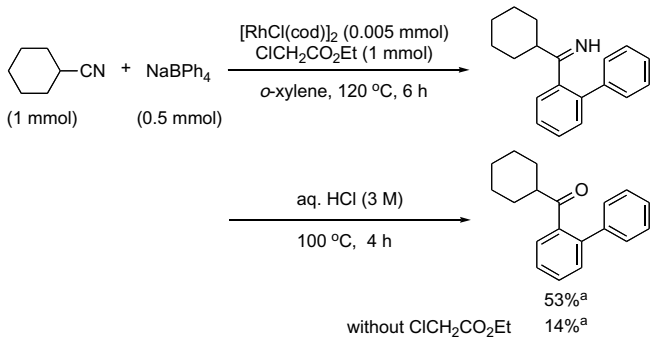


Scheme 1. Reaction of benzonitrile with sodium tetraphenylborate.

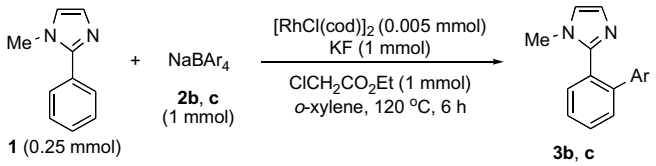


Scheme 2. Proposed mechanism for the C–H phenylation of benzophenone imine.



Scheme 3. Reaction of cyclohexanecarbonitrile with sodium tetraphenylborate. [a]Yield based on the amount of $NaBPh_4$ used.

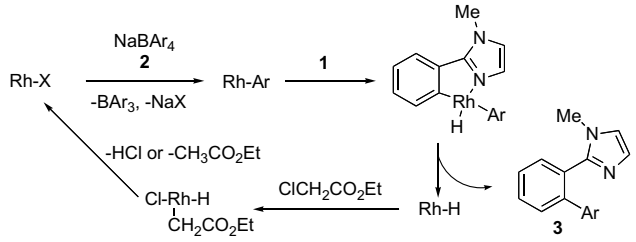
product, 2-(biphenyl-2-yl)-1-methylimidazole (**3a**), was formed in 16% yield (Entry 1 in Table 1). Addition of KF (0.5 mmol) signifi-



2	Product, Yield (%)[a]
2b: Ar = 4-$CH_3C_6H_4$	**3b**: Ar = 4-$CH_3C_6H_4$, 88
2c: Ar = 4-FC_6H_4	**3c**: Ar = 4-FC_6H_4, 21

[a] Isolated yield.

Scheme 4. Reaction of **1** with sodium tetraarylborates **2**.



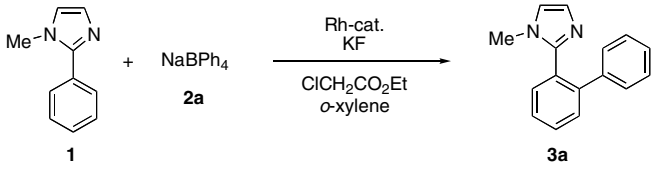
Scheme 5. Plausible mechanism for the C–H arylation of **1**.

cantly improved the yield to 46% (Entry 2). $[Rh(OMe)(cod)]_2$ showed comparable activity to that of $[RhCl(cod)]_2$, while Rh(acac)(cod) was less effective (Entries 3 and 4). At 140 °C, the yield was somewhat decreased (Entry 5). Finally, under the conditions using excess amounts of **2a** and the additives, **3a** was produced in 92% yield (Entry 6). The phenylation with phenylboronic acid (**4a**) in place of **2a** proceeded to some extent, albeit sluggishly (Entry 7).

Under the optimized conditions (Entry 6 in Table 1), sodium tetrakis(4-methylphenyl)borate (**2b**) reacted with **1** to give the corresponding *ortho*-arylated product **3b** in 88% isolated yield (Scheme 4). Sodium tetrakis(4-fluorophenyl)borate (**2c**) also underwent the reaction with **1**, although the yield of product **3c** was low.

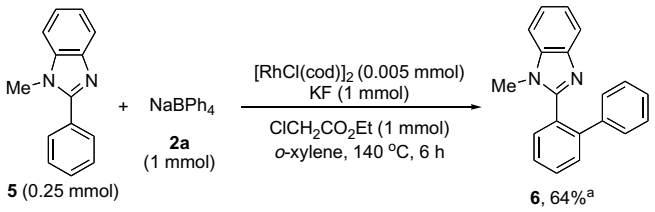
A plausible mechanism for the reaction of azole **1** with borate **2** is illustrated in Scheme 5. The reaction proceeds via C–H activation

Table 1
Reaction of 1-methyl-2-phenylimidazole (**1**) with sodium tetraphenylborate (**2a**)[a]

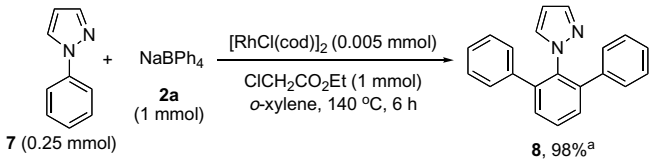


Entry	**1** (mmol)	**2a** (mmol)	Rh-cat. (mmol)	Temperature (°C)	Yield of **3a** (%)[b]
1[c]	0.5	0.5	$[RhCl(cod)]_2$ (0.005)	120	16
2	0.5	0.5	$[RhCl(cod)]_2$ (0.005)	120	46
3	0.5	0.5	$[Rh(OMe)(cod)]_2$ (0.005)	120	41
4	0.5	0.5	Rh(acac)(cod) (0.01)	120	23
5	0.5	0.5	$[RhCl(cod)]_2$ (0.005)	140	35
6[d]	0.25	1	$[RhCl(cod)]_2$ (0.005)	120	92 (92)
7[e]	0.25	1	$[RhCl(cod)]_2$ (0.005)	120	17

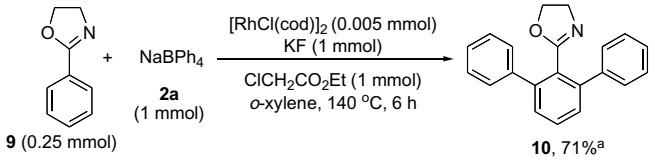
[a] Reaction conditions: KF (0.5 mmol), $ClCH_2CO_2Et$ (0.5 mmol) in *o*-xylene (5 mL) for 6 h under N_2.
[b] GC yield based on the amount of **1** used. Value in parentheses indicates isolated yield.
[c] Without KF.
[d] KF (1 mmol) and $ClCH_2CO_2Et$ (1 mmol) were used.
[e] $PhB(OH)_2$ **(4a)** (1 mmol) was used in place of **2a**.



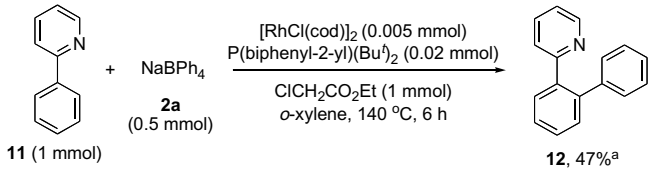
Scheme 6. Reaction of 1-methyl-2-phenylbenzimidazole (**5**) with **2a**. [a]Isolated yield.



Scheme 7. Reaction of 1-phenylpyrazole (**7**) with **2a**. [a]Isolated yield.



Scheme 8. Reaction of 2-phenyl-2-oxazoline (**9**) with **2a**. [a]Isolated yield.



Scheme 9. Reaction of 2-phenylpyridine (**11**) with **2a**. [a]GC yield.

at the 2′-position of **1** by an arylrhodium(I) intermediate, which is generated by transmetallation of a Rh(I)X species with **2**. The resulting diaryl(hydrido)rhodium species then undergoes reductive elimination to give an arylated product **3** and a Rh(I)H species. Oxidative addition of $ClCH_2CO_2Et$ to the latter and reductive elimination of HCl or CH_3CO_2Et may occur to regenerate Rh(I)X. It is cited that pinacolone [8] and tetrachloroethane solvents [14] were reported to act as hydrogen acceptors in relevant catalytic direct arylations. Use of TEMPO was also described very recently [13]. Although the significant promoting effect of KF was observed, the exact role of the additive is not definitive at the present stage.

Next, the phenylation of other phenylazoles with **2a** by using the catalyst system of $[RhCl(cod)]_2/ClCH_2CO_2Et/KF$ was examined. 1-Methyl-2-phenylbenzimidazole (**5**) underwent the reaction effectively to afford the product **6** in 64% yield (Scheme 6).

Sterically less demanding 1-phenylpyrazole (**7**) and 2-phenyl-2-oxazoline (**9**), compared with phenylimidazoles **1** and **5**, reacted with **2a** to give the corresponding diphenylated products, **8** and **10**, respectively, in good yields (Schemes 7 and 8). In these reactions, formation of monophenylated products could not be detected. In the case of **7**, the reaction took place efficiently without adding KF. It is cited that related mono- and diarylation reactions of these substrates with aryl halides or tosylates have been reported [20–23].

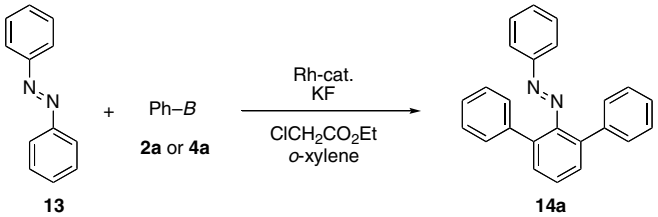
For the phenylation of 2-phenylpyridine (**11**), which is often examined as a substrate for C–H functionalizations [13,14, 21–23], addition of P(biphenyl-2-yl)(Bu^t)$_2$ as a ligand led to a better yield of product **12** (47%) (Scheme 9). The yield was reduced to 30% without using the ligand.

It was of considerable interest that phenylation of azobenzene (**13**) could also be conducted by the present method (Table 2), as no report on the transition-metal-catalyzed C–H arylation of **13** as well as **1** and **5** has to date appeared. Thus, treatment of **13** (0.25 mmol) using excess amounts of **2a**, $ClCH_2CO_2Et$, and KF in the presence of $[RhCl(cod)]_2$ gave a diphenylated product **14a** in 15% yield (Entry 1). Since a comparable result was obtained in the reaction of **13** with phenylboronic acid (**4a**) (Entry 2), **4a** was employed for further examinations. $[Rh(OMe)(cod)]_2$ was found to be more effective as a catalyst than $[RhCl(cod)]_2$ and Rh(acac)(cod) (Entries 2–4). The yield of **14a** was enhanced up to 50%, when the amount of KF was increased to 2 mmol (Entry 5).

Under the same conditions, the reactions of **13** with 4-methyl- (**4b**) and 4-chlorophenyl boronic acids (**4c**) also took place to afford the corresponding diarylated products **14b** and **14c**, respectively, in moderate yields (Scheme 10).

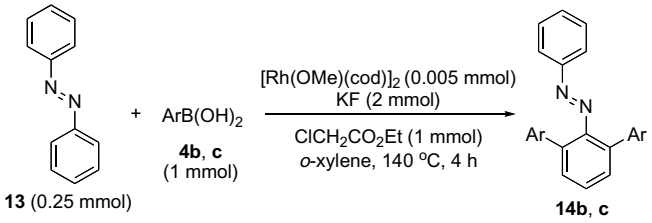
In summary, we have demonstrated that the rhodium-catalyzed direct arylation reactions of phenylazoles and related substrates including azobenzene and 2-phenylpyridine can be performed by

Table 2
Phenylation of azobenzene (**13**)[a]



Entry	Ph-*B*	Rh-cat. (mmol)	Time (h)	Yield of **14a** (%)[b]
1	$NaBPh_4$ (**2a**)	$[RhCl(cod)]_2$ (0.005)	7	15
2	$PhB(OH)_2$ (**4a**)	$[RhCl(cod)]_2$ (0.005)	4	19
3	$PhB(OH)_2$ (**4a**)	Rh(acac)(cod) (0.01)	4	20
4	$PhB(OH)_2$ (**4a**)	$[Rh(OMe)(cod)]_2$ (0.005)	4	44
5[c]	$PhB(OH)_2$ (**4a**)	$[Rh(OMe)(cod)]_2$ (0.005)	4	50 (44)

[a] Reaction conditions: **13** (0.25 mmol), **2a** or **4a** (1 mmol), KF (1 mmol), $ClCH_2CO_2Et$ (1 mmol) in *o*-xylene (5 mL) at 140 °C under N_2.
[b] GC yield based on the amount of **13** used. Value in parentheses indicates isolated yield.
[c] KF (2 mmol) was used.



4	Product, Yield (%)[a]
4b: Ar = 4-$CH_3C_6H_4$	**14b**: Ar = 4-$CH_3C_6H_4$, 40 (39)
4c: Ar = 4-ClC_6H_4	**14c**: Ar = 4-ClC_6H_4, 27 (22)

[a] GC yield. Value in parenthese indicates isolated yield.

Scheme 10. Reaction of **13** with arylboronic acids **2**.

using arylboron reagents. In these reactions, $ClCH_2CO_2Et$ acts as an effective acceptor of the eliminated hydrogen, and KF also promotes the coupling in a number of cases.

3. Experimental

3.1. General

Reactions were carried out in a 20 mL two-necked flask under N_2. $[Rh(OMe)(cod)]_2$ was prepared according to the published method [24]. ^1H and ^{13}C NMR spectra were recorded at 400 MHz and 100 MHz, respectively, for $CDCl_3$ solutions. GC analysis was carried out using a silicon OV-17 column (i.d. 2.6 mm × 1.5 m) or a CBP-1 capillary column (i.d. 0.25 mm × 25 m). GC–MS analysis was carried out using a CBP-1 capillary column (i.d. 0.25 mm × 25 m). Phenylimidazoles **1** and **5** were prepared according to the published method [25]. Other reagents were commercially available.

The following experimental procedures may be regarded as typical in methodology and scale.

3.2. Reaction of 1-methyl-2-phenylimidazole (**1**) with sodium tetraphenylborate (**2a**) (Entry 6 in Table 1)

In a flask was placed KF (1 mmol, 58 mg), which was then dried at 150 °C in vacuo for 1 h. Then, **1** (0.25 mmol, 40 mg), **2a** (1 mmol, 342 mg), $[RhCl(cod)]_2$ (0.005 mmol, 2.5 mg), $ClCH_2CO_2Et$ (1 mmol, 122 mg), dibenzyl (ca. 50 mg) as an internal standard, and *o*-xylene (5 mL) were added and the resulting mixture was stirred at 120 °C under N_2 for 6 h. After cooling, the reaction mixture was extracted with Et_2O and dried over sodium sulfate. Product **3a** (54 mg, 92%) was isolated by thin-layer chromatography on silica gel using hexane–ethyl acetate–NEt_3 (55:30:15) as eluent.

3.2.1. 2-(Biphenyl-2-yl)-1-methylimidazole (**3a**)

Oil: ^1H NMR (400 MHz, $CDCl_3$): δ 2.84 (s, 3H), 6.70 (d, J = 1.1 Hz, 1H), 7.11 (d, J = 1.1 Hz, 1H), 7.15–7.18 (m, 2H), 7.24-7.30 (m, 3H), 7.42–7.62 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 32.8, 120.5, 127.0, 127.5, 128.3, 128.4, 128.6, 129.5, 129.6, 129.6, 131.9, 140.6, 141.6, 147.7; HRMS (CI) m/z ($(M+H)^+$) Calc. for $C_{16}H_{15}N_2$: 235.1235. Found: 235.1241.

3.2.2. 2-(4′-Methylbiphenyl-2-yl)-1-methylimidazole (**3b**)

Oil, ^1H NMR (400 MHz, $CDCl_3$): δ 2.32 (s, 3H), 2.85 (s, 3H), 6.71 (d, J = 1.1 Hz, 1H), 7.03-7.09 (m, 4H) 7.11 (d, J = 1.1 Hz, 1H), 7.39–7.60 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.0, 32.8, 120.4, 127.2, 128.4, 128.5, 129.1, 129.4, 129.5, 129.6, 131.8, 136.8, 137.7, 141.6, 147.9; HRMS (CI) m/z ($(M+H)^+$) Calc. for $C_{17}H_{17}N_2$: 249.1392. Found: 249.1394.

3.2.3. 2-(4′-Fluorobiphenyl-2-yl)-1-methylimidazole (**3c**)

Oil, ^1H NMR (400 MHz, $CDCl_3$): δ 2.89 (s, 3H), 6.73 (d, J = 1.5 Hz, 1H), 6.94–6.99 (m, 2H), 7.10–7.15 (m, 3H), 7.42–7.48 (m, 2H), 7.51–7.55 (m, 1H), 7.58–7.60 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 32.8, 115.3 (d, J = 21.4 Hz) 120.5, 127.6, 128.5, 129.5, 129.5, 129.7, 130.2 (d, J = 7.7 Hz), 131.9, 136.5 (d, J = 3.8 Hz), 140.6, 147.5, 162.1 (d, J = 247.2 Hz); HRMS (CI) m/z ($(M+H)^+$) Calc. for $C_{16}H_{14}FN_2$: 253.1141. Found: 253.1144.

3.2.4. 2-(Biphenyl-2-yl)-1-methylbenzimidazole (**6**)

M.p. 137–138 °C; ^1H NMR (400 MHz, $CDCl_3$): δ 2.99 (m, 3H), 7.13–7.31 (m, 8H), 7.48–7.53 (m, 1H), 7.56–7.64 (m, 2H), 7.73–7.75 (m, 1H), 7.83–7.85 (m, 1H); δ ^{13}C NMR (100 MHz, $CDCl_3$): δ 30.2 109.4, 119.8, 122.1, 122.4, 127.3, 127.6, 128.5, 128.6, 129.0, 129.8, 130.3, 132.1, 135.5, 140.2, 141.6, 143.1, 153.9; HRMS (CI) m/z ($(M+H)^+$) Calc. for $C_{20}H_{17}N_2$: 285.1392. Found: 285.1385.

3.2.5. 1-(2,6-Diphenylphenyl)pyrazole (**8**)

M.p. 169–170 °C; ^1H NMR (400 MHz, $CDCl_3$): δ 6.05 (dd, J = 1.8, 2.4 Hz, 1H), 7.07 (dd, J = 2.4, 0.5 Hz, 1H), 7.11–7.13 (m, 4H), 7.22–7.25 (m, 6H), 7.37 (dd, J = 1.8, 0.5, 1H), 4.49–7.51 (m, 2H), 7.55-7.59 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 106.0, 127.2, 128.0, 128.2, 129.0, 130.1, 132.4, 136.5, 138.7, 139.3, 140.4; HRMS (CI) m/z ($(M+H)^+$) Calc. for $C_{21}H_{17}N_2$: 297.1392. Found: 297.1400.

3.2.6. 2-(2,6-Diphenylphenyl)-2-oxazoline (**10**)

M.p. 134–135 °C (lit. [20] m.p. 140.6–141.2); ^1H NMR (400 MHz, $CDCl_3$): δ 3.58 (t, J = 9.5 Hz, 2H), 3.89 (t, J = 9.5 Hz, 2H), 7.3 (m, 13H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 54.8, 67.3, 127.3, 128.0 (overlapped), 128.6, 128.8, 129.6, 140.8, 142.3, 164.3; HRMS (CI) m/z ($(M+H)^+$) Calc. for $C_{21}H_{18}NO$: 300.1388. Found: 300.1393.

3.3. Reaction of 2-phenylpyridine (**11**) with sodium tetraphenylborate (**2a**) (Scheme 9)

A mixture of **11** (1 mmol, 155 mg), **2a** (0.5 mmol, 171 mg), $[RhCl(cod)]_2$ (0.005 mmol, 2.5 mg), P(biphenyl-2-yl)(Bu^t)$_2$ (0.02 mmol, 6.0 mg), $ClCH_2CO_2Et$ (1 mmol, 122 mg), and dibenzyl (ca. 50 mg) as an internal standard was stirred in *o*-xylene (5 mL) at 140 °C under N_2 for 6 h. After cooling, the reaction mixture was extracted with Et_2O and dried over sodium sulfate. Product **12** (40 mg, 35%) was isolated by column chromatography on silica gel using hexane–ethyl acetate (98:2) as eluent.

3.3.1. 2-(Bipheny-2-yl)pyridine (**12**)

M.p. 84–85 °C (lit. [26] m.p. 86.5–87.5); ^1H NMR (400 MHz, $CDCl_3$): δ 6.88 (dt, J = 7.7, 1.1 Hz, 1H), 7.09 (ddd, J = 7.7, 4.8, 1.1 Hz, 1H), 7.13–7.18 (m, 2H), 7.21–7.25 (m, 3H), 7.37 (td, J = 7.7, 1.8 Hz, 1H), 7.42–7.48 (m, 3H), 7.68–7.72 (m, 1H), 8.63 (ddd, J = 4.8, 1.8, 1.1, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 121.3, 125.4, 126.7, 127.6, 128.0, 128.5, 129.6, 129.7, 130.4, 135.2, 139.5, 140.6, 141.3, 149.4, 159.2; MS (EI) m/z 231 (M^+).

3.4. Reaction of azobenzene (**13**) with phenylboronic acid (**4a**) (Entry 5 in Table 2)

In a flask was placed KF (2 mmol, 116 mg), which was then dried at 150 °C in vacuo for 1 h. Then, **13** (0.25 mmol, 46 mg), **4a** (1 mmol, 122 mg), $[Rh(OMe)(cod)]_2$ (0.005 mmol, 2.4 mg), $ClCH_2CO_2Et$ (1 mmol, 122 mg), dibenzyl (ca. 50 mg) as an internal standard, and *o*-xylene (5 mL) were added and the resulting mixture was stirred at 140 °C under N_2 for 4 h. After cooling, the reaction mixture was extracted with Et_2O and dried over sodium sulfate. Product **14a** (37 mg, 44%) was isolated by thin-layer chromatography on silica gel using hexane–ethyl acetate–NEt_3 (94.5:5:0.5) as eluent.

*3.4.1. 2,6-Diphenylazobenzene (**14a**) [27]*

Oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23–7.36 (m, 15H), 7.47–7.48 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 122.2, 126.6, 127.8, 127.9, 128.8, 130.3, 130.4, 130.8, 134.9, 139.5, 150.6, 152.5; HRMS (EI) m/z (M^+) Calc. for $C_{24}H_{18}N_2$: 334.1470. Found: 334.1472.

*3.4.2. 2,6-Bis(4-methylphenyl)azobenzene (**14b**)*

M.p. 108–109 °C, ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.33 (s, 6H), 7.09–7.17 (m, 8H), 7.32-7.37 (m, 5H), 7.4 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.1, 122.3, 127.8, 128.5, 128.8, 130.2, 130.2, 130.7, 134.6, 136.2, 136.5, 150.8, 152.6; HRMS (EI) m/z (M^+) Calc. for $C_{26}H_{22}N_2$: 362.1783. Found: 362.1779.

*3.4.3. 2,6-Bis(4-chlorophenyl)azobenzene (**14c**)*

M.p. 111–112 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18–7.49 (m, 16H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 122.3, 128.0, 128.1, 129.0, 130.6, 131.3, 131.5, 132.8, 133.9, 137.9, 150.3, 152.3; HRMS (EI) m/z (M^+) Calc. for $C_{24}H_{16}Cl_2N_2$: 402.0691. Found: 402.0674.

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