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Copper(II) Acetate-Mediated Cross-Coupling of Phenylboronic Acids with Aryloximes: Synthesis of O-Aryloximes

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Copper(II) Acetate–Mediated Cross-Coupling of Phenylboronic Acids with Aryloximes: Synthesis of O-Aryloximes

Xing-Hua Feng, Guo-Zhen Zhang, Cheng-Qun Chen, Ming-Yu Yang, Xiao-Yun Xu, and Guo-Sheng Huang
State Key Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou, China

Abstract: A novel method for the synthesis of O-aryloxime is described. It consisted of the coupling reaction between phenylboronic acids and aryloximes in the presence of catalytic quantities of copper(II) acetate. This reaction takes place in the presence of pyridine under a mild condition (DCE, Rt, 24–36 h) with moderate yield. It represents a good alternative to known methods for O-aryloxime synthesis.

Keywords: Aryloxime, copper(II) acetate, O-arylation, O-aryloxime, phenylboronic acids

During the past few years, much attention has been devoted to the synthesis of O-aryloximes because of their potential applications in medicinal and bioorganic chemistry.[1] Known methods for the synthesis of O-aryloxime ethers include the reaction of the sodium salt of oxime with fluorobenzene derivatives,[2] aldehydes/ketones with o-phenylhydroxylamine,[3] or oximes with aryl nitrates/diazonium salts.[4] However, these procedures lack generality, and the reaction often reacted at harsh conditions. Recently, De and coworkers investigated a method for synthesis of

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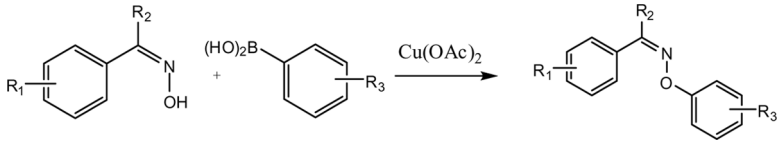

Scheme 1. Cross-coupling of aryloximes with phenylboronic acids.

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O-aryloximes using CuI-mediated cross-coupling of aryl halides with oximes.[5] However, in most of the cases, only iodobenzene gave a good yield.

Previous work by Chan,[6] Cundy,[7] and Evans[8] and their coworkers has revealed the efficacy of copper(II) acetate in the mediation of the cross-coupling of aryl boronic acids and phenols or amines to give biaryl ethers[9] and aryl alkylamines[10] or the like.[11,12] Inspired by these works, we developed a similar procedure for the synthesis of O-aryloximes, which consisted of the coupling reaction between commercially available arylboronic acids derivatives and aryloximes. Herein we report the success of this approach as shown in Scheme 1.

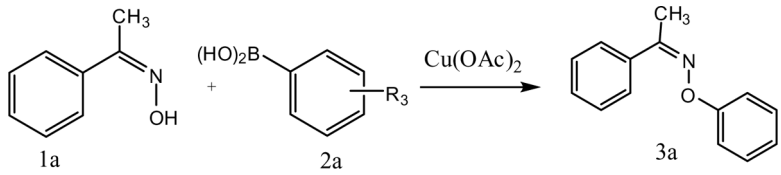
Our initial study began with the reaction of 2 equiv. of phenylboronic acids, 1 equiv. of acetophenone oxime (0.25 mmol), and 3 equiv. of Et_3N with 1 equiv. of $Cu(OAc)_2$ as the catalyst in CH_2Cl_2 at room temperature under atmosphere for 24–48 h. The desired product **3a** was isolated in a 35% yield (Table 1, entry 1). The effect of the different base on the reaction was examined. It could be seen from Table 1 that pyridine gave the best result, superior to dimethylaminopyridine (DMAP), Cs_2CO_3, and Et_3N (entries 1–4). This revealed that pyridine was by far the best base for the reaction.[11a]

According to our investigation, the optimal ratio of **1a** and **2a** is 1/2.5 (entry 8, Table 1), which gives the highest yield. Reducing the ratio of **2a** and **1a** gives less yield (table 1, entry 7), but greater quantities of phenylboronic acid has little further benefit on the reaction (entry 9, Table 1).

Further optimization of the reaction conditions revealed that 1,2-dichloroethane (DCE) was more effective solvent than dichloromethane (DCM). Furthermore, we found that a catalytic amount (20% mmol) of $Cu(OAc)_2$ is adequate for the reaction, and increasing this amount does not improve the reaction yield.

Under the optimized reaction conditions previously, the breadth and scope for the reaction were investigated. As shown in Table 2, compounds **1a**, **1b**, as well as 4-methoxybenzaldehyde oxime (**1c**) benzo[d][1,3]dioxole-5-carbaldehyde oxime (**1d**), produced the corresponding oxime ethers in good yields when react with phenylboronic acid (Table 2, entries 1, 6, 9, 11, 12). Compared to aryloxime, acetophenone oxime (**1a**) and 1-(4-methoxyphenyl)ethanone oxime (**1b**) gave good yields. When

Table 1. Optimization of the base for the cross-coupling of acetophenone oxime **1a** with phenylboronic acid **2a**



1a 2a 3a

Entry[a]	Catalyst	Base	Base equiv.	**1a**/**2a**	Yield (%)[b]
1	$Cu(OAc)_2$	Et_3N	3	1/2	35
2	$Cu(OAc)_2$	K_2CO_3	3	1/2	Trace
3	$Cu(OAc)_2$	Cs_2CO_3	3	1/2	30
4	$Cu(OAc)_2$	DMAP	3	1/2	Trace
5	$Cu(OAc)_2$	Pyridine	1	1/2	43
6	$Cu(OAc)_2$	Pyridine	3	1/2	63
7	$Cu(OAc)_2$	Pyridine	5	1/2	62
8	$Cu(OAc)_2$	Pyridine	3	1/2.5	76
9	$Cu(OAc)_2$	Pyridine	3	1/3	75

[a]Reaction conditions: 0.25 mmol acetophenone oxime, 150 mg of 4-Å molecular sieves (freshly activated), 1 equiv. $Cu(OAc)_2$, and the base in CH_2Cl_2, at room temperature for 24–36 h under an air atmosphere.

[b]Isolated yield of **3a**.

phenylboronic acid or p-tolylboronic acids are used, the corresponding coupling products are obtained in good yields (Table 2, entries 1, 4, 7). However, when p-fluorophenylboronic acid is employed, the yield is decreased notably (Table 2, entries 4, 10).[10e] In general, the reaction is affected by steric hindrance of the reaction center (Table 2, entry 5). Aryloximes with electron-withdrawing groups or electron-donating groups undergo the reaction smoothly. In all cases, the boronic acid was consumed during the reaction, and a small quantity of diphenyl ether was observed as an unwanted side product.[13] To investigate the reasons for the formation of diphenyl ether, a control experiment was carried out under the same conditions with no aryloximes. Interestingly, no diphenyl ether was observed under this condition. It seems that the aryloximes played a key role in the formation of diphenyl ethers.

In summary, we have developed a novel and mild methodology for the synthesis of O-aryloxime. The reaction of phenylboronic acids with aryloxime in the presence of catalytic quantities of copper(II) acetate affords moderate to good yields of O-aryloximes. It represents a good alternative to known methods for O-aryloxime synthesis.

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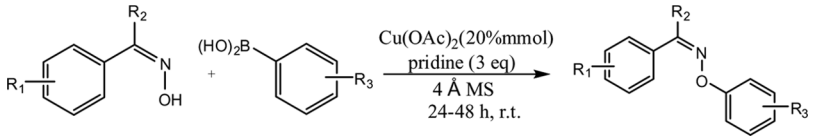
Table 2. Synthesis of O-aryloximes[a]



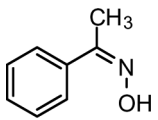
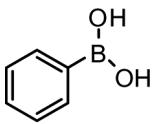
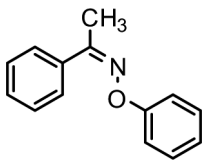
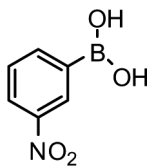
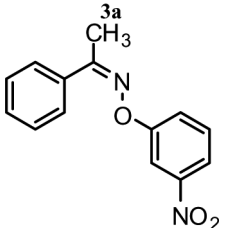
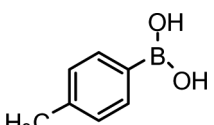
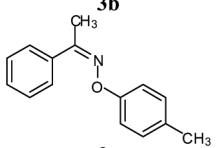
Entry	Aryloxime	Phenylboronic acids	Product	Time (h)	Yield[b] (%)
1	 **1a**	 **2a**	 **3a**	24	81
2	**1a**	 **2b**	 **3b**	24	52
3	**1a**	 **2c**	 **3c**	24	79

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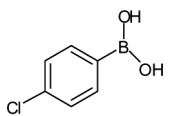
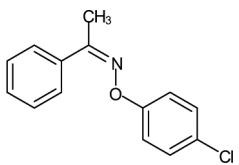
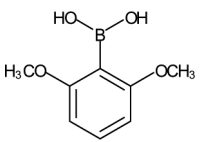
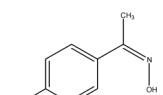
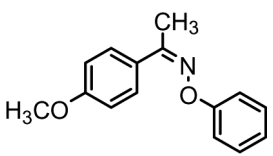
Table 2. Continued

Entry	Aryloxime	Phenylboronic acids	Product	Time (h)	Yield[b] (%)
4	**1a**	 **2d**	 **3d**	24	34
5	**1a**	 **2f**	**3e**	2d	NR[c]
6	 **1b**	**2a**	 **3f**	24	67

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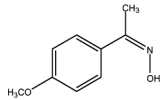
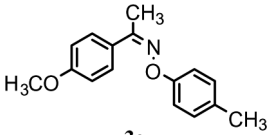
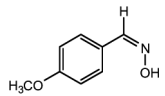
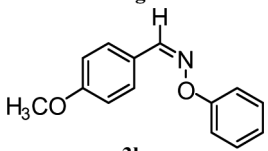
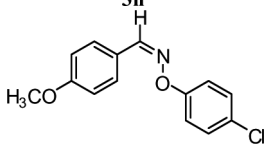
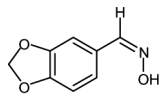
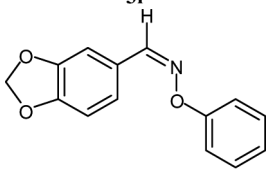
7	 **1b**	**2c**	 **3g**	24	73
8	 **1c**	**2a**	 **3h**	24	48
9	**1c**	**2d**	 **3i**	36	41
10	 **1d**	**2a**	 **3j**	48	56

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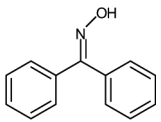
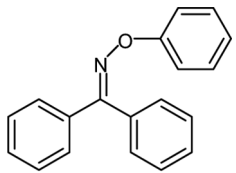
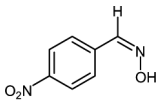
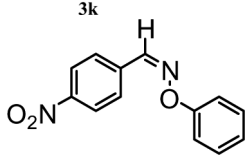
Table 2. Continued

Entry	Aryloxime	Phenylboronic acids	Product	Time (h)	Yield[b] (%)
11	 **1e**	**2a**	 **3k**	48	45
12	 **1f**	**2a**	 **3l**	48	42

[a]0. 25 mmol acetophenone oxime, 0.625 mmol phenylboronic acid, 150 mg of 4-Å molecular sieves (freshly activated), 0.05 mmol anhydrous $Cu(OAc)_2$, and 3 equiv. anhydrous pyridine in DCE (2 mL) at room temperature for 24–36 h under an air atmosphere.
[b]Isolated yields.
[c]No reaction.

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REPRESENTATIVE PRODUCTS

Acetophenone O-Phenyl Oxime (3a): General Procedure for the Preparation of O-Aryloxime

A mixture of acetophenone oxime (34 mg, 0.25 mmol), phenylboronic acid (76 mg, 0.625 mmol), $Cu(OAc)_2$ (9 mg, 0.05 mmol), freshly actived 4-Å molecular sieves (MS) (150 mg), and anhydrous pyridine (0.75 mmol) was run in DCE (2 mL) under atmosphere at room temperature. During this time, the color changed from blue to dark green. When the reaction was considered complete, the mixture was extracted with EtOAc. The combined organic extracts were washed with water and saturated brine. The organic layers were dried over $MgSO_4$ and filtered. Solvents were evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford acetophenone O-phenyl oxime (**3a**) as colorless oil (42 mg, 81%).

IR (KBr): 1592, 1489, 1216, 921, 755, 691 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 7.79–7.76 (m, 2H), 7.42–7.27 (m, 7H), 7.03 (t, 2H), 2.45 (s, 3H, $-CH_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 159.5, 157.7, 135.9, 129.7, 129.2, 128.5, 126.4, 122.1, 114.7, 13.3; MS (EI): $m/z = 211$ [M^+], 169, 118 (fission of the N-O bond), 77, 51. Anal. calcd. for $C_{14}H_{13}NO$: C, 79.59; H, 6.20. Found: C, 79.36; H, 6.18.

Acetophenone O-3-Nitrophenyl Oxime (3b)

Yellow oil. IR (KBr): 1528, 1349, 1224 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 8.19–8.17 (1H), 7.91–7.88 (1H), 7.80–7.77 (m, 2H), 7.60–7.578 (ddd, 1H), 7.50–7.45 (m, 4H), 2.49 (s, 3H, $-CH_3$). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 159.9, 159.4, 149.0, 135.1, 130.2, 129.7, 128.6, 126.6, 120.9, 116.8, 109.9, 13.6. MS (EI): $m/z = 256$ [M^+], 118 (fission of the N-O bond), 221, 77. Anal. calcd. for $C_{14}H_{12}N_2O_3$: C, 65.62; H, 4.72; N, 10.93. Found: C, 65.56; H, 4.86; N, 10.69.

Acetophenone O-4-Tolyl Oxime (3c)

Colorless oil. IR (KBr): 2924, 1503, 1216, 911, 734 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 7.79–7.73 (m, 3H), 7.40–7.38 (m, 3H), 7.21–7.10 (m, 4H) 2.401 (s, 3H, $-CH_3$), 2.31 (s, 3H, $-CH_3$). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 157.5, 157.3, 136.0, 131.4, 129.6, 129.5, 128.4, 126.4, 114.6, 20.6, 13.3. MS (EI): $m/z = 225$ [M^+], 118 (fission of the N-O bond), 77, 51. Anal. calcd. for $C_{15}H_{15}NO$: C, 79.97; H, 6.71; N, 6.22. Found: C, 79.99; H, 6.54; N, 6.52.

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Acetophenone O-4-Chlorophenyl Oxime (3d)

White solid. Mp 53–56°C. IR (KBr): 1483, 1220, 918, 823, 759, 689 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 7.78–7.74 (m, 2H), 7.44–7.41 (m, 3H), 7.30–7.21 (m, 4H), 2.44 (s, 3H, -CH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 158.2, 158.1, 135.6, 129.8, 129.1, 128.5, 126.8, 126.4, 115.9, 13.4. MS (EI): $m/z = 245$ [M^+], 118 (fission of the N-O bond), 77, 51. Anal. calcd. for $C_{14}H_{12}ClNO$: C, 68.44; H, 4.92; Cl, 14.43; N, 5.70, Found: C, 68.76; H, 4.53; Cl, 14.58; N, 5.98.

1-(4-Methoxyphenyl)ethanone O-Phenyl Oxime (3f)

Colorless solid. Mp 32–34°C. IR (KBr): 1592, 1487, 1252, 1217, 916, 753 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 7.73 (2H), 7.35–7.24 (m, 4H), 7.01 (1H), 6.92 (2H), 3.83 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 160.9, 159.6, 157.2, 129.2, 128.4, 127.8, 121.9, 114.7, 113.8, 55.3, 13.1. MS (EI): $m/z = 241$[M^+], 148 (fission of the N-O bond), 77. Anal. calcd. for $C_{15}H_{15}NO_2$: C, 74.67; H, 6.27; N, 5.81. Found: C, 74.28; 6.05; N, 5.46.

1-(4-Methoxyphenyl)ethanone O-p-Tolyl Oxime (3g)

IR (KBr): 1593, 1488, 1253, 1217, 916, 752 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 7.74–7.71 (m, 2H), 7.26–7.10 (m, 4H), 6.94–6.91 (m, 2H), 3.84 (s, 3H, -OCH_3), 2.40 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 160.9, 159.6, 157.2, 129.2, 128.4, 127.8, 121.9, 114.9, 113.8, 55.3, 20.6, 13.2. MS (EI): $m/z = 255$ [M^+], 148 (fission of the N-O bond), 77. Anal. calcd. for $C_{16}H_{17}NO_2$: C, 75.27; H, 6.71; N, 5.49. Found: C, 75.05, H, 6.24; N, 5.67.

4-Methoxybenzaldehyde O-Phenyl Oxime (3h)

Colorless oil. IR (KBr): 2933, 1604, 1593, 1512, 1487, 1255, 1219, 931, 752, 691 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 8.36 (s, 1H), 7.68–7.65 (m, 2H), 7.34–7.25 (m, 4H), 7.09–7.02 (m, 1H), 6.96–6.93 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 161.4, 159.4, 151.1, 129.2, 129.1, 123.9, 122.0, 114.3, 114.2, 55.3. MS (EI): $m/z = 227$ [M^+], 169, 134 (fission of the N-O bond), 77, 51. Anal. calcd. for $C_{14}H_{13}NO_2$: C, 73.99; H, 5.77; N, 6.16, Found: C, 73.64; H, 5.89; N, 6.27.

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4-Methoxybenzaldehyde O-4-Chlorophenyl Oxime (3i)

Waxy solid. Mp 46–48 °C. IR (KBr): 1605, 1483, 1258, 1029, 827 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 8.34 (s, 1H), 7.66–7.64 (d, $J = 8.7$ Hz, 2H), 7.269–7.263 (d, $J = 8.7$ Hz, 2H), 7.19–7.17 (d, $J = 9.6$ Hz, 2H), 6.96–6.93 (d, $J = 9.0$ Hz, 2H), 3.85 (s, 3H, OCH_3). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 161.6, 158.0, 151.6, 129.2, 129.1, 126.7, 123.6, 115.6, 114.3, 55.3. MS (EI): $m/z = 261[M^+]$, 134 (fission of the N-O bond), 77. Anal. calcd. for $C_{14}H_{12}ClNO_2$: C, 64.25; H, 4.62; Cl, 13.55; N, 5.35. Found: C, 64.68; H, 4.25; Cl, 13.67; N, 5.67.

Benzo[d][1,3]dioxole-5-carbaldehyde O-Phenyl Oxime (3j)

Colorless oil. IR (neat): 2918, 1592, 1487, 1255, 1217, 1039, 936, 753 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 8.32 (s, 1H), 7.37–7.32 (m, 3H), 7.27–7.24 (m, 2H), 7.10–7.02 (m, 2H), 6.86–6.83 (m, 1H), 6.03 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 159.3, 151.1, 149.8, 148.2, 129.2, 125.6, 123.8, 122.1, 114.3, 108.3, 105.9, 101.5. MS (EI): $m/z = 241$ $[M^+]$, 148 (fission of the N-O bond), 121, 94. Anal. calcd. for $C_{14}H_{11}NO_3$: C, 69.70; H, 4.60; N, 5.81. Found: C, 69.48; H, 4.84; N, 5.68.

Benzophenone O-Phenyl Oxime (3k)

Colorless oil. IR (KBr): 3058, 1592, 1487, 1214, 929, 753, 694 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 7.61–7.58 (m, 2H), 7.47–7.22 (m, 12H), 7.01 (t, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 159.9, 159.5, 135.9, 132.8, 129.9, 129.3, 129.2, 128.4, 128.3, 128.0, 122.2, 114.8. MS (EI): $m/z = 273[M^+]$ 180 (fission of the N-O bond), 77, 65, 51, 39. Anal. calcd. for $C_{19}H_{15}NO$: C, 83.49; H, 5.53; N, 5.12. Found: C, 83.35; H, 5.38; N, 5.16.

4-Nitrobenzaldehyde O-Phenyl Oxime (3l)

Light yellow solid. IR (KBr): 1583, 1517, 1485, 1343, 1215, 944, 849, 152, 689 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 8.48 (s, 1H), 8.30–8.28 (d, $J = 8.7$ HZ), 7.90–7.88 (d, $J = 8.7$ Hz), 7.39–7.34 (t, 2H), 7.28–7.26 (d, $J = 8.1$ Hz, 2H), 7.12–7.10 (t, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 158.9, 149.2, 137.5, 129.5, 128.2, 124.1, 123.0, 114.6, 109.7. MS (EI): $m/z = 242$ $[M^+]$, 149 (fission of the N-O bond), 94, 76, 65, 50, 39. Anal. calcd. for $C_{13}H_{10}N_2O_3$: C, 64.46; H, 4.16; N, 11.56. Found: C, 64.40; H, 4.24; N, 11.41.

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