# An Efficient Method for the Synthesis of 3-Aryl-4,7-phenanthroline Derivatives Catalyzed by Iodine

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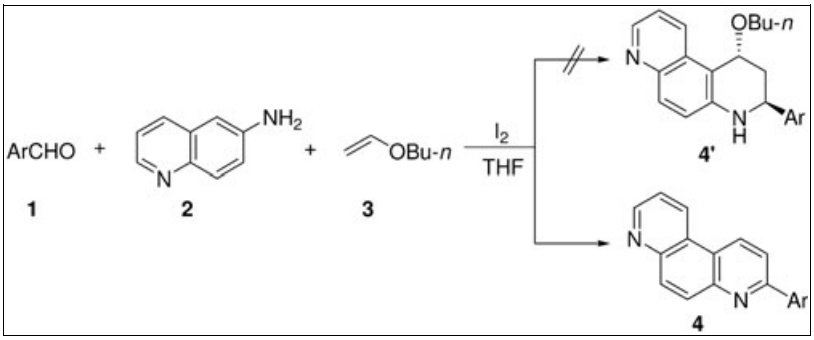


A mild and efficient method for the synthesis of 3-aryl-4,7-phenanthroline derivatives *via* three-component reaction of aromatic aldehyde, quinolin-6-amine, and *n*-butylvinyl ether is described using iodine as catalyst. The features of this procedure are mild reaction conditions, good to high yields, and operational simplicity.

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## INTRODUCTION

Multicomponent reactions (MCRs) can be distinguished from classical, sequential two-component synthetic processes in which three or more chemical starting materials are used for product formation. Up to seven starting components have been used, and often MCRs have been shown to produce higher product yields than classical chemistry [1]. They provide a powerful tool toward the one-pot synthesis of diverse and complex compounds as well as small and drug-like heterocycles [2]. Owing to their convergence and productivity, the MCRs have attracted considerable attention from the point of view of organic synthetic chemistry [3].

Phenanthroline and its derivatives are well-known compounds for their metallic complexes. The latter possess remarkable physiological and pharmacological activities. These activities include anticancer (lanthanum(III)) [4], antiinflammatory (copper(II)) [5], antitumor (Ru(II)) [6], antimicrobial (copper(II)) [7], and antibacterial activity (zinc (II)) [8]. In addition, it was reported that phenanthroline derivatives also had commendable antitumor activity [9]; therefore, the synthesis of phenanthroline received a considerable interest in recent years [10].

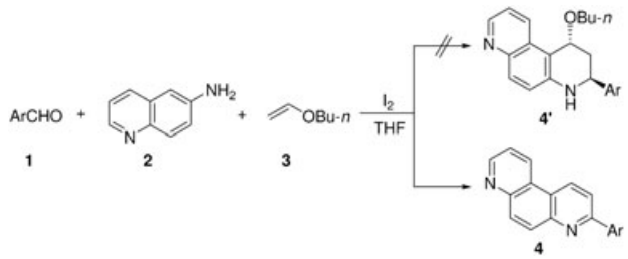
In view of the importance of phenanthroline derivatives and as a continuation of our research devoted to the development of new methods for the preparation of heterocycles *via* MCRs (catalyzed by iodine) [11], herein, we would like to synthesize 3-aryl-4,7-phenanthroline derivatives by a reaction of aromatic aldehyde, quinolin-6-amine, and *n*-butylvinyl ether in tetrahydrofuran (THF).

## RESULTS AND DISCUSSION

Treatment of aromatic aldehyde **1**, quinolin-6-amine **2**, and *n*-butylvinyl ether **3** in THF in the presence of 5 mol % iodine at reflux condition did not afford the desired *trans*-1-butoxy-1,2,3,4-tetrahydro-4,7-phenanthroline derivatives **4′**. To our surprise, the butoxy group was not found in the $^1$H-NMR, with aromatized 3-aryl-4,7-phenanthroline being obtained in good yields (Scheme 1). Obviously, this structure **4** was different from that of the simple substituted aniline-involved reactions reported by Sridharan *et al.* [12] in 2008 and 2009.

Using the conversion of 2-bromobenzaldehyde **1a**, **2**, and **3** as a model, several parameters were explored as shown in Table 1. It was found that no reaction occurred at reflux condition in the absence of iodine (Table 1, entry 1), and the yield of **4a** was much greater in the presence of various quantities of the catalyst, reaching a maximum of 82% yield with 5 mol % iodine (Table 1, entries 4–6). The yield of **4a** was also dependent on temperature (entries 2–4), proceeding smoothly at reflux in THF. Different solvents were also tested, and THF appeared to be the best medium for this transformation (entry 5 *vs.* 7–10).

**Scheme 1.** The reaction of **1**, **2** and *n*-butylvinyl ether.




This process could tolerate both electron-donating, such as alkyl and alkoxyl, and electron-withdrawing (halogen) substituents on the aromatic aldehydes (Table 2). In all cases, the reactions proceeded efficiently at reflux to afford the corresponding 3-aryl-4,7-phenanthrolines in good yields. All the compounds were characterized by $^1$H-NMR, IR, and high resolution mass spectrometer (HRMS).

According to the literatures [2]e, we think that iodine catalyzes the reaction as a mild Lewis acid [13]. The mechanism was tentatively proposed as shown in Scheme 2. First, the Schiff base **I** may be formed by the reaction of aromatic aldehyde and quinolin-6-amine. Then imino-Diels-Alder reaction between the iodine-activated Schiff base **II** and *n*-butylvinyl ether takes place selectively to form the intermediate **III** for its stability. The unexpected *n*-BuOH loses induced by iodine results in dihydrophenanthroline **IV**, which is further oxidized by air to afford aromatized 3-aryl-4,7-phenanthrolines **4**.

## EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. IR spectra were recorded on a Tensor 27 spectrometer in KBr pellet. $^1$H-NMR spectra were obtained from a solution in DMSO-$d_6$ or $CDCl_3$ with $Me_4Si$ as internal standard using a Bruker-400 spectrometer. HRMS analyses were carried out using a Bruker-micro-TOF-Q-MS analyzer. All the reagents were purchased from China National Pharmaceutical Group Corporation; the solvent of THF was dried by Na and redistilled before use.

**Table 1**

Synthetic results of **4a** under different reaction conditions.[a]

| Entry | Temp. (°C) | $I_2$ (mol %) | Solvent | Isolated yields (%) |
|---|---|---|---|---|
| 1 | Reflux | 0 | THF | 0 |
| 2 | r.t. | 5 | THF | Trace |
| 3 | 50 | 5 | THF | 76 |
| 4 | Reflux | 5 | THF | 82 |
| 5 | Reflux | 10 | THF | 82 |
| 6 | Reflux | 20 | THF | 81 |
| 7 | Reflux | 5 | $CH_3CN$ | 73 |
| 8 | Reflux | 5 | Benzene | 80 |
| 9 | 80 | 5 | DMF | 72 |
| 10 | Reflux | 5 | $CHCl_3$ | 78 |

[a]Reagents and conditions: 2-bromobenzaldehyde **1a** (0.370 g, 2.0 mmol), **2** (0.288 g, 2.0 mmol), **3** (0.220 g, 2.2 mmol), and solvent (10 mL).

**Table 2**

Synthetic results of **4** catalyzed by iodine in THF.[a]

| Entry | Ar | Products | Time (h) | Isolated yields (%) |
|---|---|---|---|---|
| 1 | 2-$BrC_6H_4$ | **4a** | 12 | 82 |
| 2 | 3-$BrC_6H_4$ | **4b** | 16 | 76 |
| 3 | 4-$BrC_6H_4$ | **4c** | 12 | 79 |
| 4 | 3-$ClC_6H_4$ | **4d** | 12 | 84 |
| 5 | 2-$FC_6H_4$ | **4e** | 20 | 82 |
| 6 | 4-$FC_6H_4$ | **4f** | 12 | 73 |
| 7 | 4-$CH_3C_6H_4$ | **4g** | 15 | 76 |
| 8 | 4-$CH_3OC_6H_4$ | **4h** | 20 | 86 |
| 9 | 2,3-$Cl_2C_6H_3$ | **4i** | 16 | 94 |
| 10 | 3,4-$Cl_2C_6H_3$ | **4j** | 12 | 90 |
| 11 | 3,4-$(CH_3O)_2C_6H_3$ | **4k** | 21 | 78 |
| 12 | 3,4-$OCH_2OC_6H_3$ | **4l** | 12 | 84 |
| 13 | 3-$CF_3C_6H_4$ | **4m** | 10 | 82 |

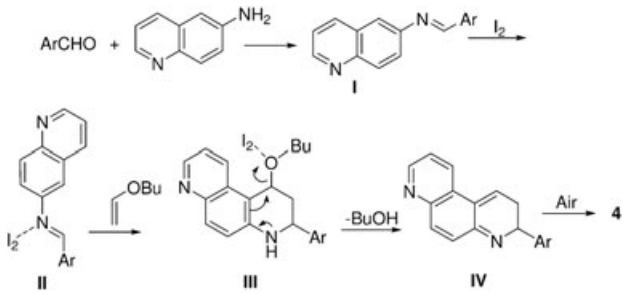
[a]Reagents and conditions: **1** (2.0 mmol), **2** (0.288 g, 2.0 mmol), **3** (0.220 g, 2.2 mmol), $I_2$ (0.1 mmol, 0.026 g), THF (10 mL), and reflux.

**General procedure for the synthesis of 3-aryl-4,7-phenanthroline 4.** A dry 50-mL flask was charged with aromatic aldehyde (2.0 mmol), quinolin-6-amine (0.288 g, 2.0 mmol), *n*-butylvinyl ether (0.220 g, 2.2 mmol), $I_2$ (0.1 mmol, 0.026 g), and THF (10 mL). The reaction mixture was stirred at reflux for 10–21 h, and then a small amount of DMF was added to the mixture until all the precipitate was dissolved. The products **4** were obtained by filtration when the mixture was allowed to cool down to room temperature.

***3-(2-Bromophenyl)-4,7-phenanthroline 4a.*** mp: 209–210°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 7.47 (t, $J$ = 7.6 Hz, 1H, ArH), 7.56–7.60 (m, 1H, ArH), 7.71 (d, $J$ = 7.6 Hz, 1H, ArH), 7.80–7.84 (m, 2H, ArH), 8.00 (d, $J$ = 8.4 Hz, 1H, ArH), 8.24 (s, 2H, ArH), 9.06 (d, $J$ = 4.0 Hz, 1H, ArH), 9.35 (d, $J$ = 8.4 Hz, 1H, ArH), 9.40 (d, $J$ = 8.8 Hz, 1H, ArH). IR (KBr): v 3075, 3051, 3009, 1580, 1526, 1483, 1447, 1434, 1417, 1387, 1356, 1330, 1300, 1284, 1102, 1078, 1022, 836, 816, 793, 764, 733, 693 cm$^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{12}BrN_2$ [M+H$^+$] 335.0184, found 335.0222.

***3-(3-Bromophenyl)-4,7-phenanthroline 4b.*** mp: 198–199°C; $^1$H-NMR ($CDCl_3$, 400 MHz): $\delta_H$ 7.42 (t, $J$ = 8.0 Hz, 1H, ArH), 7.61–7.64

**Scheme 2.** Possible mechanism for the formation of products **4**.



(m, 2H, ArH), 8.03 (d, $J$ = 8.8 Hz, 1H, ArH), 8.14 (d, $J$ = 8.0 Hz, 1H, ArH), 8.26–8.32 (m, 2H, ArH), 8.41 (s, 1H, ArH), 8.91 (d, $J$ = 8.4 Hz, 1H, ArH), 8.95 (d, $J$ = 8.8 Hz, 1H, ArH), 9.03 (d, $J$ = 4.0 Hz, 1H, ArH). IR (KBr): v 3059, 3025, 2999, 1585, 1559, 1525, 1485, 1443, 1434, 1387, 1359, 1299, 1280, 1258, 1068, 993, 839, 785, 705, 679 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{12}BrN_2$ [M+H$^+$] 335.0184, found 335.0211.

***3-(4-Bromophenyl)-4,7-phenanthroline 4c.*** mp: 236–237°C; $^1$H-NMR ($CDCl_3$, 400 MHz): $\delta_H$ 7.63 (dd, $J$ = 8.4 Hz, $J'$ = 4.4 Hz, 1H, ArH), 7.68 (d, $J$ = 8.4 Hz, 2H, ArH), 8.04 (d, $J$ = 8.4 Hz, 1H, ArH), 8.11 (d, $J$ = 8.4 Hz, 2H, ArH), 8.25–8.31 (m, 2H, ArH), 8.91 (d, $J$ = 8.0 Hz, 1H, ArH), 8.95 (d, $J$ = 8.8 Hz, 1H, ArH), 9.02–9.03 (m, 1H, ArH). IR (KBr): v 3068, 3036, 3004, 1580, 1556, 1527, 1480, 1440, 1430, 1395, 1356, 1302, 1280, 1185, 1098, 1073, 1060, 1005, 993, 851, 827, 804, 787 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{12}BrN_2$ [M+H$^+$] 335.0184, found 335.0224.

***3-(3-Chlorophenyl)-4,7-phenanthroline 4d.*** mp: 210–211°C; $^1$H NMR ($CDCl_3$, 400 MHz): $\delta_H$ 7.45–7.51 (m, 2H, ArH), 7.64 (dd, $J$ = 8.4 Hz, $J'$ = 4.4 Hz, 1H, ArH), 8.04 (d, $J$ = 8.8 Hz, 1H, ArH), 8.08–8.10 (m, 1H, ArH), 8.25–8.33 (m, 3H, ArH), 8.91 (d, $J$ = 8.4 Hz, 1H, ArH), 8.96 (d, $J$ = 8.8 Hz, 1H, ArH),9.03 (d, $J$ = 4.0 Hz, 1H, ArH). IR (KBr): v 3058, 3030, 3007, 2961, 1593, 1582, 1563, 1525, 1480, 1443, 1421, 1389, 1360, 1299, 1284, 1259, 1081, 1070, 901, 838, 784, 734, 714, 680 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{12}ClN_2$ [M+H$^+$] 291.0689, found 291.0705.

***3-(2-Fluorophenyl)-4,7-phenanthroline 4e.*** mp: 165–166°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 7.40–7.45 (m, 2H, ArH), 7.57–7.61 (m, 1H, ArH), 7.81 (dd, $J$ = 8.4 Hz, $J'$ = 4.0 Hz, 1H, ArH), 8.12–8.16 (m, 2H, ArH), 8.21–8.27 (m, 2H, ArH), 9.05 (d, $J$ = 4.4 Hz, 1H, ArH), 9.31 (d, $J$ = 8.4 Hz, 1H, ArH), 9.38 (d, $J$ = 8.8 Hz, 1H, ArH). IR (KBr): v 3050, 3027, 1614, 1585, 1526, 1486, 1441, 1390, 1363, 1307, 1283, 1210, 1112, 1068, 939, 838, 784, 751, 740 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{12}FN_2$ [M+H$^+$] 275.0985, found 275.1008.

***3-(4-Fluorophenyl)-4,7-phenanthroline 4f.*** mp: 200–201°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 7.41 (t, $J$ = 8.8 Hz, 2H, ArH), 7.79 (dd, $J$ = 8.4 Hz, $J'$ = 4.4 Hz, 1H, ArH), 8.20–8.26 (m, 2H, ArH), 8.36–8.44 (m, 3H, ArH), 9.03 (d, $J$ = 4.0 Hz, 1H, ArH), 9.32 (d, $J$ = 8.4 Hz, 1H, ArH), 9.36 (d, $J$ = 8.8 Hz, 1H, ArH). IR (KBr): v 3051, 3003, 1598, 1567, 1504, 1484, 1442, 1406, 1383, 1320, 1298, 1281, 1217, 1161, 1063, 834, 801, 775 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{12}FN_2$ [M +H$^+$] 275.0985, found 275.1011.

***3-p-Tolyl-4,7-phenanthroline 4g.*** mp: 179–180°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 2.41 (s, 3H, $CH_3$), 7.39 (d, $J$ = 8.0 Hz, 1H, ArH), 7.77 (d, $J$ = 8.4 Hz, $J'$ = 4.4 Hz, 1H, ArH), 8.18–8.26 (m, 4H, ArH), 8.34 (d, $J$ = 8.8 Hz, 1H, ArH), 9.02 (dd, $J$ = 4.4 Hz, $J'$ = 1.6 Hz, 1H, ArH), 9.29–9.34 (m, 2H, ArH). IR (KBr): v 3059, 3026, 3001, 2950, 2920, 1592, 1560, 1504, 1479, 1440, 1386, 1355, 1297, 1278, 1182, 1110, 1095, 1061, 1018, 851, 832, 807, 784, 744, 719 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{19}H_{15}N_2$ [M+H$^+$] 271.1235, found 271.1258

***3-(4-Methoxyphenyl)-4,7-phenanthroline 4h.*** mp: 200–201°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 3.86 (s, 3H, $CH_3O$), 7.14 (dd, $J$ = 6.8 Hz, $J'$ = 2.0 Hz, 2H, ArH), 7.77 (dd, $J$ = 8.0 Hz, $J'$ = 4.0 Hz, 1H, ArH), 8.17–8.23 (m, 2H, ArH), 8.30–8.33 (m, 3H, ArH), 9.01 (d, $J$ = 4.4 Hz, $J'$ = 1.6 Hz, 1H, ArH), 9.28–9.31 (m, 2H, ArH). IR (KBr): v 3054, 2955, 2835, 1603, 1596, 1566, 1531, 1508, 1483, 1441, 1415, 1391, 1304, 1289, 1254, 1186, 1111, 1027, 849, 835, 809, 783 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{19}H_{15}N_2O$ [M+H$^+$] 287.1184, found 287.1220.

***3-(2,3-Dichlorophenyl)-4,7-phenanthroline 4i.*** mp: 237–238°C; $^1$H-NMR ($CDCl_3$, 400 MHz): $\delta_H$ 7.37–7.41 (m, 1H, ArH), 7.58–7.68 (m, 3H, ArH), 7.94 (d, $J$ = 8.4 Hz, 1H, ArH), 8.30–8.33 (m, 2H, ArH), 8.96 (d, $J$ = 8.8 Hz, 1H, ArH), 9.06 (d, $J$ = 8.8 Hz, 1H, ArH), 9.05–9.06 (m, 1H, ArH). IR (KBr): v 3061, 1590, 1554, 1527, 1486, 1441, 1432, 1389, 1355, 1305, 1283, 1192, 1155, 1094, 1041, 853, 793, 775, 748, 706 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{11}Cl_2N_2$ [M+H$^+$] 325.0299, found 325.0317.

***3-(3,4-Dichlorophenyl)-4,7-phenanthroline 4j.*** mp: 216–217°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 7.60–7.65 (m, 2H, ArH), 8.02 (d, $J$ = 8.8 Hz, 1H, ArH), 8.06 (d, $J$ = 9.2 Hz, 1H, ArH), 8.26–8.31 (m, 2H, ArH), 8.37 (s, 1H, ArH), 8.90 (d, $J$ = 8.0 Hz, 1H, ArH), 8.95 (d, $J$ = 8.4 Hz, 1H , ArH), 9.04 (d, $J$ = 3.6 Hz, 1H, ArH). IR (KBr): v 3060, 3009, 1599, 1585, 1553, 1529, 1487, 1472, 1444, 1389, 1301, 1266, 1139, 1089, 1067, 1027, 914, 836, 808, 779 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{18}H_{11}Cl_2N_2$ [M+H$^+$] 325.0299, found 325.0317.

***3-(3,4-Dimethoxyphenyl)-4,7-phenanthroline 4k.*** mp: 170–171°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 3.87 (s, 3H, $CH_3O$), 3.94 (s, 3H, $CH_3O$), 7.14 (d, $J$ = 8.4 Hz, 1H, ArH), 7.77 (dd, $J$= 8.4 Hz, $J'$ = 4.0 Hz, 1H, ArH), 7.93 (d, $J$ = 8.4 Hz, 1H, ArH), 7.97 (s, 1H, ArH), 8.18–8.23 (m, 2H, ArH), 8.36 (d, $J$ = 8.8 Hz, 1H, ArH), 9.01 (d, $J$ = 4.0 Hz, 1H, ArH), 9.30 (d, $J$ = 8.8 Hz, 2H, ArH). IR (KBr): v 3008, 2963, 2936, 2910, 2837, 1596, 1564, 1530, 1510, 1481, 1459, 1441, 1415, 1359, 1338, 1274, 1224, 1217, 1161, 1135, 1021, 845, 806, 770 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{20}H_{17}N_2O_2$ [M+H$^+$] 317.1290, found 317.1324.

***3-(Methylenedioxyphenyl)-4,7-phenanthroline 4l.*** mp: 240–241°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 6.14 (s, 2H, $CH_2$), 7.11 (d, $J$ = 8.8 Hz, 1H, ArH), 7.77 (dd, $J$ = 8.0 Hz, $J'$ = 4.0 Hz, 1H, ArH), 7.91–7.93 (m, 2H, ArH), 8.17–8.21 (m, 2H, ArH), 8.30 (d, $J$ = 8.8 Hz, 1H, ArH), 9.01 (dd, $J$ = 4.0 Hz, $J'$ = 1.6 Hz, 1H, ArH), 9.29 (d, $J$ = 8.8 Hz, 2H, ArH). IR (KBr): v 3060, 2990, 2913, 1604, 1587, 1530, 1502, 1482, 1441, 1392, 1366, 1288, 1260, 1238, 1106, 1035, 927, 850, 840, 800, 788 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{19}H_{13}N_2O_2$ [M+H$^+$] 301.0977, found 301.1019.

***3-(4-(Trifluoromethyl)phenyl)-4,7-phenanthroline 4m.*** mp: 187–189°C; $^1$H-NMR (DMSO-$d_6$, 400 MHz): $\delta_H$ 7.78 (dd, $J$ = 8.4 Hz, $J'$ = 4.0 Hz, 1H, ArH), 7.92 (d, $J$ = 8.0 Hz, 2H, ArH), 8.20–8.27 (m, 2H, ArH), 8.43 (d, $J$ = 8.8 Hz, 1H, ArH), 8.54 (d, $J$ = 8.0 Hz, 2H, ArH), 9.03 (d, $J$ = 4.0 Hz, 1H, ArH), 9.31 (d, $J$ = 8.4 Hz, 1H, ArH), 9.39 (d, $J$ = 8.8 Hz, 1H, ArH). IR (KBr): v 3067, 3032, 3008, 1940, 1615, 1580, 1566, 1530, 1484, 1448, 1404, 1391, 1322, 1282, 1153, 1125, 1071, 1012, 994, 983, 862, 837, 811, 784, 719, 687 $cm^{-1}$. HRMS (ESI, *m/z*): calcd for $C_{20}H_{12}F_3N_2$ [M+H$^+$] 325.0953, found 325.0967.

## CONCLUSION

In conclusion, we found a mild and efficient method for the synthesis of 3-aryl-4,7-phenanthroline derivatives *via* three-component reaction of aromatic aldehyde, quinolin-6-amine **2**, and *n*-butylvinyl ether using iodine as catalyst. The features of this procedure are mild reaction conditions, good yields, and operational simplicity.

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