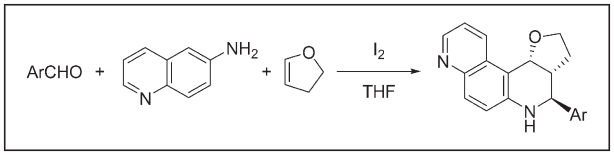
An Efficient Method for The Synthesis of 4-Arylfuro[2,3-*a*][4,7]phenanthroline Derivatives Catalyzed by Iodine

Xiang-Shan Wang*, Ming-Yue Yin, Shu-Liang Wang, Wei Wang, and Yu-Ling Li

School of Chemistry and Chemical Engineering, Xuzhou Normal University, The Key Laboratory of Biotechnology on Medical Plant, Xuzhou, Jiangsu, 221116, People's Republic of China
*E-mail: xswang@xznu.edu.cn

Received November 1, 2010
DOI 10.1002/jhet.824
View this article online at wileyonlinelibrary.com.



A mild and an efficient method for the synthesis of 4-arylfuro[2,3-*a*][4,7]phenanthroline derivatives via three-component reaction of aromatic aldehyde, quinolin-6-amine and 2,3-dihydrofuran is described using iodine as catalyst. The features of this procedure are mild reaction conditions, good yields and operational simplicity.

J. Heterocyclic Chem., **49**, 585 (2012).

INTRODUCTION

Multicomponent reactions (MCRs) can be distinguished from classical, sequential two-component synthetic processes in that they use three or more chemical starting materials for product formation [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 and pharmaceutical chemistry [3].

Phenanthroline and its derivatives are well-known compounds for their metallic compleses. The latter possess remarkable physiological and pharmacological activities. These activities include anticancer [Ln(III)] [4], antiinflammatory [Cu (II)] [5], antitumor [Pt(II)] [6], antimicrobial [Cu(II)] [7], and antibacterial activity [Zn (II)] [8]. In addition, it was reported that phenanthroline derivatives also had commendable antitumor activity [9].

To the best of our knowledge, there is no reported concerning about the synthesis of furo[2,3-*a*][4,7]phenanthroline derivatives. Such variations may contribute to the bioactivity differences and enrich the phenanthroline library for biomedical screening. As a continuation of our research devoted to the development of new methods for the preparation of heterocycles via MCRs catalyzed by iodine [10], herein, we would like to synthesize of *exo*-2,3,3*a*,4,5,11c-hexahydro-4-arylfuro [2,3-*a*][4,7]phenanthroline derivatives by a reaction of aromatic aldehyde, and quinolin-6-amine and 2,3-dihydrofuran in THF catalyzed by iodine.

RESULTS AND DISCUSSION

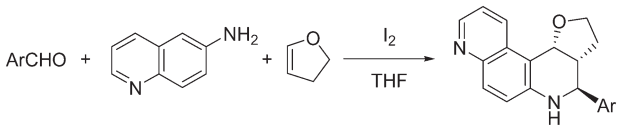
Treatment of aromatic aldehyde **1**, quinolin-6-amine **2** and 2,3-dihydrofuran **3** in THF in the presence of 5 mol % iodine at reflux condition afforded the corresponding *exo*-4-aryl-2,3,3*a*,4,5,11*c*-hexahydrofuro[2,3-*a*][4,7]phenanthrolines **4** in good yields (Scheme 1).

Using the conversion of 4-methylbenzaldehyde, quinolin-6-amine and 2,3-dihydrofuran as a model, several parameters were explored as shown in Table 1. The yield of **4a** was not detected in the absence of iodine (Table 1, Entries 1 and 2), and much greater in the presence of various quantities of the catalyst, reaching a maximum of 82% yield with 5 mol % iodine (Table 1, Entries 5–7). The yield of **4a** was also dependent on temperature (Entries 3–5), proceeding smoothly at reflux in THF. Different solvents were also tested, and THF appeared to be the best medium for this transformation (Entries 5, 8–11).

This process can tolerate both electron-donating (alkyl and alkoxy) and electron-withdrawing (halogen) substituents on the aromatic aldehydes (Table 2). In all cases, the reactions proceeded efficiently at reflux to afford the corresponding 4-arylfuro[2,3-*a*][4,7]phenanthrolines in good yields. All the compounds were characterized by ^{1}H NMR, IR, and HRMS. The *exo*-structure of **4i** is additionally confirmed by X-ray diffraction analysis, and its crystal structure is shown in Figure 1.

© 2012 HeteroCorporation

Scheme 1. The reaction of **1**, **2**, and 2,3-dihydrofuran.



1 **2** **3** **4**

According to the literatures [10]e, we think that iodine catalyzes the reaction as a mild Lewis acid. The mechanism was tentatively proposed as shown in Scheme 2. The Schiff base **I** may be formed first by the reaction of aromatic aldehyde and quinolin-6-amine. Then imino-Diels-Alder reaction between the iodine-activated Schiff base **II** and 2,3-dihydrofuran takes place selectively to form the intermediate **III** for its stability, followed by isomerization to give the final product **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 was obtained from a solution in DMSO-d_6 with Me_4Si as internal standard using a Bruker-400 spectrometer. HRMS analyses were carried out using a Bruker-micro-TOF-Q-MS analyzer. X-ray diffraction was performed on a Bruker APEX-II area-detector diffractometer.

General procedure for the synthesis of 4-arylfuro[2,3-*a*][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), 2,3-dihydrofuran (0.210 g, 3.0 mmol), THF (10 mL) and I_2 (0.026 g, 0.1 mmol). The reaction mixture was stirred at reflux for 12–22 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.

Table 1

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

Entry	Temp. (°C)	I_2 (mol %)	Solvent	Yields (%)[b]
1	r.t.	0	THF	0
2	Reflux	0	THF	0
3	r.t.	5	THF	trace
4	50	5	THF	67
5	Reflux	5	THF	82
6	Reflux	10	THF	82
7	Reflux	20	THF	81
8	Reflux	5	CH_3CN	78
9	Reflux	5	Benzene	80
10	80	5	DMF	69
11	Reflux	5	$CHCl_3$	74

[a]Reagents and conditions: 4-methylbenzaldehyde (0.240g, 2.0 mmol), **2** (0.288 g, 2.0 mmol), **3** (0.210 g, 3.0 mmol), I_2, solvent (10 mL).
[b]Isolated yields

Table 2

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

Entry	Ar	Products	Time (h)	Isolated yields (%)
1	4-$CH_3C_6H_4$	**4a**	12	82
2	3-BrC_6H_4	**4b**	18	78
3	4-BrC_6H_4	**4c**	12	84
4	3-FC_6H_4	**4d**	16	73
5	4-FC_6H_4	**4e**	22	76
6	3-ClC_6H_4	**4f**	16	83
7	4-ClC_6H_4	**4g**	21	72
8	4-$CH_3OC_6H_4$	**4h**	18	86
9	2,3-$Cl_2C_6H_3$	**4i**	12	77
10	3,4-$Cl_2C_6H_3$	**4j**	12	74
11	3,5-$(CH_3O)_2C_6H_3$	**4k**	18	89
12	3,4-$(CH_3O)_2C_6H_3$	**4l**	12	76
13	3,4-$OCH_2OC_6H_3$	**4m**	12	71

[a]Reagents and conditions: **1** (2.0 mmol), **2** (0.288 g, 2.0 mmol), **3** (0.210 g, 3.0 mmol), I_2 (0.026 g, 0.1 mmol), THF (10 mL).

Exo-2,3,3a,4,5,11c-Hexahydro-4-p-tolylfuro[2,3-a][4,7]phenanthroline 4a. M. p.: 221–222°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.551.62 (m, 1H, CH), 1.96–2.02 (m, 1H, CH), 2.342.38 (m, 4H, CH_3 + CH), 3.71 (d, J = 11.2 Hz, 1H, CH), 3.77–3.83 (m, 1H, CH), 3.91–3.97 (m, 1H, CH), 4.87 (d, J = 5.2 Hz, 1H, CH), 6.81 (s, 1H, NH), 7.24 (d, J = 7.6 Hz, 2H, ArH), 7.28 (d, J = 9.2 Hz, 1H, ArH), 7.38–7.42 (m, 3H, ArH), 7.71 (d, J = 9.2 Hz, 1H, ArH), 8.28 (d, J = 8.4 Hz, 1H, ArH), 8.54–8.55 (m, 1H, ArH). IR (KBr): ν 3363, 2966, 2884, 2874, 1620, 1576, 1462, 1378, 1349, 1267, 1134, 1097, 1030, 952, 908, 832, 811, 767, 753 cm^{-1}. HRMS (ESI, *m/z*): Calcd for $C_{21}H_{21}N_2O$ ($M + H^+$) 317.1654, found 317.1648.

Exo-4-(3-Bromophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4b. M. p.: 222–223°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.56–1.63 (m, 1H, CH), 2.02–2.05 (m, 1H, CH), 2.37–2.42 (m, 1H, CH), 3.77–3.81 (m, 2H, 2CH), 3.94–3.99 (m, 1H, CH), 4.87 (d, J = 4.8 Hz, 1H, CH), 6.91 (s, 1H, NH), 7.27 (d, J = 8.8 Hz, 1H, ArH), 7.38–7.43 (m, 2H, ArH), 7.55–7.60 (m, 2H, ArH), 7.73 (d, J =9.2 Hz, 1H, ArH), 7.76–7.77 (m, 1H, ArH), 8.29 (dd, J = 8.8 Hz, J' = 0.8 Hz, 1H, ArH), 8.56 (dd, J = 4.0 Hz, J' = 1.6 Hz, 1H, ArH). IR (KBr): ν 3359, 3049, 2961, 2938, 2871, 2831, 1621, 1595, 1570, 1520, 1467, 1454, 1426, 1379, 1348, 1269, 1171, 1072, 1028, 1017, 951, 904, 832, 811, 788, 693 cm^{-1}. HRMS

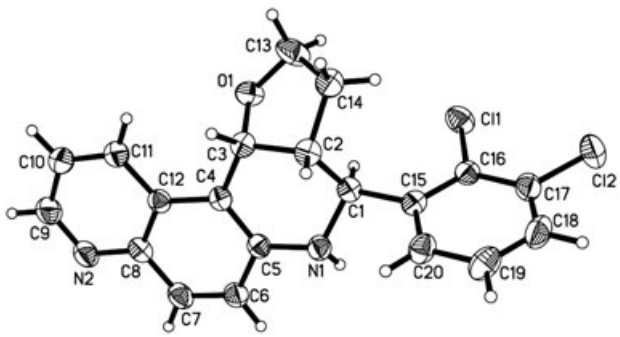
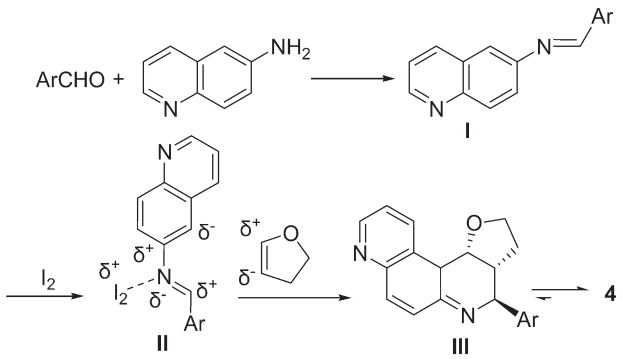


Figure 1. The crystal structure of product **4i**.

Scheme 2. The possible mechanism for the formation of product **4**.



(ESI, *m*/*z*): Calcd for $C_{20}H_{18}BrN_2O$ (M + H^+) 381.0603, found 381.0589.

Exo-4-(4-Bromophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4c. M. p.: 229–230°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.54–1.61 (m, 1H, CH), 1.98–2.04 (m, 1H, CH), 2.33–2.36 (m, 1H, CH), 3.75–3.82 (m, 2H, 2CH), 3.92–3.98 (m, 1H, CH), 4.88 (d, *J* = 5.2 Hz, 1H, CH), 6.87 (s, 1H, NH), 7.27 (d, *J* = 8.8 Hz, 1H, ArH), 7.41 (dd, *J* = 8.4 Hz, *J*′ = 4.0 Hz, 1H, ArH), 7.51 (d, *J* = 8.4 Hz, 2H, ArH), 7.63 (d, *J* =8.4 Hz, 2H, ArH), 7.71–7.73 (m, 3H, ArH), 7.87 (d, *J* = 8.4 Hz, 2H. ArH), 8.28 (d, *J* = 8.4 Hz, 1H, ArH), 8.55–8.56 (m, 1H, ArH). IR (KBr): ν 3356, 2875, 2855, 1621, 1586, 1521, 1487, 1396, 1382, 1295, 1266, 1171, 1069, 1045, 1011, 951, 824, 813, 803, 762 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{20}H_{18}BrN_2O$ (M + H^+) 381.0603, found 381.0590.

Exo-4-(3-Fluorophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4d. M. p. 193–194°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.58–1.65 (m, 1H, CH), 1.99–2.03 (m, 1H, CH), 2.38–2.42 (m, 1H, CH), 3.79–3.84 (m, 2H, 2CH), 3.94–3.99 (m, 1H, CH), 4.88 (d, *J* = 4.8 Hz, 1H, CH), 6.92 (s, 1H, NH), 7.19–7.24 (m, 1H, ArH), 7.28 (d, *J* = 9.2 Hz, 1H, ArH), 7.39–7.51 (m, 4H, ArH), 7.73 (d, *J* = 9.2 Hz, 1H, ArH), 8.29 (d, *J* = 8.4 Hz, 1H, ArH), 8.56 (d, *J* = 2.8 Hz, 1H, ArH). IR (KBr): ν 3352, 2962, 2938, 2874, 2849, 1618, 1591, 1516, 1486, 1461, 1423, 1377, 1351, 1307, 1266, 1234, 1172, 1130, 1029, 945, 861, 831, 805, 790 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{20}H_{18}FN_2O$ (M + H^+) 321.1403, found 321.1401.

Exo-4-(4-Fluorophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4e. M. p.: 233–234°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.54–1.60 (m, 1H, CH), 1.98–2.03 (m, 1H, CH), 2.33–2.39 (m, 1H, CH), 3.77–3.84 (m, 2H, 2CH), 3.92–3.98 (m, 1H, CH), 4.88 (d, *J* = 4.8 Hz, 1H, CH), 6.85 (s, 1H, NH), 7.24–7.30 (m, 3H, ArH), 7.41 (dd, *J* = 8.4 Hz, *J*′ = 4.4 Hz, 1H, ArH), 7.57–7.61 (m, 2H, ArH), 7.73 (d, *J* = 9.2 Hz, 1H, ArH), 8.55–8.56 (m, 1H, ArH). IR (KBr): ν 3355, 2974, 2937, 2872, 1620, 1605, 1577, 1511, 1468, 1420, 1378, 1269, 1219, 1170, 1157, 1094, 1030, 1016, 950, 928, 832, 806, 767 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{20}H_{18}FN_2O$ (M + H^+) 321.1403, found 321.1398.

Exo-4-(3-Chlorophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4f. M. p.: 200–201°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.56–1.63 (m, 1H, CH), 1.99–2.05 (m, 1H, CH), 2.37–2.41 (m, 1H, CH), 3.78–3.84 (m, 2H, 2CH), 3.94–3.99 (m, 1H, CH), 4.88 (d, *J* = 4.8 Hz, 1H, CH), 6.92 (s, 1H, NH), 7.28 (d, *J* = 9.2 Hz, 1H, ArH), 7.40–7.53 (m, 4H, ArH), 7.63 (s, 1H, ArH), 7.73 (d, *J* = 8.8 Hz, 1H, ArH), 8.29 (d, *J* = 8.4 Hz, 1H, ArH), 8.55–8.56 (m, 1H, ArH). IR (KBr): ν 3363, 3047, 2963, 2936, 2869, 1621, 1597, 1575, 1521, 1470, 1426, 1379, 1347, 1270, 1171, 1088, 1029, 907, 831, 811, 785, 767, 696 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{20}H_{18}ClN_2O$ (M + H^+) 337.1108, found 337.1104.

Exo-4-(4-Chlorophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4g. M. p.: 228–229°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.54–1.61 (m, 1H, CH), 1.98–2.03 (m, 1H, CH), 2.33–2.36 (m, 1H, CH), 3.77–3.82 (m, 2H, 2CH), 3.93–3.98 (m, 1H, CH), 4.88 (d, *J* = 4.8 Hz, 1H, CH), 6.87 (s, 1H, NH), 7.28 (d, *J* = 9.2 Hz, 1H, ArH), 7.41 (dd, *J* = 8.4 Hz, *J*′ = 4.0 Hz, 1H, ArH), 7.49 (d, *J* = 8.4 Hz, 2H, ArH), 7.51 (d, *J* = 8.4 Hz, 2H, ArH), 7.73 (d, *J* = 8.4 Hz, 1H, ArH), 8.28 (d, *J* = 8.4 Hz, 1H, ArH), 8.55–8.56 (m, 1H, ArH). IR (KBr): ν 3355, 3035, 2970, 2935, 2873, 1620, 1578, 1519, 1492, 1468, 1377, 1268, 1169, 1095, 1088, 1030, 1015, 928, 832, 809, 738 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{20}H_{18}ClN_2O$ (M + H^+) 337.1108, found 337.1140.

Exo-2,3,3a,4,5,11c-Hexahydro-4-(4-methoxyphenyl)furo[2,3-a][4,7]phenanthroline 4h. M. p.: 183–184°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.55–1.62 (m, 1H, CH), 1.99–2.04 (m, 1H, CH), 3.32–3.37 (m, 1H, CH), 3.70 (d, *J* =11.2 Hz, 1H, CH), 3.78–3.83 (m, 4H, CH_3O + CH), 3.91–3.97 (m. 1H, CH), 4.87 (d, *J* = 4.8 Hz, 1H, CH), 6.77 (s, 1H, NH), 6.99 (d, *J* = 8.8 Hz, 1H, ArH), 7.41 (dd, *J* = 8.4 Hz, *J*′ = 4.4 Hz, 1H, ArH), 7.45 (d, *J* = 8.4 Hz, 2H, ArH), 7.71 (d, *J* = 9.2 Hz, 1H, ArH), 8.28 (d, *J* = 8.0 Hz, 1H, ArH), 8.54–8.55 (m, 1H, ArH). IR (KBr): ν 3502, 3070, 3029, 3000, 2939, 2883, 1620, 1522, 1511, 1474, 1459, 1380, 1350, 1296, 1272, 1248, 1173, 1094, 1029, 980, 832, 815 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{21}H_{21}N_2O_2$ (M + H^+) 333.1603, found 333.1645.

Exo-4-(2,3-Dichlorophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4i. M. p.: 251–252°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.51–1.58 (m, 1H, CH), 2.07–2.16 (m, 1H, CH), 2.45–2.49 (m, 1H, CH), 3.83–3.97 (m, 2H, 2CH), 4.42 (d, *J* = 11.6 Hz, 1H, CH), 4.92 (d, *J* = 5.2 Hz, 1H, CH), 6.91 (s, 1H, NH), 7.24 (d, *J* = 8.8 Hz, 1H, ArH), 7.42 (dd, *J* = 8.8 Hz, *J*′ = 4.8 Hz, 1H, ArH), 7.47–7.51 (m, 1H, ArH), 7.67–7.72 (m, 2H, ArH), 7.75 (d, *J* = 8.8 Hz, 1H, ArH), 8.30 (dd, *J* = 8.4 Hz, *J*′ = 0.8 Hz, 1H, ArH), 8.58 (dd, *J* = 4.4 Hz, *J*′ = 1.6 Hz, 1H, ArH). IR (KBr): ν 3259, 2935, 2885, 2845, 1620, 1581, 1517, 1452, 1422, 1373, 1345, 1266, 1235, 1170, 1153, 1131, 1036, 927, 826, 787, 735, 699 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{20}H_{17}Cl_2N_2O$ (M + H^+) 371.0718, found 371.0713.

Exo-4-(3,4-Dichlorophenyl)-2,3,3a,4,5,11c-hexahydrofuro[2,3-a][4,7]phenanthroline 4j. M. p.: 204–205°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.55–1.62 (m, 1H, CH), 2.00–2.05 (m, 1H, CH), 2.37–2.43 (m, 1H, CH), 3.79–3.83 (m, 2H, 2CH), 3.94–3.99 (m, 1H, CH), 4.88 (d, *J* = 4.8 Hz, 1H, CH), 6.90 (s, 1H, NH), 7.26 (d, *J* = 9.2 Hz, 1H, ArH), 7.41 (dd, *J* = 8.4 Hz, *J*′ = 4.0 Hz, 1H, ArH), 7.55 (dd, *J* = 8.4 Hz, *J*′ = 2.4 Hz, 1H, ArH), 7.70 (d, *J* = 8.0 Hz, 1H, ArH), 7.74 (d, *J* = 9.2 Hz, 1H, ArH), 7.85 (d, *J* = 1.6 Hz, 1H, ArH), 8.29 (d, *J* = 8.4 Hz, 1H, ArH), 8.56–8.57 (m, 1H, ArH). IR (KBr): ν 3354, 3054, 3011, 2974, 2941, 2874, 2852, 1620, 1516, 1423, 1377, 1350, 1286, 1170, 1134, 1030, 931, 906, 832, 812 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{20}H_{17}Cl_2N_2O$ (M + H^+) 371.0718, found 371.0715.

Exo-2,3,3a,4,5,11c-Hexahydro-4-(3,5-dimethoxyphenyl)furo[2,3-a][4,7]phenanthroline 4k. M. p.: 202–203°C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ_H 1.64–1.71 (m, 1H, CH), 2.02–2.05 (m, 1H, CH), 2.36–2.40 (m, 1H, CH), 3.69 (d, *J* = 11.2 Hz, 1H,

CH), 3.80–3.83 (m, 7H, $2CH_3O$ + CH), 3.93–3.96 (m, 1H, CH), 4.86 (d, J = 4.8 Hz, 1H, CH), 6.50–6.51 (m, 1H, NH), 6.71 (d, J = 2.0 Hz, 2H, ArH), 6.85 (s, 1H, ArH), 7.29 (dd, J = 8.4 Hz, J' = 0.8 Hz, 1H, ArH), 8.55 (dd, J = 5.2 Hz, J' = 1.2 Hz, 1H, ArH). IR (KBr): ν 3351, 2969, 2934, 2869, 2838, 1612, 1591, 1524, 1473, 1459, 1428, 1378, 1345, 1312, 1272, 1203, 1173, 1069, 1029, 982, 920, 831, 810 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{22}H_{23}N_2O_3$ ($M + H^+$) 363.1709, found 363.1704.

Exo-2,3,3a,4,5,11c-Hexahydro-4-(3,4-dimethoxyphenyl)furo [2,3-a][4,7]phenanthroline 4l. M. p.: 241–242°C; ^1H NMR (DMSO-d_6, 400 MHz): δ_H 1.98–2.07 (m, 1H, CH), 2.34–2.39 (m, 1H, CH), 3.69 (d, J = 11.6 Hz, 1H, CH), 3.78–3.81 (m, 7H, $2CH_3O$ + CH), 3.92–3.98 (m, 1H, CH), 4.87 (d, J = 5.2 Hz, 1H, CH), 6.80 (s, 1H, NH), 6.99 (d, J = 8.0 Hz, 1H, ArH), 7.03–7.05 (m, 1H, ArH), 7.13 (s, 1H, ArH), 7.29 (d, J = 9.2 Hz, 1H, ArH), 8.41 (dd, J = 8.4 Hz, J' = 4.0 Hz, 1H, ArH), 7.71 (d, J = 8.8 Hz, 1H, ArH), 8.28 (d, J = 8.4 Hz, 1H, ArH), 8.54–8.55 (m, 1H, ArH). IR (KBr): ν 3357, 2994, 2966, 2939, 2839, 1619, 1592, 1514, 1461, 1422, 1375, 1346, 1268, 1236, 1226, 1144, 1039, 1024, 943, 841, 812, 766 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{22}H_{23}N_2O_3$ ($M + H^+$) 363.1709, found 363.1701.

Exo-4-(3,4-Methylenedioxophenyl)-2,3,3a,4,5,11c-hexahydrofuro [2,3-a][4,7]phenanthroline 4m. M. p. 199–200°C; ^1H NMR (DMSO-d_6, 400 MHz): δ_H 1.58–1.65 (m, 1H, ArH), 2.01–2.04 (m, 1H, CH), 2.31–2.37 (m, 1H, CH), 2.69 (d, J = 11.2 Hz, 1H, CH), 3.77–3.83 (m, 1H, CH), 3.91–3.97 (m, 1H, CH), 4.86 (d, J = 4.8 Hz, 1H, CH), 6.05 (s, 2H, CH_2), 6.79 (s, 1H, NH), 6.95 (d, J = 8.0 Hz, 1H, ArH), 7.00 (dd, J = 8.0 Hz, J' = 1.2 Hz, 1H, ArH), 7.12 (s, 1H, ArH), 7.28 (d, J =9.2 Hz, 1H, ArH), 7.40 (dd, J = 8.4 Hz, J' = 4.0 Hz, 1H, ArH), 7.72 (d, J = 9.2 Hz, 1H, ArH), 8.27 (d, J = 8.4 Hz, 1H, ArH), 8.55 (dd, J = 8.0 Hz, J' = 1.2 Hz, 1H, ArH). IR (KBr): ν 3280, 3073, 2975, 2944, 2869, 1619, 1519, 1441, 1386, 1349, 1270, 1251, 1236, 1202, 1190, 1104, 1041, 981, 932, 831, 814, 742 cm^{-1}. HRMS (ESI, *m*/*z*): Calcd for $C_{21}H_{19}N_2O_3$ ($M + H^+$) 347.1396, found 347.1395.

CONCLUSIONS

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

Acknowledgments. We are grateful to the National Natural Science foundation of China (20802061), the Natural Science foundation (08KJD150019) and Qing Lan Project (08QLT001) of Jiangsu Education Committee for financial support.

REFERENCES AND NOTES

[1] (a) Vijjay, N. C.; Rajesh, A. U.; Vinod, S.; Bindu, A. R.; Sreekanth, J. S.; Lakshmi, B. Acc Chem Res 2003, 36, 899; (b) Tietze, L. F. Chem Rev 1996, 96, 115.

[2] (a) Liéby-Muller, F.; Constantieux, T.; Jean Rodriguez, J. J Am Chem Soc 2005, 127, 17176; (b) Wang, X. S.; Zhang, M. M.; Li, Q.; Yao, C. S.; Tu, S. J. Synlett 2007, 3141; (c) Yao, C. S.; Lei, S.; Wang, C. H.; Li, T. J.; Yu, C. X.; Wang, X. S.; T. S. J.; J Heterocycl Chem 2010, 47, 26; (d) Bantval, S. H; Kani, V. M.; Balladka, K. S.; Boja, P. Synth Commun 2005, 35, 333; (e) Wang, X. S.; Li, Q.; Yao, C. S.; Tu, S. J. Eur J Org Chem 2008, 20, 3513; (f) Srinivasu V. N. V.; Rajashekar, B. L.; Srinivas, K. J. Heterocycl Chem 2010, 47, 687; (g) Wang, X. S.; Zhang, M. M.; Jiang, H.; Shi, D. Q.; Tu, S. J.; Wei, X. Y.; Zong, Z. M. Synthesis 2006, 4187.

[3] (a) Dolle, R. E.; Bourdonnec, B. L.; Goodman, A. J.; Morales, G. A.; Thomas, C. J.; Zhang, W. J Comb Chem 2008, 10, 753; (b) Victorio, C.; José, G.; Noel, N. J Heterocycl Chem 2010, 47, 233; (c) Wang, X. S.; Zhang, M. M.; Jiang, H.; Tu, S. J. Tetrahedron 2007, 63, 4439; (d) Wang, X. S.; Zhou, J.; Yang, K.; Zhang, M. M. Synth Commun 2010, 40, 1065.

[4] Li, F. H.; Lin, H. K. Wuji Huaxue Xuebao 2008, 24, 1949.

[5] Sharma, K. V.; Sharma, V.; Dubey, R. K.; Tripathi, U. N. J Coord Chem 2009, 62, 493.

[6] Margiotta, N.; Papadia, P.; Lazzaro, F.; Crucianelli, M.; De Angelis, F.; Pisano, C.; Vesci, L.; Natile, G. J Med Chem 2005, 48, 7821.

[7] Katsarou, M. E.; Efthimiadou, E. K.; Psomas, G.; Karaliota, A.; Vourloumis, D. J Med Chem 2008, 51, 470.

[8] Tang, D. X.; Feng, L. X.; Zhang, X. Q. Wuji Huaxue Xuebao 2006, 22, 1891.

[9] Wesselinova, D.; Neykov, M.; Kaloyanov, N.; Toshkova, R.; Dimitrov, G. Eur J Med Chem 2009, 44, 2720.

[10] (a) Wang, X. S.; Li, Q.; Zhou, J.; Tu, S. J. J Heterocycl Chem 2009, 46, 1222; (b) Wang, X. S.; Li, Q.; Wu, J. R.; Tu, S. J. J Comb Chem 2009, 11, 433; (c) Wang, X. S.; Zhou, J.; Yin, M. Y.; Yang, K.; Tu, S. J. J Comb Chem 2010, 12, 266; (d) Wang, X. S.; Yang, K.; Zhou, J.; Tu, S. J. J Comb Chem 2010, 12, 417; (e) Wang, X. S.; Zhou, J.; Yin, M. Y.; Yang, K.; Tu, S. J. J Heterocycl Chem 2010, 47, 873.