# Yb(OTf)<sub>3</sub>: An Efficient Catalyst for the Synthesis of 11-Aryl-7*H*-cyclopenta[*b*][4,7]phenanthrolin-10(11*H*)-One Derivatives

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A mild and efficient method for the synthesis of 8,9-dihydro-11-aryl-7*H*-cyclopenta[*b*][4,7]phenanthrolin-10 (11*H*)-one derivatives *via* three-component reaction of aromatic aldehyde, quinolin-6-amine and cyclopentane-1,3-dione is described catalyzed by Yb(OTf)<sub>3</sub>. The features of this procedure are mild reaction conditions, good to high yields, and operational simplicity.

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### **INTRODUCTION**

Over the past few years,  $Yb(OTf)_3$  has emerged as a powerful catalyst for various organic transformations to afford the products in good to excellent yields. Owing to several advantages such as inexpensive, nontoxic, ecofriendly nature,  $Yb(OTf)_3$  has been used as a catalyst in the investigation of different organic reactions [1].

Phenanthrolines are important core structures found in a variety of biologically important molecules [2]. It is reported that metallic complexes possess a wide range of biological activities, which confer applications as anticancer [3], antiinflammatory [4], antitumor [5], antimicrobial [6], and antibacterial agents [7]. Therefore, much attention is devoted to the synthesis of these active frameworks in recent years [8].

To the best of our knowledge, there is no report concerning the synthesis of cyclopenta[b][4,7]phenanthrolin-10(11H)-one 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 catalyzed by Yb(OTf)<sub>3</sub> [9], herein, we would like to synthesize of 8,9-dihydro-11-aryl-7H-cyclopenta[b][4,7] phenanthrolin-10(11H)-one by a reaction of aromatic aldehyde, quinolin-6-amine, and cyclopentane-1,3-dione catalyzed by Yb(OTf)<sub>3</sub>.

#### **RESULTS AND DISCUSSION**

Treatment of aromatic aldehyde, quinolin-6-amine, and cyclopentane-1,3-dione in reflux THF in the presence

of 1 mol % Yb(OTf)<sub>3</sub>, afforded the corresponding 8,9dihydro-11-aryl-7*H*-cyclopenta[*b*][4,7]phenanthrolin-10 (11H)-one derivatives **4** in good to high yields (Scheme 1).

Using the conversion of 2-fulorobenzaldehyde 1a, quinolin-6-amine and cyclopentane-1,3-dione as a model, several parameters were explored as shown in Table 1. The yield of 4a was moderate at reflux in the absence of Yb(OTf)<sub>3</sub> (62%, Table 1, entry 1) and much greater in the presence of various quantities of the catalyst, reaching a maximum of 87% yield with 1 mol % Yb (OTf)<sub>3</sub> (Table 1, entries 2–11). The yield of 4a was also dependent on temperature (entries 2, 5, and 6), proceeding smoothly at reflux. Different solvents were also tested and THF appeared to be the best medium for this transformation (entry 2 *vs.* 13–14).

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 in THF at reflux to afford the corresponding 7H-cyclopenta[b] [4,7]phenanthrolin-10(11H)-one **4a-I** in high yields. All the compounds were characterized by <sup>1</sup>H NMR, IR, and HRMS.

The formation of **4** is likely to proceed via initial condensation of aldehyde **1** with cyclopentane-1,3-dione **2** to afford 2-benzylidenecyclopentane-1,3-dione **5**, which further undergoes Michael addition with quinolin-6-amine **3** to furnish **6**. Intramolecular nucleophilic reaction of its isomer **7** could afford the compound **8**, which could eliminate one molecule of  $H_2O$  to afford title product **4** (Scheme 2). The role of the catalyst Yb(OTf)<sub>3</sub> is activating the carbonyl groups in intermediate products **5** and **7**.

Scheme 1. The reaction of 1, 2, and 3 catalyzed by Yb(OTf)<sub>3</sub>.



 Table 1

 Synthesis of 4a at different reaction conditions.<sup>a</sup>

Entry	$T(^{\circ}\mathrm{C})$	Solvent	Cat. (mol %)	Yield <sup>b</sup> (%)
1	Reflux	THF	0	62
2	Reflux	THF	$Yb(OTf)_3(1)$	87
3	Reflux	THF	$Yb(OTf)_3(5)$	86
4	Reflux	THF	Yb(OTf) <sub>3</sub> (10)	87
5	r.t	THF	$Yb(OTf)_3(1)$	Trace
6	50	THF	Yb(OTf) <sub>3</sub> (1)	73
7	Reflux	THF	AgOTf (1)	83
8	Reflux	THF	$Cu(OTf)_2(1)$	80
9	Reflux	THF	$Zn(OTf)_2(1)$	78
10	Reflux	THF	$Y(OTf)_3(1)$	85
11	Reflux	THF	$Fe(OTf)_2(1)$	79
12	Reflux	CH <sub>3</sub> CN	Yb(OTf) <sub>3</sub> (1)	85
13	Reflux	Benzene	$Yb(OTf)_3(1)$	84
14	80	DMF	$Yb(OTf)_3(1)$	80

<sup>a</sup>Reaction condition: 10 mL solvent, 2-fluorobenzaldehyde (0.248 g, 2.0 mmol), quinolin-6-amine (0.288 g, 2.0 mmol), cyclopentane-1,3dione (0.196 g, 2.0 mmol). <sup>b</sup>Isolated yields.

 Table 2

 Synthetic results of 4 in THF catalyzed by Yb(OTf)<sub>3</sub>.<sup>a</sup>

Entry	Ar	Products	Time (h)	Isolated Yields (%)
1	$2-FC_6H_4$	4a	12	87
2	$2-ClC_6H_4$	4b	12	88
3	$4-ClC_6H_4$	<b>4</b> c	14	92
4	3-BrC <sub>6</sub> H <sub>4</sub>	<b>4d</b>	14	82
5	$4-BrC_6H_4$	<b>4</b> e	16	86
6	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>4f</b>	16	90
7	3-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	4g	16	92
8	3,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	4h	10	86
9	3,4-(CH <sub>3</sub> )	4i	16	83
	$_2C_6H_3$			
10	3,4-	4j	16	86
	OCH <sub>2</sub> OC <sub>6</sub> H <sub>3</sub>	-		
11	2,3-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	4k	12	90
12	$2,4-Cl_2C_6H_3$	41	12	89

<sup>a</sup>Reagents and conditions: **1** (2.0 mmol), **2** (0.288 g, 2.0 mmol), **3** (0.196 g, 2.0 mmol), THF (10 mL). Yb(OTf)<sub>3</sub> (0.012 g, 0.02 mmol).

## CONCLUSION

In conclusion, we found a mild and efficient method for the synthesis of 11-aryl-7*H*-cyclopenta[*b*][4,7]phenanthrolin-

Scheme 2. Reaction mechanism of 1, 2, and 3 catalyzed by Yb(OTf)<sub>3</sub>.



10(11H)-one derivatives via three-component reaction of aromatic aldehyde, quinolin-6-amine, and cyclopentane-1,3-dione catalyzed by Yb(OTf)<sub>3</sub>. The features of this procedure are mild reaction conditions, good to high yields, and operational simplicity.

#### **EXPERIMENTAL**

Melting points were determined in open capillaries and are uncorrected. IR spectra were recorded on a Tensor 27 spectrometer in KBr pellet. <sup>1</sup>H NMR spectra was obtained from a solution in DMSO- $d_6$  with Me<sub>4</sub>Si as internal standard using a Bruker-400 spectrometer. HRMS analyses were carried out using a Bruker-micro-TOF-Q-MS analyzer.

General procedure for the synthesis of 7*H*-cyclopenta [*b*][4,7]phenanthrolin- 10(11*H*)-one derivatives 4. A dry 50-mL-flask was charged with aromatic aldehyde (2.0 mmol), quinolin-6-amine (2.0 mmol, 0.288 g), and cyclopentane-1,3-dione (2.0 mmol, 0.196 g), THF (10 mL) and Yb(OTf)<sub>3</sub> (0.02 mmol, 0.012 g). The reaction mixture was stirred at reflux for 12–16 h, after completion of the reaction as indicated by TLC, another portion of THF was added to the mixture until all the yellow solid was dissolved. The desired products 4 were obtained as yellow powder by filtration, when the mixture was cooled to room temperature.

**11-(2-Fluorophenyl)-8,9-dihydro-7***H***-cyclopenta[***b***][<b>4**,7]phenanthrolin-10(11*H*)-one 4a. <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.21–2.32 (m, 2H, CH<sub>2</sub>), 2.67–2.76 (m, 2H, CH<sub>2</sub>), 5.81 (s, 1H, CH), 6.90–7.19 (m, 4H, ArH), 7.41(s, 1H, ArH), 7.50 (d, *J* = 8.0 Hz, 1H, ArH), 7.90 (d, *J* = 8.0 Hz, 1H, ArH), 8.18 (d, *J* = 7.6 Hz, 1H, ArH), 8.65 (s, 1H, ArH), 10.50 (s, 1H, NH). IR (KBr): v 3242, 3173, 3092, 3040, 2923, 1668, 1624, 1609, 1576, 1530, 1487, 1416, 1399, 1342, 1272, 1241, 1219, 831, 756, 699 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>21</sub>H<sub>16</sub>FN<sub>2</sub>O (M + H<sup>+</sup>) 331.1247, found 331.1232.

**11-(2-Chlorophenyl)-8,9-dihydro-7***H***-cyclopenta[***b***][4,7] <b>phenanthrolin-10(11***H***)-one 4b.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.19–2.31 (m, 2H, CH<sub>2</sub>), 2.67–2.72 (m, 2H, CH<sub>2</sub>), 5.93 (s, 1H, CH), 7.06–7.12 (m, 3H, ArH), 7.31 (d, *J* = 7.6 Hz, 1H, ArH), 7.39 (dd, *J* = 8.4Hz, *J'* = 4.4 Hz, 1H, ArH), 7.50 (d, *J* = 8.8 Hz, 1H, ArH), 7.91 (d, *J* = 8.8 Hz, 1H, ArH), 8.16 (d, J = 8.4 Hz, 1H, ArH), 8.64 (s, 1H, ArH), 10.50 (s, 1H, NH). IR (KBr): v 3245, 3180, 3098, 3040, 2971, 2921, 1682, 1629, 1577, 1530, 1468, 1416, 1394, 1273, 1218, 1051, 1034, 834, 746, 699 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd. for C<sub>21</sub>H<sub>16</sub>ClN<sub>2</sub>O (M + H<sup>+</sup>) 347.0951, found 347.0953.

**11-(4-Chlorophenyl)-8,9-dihydro-7***H***-cyclopenta[***b***][4,7] <b>phenanthrolin-10(11***H***)-one 4c.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.27–2.34 (m, 2H, CH<sub>2</sub>), 2.67–2.74 (m, 2H, CH<sub>2</sub>), 5.70 (s, 1H, CH), 7.18–7.24 (m, 4H, ArH), 7.36 (dd, *J* = 8.4 Hz, *J'* = 4.4 Hz, 1H, ArH), 7.55 (d, *J* = 8.8 Hz, 1H, ArH), 7.95 (d, *J* = 8.8 Hz, 1H, ArH), 8.18 (d, *J* = 8.4 Hz, 1H, ArH), 8.68 (dd, *J* = 4.4 Hz, *J'* = 1.6 Hz, 1H, ArH), 10.45 (s, 1H, NH). IR (KBr): v 3273, 3179, 3095, 3052, 2969, 2931, 1668, 1625, 1578, 1517, 1489, 1466, 1388, 1338, 1272, 1242, 1218, 1087, 1013, 957, 832, 790, 768, 689 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>21</sub>H<sub>15</sub>ClN<sub>2</sub>ONa (M + Na<sup>+</sup>) 369.0771, found 369.0790.

**11-(3-Bromophenyl)-8,9-dihydro-7***H***-cyclopenta[***b***][4,7] <b>phenanthrolin-10(11***H***)-one 4d.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.25–2.35 (m, 2H, CH<sub>2</sub>), 2.64–2.77 (m, 2H, CH<sub>2</sub>), 5.71 (s, 1H, CH), 7.11 (d, *J* = 6.4 Hz, 2H, ArH), 7.24 (d, *J* = 6.4 Hz, 1H, ArH), 7.36–7.40 (m, 2H, ArH), 7.55 (d, *J* = 8.8 Hz, 1H, ArH), 7.95 (d, *J* = 8.8 Hz, 1H, ArH), 8.20 (d, *J* = 8.4 Hz, 1H, ArH), 8.68 (d, *J* = 3.2 Hz, 1H, ArH), 10.51 (s, 1H, NH). IR (KBr): v 3281, 3199, 3117, 3067, 3032, 2918, 1672, 1627, 1607, 1520, 1466, 1392, 1323, 1272, 1214, 1174, 1162, 1116, 1073, 1011, 839, 801, 787, 753, 685 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>21</sub>H<sub>16</sub>BrN<sub>2</sub>O (M + H<sup>+</sup>) 391.0446, found 391.0448.

**11-(4-Bromophenyl)-8,9-dihydro-7***H***-cyclopenta[***b***][4,7] <b>phenanthrolin-10(11***H***)-one 4e.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.27–2.34(m, 2H, CH<sub>2</sub>), 2.67–2.73 (m, 2H, CH<sub>2</sub>), 5.69 (s, 1H, CH), 7.12–7.16 (m, 2H, ArH), 7.37–7.38 (m, 3H, ArH), 7.55 (d, *J* = 9.2 Hz, 1H, ArH), 7.95 (d, *J* = 8.8 Hz, 1H, ArH), 8.18 (d, *J* = 8.4 Hz, 1H, ArH), 8.68 (dd, *J* = 4.0 Hz, *J'* = 1.6 Hz, 1H, ArH), 10.48 (s, 1H, NH). IR (KBr): v 3172, 3092, 3049, 2968, 2929, 2856, 1667, 1624, 1575, 1518, 1465, 1415, 1387, 1336, 1271, 1217, 1181, 1157, 1111, 1069, 1010, 956, 832, 788, 767, 729, 687 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>21</sub>H<sub>16</sub>BrN<sub>2</sub>O (M + H<sup>+</sup>) 391.0446, found 391.0443.

**8,9-Dihydro-11**-*p*-tolyl-7*H*-cyclopenta[*b*][4,7]phenanthrolin-**10(11***H***)-one 4f.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.14 (s, 3H, CH<sub>3</sub>), 2.21–2.33 (m, 2H, CH<sub>2</sub>), 2.68–2.70 (m, 2H, CH<sub>2</sub>), 5.61 (s, 1H, CH), 6.95 (d, *J* = 7.6 Hz, 2H, ArH), 7.06 (d, *J* = 8.0 Hz, 2H, ArH), 7.34 (dd, *J* = 8.4 Hz, *J'* = 4.4 Hz, 1H, ArH), 7.54 (d, *J* = 9.2 Hz, 1H, ArH), 7.92 (d, *J* = 9.2 Hz, 1H, ArH), 8.66 (dd, *J* = 8.0 Hz, *J'* = 1.6 Hz, 1H, ArH), 10.40 (s, 1H, NH). IR (KBr): v 3178, 3093, 3044, 3017, 2929, 2854, 1669, 1625, 1586, 1516, 1466, 1416, 1387, 1272, 1219, 1111, 1012, 957, 828, 790, 776, 690 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>21</sub>H<sub>19</sub>N<sub>2</sub>O (M + H<sup>+</sup>) 327.1497, found 327.1495.

**8,9-Dihydro-11-(3-methoxyphenyl)-7***H***-cyclopenta[***b***][<b>4**,7] **phenanthrolin-10(11***H***)-one <b>4g.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.34–2.35 (m, 2H, CH<sub>2</sub>), 2.63–2.73 (m, 2H, CH<sub>2</sub>), 3.64 (s, 3H, CH<sub>3</sub>O), 5.63 (s, 1H, CH), 6.61–6.66 (m, 2H, ArH), 6.80 (s, 1H, ArH), 7.04–7.07 (m, 1H, ArH), 7.37 (dd, *J* = 8.4 Hz, *J'* = 4.0 Hz, 1H, ArH), 7.55 (d, *J* = 8.8 Hz, 1H, ArH), 7.93 (d, *J* = 8.8 Hz, 1H, ArH), 8.22 (d, *J* = 8.4 Hz, 1H, ArH), 8.67 (d, *J* = 3.6 Hz, 1H, ArH), 10.46 (s, 1H, NH). IR (KBr): v 3238, 3170, 3091, 3028, 2930, 2835, 1668, 1627, 1606, 1528, 1487, 1465, 1438, 1394, 1311, 1259, 1217, 1143, 1046, 1013, 831, 689 cm<sup>-1</sup>. HRMS (ESI, m/z): Calcd for C<sub>22</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>Na (M + Na<sup>+</sup>) 365.1266, found 365.1259.

**11-(3,4-Dichlorophenyl)-8,9-dihydro-7***H***-cyclopenta[***b***][<b>4**,7] **phenanthrolin-10(11***H***)-one <b>4h.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.29–2.34 (m, 2H, CH<sub>2</sub>), 2.67–2.73 (m, 2H, CH<sub>2</sub>), 5.76 (m, 1H, CH), 7.03 (dd, *J* = 8.4 Hz, *J'* = 2.0 Hz, 1H, ArH), 7.37–7.42 (m, 2H, ArH), 7.54 (d, *J* = 2.0 Hz, 1H, ArH), 7.56 (d, *J* = 9.2 Hz, 1H, ArH), 7.97 (d, *J* = 9.2 Hz, 1H, ArH), 8.69(dd, *J* = 4.0 Hz, *J'* = 1.6 Hz, 1H, ArH), 10.53 (s, 1H, NH). IR (KBr): v 3177, 3083, 3053, 3020, 2962, 2927, 1668, 1624, 1590, 1511, 1465, 1415, 1388, 1342, 1273, 1241, 1218, 1187, 1132, 1029, 1014, 993, 956, 883, 832, 791, 728, 691 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>21</sub>H<sub>15</sub>Cl<sub>2</sub>N<sub>2</sub>O (M + H<sup>+</sup>) 381.0561, found 381.0574.

**8,9-Dihydro-11-(3,4-dimethylphenyl)-***TH***-cyclopenta**[*b*][**4,7**] **phenanthrolin-10(11***H***)<b>-one 4i.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.04 (s, 6H, 2CH<sub>3</sub>), 2.21–2.33(m, 2H, CH<sub>2</sub>), 2.61–2.72 (m, 2H, CH<sub>2</sub>), 5.55 (s, 1H, CH), 6.83–6.75 (m, 3H, ArH), 7.35 (dd, *J* = 8.0 Hz, *J'* = 4.0 Hz, 1H, ArH), 7.52 (d, *J* = 8.8 Hz, 1H, ArH), 7.91 (d, *J* = 8.8 Hz, 1H, ArH), 8.20 (d, *J* = 8.0 Hz, 1H, ArH), 8.65 (s, 1H, ArH), 10.39 (s, 1H, NH). IR (KBr): v 3237, 3166, 3087, 3034, 2921, 2856, 1665, 1625, 1607, 1576, 1467, 1415, 1396, 1272, 1242, 1215, 1150, 1125, 1112, 1061, 1041, 1012, 990, 957, 828, 803, 781, 760, 719, 700 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>23</sub>H<sub>20</sub>N<sub>2</sub>ONa (M + Na<sup>+</sup>) 363.1473, found 363.1484.

**11-Methylenedioxophenyl-8,9-dihydro-7***H***-cyclopenta[***b***][<b>4**,7] **phenanthrolin-10(11***H***)-one <b>4j**. <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.28–2.31 (m, 2H, CH<sub>2</sub>), 2.67–2.71 (m, 2H, CH<sub>2</sub>), 5.60 (s, 1H, CH), 5.88 (d, *J* = 11.6 Hz, 2H, CH<sub>2</sub>), 6.53 (dd, *J* = 8.0 Hz, *J'* = 1.6 Hz, 1H, ArH), 6.67 (d, *J* = 8.0 Hz, 1H, ArH), 6.78 (d, *J* = 1.6 Hz, 1H, ArH), 7.38 (dd, *J* = 8.4 Hz, *J'* = 4.4 Hz, 1H, ArH), 7.53 (d, *J* = 9.2 Hz, 1H, ArH), 7.92 (d, *J* = 8.8 Hz, 1H, ArH), 8.24 (d, *J* = 8.4 Hz, 1H, ArH), 8.66–8.67 (m, 1H. ArH), 10.45 (s, 1H, NH). IR (KBr): v 3237, 3165, 3086, 3036, 2924, 2853, 1665, 1610, 1536, 1502, 1488, 1467, 1397, 1363, 1254, 1216, 1180, 1036, 922, 829, 810, 792 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>22</sub>H<sub>17</sub>N<sub>2</sub>O<sub>3</sub> (M + H<sup>+</sup>) 357.1239, found 357.1243.

**11-(2,3-Dichlorophenyl)-8,9-dihydro-***TH***-cyclopenta**[*b*][**4,7**] **phenanthrolin-10(11***H***)<b>-one 4k.** <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400 MHz):  $\delta_{\rm H}$  2.20–2.34 (m, 2H, CH<sub>2</sub>), 2.89 (s, 2H, CH<sub>2</sub>), 6.01 (s, 1H, CH), 7.13–7.17 (m, 2H, ArH), 7.35 (d, *J* = 8.8 Hz, 1H, ArH), 7.42 (dd, *J* = 8.8 Hz, *J'* = 4.4 Hz, 1H, ArH), 7.53 (d, *J* = 9.2 Hz, 1H, ArH), 7.92–7.95 (m, 1H, ArH), 8.06 (d, *J* = 8.4 Hz, 1H, ArH), 8.67 (d, *J* = 3.6 Hz, 1H, ArH), 10.59 (s, 1H, NH). IR (KBr): v 3233, 3165, 3082, 3015, 2928, 2860, 1673, 1625, 1594, 1523, 1464, 1417, 1389, 1270, 1240, 1219, 1174, 1155, 1086, 1042, 1013, 957, 827, 738, 709, 635 cm<sup>-1</sup>. HRMS (ESI, *m/z*): Calcd for C<sub>21</sub>H<sub>15</sub>Cl<sub>2</sub>N<sub>2</sub>O (M + H<sup>+</sup>) 381.0561, found 381.0565.

**11-(2,4-Dichlorophenyl)-8,9-dihydro-7***H***-cyclopenta[***b***][4,7] <b>phenanthrolin-10(11***H***)-one 41.** <sup>1</sup>H NMR (DMSO- $d_6$ , 400 MHz):  $\delta_{\rm H}$  2.19–2.34 (m, 2H, CH<sub>2</sub>), 2.73 (m, 2H, CH<sub>2</sub>), 5.92 (s, 1H, CH), 7.14 (d, *J* = 8.0 Hz, 1H, ArH), 7.22 (dd, *J* = 8.4 Hz, *J'* = 1.6 Hz, 1H, ArH), 7.41 (dd, *J* = 8.4 Hz, *J'* = 4.4 Hz, 1H, ArH), 7.48 (d, *J* = 1.6 Hz, 1H, ArH), 7.51 (d, *J* = 8.8 Hz, 1H, ArH), 7.92 (d, *J* = 8.8 Hz, 1H, ArH), 8.07 (d, *J* = 8.8 Hz, 1H, ArH), 8.66–8.67 (m, 1H, ArH), 10.56 (s, 1H, NH). IR (KBr): v 3238, 3172, 3095, 3028, 2961, 2920, 1670, 1625, 1598, 1526, 1466, 1437, 1390, 1269, 1239, 1221, 1097, 1044, 1013, 957, 845, 827, 769, 690, 645 cm<sup>-1</sup>. HRMS (ESI, m/z): Calcd for C<sub>21</sub>H<sub>15</sub>Cl<sub>2</sub>N<sub>2</sub>O (M + H<sup>+</sup>) 381.0561, found 381.0539.

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