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An Efficient Method for the Synthesis of Benzo[*f*]quinoline and Benzo[*a*]phenanthridine Derivatives Catalyzed by Iodine by a Three-Component Reaction of Arenecarbaldehyde, Naphthalen-2-amine, and Cyclic Ketone

Xiang-Shan Wang,*[a,b] Qing Li,[a] Chang-Sheng Yao,[a] and Shu-Jiang Tu[a,b]

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A mild, efficient, and general method for the synthesis of benzo[*f*]quinoline and benzo[*a*]phenanthridine derivatives by a three-component reaction of arenecarbaldehyde, naphthalen-2-amine, and cyclic ketone using iodine as catalyst is described. A possible reaction mechanism for the formation of the product is proposed based on further experimental results.

Introduction

Multicomponent reactions (MCRs) are of paramount importance in the context of green chemistry. They offer a convenient strategy for the rapid and convergent construction of complex organic molecules without the need to isolate and purify the intermediates, resulting in a substantial minimization of waste, labor, time, and cost. Thus, MCRs have become increasingly popular in the last decade.[1] Benzo[*f*]quinoline and its derivatives are very useful compounds in various fields of chemistry, including biological and pharmacological chemistry. Some of these compounds exhibit antibacterial activity,[2] UDP (uridine diphosphate) glucuronosyl transferase activity,[3] inhibitory activity of recombinant human type 1 and 2 steroid 5a-reductase in vitro,[4] antimicrobial activity,[5] antimalarial activity,[6] agonistic activity at postjunctional α_1-receptors,[7] antipsychotic activity,[8] and peripheral dopaminergic activity in cat cardioaccelerator nerve assays.[9]

In view of the importance of benzo[*f*]quinoline and its derivatives, several methods for the synthesis of benzo[*f*]-quinoline and its derivatives have been developed by Kozlov and co-workers[10] and other groups.[11] However, in spite of their potential utility, many of these reported methods suffer from drawbacks such as harsh reaction conditions, unsatisfactory yields, prolonged reaction times, and cumbersome procedures for product isolation. The development of an efficient, mild, and high-yielding process for the preparation of biologically active benzo[*f*]quinoline derivatives appeared interesting.

In recent years, the use of iodine in organic synthesis has received considerable attention as an inexpensive, nontoxic, readily available, mild Lewis acid catalyst for organic transformations, such as the dehydration of tertiary alcohols to alkenes,[12] the synthesis of alkyl benzyl ethers,[13] mixed ethers under hydrogen pressure,[14] and biindoles,[15] the deprotection of acetals,[16] esterification,[17] transesterification,[18] Michael addition,[19] and many other transformations.[20,21]

To the best of our knowledge, the literature records only a few reports describing the synthesis of benzo[*f*]quinolines from arenecarbaldehyde, naphthalen-2-amine, and cyclic ketone. Kozlov and Basalaeva[22] recently reported the formation of benzo[*a*]phenanthridines by a multistep reaction; arenecarbaldehyde and naphthalen-2-amine in EtOH were first converted into the Schiff base, which then reacted with cyclohexanone catalyzed by HCl to form benzo[*f*]quinolines. However, this protocol suffered from the disadvantages of multistep reactions, including low yields, which were obtained with cycloheptanone as the substrate in 1971[23a] and with methylcyclohexanone in 2004.[23b] The work presented herein is part of our continuing drive to prepare heterocyclic compounds by multicomponent reactions.[24] Herein we report the successful three-component reaction of arenecarbaldehyde, naphthalen-2-amine, and cyclic ketone catalyzed by iodine in THF without having to isolate and purify the intermediates, affording benzo[*f*]quinolines or benzo[*a*]phenanthridines in good-to-high yields, including benzo[*f*]cyclopenta[*c*]quinoline, benzo[*a*]phenanthridine, benzo[*f*]cyclohepta[*c*]quinoline, benzo[*f*]cycloocta[*c*]quinoline, and benzo[*f*]cyclododeca[*c*]quinoline derivatives.

[a] School of Chemistry and Chemical Engineering, Xuzhou Normal University,
Xuzhou Jiangsu 221116, P. R. China
Fax: +86-516-83500065
E-mail: xswang1974@yahoo.com
[b] The Key Laboratory of Biotechnology on Medical Plants, Xuzhou, Jiangsu 221116, P. R. China
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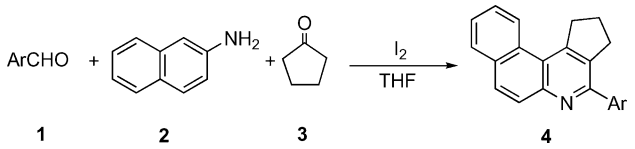
Results and Discussion

The treatment of arenecarbaldehyde **1**, naphthalen-2-amine (**2**), and cyclopentanone (**3**) in THF in the presence of 5 mol-% iodine at reflux afforded the corresponding benzo[*f*]cyclopenta[*c*]quinoline derivatives **4** in high yields (Scheme 1).



Scheme 1.

In our initial study, the reaction of 2-chlorobenzaldehyde (**1a**), naphthalen-2-amine (**2**), and cyclopentanone (**3**) was used as a model reaction to optimize the conditions. The reaction was first carried out in THF in the absence of I_2. No reaction occurred at room temperature and under reflux conditions (Table 1, Entries 1 and 2). Similar reactions were then attempted in the presence of 5, 10, and 20 mol-% of I_2. The results in Table 1 (Entries 5–7) show that the use of 5 mol-% of I_2 at reflux in THF is sufficient to push the reaction forward. Higher loading of the catalyst had no significant influence on the reaction yield. To find the optimum reaction temperature, the reaction was carried out with 5 mol-% of I_2 at room temperature, 50 °C, and at reflux temperature, which resulted in the isolation of **4a** in a trace amount and 80 and 91% yields (Table 1, Entries 3–5), respectively. Thus, 5 mol-% of I_2 and a reaction temperature at reflux were the optimal conditions. In addition, CH_3CN, benzene, DMF, and $ClCH_2CH_2Cl$ (Table 1, Entries 8–11) were also tested as the solvents. In these cases, product **4a** was formed in slightly lower yields (Table 1, Entries 8–11).

Table 1. Synthesis of **4a** under different reaction conditions.[a]

Entry	Temp. [°C]	I_2 [mol-%]	Solvent	Yield [%][b]
1	room temp.	0	THF	0
2	reflux	0	THF	0
3	room temp.	5	THF	trace
4	50	5	THF	80
5	reflux	5	THF	91
6	reflux	10	THF	89
7	reflux	20	THF	91
8	reflux	5	CH_3CN	81
9	reflux	5	benzene	86
10	80	5	DMF	78
11	reflux	5	$ClCH_2CH_2Cl$	81

[a] Reagents and conditions: **1** (2 mmol), **2** (2 mmol, 0.286 g), **3** (2 mmol, 0.168 g), solvent (10 mL). [b] Isolated yields.

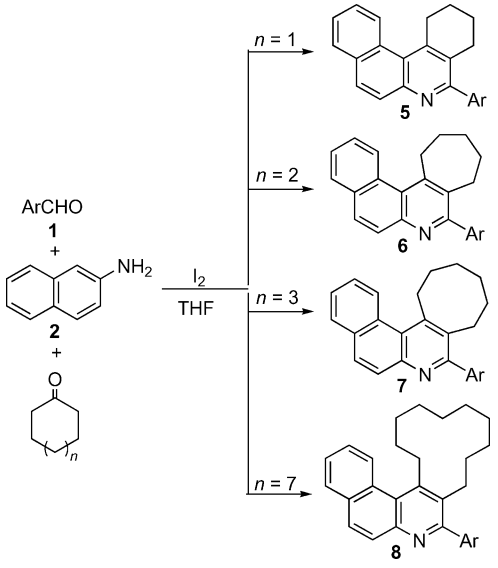
Under the optimized conditions, various benzaldehydes **1** reacted with naphthalen-2-amine (**2**) and cyclopentanone (**3**) to give 4-aryl-2,3-dihydro-1*H*-benzo[*f*]cyclopenta[*c*]-quinolines **4** in high yields within a few hours. The results are summarized in Table 2. It can be observed that the process tolerates both electron-donating and -withdrawing substituents in the benzaldehydes. In all cases, the reactions proceeded efficiently at reflux under mild conditions to afford the corresponding benzo[*f*]cyclopenta[*c*]quinolines in high yields. However, we failed to obtain the expected products when other amines such as naphthalen-1-amine or *p*-toluidine were used. We think a possible reason for this is that *p*-toluidine or naphthalen-1-amine are less reactive than naphthalene-2-amine. All the products were characterized by ^1H and ^{13}C NMR and IR spectroscopy and elemental analyses.

Table 2. I_2-catalyzed reactions of benzaldehyde, naphthalen-2-amine, and cyclopentanone in THF.[a]

Entry	Ar	Product	Time [h]	Yield [%][b]
1	2-ClC_6H_4	**4a**	7	91
2	2-thienyl	**4b**	8	82
3	2,4-$Cl_2C_6H_3$	**4c**	8	88
4	3-ClC_6H_4	**4d**	9	87
5	4-BrC_6H_4	**4e**	9	88
6	4-FC_6H_4	**4f**	8	96
7	3-BrC_6H_4	**4g**	8	87
8	4-ClC_6H_4	**4h**	8	89
9	2-$O_2NC_6H_4$	**4i**	6	90
10	2,3-$(H_3CO)_2C_6H_3$	**4j**	12	86
11	4-$H_3CC_6H_4$	**4k**	10	84

[a] Reagents and conditions: **1** (2 mmol), **2** (2 mmol, 0.286 g), **3** (2 mmol, 0.168 g), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

As expected, the reaction could be extended to other cyclic ketones. Cyclohexanone, -heptanone, and -octanone were also chosen to react with benzaldehyde and naphthalen-2-amine (Scheme 2) and were found to generate the corresponding benzo[*a*]phenanthridines **5a**–**5k**, benzo[*f*]cyclohepta[*c*]quinolines **6a**–**6k**, and benzo[*f*]cycloocta[*c*]quinolines **7a**–**7d** in good-to-high yields, respectively. Even with cyclododecanone, the desired reaction took place successfully to afford benzo[*f*]cyclododeca[*c*]quinolines **8a**,**8b** in satisfactory yields. The results are summarized in Table 3.



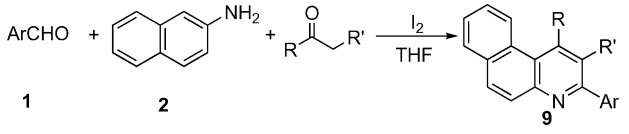
Scheme 2.

Table 3. I_2-catalyzed reactions of benzaldehyde, naphthalen-2-amine, and cyclic ketones in THF.[a]

Entry	Ar	*n*	Product	Time [h]	Yield [%][b]
1	3-ClC_6H_4	1	**5a**	8	86
2	4-BrC_6H_4	1	**5b**	9	89
3	3-BrC_6H_4	1	**5c**	10	92
4	4-FC_6H_4	1	**5d**	8	90
5	2-ClC_6H_4	1	**5e**	8	96
6	2-thienyl	1	**5f**	10	87
7	2,3-$Cl_2C_6H_3$	1	**5g**	8	90
8	4-ClC_6H_4	1	**5h**	10	84
9	4-$O_2NC_6H_4$	1	**5i**	8	86
10	4-$H_3COC_6H_4$	1	**5j**	12	84
11	3-HOC_6H_4	1	**5k**	14	78
12	3-ClC_6H_4	2	**6a**	8	89
13	2,4-$Cl_2C_6H_3$	2	**6b**	8	90
14	3-BrC_6H_4	2	**6c**	10	86
15	4-ClC_6H_4	2	**6d**	12	85
16	4-BrC_6H_4	2	**6e**	12	86
17	4-FC_6H_4	2	**6f**	10	86
18	4-$H_3COC_6H_4$	2	**6g**	12	88
19	3,5-$(H_3CO)_2C_6H_3$	2	**6h**	12	82
20	2,3-$(H_3CO)_2C_6H_3$	2	**6i**	12	79
21	4-$O_2NC_6H_4$	2	**6j**	8	85
22	2-$O_2NC_6H_4$	2	**6k**	8	79
23	4-BrC_6H_4	3	**7a**	16	76
24	3-BrC_6H_4	3	**7b**	20	72
25	4-ClC_6H_4	3	**7c**	18	80
26	3-ClC_6H_4	3	**7d**	16	71
27	3-BrC_6H_4	7	**8a**	18	73
28	4-ClC_6H_4	7	**8b**	20	76

[a] Reagents and conditions: **1** (2 mmol), **2** (2 mmol, 0.286 g), **3** (2 mmol), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

In order to show the general scope of this iodine-catalyzed reaction, we carried out the reaction with acetone or propionaldehyde instead of cyclic ketone (Scheme 3). The reaction proceeded smoothly as expected in high yields. The results are summarized in Table 4.



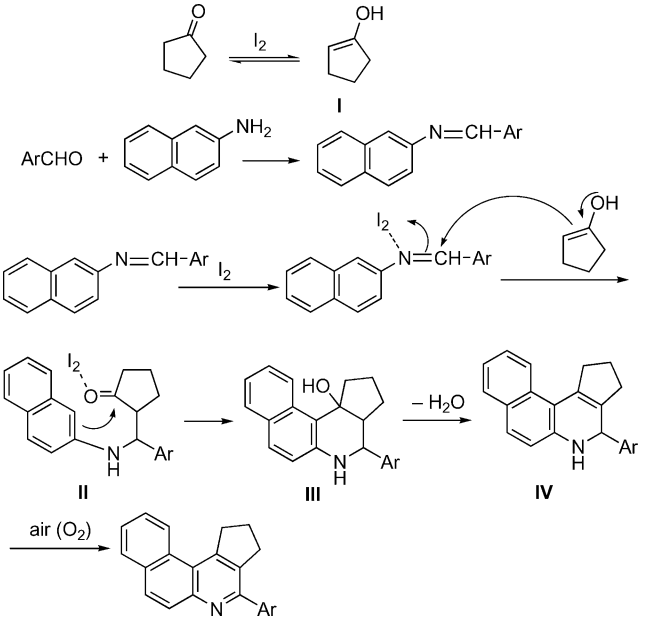
Scheme 3.

Table 4. I_2-catalyzed reactions of benzaldehyde, naphthalen-2-amine, and acetone or propionaldehyde in THF.[a]

Entry	Ar	R	R′	Product	Time [h]	Yield [%][b]
1	2,3-$Cl_2C_6H_3$	Me	H	**9a**	8	92
2	2-FC_6H_4	Me	H	**9b**	8	90
3	3-$O_2NC_6H_4$	H	Me	**9c**	6	84
4	4-$H_3COC_6H_4$	H	Me	**9d**	6	86

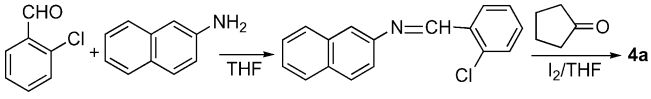
[a] Reagents and conditions: **1** (2 mmol), **2** (2 mmol, 0.286 g), acetone or propionaldehyde (2.5 mmol), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

In accord with the literature,[25] we think that iodine catalyzes the reaction as a mild Lewis acid. The mechanism proposed is shown in Scheme 4. In the presence of iodine, cyclopentanone is in equilibrium with the enol form **I**. The enol immediately attacks the iodine-activated Schiff base to form intermediate **II**, which then undergoes an intramolecular Friedel–Crafts cyclization to give **III**. The subsequent dehydration of **III** results in dihydroquinoline **IV**, which is then oxidized by air to afford aromatized benzo[*f*]cyclopenta[*c*]quinoline **4**.



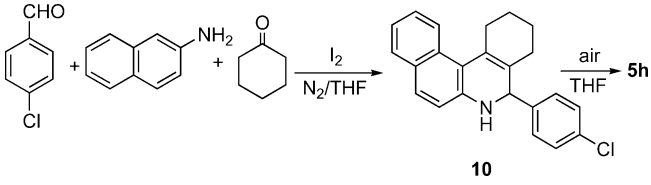
Scheme 4.

To verify the mechanism, we performed each step individually. The Schiff base, *N*-arylidenenaphthalen-2-amine, was obtained in 92% yield when 2-chlorobenzaldehyde (**1**, Ar = 2-ClC_6H_4) was treated with naphthalen-2-amine in THF at ambient temperature (Scheme 5). As expected, the Schiff base reacted smoothly with cyclopentanone to give the corresponding 4-(2-chlorophenyl)-2,3-dihydro-1*H*-benzo[*f*]cyclopenta[*c*]quinoline (**4a**) at reflux in the presence of 5 mol-% of iodine. This result indicates that the Schiff base was formed during the reaction. However, the overall yield was low (71%) in this multistep reaction, as mentioned above.



Scheme 5.

It should be noted that oxygen is important for the last aromatization step. In order to gain more insight into the reaction mechanism, we performed the three-component reaction with 4-chlorobenzaldehyde, naphthalen-2-amine, and cyclohexanone under dry N_2 (Scheme 6): Unaromatized



Scheme 6.

product **10** was obtained in 82% yield. Subsequent treatment of intermediate **10** in THF at reflux in the absence of I_2 in air gave the corresponding aromatized compound **5h** in 80% yield.

Conclusion

We have found an efficient method for the synthesis of benzo[*f*]quinoline and benzo[*a*]phenanthridine derivatives by a three-component reaction of arenecarbaldehyde, naphthalen-2-amine, and cyclic ketone using 5 mol-% of iodine as the catalyst. The features of this procedure are mild reaction conditions, high yields, operational simplicity, and the environmental friendlyness.

Experimental Section

General: Melting points were determined in open capillaries and are uncorrected. IR spectra were recorded with a TENSOR 27 spectrometer using KBr pellets. ^{1}H and ^{13}C NMR spectra were obtained in [D_6]DMSO or $CDCl_3$ with Me_4Si as the internal standard with a Bruker 400 spectrometer. Elemental analyses were carried out with a Perkin-Elmer 2400 II analyzer.

General Procedure for the Synthesis of Benzo[*f*]quinolines 4, 6–8 and Benzo[*a*]phenanthridines 5: A dry 50 mL flask was charged with arenecarbaldehyde (2.0 mmol), naphthalen-2-amine (2.0 mmol, 0.286 g), cyclic ketone (2.0 mmol), I_2 (0.1 mmol, 0.026 g), and THF (10 mL). The reaction mixture was stirred at reflux for 6–20 h. After completion of the reaction, as indicated by TLC, a little DMF was added to the mixture until all the yellow solid had dissolved. Crystals formed when the mixture was cooled to room temperature and were collected by filtration to give **4**, **5**, **6**, **7**, or **8**.

4-(2-Chlorophenyl)-2,3-dihydro-1*H*-benzo[*f*]cyclopenta[*c*]quinoline (4a): Pale-yellow crystals (0.599 g, 91%), m.p. 165–167 °C. IR (KBr): $\tilde{\nu}$ = 3047, 2956, 2849, 1593, 1560, 1487, 1474, 1449, 1428, 1371, 1296, 1281, 1260, 1124, 1086, 1051, 1036, 982, 861, 825, 789, 756, 739, 703, 673 cm^{-1}. ^{1}H NMR ([D_6]DMSO, 400 MHz): δ = 2.21–2.27 (m, 2 H, CH_2), 2.90 (t, J = 7.2 Hz, 2 H, CH_2), 3.83 (t, J = 7.2 Hz, 2 H, CH_2), 7.51–7.56 (m, 3 H, ArH), 7.62–7.65 (m, 1 H, ArH), 7.73–7.81 (m, 2 H, ArH), 7.95 (d, J = 9.2 Hz, 1 H, ArH), 8.08–8.12 (m, 2 H, ArH), 8.78 (d, J = 8.0 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 25.2, 31.1, 37.5, 123.7, 126.5, 126.6, 126.8, 127.0, 128.8, 129.2, 129.6, 129.7, 130.2, 130.6, 130.8, 132.7, 132.8, 138.3, 139.2, 147.7, 150.1, 153.7 ppm. $C_{22}H_{16}ClN$ (329.82): calcd. C 80.11, H 4.89, N 4.25; found C 80.01, H 4.92, N 4.80.

2,3-Dihydro-4-(2-thienyl)-1*H*-benzo[*f*]cyclopenta[*c*]quinoline (4b): Pale-yellow crystals (0.496 g, 82%), m.p. 176–178 °C. IR (KBr): $\tilde{\nu}$ = 3072, 2955, 2857, 1605, 1552, 1530, 1486, 1450, 1436, 1324, 1261, 1221, 1068, 983, 945, 864, 832, 749, 721, 675 cm^{-1}. ^{1}H NMR ([D_6]-DMSO, 400 MHz): δ = 2.27–2.34 (m, 2 H, CH_2), 3.38 (t, J = 7.2 Hz, 2 H, CH_2), 3.76 (t, J = 7.2 Hz, 2 H, CH_2), 7.25–7.27 (m, 1 H, ArH), 7.68–7.76 (m, 4 H, ArH), 7.89 (d, J = 9.2 Hz, 1 H, ArH), 8.04–8.07 (m, 2 H, ArH), 8.70 (d, J = 8.4 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 25.2, 33.0, 37.1, 122.8, 126.37, 126.42, 126.6, 127.0, 127.9, 128.0, 128.8, 129.0, 130.2, 130.6, 132.5, 134.6, 145.6, 147.7, 148.1, 151.2 ppm. $C_{20}H_{15}NS$ (301.40): calcd. C 79.70, H 5.02, N 4.65; found C 79.88, H 4.92, N 4.75.

5-(3-Chlorophenyl)-1,2,3,4-tetrahydrobenzo[*a*]phenanthridine (5a): Pale-yellow crystals (0.593 g, 86%), m.p. 139–140 °C. IR (KBr): $\tilde{\nu}$ = 3054, 2939, 2860, 1595, 1561, 1475, 1446, 1433, 1410, 1342, 1318, 1255, 1134, 1077, 1044, 978, 884, 829, 799, 773, 747, 714, 700 cm^{-1}. ^{1}H NMR ([D_6]DMSO, 400 MHz): δ = 1.79–1.85 (m, 4 H, 2CH_2), 2.82 (t, J = 6.4 Hz, 2 H, CH_2), 3.59 (t, J = 5.6 Hz, 2 H, CH_2), 7.51–7.54 (m, 3 H, ArH), 7.62 (s, 1 H, ArH), 7.70–7.73 (m, 2 H, ArH), 7.83 (d, J = 8.8 Hz, 1 H, ArH), 8.04 (d, J = 8.4 Hz, 1 H, ArH), 8.08–8.10 (m, 1 H, ArH), 8.83 (d, J = 8.8 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 22.2, 23.2, 28.4, 33.4, 125.1, 125.7, 126.4, 127.1, 128.1, 128.5, 128.8, 129.0, 129.06, 129.11, 129.7, 130.1, 130.3, 133.5, 134.3, 143.0, 145.5, 146.5, 157.7 ppm. $C_{23}H_{18}ClN$ (343.85): calcd. C 80.34, H 5.28, N 4.07; found C 80.45, H 5.29, N 4.00.

5-(2-Chlorophenyl)-1,2,3,4-tetrahydrobenzo[*a*]phenanthridine (5e): Pale-yellow crystals (0.659 g, 96%), m.p. 168–170 °C. IR (KBr): $\tilde{\nu}$ = 3056, 2928, 2863, 1594, 1560, 1513, 1475, 1435, 1370, 1342, 1320, 1261, 1227, 1211, 1054, 1032, 1005, 967, 950, 872, 830, 750, 704, 681 cm^{-1}. ^{1}H NMR ([D_6]DMSO, 400 MHz): δ = 1.71–1.86 (m, 4 H, 2CH_2), 2.57–2.61 (m, 2 H, CH_2), 3.54–3.67 (m, 2 H, CH_2), 7.45–7.54 (m, 3 H, ArH), 7.61–7.63 (m, 1 H, ArH), 7.72–7.74 (m, 2 H, ArH), 7.83 (d, J = 8.8 Hz, 1 H, ArH), 8.05 (d, J = 9.2 Hz, 1 H, ArH), 8.09–8.11 (m, 1 H, ArH), 8.05 (d, J = 9.2 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 22.1, 23.3, 27.5, 33.5, 125.4, 125.7, 126.3, 127.2, 128.5, 128.9, 129.2, 129.4, 129.6, 129.7, 130.1, 130.21, 130.24, 132.7, 133.5, 140.1, 145.1, 146.6, 157.3 ppm. $C_{23}H_{18}ClN$ (343.85): calcd. C 80.34, H 5.28, N 4.07; found C 80.30, H 5.44, N 4.12.

8-(3-Chlorophenyl)-10,11,12,13-tetrahydro-9*H*-benzo[*f*]cyclohepta-[*c*]quinoline (6a): Pale-yellow crystals (0.638 g, 89%), m.p. 101–102 °C. IR (KBr): $\tilde{\nu}$ = 3058, 2925, 2853, 1594, 1557, 1480, 1440, 1416, 1384, 1368, 1338, 1256, 1234, 1216, 1165, 1091, 1076, 1045, 986, 891, 865, 832, 802, 785, 758, 740, 715, 700 cm^{-1}. ^{1}H NMR ([D_6]DMSO, 400 MHz): δ = 1.74 (br., 2 H, CH_2), 1.94 (br., 2 H, CH_2), 2.03 (br., 2 H, CH_2), 2.89–2.91 (m, 2 H, CH_2), 3.39–3.41 (m, 2 H, CH_2), 7.41–7.43 (m, 1 H, ArH), 7.49 (d, J = 5.2 Hz, 2 H, ArH), 7.56 (s, 1 H, ArH), 7.63–7.65 (m, 2 H, ArH), 7.72 (d, J = 8.8 Hz, 1 H, ArH), 7.94 (d, J = 9.2 Hz, 1 H, ArH), 7.99–8.01 (m, 1 H, ArH), 8.35–8.38 (m, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 25.9, 27.4, 30.6, 31.7, 32.8, 124.7, 125.7, 126.6, 127.4, 127.9, 128.0, 128.6, 128.7, 129.4, 129.5, 129.8, 130.3, 133.2, 134.3, 135.2, 143.3, 146.7, 150.4, 156.2 ppm. $C_{24}H_{20}ClN$ (357.88): calcd. C 80.55, H 5.63, N 3.91; found C 80.68, H 5.70, N 3.80.

8-(2,4-Dichlorophenyl)-10,11,12,13-tetrahydro-9*H*-benzo[*f*]cyclohepta[*c*]quinoline (6b): Pale-yellow crystals (0.705 g, 90%), m.p. 161–163 °C. IR (KBr): $\tilde{\nu}$ = 3054, 2922, 2850, 1586, 1552, 1473, 1445, 1379, 1355, 1320, 1236, 1216, 1141, 1098, 1082, 1048, 1033, 988, 953, 905, 858, 832, 811, 780, 748 cm^{-1}. ^{1}H NMR ([D_6]DMSO, 400 MHz): δ = 1.70 (br., 2 H, CH_2), 1.94–2.06 (m, 4 H, 2CH_2), 2.78 (d, J = 6.4 Hz, 2 H, CH_2), 3.41–3.46 (m, 1 H, CH), 3.55–3.59 (m, 1 H, CH), 7.50 (d, J = 8.4 Hz, 1 H, ArH), 7.58 (dd, J = 8.0, 1.6 Hz, 1 H, ArH), 7.69–7.79 (m, 4 H, ArH), 8.01 (d, J = 9.2 Hz, 1 H, ArH), 8.06–8.09 (m, 1 H, ArH), 8.47–8.49 (m, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 25.9, 27.3, 30.8, 31.9, 33.1, 125.4, 125.8, 126.7, 127.4, 127.9, 128.6, 128.7, 129.3, 129.8, 130.3, 131.7, 133.2, 134.0, 134.5, 136.4, 139.0, 146.9, 150.2, 154.5 ppm. $C_{24}H_{19}Cl_2N$ (392.32): calcd. C 73.47, H 4.88, N 3.57; found C 73.40, H 4.97, N 3.80.

8-(4-Bromophenyl)-9,10,11,12,13,14-hexahydrobenzo[*f*]cycloocta[*c*]-quinoline (7a): Pale-yellow crystals (0.635 g, 76%), m.p. 240–242 °C. IR (KBr): $\tilde{\nu}$ = 3055, 2922, 2848, 1587, 1557, 1542, 1500, 1476, 1436, 1392, 1362, 1327, 1269, 1253, 1176, 1102, 1085, 1067, 1005, 943, 862, 840, 805, 763, 744, 717 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.55–1.58 (m, 2 H, CH_2), 1.65–1.74 (m, 4 H, 2CH_2),

2.27–2.30 (m, 2 H, CH_2), 2.96–2.99 (m, 2 H, CH_2), 3.64 (s, 2 H, CH_2), 7.41 (d, J = 8.0 Hz, 2 H, ArH), 7.64 (d, J = 8.4 Hz, 2 H, ArH), 7.67–7.72 (m, 2 H, ArH), 7.92–7.99 (m, 3 H, ArH), 8.85 (d, J = 8.0 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 25.8, 27.3, 28.0, 30.7, 31.0, 31.1, 122.1, 124.8, 126.3, 126.4, 127.7, 129.3, 129.5, 130.1, 130.4, 130.6, 131.3, 132.9, 133.5, 141.2, 147.4, 148.7, 158.3 ppm. $C_{25}H_{22}BrN$ (416.35): calcd. C 72.12, H 5.33, N 3.36; found C 72.25, H 5.20, N 3.55.

8-(3-Bromophenyl)-9,10,11,12,13,13,14-hexahydrobenzo[*f*]cycloocta[*c*]-quinoline (7b): Pale-yellow crystals (0.600 g, 72%), m.p. 142–144 °C. IR (KBr): $\tilde{\nu}$ = 3063, 2922, 2856, 1594, 1556, 1545, 1492, 1474, 1449, 1435, 1405, 1376, 1348, 1269, 1255, 1232, 1070, 1011, 890, 848, 836, 806, 799, 767, 752, 722, 702, 678 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.53–1.55 (m, 2 H, CH_2), 1.63–1.74 (m, 4 H, $2CH_2$), 2.23–2.29 (m, 2 H, CH_2), 2.94–2.97 (m, 2 H, CH_2), 3.61 (s, 2 H, CH_2), 7.33–7.37 (m, 1 H, ArH), 7.43 (d, J = 7.6 Hz, 1 H, ArH), 7.58 (d, J = 8.0 Hz, 1 H, ArH), 7.64–7.69 (m, 3 H, ArH), 7.89–7.97 (m, 3 H, ArH), 8.82 (d, J = 7.6 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 25.8, 27.3, 28.0, 30.7, 31.0, 31.1, 122.4, 124.8, 126.3, 126.5, 127.4, 127.8, 129.3, 129.5, 129.7, 130.1, 130.7, 130.9, 131.8, 132.9, 133.5, 144.2, 147.3, 148.8, 157.9 ppm. $C_{25}H_{22}BrN$ (416.35): calcd. C 72.12, H 5.33, N 3.36; found C 72.28, H 5.30, N 3.50.

8-(3-Bromophenyl)-9,10,11,12,13,14,15,16,17,18-decahydrobenzo-[*f*]cyclododeca[*c*]quinoline (8a): Pale-yellow crystals (0.694 g, 73%), m.p. 205–207 °C. IR (KBr): $\tilde{\nu}$ = 3057, 2927, 2905, 2863, 2841, 1557, 1542, 1492, 1472, 1430, 1409, 1374, 1357, 1293, 1252, 1229, 1190, 1159, 1069, 996, 876, 844, 815, 783, 765, 753, 695, 685 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.35–1.68 (m, 14 H, $7CH_2$), 1.94–1.96 (m, 2 H, CH_2), 2.92–2.94 (m, 2 H, CH_2), 3.55–3.59 (m, 2 H, CH_2), 7.33–7.37 (m, 1 H, ArH), 7.46 (d, J = 8.4 Hz, 1 H, ArH), 7.57 (d, J = 8.0 Hz, 1 H, ArH), 7.62–7.69 (m, 3 H, ArH), 7.87–7.94 (m, 3 H, ArH), 8.77–8.79 (m, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 23.0, 23.4, 27.0, 27.1, 27.4, 27.9, 28.4, 28.9, 30.7, 122.4, 125.0, 126.2, 126.5, 127.1, 127.4, 129.2, 129.3, 129.7, 130.4, 130.6, 130.9, 131.9, 133.2, 133.3, 144.2, 147.1, 148.2, 158.3 ppm. $C_{29}H_{32}BrN$ (474.48): calcd. C 83.41, H 6.80, N 2.95; found C 73.53, H 6.70, N 2.81.

8-(4-Chlorophenyl)-9,10,11,12,13,14,15,16,17,18-decahydrobenzo-[*f*]cyclododeca[*c*]quinoline (8b): Pale-yellow crystals (0.653 g, 76%), m.p. 218–220 °C. IR (KBr): $\tilde{\nu}$ = 2919, 2854, 1594, 1557, 1544, 1490, 1467, 1436, 1394, 1380, 1335, 1263, 1234, 1089, 1014, 949, 867, 841, 828, 748, 725, 706, 688 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.34–1.68 (m, 14 H, $7CH_2$), 1.94–1.97 (m, 2 H, CH_2), 2.89–2.96 (m, 2 H, CH_2), 3.55–3.60 (m, 2 H, CH_2), 7.47 (s, 4 H, ArH), 7.62–7.65 (m, 2 H, ArH), 7.87–7.94 (m, 3 H, ArH), 8.78 (d, J = 8.4 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 23.0, 23.4, 27.0, 27.1, 27.4, 27.9, 28.4, 28.8, 30.7, 124.9, 126.2, 126.4, 127.1, 128.4, 129.2, 129.3, 130.2, 130.4, 130.5, 133.27, 133.30, 133.8, 140.6, 147.1, 148.1, 158.7 ppm. $C_{29}H_{32}ClN$ (430.02): calcd. C 81.00, H 7.50, N 3.26; found C 80.79, H 7.55, N 3.40.

General Procedure for the Synthesis of Benzo[*f*]quinolines 9: A dry 50 mL flask was charged with arenecarbaldehyde (2.0 mmol), naphthalen-2-amine (2.0 mmol, 0.286 g), acetone or propionaldehyde (2.5 mmol), I_2 (0.1 mmol, 0.026 g), and THF (10 mL). The reaction mixture was stirred at 50 °C for 6–8 h. After completion of the reaction, as indicated by TLC, a little DMF was added to the mixture until all the yellow solid had dissolved. Crystals formed when the mixture was cooled to room temperature and were collected by filtration to give **9**.

3-(2,3-Dichlorophenyl)-1-methylbenzo[*f*]quinoline (9a): Pale-yellow crystals (0.624 g, 92%), m.p. 201–203 °C. IR (KBr): $\tilde{\nu}$ = 3052, 2978, 2870, 1681, 1606, 1578, 1549, 1485, 1446, 1412, 1387, 1353, 1254, 1193, 1047, 1025, 866, 834, 794, 744, 708 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 3.20 (s, 3 H, CH_3), 7.33–7.37 (m, 1 H, ArH), 7.56 (dd, J = 8.0, 1.6 Hz, 1 H, ArH), 7.61 (dd, J = 7.6, 1.6 Hz, 1 H, ArH), 7.65–7.72 (m, 3 H, ArH), 7.97–8.01 (m, 2 H, ArH), 8.05 (d, J = 8.8 Hz, 1 H, ArH), 8.87 (d, J = 8.0 Hz, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 26.9, 125.0, 126.3, 126.5, 126.7, 127.5, 127.7, 129.1, 129.3, 129.9, 130.5, 130.7, 131.1, 131.4, 133.1, 133.7, 141.5, 145.0, 149.5, 155.1 ppm. $C_{20}H_{13}Cl_2N$ (338.23): calcd. C 71.02, H 3.87, N 4.14; found C 70.87, H 3.92, N 4.20.

2-Methyl-3-(3-nitrophenyl)benzo[*f*]quinoline (9c): Pale-yellow crystals (0.528 g, 84%), m.p. 187–188 °C. IR (KBr): $\tilde{\nu}$ = 3085, 2960, 1533, 1487, 1449, 1383, 1344, 1094, 1032, 895, 867, 828, 811, 749, 687 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 2.61 (s, 3 H, CH_3), 7.26–7.73 (m, 3 H, ArH), 7.93–8.04 (m, 4 H, ArH), 8.30–8.33 (m, 1 H, ArH), 8.57–8.58 (m, 1 H, ArH), 8.64 (d, J = 8.0 Hz, 1 H, ArH), 8.84 (s, 1 H, ArH) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 20.7, 122.8, 123.1, 124.4, 125.0, 127.2, 127.5, 127.9, 128.77, 128.81, 129.1, 129.4, 130.7, 132.0, 132.9, 135.3, 142.3, 146.5, 148.3, 156.4 ppm. $C_{20}H_{14}N_2O_2$ (314.34): calcd. C 76.42, H 4.49, N 8.91; found C 76.25, H 4.59, N 8.80.

General Procedure for the Synthesis of 5-(4-Chlorophenyl)-1,2,3,4,5,6-hexahydrobenzo[*a*]phenanthridine (10): A dry 50 mL flask was charged with 4-chlorobenzaldehyde (2.0 mmol, 0.281 g), naphthalen-2-amine (2.0 mmol, 0.286 g), cyclohexanone (2.0 mmol), I_2 (0.1 mmol, 0.026 g), and THF (10 mL). The reaction mixture was stirred at reflux under dry N_2 for 6 h. After completion of the reaction, as indicated by TLC, pale-yellow crystals formed when the mixture was cooled to room temperature and were collected by filtration to give **10** (0.566 g, 82%), m.p. 166–168 °C. IR (KBr): $\tilde{\nu}$ = 3419, 3387, 3054, 2934, 2855, 2830, 1633, 1611, 1517, 1480, 1409, 1386, 1338, 1316, 1294, 1274, 1239, 1208, 1182, 1143, 1117, 1104, 1084, 1012, 917, 831, 812, 748, 696 cm^{-1}. ^{1}H NMR ([D$_6$]DMSO, 400 MHz): δ = 1.09–1.17 (m, 1 H, CH), 1.46–1.51 (m, 2 H, CH_2), 1.68–1.72 (m, 1 H, CH_2), 2.16–2.21 (m, 1 H, CH), 2.37–2.42 (m, 1 H, CH), 2.47–2.50 (m, 1 H, CH_2), 4.27 (d, J = 10.8 Hz, 1 H, CH), 6.25 (s, 1 H, CH), 6.50 (s, 1 H, NH), 6.91 (d, J = 8.4 Hz, 1 H, ArH), 7.07–7.11 (m, 1 H, ArH), 7.29–7.32 (m, 1 H, ArH), 7.45–7.48 (m, 5 H, ArH), 7.63 (d, J = 8.0 Hz, 1 H, ArH), 8.19 (d, J = 8.4 Hz, 1 H, ArH) ppm. ^{13}C NMR ([D$_6$]DMSO, 100 MHz): δ = 20.0, 26.2, 26.3, 37.5, 62.7, 111.0, 117.5, 120.7, 123.1, 125.1, 126.1, 127.7, 128.0, 128.48, 128.54, 130.0, 131.4, 132.2, 141.9, 142.2 ppm. $C_{23}H_{20}ClN$ (345.86): calcd. C 79.87, H 5.83, N 4.05; found C 79.99, H 5.92, N 3.92.

Supporting Information (see footnote on the first page of this article): Detailed experimental procedures, melting points, elemental analyses, and spectroscopic data (^{1}H and ^{13}C NMR, IR) for **4c**–**4k**, **5b**–**5d**, **5f**–**5k**, **6c**–**6k**, **7c**–**7d**, **9b**, and **9d**.

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