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SYNTHESIS OF PHENANTHRIDINES AND RELATED COMPOUNDS BY PALLADIUM-CATALYZED DIRECT COUPLING VIA C–H AND N–H BOND CLEAVAGES

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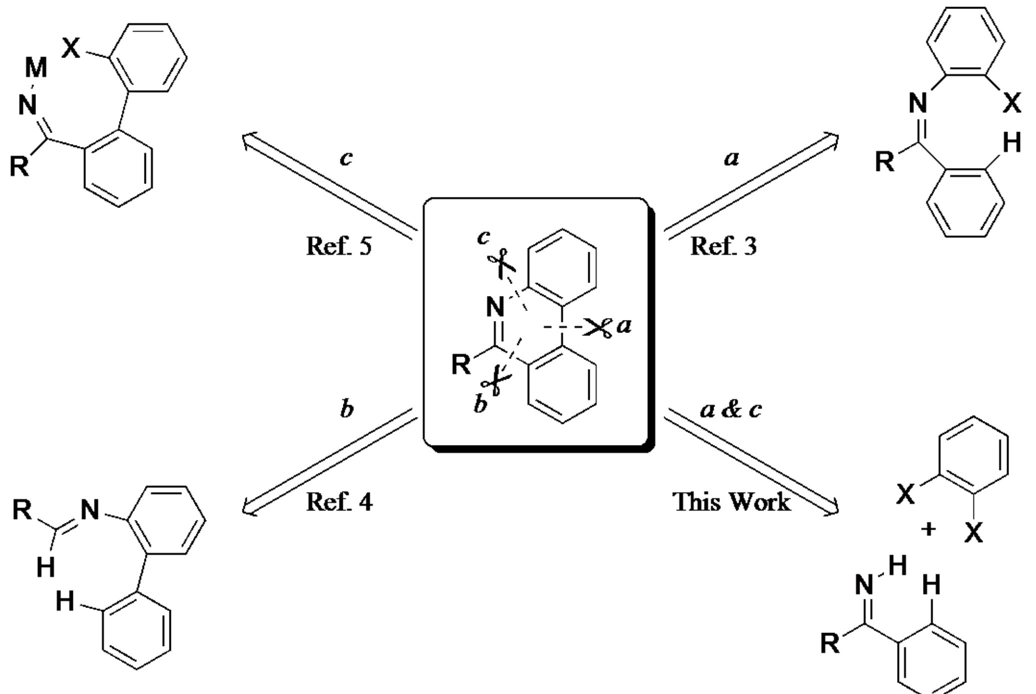
Abstract – The palladium-catalyzed direct annulation of benzophenone imines with *o*-dihalobenzenes proceeds through C–H and N–H bond cleavages to produce 6-arylphenanthridine derivatives. 2-Phenyl-imidazole, -benzimidazole, and -indole also undergo the annulation to form the corresponding tetra- and pentacyclic compounds.

INTRODUCTION

Nitrogen-containing fused heteroaromatic compounds including phenanthridines can be seen in a wide range of π-conjugated functional materials such as organic semiconductors and luminescent materials as well as pharmaceuticals and natural products.[1] Among them, 6-arylphenanthridine derivatives are of particular interest due to their applicability as ligands for phosphorescent iridium complexes, which are promising emitters in pure red region.[2] Therefore, their effective construction has attracted much attention and a number of synthetic approaches through transition-metal-catalyzed cyclization reactions have been developed (Scheme 1). However, in reported procedures shown as routes *a*,[3] *b*,[4] and *c*,[5] starting materials have been prepared through complicated multistep processes.

Dedicated to Professor Ei-ichi Negishi on the occasion of his 77th birthday.



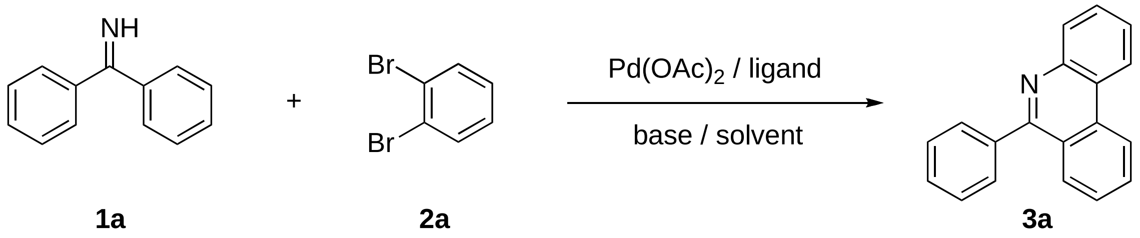
Scheme 1

In contrast, the annulation between two aromatic substrates (routes *a & c*) appears to be attractive because of the substrate availability and applicability.[6] In the course of our recent studies on transition-metal-catalyzed annulation reactions involving C–H bond cleavage,[7] we succeeded in conducting the one step synthesis of 6-arylphenanthridines through the direct coupling of benzophenone imines with *o*-dihalobenzenes under palladium catalysis, accompanied by C–H and N–H bond cleavages. The catalyst was also found to be applicable to the related annulation of 2-phenyl-imidazoles and -indole. These new findings are described herein.

RESULTS AND DISCUSSION

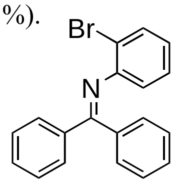
In an initial attempt, benzophenone imine (**1a**, 0.5 mmol) was treated with *o*-dibromobenzene (**2a**, 0.4 mmol) in the presence of $Pd(OAc)_2$ (0.02 mmol), PCy_3 (0.04 mmol), and K_2CO_3 (0.9 mmol) as a catalyst, ligand, and base, respectively, in DMF at 160 °C for 9 h. As a result, 6-phenylphenanthridine (**3a**) was obtained in 53% yield (Table 1, Entry 1). The use of other bases such as Na_2CO_3, Cs_2CO_3, $KOBu^t$, and K_3PO_4 in place of K_2CO_3 decreased the product yield (Entries 2-5). The reaction proceeded more efficiently at 170 °C in DMAc to afford **3a** in 65% yield (Entry 6). Even at 170 °C, the **3a** yield decreased in NMP, diglyme, or mesitylene (Entries 7-9). Without PCy_3, the reaction did not proceed catalytically (Entry 10). Increasing the amount of PCy_3 to 0.08 mmol decreased the product yield (Entry 11). Other mono- (Entries 12 and 13) and diphosphine ligands (Entries 14-17) were less effective than PCy_3. In the case using xantphos (Entry 17), a significant amount (45%) of monobromide **4** was formed together with **3a**.

Table 1. Reaction of benzophenone imine (**1a**) with *o*-dibromobenzene (**2a**)[a]



Entry	Ligand (mmol)[b]	Base	Solvent	Temp. /°C	Time /h	Yield of **3a** /%[c]
1	PCy_3 (0.04)	K_2CO_3	DMF	160	9	53
2	PCy_3 (0.04)	Na_2CO_3	DMF	160	9	11
3	PCy_3 (0.04)	Cs_2CO_3	DMF	160	9	23
4	PCy_3 (0.04)	$KOBu^t$	DMF	160	9	0
5	PCy_3 (0.04)	K_3PO_4	DMF	160	9	41
6	PCy_3 (0.04)	K_2CO_3	DMAc	170	6	65 (64)
7	PCy_3 (0.04)	K_2CO_3	NMP	170	6	51
8	PCy_3 (0.04)	K_2CO_3	diglyme	170	9	20
9	PCy_3 (0.04)	K_2CO_3	mesitylene	170	9	16
10	–	K_2CO_3	DMAc	170	9	4
11	PCy_3 (0.08)	K_2CO_3	DMAc	170	9	40
12	PCy_2(*o*-biph) (0.04)	K_2CO_3	DMAc	170	6	20
13	PPh_3 (0.04)	K_2CO_3	DMAc	170	6	34
14	dppm (0.02)	K_2CO_3	DMAc	170	6	15
15	dppb (0.02)	K_2CO_3	DMAc	170	9	34
16	dppf (0.02)	K_2CO_3	DMAc	170	6	28
17	xantphos (0.02)	K_2CO_3	DMAc	170	6	16[d]

[a] Reaction conditions: **1a** (0.5 mmol), **2a** (0.4 mmol), $Pd(OAc)_2$ (0.02 mmol), Base (0.9 mmol), Solvent (2.5 mL), under N_2. [b] PCy_3 = tricyclohexylphosphine, PCy_2(*o*-biph) = [1,1'-biphenyl]-2-yldicyclohexylphosphine. [c] GC yield based on the amount of **2a** used. Value in parentheses indicates yield after purification. [d] A monobromide **4** was also formed (45%).



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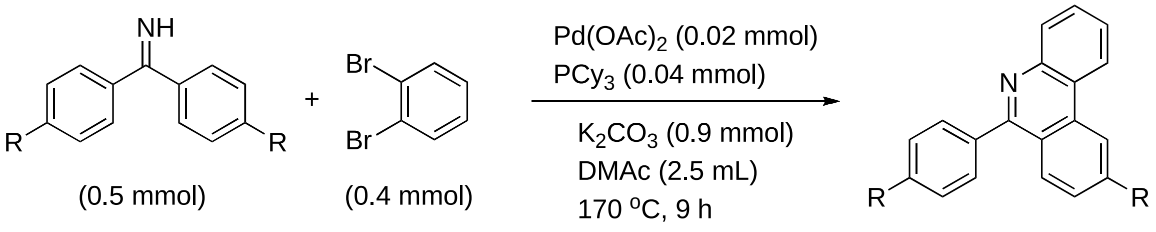
Under the optimized reaction conditions (Table 1, Entry 6), the reactions of a series of *p*-substituted benzophenone imines **1b**-**d** with **2a** were next examined (Scheme 2). 4,4'-Dimethyl- and 4,4'-di-*tert*-butylbenzophenone imines (**1b** and **1c**) coupled with **2a** effectively to form the corresponding phenanthridines **3b** and **3c** in 51 and 60% yields, respectively. In the reaction of 4,4'-dimethoxybenzophenone imine (**1d**), only a small amount of coupling product was detected by GC and GC-MS. The poor result seems to be due, at least in part, to the poorer solubility of **1d** and/or the product.[8]



1b (R = Me)
1c (R = Bu^t)
1d (R = OMe)

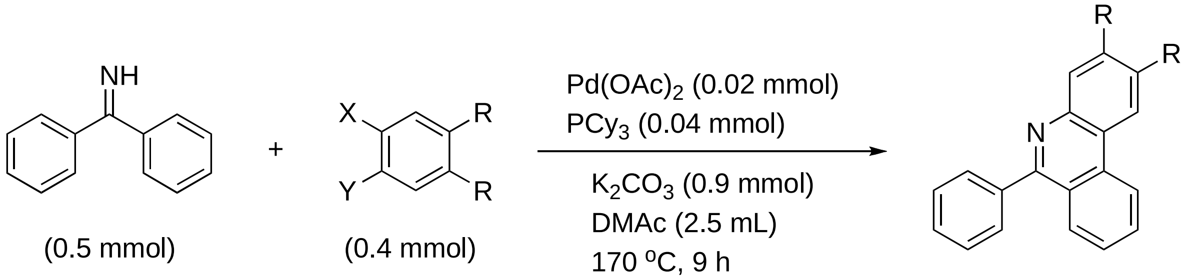
2a

3b (R = Me) (51%)^a
3c (R = Bu^t) 60% (58%)^a
3d (R = OMe)^b 7%^{a}

[a] GC yield based on the amount of **2a** used. Value in parentheses indicates yield after purification.
[b] Detected by GC and GC-MS.

Scheme 2

Under similar conditions, the reactions of **1a** with *o*-bromochlorobenzene (**2b**) and *o*-dichlorobenzene (**2c**) gave **3a** in low yields (Scheme 3). 1,2-Dibromo-4,5-dimethylbenzene (**2d**) coupled with **1a** effectively to produce **3e** in 52% yield. In contrast, the reaction of an electron-deficient dibromide, 1,2-dibromo-4,5-difluorobenzene (**2e**), was sluggish.



1a

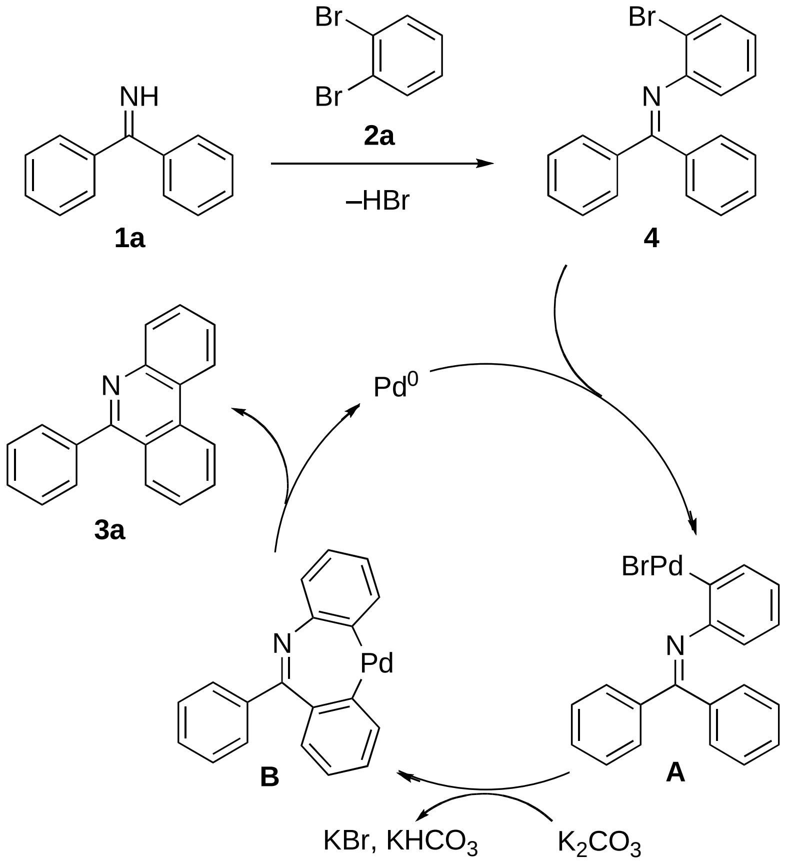
2b (R = H, X = Cl, Y = Br)
2c (R = H, X = Y = Cl)
2d (R = Me, X = Y = Br)
2e (R = F, X = Y = Br)

3a (R = H) 26%^{a}
3a (R = H) 21%^{a}
3e (R = Me) (52)%^{a}
3f (R = F)^b 5%^{a}

[a] GC yield based on the amount of **2** used. Value in parentheses indicates yield after purification.
[b] Detected by GC and GC-MS.

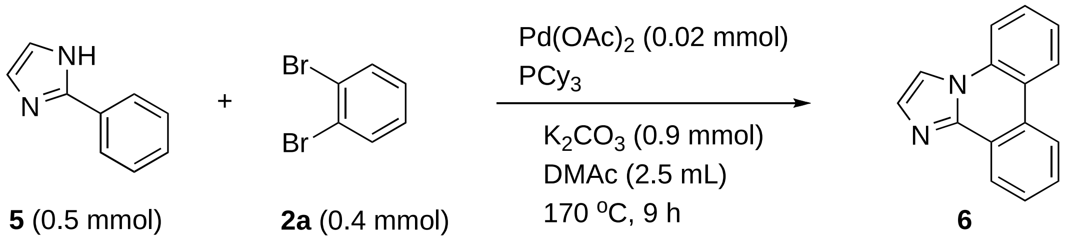
Scheme 3

A plausible mechanism for the coupling of **1a** with **2a** is illustrated in Scheme 4, in which neutral ligands are omitted. Taking account of a fact that the *N*-arylation of **1a** is known to occur under mild conditions at 65 °C,[9] the arylation seems to first occur on the imino nitrogen of **1a** to produce a monobromide **4**. Actually, **4** was obtained in the case using xantphos ligand (Table 1, Entry 17), as described above. Then, the next intramolecular C–H arylation may occur as follows: Oxidative addition of the remaining C–Br bond in **4** to Pd^0 species gives arylpalladium species **A**. Subsequent cyclopalladation and reductive elimination take place to form **3a** and regenerate Pd^0 species.



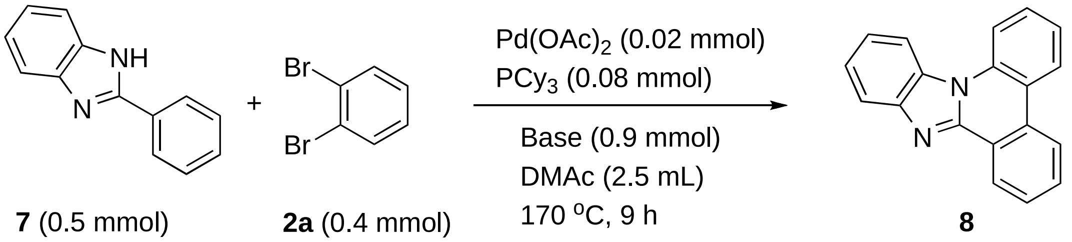
Scheme 4

Under similar conditions, 2-phenylimidazole (**5**) also underwent the annulation with **2a**, accompanied by C–H and N–H bond cleavages to afford a tetracyclic product **6** in 20% yield (Scheme 5). Increase of the amount of PCy_3 ligand to 0.08 mmol improved the product yield up to 57%. Although the exact effect of increased ligand is not clear at the present stage, the deposition of palladium black was observed to be slightly retarded. Similarly, 2-phenylbenzimidazole (**7**) and 2-phenylindole (**9**) also coupled with **2a** under modified conditions using Cs_2CO_3 as a base in place of K_2CO_3 to produce **8** and **10**, respectively. In the latter case, the use of 0.12 mmol of PCy_3 gave the best result.



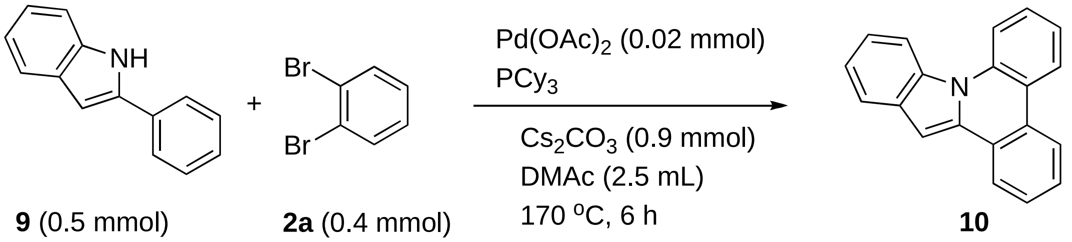
5 (0.5 mmol) **2a** (0.4 mmol) **6**

PCy$_3$ (mmol)	Yield of **6** /%[a]
0.04	20
0.08	57 (55)
0.12	54



7 (0.5 mmol) **2a** (0.4 mmol) **8**

Base	Yield of **8** /%[a]
K_2CO_3	19
Cs_2CO_3	39 (39)



9 (0.5 mmol) **2a** (0.4 mmol) **10**

PCy$_3$ (mmol)	Yield of **10** /%[a]
0.08	43
0.12	62 (61)

[a] GC yield based on the amount of **2a** used. Value in parentheses indicates yield after purification.

Scheme 5

In summary, we have demonstrated that the synthesis of phenanthridine derivatives from the readily available building blocks, benzophenone imines and *o*-dihalobenzenes, can be achieved through C–H and N–H bond cleavages under palladium catalysis. The procedure is also applicable to the construction of related fused tetra- and pentacyclic heteroarenes.

EXPERIMENTAL

General. ^{1}H and ^{13}C NMR spectra were recorded at 400 and 100 MHz for $CDCl_3$ solutions. MS data were obtained by EI. GC analysis was carried out using a silicon OV-17 column (i. d. 2.6 mm x 1.5 m). GC-MS analysis was carried out using a CBP-1 capillary column (i. d. 0.25 mm x 25 m). The structures of all products listed below were unambiguously determined by ^{1}H and ^{13}C NMR with the aid of NOE, COSY, HMQC, and HMBC experiments.

Starting Materials. Imines **1b-d** were prepared according to published procedures.[10] Other starting materials were commercially available.

Typical Procedure for the Reactions of Benzophenone Imines 1, 2-Phenylimidazoles 5 and 7, and 2-Phenylindole 9 with *o*-Dihalobenzene 2. A mixture of nitrogen-containing substrate **1**, **5**, **7**, or **9** (0.5 mmol), *o*-dihalobenzene **2** (0.4 mmol), $Pd(OAc)_2$ (0.02 mmol, 4.5 mg), PCy_3 (0.04-0.12 mmol), base (0.9 mmol), and dibenzyl (ca. 40 mg) as internal standard was stirred in DMAc (2.5 mL) under nitrogen at 170 °C for 6-9 h. GC and GC-MS analyzes of the mixtures confirmed formation of product **3**, **6**, **8**, or **10**. After cooling, the reaction mixture was extracted with ethyl acetate (100 mL). The organic layer was washed with water (100 mL, three times), and dried over Na_2SO_4. After evaporation of the solvent under vacuum, the product **3**, **6**, **8**, or **10** was isolated by column chromatography on silica gel using hexane-ethyl acetate as eluant.

Characterization Data of Products.

6-Phenylphenanthridine (3a):[6d] mp 99-101 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.63 (m, 4H), 7.67-7.78 (m, 4H), 7.84-7.88 (m, 1H), 8.11 (d, *J* = 8.4 Hz, 1H), 8.25 (d, *J* = 7.8 Hz, 1H), 8.62 (d, *J* = 8.2 Hz, 1H), 8.71 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 121.9, 122.2, 123.7, 125.2, 126.9, 127.1, 128.4, 128.7, 128.8, 128.9, 129.7, 130.3, 130.6, 133.4, 139.8, 143.8, 161.3; HRMS m/z (M^+) Calcd for $C_{19}H_{13}N$: 255.1048. Found 255.1045.

9-Methyl-6-(4-methylphenyl)phenanthridine (3b):[6d] mp 70-72 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.47 (s, 3H), 2.64 (s, 3H), 7.36 (d, *J* = 7.3 Hz, 2H), 7.42 (d, *J* = 8.7 Hz, 1H), 7.62-7.66 (m, 3H), 7.69-7.74 (m, 1H), 8.01 (d, *J* = 8.7 Hz, 1H), 8.21 (d, *J* = 8.2 Hz, 1H), 8.46 (s, 1H), 8.58 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.4, 22.2, 121.8, 121.9, 123.45, 123.54, 126.5, 128.6, 128.7, 128.9, 129.0, 129.7, 130.3, 133.6, 137.1, 138.5, 140.8, 144.1, 161.1; HRMS m/z (M^+) Calcd for $C_{21}H_{17}N$: 283.1361. Found 283.1358.

9-(*tert*-Butyl)-6-[4-(*tert*-butyl)phenyl]phenanthridine (3c): mp 178-179 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.41 (s, 9H), 1.50 (s, 9H), 7.56-7.58 (m, 2H), 7.65-7.75 (m, 5H), 8.12 (d, *J* = 8.7 Hz, 1H), 8.23 (d, *J* = 8.7 Hz, 1H), 8.64-8.68 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.3, 31.4, 34.7, 35.5, 117.7, 121.8, 123.4, 123.9, 125.35, 125.38, 126.5, 128.5, 128.8, 129.4, 130.3, 133.2, 137.0, 144.1, 151.6, 153.7, 161.0; HRMS m/z (M^+) Calcd for $C_{27}H_{29}N$: 367.2300. Found 367.2302.

2,3-Dimethyl-6-phenylphenanthridine (3e):[6d] mp 92-94 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 2.50 (s, 3H), 2.55 (s, 3H), 7.48-7.57 (m, 4H), 7.71-7.73 (m, 2H), 7.79-7.83 (m, 1H), 8.01 (s, 1H), 8.07 (d, *J* = 8.2 Hz, 1H), 8.34 (s, 1H), 8.65 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 20.1, 20.4, 121.7, 121.96, 121.98, 125.0, 126.5, 128.4, 128.5, 128.8, 129.8, 130.23, 130.24, 133.2, 136.4, 138.4, 140.0, 142.6, 160.3; HRMS m/z (M$^+$) Calcd for $C_{21}H_{17}N$: 283.1361. Found 283.1359.

2-Bromo-*N*-(diphenylmethylene)aniline (4):[11] mp 97-99 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 6.53 (d, *J* = 7.8 Hz, 1H), 6.76-6.80 (m, 1H), 7.00-7.04 (m, 1H), 7.19-7.27 (m, 5H), 7.40-7.51 (m, 4H), 7.82 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 115.2, 121.2, 124.2, 127.4, 127.9, 128.2, 128.7, 128.8, 129.6, 131.1, 132.4, 136.0, 138.8, 150.2, 169.4; HRMS m/z (M$^+$) Calcd for $C_{19}H_{14}BrN$: 335.0310. Found 335.0308.

Imidazo[1,2-f]phenanthridine (6):[12] mp 115-124 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.49 (td, *J* = 8.2 and 1.4 Hz, 1H), 7.58-7.66 (m, 4H), 7.84 (dd, *J* = 8.2 and 0.9 Hz, 1H), 7.97 (d, *J* = 1.4 Hz, 1H), 8.33-8.37 (m, 1H), 8.43 (dd, *J* = 8.3 and 1.4 Hz, 1H), 8.63-8.97 (m, 1H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 112.0, 115.8, 121.7, 122.3, 123.7, 124.11, 124.12, 125.1, 127.4, 128.55, 128.60, 128.8, 131.5, 131.7, 142.5; HRMS m/z (M$^+$) Calcd for $C_{15}H_{10}N_2$: 218.0844. Found 218.0842.

Benzo[4,5]imidazo[1,2-f]phenanthridine (8):[13] mp 145-147 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.44-7.54 (m, 3H), 7.64-7.73 (m, 3H), 8.03-8.06 (m, 1H), 8.33 (t, *J* = 8.3 Hz, 2H), 8.43 (dd, *J* = 8.0 and 1.2 Hz, 1H), 8.52 (d, *J* = 7.8 Hz, 1H), 8.85 (dd, *J* = 7.8 and 1.8 Hz, 1H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 113.9, 116.0, 120.3, 121.7, 122.3, 122.9, 123.4, 124.1, 124.2, 124.4, 126.0, 128.6, 129.1, 129.5, 130.4, 131.9, 134.4, 144.5, 147.5; HRMS m/z (M$^+$) Calcd for $C_{19}H_{12}N_2$: 268.1000. Found 268.1003.

Indolo[1,2-f]phenanthridine (10):[14] mp 146-148 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.28 (s, 1H), 7.33-7.42 (m, 3H), 7.48-7.53 (m, 2H), 7.57-7.61 (m, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 8.13-8.17 (m, 1H), 8.22-8.25 (m, 1H), 8.34 (d, *J* = 7.8 Hz, 1H), 8.40 (d, *J* = 8.2 Hz, 1H), 8.56 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 96.1, 114.2, 116.3, 121.0, 121.7, 121.95, 122.01, 122.3, 123.0, 123.9, 124.1, 126.1, 126.8, 127.8, 128.1, 128.7, 130.2, 133.8, 135.2, 135.9; HRMS m/z (M$^+$) Calcd for $C_{20}H_{13}N$: 267.1048. Found 267.1044.

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