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# Fused Phenanthridines: Domino Cyclization of 1,6-Diynes with Bromo(iso)quinoline

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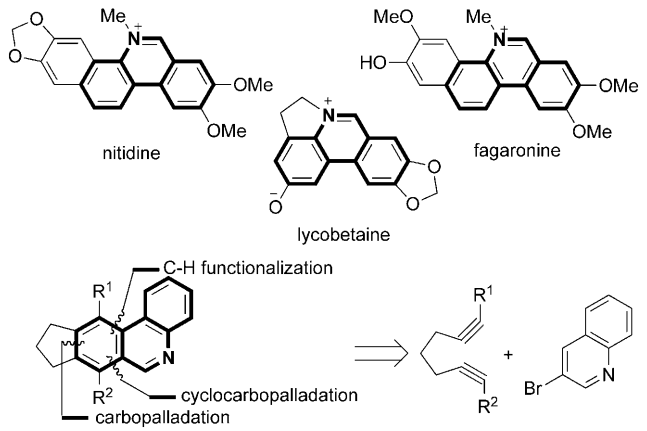
Fused phenanthridines are an important class of heterocycles that show a broad range of biological and pharmaceutical activity.[1] These structural motifs are found in a plethora of different natural products (Scheme 1).[2] Molecules containing phenanthridine are the subject of considerable interest as potent antitumor, antimicrobial, and antiviral agents.[3] Therefore, numerous synthetic strategies for the preparation of these scaffolds have been developed.[4] Recently, Cronin and co-workers[5] developed a new C–C bond-forming annulation reaction leading to a class of 2,3-dihydro-12*H*-pyrrolo-[1,2-*f*]phenanthridine derivatives that exhibit a reversible "lockable molecular switch"/ring-opening-cyclization process. Lautens and co-workers have reported a powerful strategy for the synthesis of diversely substituted phenanthridines and benzo[*c*]phenanthridines involving a sequence of domino *ortho*-functionalization with palladium-catalyzed cross-coupling processes.[6] Much effort has been invested in synthesizing the derivatives of these phenanthridines to deduce structure–activity relationships and discover new analogues with improved properties.[7–9] Notably, Yu and co-workers reported the first example of directed transition-metal-catalyzed C–H functionalizations of a pyridine ring at the 3- or 4-position.[10] Our group has also employed a domino strategy to devise alternative processes for the efficient construction of benzocyclo[penta- to octa-]isoindole.[11]




Scheme 1. Examples of natural products containing phenanthridine alkaloids and retrosynthetic analysis of fused phenanthridines.

We envisioned a C–H activation/cross-coupling approach toward the synthesis of diversely polycyclic phenanthridine derivatives (Scheme 1). This cascade series consists of inter- and intramolecular Heck reactions and a subsequent regioselective direct arylation by C–H activation of the quinoline ring at the 2- or 4-positions. Herein, we report the palladium-catalyzed domino reactions of dimethyl 2,2-bis(3-phenylprop-2-ynyl)malonate (**1a**), diethyl 2,2-bis(3-phenylprop-2-ynyl)malonate (**1b**), 1-ethyl 3-methyl 2,2-bis(3-phenylprop-2-ynyl)malonate (**1c**), dimethyl 2,2-bis(3-(4-chlorophenyl)-prop-2-ynyl)malonate (**1d**), diethyl 2,2-bis(3-(4-chlorophenyl)prop-2-ynyl)malonate (**1e**), 1-ethyl 3-methyl 2,2-bis(3-(4-chlorophenyl)prop-2-ynyl)malonate (**1f**), dimethyl 2,2-bis(3-p-tolylprop-2-ynyl)malonate (**1g**), diethyl 2,2-bis(3-p-tolylprop-2-ynyl)malonate (**1h**), diethyl 2,2-bis(3-(4-methoxyphenyl)prop-2-ynyl)malonate (**1i**), diethyl 2,2-bis(3-(4-fluorophenyl)prop-2-yn-1-yl)malonate (**1j**), and dimethyl 2,2-bis(3-(4-fluorophenyl)prop-2-yn-1-yl)malonate (**1k**) with 3-bromo(iso)quinoline or 3-bromopyridine, in the direct, efficient, and economic construction of phenanthridine and quinoline through both C–C bond coupling and C–H bond activation. Optimization of the reaction conditions was performed using diethyl 2,2-bis(3-(4-chlorophenyl)prop-2-ynyl)-malonate (**1e**) and 3-bromoquinoline (Table 1). Reaction of **1e** with 3-bromoquinoline in *N*,*N*-dimethylformamide

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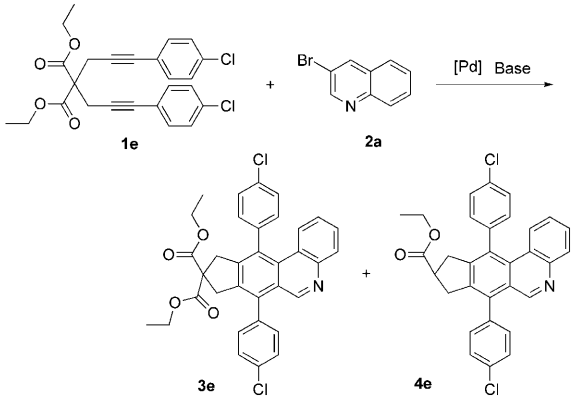
Table 1. Palladium-catalyzed one-pot cascade reaction.




| Entry | [Pd] [mol%] | Base (equiv) | Solvent | $t$ [h] | $T$ [°C] | Yield [%] 3e | 4e |
|---|---|---|---|---|---|---|---|
| 1 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $nBu_3N$ (2) | MeCN | 9 | 110 | – | – |
| 2 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $nBu_3N$ (2) | toluene | 10 | 120 | 15 | trace |
| 3 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $nBu_3N$ (2) | DMF | 11 | 100 | trace | – |
| 4 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $nBu_3N$ (2) | DMF | 11 | 110 | 41 | 14 |
| 5 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $nBu_3N$ (2) | DMF | 16 | 120 | 59 | 30 |
| 6 | $[Pd(OAc)_2]/PPh_3$ (1:2) | $nBu_3N$ (2) | DMF | 16 | 120 | 49 | 12 |
| 7 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $nBu_3N$ (2) | DMF | 14 | 120 | 64 | 25 |
| 8 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $K_2CO_3$ (2) | DMF | 14 | 120 | trace | – |
| 9 | $[Pd(OAc)_2]/PPh_3$ (2:4) | $NaHCO_3$ (2) | DMF | 14 | 120 | trace | – |
| 10 | $[PdCl_2]/PPh_3$ (2:4) | $nBu_3N$ (2) | DMF | 14 | 120 | 19 | 7 |
| 11 | $Pd(PPh_3)_4$ (2) | $Cs_2CO_3$ (2) | DMF | 14 | 120 | 25 | 6 |
| 12 | $[Pd(dba)_2]$ (2) | $nBu_3N$ (2) | DMF | 14 | 120 | 24 | 8 |

(DMF) in the presence of a catalytic amount of $Pd(OAc)_2$ at 120 °C for 14 hours produced diethyl 7,11-bis(4-chlorophenyl)-8H-cyclopenta[*j*]phenanthridine-9,9(10H)-dicarboxylate (**3e**) and ethyl 7,11-bis(4-chlorophenyl)-9,10-dihydro-8H-cyclopenta[*j*]phenanthridine-9-carboxylate (**4e**) in 89% yield. The output of the multi-cascade route to product **3e** was found to be greatly affected by reaction temperature (Table 1, entries 1–4), the additive base (Table 1, entries 8, 9, 11), the catalytic system (Table 1, entries 10–12), and by the solvent used in the reaction (Table 1, entries 1–2). Thus, the following optimized reaction conditions were selected for carrying out the following studies: diyne (1 equiv) was reacted with 3-bromoquinoline (1.2 equiv) in the presence of 2 mol% palladium(II) catalyst and 4 mol% $Ph_3P$ with $nBu_3N$ (2 equiv) as an additive in *N*,*N*-dimethylformamide at 120 °C.

Illustrative examples of the scope of the cascade reaction are shown in Table 2. Various diynes and 3-bromo-(iso)quinoline or 3-bromopyridine were compatible with this palladium-catalyzed domino reaction. A range of 7,11-diphenyl-8H-cyclopenta[*j*]phenanthridine compounds were readily isolated in good to excellent yields, except in the case of **5c** or **5j**, when 1,7-diphenylhepta-1,6-diyne with a variety of substituted groups was employed. The substituted groups could be methoxycarbonyl, ethoxycarbonyl, or *para*-substituted groups on the benzene ring (chloro, fluoro, methyl and methoxyl). Using 3-bromoquinoline with diyne subatrates (**a**-**k**), the reaction affords 7,11-diphenyl-9,10-dihydro-8H-cyclopenta[*j*]phenanthridine **3a+4a**, **3b+4b, 3d+4d**, and **3e+4e**, respectively, in yields beyond 81%. The yields of compound (**3a+4a**) and (**3d+4d**) were the highest at 89%. Simultaneously, when 3-bromo-(iso)quinoline was used in the reaction with **c**, **f**–**k**, the desired phenanthridines were obtained in good yields ranging from 62–78% (**3c**, **3f**, **4g**, **3i**, **3j**, **3k** and **6b**). The reaction of 3-bromoquinoline with diyne having a methoxycarbonyl or ethoxycarbonyl group (**4a**, **4b**, **4d** and **4e**) gave much lower yields of the products than that of **4a**, **4b**, **4d**, and **4e**. Note that the yield of compound **4g** was highest in all deoxycarbonyl decomposition[12] of the desired products at 65%. Interestingly, compound **5c** and **5j** are low (15% and 18%), but this is a novel cyclopenta[*b*]acridine. To test this idea, 3-bromoisoquinoline was employed to give the single compound **6b** (75%). Lastly, with these conditions in hand, the generality of the cyclization has been studied using 3-bromopyridine to take the place of 3-bromoquinoline. Results showed that the deoxycarbonyl cyclopenta[*g*]isoquinolines **7a** and **7d** give moderate yields 68% and 67%, respectively.

The structures of all of the resulting fused phenanthridine compounds were confirmed by various spectroscopic tech-

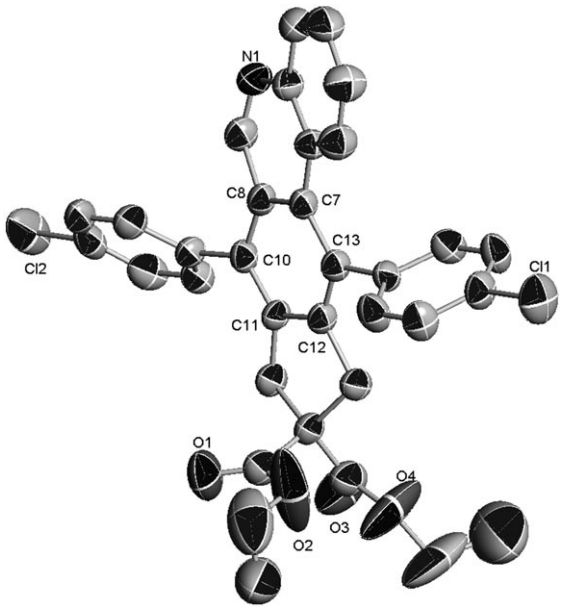
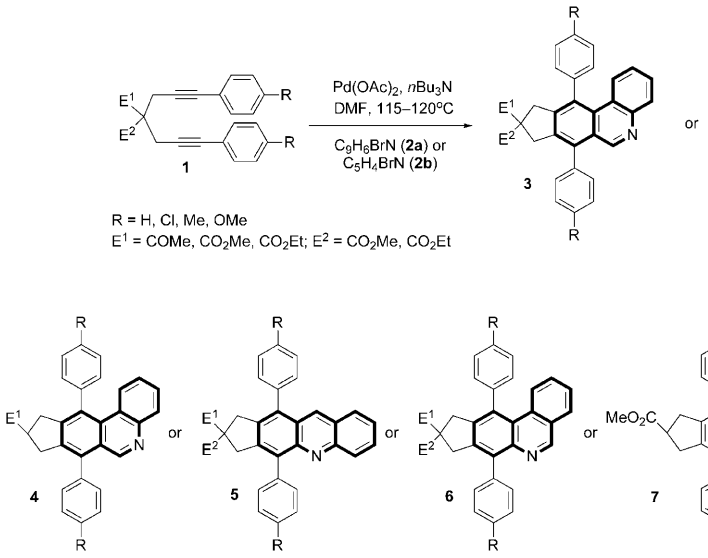



Figure 1. Molecular structure of compound **3e**. Ellipsoids are drawn at 30% probability. Selected bond lengths (Å) and angles (°) C7–C13 1.435(6), C8–C10 1.434(7), C10–C11 1.384(6), C11–C12 1.394(6), C12–C13 1.391(6), C7-C8-C10 122.3(5), C8-C10-C11 117.5(4), C10-C11-C12 120.2(5), C11-C12-C13 123.4(5), C12-C13-C7 117.8(4).

Table 2. The palladium-catalyzed one-pot formation of fused phenanthridines.[a]




| Entry | $E^1$ | $E^2$ | R | Yield [%][b] |
|---|---|---|---|---|
| **3a** | $CO_2Me$ | $CO_2Me$ | – | 52 |
| **3b** | $CO_2Et$ | $CO_2Et$ | – | 61 |
| **3c** | COMe | $CO_2Et$ | – | 62 |
| **3d** | $CO_2Me$ | $CO_2Me$ | Cl | 55 |
| **3e** | $CO_2Et$ | $CO_2Et$ | Cl | 64 |
| **3f** | COMe | $CO_2Et$ | Cl | 78 |
| **3h** | $CO_2Et$ | $CO_2Et$ | Me | 69 |
| **3i** | $CO_2Et$ | $CO_2Et$ | OMe | 62 |
| **3j** | $CO_2Et$ | $CO_2Et$ | F | 65 |
| **3k** | $CO_2Me$ | $CO_2Me$ | F | 63 |
| **4a** | $CO_2Me$ | – | – | 27 |
| **4b** | $CO_2Et$ | – | – | 23 |
| **4d** | $CO_2Me$ | – | Cl | 26 |
| **4e** | $CO_2Et$ | – | Cl | 25 |
| **4g** | $CO_2Me$ | – | Me | 65 |
| **5c** | COMe | $CO_2Et$ | – | 15 |
| **5j** | $CO_2Et$ | $CO_2Et$ | F | 18 |
| **6b** | $CO_2Et$ | $CO_2Et$ | – | 75 |
| **7a** | $CO_2Me$ | – | – | 68 |
| **7d** | $CO_2Me$ | – | Cl | 67 |

[a] Reaction conditions: **1** (1.0 equiv), bromo(iso)quinoline (1.2 equiv), $Pd(OAc)_2$ (2 mol%), $PPh_3$ (4 mol%), $nBu_3N$ (2 equiv), DMF 10 mL, 115–120°C, 14 h. [b] Yield of isolated product after column chromatography on silica gel.

niques ($^1H$ and $^{13}C$ NMR spectroscopy, and UV/Vis, IR, and HRMS). The molecular structure and relative configuration of **3e** was unambiguously confirmed by X-ray diffraction (Figure 1).[13] Further details are provided in the Supporting Information.

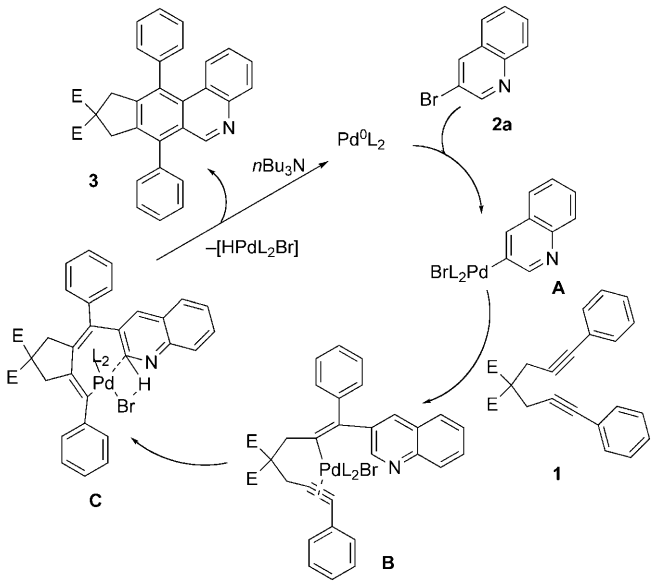
Simplified catalytic cycle mechanisms for the selective formation of polycyclic phenanthridines were proposed in Scheme 2. Oxidative addition of the bromo(iso)quinoline (**2a**) would afford aryl-palladium intermediate **A** which can subsequently undergo carbopalladation with the diyne moiety (**1**) to yield intermediate **B**, which then reacts through a carbopalladation reaction with the carbon–carbon double bond and σ-bond metathesis onto the aryl group via intermediate **C**. Proton-abstraction[14] by the base affords polycyclic phenanthridine **3**.

In conclusion, we have developed a practical and efficient domino method for the preparation of diversely fused phenanthridines from readily available precursors through the multistep C–C bond-formation and the C–H activation of the quinoline or isoquinoline ring. This series produces diphenylcyclopentaphenanthridine derivatives in moderate to very good yields and excellent regioselectivities. This methodology will certainly aid the effort to generate more-interesting structures for use in biological studies. Further investigation to understand this catalytic transformation, evaluation of the process with a broader scope of substrates, and the synthesis of more-complex π-system heterocycles are underway.

## Experimental Section

*Typical Procedure for the Palladium-Catalyzed Domino Reaction of 1,6-Diynes with Bromo(iso)quinoline*

Diyne **1f** (1.0 equiv), 3-bromoquinoline **2a** (1.2 equiv), $Pd(OAc)_2$ (2 mol%), and $PPh_3$ (4 mol%) were added to the degassed solution of $nBu_3N$ (2 equiv) in *N,N*-dimethylformamide (DMF; 5 mL). and the mixture was stirred at room temperature for 40 min then heated at 115–120°C for 18 h. The reaction mixture was cooled, quenched with water, and extracted with EtOAc (3×5 mL). The combined organic layers were washed with hydrochloric acid (5%), aqueous sodium carbonate (5%), and saturated aqueous sodium chloride solution. After separation, the organic layer was dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. Flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 6:1) afforded **3f** (78%) as a yellow powder; 431 mg (78% yield); m.p. 224–225°C; $R_f = 0.36$; $^1H$ NMR (300 MHz, $CDCl_3$): $\delta = 9.03$ (s, 1H, Ar–H), 8.08 (d, 1H, $J = 7.5$ Hz, Ar–H), 7.57 (s, 6H, Ar–H), 7.38 (d, 2H, $J = 7.8$ Hz, Ar–H), 7.32 (d, 2H, $J = 7.2$ Hz, Ar–H), 7.19 (d, 1H, $J = 6.9$ Hz, Ar–H), 4.18 (q, 2H, $J = 6.9$ Hz, $CH_3CHH$–O–), 3.49 (s, 2H, $CH_2$), 3.38 (s, 2H, $CH_2$), 2.15 (s, 3H, $CH_3CO$), 1.23 ppm (t, $J = 6.9$ Hz, 3H, $CH_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$): $\delta = 201.9$, 171.7, 151.4, 145.3, 143.9, 139.9, 138.3, 135.9, 135.4, 134.2, 133.9, 133.6, 131.3, 131.2, 130.6, 130.1, 129.0, 128.0, 126.6, 125.9, 125.3, 124.0, 66.2, 62.1, 39.8, 38.7, 26.1, 14.0 ppm; UV/Vis ($CH_3CN$): 261 nm; FT-IR (KBr): $\tilde{\nu}_{max} = 1739$, 1710, 1244, 1093, 1014, 817, 773, 524 $cm^{-1}$; HRMS (ESI): $m/z$ $[M+H]^+$ calcd. for $C_{33}H_{26}Cl_2NO_3$: 554.1290; found: 554.1303.




Scheme 2. Proposed catalytic cycle.

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**Keywords:** C–H activation · cyclization · domino reactions · fused phenanthridines · palladium

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[13] Crystal structure determination: $C_{34}H_{27}Cl_2NO_4$ **3e**, M = 584.47, triclinic, space group P-1, $a = 8.996(2)$, $b = 26.334(6)$, $c = 12.674(3)$ Å, U = 2853.4(1) Å$^3$, $T = 293$ K, $Z = 4$, 2023 reflections measured, 6597 unique ($R_{int} = 0.0819$) which were used in all calculations. The final $wR(F^2)$ was 0.1778 (all data). CCDC 792043 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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