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N-Bromosuccinimide-Induced C−H Bond Functionalization: An Intramolecular Cycloaromatization of Electron Withdrawing Group Substituted 1-Biphenyl-2-ylethanone for the Synthesis of 10-Phenanthrenol

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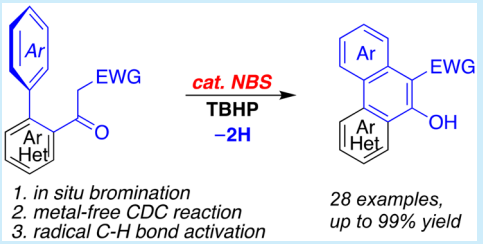
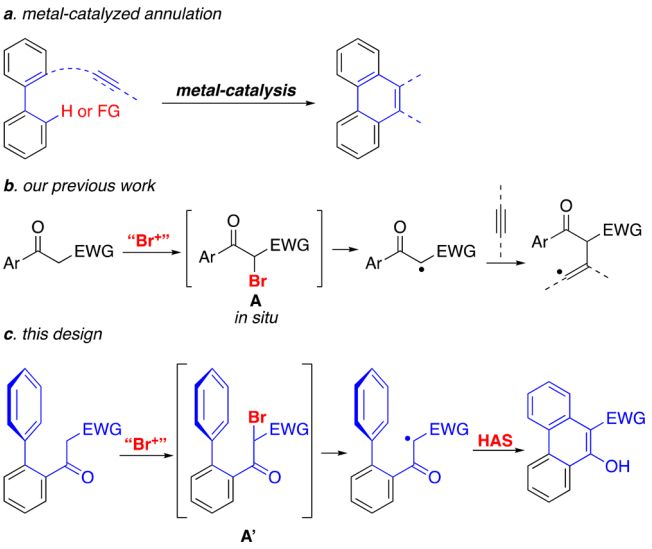
Supporting Information

ABSTRACT: An NBS-induced intramolecular cycloaromatization for the synthesis of 10-phenanthrenols from electron-withdrawing group substituted 1-biphenyl-2-ylethanones is described. The in situ generated bromide was designed to act as an initiator for the radical C−H bond activation. An oxidative cross-dehydrogenative coupling reaction of a highly active C−H bond with an inert C−H bond readily occurs under mild conditions without the need for transition metals or strong oxidants.



Natural products containing a phenanthrene skeleton show various biological activities[1] such as antitumor,[2] antimicrobial,[3] antiallergic,[4] and antimalarial activities.[5] Further, many synthetic phenanthrene frameworks are frequently found in organic photoelectronic materials,[6] carbon nanotubes,[7] superconducting materials,[8] and chelating ligands.[9] The traditional approaches toward the synthesis of phenanthrene include photocyclization[10] and McMurry coupling reaction.[11] These early procedures are not always satisfying, and they suffer from side reactions, low selectivity, and harsh conditions. To address these drawbacks, much attention has been paid to the development of new methods for the synthesis of phenanthrene.[12] Most research efforts have focused on the transition-metal-catalyzed annulation reaction of functionalized biphenyls with alkenes or alkynes (Scheme 1, a). A number of elegant cycloaromatization reactions such as Ru-,[13] Mo-,[14] or Fe[15]-catalyzed olefin metathesis, Fe-,[16] Pd-,[17] Cu-,[18] or Ir[19]-catalyzed [4 + 2] benzannulation, and Pt-,[20] Au-,[21] Ag-,[22] or Cr[23]-catalyzed intramolecular annulation have been reported. Recently, we have reported an efficient bromide-induced cycloaromatization of arylethanone derivatives with alkynes, which is applicable to the synthesis of naphthols and carbazoles.[24] During our studies on the reaction mechanism, a key intermediate **A** was obtained from arylethanone and "Br^+" through bromination (Scheme 1, b). α-Arylethanone radical was then formed from bromide **A** and initiated the whole radical cycle. This observation led us to design a new synthetic strategy for the construction of a phenanthrene skeleton. The similar α-bromination of 2-acetyl biphenyls may occur to give a bromide intermediate **A′**, which could provide the corresponding radical intermediate. An intramolecular homolytic aromatic substitution (HAS) reaction enables it to close the ring (Scheme 1, c).[25] On the basis of this design

Scheme 1. Reported Methods and Our Design for the Phenanthrene Synthesis



principle, we report a bromide-induced cross-dehydrogenative coupling(CDC) reaction for the synthesis of 10-phenanthrenols. This metal-free CDC reaction affords the annulation products in good to excellent yields by utilizing the readily available EWG-substituted 1-biphenyl-2-ylethanones as starting materials and NBS as a catalyst under mild conditions. A cross-coupling reaction between a highly active C−H bond and an inert C−H bond was achieved.

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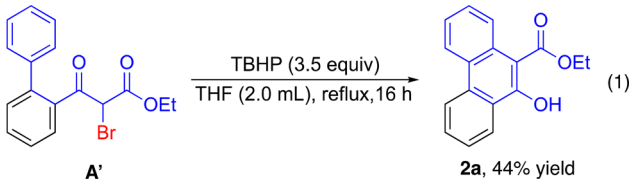
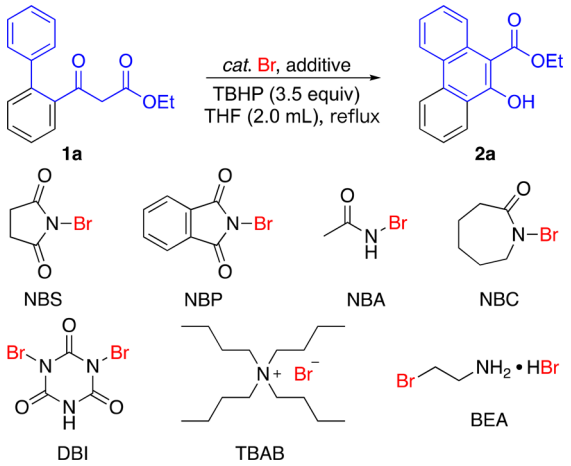
To validate our hypothesis in Scheme 1, c, an initial experiment was performed using ethyl 2-bromo-3-([1,1′-biphenyl]-2-yl)-3-oxopropanoate **A′** and TBHP in refluxing THF. As expected, the desired phenanthrene **2a** was obtained in 44% isolated yield (eq 1). The catalytic cycloaromatization



reaction was then conducted under bromide-induced conditions using ethyl 3-([1,1′-biphenyl]-2-yl)-3-oxopropanoate **1a** instead of **A′** as the starting material (Table 1). As shown in

Table 1. Screening of the Reaction Conditions[a]

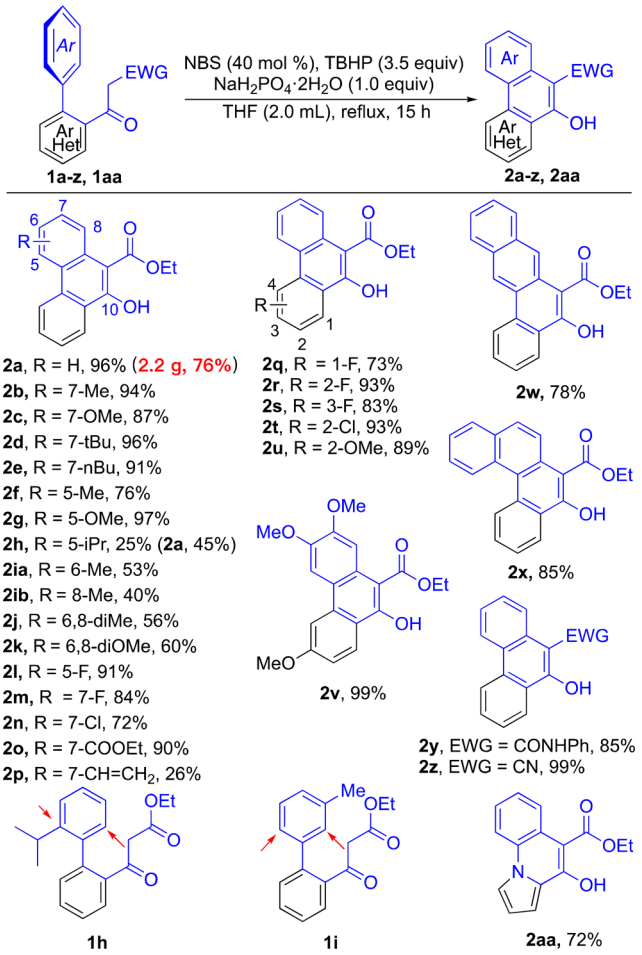


entry	cat. Br	additive	yield (%)
1	Br_2		49
2	NBS		72
3	NBP		69
4	NBA		10
5	NBC		64
6	DBI		20
7	TBAB		10
8	HBr		39
9	KBr		10
10	BEA		25
11	$Ph_3P{\cdot}HBr$		47
12	NBS	AcOH	78
13	NBS	citric acid	82
14	NBS	$NaH_2PO_4{\cdot}2H_2O$	96
15	Br_2	$NaH_2PO_4{\cdot}2H_2O$	78

[a]Reaction conditions: **1a** (0.25 mmol), bromine catalyst (40 mol %), additive (1.0 equiv), TBHP (3.5 equiv, 70% aqueous solution), under air; isolated yields.

Table 1, several well-known brominating reagents such as Br_2, *N*-bromosuccinimide (NBS), *N*-bromophthalimide (NBP), *N*-bromoacetamide (NBA), and *N*-bromo-ε-caprolactam (NBC) were investigated first (Table 1, entries 1−5). Among all of these bromine reagents, NBS gave the best result, and the desired product **2a** was obtained in 72% yield (Table 1, entry 2). Dibromoisocyanuric acid (DBI) was also examined in the transformation. Although DBI belongs to the *N*,*N*-dibromoamides, partial cycloaromatization and low yield of **2a** were observed (Table 1 entry 6). Other readily available bromine sources led to the formation of **2a** in only low to moderate yields (Table 1, entries 7−11). It has been reported that the mildly acidic or buffered reaction conditions are necessary for efficient oxidative halogenation.[26] We found that the addition of an acid did promote the present reaction (Table 1, entries 12−14). An excellent yield of **2a** was obtained in the presence of 1.0 equiv of $NaH_2PO_4{\cdot}2H_2O$ (Table 1 entry 14). When Br_2 was used under the acidic conditions, a lower yield of **2a** was observed. (entry 15). With the optimal conditions in hand, the reaction was further conducted on multigram scale, which allowed the formation of 2.2 g of **2a** in 76% yield (Scheme 2, **2a**). In addition, the structure of **2a** was unambiguously established by single-crystal X-ray analyses.

Scheme 2. Scope of the Substrates[a]



[a]Reaction conditions: **1** (0.25 mmol), NBS (40 mol %), $NaH_2PO_4{\cdot}H_2O$ (1.0 equiv), TBHP (3.5 equiv, 70% aqueous solution), under air; isolated yields.

We next examined the scope of this transformation, and biaryl substrates with various acetyl subunits were subjected to the optimized conditions. As illustrated in Scheme 2, both electron-donating and electron-withdrawing groups on the above aromatic ring were tolerated (**2a−p**). All substrates resulted in good to excellent conversion except for compound **1p** bearing an ethenyl group since it underwent complicated side reactions (**2p**). Interestingly, when *o*-isopropyl-substituted

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substrate **1h** was employed in the reaction, in addition to yielding the expected product **2h** in 25% yield, the cleavage of the isopropyl substituent occurred to provide **2a** in 45% yield. The studies on this unexpected C−C bond-forming reaction are ongoing in our laboratory. The cycloaromatization may take place at two possible positions of *m*-methyl-substituted substrate **1i**. The results indicated that two isomers **2ia** and **2ib** were formed, respectively, and the yield of **2ib** was slightly lower than that of **2ia**. We think that the attack at the *ortho*-position is hindered, which accounts for the observed regioselectivity. Similarly, substrates **1j** and **1k** afforded the corresponding products **2j** and **2k** in only moderate yields due to the steric hindrance of methyl or methoxyl. The annulation of **1w** took place at the β position of nathphalene to afford a single product **2w** in 78% yield. We performed a computational study to explain the complete regioselectivity. The detailed results are provided in the Supporting Information. Biphenyls with F, Cl, and MeO on the lower aromatic ring proved to be efficient substrates, resulting in high yields of products (**2q**−**v**). It is noteworthy that *p*-fluorobiphenyl **1r** give the highest yield among the three isomers (**2q**−**s**). 3,6,7-Trimethoxylphenanthrene **2v**, which is a common structural motif in phenanthroindolizidine and phenanthroquinolizine alkaloids such as cryptopleurine and tylocrebrine,[1b] was prepared in almost quantitative yield using the present method. Two polycyclic aromatic compounds **2w** and **2x** were also synthesized successfully in high yields. In addition to the COOEt-substituted biphenyl-2-ylethanone, substrates bearing *N*-phenylamide and a cyano group formed the phenanthrenes **2y** and **2z** in 85% and 99% yield, respectively. Furthermore, substrate **1aa** incorporating a pyrrole ring underwent the desired transformation in satisfactory yield (**2aa**).

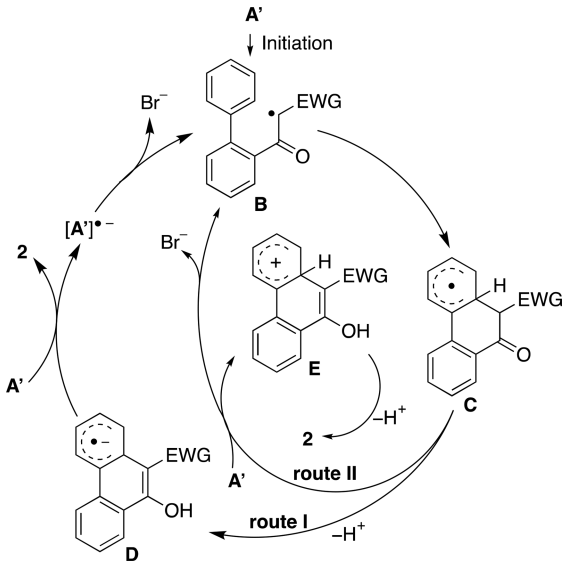
The mechanism of the cycloaromatization was investigated after we established the substrate scope (Scheme 3).

Scheme 3. Control Experiments

1a	standard conditions, without NBS and TBHP	**2a**	none	(2)
1a	standard conditions, without NBS	**2a**	trace	(3)
1a	standard conditions, with 1.0 equiv of NBS and without TBHP	**2a**	trace	(4)
1a	standard conditions, radical scavenger	**2a**	TEMPO, 25% yield BHT, 29% yield	(5)
A'	standard conditions	**2a**	95% yield	(6)
A'	standard conditions, TEMPO	**2a**	65% yield	(7)

The formation of bromide **A′** was observed during the reaction, and its structure was determined by NMR (eq 1). This observation and the result of eq 1 suggested that the cyclization involved an in situ bromination. The annulation of **1a** did not take place in the absence of NBS and TBHP (Scheme 3 eq 2). Moreover, both NBS and TBHP were necessary for the reaction to occur (eqs 3 and 4). Radical scavenger experiments were conducted with TEMPO and BHT under standard conditions. The yields of **2a** were significantly decreased (eq 5). Moreover, when intermediate **A′** was subjected to the standard conditions, the addition of TEMPO retarded the annulation (eqs 6 and 7). The results indicate that a radical pathway may be involved in the present reaction (eqs 6 and 7). On the basis of our results and previous reports,[27] a plausible reaction mechanism is proposed in Scheme 4. The reaction begins with the formation of

Scheme 4. Proposed Mechanism



intermediate **A′**, which yields radical **B** to initiate the radical cycle through the homolytic cleavage of C−Br bond.[27] Then an intramolecular HAS reaction[25] of **B** takes place to provide radical **C**. Further, radical anion **D** is generated from **C** through deprotonation. Oxidation of **D** by **A′** gives product **2** and radical anion $[\mathbf{A}']^{\bullet-}$. Finally, radical **B** and Br^- are regenerated through fragmentation of $[\mathbf{A}']^{\bullet-}$ (route I). Alternatively, radical **C** undergoes an oxidation to form intermidate **E**, which produces the final product **2** through deprotonation (route II).

In summary, we have developed an efficient method for the synthesis of 10-phenanthrenol from readily available starting materials under mild conditions. A metal-free intramolecular CDC reaction of a highly active C−H bond with an inert C−H bond has been achieved. The catalytic utilization of bromine reagents in C−H bond functionalization has been realized through an in situ bromination and followed homolytic cleavage. The present example disclosed a new strategy for radical C−H bond activation, which would be valuable for the development of green and sustainable chemistry.[28]

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b01160.

Detailed experimental procedures and spectral data (PDF)

Accession Codes

CCDC 1535154 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; fax: + 44 1223 336033.

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Notes

The authors declare no competing financial interest.

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