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Synthesis of Functionalized Benzo[g]indoles and 1-Naphthols via Carbon−Carbon Triple Bond Breaking/Rearranging

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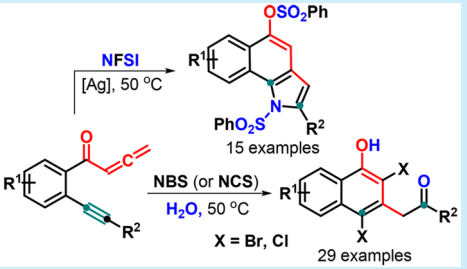
Supporting Information

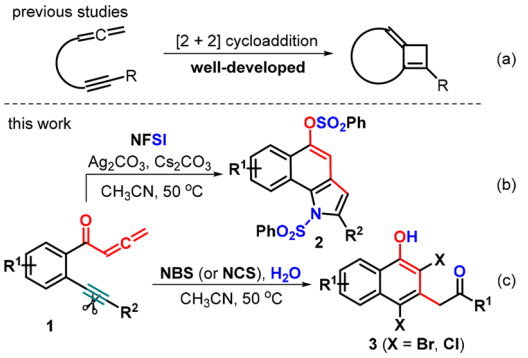
ABSTRACT: Novel carbon−carbon triple bond breaking and rearranging reactions of benzene-linked allene−ynes have been established. The reactions can be selectively controlled toward the formation of two families of skeletally diverse benzo[g]indoles and 1-naphthols under mild conditions. Silver salt was found to efficiently promote indole annulation to give multifunctional benzo[g]indoles with the installation of two sulfonyl groups into the indole ring via N−S and N−F bond cleavage of NFSI, whereas NBS and NCS-mediated benzannulations occurred with the formation of dihalogenated 1-naphthols.



The study of carbon−carbon triple bond breaking, rearrangement, and metathesis has been an extremely challenging topic in chemical and material sciences and has stimulated widespread interest in organic and organometallic disciplines.[1] Differentiated from traditional strategies, this methodology can enable molecular skeletons to be reorganized through more atom-economical retrosynthetic analysis so as to assemble challenging structural units in straightforward and practical fashions.[2] Besides alkyne metathesis,[3] previous work on the cleavage of the carbon−carbon triple bond has been heavily dependent on the use of stoichiometric organometallic reagents, such as alkyne−ligand scission on metal complexes[4] and oxidative cleavage,[5] thereby making their applications less desirable. Lately, much effort has been made toward transition-metal catalysis involving Au,[6] Pd,[7] Ru,[8] Rh,[9] Cu,[10] and Ag[11] species in catalytic systems. In contrast, metal-free cleavage of the unactivated carbon−carbon triple bond has been much less investigated and still remains an extremely difficult task.[12]

In recent years, our laboratories have been investigating a series of domino cyclization reactions for the formation of multiple functional rings.[13] During this study, we prepared allene−yne-anchored starting materials for various tandem [2 + 2] cycloadditions and functionalization (Scheme 1a).[14] Surprisingly, we found the cyclobuta[a]naphthalen-4-ols cannot be generated as we originally planned.[15] Instead, we found unexpected cleavage of the carbon−carbon triple bond occurring under mild conditions, leading to the formation of benzo[g]indoles and 1-naphthols. Further investigation revealed two types of domino reactions involving alkyne breaking and rearranging steps: (i) Ag-mediated indole annulation led to functionalized benzo[g]indoles **2** using *N*-fluorobenzenesulfonimide (NFSI) as both a nitrogen source of indole synthesis and a sulfonyl anchor for sulfonate formation (Scheme 1b); (ii) the use of *N*-bromosuccinimide (NBS) and *N*-chlorosuccinimide (NCS) as halogen sources resulted in dibrominated and dichlorinated 1-naphthols **3** (Scheme 1c) chemoselectively. To the best of our knowledge, this is the first breaking/rearranging of unactivated carbon−carbon triple bonds for the formation of the above functionalized benzo[g]indoles and 1-naphthols. In the former process, Ag-mediated the cleavage of N−F and N−S bonds of NFSI to facilitate the ring-opening of cyclobutenes and formation of pyrrole ring under oxidative conditions. In the latter, NBS and NCS played dual roles as both promoters and electrophiles. Here, we report these novel transformations, as shown in Scheme 1b,c.

Scheme 1. Allene−Yne-Based Cycloadditions



Our initial investigation started with the treatment of benzene-tethered allene−yne **1a** with NFSI in the presence of $AgNO_3$ and Cs_2CO_3. The reaction was performed in

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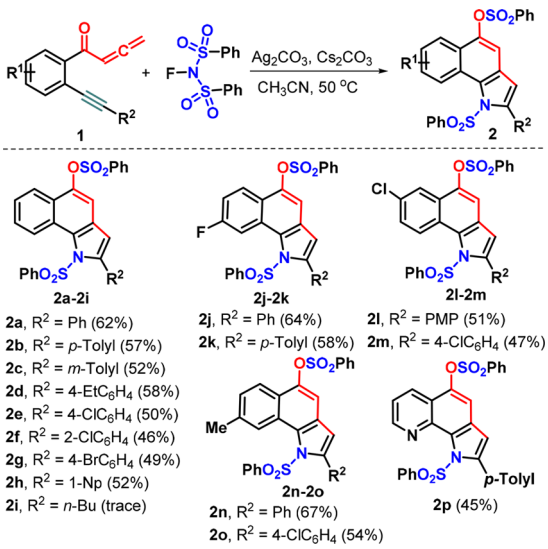
acetonitrile solvent at 50 °C under air atmosphere, delivering the unexpected benzo[*g*]indole **2a** through indole annulation, albeit with a low yield of 19% (Table S1, entry S1). The result is very interesting since it is the first finding that NFSI can act as both nitrogen and sulfonyl sources for the synthesis of benzo[*g*]indol-5-yl sulfonates **2**, rather than as the fluorination reagent often reported in the literature. This discovery prompted us to further search for the optimal conditions for this indole annulation. Several others silver salts including AgOTf, AgTFA, Ag_2O, and Ag_2CO_3, that are often employed in the catalytic transformations, were screened for this transformation by using 2.0 equiv of NFSI (entries S2−S5). Silver salts such as AgOTf, AgTFA, or Ag_2O gave lower concersions as compared with $AgNO_3$ (entries S2−S4 vs S1), whereas the use of Ag_2CO_3 led to a slightly higher yield of **2a** (25%, entry S5). Increasing Ag_2CO_3 loading is beneficial to the transformation (entries S6−S8), and its 0.8 equiv gave the best outcome (49% entry S7). Adjusting the ratio of **1a** with NFSI to 1:3 improved the yield of **2a** to 62% (entry S9). Further increase of the dosage of NFSI showed no positive impact on the reaction efficiency (entry S10). We next optimized conditions by using different bases such as K_2CO_3, Na_2CO_3, K_3PO_4, AcONa, EtONa, and *t*-BuOK and found all these attempts did not show any improvements with respect to the reaction yield (entries S11−S17). Lower conversion into **2a** was obtained with the decrease or increase of Cs_2CO_3 loading (entries S18 and S19). Changing the solvents, such as *N*,*N*-dimethylformamide (DMF), 1,4-dioxane, 1,2-dichloroethane (DCE), trifluorotoluene and tetrahydrofuran (THF), revealed that all these media completely suppressed the reaction process (entries S20−S24). The reaction gave a relatively lower conversion when temperature was changed to either 30 or 70 °C (entries S25−S26). The reaction under argon conditions gave a very inferior outcome as compared with air conditions (entry S27).

Under the above optimal conditions, we explored the reaction scope by using a variety of the preformed benzene-tethered allene−ynes **1** (Scheme 2). A wide range of substituted benzo[*g*]indoles **2a−o** were isolated in modest to good yields (46%−67%). Both electron-donating (methyl **1b** and **1c**, ethyl **1d**) and electron-withdrawing (chloro **1e** and **1f**, bromo **1g**) groups at different positions of arylalkynyl motifs can all tolerate this oxidative system. Among them, a sterically encumbered *o*-chlorophenyl analogue **1f** turned out to be a suitable reaction partner. Similarly, allene−yne **1h** carrying a 1-naphthyl (1-Np) group still showed high reactivity. However, exchanging a 1-naphthyl group with an *n*-butyl counterpart **1i** failed to afford product **2i**. Alternatively, substituents with electronically poor and rich nature on 4- or 5-positions of the internal arene rings of allene-ynes **1** did not hamper indole annulation. Various functional groups, such as fluoro (**1j** and **1k**), chloro (**1l** and **1m**), and methyl (**1n** and **1o**), were adaptable to this reaction, enabling Ag-mediated carbon−carbon triple bond breaking/rearranging to access the corresponding benzo[*g*]indoles **2j−o** in yields ranging from 54% to 67%. Notably, pyridine-linked allene−yne **1p** was successfully engaged into the current transformation, providing the corresponding pyrrolo[3,2-*h*]quinolin-5-yl sulfonate **2p** albeit with 45% yield.

After the successful formation of benzo[*g*]indoles **2**, we then employed NBS to replace NFSI for this transformation. Allene−yne **1a** was first subjected to the reaction with 2.0 equiv of NBS under the standard conditions, and the

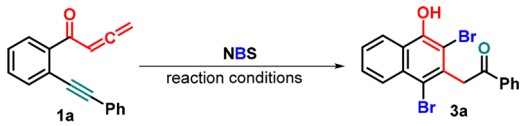
Scheme 2. Substrate Scope for Forming Products 2[a]



[a]Reaction conditions: benzene-tethered allene−ynes **1** (0.2 mmol), NFSI (0.6 mmol), Ag_2CO_3 (0.16 mmol), Cs_2CO_3 (0.2 mmol), CH_3CN (3.0 mL), 50 °C, 5 h. Isolated yields in parentheses are based on **1**.

unexpected dibrominated 1-naphthol **3a** was obtained in 39% yield through carbon−carbon triple bond cleavage (Scheme 3).

Scheme 3. Synthesis of Dibrominated 1-Naphthol 3a



reaction conditions: i) Ag_2CO_3, Cs_2CO_3, CH_3CN, 50 °C, **3a** (39%)
ii) H_2O (1.5 equiv), CH_3CN, 50 °C, **3a** (75%)

After a careful screening of conditions was conducted, the chemical yield can be increased to 75% by adding 1.5 equiv of water into the system in the absence of Ag salts and bases. We then continued examining the scope and limitation of the dibromobenzannulation by changing allene−yne substrates.

As shown in Scheme 4, allene−ynes **1** carrying electron-neutral, -donating, and -withdrawing substituents on the arylalkynyl moiety were all compatible for this system. The presence of electron-rich substituents (Me, *t*-Bu, and MeO) at the *para*-position of the arylalkynyl moiety resulted in the corresponding products **3b,c**, **3h−3j**, and **3m** in slightly lower isolated yields than those with electron-withdrawing groups (**3d,e**, **3k**, and **3n**). Next, NCS was utilized to replace NBS in an attempt to expand the scope of allene-ynes **1**. As expected, the bis-chlorobenzannulation reaction occurred readily under the above conditions with a variety of functional groups on both internal arene rings and arylalkynyl moieties of substrates **1**. Typical functional groups, such as chloro, fluoro, methyl, *tert*-butyl, and ethyl, attached on the arylalkynyl moiety were all proven to be compatible with the present reaction system. It was found that electron-withdrawing groups at the C5-position relative to the internal arene ring showed much better yields than those with electron-donating ones (**3v** vs **3aa**, **3z** vs **3cc**), indicating that the reactivity of the reaction would be dominated by the electronic nature of substituents. Among them, a much lower yield was observed for the substrate with a

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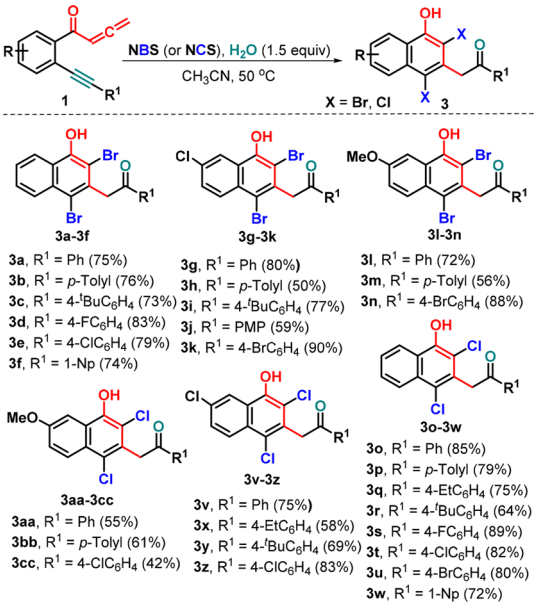
Scheme 4. Substrate Scope for Forming Products 3[a]



[a]Reaction conditions: allene−ynes **1** (0.2 mmol), H_2O (0.3 mmol), CH_3CN (5.0 mL), 50 °C, 4−8 h. For products **3a**−**3n**: NBS (0.4 mmol). Isolated yields in parentheses are based on **1**. For products **3o**−**dd**: NCS (0.3 mmol). Isolated yields in parentheses are based on NCS.

chloro group linked to the arylalkynyl moiety and a methoxy group located at C5-position of the internal arene ring for the formation of the dichlorinated product **3cc**. It should be noted that this is the first method for the multicomponent assembly of these multifunctionalized 1-naphthols through a sequential [2 + 2] cycloaddition/C−C cleavage/bis-halogenation pathway in a one-pot manner. The structures of the products **2a** and **3b** were determined by X-ray diffraction analysis (Figure 1 and see Supporting Information).

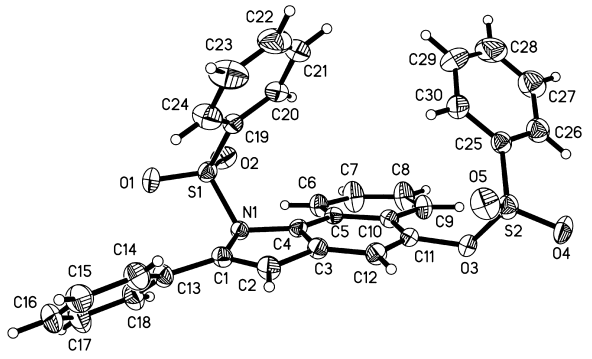
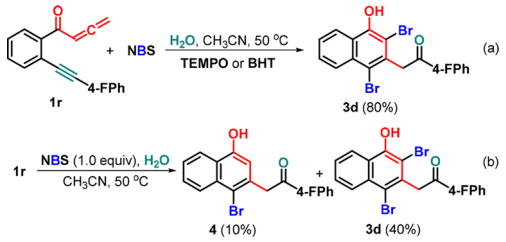


Figure 1. ORTEP drawing of **2a**.

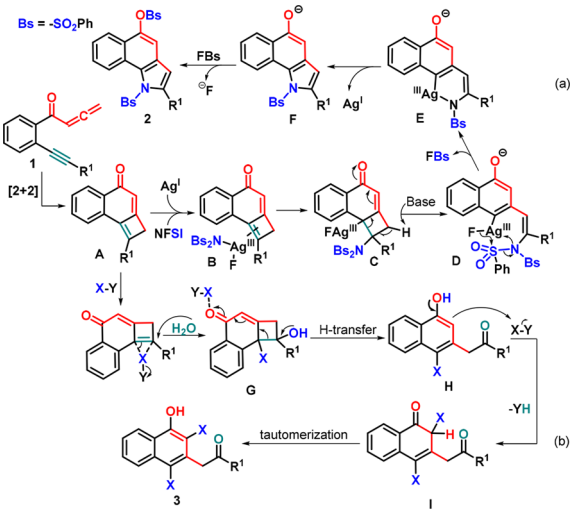
Considering NBS and NCS can readily trigger the radical species, the substrate **1r** was first subjected to the reaction with 1.5 equiv of TEMPO or BHT, and the expected product **3d** was afforded in 80% yield; this observation would confirm that the mechanism does not involve a radical process (Scheme 5a). Next, substrate **1r** was treated with 1.0 equiv of NBS under standard conditions, delivering the expected product **3d** in 40% yield along with monobrominated product **4** in 10% yield, which indicates bromination occurred at C4 position prior to C2 position during the reaction process (Scheme 5b).

Scheme 5. Control Experiments



Based on the above observations, LC−MS analysis, and literature survey,[14,16] the mechanisms for forming products **2** and **3** were proposed as shown in Scheme 6. First, the

Scheme 6. Plausible Reaction Pathways



intramolecular [2 + 2] cycloaddition of allene−ynes **1** spontaneously occurs to afford cyclobutene intermediate **A**, which undergoes oxidation−coordination with silver salt and NFSI to afford Ag^{III} complex intermediate **B**,[16] followed by migratory insertion[17] and base-promoted ring-opening of cyclobutane intermediate to access vinyl−Ag^{III} fluoride **D**. Next, the fluoride substitutes the sulfonyl group to liberate benzenesulfonyl fluoride (detected by LC−MS)[18] and intermediate **E** via N−S bond cleavage, and the following reductive elimination gives benzo[*g*]indole intermediate **F**. Finally, base-promoted nucleophilic substitution between **F** and benzenesulfonyl fluoride yields polysubstituted benzo[*g*]indole products **2** (Scheme 6a). Different from the above, the latter two processes undergo spontaneous [2 + 2] cycloaddition and the following halohydroxylation of cyclobutene intermediates in the presence of **X**−**Y** (NBS and NCS) and H_2O to give **G**. Subsequently, the ring-opening of cyclobutene and hydrogen transfer proceed to form intermediates **H** followed by electrophilic substitution with NBS and NCS to yield the products **3** (Scheme 6b).

In summary, we have discovered unprecedented C≡C bond breaking and rearranging reactions of benzene-linked allene−ynes under mild conditions. These transformations provide general and practical protocols toward the formation of a range of richly decorated benzo[*g*]indoles and 1-naphthols of chemical and biomedical importance. The original alkyne motif was split into two parts and was enabled them to remerge into one molecular framework, thereby maximizing atom

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economy of the present strategy. These benzannulation reactions feature bond-forming efficiency, accessibility of starting materials, and functional group tolerance, making them feasible and powerful. Further studies toward confirming the mechanism and their synthetic applications are currently underway in our laboratories.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b03410.

Experimental procedures and spectroscopic data for all new compounds **2a**–**p** and **3a**–**cc** (PDF)

Accession Codes

CCDC 1517855 and 1577815 contain 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 CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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