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Transition-Metal-Free Cleavage of C−C Triple Bonds in Aromatic Alkynes with S_8 and Amides Leading to Aryl Thioamides

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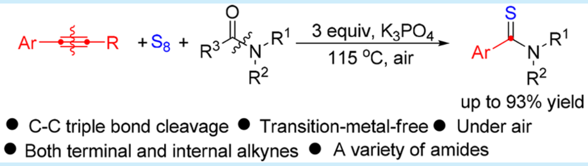
Supporting Information

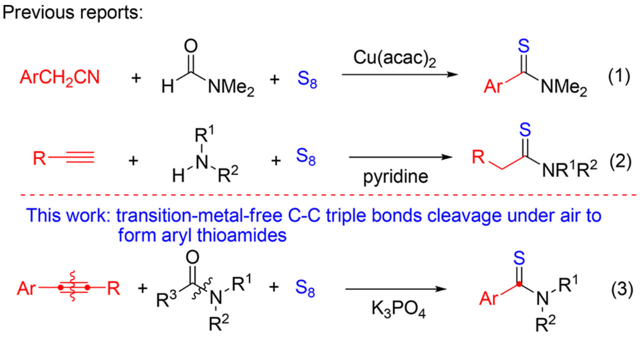
ABSTRACT: A novel transition-metal-free cleavage reaction of C−C triple bonds in aromatic alkynes with S_8 and amides furnishes aryl thioamides in moderate to excellent yields. The remarkable features of this thioamidation include the metal-free cleavage of C−C triple bond, mild reaction conditions, as well as wide substrate scope that is particularly compatible with some internal aromatic alkynes and acetamides.



The cleavage and the transformation of C−C triple bonds, compared with those of C−C single bonds, are less explored and considered to be one of most challenging targets in current organic synthesis.[1] Except for the metathesis of alkynes,[2] previous methods for C−C triple bonds cleavage often suffered from the use of stoichiometric amounts of organometallic reagents or oxidants.[3] In recent years, several efficient and desirable transition-metal-catalyzed processes[4] such as Rh-catalyzed cleavage of C−C triple bonds via hydroiminoacylation[4a] or hydroacylation,[4b] Ru-catalyzed C−C triple bonds cleavage of diynes through hydroamination,[4c] Ru-catalyzed C−C triple bonds cleavage of ethynyl alcohols,[4d] Au-catalyzed C−C triple bonds cleavage of (Z)-enynols with molecular oxygen,[4e] Pd-catalyzed C−C triple bonds cleavage of alkynes with molecular oxygen,[4f] Ni-catalyzed C−C triple bonds cleavage of alkynes with molecular oxygen,[4g] Ag-catalyzed C−C triple bonds cleavage and nitrogenation of alkynes,[4h] Mn-catalyzed C−C triple bonds cleavage of alkynes via oxidative rearrangement,[4i] Fe/Cu-catalyzed C−C triple bonds cleavage of alkynones,[4j] and visible-light-induced Cu-catalyzed C−C triple bond cleavage of terminal alkynes[4k] were presented. Undoubtedly, the transition-metal-free approaches developed very recently are more attractive, delivering very useful building blocks such as acids, nitriles, esters, and amides.[5] Despite these significant developments, transition-metal-free approaches for C−C triple bond cleavage with inexpensive, stable, and readily available reagents are still highly desirable.

Thioamides constitute a kind of useful intermediate for the synthesis of heterocyclic compounds, protein, pharmaceuticals, and materials.[6] The usefulness of aryl thioamides has made their synthesis the subject of extensive research.[7] Apart from the traditional methods such as thionation of cabonamides and the Willgerodt−Kindler-type reactions,[8] the strategies involving the step-economic and easily handled three-component reactions[9] have become an attractive alternative recently. In detail, most reports involving three-component reactions focused on the condensation of various substrates such as benzylamines,[9a] nitriles,[9b] aldehydes,[9c,g] alkynes,[9d,f] or arylacetic and cinnamic acids[9e,h] with amines or amides promoted by S_8. Among them, $C(sp^2)-C(sp^3)$ bond cleavage was observed in the Cu-catalyzed reactions of dimethylformamide with arylacetonitriles and sulfur[9b] (Scheme 1, eq 1). In 2014, Nguyen and co-workers presented an efficient three-component synthesis of aliphatic thioamides from alkynes, elemental sulfur, and amines[9d] (Scheme 1, eq 2). Meanwhile, elemental sulfur has been used in many multicomponent reactions as well as useful metal-free transformations.[10] As far as we know, there is no report on the synthesis of thioamides via C−C triple bonds cleavage. Herein, we report a novel synthesis of aryl thioamides by three-component reactions from alkynes,

Scheme 1. Thioamide Synthesis



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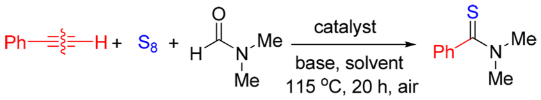
elemental sulfur, and amides via unexpected transition-metal-free C−C triple bonds cleavage (Scheme 1, eq 3).

To our surprise, the reaction of phenylacetylene, sulfur power and *N*,*N*-dimethylformamide (DMF) in the presence of NaO^{t}Bu afforded *N*,*N*-dimethylbenzothioamide (Table 1, entry 1). The selected results for reaction condition optimization are summarized in Table 1. In general, the bases tested could deliver the target product (Table 1, entries 1−7), and K_3PO_4 (Table 1, entry 3) was found to be the best with an isolated yield up to 84% comparable with the yield of 80% given by K_2CO_3 (Table 1, entry 2). Regarding to the effects of different solvents, the use of DMF without combination of another solvent exhibited a favorable result (Table 1, entry 3 vs 8). In particular, when the reaction proceeded in a mixed solvent of DMF/H_2O (1:1), a messy conversion was observed. Some control experiments were also explored (Table 1, entries 9−15). The addition of metal salts, performing the reaction under Ar in place of air, the use of sublimated sulfur powder, or a slight decrease in the temperature had little influence on the yield (Table 1, entries 9−13). However, the yield dropped obviously with a decreasing amount of sulfur powder (Table 1, entry 3 vs entry14). Note that no target product was isolated in the absence of base (Table 1, entry 15).

Table 1. Optimization of Reaction Conditions[a]

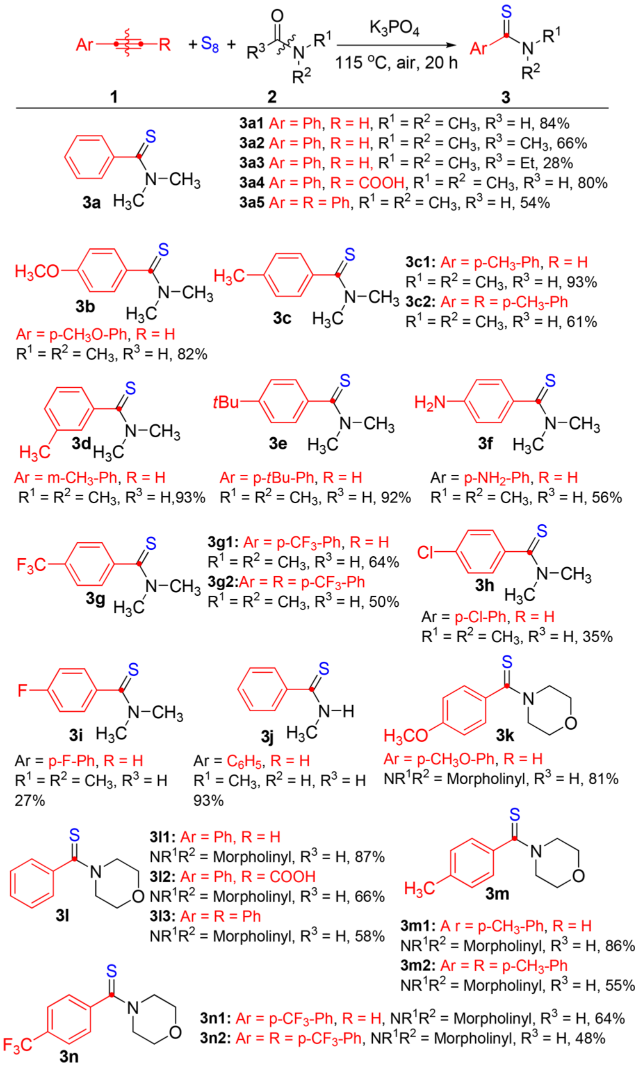


entry	catalyst	base	solvent	yield[b] (%)
1	no	NaO^{t}Bu	DMF	78
2	no	K_2CO_3	DMF	80
3	**no**	**K_3PO_4**	**DMF**	**84**
4	no	KOAc	DMF	57
5	no	KF	DMF	68
6	no	Cs_2CO_3	DMF	77
7	no	NaOH	DMF	75
8	no	K_3PO_4	DMF/toluene (1:1)	84
9	$FeCl_3$	K_3PO_4	DMF	84
10	$NiCl_2$	K_3PO_4	DMF	82
11[c]	no	K_3PO_4	DMF	81
12[d]	no	K_3PO_4	DMF	83
13[e]	no	K_3PO_4	DMF	80
14[f]	no	K_3PO_4	DMF	66
15	no	no	DMF	<5

[a] Reagents and conditions: phenylacetylene (0.4 mmol), S_8 (9.6 mmol), base (1.2 mmol), and the catalyst (10 mol %) in solvent (2 mL) at 115 °C under air for 20 h. [b] Isolated yield based on the amount of phenylacetylene. [c] Under Ar. [d] Using sublimated sulfur powder. [e] At 90 °C. [f] S_8 (4.8 mmol).

The substrate scope was studied under optimized reaction conditions (Scheme 2). Overall, several aromatic alkynes and amides could be amenable to the reaction and afford the corresponding products. When terminal aromatic alkynes were treated with DMF and S_8, electron-neutral and electron-rich ones displayed higher reactivity than the electron-deficient ones (**3a1**, **3b**, **3c1**, **3d**, **3e** vs **3g1**, **3h**, **3i**). Surprisingly, the substituents NH_2 and CF_3 on the aromatic alkynes could also survive in this transformation (**3f** and **3g**). While ethynyl-2-methylbenzene could not deliver the corresponding product, ethynyl-3-methylbenzene reacted smoothly to generate **3d** in excellent yield, presumably due to the effect of steric hindrance.

Scheme 2. Substrate Scope[a]

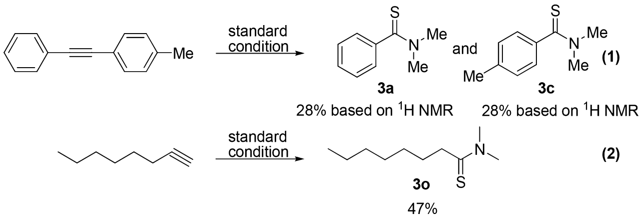


[a] Reagents and conditions: aromatic alkyne (0.4 mmol), S_8 (9.6 mmol), and K_3PO_4 (1.2 mmol) in amide (2 mL) at 115 °C under air for 20 h. Yield of isolated product based on the amount of aromatic alkyne.

The secondary thioamide **3j** was generated in 93% yield when *N*-methylformamide was subjected to the reaction conditions. In addition, replacement of DMF with formylmorpholine led to the formation of thioamides **3k** to **3n** in good yields (**3k**−**n**). Both *N*,*N*-dimethylacetamide and *N*,*N*-dimethylpropionamide could take part in the reaction producing the same product **3a** as DMF did, albeit in lower yields (**3a1** vs **3a2**, **3a3**), which indicated that the amides were more likely to undergo scission of the C(sp^2)−N bond in a basic environment[9g,h] offering the N$(Me)_2$ moiety. Remarkably, internal aromatic alkynes were also tolerant to the reaction, which provided **3a**, **3c**, **3g**, **3l**, **3m**, and **3n**, respectively (**3a4**, **3a5**, **3c2**, **3g2**, **3l2**, **3l3**, **3m2**, **3n2**). In addition, in N-substituted acetamides, some aromatic alkynes worked well to afford the corresponding aryl thioamides in moderate yields from 40% to 60% (Table 1, see the Supporting Information (SI) for more details).

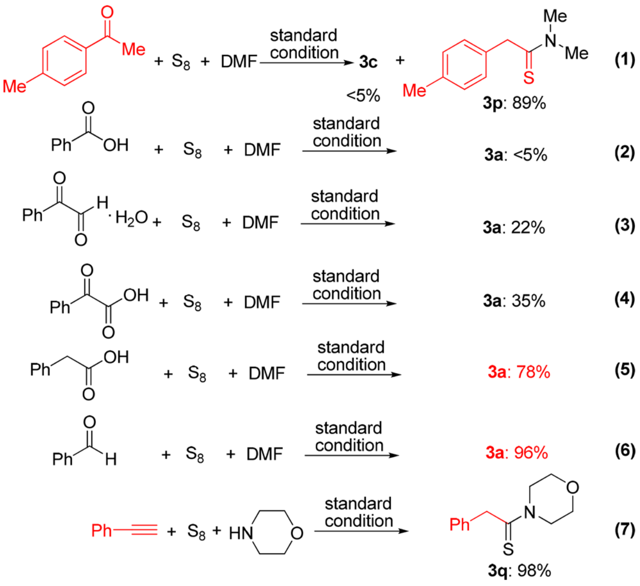
In particular, 1-methyl-4-(phenylethynyl)benzene was equally split into **3a** and **3c** in the presence of DMF and S_8 (Scheme 3, eq 1). Moreover, when 1-octyne was added to the standard conditions, *N*,*N*-dimethyloctanethioamide (**3o**), the

Scheme 3. Thioamidation of Alkynes by DMF and S_8



product without C−C triple bond breaking, was obtained in 47% yield (Scheme 3, eq 2).

Scheme 4. Mechanism Investigation



Some possible intermediates including aryl methyl ketones exemplified as 1-(4-methylphenyl)ethanone, benzoic acid, phenylglyoxal monohydrate, and 2-oxo-2-phenylacetic acid were subjected to the standard conditions, but none of them gave cracked products in good yield, which ruled out their formation to a large extent in the reaction process (Scheme 4, eqs 1−4). As expected, both phenylacetic acid and benzaldehyde could give rise to **3a** in good to excellent yields under the standard conditions (Scheme 4, eqs 5 and 6). In addition, to evaluate the sequence of the three-component reaction, replacement of formyl morpholine with morpholine resulted in the formation of **3q** rather than **3l**, which is in accordance with Nguyen's report (Scheme 4, eq 7).[9d] Thus, the scission of the C(sp^2)−N bond in amides for the synthesis of aryl thioamides might not be the first step. On the basis of the results mentioned above and relevant literature,[11] a plausible mechanistic pathway for cleavage of C−C triple bond in aromatic alkynes promoted by S_8 and amides is depicted (Schemes 1 and 2, see the SI for more details).

In conclusion, we developed a general and efficient three-component reaction between aromatic alkynes, S_8, and amides, affording various aryl thioamides in moderate to excellent yields. Notably, the metal-free cleavage of a C−C triple bond promoted by the economical and readily available S_8 without external oxidants has been achieved. Meanwhile, this three-component reaction could even tolerate various internal aromatic alkynes and relatively inactive N-substituted acetamides. Further mechanistic studies and synthetic applications of these methodologies are currently underway in our laboratory.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b00573.

Experimental procedures; spectroscopic data for new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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