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# Cu-Catalyzed Transformation of Alkynes and Alkenes with Azide and Dimethyl Sulfoxide Reagents

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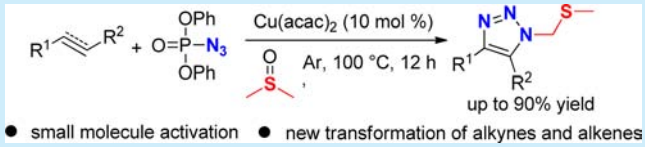
Supporting Information

**ABSTRACT:** A novel and efficient Cu-catalyzed nitrogenation of alkynes and alkenes for the direct synthesis of sulfur-containing triazoles is described. Simple and readily available sulfoxides and azides are employed as the S- and N-source to prepare highly value N-,S-containing compounds from simple alkynes and alkenes.

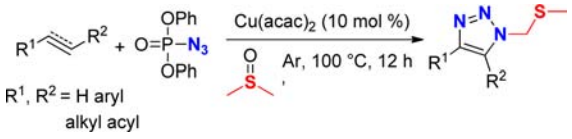



The activation of dimethyl sulfoxide (DMSO) has become a hot topic in recent years,[1−7] not only because it is an inexpensive, common, low-toxic solvent,[1] but also due to the versatile role it plays in organic synthesis as an important small molecule. In the past decades, DMSO has also been widely utilized as an oxygen source,[2] a carbon source,[3] and a sulfur source such as MeS,[4] MeS(O),[5] $MeSCH_2$,[6] and $MeS(O)CH_2$[7] for the incorporation of functional groups into target molecules. Among them, the Pummerer rearrangement[8] through the in situ generation of thionium ion intermediates provides a very powerful protocol for the construction of thioethers. Various nucleophiles, such as amides, alkenes, phenols, and arenes, have been reported for attacking the thionium ions in the valuable synthetic processes since the first Pummerer reaction was reported in 1909.[9,10] However, a Pummerer reaction that employs azides as nucleophiles has rarely been reported.[11]

Copper catalyzed click chemistry provides a very powerful protocol for the construction of highly valuable products in supramolecule chemistry, materials science, drug discovery, polymer chemistry, and biotechnology.[12] Meanwhile, thioether fragments exist in large numbers of pharmaceutically and agrochemically active compounds, such as drugs for treatment of Alzheimer's disease, breast cancer, HIV, and inflammatory disease.[13] Sulfur-containing triazoles, which constitute a class of pharmaceutically important triazoles, show special inhibition on the activity of mPGES-1[14] and are potentially new anti-inflammatory agents. Encouraged by the exploration of nitrogenation of alkynes employing azides as a nitrogen source[15] and DMSO activation[2a,b,16] in our group, we hope to try other types of direct nitrogenation of alkynes that could combine with DMSO activation. Compared to the traditional synthesis of sulfur-containing triazoles,[14,17] there is no doubt that a direct approach from simple hydrocarbons is highly attractive but challenging.

Herein, we report a novel and efficient Cu-catalyzed direct transformation of alkynes and alkenes for the synthesis of highly pharmaceutically valuable sulfur-containing triazoles (Scheme 1). The significance of the present protocol is threefold: (1) The nitrogen and sulfur atoms can be significantly incorporated into simple alkynes and alkenes in one step. (2) DMSO not only is employed as a common solvent but also is activated as a sulfur source in this transformation. (3) This chemistry provides a direct and practical approach to sulfur-containing triazoles.

**Scheme 1. Cu-Catalyzed Direct Nitrogenation of Alkynes and Alkenes**




*p*-Methoxy phenylacetylene **1a** was initially evaluated for the direct nitrogenation of alkynes. Disappointingly, Lewis acids such as $Ag_2CO_3$, $AuCl_3$, $NiCl_2$ could hardly deliver the desired product (Table 1, entries 1−3). In contrast, the copper salts, which have been proven to be an efficient catalyst for click reactions,[12] could enable this transformation to produce sulfur-containing triazoles **2a** in moderate yields (Table 1, entries 4, 8, and 9). The kind of azides used is a key factor for this transformation. $NaN_3$, $TsN_3$ could not give the desired product (Table 1, entries 6 and 7). It is noteworthy that the yield was significantly improved to 86% with DPPA as the $N_3$ source (Table 1, entry 5). **2a** could not be obtained in the absence of a transition metal catalyst (Table 1, entry 10). Undesirable results were observed when reducing the reaction temperature (Table 1, entries 11 and 12) or performing the reaction under $O_2$ (Table 1, entry 13). However, the reaction that proceeded with a stoichiometric amount of dimethyl sulfoxide gave a very low yield (Table 1, entry 14; also see Supporting Information (SI)).

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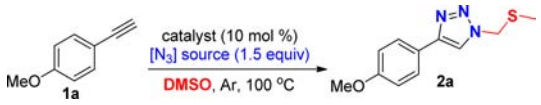
**Table 1. Screening of the Reaction Conditions**[a]

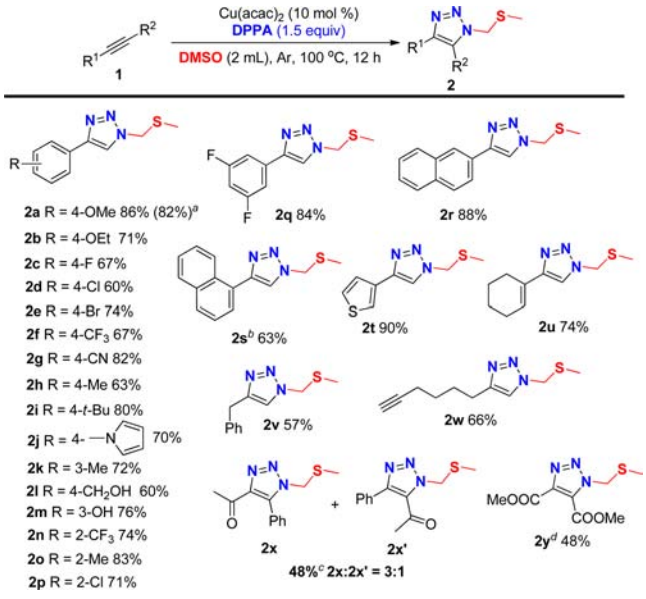



| entry | catalyst | [$N_3$] source | yield of **2a**[b] (%) |
|---|---|---|---|
| 1 | $Ag_2CO_3$ | $TMSN_3$ | 8 |
| 2 | $AuCl_3$ | $TMSN_3$ | 0 |
| 3 | $NiCl_2$ | $TMSN_3$ | 0 |
| 4 | $Cu(acac)_2$ | $TMSN_3$ | 45 |
| 5 | **$Cu(acac)_2$** | **DPPA** | **86** |
| 6 | $Cu(acac)_2$ | $NaN_3$ | 0 |
| 7 | $Cu(acac)_2$ | $TsN_3$ | 0 |
| 8 | $Cu(OAc)_2$ | DPPA | 61 |
| 9 | CuBr | DPPA | 52 |
| 10 | – | DPPA | 0 |
| 11[c] | $Cu(acac)_2$ | DPPA | 0 |
| 12[d] | $Cu(acac)_2$ | DPPA | 44 |
| 13[e] | $Cu(acac)_2$ | DPPA | 31 |
| 14[f] | $Cu(acac)_2$ | DPPA | 33 |

[a]Reaction conditions: **1a** (0.5 mmol), catalyst (0.05 mmol), $N_3$ source (0.75 mmol), dry DMSO (2 mL), stirred at 100 °C under Ar for 12 h. [b]Isolated yields. [c]At 50 °C. [d]At 80 °C. [e]The reaction was carried out under $O_2$. [f]10.0 equiv of DMSO were used. DPPA = diphenylphosphoryl azide, TMS = trimethylsilyl, DMSO = dimethyl sulfoxide, Ts = *p*-toluenesulfonyl.

With the optimied reaction conditions in hand (Table 1, entry 5), we subsequently investigated the substrate scope of this transformation (Scheme 2). The results indicate that various phenylacetylene derivatives bearing both electron-donating and -withdrawing group substituents at the aryl ring afforded the sulfur-containing triazole products in good to excellent yields (**2a**–**j**). The halo-substituted phenylacetylenes worked well under these mild reaction conditions to produce the corresponding halo-substituted sulfur-containing triazoles (**2c**–**e**). The structure of **2e** was further confirmed by single-crystal X-ray (Figure 1). Moreover, enynes and the relatively inactive aliphatic alkynes could also generate the desired products in good yields (**2u**–**w**). To our delight, the internal alkynes could also produce the trisubstituted sulfur-containing triazoles (**2x** and **2y**). It is noted that **2x** and **2x′** were obtained in the ratio 3:1 (the structure of **2x** was confirmed by NOE spectroscopy; see SI).

**Scheme 2. Direct Conversion of Alkynes 1 to Sulphur-Containing Triazoles 2**




[a]10 mmol scale reaction. [b]This reaction was carried out at 100 °C for 24 h. [c]This reaction was carried out at 100 °C for 36 h, with 42% of alkyne **1** recovered. [d]This reaction was carried out at 100 °C for 24 h, with 33% of alkyne **1** recovered.

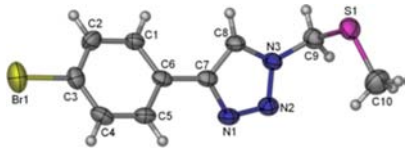
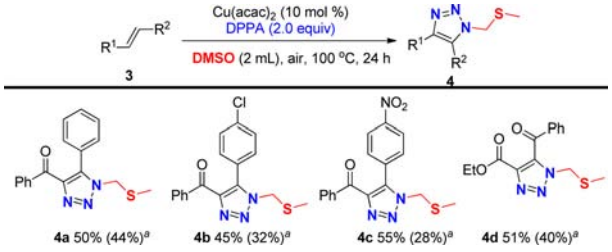


**Figure 1.** X-ray crystallography structure of compound **2e**.

Encouraged by the above-mentioned results with alkynes, the reaction with alkenes was then investigated (Scheme 3).

**Scheme 3. Direct Conversion of *α,β*-Unsaturated Ketones 3 to Trisubstituted Sulphur-Containing Triazoles 4**




[a]Recovery of the alkenes.

Compared to alkynes, those alkenes were predicted to be more difficult in undergoing [3 + 2] cycloaddition with azides. We were very interested to find that *α,β*-unsaturated ketones could be highly regioselectively converted into the desired products in moderate yields by using these internal alkene substrates under air (Scheme 3).[18] The structure of these products were further confirmed by NOE spectroscopy. When the reaction of **3a** was performed under Ar, **4a** was obtained in only 9% yield (see SI), which indicates that the oxidation process occurred under this Cu–air system to form the C–C double bond in the triazole products.[18]

Other MeS(=O)R-type sulfoxide derivatives could also react in a similar manner to give the desired corresponding sulfur-containing triazoles (Scheme 4). It is interesting to find that the reaction highly selectively occurs at a 1° C–H bond rather than a 2° or 3° C–H bond to give sulfur-containing triazoles with a longer alkyl chain in good yields (**6a**–**d**). It is noteworthy that a disulfide-bond-containing triazole (**6e**) was obtained in moderate yield when $CH_3S(=O)SCH_3$ was employed as solvent.

Sulfur-containing triazole moieties are widely found to be key motifs in biologically active substances.[14] In order to test the further application of this protocol in the late-stage nitrogenation of natural product derivatives, the modification of alkynes **7** and **9** derived from estrone and estradiol were investigated under the optimized conditions (Scheme 5). Interestingly, the direct nitrogenation took place efficiently to



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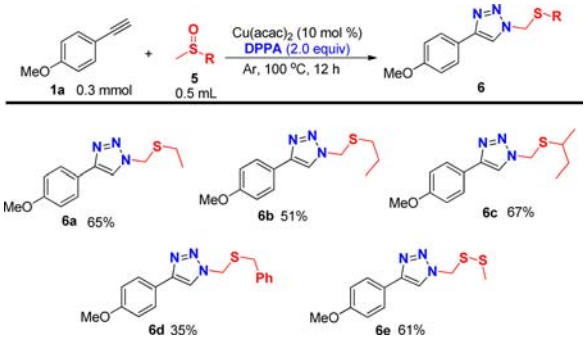
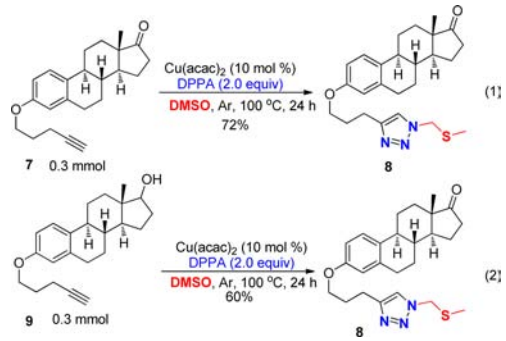
**Scheme 4. Direct Conversion of Sulfoxide 5 to Sulphur-Containing Triazoles 6**

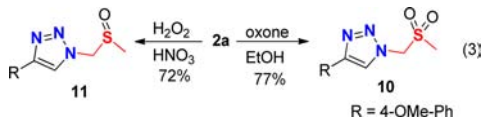



**Scheme 5. Late-Stage Modification of Estradiol Derivatives**

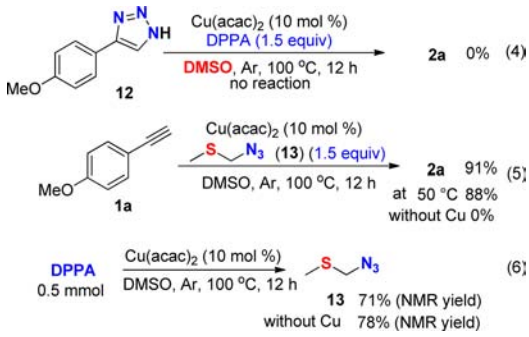



give the same sulfur-containing triazole **8** in 72% and 60% yields, respectively. An unexpected oxidation occurred in eq 2 under this condition.

As different oxidative states of thioether derivatives, sulfone and sulfoxide also show different divergent functions and potencies.[13c] The model product **2a** could be obtained in 82% yield in gram scale under standard conditions (1.93 g, Scheme 2; also see SI), showing the efficiency and practicability of this protocol. Then we explored further transformations of sulfur-containing triazole **2a**; the oxidation of **2a** with oxone or $H_2O_2$ provided sulfone **10** and sulfoxide **11** (eq 3) in 77% and 72% yields, respectively.



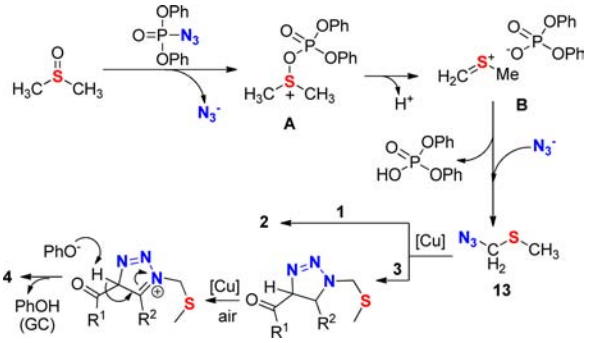

To further understand the mechanism, control experiments were investigated. The model reaction was not inhibited in the presence of equivalent TEMPO or BHT (eqs S1–S2; see SI), which might rule out a radical process in this transformation. In order to prove whether the click reaction intermediate **12**, which is observed in some cases, is or is not involved in this transformation, triazole **12** was prepared and investigated under the standard conditions (eq 4). However, no desired product **2a** was detected (eq 6), excluding the initial click reaction and subsequent DMSO activation relay. Moreover, (azidomethyl)(methyl)sulfane **13** was prepared and investigated. It is noted that **13** reacted smoothly with **1a** and afforded the desire product **2a** in 91% yield (eq 5), which demonstrates that (azidomethyl)(methyl)sulfane **13** *in situ* generated from DPPA and DMSO might be the key intermediate in this transformation. The click reaction performed well even at 50 °C. but




did not work in the absence of a Cu catalyst (eq 5). To further verify the role of the Cu catalyst, the initial reaction of DPPA and DMSO in the absence of alkynes was evaluated (eq 6). Similar results (71% and 78% yields) for intermediate **13** were obtained with/without a Cu catalyst (eq 6), which indicates that a Cu catalyst was not necessary at the initiation step. These results illustrated that high temperature is necessary for the initiation of Pummerer reaction, and a copper catalyst is required for the following click process.

The possible mechanism is proposed on the basis of these preliminary results and mechanism of the Pummerer reaction (Scheme 6). Initially, electrophilic substitution occurs between

**Scheme 6. Proposed Mechanism**




DPPA and solvent DMSO with heat to afford intermediate **A** by releasing an azide ion, which successively generates classical thionium ion **B**.[8,10a] The subsequent nucleophilic attack reaction with the azide ion gives the key intermediate **13** along with the release of diphenyl phosphate. Then intermediate **13** undergoes a click reaction with alkyne **1** to produce the sulfur-containing triazole **2** under copper catalytic conditions. Alternatively, the triazole **4** is finally produced through the relay of a click reaction of alkenes **3** and oxidation by the Cu–air oxidative system.[18]

In summary, we have demonstrated an efficient Cu-catalyzed direct transformation of alkynes and alkenes for the synthesis of sulfur-containing triazoles. Simple and readily available DMSO and azide are employed as the S- and N-source to prepare highly valued N-,S-containing compounds. Further studies on the substrate scope and synthetic applications of this transformation are ongoing in our group.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b03179.



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Experimental procedures, full characterization of products, and copies of NMR spectra (PDF)
Crystallographic data (CIF)

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### Notes

The authors declare no competing financial interest.

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