OL | Organic Letters

Cite This: Org. Lett. XXXX, XXX, XXX−XXX

Letter

pubs.acs.org/OrgLett

Copper-Catalyzed Three-Component Reaction of Alkynes, $TMSN_3$, and Ethers: Regiocontrollable Synthesis of N^1- and N^2-Oxyalkylated 1,2,3-Triazoles

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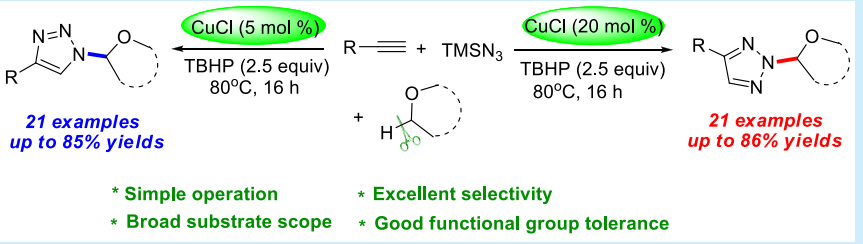
Supporting Information



ABSTRACT: A new copper-catalyzed selective synthesis of N^1- and N^2-oxyalkylated 1,2,3-triazoles has been developed through a three-component reaction of alkynes, $TMSN_3$, and ethers. Through this methodology, a series of N^1- and N^2-oxyalkylated 1,2,3-triazoles could be efficiently and regioselectively obtained from simple and readily available starting materials with favorable functional group tolerance.

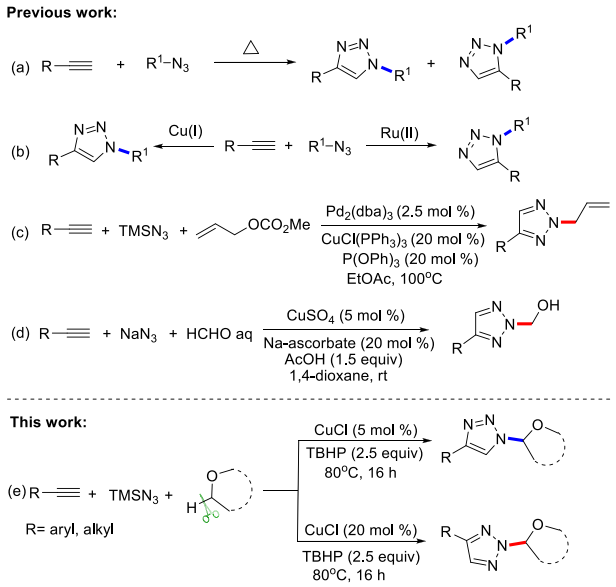
As one of the most important classes of N-heterocyclic compounds, N-substituted 1,2,3-triazoles have exhibited widespread applications in medicinal chemistry,[1] organic synthesis,[2] and material science.[3] Consequently, numerous efforts have been paid to construct N-substituted 1,2,3-triazoles.[4−9] Representatively, N-substituted 1,2,3-triazoles are synthesized by thermo- or copper/ruthenium-catalyzed cycloaddition of alkynes with organic azides (Scheme 1a and 1b).[5] Alternative cycloaddition reactions of organic azides with nitroolefins,[6] enaminones,[7] and cinnamic acids[8] as well as azide-free synthetic methods[9] have also been developed. Through these methodologies, only N^1-substituted 1,2,3-triazoles can be obtained owing to the nature of organic azides. N^2-Substituted 1,2,3-triazoles are extremely interesting compounds that possess a wide range of biological activities such as antiviral, antiarrhythmic, antiarthritic, and antiherpetic properties.[10] Nevertheless, in comparison with N^1-substituted 1,2,3-triazoles, the strategies for the synthesis of N^2-substituted triazoles remained largely unexplored.[11] Generally, N^2-substituted 1,2,3-triazoles are prepared by the *N*-2 alkylations or *N*-2 arylations of the preformed NH-1,2,3-triazoles with alkylation/arylation reagents.[11] These reactions often require harsh reaction conditions and suffer from low selectivities. Multicomponent reactions (MCRs) are exceptionally attractive tools for accomplishing the goal of step and energy economy in the construction of structurally diverse compounds from simple starting materials.[12] In this context, the Yamamoto group reported an elegant Pd/Cu cocatalyzed synthesis of 2-allyl-1,2,3-triazoles via three-component reaction of alkynes, allyl methyl carbonate, and $TMSN_3$ (Scheme 1c).[13] Fokin and co-workers reported a copper-catalyzed three-component reaction of alkynes, sodium azide, and formaldehyde leading to 2-hydroxymethyl-2*H*-1,2,3-triazoles (Scheme 1d).[14] Although some progress has been made, there is still a high demand for the development of facile and efficient methods to selectively construct N^1- and N^2-substituted 1,2,3-triazoles from simple and readily available materials.

Cyclic ethers are privileged structural motifs in many biologically active molecules and natural products, which exhibited a broad spectrum of biological and pharmaceutical properties.[15] Recently, the direct alkylation of NH-1,2,3-triazoles with cyclic ethers and vinyl ethers has been developed for the construction of the N^1- or N^2-oxyalkylated azoles.[16] However, the need of extra steps to prepare various NH-1,2,3-triazoles and the formation of a mixture of the isomeric products are the major drawbacks of this strategy. With our continued interests in the synthesis of heterocyclic compounds,[17] here, we report an efficient and regiocontrollable method for the synthesis of various N^1- and N^2-oxyalkylated 1,2,3-triazoles via copper-catalyzed three-component reaction of alkynes, $TMSN_3$, and ethers (Scheme 1e).

Received: July 3, 2019

DOI: 10.1021/acs.orglett.9b02295
Org. Lett. XXXX, XXX, XXX−XXX

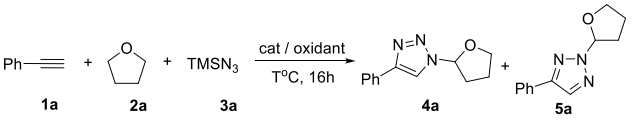
Scheme 1. Synthetic Approaches to N-Substituted 1,2,3-Triazoles



Initially, phenylacetylene (**1a**), $TMSN_3$ (**2a**), and THF (**3a**) were chosen as model substrates to optimize the reaction conditions. When the model reaction was conducted at 80 °C in the presence of CuI (5 mol %) and TBHP (2.5 equiv), the N^1-substituted triazole **4a** and N^2-substituted triazole **5a** were obtained in 30% and 15% yields, respectively (Table 1, entry 1). Subsequently, a series of metal catalysts (5 mol %) were investigated to improve the yield and selectivity (Table 1, entries 2−6). Among various metal catalysts examined, the copper salt, especially CuCl, was found to be the best one to selectively afford the N^1-substituted triazole **4a** with 85% yield (Table 1, entry 3). None of the products were detected when other metal salts such as $AgNO_3$, $Pd(OAc)_2$, and $FeCl_3$ were used (Table 1, entries 7−9). Then, the effect of other oxidants was further screened. As shown in entries 10−12, lower yield and selectivity were observed by using DTBP, H_2O_2, or BPO as the oxidant. The reaction did not occur in the presence of DDQ or $K_2S_2O_8$ (entries 13 and 14). The decrease of TBHP loading to 1 equiv or 1.5 equiv would lead to lower yield of **4a**. When other solvents such CH_3CN were used in this reaction, only 11% yield of **4a** and a trace amount of product **5a** were obtained (Table 1, entry 17). To our delight, significantly improved reaction efficiency of N^2-substituted triazole **5a** was observed with the increase of CuCl loading (Table 1, entries 18−21), and 20 mol % of CuCl gave the best yield (86%) of **5a** (Table 1, entry 20). Further optimization of reaction temperature found that 80 °C was the optimal choice, and the decrease or increase of reaction temperature had a negative effect on the yield of **4a** and **5a** (see Supporting Information).

With the optimized conditions in hand, the substrate scope for the synthesis of various N^1-substituted triazoles was first investigated. As shown in Scheme 2, aromatic alkynes with a methyl group at a different position of the benzene ring all reacted well to give the desired products **4b**−**4d** in good yields. Other electron-rich groups such as *tert*-butyl and methoxy at the *para* position of phenylacetylene were also compatible with this reaction. Electron-withdrawing substituents including fluorine, chloro, bromo, trifuoromethyl, and cyano were also tolerated to afford the corresponding products **4g**−**4m**, which

Table 1. Optimization of the Reaction Conditions[a]



entry	catalyst (mol %)	oxidant (2.5 equiv)	**4a** yield (%)[b]	**5a** yield (%)[b]
1	CuI (5)	TBHP	30	15
2	CuBr (5)	TBHP	25	30
3	**CuCl (5)**	**TBHP**	**85**	**trace**
4	$Cu(OAc)_2$ (5)	TBHP	50	trace
5	$CuCl_2$ (5)	TBHP	75	trace
6	$CuBr_2$ (5)	TBHP	45	30
7	$AgNO_3$ (5)	TBHP	0	0
8	$Pd(OAc)_2$ (5)	TBHP	0	0
9	$FeCl_3$(5)	TBHP	0	0
10	CuCl (5)	DTBP	12	10
11	CuCl (5)	H_2O_2	10	15
12	CuCl (5)	BPO	50	37
13	CuCl (5)	DDQ	0	0
14	CuCl (5)	$K_2S_2O_8$	0	0
15	CuCl(5)	TBHP(1eq)	65	trace
16	CuCl (5)	TBHP(1.5eq)	70	trace
17[c]	CuCl (5)	TBHP	11	trace
18	CuCl (10)	TBHP	30	30
19	CuCl (15)	TBHP	25	45
20	**CuCl (20)**	**TBHP**	**<5**	**86**
21	CuCl (25)	TBHP	<5	84

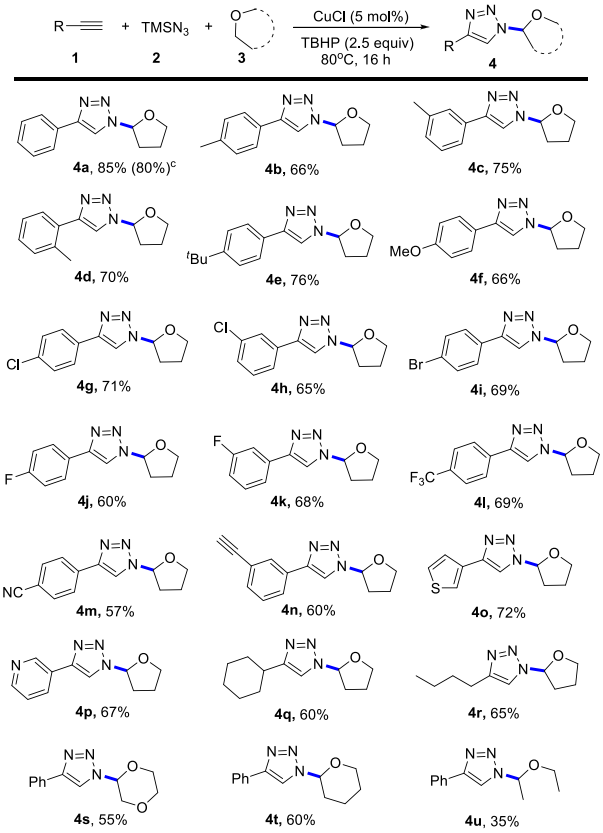
[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), THF **3a** (2 mL), catalyst (5−25 mol %), oxidant (0.5 mmol), 16 h, air, 80 °C. [b]Isolated yield based on **1a**. TBHP (*tert*-butyl hydroperoxide, 70% solution in water). [c]THF (2 mmol), CH_3CN (2 mL).

could be employed for further transformation. It should be noted that the product **4n** was selectively obtained in 60% yield when 1,3-diethynylbenzene was used as the substrate. Notably, heterocyclic aromatic alkynes such as 3-ethynylthiophene and 3-ethynylpyridine were also suitable substrates to produce the desired products **4o** and **4p** in 72% and 67% yields, respectively. Moreover, aliphatic alkynes such as ethynylcyclohexane and hex-1-yne could also react smoothly to afford the corresponding products **4q** and **4r** in good yields. Furthermore, the scope of ether was also investigated under the standard conditions. In addition to tetrahydrofuran, other cyclic ethers such as 1,4-dioxane and tetrahydro-2*H*-pyran were also tolerated in this process to give the desired products in moderate yields. Linear ether such as diethyl ether was also a suitable substrate but leading to the desired product **4u** in relatively lower yield.

After successful demonstration of the synthesis of N^1-substituted triazoles, the scope of the three-component reactions of alkynes, $TMSN_3$, and ethers to selectively construct N^2-substituted triazoles was studied (Scheme 3). Similar to the scope for the construction of N^1-substituted triazoles, it was found that various aromatic alkynes containing both electron-donating and electron-withdrawing groups underwent construction smoothly to afford the desired products **5b**−**5m** in good yields. Heterocyclic aromatic alkyne (i.e., 3-ethynylthiophene) could also provide the corresponding product **5n** in 77% yield. Aliphatic alkynes were also suitable substrates to give the desired products **5o** and **5p** with good yields. With respect to ethers, both cyclic ethers and

DOI: 10.1021/acs.orglett.9b02295
Org. Lett. XXXX, XXX, XXX−XXX

Scheme 2. Scope for the Synthesis of N^1-Oxyalkylated Triazoles[a,b]

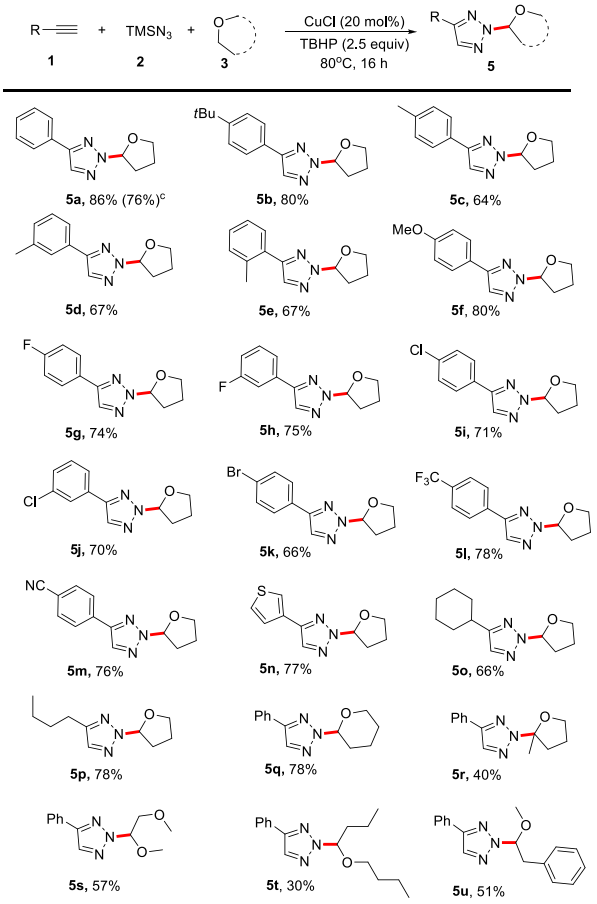


[a]Reaction conditions: **1** (0.2 mmol), **2** (0.4 mmol), ether **3** (2 mL), CuCl (5 mol %), TBHP (0.5 mmol), 16 h, air, 80 °C. [b]Isolated yield based on **1**. [c]**1a** (5 mmol), **2a** (10 mmol), and **3a** (20 mL).

linear ethers were suitable for this protocol (**5q**−**5t**). In general, cyclic ethers showed better activities than linear ethers. Notably, when an asymmetric chain ether such as 2-phenylethyl methyl ether was employed in this reaction system, the desired product **5u** was selectively obtained in 51% yield.

A series of control experiments were performed to probe the possible reaction mechanism, and the results were demonstrated in Scheme 4. First, when TEMPO was added in the model reaction system, the reaction was suppressed, and the TEMPO-trapped complex (TEMPO-THF) was observed by LC-MS, confirming that a radical process should be involved in the present system (Scheme 4a). Subsequently, when THF reacted separately with $TMSN_3$ under the standard reaction conditions, oxyalkylated azide **8a** was detected (Scheme 4b). This result suggested oxyalkylated azide might be involved in the reaction. Moreover, the 4-phenyl-1*H*-1,2,3-triazole **10a** was isolated in 17% yield when the model reaction was carried out in the absence of TBHP (Scheme 4c). The reaction of 4-phenyl-1*H*-1,2,3-triazole **10a** with THF **3a** in CuCl (5 mol %) catalyzed conditions gave the products **4a** and **5a** in 46% and 23% yields, respectively (Scheme 4d). Furthermore, the treatment of 4-phenyl-1*H*-1,2,3-triazole **10a** with THF **3a** under the standard reaction conditions (CuCl, 20 mol %) would produce the products **4a** and **5a** in 16% and 27% yields, respectively (Scheme 4e). These results indicated that phenyl-1*H*-1,2,3-triazole might also be an intermediate in this reaction. In addition, when product **4f** was directly stirred with CuCl (5 mol % and 20 mol %) in THF in the absence of TBHP, the product **5f** could be isolated in 50% and 71% yields, respectively (Scheme 4f and 4g).

Scheme 3. Scope for the Synthesis of N^2-Oxyalkylated Triazoles[a,b]



[a]Conditions: **1** (0.2 mmol), **2** (0.4 mmol), **3** (2 mL), CuCl (20 mol %), TBHP (0.5 mmol), 16 h, air, 80 °C. [b]Isolated yield based on **1**. [c]**1a** (5 mmol), **2a** (10 mmol), and **3a** (20 mL).

Based on the above experimental results and previous reports,[4,5,16,18] we propose possible mechanisms as described in Scheme 5. One pathway for the formation of N^1-oxyalkylated 1,2,3-triazoles could be the initial copper-catalyzed homolytic decomposition of TBHP which generates radical species *t*-BuOO· and *t*-BuO·,[18] which abstract a hydrogen from α-C−H of THF (**3a**) to form alkoxyalkyl radical **6a**. Then, the α-alkoxyalkyl radical **6a** is further oxidized to generate α-alkoxyalkyl cation **7a**. Next, the nucleophilic attack of $TMSN_3$ to **7a** produces oxyalkylated azide **8a**, which undergoes the click reaction with phenylacetylene **1a** to give the desired product **4a** (path A). Another probable pathway is that phenylacetylene **1a** first reacts with $TMSN_3$ leading to 4-phenyl-^{1}*H*-1,2,3-triazole **10a**, which further attacks the α-alkoxyalkyl cation **7a** to give the product **4a** (path B). The pathway for the formation of **5a** should involve the copper-promoted 1,2-shift of the ether group in **4a** via a sequential electronic transfer process.

In summary, we have developed a convenient and regiocontrollable synthetic method to access N^1- and N^2-oxyalkylated 1,2,3-triazoles via a copper-catalyzed three-component reaction of alkynes, $TMSN_3$, and ethers. Through

DOI: 10.1021/acs.orglett.9b02295
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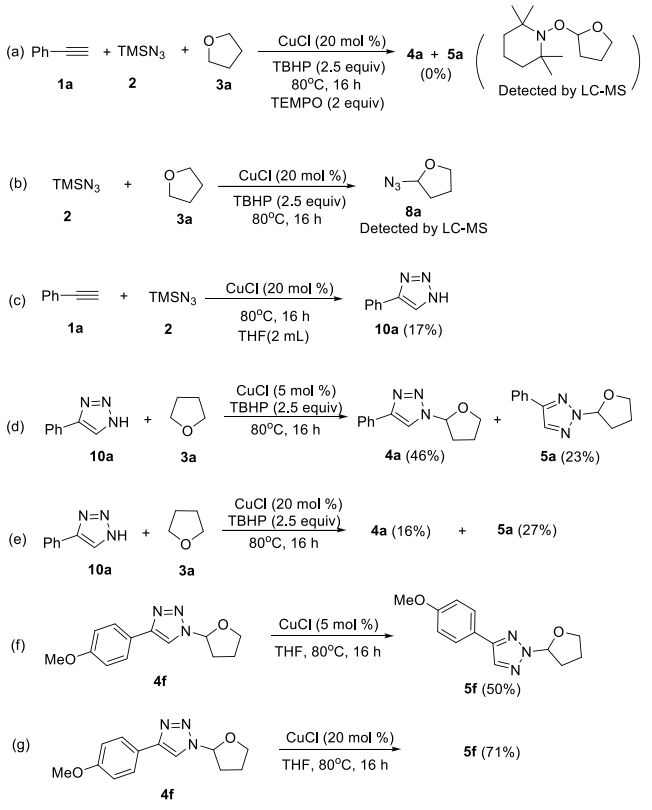
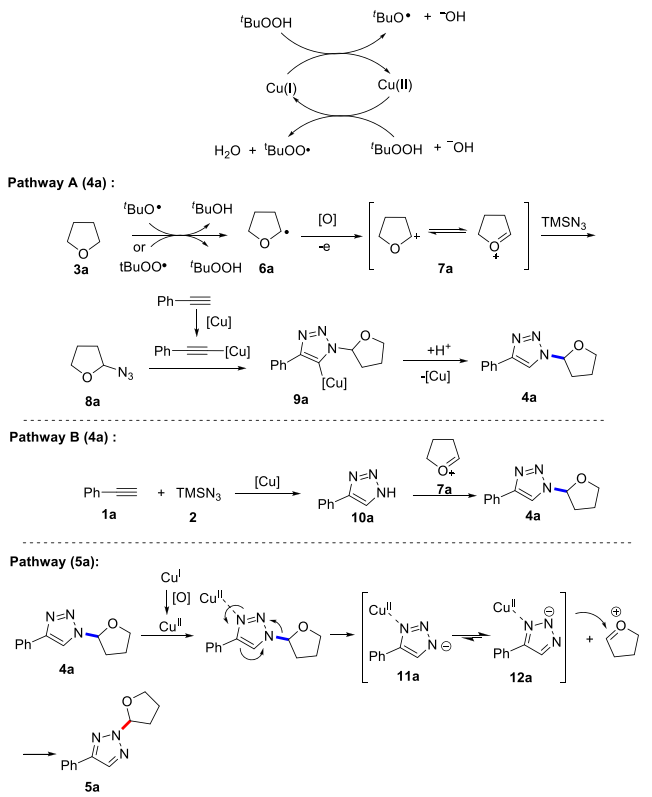
Scheme 4. Control Experiments



Scheme 5. Possible Reaction Mechanism



this protocol, a series of N^1- and N^2-oxyalkylated 1,2,3-triazoles could be efficiently and selectively obtained with moderate to good yields from simple and readily available substrates by use of inexpensive copper salts as catalyst. This methodology exhibits good regioselectivity, favorable functional group tolerance, and a broad substrate scope. Further investigations of the detailed reaction mechanism and synthetic application are currently underway 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.9b02295.

Experimental details and compound characterization (PDF)

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

This work was supported by the Natural Science Foundation of Shandong Province (ZR2018MB009), the International Cooperation Project of Qinghai Province (2018-HZ-806 and 2018-HZ-815), the Qinghai Key Laboratory of Tibetan Medicine Research (2017-ZJ-Y11), and the National Natural Science Foundation of China (No. 21302109).

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DOI: 10.1021/acs.orglett.9b02295
Org. Lett. XXXX, XXX, XXX−XXX