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Consecutive Cycloaddition/S_NAr/Reduction/Cyclization/Oxidation Sequences: A Copper-Catalyzed Multicomponent Synthesis of Fused N-Heterocycles

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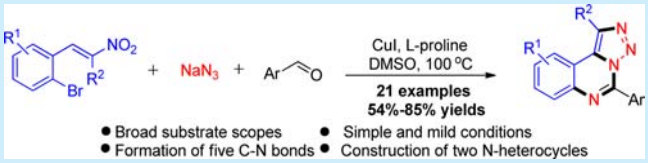
Supporting Information

ABSTRACT: A highly efficient multicomponent domino protocol has been developed for the synthesis of 5-phenyl-[1,2,3]triazolo[1,5-*c*]quinazolines from simple and readily available (*E*)-1-bromo-2-(2-nitrovinyl)benzenes, aldehydes, and sodium azide. This elegant domino process involved consecutive [3 + 2] cycloaddition, copper-catalyzed S_NAr, reduction, cyclization, and oxidation sequences. Notably, sodium azide acted as a dual nitrogen source in the construction of this novel fused N-heterocycle.



Owing to the significance of molecular diversity and complexity in drug discovery and other types of research programs, development of an efficient strategy for rapid synthesis of complex molecular scaffolds is a prevalent theme in organic synthetic chemistry. Among the various synthetic approaches to these substances, those that utilize domino reactions of readily accessible starting materials are highly attractive due to their step and atom economy.[1] [1,2,3]-Triazolo[1,5-*c*]quinazolines belong to a class of structurally novel N-fused heterocycles containing molecules of quinazoline[2] and 1,2,3-triazole[3] frameworks. It is assumed that this novel skeleton may feature promising bioactivity for screening considering analogue fused 1,2,3-triazoles[4] and their component units. To the best of our knowledge, only rare approaches[5] have been reported thus far to construct this novel structure from complex preparative substrates. Therefore, it is fascinating and desirable to design and assemble this structurally novel fused N-heterocycles from simple materials via an elegant domino strategy.

Sodium azide (NaN_3), which is a convenient nitrogen source, has been widely applied in organic synthesis.[6−13] One common function of sodium azide is acting as a 1,3-dipole to react with electron-deficient olefins[7] or alkynes[8] for the synthesis of 1,2,3-triazoles. Many appealing fused N-heterocycles containing the 1,2,3-triazole core have also been constructed via a domino strategy based on this typical [3 + 2] cycloaddition.[9] The nucleophilicity of the nitrogen atom in 1,2,3-triazole generated in situ from [3 + 2] cycloaddition was regarded as the most key point. Additionally, sodium azide was utilized as a coupling partner participating in copper-catalyzed S_NAr reactions.[10] This fundamental reaction was also incorporated with copper-catalyzed azide−alkyne cycloaddition (CuAAC) for the synthesis of 1,4-substituted 1,2,3-triazoles.[11] Recently, transition-metal-catalyzed decomposition reactions of aryl azides to provide nitrenoids have exhibited their potential in N atom transfer reactions.[12] Substantial progress has been made in developing domino reactions that combined copper-catalyzed coupling with subsequent intramolecular N-atom transfer reactions.[13] Despite the above-mentioned advancements, there is still a demand for developing new domino processes related to NaN_3 that allow for efficient construction of complex and diverse N-heterocycles. Herein we envisioned a copper-catalyzed domino strategy for the synthesis of 5-phenyl-[1,2,3]triazolo[1,5-*c*]quinazolines from simple and readily available (*E*)-1-bromo-2-(2-nitrovinyl)benzenes, aldehydes, and sodium azide. In our hypothesis, this elegant domino process integrated consecutive [3 + 2] cycloaddition, copper-catalyzed S_NAr, reduction, cyclization, and oxidation sequences. Notably, 1,2,3-triazoles generated in situ would act as a multitasking functional group to play dual roles in this transformation: (1) the directing group to facilitate following copper-catalyzed S_NAr and reduction; (2) the building block incorporated into fused N-heterocycles (Scheme 1).

Our study commenced with the reaction of (*E*)-1-bromo-2-(2-nitrovinyl)benzene (**1a**), benzaldehyde (**2a**), and sodium azide in the presence of CuI in DMSO at 100 °C in a sealed vessel under air. Gratifyingly, the desired 5-phenyl-[1,2,3]-triazolo[1,5-*c*]quinazoline (**3aa**) identified by NMR spectra was obtained in 47% yield (Table1, entry 1). To further improve the yield, various reaction parameters were evaluated systematically, including ligands, copper salts, solvents, and temperatures; all cases are shown in Table 1. First, a series of ligands were screened for this domino reaction, such as DMEDA,

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Scheme 1. Rational Design for the Synthesis of [1,2,3]Triazolo[1,5-*c*]quinazolines via a Copper-Catalyzed Domino Strategy

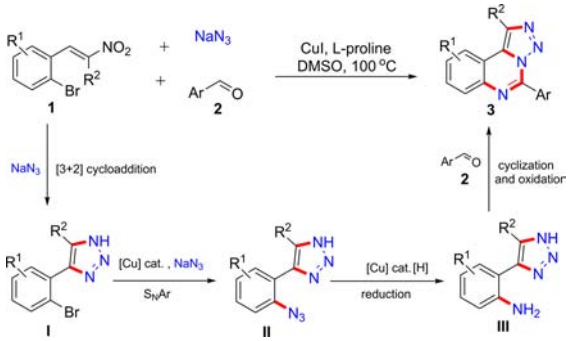
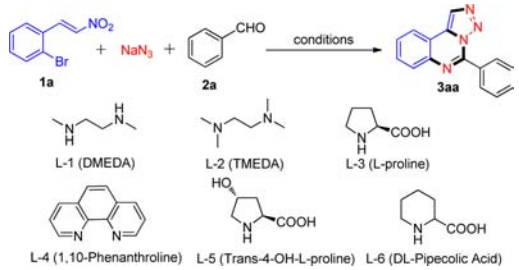


Table 1. Optimization of the Reaction Conditions[a]



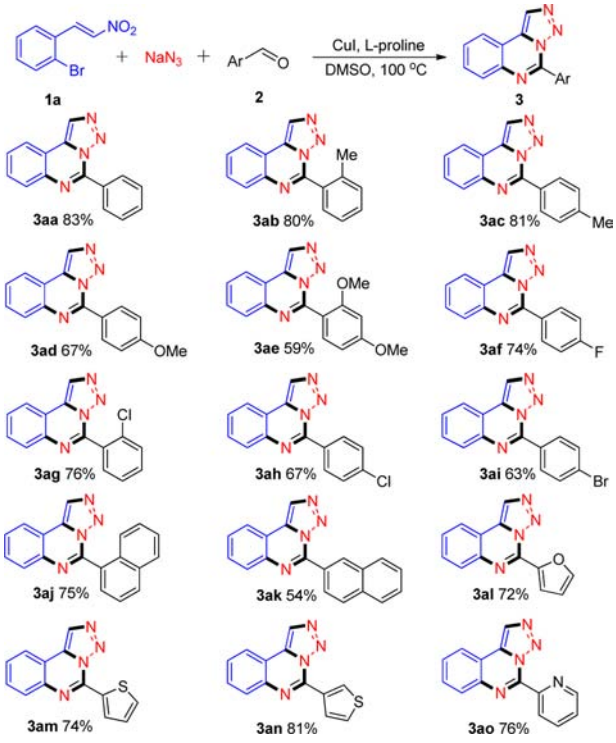
entry	catalyst	ligand	solvent	temp (°C)	yield (%)[b]
1	CuI	–	DMSO	100	47
2	CuI	L-1	DMSO	100	64
3	CuI	L-2	DMSO	100	68
4	**CuI**	**L-3**	**DMSO**	**100**	**83**
5	CuI	L-4	DMSO	100	12
6	CuI	L-5	DMSO	100	74
7	CuI	L-6	DMSO	100	76
8	CuCl	L-3	DMSO	100	74
9	CuBr	L-3	DMSO	100	76
10	Cu_2O	L-3	DMSO	100	78
11	$CuCl_2$	L-3	DMSO	100	71
12	$CuBr_2$	L-3	DMSO	100	73
13	$Cu(OAc)_2$	L-3	DMSO	100	67
14	$Cu(OTf)_2$	L-3	DMSO	100	74
15	CuI	L-3	DMF	100	61
16	CuI	L-3	1,4-dioxane	100	trace
17	CuI	L-3	toluene	100	trace
18	CuI	L-3	DMSO	80	71
19	CuI	L-3	DMSO	110	81
20	–	L-3	DMSO	100	trace

[a]Reactions conditions: **1a** (0.5 mmol), **2a** (0.5 mmol), NaN_3 (1.5 mmol), catalyst (10%), and ligand (20%) were heated in 3 mL of solvent in a sealed vessel under air for 2 h. [b]Isolated yield.

TMEDA, L-proline, 1,10-phenanthroline, *trans*-4-OH-L-proline, and DL-pipecolic acid (Table 1, entries 2–7), and L-proline showed the highest efficiency (Table 1, entry 4). Then, various copper salts were screened against the reaction (Table 1, entries 8–14), and the desired product **3aa** was again obtained in a good yield (67%–78%). Moreover, several solvents were tested (Table 1, entries 15–17), and DMSO proved to be the most effective solvent (compare entries 4 and 15–17). Increasing or decreasing the temperature of the reaction did not lead to any further improvements in the yield (entries 18–19). A control experiment confirmed that the target product was not observed without the copper catalyst (Table 1, entry 20). Finally, the optimized reaction conditions were eventually identified as **1a** (0.5 mmol), 1.0 equiv of **2a**, 3.0 equiv of sodium azide, 10 mol % of CuI, and 20 mol % of L-proline in 3 mL of DMSO at 100 °C in a sealed vessel under air.

To test the substrate generality of this multicomponent reaction (MCR), a series of substituted aromatic aldehydes were investigated under the optimized reaction conditions. To our delight, the reaction demonstrated good compatibility with various aldehydes as shown in Scheme 2. Reactions with

Scheme 2. Scope of Aldehydes[a,b]



[a]Reaction conditions: **1a** (0.5 mmol), **2** (0.5 mmol), NaN_3 (1.5 mmol), CuI (0.05 mmol), and L-proline (0.1 mmol) in DMSO (3 mL) at 100 °C in a sealed vessel under air for 2–4 h. [b]Isolated yields.

aromatic aldehydes containing electron-neutral (4-H, 2-Me, 4-Me) and electron-donating (4-OMe, 2,4-$(OMe)_2$) groups proceeded smoothly to afford the corresponding products in moderate to good yields (59%–83%, **3aa**–**3ae**). Much to our satisfaction, halo-substituted aldehydes were successfully converted directly into target products **3af**–**3ai** in 63% to 76% yields, which provided the possibility for further functionalization. Furthermore, sterically hindered substrates such as 1- and 2-naphthaldehyde were also found to be suitable for this transformation (**3aj**–**3ak**, 75% and 54%). Considering that novel, valuable compounds with multiheterocyclic scaffolds would exhibit enhanced biological activity or vagarious properties, we then explored the reactivity of other representative heteroaryl aldehydes including furanyl, thienyl, and pyridinyl aldehydes. Gratifyingly, all these heteroaryl substrates proved to be compatible under the optimal conditions (**3al**–**3ao**, 72%–81%). Furthermore, the structure of **3ab** was unambiguously determined by X-ray crystallographic analysis (see Supporting Information).

We then examined the scope of this MCR with respect to the nitroolefin's coupling partner. Gratifyingly, the attachment of

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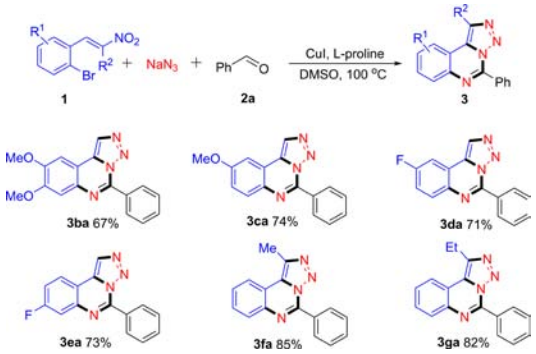
an electron-donating group or halogen to the phenyl group of **1** was well tolerated, with the corresponding products obtained in good yields (Scheme 3, 67%−74%, **3ba**−**3ea**). Moreover, (*E*)-1-bromo-2-(2-nitroprop-1-en-1-yl)benzene (**1f**) and (*E*)-1-bromo-2-(2-nitrobut-1-en-1-yl)benzene (**1g**) also exhibit good reactivity under the optimized conditions (Scheme 3, **3fa**−**3ga**, 85% and 82%).

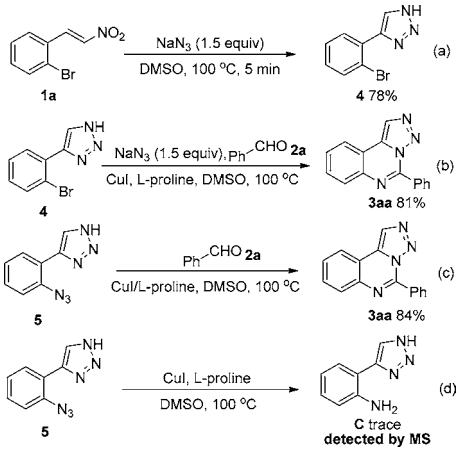
Scheme 3. Scope of Nitroolefins[a,b]



[a]Reaction conditions: **1** (0.5 mmol), **2a** (0.5 mmol), NaN_3 (1.5 mmol), CuI (0.05 mmol), and L-proline (0.1 mmol) in DMSO (3 mL) at 100 °C in a sealed vessel under air for 2−4 h. [b]Isolated yields.

With the scope of the method established, we then turned our attention to evaluate the reaction mechanism. Initially, (*E*)-1-bromo-2-(2-nitrovinyl)benzene (**1a**) with sodium azide (1.5 equiv) were heated in DMSO at 100 °C for 5 min, and 4-(2-bromophenyl)-1*H*-1,2,3-triazole **4** was obtained in 78% yield (Scheme 4a). Substrate 4-(2-bromophenyl)-1*H*-1,2,3-triazole **4**

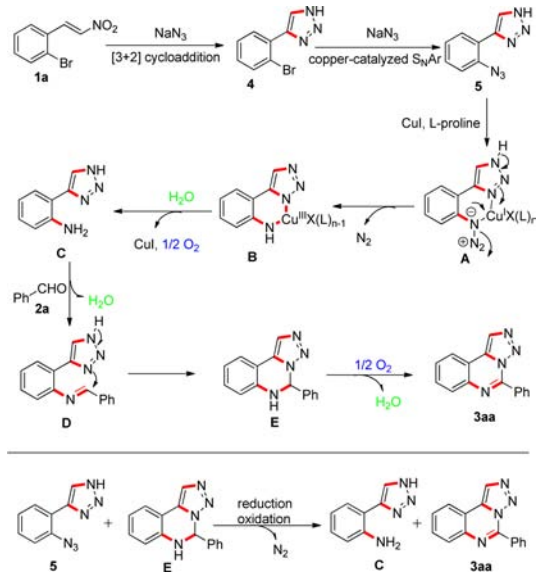
Scheme 4. Control Experiments



was treated with benzaldehyde (**2a**) under standard conditions, and the target product 5-phenyl-[1,2,3]triazolo[1,5-*c*]-quinazoline **3aa** could be isolated in 81% yield (Scheme 4b). Additionally, the reaction of 4-(2-azidophenyl)-1*H*-1,2,3-triazole **5** and benzaldehyde (**2a**) was conducted under standard conditions, and the desired product **3aa** was obtained in 84% yield (Scheme 4c). These results demonstrated that 4-(2-bromophenyl)-1*H*-1,2,3-triazole **4** and 4-(2-azidophenyl)-1*H*-1,2,3-triazole **5** may be key intermediates. When 4-(2-azidophenyl)-1*H*-1,2,3-triazole **5** was treated with CuI and L-proline in DMSO at 100 °C, only a trace of reductive product **C** could be detected by MS. This result could be attributed to the lack of a suitable hydrogen source (Scheme 4d).[10c−e]

Although mechanistic details are not clear at this point, on the basis of experimental results and the literature precedent,[7,10,14−16] a possible reaction mechanism for the copper-catalyzed domino process was proposed using (*E*)-1-bromo-2-(2-nitrovinyl)benzene (**1a**), benzaldehyde (**2a**), and sodium azide as an example (Scheme 5). Initially, the 4-(2-bromophenyl)-1*H*-1,2,3-triazole **4** was formed though a [3 + 2] cycloaddition of (*E*)-1-bromo-2-(2-nitrovinyl)benzene (**1a**) with NaN_3.[7] Subsequently, copper-catalyzed S_NAr of the intermediate **4** with NaN_3 proceeds to form 5-(2-azidophenyl)-2*H*-tetrazole **5** despite the *ortho*-substituent effect.[10,14] Copper-catalyzed denitrogenation of **5** was followed by reduction with the aid of trace H_2O in DMSO to give intermediate 2-(1*H*-1,2,3-triazol-5-yl)aniline **C**. Coordination of azide to copper and further electrocyclization with release of N_2 could generate Cu(III) complex **B**.[15] Next, 2-(1*H*-1,2,3-triazol-5-yl)aniline **C** could easily condense with benzaldehyde (**2a**) to give imine intermediate **D**. Then intramolecular nucleophilic attack of nitrogen to imine in **D** led to **E**. Eventually, the target product **3aa** was obtained after final oxidative dehydrogenation. There is a considerable possibility that the target product **3aa** and 2-(1*H*-1,2,3-triazol-5-yl)aniline **C** could be obtained through synergetic oxidation−reduction occurring between the intermediate **5** and **E**.[16] Further mechanistic studies of the detailed process of reduction and oxidation in this reaction system are in progress.

Scheme 5. A Possible Mechanism



In conclusion, we have developed a highly efficient domino protocol for the rapid synthesis of 5-phenyl-[1,2,3]triazolo[1,5-*c*]quinazolines derivatives from simple and readily available (*E*)-1-bromo-2-(2-nitrovinyl)benzenes, aldehydes, and sodium azide. This elegant domino process integrated consecutive [3 + 2] cycloaddition, copper-catalyzed S_NAr, reduction, cyclization, and oxidation sequences. Notably, sodium azide acted as a dual nitrogen source in the construction of these fused N-heterocycles. Further application of this self-sequence strategy utilizing NaN_3 as a simple nitrogen donor for the synthesis of other fascinating N-heterocycles is underway in our laboratory.

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■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures, product characterizations, crystallographic data, and copies of the ^{1}H and ^{13}C NMR spectra are involved. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b01242.

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Notes

The authors declare no competing financial interest.

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