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Letter

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Conversion of 2-Furylcarbinols with Alkyl or Aryl Azides to Highly Functionalized 1,2,3-Triazoles via Cascade Formal [3 + 2] Cycloaddition/Ring-Opening

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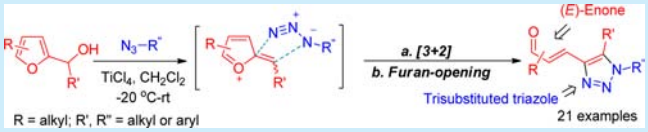
Supporting Information

ABSTRACT: A Lewis acid promoted cascade cycloaddition/ring-opening of 2-furylcarbinols with alkyl or aryl azides is described. The reaction features an initial formal [3 + 2] cycloaddition to form a trisubstitued triazole motif, followed by a ring opening of furan to generate the (*E*)-configuration of the enone. A wide range of highly functionalized triazoles is expediently and efficiently synthesized in a highly step-economical manner.



The rapid and reliable construction of heterocyclic frameworks is an integral part of programs directed toward drug discovery and the development of molecular tools that probe biochemical and cellular function. As important privileged scaffolds, 1,2,3-triazoles are endowed with a broad spectrum of biological activities.[1] They have been extensively used in organic synthesis,[2] biochemistry,[3] and material science.[4] In this context, derivatives of 1,2,3-triazoles, when conjugated with a α,β-unsaturated carbonyl skeleton, represent a family of useful molecules. Such frameworks are especially attractive for drug discovery because of high hit rates and pharmacological profiles (Figure 1). For example, BRL-42715 (**1**) has been shown to be a

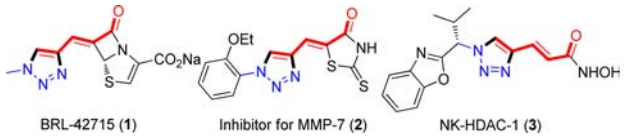


Figure 1. Selected useful 1,2,3-triazoles with a (*E*)-enone.

potent inhibitor of most bacterial β-lactamases including the class I β-lactamase, which is resistant to other β-lactamase inhibitors,[5] and triazole (**2**) is a potent inhibitor for matrix metalloproteases (MMPs) that is linked to many serious human diseases.[6] NK-HDAC-1 (**3**) is promising lead for the inhibition of histone deacetylase with great bioavailability and in vivo half-life.[7]

However, although this core skeleton is useful, the current strategies for its synthesis generally require tedious multistep syntheses, and no simple and direct approach from easily available precursors is known so far. Here, we report an efficient synthesis of 1,2,3-triazole motif which conjugated with a α,β-unsaturated carbonyl by a Lewis acid-promoted cascade formal [3 + 2] cycloaddition/ring-opening of alkyl azides with 2-furylcarbinols.

Huisgen 1,3-dipolar azide−alkyne cycloadditions (AAC) have been extensively developed,[8] among which the copper-catalyzed conditions (CuAAC),[9] metal-activated AAC,[10] and AAC using reactive strained alkynes have been powerful synthetic tools with numerous applications.[11] Compared to general alkynes, C−C triple bonds activated by electron-withdrawing groups furnish triazolations under mild conditions.[12] Enamine and enolate conjugated with carbonyls also afford triazoles via a cycloaddition/elimination process.[13] However, all of these methods lack synthetic capabilities for the structurally complicated triazoles mentioned above. Thus, more versatile and general strategies of rapid triazole constructions should be studied.

The low resonance energy of the furan ring[14] allows for its facile recyclization reactions into different carbo-[15] and heterocycles.[16] In particular, furfuryl cations have also served as well-known reactive intermediates.[17] In considering the significance of the above-mentioned 1,2,3-triazole skeleton and inspired by the high degree of synthetic flexibility of furfurylcarbinol, we questioned whether a carbon-cation induced cascade strategy could expedite the assembly of the highly substituted triazole core linked to α,β-unsaturated carbonyl. We envisioned that the key structural architecture of **3** could be directly generated from highly polarized olefin in a furfuryl cation by a [3 + 2] cycloaddition with alkyl azide to produce the triazole framework and subsequent furan ring-opening to form the α,β-unsaturated carbonyl (Figure 2).

Following this designed strategy, we chose the 5-methylfurfurylcarbinol **4a** and benzyl azide **6a** as the standard substrates for

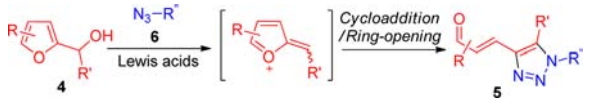


Figure 2. Strategy for construction of conjugated 1,2,3-triazoles.

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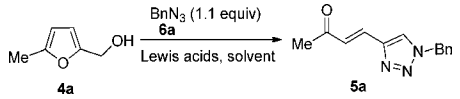
our attempt to seek an effective cation-generating condition for this domino reaction. Initially, no product formation was observed in the presence of the Brønsted acids such as TFA, TfOH, and *p*-TsOH at room temperature in acetonitrile, and the reflux was also not suitable for this conversion and led to a complicated reaction mixture (Table 1, entries 1−3). Strong

Table 1. Optimization Studies for the Synthesis of 5a



entry[a]	acid	solvent	equiv	t(°C)	time (min)	yield[b] (%)
1	TFA	CH_3CN	1.1	rt to 80	120	ND[c]
2	TfOH	CH_3CN	1.1	rt to 80	120	ND[c]
3	*p*-TsOH	CH_3CN	1.1	rt to 80	120	ND[c]
4	$AlCl_3$	CH_2Cl_2	1.1	−20 to rt	30	46
5	$FeCl_3$	CH_2Cl_2	1.1	−20 to rt	30	20
6	$TiCl_4$	CH_2Cl_2	1.1	−20 to rt	30	71
7	$TiCl_4$	CH_2Cl_2	0.1	−20 to rt	30	5
8	$InCl_3$	CH_2Cl_2	1.1	rt to 80	120	trace
9	$Sc(OTf)_3$	CH_3CN	0.1	rt to 80	120	trace
10	$Ir(OTf)_3$	CH_3CN	0.1	rt to 80	120	ND[c]
11	TMSOTf	CH_2Cl_2	1.1	−20 to rt	120	ND[c]
12	no acid	toluene		reflux	120	NR[d]

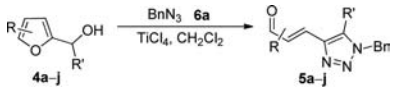
[a]Conditions: 5-methylfurfurylcarbinol **4a** (1.0 mmol), benzyl azide **6a** (1.1 mmol), solvent (5 mL). [b]Isolated yield. [c]ND: not detected. [d]NR: no reaction.

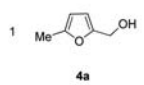
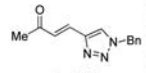
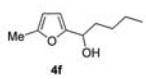
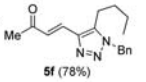
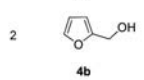
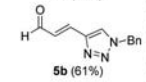
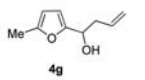
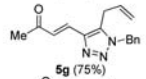
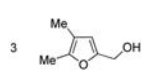
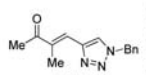
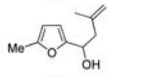
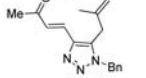
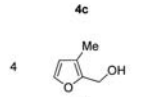
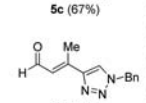
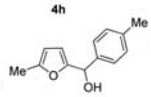
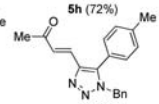
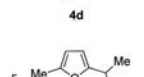
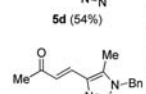
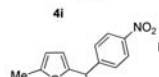
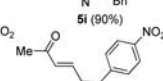
Lewis acids were then investigated. We were pleased to find that the desired triazole could be generated using $AlCl_3$ and $FeCl_3$ at ambient temperature and dichloromethane as the solvent in 46% and 20% yields (entries 4 and 5). Among them, $TiCl_4$ gave the best yield (71%) with a short reaction time (entry 6). However, the extended reaction time cannot be beneficial. On the other hand, a low product yield was observed when the reaction was performed with 10 mol % of $TiCl_4$ (entry 7). Meanwhile, weaker Lewis acids $InCl_3$ and $Sc(OTf)_3$ also induced this transformation, but the reaction was much slower and a certain amount of unidentical byproducts was obtained (entries 8 and 9). Other Lewis acids (e.g., $Ir(OTf)_3$ and TMSOTf) were proven to be ineffective or result in decomposition of starting material (entries 10 and 11). Finally, no desired product was observed when there was no Lewis acid at reflux in toluene (entry 12).

It is worthy of note that in this reaction, the azide group in **6a** together with the methylene and C2 of furfurylcarbinol **4a** are transferred to the triazole skeleton, while the remaining C2−C4 carbons of furan are converted into the enone moiety in product **5a**. Remarkably, only (*E*)-enone was observed (coupling constant of two olefinic hydrogens is 16 Hz); the (*Z*)-isomer was not detected according to the ^{1}H NMR of the crude reaction mixture.

We next examined a range of different substrates under the optimal reaction conditions ($TiCl_4$, −20 °C to rt, 30 min). The results are summarized in Table 2. Simple furfurylcarbinol **4b** gave aldehyde **5b** in 61% yield (Table 2, entry 2). By investigating the substituents on furans, 4,5-dimethyl and 3-methyl substrates **4c** and **4d** were found to be effective to generate corresponding trisubstituted olefins **5c** and **5d**, respectively (entries 3 and 4). The carbonyls and triazoles were individually located at the *trans*-positions to double bonds. The yields with secondary alcohols (**4e**−**h**) were generally good for benzyl azide **6a**, and

Table 2. Variations of Furfurylcarbinol 4



entry[a]	substrate	product[b] (yield[c])	entry[a]	substrate	product[b] (yield[c])
1		 5a (71%)	6		 5f (78%)
2		 5b (61%)	7		 5g (75%)
3		 5c (67%)	8		 5h (72%)
4		 5d (54%)	9		 5i (90%)
5		 5e (82%)	10		 5j (79%)

[a]Conditions: 2-furylcarbinols **4** (1.0 mmol), benzyl azide **6a** (1.1 mmol), $TiCl_4$ (1.1 mmol), CH_2Cl_2 (5 mL), −20 °C to rt. [b]Only the *E*-isomer was observed by ^{1}H NMR in each example. [c]Isolated purified yield (average of two runs).

trisubstituted triazoles (**5e**−**h**) were obtained, which could be well rationalized by the enhancement of the reactivity and stability of secondary cation compared to primary cation. Homoallylic alcohols **4g** and **4h** were well tolerated during the reaction, affording **5g** and **5h** in 75% and 72% yields, respectively (entries 7 and 8). Benzylic **4i** and **4j** led to significant increases in yields (entries 9 and 10). Considering the electronic effect of the aromatic ring for benzyl cations revealed that the electron-donating group could enhance reactivity.

The present method could be applied successfully to various azides **6**, whether alkyl or aryl, to provide the corresponding (*E*)-enones or -enals in generally good to high yields (Table 3). The aromatic ring of benzyl azides bearing an electron-withdrawing group (−Br) or an electron-donating group (−OMe) were all compatible, furnishing the corresponding enones **5l** and **5m** in 74% and 72% yields, respectively (entries 1 and 2). The alkyl-substituted azide **6d** underwent recyclization with furfurylcarbinol **4b** smoothly, leading to **5n** in 68% yield (entry 3). Secondary and tertiary azides (**6e**, **6f**, and **6g**) were also reacted with furfurylcarbinol in moderate yields, although in those cases, prolonged reaction times were required, presumably due to the more steric bulkiness of the substrates. Allyl and ester azides **6h** and **6i** could also be used in the reaction, and a 73% yield of **5r** and a 66% yield of **5s** were achieved (entries 7 and 8). In contrast, aromatic azides, which are known to be relatively stable (entries 9 and 10), can be applicable to the domino processes as well.

Then, triazole **5k** was easily afforded from cinnamyl azide **6l** and 5-methyl furfuralcohol **4a** with a single (*E*)-enone in good yield (Scheme 1). This product has a similar core structure of NSC746457, which was discovered by Wang's group as a lead compound for the inhibition of histone deacetylase.[7a] By contrast, in order to construct *trans*-olefin and triazole with propiolic acid as starting material, a series of key reactions were applied in the literature synthesis, including hydroiodination, Sonogashira coupling, and click reaction. The result clearly

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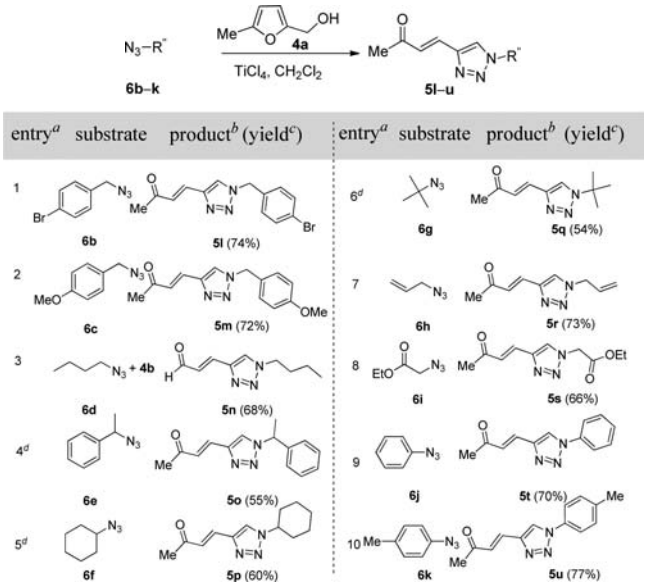
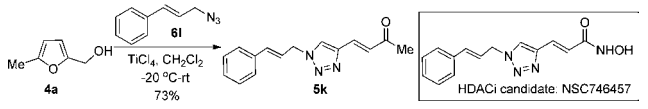
Table 3. Variations of Azides 6



[a]Conditions: 2-furylcarbinols **4a** or **4b** (1.0 mmol), azides **6** (1.1 mmol), $TiCl_4$ (1.1 mmol), CH_2Cl_2 (5 mL), −20 °C−rt. [b]Only the *E*-isomer was observed by ^1H NMR in each example. [c]Isolated purified yield (average of two runs). [d]The reaction was prolonged to 120 min.

Scheme 1. Synthetic Utility



demonstrates the exceptional advantages of our approach, which would pave a feasible way for the inhibitor synthesis.

To assign the substitution positions of triazoles and the *trans*-stereospecificity of double bonds beyond a doubt, the structures of **5l** and **5n** were unambiguously confirmed by X-ray crystallographic analysis, which clearly showed the alkyl chain and enone (or enal) were located at the 1 and 4 positions of triazoles individually and that the configuration of the double bonds was trans (Figure 3). In the solid structure, the C═C−C═O unit is oriented approximately perpendicular to the central triazole ring.

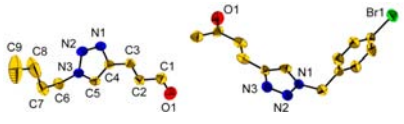
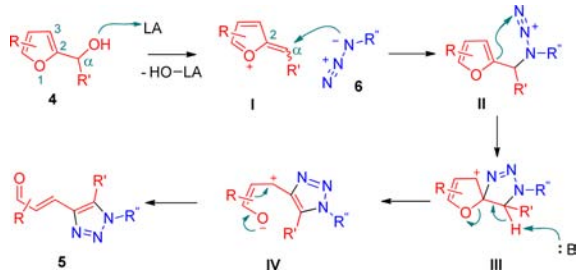


Figure 3. ORTEP drawing of **5n** (left CCDC no. 1005591) and **5l** (right CCDC no. 999263).

The proposed mechanism for cascade formal [3 + 2] cycloaddition/ring-opening implies initial dehydroxylation of 2-furylcarbinols **4** with Lewis acids to produce oxocarbenium **I**, in which the more electron poor α-C, rather than C2 of furan, undergoes nucleophilic attack of the azides to form the intermediate aminodiazonium **II** (Scheme 2). This step leads to exclusive regioselectivity of the final triazole. The subsequent intramolecular Friedel−Crafts-like reaction between C-2 position of the furans and azides closes to spiro-dihydrofuran cations **III**, which is configured to aromatize to triazole by β−H elimination in the vicinity of oxygen, followed by ring-opening of

Scheme 2. Proposed Reaction Pathway (See Text for Details)



furan to deliver the enolate zwitterions **IV**. Finally, tautomerization of enolates leads to the formation of the corresponding enones or enals in an exclusive *trans*-configuration, which is thermodynamically favored in this single-step transformation. Further studies are underway to help elucidate the mechanism and the stereoselectivity event of this cascade transformation.

In summary, we have developed a method for generating highly functionalized 1,2,3-triazoles based on a novel cascade reaction of 2-furylcarbinols and azides. This method proved to have wide substrate scope, and the valuable assets of this reaction are further represented by its high step economy, diversity, and high (*E*)-stereospecificity. This efficient process, employing readily available substrates and reagents, is expected to find applications in the construction of a diverted library for chemical biology and drug discovery.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures, product characterization, copies of NMR spectra, and crystallographic data for **5l** and **5n** (CIF). This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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