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One-Pot Synthesis of 1-Monosubstituted 1,2,3-Triazoles from Propargyl Alcohol

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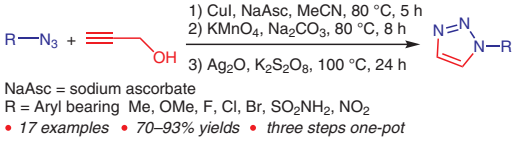

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Abstract A one-pot synthesis of 1-monosubstituted-1,2,3-triazoles from propargyl alcohol and various aryl azides was achieved. This simple method provides concise and efficient access to various 1-monosubstituted 1,2,3-triazole derivatives through a three-step one-pot sequence in good to excellent yields.

Key words triazoles, propargyl alcohol, aryl azides, copper catalysis

As an important group of heterocyclic compounds containing a five-membered ring with three nitrogen atoms, 1,2,3-triazole derivatives are widely applied in many fields, such as biology,[1] materials science,[2] and medicinal[3] and synthetic organic chemistry.[4] In particular, in the last ten years many compounds of this type have found use as clinical and commercial drugs such as antibiotics,[5] indoleamine 2,3-dioxygenase (IDO) inhibitors,[6] antiviral drugs,[7] and histone deacetylase inhibitors (HDIs) (Figure 1).[8]

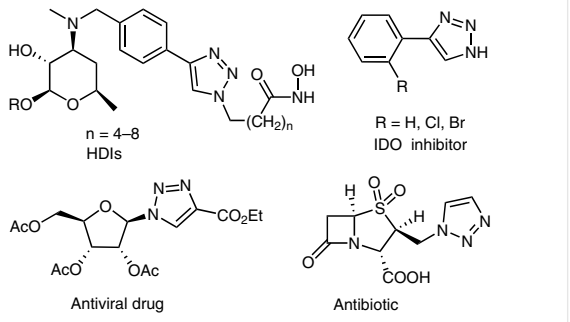

Figure 1 Some 1,2,3-triazoles possessing various pharmaceutical activities

Owing to their wide range of uses, several strategies for the syntheses of 1,2,3-triazoles have been reported. The first method that was used to construct the 1,2,3-triazole ring was the Huisgen dipolar cycloaddition, which gives 1,4- and 1,5-disubstituted regioisomers without regioselectivity; in this reaction, an alkyne and an azide are mixed and heated.[9] In 2002, the Sharpless group[10] developed a copper-catalyzed 1,3-dipolar cycloaddition reaction of terminal alkynes and azides for the regioselective construction of 1,4-disubstituted 1,2,3-triazoles. This method is simple and vigorous. Subsequently, these compounds came into the limelight, attracting interested researchers who explored more-effective methods for the construction of this type of molecule through various approaches.[11] For example, the Fokin group[12] used a triazole ligand to stabilize Cu(I), which can vigorously catalyze the Huisgen cycloaddition reaction to form 1,4-substituted 1,2,3-triazoles at ambient temperatures. Orgueira et al.[13] found that active nanoparticulate copper also catalyzes the Huisgen cycloaddition reaction with high efficiency in a broad range of solvents, including THF, MeOH, MeCN, DMSO, and DMF.[14] Ramachary et al.[15] reported an organocatalytic enolate-mediated synthesis of 1,2,3-triazoles from aldehydes and aryl azides as starting materials, which constitutes an important alternative method. Meanwhile, syntheses of 1,5-disubstituted 1,2,3-triazoles were reported, in which ruthenium or a base was usually applied as a catalyst.[16] Recently, 1,4,5-trisubstituted 1,2,3-triazoles have been synthesized by using a three-component system or from starting materials other than terminal alkynes and azides.[17] Some simple one-pot syntheses have been demonstrated that use aryldiazonium silica sulfates,[18] arylboronic acids,[19] aryl halides,[20] or aromatic amines[21] as starting materials.

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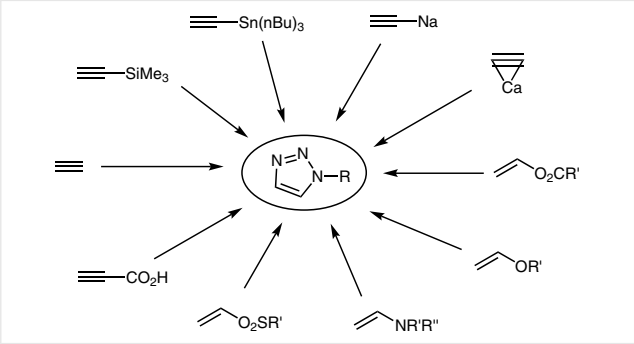
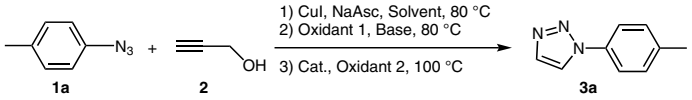


Scheme 1 Methods for synthesizing 1-monosubstituted 1,2,3-triazoles

Owing to their recently identified particular biological activity, 1-monosubstituted 1,2,3-triazole derivatives have attracted a great deal of attention, especially in relation to their preparation, and they have been mainly prepared from azides and various acetylene sources, including acetylene[22] and its derivatives, such as acetylides [ethynyl(trimethyl)silane, ethynyl(tributyl)tin, sodium acetylide, or calcium carbide];[23] vinyl compounds (vinyl acetate, vinyl ethers, vinyl amines, or vinyl sulfoxides);[24] or propiolic acid (Scheme 1).[25]

As part of our continuing interest in the synthesis and modification of various 1,2,3-triazole derivatives,[26] we describe a convenient and efficient one-pot three-step method for the preparation of monosubstituted 1,2,3-triazoles **3**

Table 1 Selected Optimizations of the Reaction Conditions[a]



Entry	Solvent	Oxidant 1 (2.5 equiv)	Base (1.5 equiv)	Catalyst (mol%)	Oxidant 2 (2 equiv)	Yield[b] (%)
1	DMF	–	–	–	–	62[c]
2	DMF–H_2O (8:1)	–	–	–	–	76[c]
3	MeCN–H_2O (8:1)	–	–	–	–	68[c]
4	MeCN	–	–	–	–	96[c]
5	MeCN	^tBuOOH	–	–	–	74[d]
6	MeCN	$K_2S_2O_8$	–	–	–	88[d]
7	MeCN	$KMnO_4$	–	–	–	72[d]
8	MeCN	$KMnO_4$	KOH	–	–	82[d]
9	MeCN	$KMnO_4$	K_2CO_3	–	–	86[d]
10	MeCN	$KMnO_4$	Na_2CO_3	–	–	92[d]
11	MeCN	$KMnO_4$	Na_2CO_3	$PdCl_2$ (20)	$Cu(OAc)_2$	0[e]
12	MeCN	$KMnO_4$	Na_2CO_3	$Pd(OAc)_2$ (20)	$Cu(OAc)_2$	25[e]
13	MeCN	$KMnO_4$	Na_2CO_3	AgOAc (20)	$Cu(OAc)_2$	30[e]
14	MeCN	$KMnO_4$	Na_2CO_3	AgOAc (20)	$K_2S_2O_7$	36[e]
15	MeCN	$KMnO_4$	Na_2CO_3	Ag_2CO_3 (20)	$K_2S_2O_7$	30[e]
16	MeCN	$KMnO_4$	Na_2CO_3	$AgNO_3$ (20)	$K_2S_2O_7$	52[e]
17	MeCN	$KMnO_4$	Na_2CO_3	Ag_2O (20)	$K_2S_2O_7$	86[e]
18	MeCN	$KMnO_4$	Na_2CO_3	Ag_2O (20)	$(NH_4)_2S_2O_7$	74[e]
19	MeCN	$KMnO_4$	Na_2CO_3	Ag_2O (20)	$KMnO_4$	26[e]
20	MeCN	$KMnO_4$	Na_2CO_3	Ag_2O (20)	–	32[e]
21	MeCN	$KMnO_4$	Na_2CO_3	Ag_2O (10)	$K_2S_2O_7$	88[e]
22	MeCN	$KMnO_4$	Na_2CO_3	Ag_2O (5)	$K_2S_2O_7$	65[e]

[a] Reaction conditions: (1) 1-azido-4-methylbenzene (**1a**; 0.3 mmol), propargyl alcohol (**2**; 0.36 mmol), CuI (0.03 mmol), NaAsc (0.06 mmol), solvent (2 mL) solvent, 15 mL sealed pressure tube, stirring, 80 °C; (2) oxidant 1 (0.75 mmol), base (0.45 mmol), stirring, 80 °C; (3) catalyst and oxidant 2 (0.6 mmol), stirring, 100 °C.
[b] Isolated yield.
[c] Yield of [1-(4-tolyl)-1*H*-1,2,3-triazol-4-yl]methanol.
[d] Yield of 1-(4-tolyl)-1*H*-1,2,3-triazole-4-carboxylic acid.
[e] Yield of 1-(4-tolyl)-1*H*-1,2,3-triazole (**3a**).

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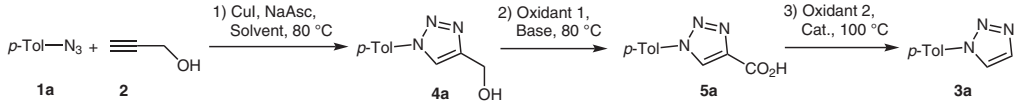
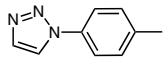
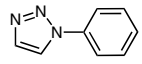
by using aryl azides **1** and propargyl alcohol (**2**) as starting materials.

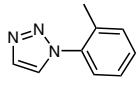
We chose the reaction of 1-azido-4-methylbenzene (**1a**) and propargyl alcohol (**2**) as a model system (Table 1). Initially, we explored the first step of the process by using CuI and sodium ascorbate (NaAsc) as a catalyst system in various solvents, and we obtained the intermediate product [1-(4-tolyl)-1*H*-1,2,3-triazol-4-yl]methanol in 62% yield by using DMF as a solvent, through a Cu-catalyzed azide–alkyne Huisgen cycloaddition (Table 1, entry 1). DMF–H_2O (8:1), MeCN–H_2O (8:1), and MeCN were also examined as solvents, and an excellent yield of 96% was obtained in MeCN after five hours (entries 2–4). We then studied the second process of oxidizing the intermediate product [1-(4-tolyl)-1*H*-1,2,3-triazol-4-yl]methanol to 1-(4-tolyl)-1*H*-1,2,3-triazole-4-carboxylic acid by using various oxidants and bases (entries 5–10). A combination of $KMnO_4$ and Na_2CO_3 was the best choice, giving a 92% yield after eight hours (entry 10). *t*-BuOOH, $K_2S_2O_8$, $KMnO_4$, $KMnO_4$–KOH, and $KMnO_4$–K_2CO_3 gave inferior results as oxidants (entries 5–9). Encouraged by these results, we screened various catalysts and oxidants for the final step of the process (entries 11–20). None of the target molecule was detected when we used $PdCl_2$ as a catalyst and $Cu(OAc)_2$ as the oxidant (entry 11). When the $PdCl_2$ catalyst was replace with $Pd(OAc)_2$, the reaction gave 1-(4-tolyl)-1*H*-1,2,3-triazole (**3a**) in 25% yield (entry 12). We then investigated other catalysts (AgOAc, Ag_2CO_3, $AgNO_3$, and Ag_2O) (entries 13–17) and we found that Ag_2O was the most efficient. Oxidant screening showed that $K_2S_2O_7$ was the best choice, giving an 86% yield (entry 17). An oxidant is essential for this coupling, as a very low yield was obtained in the absence of an oxidant (entry 20). Next, we examined the amount of Ag_2O and we found that 10% $AgNO_3$ was efficient in this transformation, affording the desired product **3a** in 88% yield (entry 21); reducing the amount to 5% an inferior result was obtained(entry 22).

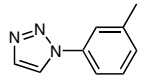
Table 2 Substrates scope[a,b]

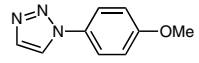


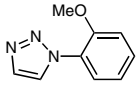

3a; 88%

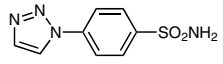

3b; 83%

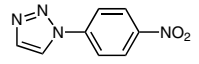

3c; 81%

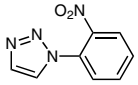

3d; 83%

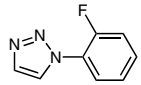

3e; 93%

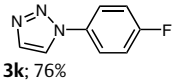

3f; 87%

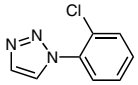

3g; 79%

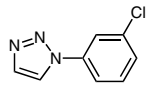

3h; 73%

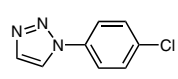

3i; 70%

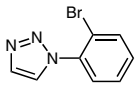

3j; 70%

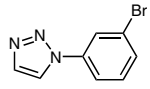

3k; 76%

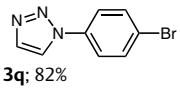

3l; 73%


3m; 75%


3n; 81%


3o; 77%


3p; 80%


3q; 82%

[a] *Reaction conditions*: (1) azide **1** (0.3 mmol), propargyl alcohol (**2**; 0.36 mmol), CuI (0.03 mmol), NaAsc (0.06 mmol), MeCN (2 mL), sealed 15 mL pressure tube, 80 °C, 5 h. (2) $KMnO_4$ (0.75 mmol), Na_2CO_3 (0.45 mmol), 80 °C, 8 h; (3) Ag_2O (0.03 mmol), $K_2S_2O_7$ (0.6 mmol), 100 °C, 24 h.[27]
[b] Yields of the isolated products after column chromatography are reported.

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By using the optimized reaction conditions, we then explored the scope of the aryl azide in this transformation (Table 2). A broad spectrum of substrates bearing various substituents was investigated. All the reactions proceeded smoothly and they consistently gave the target molecules **3a–q** in good to excellent yield, regardless of whether the substrates bore electron-donating or electron-withdrawing substituents (Table 2, 3a–q). The reactions of aryl azides with electron-donating groups such as methyl or methoxy in the *ortho*-, *meta*-, or *para*-position all gave the corresponding products in good yields (**3a–f**). Substrates with an electron-withdrawing group such as as sulfonamide, nitro, fluoro, chloro, or bromo also reacted smoothly, although the yields were somehow lower (**2g–q**).

Aryl azides with substituents in the *para*-position produced higher yields than did those bearing groups in the *meta*- or *ortho*-position, probably owning to the steric effects (Table 2, 3a versus **3c** and **3d** or **3e** versus **3f**). Note that substrates possessing more-electron-rich groups gave higher yields of the corresponding products. Furthermore, a substrate bearing group a sulfonamido group was also suitable for this transformation, giving a good yield of the corresponding product **3g**.

In summary, we successfully synthesized 1-monosubstituted 1,2,3-triazoles from propargyl alcohol and various aryl azides as starting materials. The 1-monosubstituted 1,2,3-triazole derivatives were readily prepared in good to excellent yields by a simple three-step one-pot sequence.

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Supporting Information

Supporting information for this article is available online at https://doi.org/10.1055/s-0036-1589157.

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(27) **1-Substituted 1*H*-1,2,3-Triazoles; General Procedure**
Aryl azide **1** (0.3 mmol), propargyl alcohol (**2**; 0.36 mmol), CuI (0.03 mmol), NaAsc (0.06 mmol), and MeCN (2 mL) were added to a 15 mL pressure tube. The tube was sealed and the mixture was stirred at 80 °C for 5 h until the reaction was complete. $KMnO_4$ (0.75 mmol) and Na_2CO_3 (0.45 mmol) were added, and the mixture was stirred at 80 °C for 8 h. Ag_2O (0.03 mmol) and $K_2S_2O_7$ (0.6 mmol) were then added, and the mixture was heated at 100 °C for 24 h until the reaction was complete (TLC). H_2O (25 mL) was added, and the mixture was extracted with EtOAc (3 × 20 mL). The organic layers were combined, washed with brine (3 × 5 mL), dried (Na_2SO_4), and concentrated under reduced pressure to afford a crude product that was purified by column chromatography [silica gel, EtOAc–PE (1:3)].

1-(4-Tolyl)-1*H*-1,2,3-triazole (3a)
White solid; yield: 42 mg (88%); mp 85.5–86.5 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.96 (d, *J* = 0.8 Hz, 1 H), 7.83 (s, 1 H), 7.62 (d, *J* = 8.4 Hz, 2 H), 7.32 (d, *J* = 8.2 Hz, 2 H), 2.43 (s, 3 H).

1-(2-Methoxyphenyl)-1*H*-1,2,3-triazole (3f)
White solid; yield: 46 mg (87%); mp 81–82.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.13 (d, *J* = 1.0 Hz, 1 H), 7.82 (d, *J* = 1.0 Hz, 1 H), 7.79 (dd, *J* = 7.9, 1.7 Hz, 1 H), 7.46–7.41 (m, 1 H), 7.14–7.08 (m, 2 H), 3.89 (s, 3 H).

4-(1*H*-1,2,3-Triazol-1-yl)benzenesulfonamide (3g)
White solid; yield: 53 mg (79%); mp 187–187.5 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.93 (s, 1 H), 8.14 (d, *J* = 8.6 Hz, 2 H), 8.04 (d, *J* = 6.1 Hz, 3 H), 7.55 (s, 2 H).

1-(3-Chlorophenyl)-1*H*-1,2,3-triazole (3m)
White solid; yield: 40 mg (75%); mp 91.6–92.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.02 (d, *J* = 1.1 Hz, 1 H), 7.86 (d, *J* = 1.0 Hz, 1 H), 7.80 (t, *J* = 2.0 Hz, 1 H), 7.66 (ddd, *J* = 7.9, 2.0, 1.3 Hz, 1 H), 7.51–7.40 (m, 2 H).

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