



Synthesis of 1,4-Disubstituted-1,2,3-Triazoles Catalyzed by Part-per-Million CuI in Magnetized Distilled Water

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Abstract

The use of an extremely low quantity of copper is always a significant factor in Cu-catalyzed azide-alkyne cycloaddition reaction (CuAAC). A significant quantity of CuI showed excellent catalytic activity for the click reaction of terminal alkynes, benzyl chloride derivatives, and sodium azide in magnetized distilled water (MDW) as a green solvent.

Keywords: Triazole, Copper Iodide, Terminal alkyne, Ligand-free, MDW.

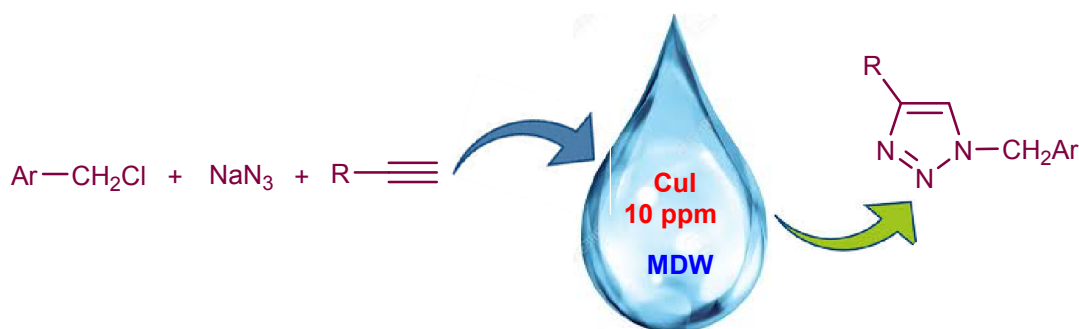
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Introduction

In recent years, 1,2,3-triazoles have been used in organic chemistry, agrochemicals, dyes, and medicinal chemistry [1,2]. For example, 1,2,3-triazoles have displayed broad biological activities such as anti-helminthic, antitumor, and pharmacological properties [3-6]. Copper-catalyzed azide-alkyne cycloaddition (Cu AAC) reaction access to 1,2,3-triazoles was reported by Sharpless et.al [7]. Recently, various copper catalysts such as CuI [8, 9], Cu₂O [10], CuBr (PPh₃)₃ [11], CuSO₄[12, 13], Cu/C [14], and Cu/SiO₂[15] have been reported for this purpose.

A large quantity of a copper metal catalyst is employed in the classic CuAAC reactions. While Cu salts pollute the biologically related compounds and are toxic, their complete elimination from the products is difficult. This complication prevents the use of CuAAC reactions in biomedicine and clinical research. The ligands permit the usage of Cu catalysts in reduced amounts compared to the original CuAAC reactions, which is yet the most typically used catalyst but in more significant quantities that are often even stoichiometric or higher to stoichiometry [16, 17]. For this purpose, the development of ligand-free, using an extremely low quantity of copper, cheaper, highly efficient, more general, and using water instead of organic solvents is favorable for the preparation of 1,2,3-triazoles.

Water magnetization is a simple procedure without extreme utilization when a permanent external magnet is used. Two primary methods of making MDW has been reported. The first method is passing water through a magnetic field, and the second is using a permanent magnet near a particular volume of water. The magnetic field intensity and the time of magnetization have a significant effect on the properties of MDW [18-22]. In continuation of our recent studies on the synthesis of heterocyclic compounds in MDW [23-26], we wish to report a ligand-free, green, simple, and efficient method for the synthesis of 1,4-disubstituted-1,2,3-triazoles catalyzed by CuI (10 ppm) in MDW (Scheme 1).



Scheme 1. Synthesis of 1,4-disubstituted-1,2,3-triazoles in MDW.

Experimental

Material and equipment

All chemicals were used as reagent grade, and received without further purification. ^1H NMR spectra were recorded using Bruker Advance DPX-250 NMR FT-300 MHz spectrometers instrument in $\text{DMSO-}d_6$. Coupling constants (J) are reported in hertz (Hz), and multiplicities are indicated as follows: s (singlet), d (doublet), dd (doublet of doublet), t(triplet), q (quartet), m (multiplet). IR spectra were recorded on a Shimadzu IR-435 grating spectrophotometer.

Preparation of magnetized distilled water

Distilled water (3 mL) was put in a test tube, which was then put between two neodymium magnets NdFeB (10 cm \times 5 cm \times 4 cm) with a magnetic field of 0.8 T for 15 min. In the next step, the test tube containing MDW was taken out from the apparatus and used for the reaction.

General procedure for the synthesis of 1,4-disubstituted-1,2,3-triazoles (**4a-r**)

A benzyl chloride (1.0 mmol), sodium azide (1.1 mmol), a terminal alkyne (1.0 mmol), were stirred at 80 °C for 3 h in MDW (3 mL). After the reaction was completed by TLC detection, cooled, and the crude product was separated and recrystallized with ethanol to get product **4** (Table 2).

4-((4-nitrophenoxy) methyl) - 1-benzyl-1H-1, 2, 3-triazole (**4d**)

Yellow solid (90%); ^1H NMR ($\text{DMSO-}d_6$, 300 MHz) δ = 5.3(s, 2H, CH_2), 5.6 (s, 2H, CH_2), 7.2(d, J =9.3Hz, 2.1Hz, 2H, ArH), 7.3-7.4(m, 5H, ArH), 8.2 (d, J =9.3 Hz, 2H, ArH), 8.3 (s, 1H, CH of triazole) ppm.

1-(2-chlorobenzyl)-4-((4-nitrophenoxy) methyl)-1H-1, 2, 3-triazole (**4e**)

Yellow solid (85%); ^1H NMR ($\text{DMSO-}d_6$, 300 MHz) δ = 5.3 (s, 2H, CH_2), 5.7 (s, 2H, CH_2), 7.2-7.3 (m, 3H, ArH), 7.3-7.4 (m, 2H, ArH), 7.5 (dd, J = 7.5 Hz, 1.8 Hz, 1H, ArH), 8.2 (d, J = 9.3 Hz, 2H, ArH), 8.3 (s, 1H, CH of triazole) ppm; ^{13}C NMR ($\text{DMSO-}d_6$, 75 MHz) δ = 51.1, 62.3, 115.8, 125.9, 126.3, 128.2, 130.1, 130.8, 131.0, 133.1, 133.6, 141.5, 142.3, 163.7 ppm; IR (KBr): 1595 (C=C), 1340 (N=O), 1255 (C-O) cm^{-1} .

1-(4-chlorobenzyl)-4-((4-nitrophenoxy) methyl)-1H-1, 2, 3-triazole (**4f**)

Yellow solid (90%); ^1H NMR ($\text{DMSO-}d_6$, 300 MHz) δ = 5.3 (s, 2H, CH_2), 5.6 (s, 2H, CH_2), 7.2(d, J =9 Hz, 2H, ArH), 7.3 (d, J =8.4 Hz, 2H, ArH), 7.4 (d, J =8.4 Hz, 2H, ArH), 8.23 (d, J =9.3 Hz, 2H, ArH), 8.37 (s, 1H, CH of triazole) ppm; ^{13}C NMR ($\text{DMSO-}d_6$, 75 MHz) δ = 52.5,

62.3, 115.8, 125.6, 126.3, 129.2, 130.4, 133.4, 135.3, 141.5, 142.5, 163.7 ppm; IR (KBr): 1597 (C=C), 1338 (N=O), 1261 (C-O) cm^{-1} .

2-((1-benzyl-1H-1,2,3-triazol-4-yl)methoxy)benzaldehyde (4g)

White solid (94%); ^1H NMR (DMSO- d_6 , 300 MHz) δ = 5.3 (s, 2H, CH_2), 5.6 (s, 2H, CH_2), 7.1 (t, J = 7.35 Hz, 1H, ArH), 7.3-7.4(m, 6H, ArH), 7.6-7.7 (m, 2H, ArH), 8.4 (s, 1H, CH of triazole), 10.3 (s, 1H, CHO) ppm.

2-((1-(2-chlorobenzyl)-1H-1, 2, 3-triazol-4-yl) methoxy) benzaldehyde (4h)

White solid (88%); ^1H NMR (DMSO- d_6 , 300 MHz) δ = 5.3 (s, 2H, CH_2), 5.7 (s, 2H, CH_2), 7.1(t, J = 7.35 Hz, 1H, ArH), 7.2 (dd, J = 7.2, 2.1 Hz, 1H, ArH), 7.3-7.4 (m, 3H, ArH), 7.5 (dd, J = 7.5, 1.5 Hz, 1H, ArH), 7.6-7.7 (m, 2H, ArH), 8.3(s, 1H, CH of triazole), 10.3 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO- d_6 , 75 MHz) δ = 51.1, 62.6, 114.8, 121.6,125,125.7, 128.1, 128.2,130.1,130.7,130.9,133.0, 133.7, 136.8, 142.9, 160.8, 189.6 ppm; IR (KBr): 1595 (C=C), 1680 (C=O), 1296 (C-O) cm^{-1} .

2-((1-(4-chlorobenzyl)-1H-1,2,3-triazol-4-yl)methoxy)benzaldehyde (4i)

White solid (90%); ^1H NMR (DMSO- d_6 , 300 MHz) δ = 5.3 (s, 2H, CH_2), 5.6 (s, 2H, CH_2), 7.1 (t, J = 7.05 Hz, 1H, ArH), 7.3-7.4 (m, 5H, ArH), 7.6-7.7 (m, 2H, ArH), 8.4(s, 1H, CH of triazole), 10.3 (s, 1H, CHO) ppm; IR (KBr): 1595 (C=C), 1672 (C=O), 1242 (C-O) cm^{-1} .

4-((1-benzyl-1H-1, 2, 3-triazol-4-yl) methoxy) benzaldehyde (4j)

White solid (91%); ^1H NMR (DMSO- d_6 , 300 MHz) δ = 5.3 (s, 2H, CH_2), 5.6 (s, 2H, CH_2), 7.25(d, J = 8.7 Hz, 2H, ArH), 7.3-7.4 (m, 5H, ArH), 7.9 (d, J = 8.7, 2H, ArH), 8.3(s, 1H, CH of triazole), 9.9 (s, 1H, CHO) ppm.

3-((1-benzyl-1H-1, 2, 3-triazol-4-yl) methoxy)-4-methoxybenzaldehyde (4k)

Brown solid (70%); ^1H NMR (DMSO- d_6 , 300 MHz) δ = 3.8 (s, 3H, OCH_3), 5.2 (s, 2H, CH_2), 5.6(s, 2H, CH_2), 7.2 (d, J = 8.7 Hz, 1H, ArH), 7.3-7.4 (m, 5H, ArH), 7.5-7.6 (m, 2H, ArH), 8.3(s, 1H, CH of triazole), 9.8 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO- d_6 , 75 MHz) δ = 53.3, 56.3, 62.1, 112.0, 112.1,125.4, 126.8, 128.4,128.6, 129.2, 130.0, 136.4, 143.0, 148.2, 154.9, 191.8 ppm; IR (KBr): 1595 (C=C), 1687 (C=O), 1261 (C-O) cm^{-1} .

3-((1-(2-chlorobenzyl)-1H-1,2,3-triazol-4-yl)methoxy)-4-methoxy benzaldehyde(4l)

Brown solid (79%); ^1H NMR (DMSO- d_6 , 300 MHz) δ = 3.8(s, 3H, OCH₃), 5.2 (s, 2H, CH₂), 5.7 (s, 2H, CH₂), 7.1-7.2 (m, 2H, ArH), 7.3-7.4 (m, 2H, ArH), 7.5-7.6 (m, 3H, ArH), 8.2 (s, 1H, CH of triazole), 9.8 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO- d_6 , 75 MHz) δ = 51.0, 56.3, 62.0, 112.1, 112.2, 125.8, 126.8, 128.2, 130.0, 130.1, 130.7, 131.0, 133.1, 133.7, 142.9, 148.1, 154.9, 191.8 ppm.

3-((1-(4-chlorobenzyl)-1H-1,2,3-triazol-4-yl)methoxy)-4-methoxybenzaldehyde (4m)

Brown solid (75%); ^1H NMR (DMSO- d_6 , 300 MHz) δ = 3.8 (s, 3H, OCH₃), 5.22 (s, 2H, CH₂), 5.6 (s, 2H, CH₂), 7.2 (d, J = 8.7 Hz, 1H, ArH), 7.3(d, J = 8.7 Hz, 2H, ArH), 7.4 (d, J = 8.7 Hz, 2H, ArH), 7.5-7.6 (m, 2H, ArH), 8.3 (s, 1H, CH of triazole), 9.8 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO- d_6 , 75 MHz) δ = 52.5, 56.3, 62.1, 112.0, 112.1, 125.4, 126.8, 129.2, 130.0, 130.4, 133.4, 135.4, 143.1, 148.2, 154.9, 191.7 ppm; IR (KBr): 1591 (C=C), 1685 (C=O), 1265 (C-O) cm^{-1} .

Results and discussion

Magnetized distilled water was prepared similar to priorly reported [25]. The test tube containing distilled water (3 mL) was put in a magnetic field (0.8 T) for 15 minutes. Subsequently, the prepared MDW was used for the reaction. (Figure 1).

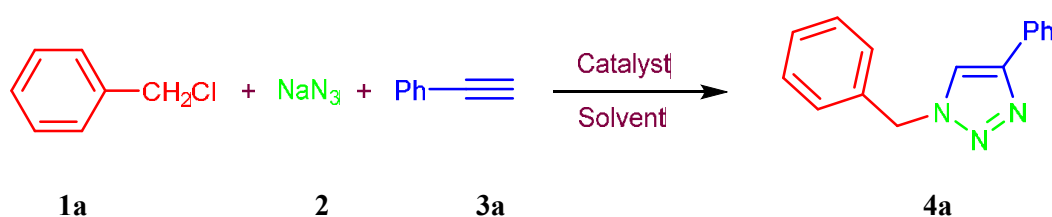


Figure 1. The pilot for solvent magnetization apparatus.

To demonstrate the effect of MDW in the synthesis of 1,2,3-triazoles, initially, the reaction of benzyl chloride **1a**, sodium azide **2**, and phenylacetylene **3a**, as a model reaction, was performed in the presence of CuI, Cu₂O, CuO, CuSO₄/NaAsc, and Cu(OAc)₂/NaAsc as a catalyst in MDW at 80 °C for 3 h (Table 1, entries 1-5). As shown in Table 1, the reaction in the presence of the CuI afforded a higher isolated yield (Table 1, entry 1). Thus, we tried to optimize the reaction conditions using CuI as a catalyst. At 50 °C, the click reaction did not progress well, and the reaction had a low product yield (Table 1, entry 6). Remarkably,

when MDW was replaced with other solvents such as H₂O, EtOH, MeOH, DMF, dioxane, and CH₃CN, product yield was drastically decreased (Table 1, entries 7-12). Furthermore, the efficiency of this method has been examined again for the model reaction with various amounts of CuI (Table 1, entries 13-17). According to Table 1, when the amount of CuI is 500 ppm, the product yield **4a** is 95% in 3 h (Table 1, entry 1). Interestingly, when the reaction was performed using 10 ppm of CuI, product **4a** was 94% yield (Table 1, entry 16). Upon decreasing the CuI (1 ppm), product **4a** was found to be an 85% yield (Table 1, entry 17).

Table 1. Effects of various parameters; solvent, catalyst, and temperature on the synthesis of 1-benzyl-4-phenyl-1*H*-1,2,3-triazole **4a**^a.



Entry	Catalyst (ppm)	Solvent	Temperature	Yield ^b (%)
1	CuI (500)	MDW	80	95
2	Cu ₂ O (500)	MDW	80	64
3	CuO (500)	MDW	80	55
4 ^c	CuSO ₄ (500)	MDW	80	45
5 ^c	Cu(OAc) ₂ (500)	MDW	80	61
6	CuI (500)	MDW	50	75
7	CuI (500)	H ₂ O	80	70
8	CuI (500)	EtOH	80	65
9	CuI (500)	MeOH	65	61
10	CuI (500)	DMF	80	24
11	CuI (500)	CH ₃ CN	80	40
12	CuI (500)	Dioxane	80	29
13	CuI (400)	MDW	80	95
14	CuI (200)	MDW	80	95
15	CuI (100)	MDW	80	94
16	CuI (10)	MDW	80	94
17	CuI (1)	MDW	80	85

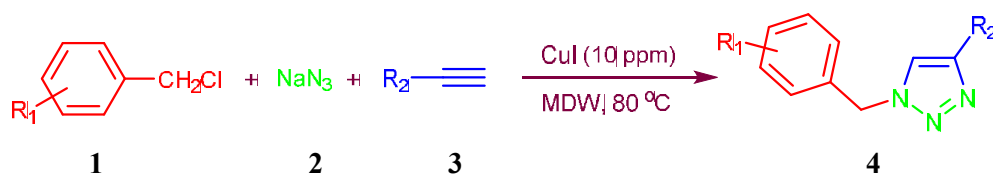
^aReaction conditions: benzyl chloride **1a** (1.0 mmol), sodium azide **2** (1.1 mmol), phenyl acetylene **3a** (1.0 mmol), solvent (3 mL). ^bIsolated yield. ^cSodium ascorbate (1000 ppm).

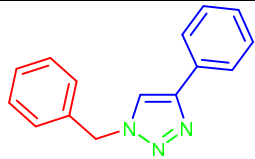
It is noteworthy that a meager amount of CuI has never been successful for ligand-free Cu-catalyzed azide-alkyne cycloaddition reactions before the present study. Recently, Astruc *et al.* have

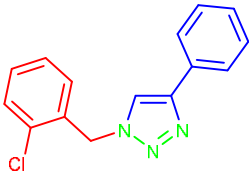
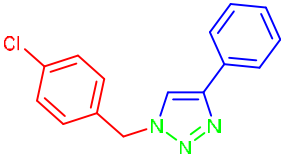
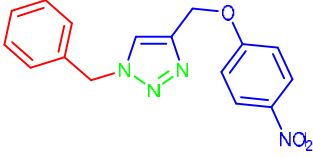
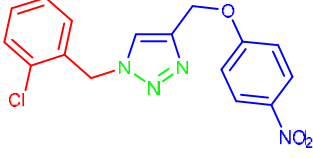
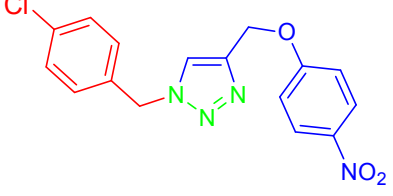
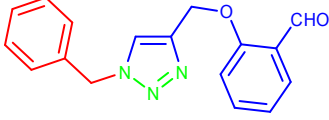
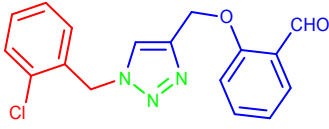
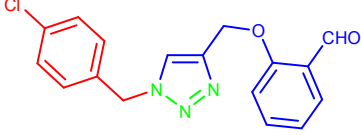
reported the CuAAC reactions of alkyl/aryl azides with terminal alkynes catalyzed by CuI (4-200 ppm) in the presence of dendrimer ligand at 30 °C for 24 h [27]. Moreover, Vincent et al. have used the copper (I)-tren catalyst (500 ppm) for the synthesis of 1,2,3-triazoles in toluene at 60 °C for 24 h [28]. They also reported that using 10 ppm of copper (I)-tren catalyst, 1-benzyl-4-phenyl-1*H*-1,2,3-triazole **4a** was obtained in 54%. Whereas in the present work, the ligand-free click reactions catalyzed by CuI (10 ppm) in MDW at 80 °C for 3 h, an excellent reaction yield (94%) was obtained.

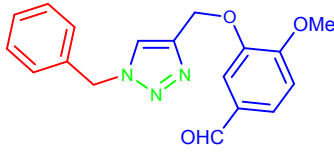
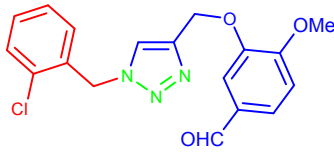
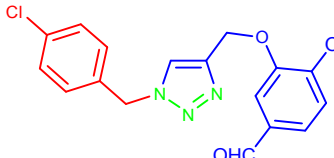
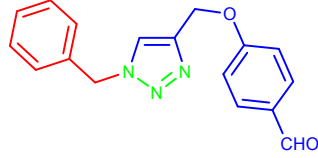
Under the optimized conditions (Table 1, entry 16), the click reaction has been performed in MDW for seventeen other substrates to study the generality of the procedure for this reaction. Various terminal alkynes, and benzyl chloride derivatives were investigated in the reaction (Table 2). According to Table 2, the CuAAC reactions of various substrates, leading to 1,4-disubstituted triazoles in good-to-excellent yields (70-95%). Notably, various aromatic alkynes bearing withdrawing groups undergo the CuAAC reaction to give the 1,2,3-triazoles in high yields (Table 2, entries 4-10). The aryl alkynes with electron-donating groups (OCH₃) provided the corresponding 1,4-disubstituted triazoles in good yields (Table 2, entries 11-13). Moreover, various propargyl alcohols gave the products **4n**, **4o**, **4p**, **4q**, and **4r** with yields of about 93, 76, 70, 91, and 85%, respectively (Table 2, entries 14-18).

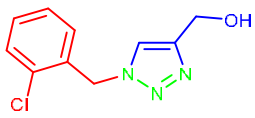
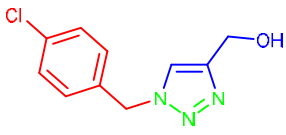
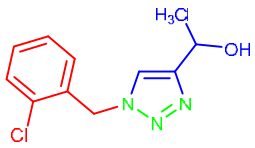
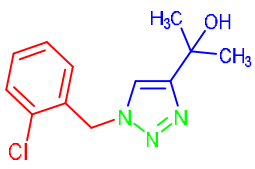
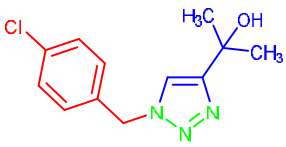
Table 2. Reaction times, melting points, and yields of 1,4-disubstituted-1,2,3-triazoles **4^a**.



Entry	R ₁	R ₂	Time (h)	Product	m.p. (°C)	Yield (%) ^b
					Observed/Reported [Ref.]	
1	H	Ph	3		125-127	128-129 [29] 95

2	2-Cl	Ph	4		86-88	88-89 [30]	81
				4b			
3	4-Cl	Ph	3		130-132	132-134 [31]	88
				4c			
4	H	4-NO ₂ - C ₆ H ₄ -OCH ₂	5		93-95	94-96 [31]	90
				4d			
5	2-Cl	4-NO ₂ - C ₆ H ₄ -OCH ₂	5		100-102	This work	85
				4e			
6	4-Cl	4-NO ₂ - C ₆ H ₄ -OCH ₂	5		98-100	This work	90
				4f			
7	H	2-CHO- C ₆ H ₄ -OCH ₂	5		128-130	130-132 [31]	94
				4g			
8	2-Cl	2-CHO- C ₆ H ₄ -OCH ₂	3		100-102	This work	88
				4h			
9	4-Cl	2-CHO- C ₆ H ₄ -OCH ₂	3		118-120	This work	90
				4i			

10	H	2-MeO-4-CHO-C ₆ H ₃ -OCH ₂	4		121-123	This work	70
				4k			
11	2-Cl	2-MeO-4-CHO-C ₆ H ₃ -OCH ₂	5		95-97	This work	79
				4l			
12	4-Cl	2-MeO-4-CHO-C ₆ H ₃ -OCH ₂	5		84-86	This work	75
				4m			
13	H	4-CHO-C ₆ H ₄ -OCH ₂	4		85-87	85-87 [32]	91
				4j			

14	2-Cl	CH ₂ OH	5		109-111	110-112 [33]	93
				4n			
15	4-Cl	CH ₂ OH	4		76-78	79-80 [33]	76
				4o			
16	2-Cl	CH ₃ CHOH	4		58-60	59-61 [33]	70
				4p			
17	2-Cl	(CH ₃) ₂ COH	5		98-100	100-102 [33]	91
				4q			
18	4-Cl	(CH ₃) ₂ COH	5		95-97	97-99 [33]	85
				4r			

^aReaction conditions: benzyl chloride **1** (1.0 mmol), sodium azide **2** (1.1 mmol), terminal alkyne **3** (1.0 mmol), MDW (3 mL), magnetization time (15 min), 80 °C.

^bIsolated yield.

Conclusions

We have developed a practical, simple, and eco-friendly synthesis of 1,4-disubstituted 1,2,3-triazoles by the reaction of benzyl chloride derivatives, sodium azide, and terminal alkynes *via* the copper-catalyzed click reactions in MDW. In addition, CuAAC reactions were carried out at 80 °C with 10 ppm of commercial CuI for quantitative yields and 1 ppm with 85% yield. This new procedure furnishes an occasion to use MDW and offers numerous advantages, including high reaction yield, cleaner reaction profile, low costs, short reaction times, environmentally benign, and easy workup.

Acknowledgements

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