Inorganica Chimica Acta 506 (2020) 119470

Contents lists available at ScienceDirect

Inorganica Chimica Acta

journal homepage: www.elsevier.com/locate/ica

Research paper

Click reactions catalyzed by Cu(I) complexes supported with dihydrobis(2-mercapto-benzimidazolyl)borate and phosphine ligands

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ARTICLE INFO

Keywords:
Cu(I) complex
Dihydrobis(2-mercapto-benzimidazolyl)borate
1,4-Disubstituted 1,2,3-triazole
Bioactive ligand
Epoxides

ABSTRACT

Four Cu(I) complexes bearing dihydrobis(2-mercapto-benzimidazolyl)borate and phosphine co-ligands were synthesized and their catalytic activity was investigated in azide-alkyne reactions. These complexes were tested as catalyst system and among them a Cu(I) complex bearing tricyclohexylphosphine ligand, [Cu(Bb)(PCy_3)], showed superior catalytic activity in water. The introduced ligands are advantageous due to their nontoxicity, strong σ-donating ability, and the ease of their handling process. The selected catalyst allowed the clean preparation of triazoles in water which is considered a safe medium according to the principles of green chemistry. These catalysts were prepared by reacting copper iodide with sodium precursor (NaBb) [Bb = dihydrobis(2-mercapto-benzimidazolyl)borate in methanol in the presence of selected phosphine coligands (PPh_3, PCy_3, PPh_2Me, PPh_2Py). All of the resulting Cu(I) complexes were formed as predominantly a single monomeric isomer and were characterized using a combination of ^1H, $^{13}C\{^1H\}$ and $^{31}P\{^1H\}$ NMR spectroscopy.

1. Introduction

Five-membered *N*-heterocycles, as important structural motifs, are present in a large class of drugs, natural products, and biologically active compounds. They also display various medicinal activities ranging from antibiotic, antitumor, antibacterial, antifungal, and antiviral properties. Considering these activities as well as being valuable intermediates in the synthesis of organic compounds, the facile and efficient preparation of these compounds encourage further investigations [1–3].

Among *N*-heterocycles, the 1,2,3-triazole scaffolds have been well exploited for the creation of numerous medicinal moieties exhibiting anticancer, anti-HIV, and antibacterial properties [4–9]. There are structurally diverse bioactive compounds bearing 1,2,3-triazole moiety and some of them are also well acknowledged for their therapeutic effects as elucidated in Fig. 1. Some of these compounds serve as important intermediates in numerous industrial and laboratory applications including corrosion inhibitors [10,11], agrochemicals [12], ligands [13–21], dendrimers, polymers [22,23], linkers, catalysts, and pigments [24].

Traditionally, triazole products have been prepared *via* thermal 1,3-dipolar [3 + 2] cycloaddition of alkynes with organic azides [25]. The major obstacles of exiting CuAAC methodologies include high reaction temperatures, long reaction times and the presence of reducing agents. These issues restrict their applications in practical processes. To overcome these issues, great efforts have been made by researchers in the past several years. For example, Cu(I)-catalyzed azide-alkyne cycloaddition (CuAAC), introduced by Sharpless and coworkers in 2001, has found a widespread application in chemistry, biochemistry and material science [26,27].

The Sharpless-Meldal C—N bond forming reactions are catalyzed by a variety of copper sources and ligands. The importance of these ligands in the formation of triazole products has promoted widespread studies toward their synthesis. For instance, Sharpless et al. reported polydentate triazole ligands in “click” reaction were responsible for enhanced catalytic activity of Cu(I) complexes through binding to the metal [28]. Sharghi et al. studied the auxiliary effect of many ligands in copper catalyzed Sharpless-Meldal C—N bond-forming reactions [29–32].

The derivatives of 2-mercaptobenzimidazole are associated with a wide range of pharmacological activities including antihistaminic, antiviral, anticancer, and antifungal activities [33–35]. They have also been utilized as supporting ligands for late transition metals such as copper ions [36–40]. We recently reported the synthesis,

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https://doi.org/10.1016/j.ica.2020.119470
Received 12 December 2019; Received in revised form 24 January 2020; Accepted 25 January 2020
Available online 31 January 2020
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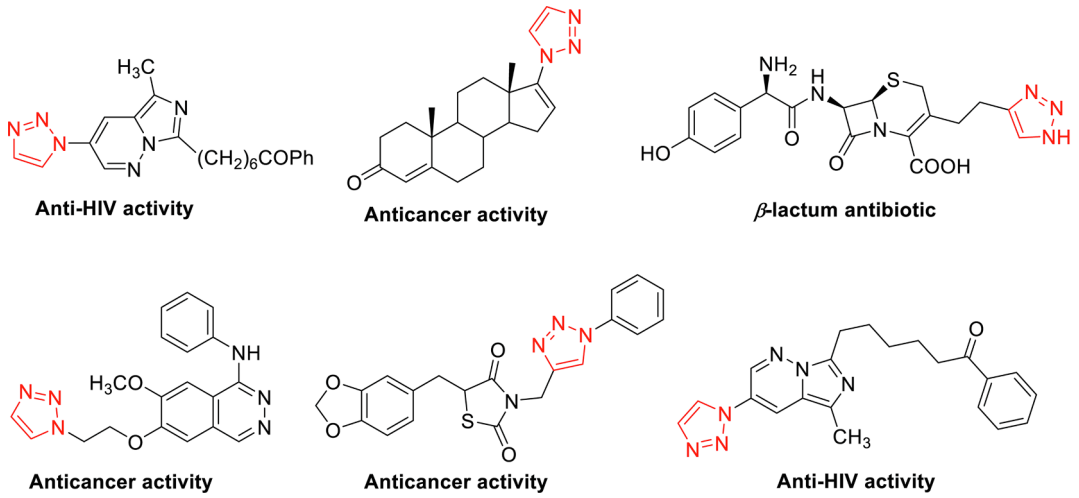


Fig. 1. Chemical structure of some commercial drugs and bioactive molecules bearing triazole moiety.

characterization and photophysical properties of a series of Cu(I) complexes supported with these types of compounds. In our work, two 2-mercaptobenzothiazole or 2-mercaptobenzimidazole moieties were tethered to a bridgehead boron atom commonly providing a chelating ligand system known as "scorpionate ligands" [41]. These softer congeners of multi-dentate borate based "scorpionate ligands" play a crucial role in the development of stable coordination compounds [42,43]. The coordination chemistry of bipodal and tripodal sulfur donor scorpionate ligands with electron rich middle to late transition metals has been the subject of extensive research studies. The main feature of borate based ligands include their ability to act as bidentate or tridentate ligands and to stabilize a multitude of metal ions with different oxidation states. This feature has been demonstrated with a lot of reports regarding synthesis and applications of metal complexes bearing borate based ligands. The majority of literature reports regarding polydentate N, P or S donor borate based ligands include description of their interesting structural features [44–47], agostic interactions of metal centers with B-H moiety of borate ligands [43,48–52], bioinorganic aspects of metal complexes bearing such ligands, as well as their catalytic activities [53–56]. Since active species in copper catalyzed alkyne-azide reactions are Cu(I) ions, developments in the synthesis and optimized reactions conditions including copper complexes of borate based ligands has resulted in a surge in the application of borate based copper and their closed counterparts based on silver complexes in click reactions [57–59].

In this study. we explored the catalytic activities of a series of Cu(I) complexes supported with bidentate sulphur donor ligands based on 2-mercaptobenzimidazole and phosphine co-ligands for the efficient synthesis of 1,2,3-triazole compound *via* a three-component coupling reaction of various alkyl halides/epoxides with alkynes in the presence of sodium azide. It is notable that in these reactions, no reducing agents or bases were utilized. Besides, some non-activated terminal alkynes were successfully employed in coupling reactions.

2. Experimental section

2.1. Instrumentation, analyses, and starting material

NMR spectra were recorded on a Brucker Avance DPX-250 (^{1}H NMR 250 MHz and ^{13}C NMR 62.9 MHz) and DPX-400 (^{1}H NMR 400 MHz and ^{13}C NMR 100 MHz) spectrometer in pure deuterated chloroform ($CDCl_3$) and deuterated dimethylsulfoxide (DMSO-d_6) solutions with tetramethylsilane (TMS) as an internal standard. Chemical shifts were reported in ppm (δ) and coupling constants in Hz (J). Fourier-transform infrared (FT-IR) spectroscopy with a Shimadzu FTIR-8300 spectrophotometer were employed for characterization of the catalysts and products. Melting points were determined in open capillary tubes using a Barnstead electro thermal 9100 BZ circulating oil melting point apparatus. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica gel PolyGram SILG/UV 254 plate. Column Chromatography was carried out on columns of silica gel 60 (70–230 mesh) in glass columns using 15–30 g of silica gel per one-gram crude mixture.

2.2. General procedure for the "Click" synthesis of 1,4-Disubstitude 1,2,3-Triazoles

A mixture of organic halide or epoxide (1.0 mmol), sodium azide (1.2 mmol), and alkyne (1.0 mmol) was stirred in water (3.0 mL) as solvent. Then, Cu(Bb)(PCy_3) (5 mg) was added to the reaction mixture and it was further stirred at 60 °C for a period of time. After completion of the reaction (monitored by TLC), the reaction mixture was extracted by ethyl acetate (5 mL, 3 times) and dried over anhydrous Na_2SO_4 and the procedure was followed by concentration under reduced pressure. The crude products were purified by column chromatography on silica gel (silica gel, *n*-hexane-EtOAc) to give the pure products.

2.3. General procedure for the synthesis of 1, 2, 3 and 4

The synthesis of sodium dihydrobis(2-mercapto-benzimidazolyl) borate (NaBb), and the catalysts tested in this study were carried out by following the procedure described in the literature [41]. In summary, NaBb (0.050 g, 0.148 mmol) and 1 equivalent of an appropriate phosphine were dissolved in methanol (5.0 mL) and to this solution was added CuI (0.028 mg, 0.148 mmol). The solution turned cloudy with a precipitate forming, and the mixture was stirred for 12 h. The precipitate was filtered and it was washed with 5.0 mL cold methanol. The product was dried under vacuum, yielding **1, 2, 3** as white and **4** as yellow solid. Yields: (**1**) 78%, (**2**) 55%, (**3**) 55%, (**4**) 53%.

2.4. 1-Benzyl-4-phenyl-1H-1,2,3-triazole (7a)

White solid, mp 126–128 °C (lit. [60] mp 128–131 °C); FT-IR (KBr cm^{-1}): 3093, 2901, 1612, 1458, 1357, 1072, 763, 478; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 5.59 (s, 2H), 7.32–7.36 (m, 3H), 7.40–7.44 (m, 5H), 7.71 (s, 1H), 7.83 (d, J = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 54.2, 119.6, 125.7, 128.1, 128.2, 128.8, 128.9, 129.2, 130.6, 134.7, 148.2.

2.5. 1-(4-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole (7b)

White solid, mp 128–130 °C (lit. [61] mp 129–131 °C); FT-IR (KBr cm^{-1}): 3085, 2923, 2854, 1604, 1510, 1458.1, 1350, 1226, 1072, 771, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 5.52 (s, 2H), 7.06 (t, J = 8.6 Hz, 2H), 7.28–7.35 (m, 3H), 7.41 (t, J = 7.5 Hz, 2H), 7.73 (s, 1H), 7.82 (d, J = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 53.4, 116.11 (d, J = 21.2 Hz), 119.6, 125.7, 128.3, 128.9, 129.96 (d, J = 9.1 Hz), 130.5, 130.65 (d, J = 3.0 Hz), 148.3, 162.83 (d, J = 248.5 Hz).

2.6. 4-((4-Phenyl-1H-1,2,3-triazol-1-yl)methyl)benzonitrile (7c)

White solid, mp 132–135 °C (lit. [62] mp 132–134 °C); FT-IR (KBr cm^{-1}): 3085, 2923, 2854, 2229, 1612, 1465, 1434, 1342, 1218, 1188, 1080, 1041, 771, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 5.64 (s, 2H), 7.33–7.44 (m, 5H), 7.64 (d, J = 8.1 Hz, 2H), 7.80 (s, 2H), 7.82 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 53.4, 112.6, 118.3, 120.0, 125.7, 128.4, 128.5, 128.9, 130.2, 132.9, 140.0, 148.5.

2.7. 1-(3-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole (7d)

White solid, mp 103–105 °C (lit. [63] mp 102–105 °C); FT-IR (KBr cm^{-1}): 3085, 2923, 1589, 1481, 1450, 1342, 1249, 1195, 1141, 1080, 972, 748, 694, 516; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 5.56 (s, 2H), 6.96–7.09 (m, 3H), 7.30–7.45 (m, 4H), 7.71 (s, 1H), 7.79–7.83 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 53.6, 115.00 (d, J = 22.2 Hz), 115.81 (d, J = 21.2 Hz), 119.6, 123.52 (d, J = 3.0 Hz), 125.7, 128.3, 128.8, 130.3, 130.82 (d, J = 8.1 Hz), 137.04 (d, J = 8.1 Hz), 148.4, 163.02 (d, J = 248.5 Hz).

2.8. 1-Allyl-4-phenyl-1H-1,2,3-triazole (7e)

White solid, mp 74–76 °C (lit. [64] mp 76–77 °C); FT-IR (KBr cm^{-1}): 3121, 3095, 2943, 2428, 1855, 1611, 1464, 1445, 1354, 1334, 1223, 1204, 1169, 1076, 1050, 991, 976, 943, 827, 690, 511; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 5.02 (dt, J = 6.1, 1.4 Hz, 2H), 5.30–5.40 (m, 2H), 5.98–6.14 (m, 1H), 7.29–7.45 (m, 3H), 7.76 (s, 1H), 7.81–7.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 52.3, 119.4, 120.3, 125.5, 128.0, 128.8, 130.7, 131.5, 147.5.

2.9. 1-Butyl-4-phenyl-1H-1,2,3-triazole (7f)

White solid, mp 42–45 °C (lit. [61] mp 42–44 °C); FT-IR (KBr cm^{-1}): 3085, 2960, 2869, 1651, 1465, 1380, 1080, 763, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.01 (t, J = 7.4 Hz, 3H), 1.43 (sext, J = 7.4 Hz, 2H), 1.97 (quin, J = 7.3 Hz, 2H), 4.44 (t, J = 7.2 Hz, 2H), 7.36 (t, J = 7.3 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 7.78 (s, 1H), 7.87 (d, J = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 13.5, 19.7, 32.3, 50.2, 119.4, 125.7, 128.1, 128.8, 130.7, 147.7.

2.10. 1-Octyl-4-phenyl-1H-1,2,3-triazole (7g)

White solid, mp 76–79 °C (lit. [64] mp 78–79 °C); FT-IR (KBr cm^{-1}): 3121, 3094, 2955, 2919, 2849, 1599, 1460, 1217, 1191, 1079, 839, 762, 695; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 0.91 (t, J = 6.7 Hz, 3H), 1.30–1.38 (m, 10H), 1.98 (quin, J = 7.2 Hz, 2H), 4.43 (t, J = 7.2 Hz, 2H), 7.36 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 7.78 (s, 1H), 7.87 (d, J = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 14.1, 22.6, 26.5, 29.0, 29.1, 30.4, 31.7, 50.5, 119.4, 125.7, 128.1, 128.8, 130.8, 147.7.

2.11. 1-Isopropyl-4-phenyl-1H-1,2,3-triazole (7i)

Yellow solid, mp 42–45 °C (lit. [65] mp 43.5 °C); FT-IR (KBr cm^{-1}): 3124, 2939, 1612, 1458, 1365, 1226, 1188, 1080, 1033, 972, 763, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.62 (d, J = 6.8 Hz, 6H), 4.87 (sep, J = 6.7 Hz, 1H), 7.33 (t, J = 7.4 Hz, 1H), 7.42 (t, J = 7.4 Hz, 2H), 7.82 (s, 1H), 7.86 (d, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 23.0, 53.1, 117.3, 125.7, 128.1, 128.8, 130.8, 147.5.

2.12. 1-(sec-Butyl)-4-phenyl-1H-1,2,3-triazole (7j)

Yellow solid, mp 55–57 °C (lit. [66] mp 54–55 °C); FT-IR (KBr cm^{-1}): 3078, 2970, 2877, 1643, 1458, 1365, 1226, 1080, 1049, 763, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 0.92 (t, J = 7.4 Hz, 3H), 1.63 (d, J = 6.8 Hz, 3H), 1.90–2.05 (m, 2H), 4.67 (sext, J = 6.7 Hz, 1H), 7.35 (t, J = 7.3 Hz, 1H), 7.45 (t, J = 7.5 Hz, 2H), 7.78 (s, 1H), 7.88 (d, J = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 10.5, 21.0, 30.4, 59.0, 117.4, 125.7, 128.0, 128.8, 130.9, 147.5.

2.13. 1-Cyclopentyl-4-phenyl-1H-1,2,3-triazole (7k)

Light yellow solid, mp 69–71 °C (lit. [66] mp 68–69 °C); FT-IR (KBr cm^{-1}): 3116, 2962, 2862, 1651, 1458, 1380, 1080, 972, 879, 763, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.75–1.85 (m, 2H), 1.90–2.00 (m, 2H), 2.08–2.16 (m, 2H), 2.27–2.35 (m, 2H), 4.96–5.03 (m, 1H), 7.33–7.37 (m, 1H), 7.44 (t, J = 7.6 Hz, 2H), 7.80 (s, 1H), 7.86 (d, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 24.1, 33.4, 62.0, 118.2, 125.7, 128.1, 128.9, 130.7, 147.6.

2.14. 1-Cyclohexyl-4-phenyl-1H-1,2,3-triazole (7l)

White solid, mp 107–110 °C (lit. [67] mp 107–108 °C); FT-IR (KBr cm^{-1}): 3124, 2931, 2854, 1651, 1458, 1373, 1218, 1080, 1002, 763, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.29–1.40 (m, 2H), 1.46–1.58 (m, 2H), 1.81–1.85 (m, 2H), 1.97–2.00 (m, 2H), 2.28–2.31 (m, 2H), 4.50–4.57 (m, 1H), 7.35 (t, J = 7.4 Hz, 1H), 7.45 (t, J = 7.6 Hz, 2H), 7.80 (s, 1H), 7.87 (d, J = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 25.2, 25.2, 33.6, 60.2, 117.3, 125.7, 128.0, 128.8, 130.9, 147.3.

2.15. 1-Benzyl-4-((naphthalen-1-yloxy)methyl)-1H-1,2,3-triazole (7m)

Light brown solid, mp 127–130 °C (lit. [68] mp 128–129 °C); FT-IR (KBr cm^{-1}): 3082, 2926, 2874, 2516, 2110, 1578, 1459, 1378, 1329, 1307, 1268, 1240, 1213, 1125, 1098, 1070, 1054, 877, 791, 769, 723, 703; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 5.20 (s, 2H), 5.34 (s, 2H), 6.79 (d, J = 7.5 Hz, 1H), 7.08–7.12 (m, 2H), 7.18–7.26 (m, 4H), 7.29–7.37 (m, 3H), 7.43 (s, 1H), 7.63–7.66 (m, 1H), 8.07–8.11 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 54.3, 62.5, 105.5, 121.0, 121.7, 122.1, 122.8, 125.4, 125.7, 126.0, 126.6, 127.6, 128.1, 128.9, 129.2, 134.6, 144.8, 154.1.

2.16. 1-(4-Fluorobenzyl)-4-((naphthalen-1-yloxy)methyl)-1H-1,2,3-triazole (7n)

White solid, mp 99–101 °C; FT-IR (KBr cm^{-1}): 3129, 3091, 3057, 2947, 1581, 1509, 1390, 1265, 1227, 1157, 1128, 1094, 1059, 1017, 840, 810, 787, 765, 530; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 5.40 (s, 2H), 5.50 (s, 2H), 6.98 (d, J = 7.5 Hz, 1H), 7.05–7.11 (m, 2H), 7.25–7.29 (m, 2H), 7.40 (t, J = 7.9 Hz, 1H), 7.45–7.53 (m, 3H), 7.61 (s, 1H), 7.82–7.84 (m, 1H), 8.24–8.26 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 53.5, 62.5, 105.5, 116.16 (d, J = 22.2 Hz), 120.9, 122.0, 122.5, 125.3, 125.6, 125.8, 126.5, 127.5, 129.97 (d, J = 8.1 Hz), 130.44 (d, J = 3.0 Hz), 134.6, 144.9, 153.9, 162.88 (d, J = 248.5 Hz).

2.17. 4-((4-((Naphthalen-1-yloxy)methyl)-1H-1,2,3-triazol-1-yl)methyl) benzonitrile (7o)

Yellow solid, mp 99–100 °C; FT-IR (KBr cm^{-1}): 3152, 2924, 2854, 2229, 2106, 1574, 1504, 1458, 1388, 1265, 1226, 1095, 1056, 987, 760, 547; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 5.34 (s, 2H), 5.52 (s, 2H),

6.87 (d, J = 7.5 Hz, 1H), 7.17–7.19 (m, 1H), 7.23–7.29 (m, 2H), 7.33–7.44 (m, 3H), 7.55–7.60 (m, 3H), 7.72 (d, J = 7.8 Hz, 1H), 8.12 (d, J = 8.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 53.3, 62.3, 105.5, 112.5, 118.3, 121.0, 121.9, 123.2, 125.4, 125.9, 126.6, 127.6, 128.4, 132.6, 132.8, 134.5, 139.8, 145.0, 153.8.

2.18. 1-(3-Fluorobenzyl)-4-((naphthalen-1-yloxy)methyl)-1H-1,2,3-triazole (7p):

White solid, mp 83–85 °C; FT-IR (KBr cm^{-1}): 3055, 2900, 2854, 1612, 1573, 1458, 1396, 1242, 1103, 1041, 802, 779; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 5.31 (s, 2H), 5.43 (s, 2H), 6.84–6.90 (m, 2H), 6.93–7.00 (m, 2H), 7.16–7.25 (m, 1H), 7.27–7.31 (m, 1H), 7.34–7.43 (m, 3H), 7.53 (s, 1H), 7.69–7.72 (m, 1H), 8.11–8.15 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 53.5, 62.4, 105.4, 115.00 (d, J = 22.2 Hz), 115.82 (d, J = 21.2 Hz), 120.9, 122.0, 122.7, 123.54 (d, J = 3.0 Hz), 125.3, 125.6, 125.8, 126.5, 127.5, 130.82 (d, J = 8.1 Hz), 134.5, 136.94 (d, J = 8.1 Hz), 145.0, 153.9, 163.00 (d, J = 248.5 Hz).

2.19. 1-Allyl-4-((naphthalen-1-yloxy)methyl)-1H-1,2,3-triazole (7q)

Yellow solid, mp 78–80 °C; FT-IR (KBr cm^{-1}): 3136, 3056, 2932, 1584, 1591, 1460, 1393, 1268, 1239, 1178, 1156, 1097, 1068, 1050, 1019, 987, 793, 772, 572; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 5.01 (d, J = 5.9 Hz, 2H), 5.31–5.40 (m, 2H), 5.44 (s, 2H), 6.00–6.10 (m, 1H), 6.99 (d, J = 7.6 Hz, 1H), 7.41 (t, J = 7.9 Hz, 1H), 7.48–7.52 (m, 3H), 7.69 (s, 1H), 7.83 (d, J = 7.9 Hz, 1H), 8.29 (d, J = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 52.8, 62.4, 105.4, 120.4, 120.9, 122.0, 122.5, 125.3, 125.6, 125.9, 126.5, 127.5, 131.1, 134.6, 144.6, 154.0.

2.20. 1-Benzyl-4-((4-benzylphenoxy)methyl)-1H-1,2,3-triazole (7r)

White solid, mp 101–102 °C; FT-IR (KBr cm^{-1}): 3741, 3031, 2877, 1612, 1512, 1458, 1380, 1242, 1180, 1110, 1002, 833, 725; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.92 (s, 2H), 5.16 (s, 2H), 5.52 (s, 2H), 6.85–6.91 (m, 2H), 7.08–7.25 (m, 6H), 7.27–7.40 (m, 6H), 7.51 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 54.3, 62.2, 114.8, 122.6, 126.1, 128.2, 128.5, 128.9, 129.2, 129.9, 133.9, 134.5, 141.5, 144.8, 156.6.

2.21. 4-((4-Benzylphenoxy)methyl)-1-(4-fluorobenzyl)-1H-1,2,3-triazole (7s)

White solid, mp 107–108 °C; FT-IR (KBr cm^{-1}): 3085, 2908, 1604, 1512, 1450, 1249, 1172, 1110, 1049, 840, 732, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 3.96 (s, 2H), 5.20 (s, 2H), 5.52 (s, 2H), 6.93 (d, J = 8.4 Hz, 2H), 7.07–7.15 (m, 4H), 7.20–7.25 (m, 3H), 7.28–7.34 (m, 4H), 7.55 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 53.5, 62.2, 114.8, 116.17 (d, J = 21.2 Hz), 122.5, 126.1, 128.5, 128.8, 129.97 (d, J = 2.0 Hz), 130.1, 130.41 (d, J = 3.0 Hz), 134.0, 141.4, 144.9, 156.6, 162.90 (d, J = 249.5 Hz).

2.22. 4-((4-((4-Benzylphenoxy)methyl)-1H-1,2,3-triazol-1-yl)methyl) benzonitrile (7t)

Gray solid, mp 99–101 °C; FT-IR (KBr cm^{-1}): 3093, 2908, 2229, 1612, 1512, 1450, 1242, 1180, 1110, 1049, 840, 810, 732, 694; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 3.95 (s, 2H), 5.20 (s, 2H), 5.59 (s, 2H), 6.92 (d, J = 8.5 Hz, 2H), 7.14 (d, J = 8.4 Hz, 2H), 7.20–7.25 (m, 3H), 7.30–7.35 (m, 4H), 7.64 (d, J = 4.0 Hz, 2H), 7.66 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 53.4, 62.1, 112.7, 114.8, 118.2, 123.0, 126.1, 128.4, 128.5, 128.9, 130.0, 132.9, 134.1, 139.8, 141.4, 145.2, 156.6.

2.23. 4-((4-Benzylphenoxy)methyl)-1-(3-fluorobenzyl)-1H-1,2,3-triazole (7u)

Yellow Solid, mp 57–59 °C; FT-IR (KBr cm^{-1}): 3741, 3070, 3024, 2916, 1697, 1589, 1512, 1450, 1243, 1172, 1049, 794, 694, 509; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.82 (s, 2H), 5.07 (s, 2H), 5.40 (s, 2H), 6.64–6.67 (m, 1H), 6.77–6.95 (m, 5H), 6.97–7.06 (m, 4H), 7.15–7.22 (m, 3H), 7.45 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 53.3, 62.0, 114.9, 114.94 (d, J = 46.5 Hz), 115.68 (d, J = 23.2 Hz), 123.4, 123.72 (d, J = 3.0 Hz), 126.2, 128.6, 129.0, 130.0, 130.66 (d, J = 9.1 Hz), 130.85 (d, J = 8.1 Hz), 134.1, 141.6, 144.7, 156.8, 162.96 (d, J = 248.5 Hz).

2.24. 1-Allyl-4-((4-benzylphenoxy)methyl)-1H-1,2,3-triazole (7v):

White solid, mp 59–61 °C; FT-IR (KBr cm^{-1}): 3128, 3082, 3025, 2919, 2870, 1607, 1508, 1493, 1380, 1291, 1243, 1181, 1107, 1057, 1019, 987, 937, 834, 793, 726, 695, 598; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.93 (s, 2H), 4.94–4.98 (m, 2H), 5.17–5.19 (m, 2H), 5.24–5.38 (m, 2H), 5.93–6.10 (m, 1H), 6.89–6.93 (m, 2H), 7.08–7.29 (m, 7H), 7.58–7.60 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 52.8, 62.2, 114.8, 120.3, 122.6, 126.1, 128.5, 128.9, 130.0, 131.2, 134.0, 141.5, 144.6, 156.7.

2.25. 4-((4-Benzylphenoxy)methyl)-1-butyl-1H-1,2,3-triazole (7w)

Yellow solid, mp 51–53 °C; FT-IR (KBr cm^{-1}): 3133, 3084, 3028, 1605, 1508, 1466, 1383, 1298, 1243, 1177, 1110, 1037, 1011, 844, 796, 696, 594; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 0.98 (t, J = 7.3 Hz, 3H), 1.31–1.38 (m, 2H), 1.83–1.90 (m, 2H), 3.95 (s, 2H), 4.31 (t, J = 7.0 Hz, 2H), 5.19 (s, 2H), 6.97 (d, J = 8.2 Hz, 2H), 7.15 (d, J = 8.2 Hz, 2H), 7.21–7.23 (m, 3H), 7.29–7.34 (m, 2H), 7.61 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 13.5, 19.7, 32.2, 41.1, 50.1, 62.1, 114.9, 122.8, 126.1, 128.5, 128.9, 130.0, 133.9, 141.5, 144.1, 156.8.

2.26. 4-((4-Benzylphenoxy)methyl)-1-cyclopentyl-1H-1,2,3-triazole (7x)

White solid, mp 73–75 °C; FT-IR (KBr cm^{-1}): 3071, 3025, 2966, 2869, 1601, 1507, 1313, 1248, 1177, 1106, 1045, 839, 792, 729, 695, 598; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.76–1.98 (m, 4H), 2.04–2.23 (m, 4H), 3.97 (s, 2H), 4.95 (quin, J = 8.2, 7.6 Hz, 1H), 5.22 (s, 2H), 6.98 (d, J = 8.5 Hz, 2H), 7.16 (d, J = 8.4 Hz, 2H), 7.22–7.26 (m, 3H), 7.33 (t, J = 7.3 Hz, 2H), 7.65 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 24.1, 33.4, 41.1, 62.0, 62.3, 114.8, 121.3, 126.1, 128.5, 128.9, 130.0, 133.9, 141.5, 144.0, 156.8.

2.27. 2-Phenyl-2-(4-phenyl-1H-1,2,3-triazol-1-yl)ethan-1-ol (9a)

White solid, mp 126–128 °C (lit. [69] mp 125–127 °C); FT-IR (KBr cm^{-1}): 3402, 3085, 2931, 1959, 1890, 1604, 1458, 1380, 1226, 1180, 1072, 910, 833, 763, 694, 555; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 4.00–4.10 (m, 1H), 4.24–4.27 (m, 1H), 4.63–4.68 (m, 1H), 5.72 (dd, J = 8.2, 3.6 Hz, 1H), 7.32–7.35 (m, 2H), 7.39–7.42 (m, 6H), 7.73–7.75 (m, 2H), 7.77 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 65.0, 67.4, 120.6, 125.7, 127.2, 128.3, 128.8, 129.0, 129.1, 130.2, 136.1, 147.6.

2.28. 2-(4-Phenyl-1H-1,2,3-triazol-1-yl)cyclopentan-1-ol (9b)

White solid, mp 162–164 °C (lit. [70] mp 165–167 °C); FT-IR (KBr cm^{-1}): 3841, 3134, 2914, 2876, 1609, 1460, 1429, 1375, 1315, 1247, 1221, 1080, 1071, 1041, 828, 769, 697, 512; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.73–1.82 (m, 1H), 1.84–1.91 (m, 2H), 2.10–2.20 (m, 2H), 2.27–2.35 (m, 1H), 4.53–4.59 (m, 2H), 5.18 (s, 1H), 7.27–7.36 (m, 3H), 7.61 (d, J = 7.0 Hz, 2H), 7.68 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 20.2, 29.5, 31.8, 68.8, 77.6, 119.6, 125.5, 128.1, 128.8, 130.2, 146.9.

2.29. 2-(4-Phenyl-1H-1,2,3-triazol-1-yl)cyclohexan-1-ol (9c)

White solid, mp 177–180 °C (lit. [71] mp 177–179 °C); FT-IR (KBr cm^{-1}): 3309, 3116, 2939, 2862, 1612, 1558, 1442, 1365, 1234, 1080, 848, 771, 702, 640, 516; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.43–1.58 (m, 3H), 1.89–1.94 (m, 2H), 2.02–2.06 (m, 1H), 2.21–2.28 (m, 2H), 4.02 (brs, 1H), 4.11–4.21 (m, 2H), 7.31–7.34 (m, 1H), 7.36–7.40 (m, 2H), 7.67–7.70 (m, 2H), 7.77 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 24.1, 24.8, 31.6, 33.8, 67.3, 72.6, 119.8, 125.5, 128.0, 128.8, 130.3, 146.8.

2.30. 1-(4-Phenyl-1H-1,2,3-triazol-1-yl)propan-2-ol (9d)

White solid, mp 117–120 °C (lit. [72] mp 118–120 °C); FT-IR (KBr cm^{-1}): 3255, 3139, 2962, 2923, 1612, 1465, 1434, 1365, 1226, 1141, 1087, 979, 817, 760, 694, 509; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 1.32 (d, J = 6.2 Hz, 3H), 3.21–3.25 (m, 1H), 4.22 (dd, J = 13.3, 7.8 Hz, 1H), 4.31–4.42 (m, 1H), 4.47 (dd, J = 13.3, 2.6 Hz, 1H), 7.32–7.42 (m, 3H), 7.70–7.74 (m, 2H), 7.81 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 20.5, 57.6, 66.4, 121.2, 125.4, 128.1, 128.8, 130.1, 147.1.

2.31. 1-(4-Phenyl-1H-1,2,3-triazol-1-yl)butan-2-ol (9e)

White solid, mp 145–147 °C (lit. [73] mp 148–150 °C); FT-IR (KBr cm^{-1}): 3247, 3139, 2962, 2931, 1651, 1612, 1458, 1373, 1226, 1134, 1095, 979, 918, 825, 763, 694, 516; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 1.06 (t, J = 7.4 Hz, 3H), 1.55–1.67 (m, 2H), 3.15 (d, J = 4.6 Hz, 1H), 4.05–4.11 (m, 1H), 4.20–4.29 (m, 1H), 4.50 (dd, J = 13.8, 2.7 Hz, 1H), 7.30–7.41 (m, 3H), 7.70–7.74 (m, 2H), 7.83 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 9.8, 27.5, 56.1, 71.7, 121.3, 125.6, 128.1, 128.8, 130.1.

2.32. 1-(4-Phenyl-1H-1,2,3-triazol-1-yl)decan-2-ol (9f)

White solid, mp 99–101 °C; IR (KBr cm^{-1}): 3402, 3245, 3144, 2928, 2854, 1674, 1457, 1231, 1136, 1105, 1090, 823, 765, 695, 512; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.81 (t, J = 6.4 Hz, 3H), 1.17–1.45 (m, 14H), 3.22 (brs, 1H), 4.02–4.20 (m, 2H), 4.42 (dd, J = 13.3, 2.1 Hz, 1H), 7.19–7.34 (m, 3H), 7.61–7.68 (m, 2H), 7.75 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 14.2, 22.7, 25.6, 29.3, 29.6, 29.6, 31.9, 34.5, 56.5, 70.3, 121.4, 125.4, 128.0, 128.8, 130.2, 147.0.

2.33. 1-(4-Phenyl-1H-1,2,3-triazol-1-yl)hex-5-en-2-ol (9g)

White solid, mp 104–106 °C (lit. [74] mp 104.9–107.9 °C); FT-IR (KBr cm^{-1}): 3393, 3233, 3141, 2934, 2920, 1636, 1441, 1228, 1206, 1160, 1110, 1091, 1023, 910, 826, 767, 696, 513; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.63–1.68 (m, 2H), 2.20–2.39 (m, 2H), 4.17–4.20 (m, 1H), 4.22–4.24 (m, 1H), 4.29 (brs, 1H), 4.45–4.48 (m, 1H), 5.01–5.04 (m, 1H), 5.07–5.12 (m, 1H), 5.81–5.91 (m, 1H), 7.29–7.38 (m, 3H), 7.63–7.65 (m, 2H), 7.80 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 29.7, 33.5, 56.4, 69.7, 115.4, 121.3, 125.5, 128.1, 128.8, 130.2, 137.8, 147.1.

2.34. 1-Butoxy-3-(4-phenyl-1H-1,2,3-triazol-1-yl)propan-2-ol (9h)

White solid, mp 60–62 °C; FT-IR (KBr cm^{-1}): 3255, 3139, 2931, 2869, 1612, 1434, 1373, 1226, 1126, 1087, 979, 871, 825, 763, 694, 516; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 0.92 (t, J = 7.3 Hz, 3H), 1.30–1.44 (m, 2H), 1.51–1.62 (m, 2H), 3.20 (brs, 1H), 3.36–3.54 (m, 4H), 4.19–4.30 (m, 1H), 4.43 (dd, J = 14.0, 6.8 Hz, 1H), 4.59 (dd, J = 14.0, 3.7 Hz, 1H), 7.28–7.35 (m, 1H), 7.37–7.44 (m, 2H), 7.76–7.81 (m, 2H), 7.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 13.9, 19.2, 31.5, 53.5, 68.9, 71.3, 71.9, 121.7, 125.4, 127.9, 128.7, 130.3, 147.1.

2.35. 1-Isopropoxy-3-(4-phenyl-1H-1,2,3-triazol-1-yl)propan-2-ol (9i)

Yellow solid, mp 61–63 °C (lit. [68] mp 62–64 °C); FT-IR (KBr cm^{-1}): 3201, 2970, 2869, 1643, 1558, 1463, 1365, 1334, 1303, 1226, 1072, 979, 925, 825, 763, 702, 516; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 1.17 (d, J = 3.2 Hz, 3H), 1.19 (d, J = 3.2 Hz, 3H), 3.42 (dd, J = 9.6, 5.7 Hz, 1H), 3.52 (dd, J = 9.6, 5.0 Hz, 1H), 3.62 (hep, J = 6.1 Hz, 1H), 3.74 (brs, 1H), 4.21–4.30 (m, 1H), 4.43 (dd, J = 14.0, 7.1 Hz, 1H), 4.60 (dd, J = 14.0, 3.6 Hz, 1H), 7.30–7.36 (m, 1H), 7.40 (t, J = 7.4 Hz, 2H), 7.75–7.78 (m, 2H), 7.90 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 22.0, 53.3, 69.0, 69.4, 72.5, 121.3, 125.6, 128.1, 128.8, 130.5, 147.4.

2.36. 1-Phenoxy-3-(4-phenyl-1H-1,2,3-triazol-1-yl)propan-2-ol (9j)

White solid, mp 125–126 °C (lit. [69], mp 125–127 °C); FT-IR (KBr cm^{-1}): 3417, 3085, 2923, 2877, 1697, 1596, 1488, 1373, 1296, 1242, 1172, 1118, 1080, 1041, 879, 833, 756, 686, 516; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.93–3.96 (m, 2H), 4.26 (brs, 1H), 4.37–4.47 (m, 2H), 4.58–4.65 (m, 1H), 6.81–6.91 (m, 3H), 7.21–7.31 (m, 5H), 7.55–7.60 (m, 2H), 7.76 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 29.7, 53.3, 68.9, 114.6, 121.5, 125.6, 128.2, 128.9, 129.5, 129.7, 130.2, 147.5, 158.2.

2.37. 1-(Allyloxy)-3-(4-phenyl-1H-1,2,3-triazol-1-yl)propan-2-ol (9k)

White solid, mp 71.5–72 °C (lit. [75], mp 71.4 °C); FT-IR (KBr cm^{-1}): 3240, 3139, 2931, 2869, 1651, 1612, 1434, 1357, 1234, 1087, 995, 918, 871, 825, 763, 694, 509; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.28 (d, J = 4.9 Hz, 1H), 3.43 (dd, J = 9.7, 5.9 Hz, 1H), 3.54 (dd, J = 9.7, 4.7 Hz, 1H), 4.03 (dt, J = 5.7, 1.4 Hz, 2H), 4.23–4.33 (m, 1H), 4.44 (dd, J = 14.0, 6.9 Hz, 1H), 4.60 (dd, J = 14.0, 3.6 Hz, 1H), 5.19–5.33 (m, 2H), 5.82–5.98 (m, 1H), 7.29–7.44 (m, 3H), 7.75–7.80 (m, 2H), 7.88 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 53.5, 69.1, 71.3, 72.4, 117.6, 121.5, 125.5, 128.1, 128.8, 130.3, 134.3, 147.2.

2.38. 1,3-Bis(4-phenyl-1H-1,2,3-triazol-1-yl)propan-2-ol (9l)

White solid, mp 231–233 °C (lit. [69] mp 233–233 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 3.58–3.66 (m, 2H), 3.75–3.79 (m, 1H), 4.25 (brs, 1H), 4.45–4.51 (m, 1H), 4.56–4.60 (m, 1H), 7.33–7.38 (m, 2H), 7.42 (t, J = 7.4 Hz, 3H), 7.72–7.98 (m, 5H), 8.02 (s, 2H).

2.39. 2-(4-((Naphthalen-1-yloxy)methyl)-1H-1,2,3-triazol-1-yl)-2-phenylethan-1-ol (9m)

Light yellow solid, mp 103–105 °C; FT-IR (KBr cm^{-1}): 3271, 3063, 2935, 2873, 1580, 1457, 1387, 1333, 1269, 1242, 1131, 1096, 1061, 983, 796, 774, 703, 530; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 4.21–4.24 (m, 1H), 4.32 (brs, 1H), 4.56–4.61 (m, 1H), 5.30 (s, 2H), 5.69–5.71 (m, 1H), 6.93 (d, J = 7.5 Hz, 1H), 7.21–7.30 (m, 2H), 7.35–7.40 (m, 4H), 7.43–7.52 (m, 3H), 7.72 (s, 1H), 7.83 (d, J = 8.0 Hz, 1H), 8.26 (d, J = 8.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 62.3, 64.7, 67.3, 105.5, 121.0, 122.1, 123.8, 125.4, 125.6, 125.9, 126.6, 127.2, 127.6, 128.9, 129.1, 134.6, 136.1, 144.0, 154.0.

2.40. 2-(4-((4-Benzylphenoxy)methyl)-1H-1,2,3-triazol-1-yl)-2-phenylethan-1-ol (9n)

White solid, mp 64–66 °C; FT-IR (KBr cm^{-1}): 3309, 3062, 2916, 1604, 1504, 1380, 1342, 1296, 1242, 1180, 1126, 1049, 910, 840, 725, 694, 594, 532; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.09–3.23 (m, 1H), 3.89–3.92 (m, 2H), 4.11–4.24 (m, 1H), 4.50–4.63 (m, 1H), 5.13–5.15 (m, 2H), 5.57–5.65 (m, 1H), 6.82–6.90 (m, 2H), 7.05–7.26 (m, 9H), 7.32–7.39 (m, 3H), 7.53–7.56 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 62.0, 64.8, 67.3, 114.8, 123.8, 126.1, 127.2, 128.5, 128.9, 129.0, 129.1, 130.0, 134.0, 136.1, 141.5, 144.0, 156.7.

2.41. 1-(4-((4-Benzylphenoxy)methyl)-1H-1,2,3-triazol-1-yl)-3-phenoxypropan-2-ol (9o)

White solid, mp 104–106 °C; FT-IR (KBr cm^{-1}): 3255, 3025, 2919, 1594, 1495, 1292, 1253, 1232, 1182, 1115, 1038, 1015, 853, 813, 755, 693, 600; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.01 (brs, 1H), 3.84 (s, 2H), 3.87–3.99 (m, 2H), 4.35–4.50 (m, 2H), 4.58–4.64 (m, 1H), 5.10 (s, 2H), 6.79–6.84 (m, 4H), 6.88–6.95 (m, 1H), 7.01–7.18 (m, 7H), 7.22–7.25 (m, 2H), 7.67 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 53.2, 61.9, 68.8, 68.8, 114.6, 114.8, 121.6, 124.6, 126.1, 128.5, 128.9, 129.7, 130.0, 134.0, 141.5, 144.1, 156.6, 158.1.

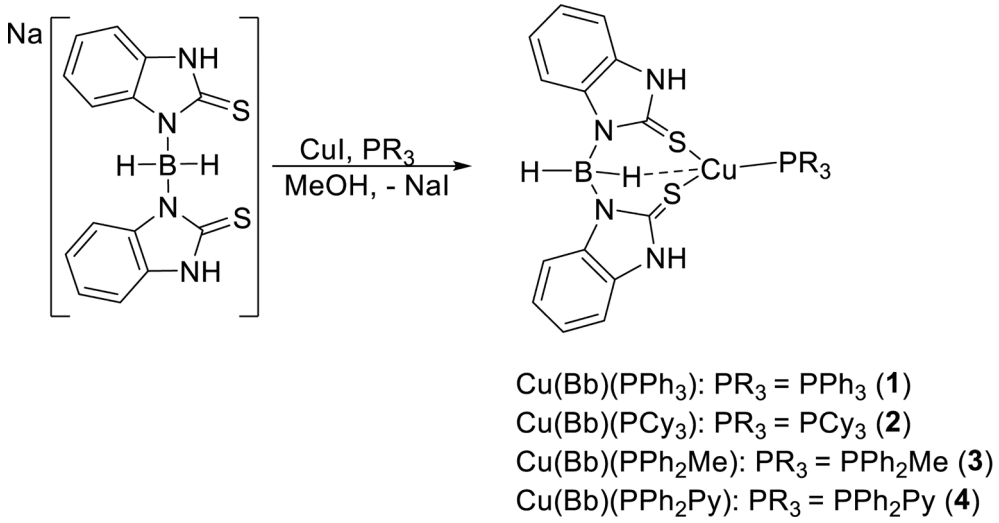
2.42. 1-(Allyloxy)-3-(4-((4-benzylphenoxy)methyl)-1H-1,2,3-triazol-1-yl) propan-2-ol (9p)

Yellow oil; FT-IR (KBr cm^{-1}): 3393, 2903, 2868, 1609, 1508, 1458, 1299, 1241, 1177, 1109, 1060, 1013, 928, 797, 726, 698, 599; ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm) 3.28 (dd, J = 9.8, 5.6 Hz, 1H), 3.35 (dd, J = 9.8, 4.9 Hz, 1H), 3.61 (brs, 1H), 3.80 (s, 2H), 3.87 (dt, J = 5.6, 1.3 Hz, 2H), 4.05–4.15 (m, 1H), 4.26 (dd, J = 14.0, 7.1 Hz, 1H), 4.42 (dd, J = 14.0, 3.7 Hz, 1H), 5.01 (s, 2H), 5.05–5.19 (m, 2H), 5.68–5.84 (m, 1H), 6.75–6.81 (m, 2H), 6.97–7.02 (m, 2H), 7.04–7.11 (m, 3H), 7.14–7.19 (m, 2H), 7.63 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 41.1, 53.4, 61.8, 69.0, 71.3, 72.4, 114.9, 117.6, 124.8, 126.2, 128.6, 128.9, 130.1, 134.0, 134.4, 141.6, 143.7, 156.7.

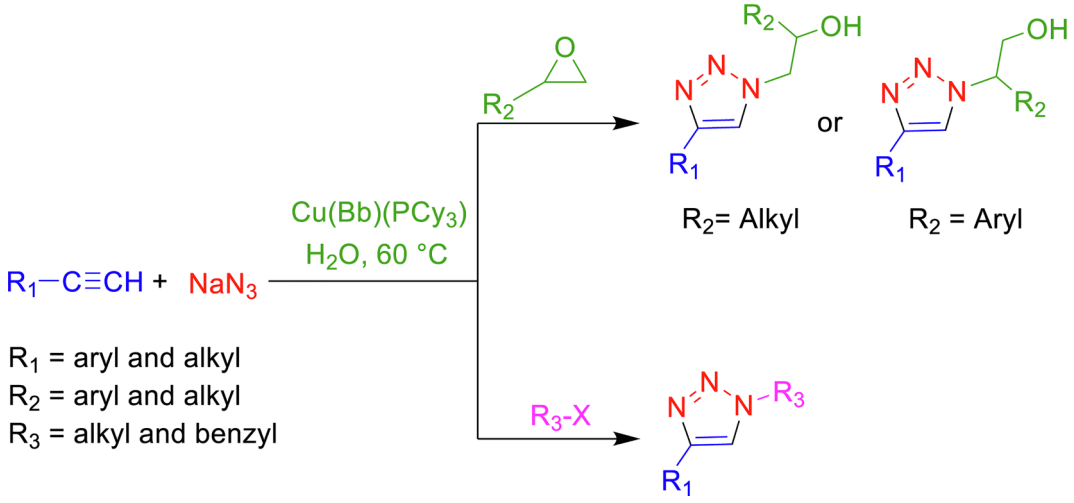
3. Results and discussion

Cu(I) complexes **1**–**4** were synthesized according to our procedure reported previously in the literature [41]. The characterization of copper complexes was carried out by FT-IR, ^{1}H NMR, ^{13}C NMR, ^{31}P NMR and elemental analysis (Scheme 1).

The molecular structure of **1** and **2** were reported previously [41]. In complex **1**, copper atom adopts a trigonal geometry. Out of four Cu(I) complexes, the Cu(Bb)(PCy_3) (**2**) showed promising results in our initial tests and it was selected as catalyst in the "click" reaction (Scheme 2).



Scheme 1. Synthesis of Cu (I) complexes.



Scheme 2. The one-pot three-component route to 1,4-disubstituted 1,2,3-triazoles.

3.1. The catalytic application of Cu(Bb)(PCy3) in azide-alkyne cycloaddition

The "click" reaction was performed to investigate the catalytic activity of Cu (I) complexes as depicted in Scheme 1. For this purpose, the one-pot three-component reaction between phenylacetylene **5a** (1.0 mmol), sodium azide (1.2), and benzyl bromide **6a** (1.0 mmol) was chosen as a model reaction to screen various conditions including solvent, temperature, and catalytic amount of the Cu (I) complexes (Table 1). First, the "click" reaction was performed in the absence of the catalyst in water medium at 60 °C and no triazole product was observed even after 12 h (Table 1, entry 1). In the next step, a series of Cu (I) complexes, including Cu(Bb)(PPh_3) (**1**), Cu(Bb)(PCy_3) (**2**), Cu(Bb)(PPh_2Me) (**3**) and Cu(Bb)(PPh_2Py) (**4**) were tested in this reaction (Table 1, entries 2–5). Cu(Bb)(PCy_3) complex proved to be highly active in terms of yield, selectivity, and time for the synthesis of 1,4-disubstituted 1,2,3-triazole derivatives in water (Table 1, entry 2). Then, various amounts of catalyst loading were studied. As shown in Table 1, the highest reaction yield was achieved by using 5 mg of the catalyst (Table 1, entries 6 and 7). After optimizing the catalyst loading, the effect of various solvents was also investigated for the model reaction (Table 1, entries 8–15). It was observed that water was the most effective solvent to furnish the coupled product **7a**. This could be perhaps due to better solublity of the catalyst in aquous medium *via* establishing hydrogen bonding through N—H moiety of benzimidazole ring. Subsequently, the reaction temperature was also optimized and the best result was obtained at 60 °C (Table 1, entries 2, 16–18). As can be seen from Table 1, maximum yield of triazole product was obtained with 5 mg of the catalyst in water medium at 60 °C.

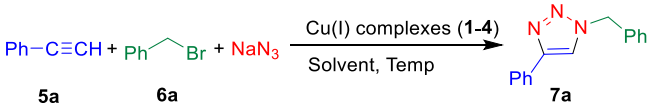
The initial results inspired us to investigate further experiments with a wide range of organic halides or epoxides and terminal alkynes. Under the optimized reaction condition, benzyl bromide **6a**, was converted into the 1-benzyl-4-phenyl-1*H*-1,2,3-triazole **7a** in 97% yield within 40 min. Benzyl chloride with electron-withdrawing groups such as cyano and fluoro were successfully reacted with phenylacetylene, yielding corresponding triazoles **7b-c** in excellent yields (Table 2,entries 2 and 3). Also, meta-substituted benzyl bromide afforded the corresponding product **7d** with excellent yield (entry 4). For further investigation of reaction scope, we found out that primary alkyl halides in reaction with phenylacetylene can be transformed to the corresponding 1,4-disustituted 1,2,3-triazole products **7e-g** in good to excellent yields (entries 5–8). Notably, when secondary aliphatic alkyl halides such as 2-bromopropane, 2-bromobuatne, bromocyclopentane and bromocyclohexane were employed as substrates, moderate yields of triazoles **7i-l** were obtained (entries 9–12). In addition to aromatic alkyne, aliphatic alkynes also worked well and reacted with different benzyl and alkyl halides in the presence of a catalytic amount of Cu(Bb)(PCy_3) to furnish the corresponding 1,4-disustituted 1,2,3-triazole derivatives **7 m-x** (Table 2,entries 13–24). The aliphatic alkynes are known for their low reactivity in the "click" chemistry [76].

It is noteworthy that in this process, alkyl halides play the roles of synthetic equivalent of potentially explosive azides which are known to be difficult to handle and store. Having successfully established the Cu(Bb)(PCy_3) catalyzed CuAAC click transformation for the synthesis of 1,4-disubstituted triazoles, it was suggested that β-hydroxy-1,2,3-triazoles might also be synthesized under the same reaction conditions by replacing alkyl halides with epoxides (Table 3). According to the literature reports, primary β-hydroxy triazole derivatives were formed using styrene oxide derivatives and in these cases, electronic factors predominate over steric factors, while secondary alcohols as another possible regional isomers were formed using aliphatic oxide derivatives (Scheme 3).

In the following step, bicyclic epoxides such as **1,2-**Epoxycyclopentane and 1,2-Epoxycyclohexane gave the corresponding 1,2,3-triazoles **9b-c** in 89% and 92%, respectively (Table 3, entries 2 and 3). Moreover, alkyl substituted epoxides were also applicable to the present transformation, and the corresponding 1,4-disubstituted 1,2,3-triazoles **9d-f** were obtained in excellent yields (Table 3, entries 4–6). The results indicated that the epoxides with ether groups including 2-(butoxymethyl)oxirane, 2-(isopropoxymethyl)oxirane, 2-(phenoxymethyl)oxirane and 2-((allyloxy)methyl)oxirane were tolerated in the reaction and afforded the desired triazole products **9h-k** in high yields. (Table 3, entries 8–11). Surprisingly, aliphatic alkynes appeared as good substrates under these conditions and reacted with epoxides to give corresponding 1,4-disubstituted 1,2,3-triazoles **9m-p** with reasonable yields (Table 3, entries 13–16). Generally, all these "click" reactions proceeded efficiently and the corresponding triazole products were achieved in aqueous medium in good to excellent yields.

Herein, according to the mechanistic studies reported in the literature regarding alternative "click" reactions [29], we proposed likely mechanism for the one-pot synthesis of 1,4-disubstituted 1,2,3-triazole compounds *via* a multicomponent reaction in the presence of Cu(Bb)(PCy_3) complex **2**. In the first step of the catalytic cycle, Cu(I)-acetylide intermediate **A** is created. In the next step, the azide intermediate is coordinated to the copper(I) center to generate intermediate **B**. Finally, the cyclization reaction of azide with activated alkyne along with protonolysis is carried out to provide 1,4-disubstituted 1,2,3-triazole compounds (Scheme 4).

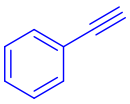
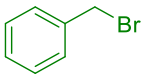
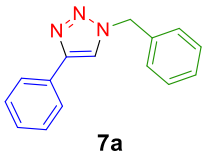
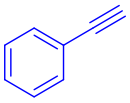
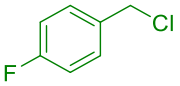
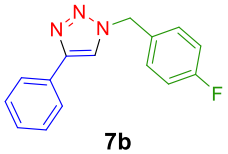
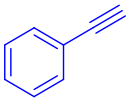
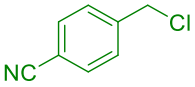
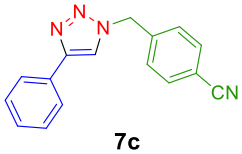
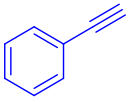
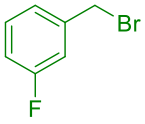
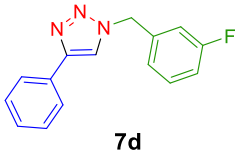
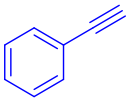
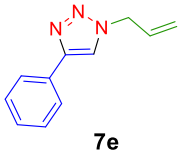
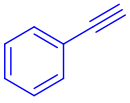
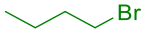
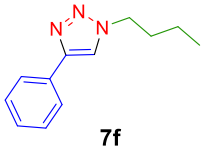
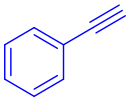
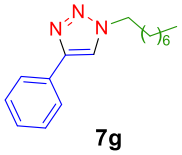
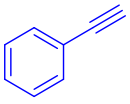
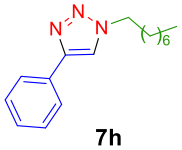
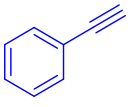
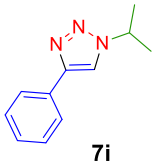
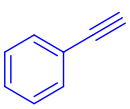
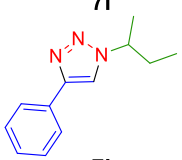
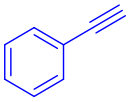
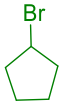
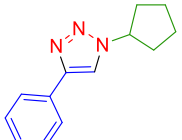
Table 1
Optimization of the reaction conditions for the synthesis of 1-benzyl-4-phenyl-1*H*-1,2,3-triazole.



Entry	Catalyst	mg	Solvent	Temp (°C)	Time (h)	Yield[a] (%)
1	–	–	H_2O	60	12	0
2	**Cu(Bb)(PCy_3)**	**5**	**H_2O**	**60**	**0.7**	**97**[b]
3	Cu(Bb)(PPh_2Py)	5	H_2O	60	3	70
4	Cu(Bb)(PPh_3)	5	H_2O	60	4	63
5	Cu(Bb)(PPh_2Me)	5	H_2O	60	6	58
6	Cu(Bb)(PCy_3)	10	H_2O	60	5	83
7	Cu(Bb)(PCy_3)	3	H_2O	60	5	71
8	Cu(Bb)(PCy_3)	5	CH_3CN	Reflux	5	0
9	Cu(Bb)(PCy_3)	5	DMF	80	2	54
10	Cu(Bb)(PCy_3)	5	THF	Reflux	3	46
11	Cu(Bb)(PCy_3)	5	EtOH	Reflux	5	85
12	Cu(Bb)(PCy_3)	5	CH_2Cl_2	Reflux	5	0
13	Cu(Bb)(PCy_3)	5	Dioxane	Reflux	5	0
14	Cu(Bb)(PCy_3)	5	H_2O/ Dioxane	Reflux	5	60
15	Cu(Bb)(PCy_3)	5	H_2O/ *t*-BuOH	80	5	83
16	Cu(Bb)(PCy_3)	5	H_2O	80	1	93
17	Cu(Bb)(PCy_3)	5	H_2O	100	3	54
18	Cu(Bb)(PCy_3)	5	H_2O	r.t.	3	46

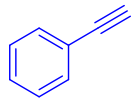
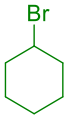
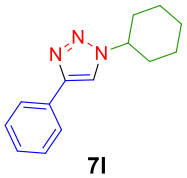
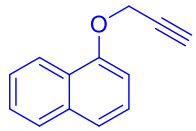
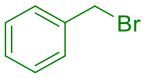
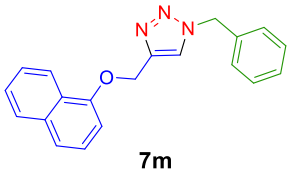
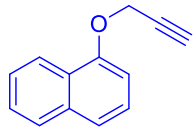
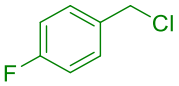
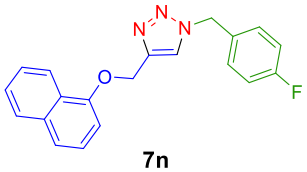
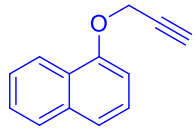
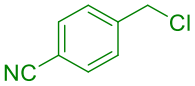
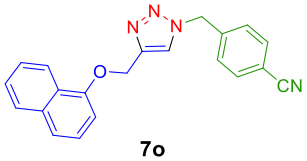
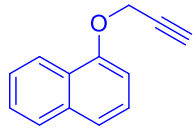
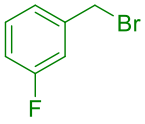
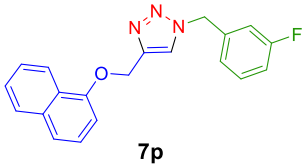
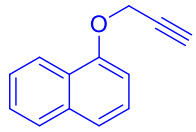
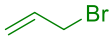
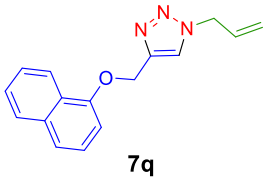
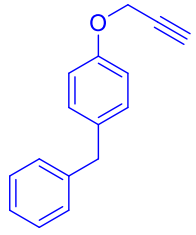
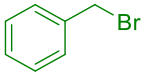
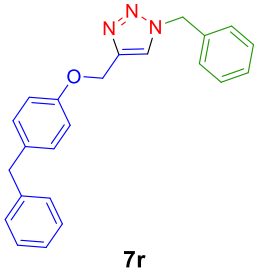
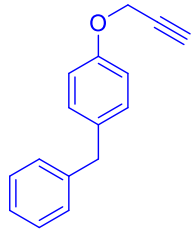
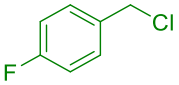
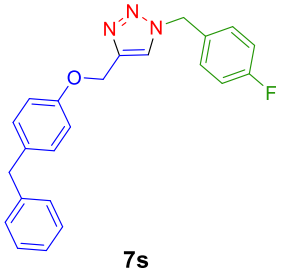
[a] Yield of pure isolated product.
[b] Experimental conditions: phenylacetylene **5a** (1.0 mmol), benzyl bromide **6a** (1.0 mmol), sodium azide (1.2 mmol), Cu (I) complexes as catalyst (type indicated), and H_2O (3 mL).

Table 2
Synthesis of 1,2,3-triazoles by one-pot three-component reaction using homogeneous Cu(Bb)(PCy_3) catalyst at 60 °C.[a]

Entry	Alkyne	Organic halide	Product	Time (min)	Yield (%)[b]
1				40	97
2			**7a** 	50	94
3			**7b** 	50	95
4			**7c** 	55	92
5			**7d** 	45	90
6			**7e** 	60	85
7			**7f** 	120	77
8			**7g** 	60	90
9			**7h** 	70	90
10			**7i** 	75	81
11			**7j**  **7k**	80	80

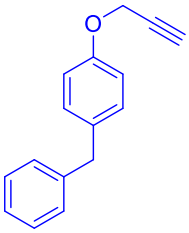
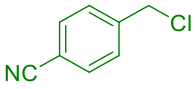
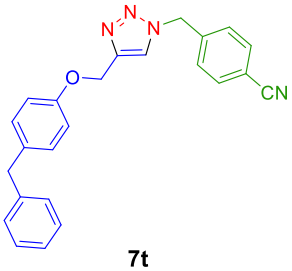
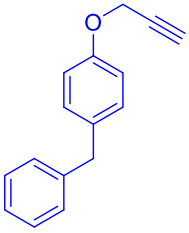
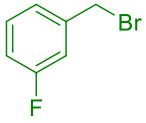
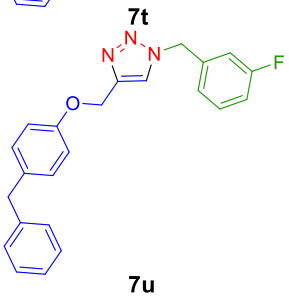
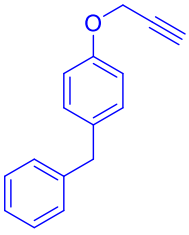
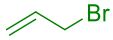
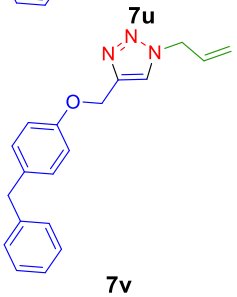
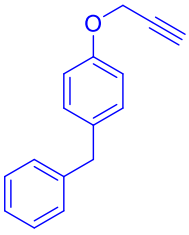
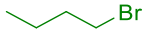
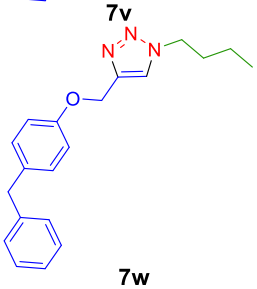
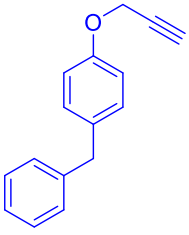
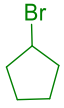
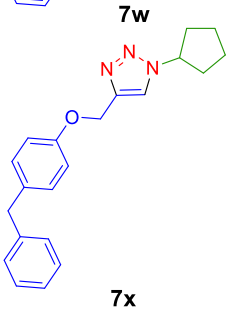
(*continued on next page*)

Table 2 (*continued*)

Entry	Alkyne	Organic halide	Product	Time (min)	Yield (%)[b]
12			 **7l**	90	78
13			 **7m**	45	93
14			 **7n**	55	92
15			 **7o**	70	88
16			 **7p**	80	85
17			 **7q**	50	90
18			 **7r**	45	95
19			 **7s**	50	90

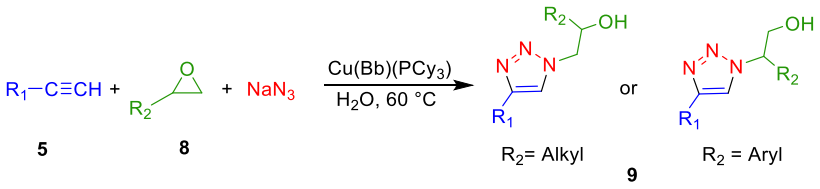
(*continued on next page*)

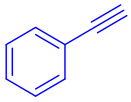
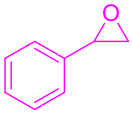
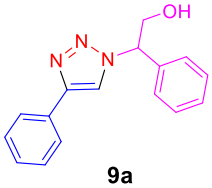
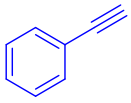
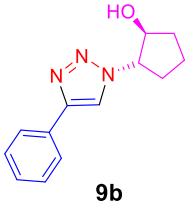
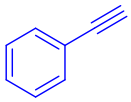
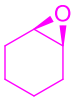
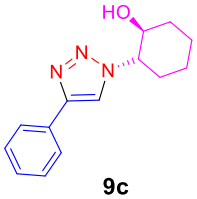
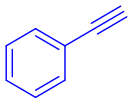
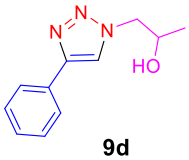
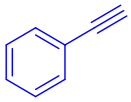
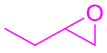
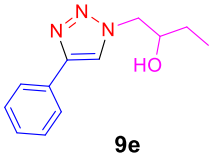
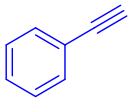
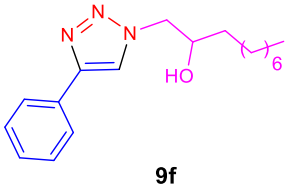
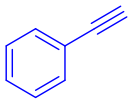
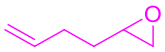
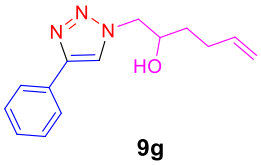
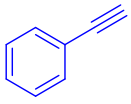
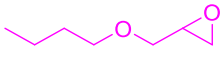
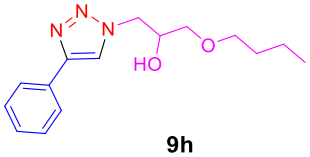
Table 2 (*continued*)

Entry	Alkyne	Organic halide	Product	Time (min)	Yield (%)[b]
20				55	92
21			**7t** 	100	82
22			**7u** 	50	91
23			**7v** 	65	89
24			**7w**  **7x**	90	80

[a] Reaction conditions: alkyne (1.0 mmol), organic halide (1.0 mmol), sodium azide (1.2 mmol), Cu(Bb)(PCy_3) (5 mg) in water (3.0 mL), 60 °C.
[b] Isolated yields.

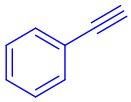
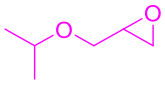
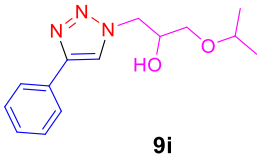
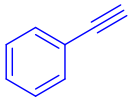
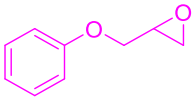
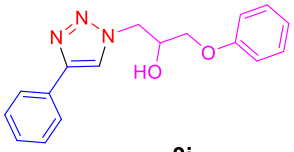
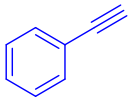
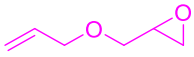
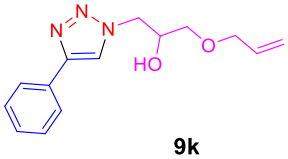
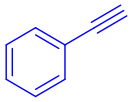
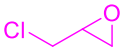
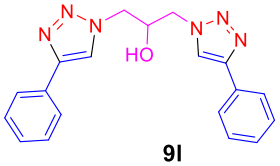
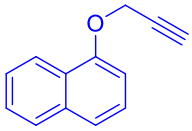
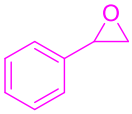
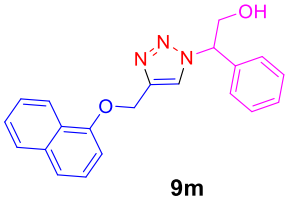
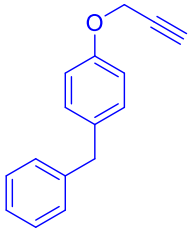
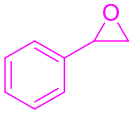
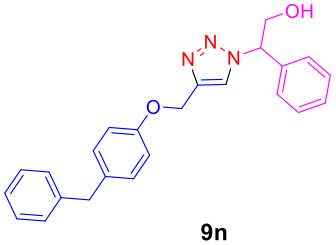
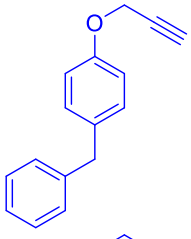
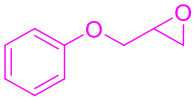
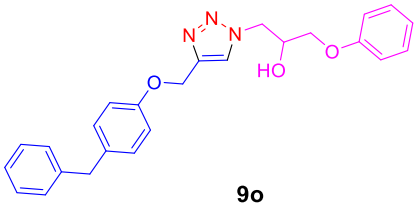
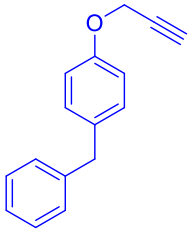
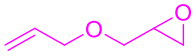
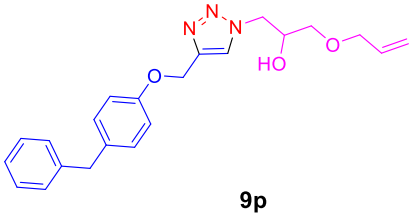
Table 3
Synthesis of 1,2,3-triazoles *via* a one-pot three-component reaction between epoxides, sodium azide and terminal alkynes using Cu(Bb)(PCy_3).[a]



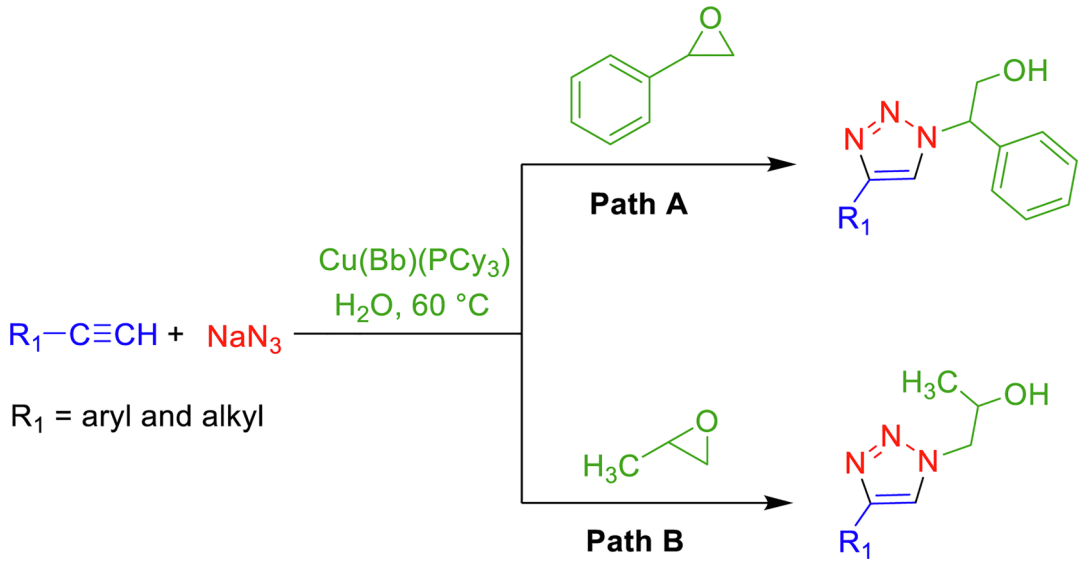
Entry	Alkyne	Epoxide	Product	Time (min)	Yield[b] (%)
1				35	96
2			**9a** 	60	89
3			**9b** 	55	92
4			**9c** 	40	92
5			**9d** 	40	94
6			**9e** 	65	93
7			**9f** 	40	95
8			**9g**  **9h**	50	89

(*continued on next page*)

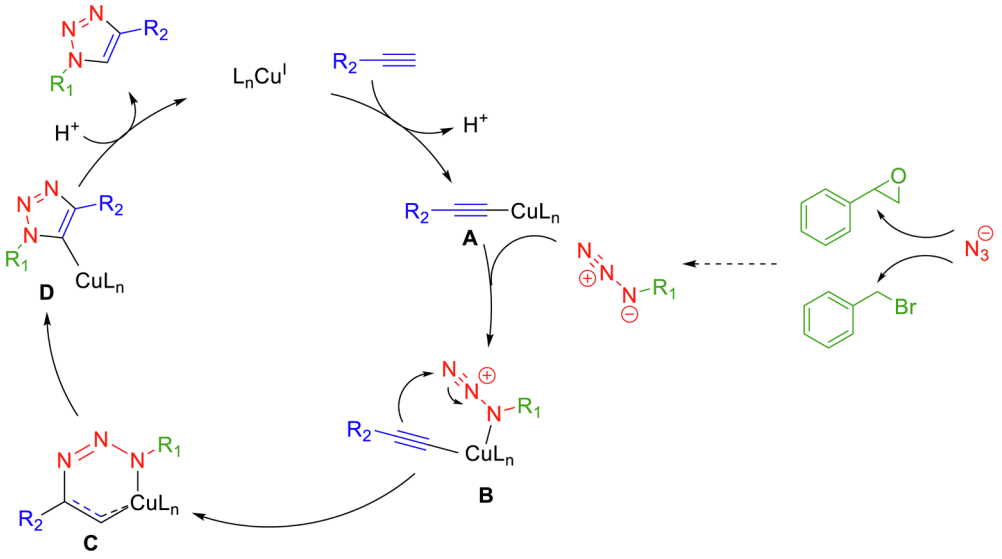
Table 3 (*continued*)

Entry	Alkyne	Epoxide	Product	Time (min)	Yield[b] (%)
9			 **9i**	45	90
10			 **9j**	70	75
11			 **9k**	65	88
12			 **9l**	120	65
13			 **9m**	70	91
14			 **9n**	80	93
15			 **9o**	80	80
16			 **9p**	110	75

[a] Reagents and conditions: alkyne (1.0 mmol), epoxides (1.0 mmol), sodium azide (1.2 mmol), water (3.0 mL), Cu(Bb)(PCy_3) (5 mg). 60 °C.
[b] Isolated yield.



Scheme 3. Synthesis of 1,4-disubstituted 1,2,3-triazoles *via* phenyl acetylene, sodium azide and 2-phenyloxirane (path A) or 2-methyloxirane (path B).



Scheme 4. Proposed mechanism for the synthesis of 1,4-Disubstitude 1,2,3-Triazoles catalysed by Cu(Bb)(PCy_3).

4. Conclusion

In summary, a copper(I) complex containing homoscorpionate bis(2-mercaptobenzimidazolyl) and tricyclohexylphosphine ligands was used as a catalyst for the synthesis of 1,4-disubstitud 1,2,3-triazole derivatives *via* one-pot and three-component reaction of organic halides or epoxides, sodium azide, and terminal alkynes. The major advantages of this protocol include mild and green reaction conditions, short reaction times, clean reaction profile, high diversity, and good to excellent yields.

Acknowledgements

We gratefully appreciate the Shiraz University Research Council for the financial support of this work.

Conflicts of interest

Authors do not have any conflict of interest.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ica.2020.119470.

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