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Copper-catalyzed one-pot synthesis of amide linked 1,2,3-triazoles bearing aryloxy skeletons

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ABSTRACT

In this paper, novel amide linked 1,2,3-triazoles containing aryloxy derivatives (8a–l) are synthesized via copper-catalyzed one-pot sequential hydroxylation-*O*-alkylation/click reaction of 2-bromo-*N*-prop-2-ynyl-benzamides. The products are synthesized in an efficient way in high isolated yields. The synthetic method involves the use of 2-bromo-*N*-prop-2-ynyl-benzamide and various benzyl halides over a one-pot copper-catalyzed hydroxylation-*O*-alkylation/Click reaction. The products are characterized by ^{1}H NMR, ^{13}C NMR, mass spectrometry, FT-IR, elemental analysis, melting point, and single crystal X-ray diffraction. In-situ prepared phenol moiety in H_2O/DMF as a solvent\co-solvent system prompted to perform a reaction between benzyl halide and phenols. The step economic feature of the method leads to the synthesis of the products in high isolated yields.

Introduction

1,4-Disubstituted 1,2,3-triazole compounds are important due to their various biological applications, including antibiotic [1], antitumor [2], and antifungal [3] activity. Recently, many antiproliferative agents were reported with the 1,4-disubstituted 1,2,3-triazole moiety in their molecular structure as an important pharmacophoric core (compounds A and B Fig. 1) [4–12]. This interesting biologically active core conjugated amide moiety has a high binding affinity to many biological targets [13–16]. Some molecular structures of antiproliferative agents bearing amide and ethereal linked 1,2,3-triazolyl skeletons have been shown in Fig. 1 (compounds C and D) [17,18]. Furthermore, it was found that the benzyloxy substituent, the Amide linkage, and the triazole moiety are significant for both inhibition of mitoxantrone efflux and inhibition of basal ATPase activity of ABCG2 (Fig. 1 compound E) [8]. Based on the biological activity of 1,4-disubstituted 1,2,3-triazole compounds, and their affinity to various biological targets, in this paper, a novel series of this class of compounds are synthesized. The structure of one of the synthesized compounds in this paper (8a) is presented in Fig. 1.

Regarding the significance and the biological activities of 1,2,3-triazole derivatives, several efforts have been focused on the synthesis of these compounds. The most common way for the synthesis of 1,4-disubstituted 1,2,3-triazoles is 1,3-dipolar cyclization reaction between an azide and terminal alkyne [19–22]. Regarding this efficient way for the synthesis of the mentioned compounds, several modifications have been reported by introducing novel catalysts with improved activity and selectivity [23–26].

The most prominent classical methods for the preparation of phenols are the conversion of aryl halides to phenols. This reaction is efficient and is reported by several reagents and catalysts, including copper [27–29], palladium [30–35], Iridium [32], or Iron [36–38] catalysts, using various hydroxide salts. Copper-catalyzed direct hydroxylation of aryl halides is reported using various ligands including 1,3-diketones [39,40], pyridine-2-aldoxime [41], D-glucose [42], *N*-heterocyclic carbenes [43,44], and glycolic acid in an H_2O/co-solvent system [45].

In continuation of our endeavor to find efficient methods for the synthesis of biologically active triazolic skeletons [46,47], we became interested in the design and synthesis of new triazole containing derivatives, including amide linkage and ether functional

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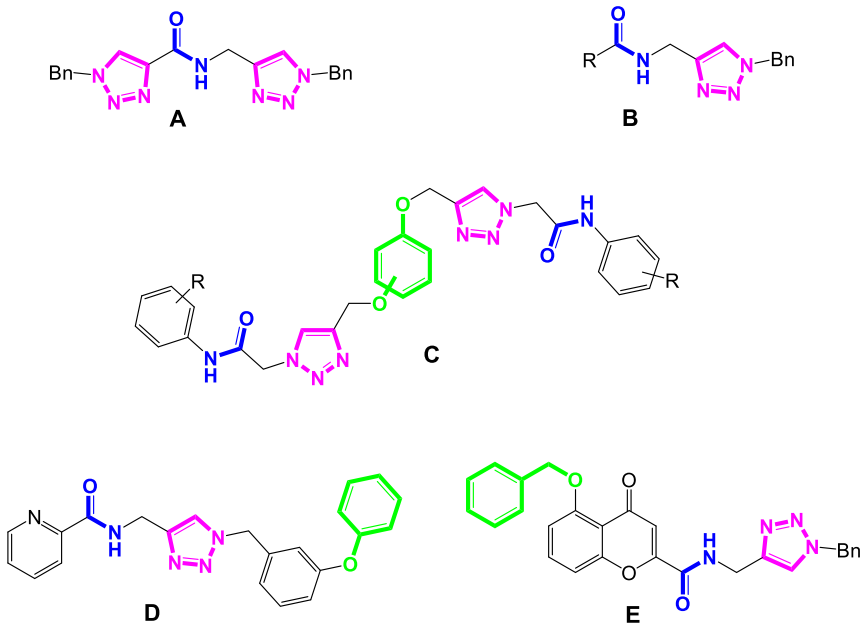
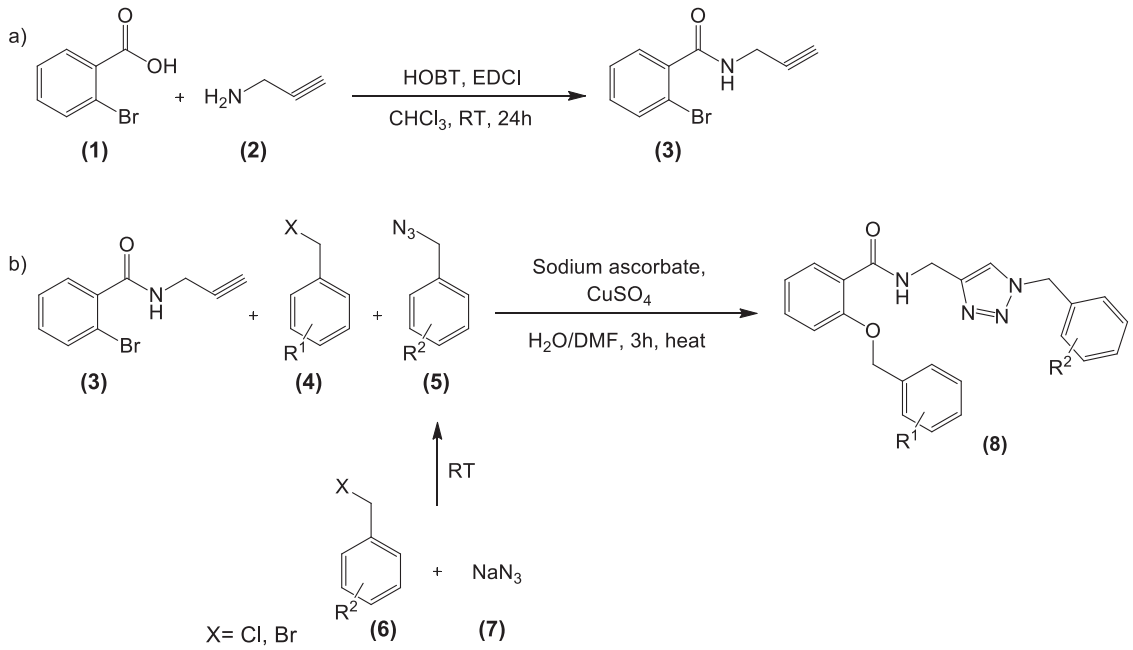


Fig. 1. Ether and amide linked 1,2,3-triazolyl scaffolds as antiproliferative agents.



Scheme 1. Synthesis of *N*-((1-aryl-1*H*-1,2,3-triazol-4-yl)methyl)-2-(aryloxymethyl)benzamide derivatives.

group with a novel and step economy method. Hereby, we describe the copper-catalyzed synthesis of amide linked 1,2,3-triazoles bearing aryloxy moiety in a single molecular framework over an interesting one-pot procedure. The Cu(I)-catalyzed azide-alkyne [3 + 2] cycloaddition (CuAAC) click reaction [14,15,48–53], is an efficient and regiospecific route for the synthesis of 1,4-disubstituted 1,2,3-triazoles with numerous applications [13,51,52,54–57]. Inspired by the idea to synchronize several catalytic reactions in a one-pot manner, including sequential, domino or tandem catalysis [58–66], we set out to combine hydroxylation of aryl bromide following by *O*-Alkylation of in-situ prepared phenol with click reaction of terminal alkynes to have consecutively occurred in a one-pot fashion in a molecule (Scheme 1b). The reaction has had the best performance in the mixture of water and

Table 1
Optimization conditions of the synthesis of *N*-((1-aryl-1*H*-1,2,3-triazol-4-yl)methyl)-2-(aryloxymethyl)benzamide derivatives by using of the compound 8a (R_1, R_2 = H) as the model compound.

Entry	Solvent	Catalyst (mol%)	Temperature (°C)	Yield (%) b
1	H_2O	$CuSO_4$ (10)	Reflux	31
2	EtOH	$CuSO_4$ (10)	Reflux	40
3	MeOH	$CuSO_4$ (10)	Reflux	35
4	CH_2Cl_2	$CuSO_4$ (10)	Reflux	21
5	DMF	$CuSO_4$ (10)	Reflux	62
6	DMSO	$CuSO_4$ (10)	Reflux	55
7	H_2O/EtOH a	$CuSO_4$ (10)	Reflux	42
8	H_2O/DMF a	$CuSO_4$ (10)	Reflux	84
9	H_2O/DMF	CuI (10)	Reflux	69
10	H_2O/DMF	$CuCl_2$ (10)	Reflux	58
11	H_2O/DMF	$Cu(OH)_2$ (10)	Reflux	33
12	H_2O/DMF	$CuSO_4$ (5)	Reflux	17
13	H_2O/DMF	$CuSO_4$ (7)	Reflux	59
14	H_2O/DMF	$CuSO_4$ (15)	Reflux	84
15	H_2O/DMF	$CuSO_4$ (10)	25	9
16	H_2O/DMF	$CuSO_4$ (10)	75	42
17	H_2O/DMF	$CuSO_4$ (10)	100	72

[a] 1:1 mixture; [b] isolated yield.

DMF, which is similar to the common solvent/co-solvent system for click reaction, catalyzed by a copper catalyst [67,68]. Utilizing intermediate 3 as a relative activated aryl-bromide, the hydroxylation reaction can proceed efficiently. On the other hand, the substitution reaction between phenolic moiety and benzyl halides is well understood [69–72] and this could be an interesting sequential one-pot reaction once phenol part is prepared in situ. The advantage of this method is the use of a novel one-pot multi-component reaction as a step economic procedure for the synthesis of aryloxy containing amide linked 1,2,3-triazole analogues. The synthetic route is presented in Scheme 1.

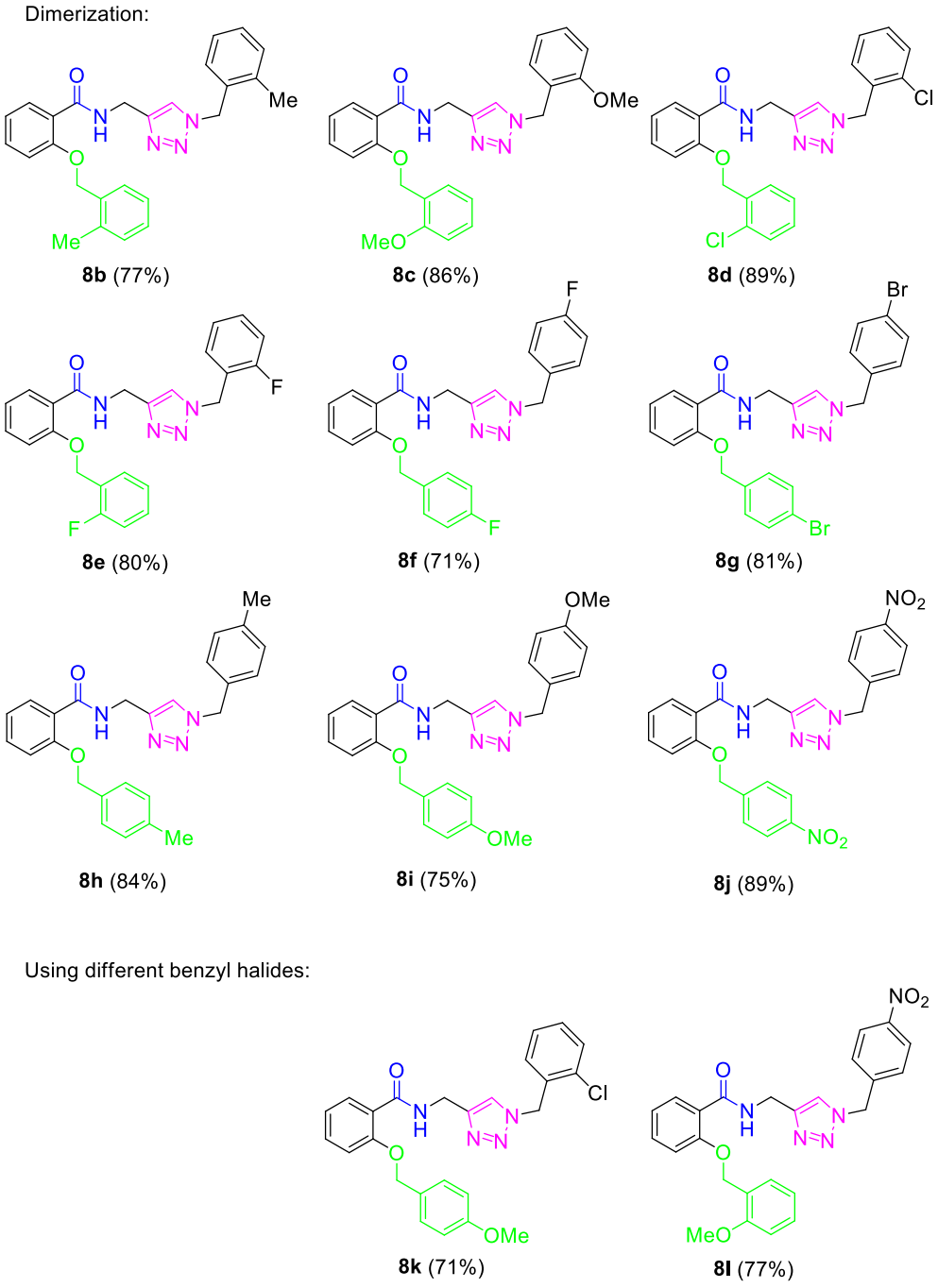
Results and discussion

In this paper, a novel and efficient method are reported for the synthesis of *N*-((1-aryl-1*H*-1,2,3-triazol-4-yl)methyl)-2-(aryloxymethyl)benzamide derivatives. The synthesis of new aryloxy containing amide linked 1,2,3-triazoles (8a-l) is carried out via a one-pot manner is illustrated in Schemes 1. The multicomponent reaction is passed on the reaction of 2-bromo-*N*-(prop-2-yn-1-yl) benzamide (3), benzyl halide derivatives (4), and benzyl azide derivatives (5) in a mixture of H_2O/DMF as a solvent and in the presence of sodium ascorbate and a copper catalyst. 2-Bromo-*N*-(prop-2-yn-1-yl)benzamide was separately synthesized from the reaction of 2-bromobenzoic acid (1) and propargylamine (2) via amidation reaction, as described in Scheme 1a. Besides, benzyl azides were obtained from the reaction of the corresponding benzyl halide (6) and sodium azide (7) at room temperature. After in-situ preparation of the azidomethyl benzene derivatives (5) from corresponding benzyl halide and sodium azide in DMF/H_2O (1:1) at room temperature, the compounds 3 and 4 were added to the reaction vessel and the reaction was carried out in the presence of copper iodide and sodium ascorbate at 150 °C. The reaction was completed after 3 h, which resulted into the formation of corresponding aryloxy containing amide linked 1,2,3-triazoles (8a–l) in excellent yields.

For the synthesis of *N*-((1-aryl-1*H*-1,2,3-triazol-4-yl)methyl)-2-(aryloxymethyl)benzamide derivatives from the multicomponent reaction of 2-bromo-*N*-(prop-2-yn-1-yl)benzamide, benzyl azide and benzyl halide with the most efficiency, the reaction was performed in various conditions and the effect of different variables on the reaction performance was optimized. For this purpose, the reaction was performed in protic and aprotic solvents, and mixture of water and DMF as solvent. In addition, several copper sources were used as catalyst for the reaction performance. For finding the optimal reaction conditions, the reaction of 2-bromo-*N*-(prop-2-yn-1-yl)benzamide, 4-methylbenzyl chloride was selected and the model reaction. Studying the reaction performance in various conditions showed that the highest isolated yields of the products were obtained when the reaction was performed in the presence of 10 mol% of copper sulfate a 1:1 (v/v) mixture of H_2O/DMF under reflux conditions. Performing the reaction in various protic or aprotic solvents with different polarities showed that a 1:1 (v/v) mixture of H_2O/DMF is the best solvent and the highest isolated yields were obtained in this solvent system. In addition, several copper sources were used as the catalyst. 10 mol% of copper sulfate showed to be the best catalyst for the reaction performance. It should be noted that the presence of a catalytic amount of sodium ascorbate is critical for the reaction performance. The results of the optimization conditions are presented in Table 1.

The scope and the generality of the method were studied by using various starting materials. For this purpose, several substrates with different functionalities were used as starting materials. The structure of the products is presented in Scheme 2. It could be observed that the reactions with all the applied substrates have led to the desired products in high isolated yields. The substrates with electron-donating or electron-withdrawing functionalities have given the products in very good isolated yields. All the synthesized compounds have been characterized by physical data and spectral analysis (the copy of the ^{1}H NMR and ^{13}C NMR spectra are available in supporting information).

The structures of the products (8a-l) were fully characterized by several characterization techniques, including ^{1}H NMR, ^{13}C NMR,



Scheme 2. Structures of synthesized new analogs of aryloxy containing amide linked 1,2,3-triazoles (8a-l).

MASS, FT-IR, elemental analysis, and melting point. In addition, for the exact characterization of the products, single-crystal X-ray diffraction method was applied. The structure of *N*-((1-(2-chlorobenzyl)-1*H*-1,2,3–triazol–4-yl)methyl)-2-((2-chlorobenzyl) oxy)benzamide (8d) was confirmed by single crystal XRD. The structure of these compounds, which was obtained by single-crystal XRD results, is presented in Fig. 2. As can be seen in the crystal structure, the new bond has been formed between aryl bromide (C-4) and the oxygen atom with the bond length of 1.38 Å.

For studying the mechanism of the synthesis of amide linked 1,2,3-triazoles bearing aryloxy skeletons, a possible mechanism was proposed according to DFT calculations. The proposed mechanism is presented in Fig. 3. The mechanism is calculated for the participation of compound 3 in the multicomponent reaction to form compounds 8. The key role of copper catalyst is presented in the suggested mechanism. Coordination of copper to "N" and "Br" atoms lead to C-Br bond weakening. The C-Br bond weakening results in the C—O bond formation in the transition state. C-Br

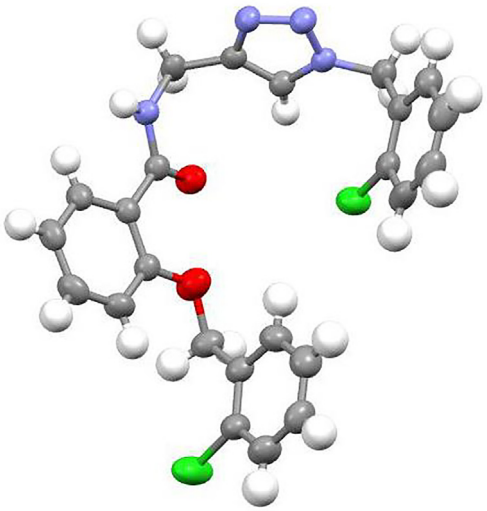


Fig. 2. ORTEP single crystal XRD of compound 7e.

bond cleavage leads to the formation of C—O bond in intermediate, which reacts with benxyl halide.

Conclusion

In this paper, a novel method is introduced for the synthesis of *N*-((1-(2-aryl)-1*H*-1,2,3–triazol–4-yl)methyl)–2-(2-aryloxy)benzamide derivatives. The method is based on a multicomponent copper-catalyzed one-pot sequential hydroxylation-*O*-alkylation/click reaction of 2-bromo-*N*-prop-2-ynyl-benzamides, benzyl chlorides, and benzyl azides. The synthesis involved simultaneous reactions, including click reaction, phenol formation reaction, and the reaction of the phenol moiety with benzyl halide derivatives. The advantage of this method is performing the mentioned reactions at the same time via an efficient multicomponent reaction. The products are synthesized in an efficient way in high isolated yields. The reaction is performed in a 1:1 (v/v) mixture of H_2O/DMF as solvent under reflux conditions. A wide scope of substrates was used in the reaction and all the substrates led to the desired products in high isolated yields. The products are characterized by ^{1}H NMR, ^{13}C NMR, mass spectroscopy, FT-IR, elemental analysis, melting point, and single-crystal X-ray diffraction. Single-crystal XRD methods proved the bond formation between aryl bromide (C-4) and the oxygen atom with a bond length of 1.38 Å. In general, the method is efficient and simple and the products are obtained in very good isolated yields.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data (experimental, spectral data and the copies of ^{1}H NMR and ^{13}C NMR spectra) to this article can be found online at https://doi.org/10.1016/j.tetlet.2020.152765.

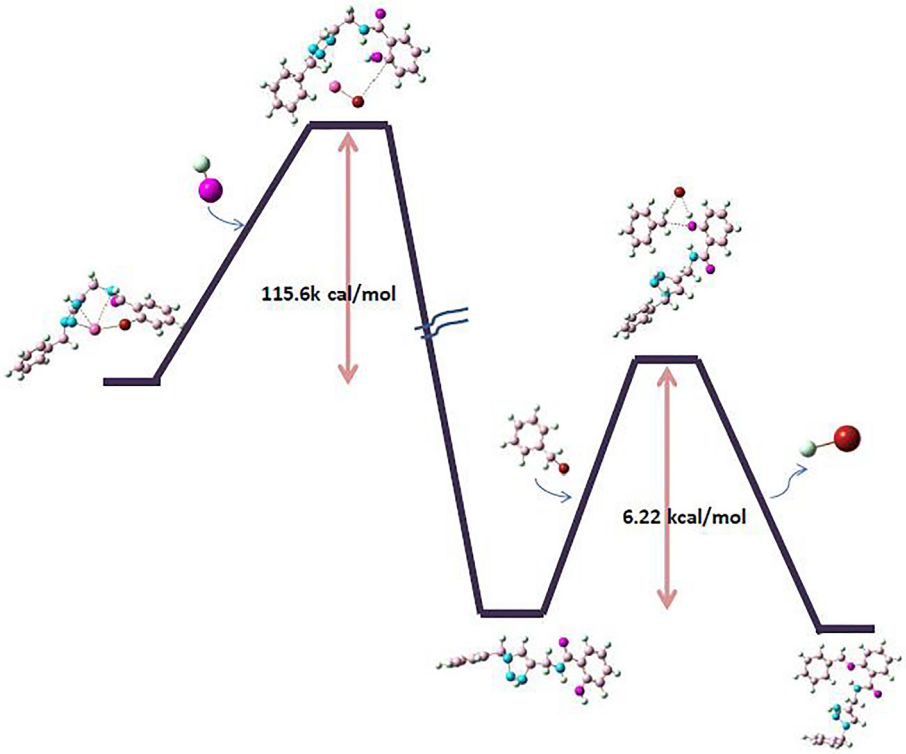


Fig. 3. DFT calculation of the mechanism of amide linked 1,2,3-triazoles bearing aryloxy skeletons synthesis.

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