One Pot Multicomponent Synthesis of Highly Commutated 1,2,3-Triazoles using some Pyrazole aldehyde through “Click” Reaction

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ABSTRACT

1,2,3 Trizole compounds are widely applied in major several technical and research areas especially in drug discovery new chemical entities like trizoles are developed via click reactions. Synthesis of heterocycles through cycloaddition reaction between azides and alkynes by employing azides using copper as catalyst is said to be Click reaction. Most commonly triazoles are utilized in medicinal field as a drug linkers for bioconjugation. It found to have potential multiple applications in biological as well as medical sciences. We describe herein the novel and efficient three step multicomponent synthesis of highly substituted 1,2,3-triazole derivatives from pyrazole aldehyde, diaminobenzene via N-alkylation by Click reaction. For the future, our perspective is studies of anti-cancer, anti-viral and antimicrobial activities in 1,2,3-triazole.

Keyword: 1,2,3-triazole, Pyrazole aldehyde, Click reaction, CuAAC.

INTRODUCTION

“Click chemistry” includes reactions which have simple and easy reaction conditions devoid of chromatographic separations, very high% of product formation, highly stereospecific, involving quick and easily removable solvents, mostly carried out at room temperature. This methodology includes carbon–carbon multiple bond addition reaction, epoxide formation, heterocycle synthesis etc… that enables the synthesis of many biologically potential drug like organic compounds¹. Cycloaddition of azides with alkynes by sharpless et al.,² is one such reaction that satisfies all the above requisites involving water as solvent that resulting in formation of 1,4-disubstituted 1,2,3-triazoles³⁻⁵. The unique characteristic of the 1,4-disubstituted 1,2,3-triazole ring, based on its ability to contribute in hydrogen bonding and dipolar interactions, makes click chemistry even more beneficial for a several synthesis
applications. This reaction seems to be far better than Huisgen reaction as reported by A. Michael that includes the catalyst free reaction between phenyl azide and diethylacetylenedicarboxylate to form triazoles accompanied by two to four by-products\(^6\)\(^{-8}\). triazoles are 5-membered heterocycles with N as heteroatom’s which are most suitable moieties utilized in preparation of pharmaceuticals\(^9\)\(^{-12}\), biologically active compounds\(^13\) such as fluconazole\(^14\) and found useful in material science. This triazole core is also found in medicines for diseases like cancer\(^15\), HIV\(^16\), bacterial\(^17\), malarial infections\(^18\) and for tuberculosis\(^19\). Moreover they show potential resistance to oxidation, hydrolysis and other metabolic degradation\(^20\).

An efficient method to synthesis highly regioselective, 4-disubstituted 1,2,3-triazoles (1,4-DTs) using Cu(I)–CuAAC was reported.\(^21\) Subsequently, 1,5-disubstituted 1,2,3-triazoles were synthesized by the RuAAC reaction.\(^22\) IrAAC reactions and the Pd-catalyzed alkynyl bromide-acid cyclodition were developed for the triazole synthesis.\(^23\)\(^{-25}\)

Considering the biological significance of the 1,4-DTs and its derivatives various synthetic methods were proposed using transition metal catalysts such as silver, zinc, ruthenium, and iridium, but on contrary their biological applications got further limited.

In this investigation, we reported the synthetic route for a series of 1,4-DTs that processed via one pot condensation of pyrazole aldehyde, diaminobenzene via, N-alkylation through Click reaction. All the synthesized triazoles were characterized by FTIR, \(^1\)H, \(^13\)CNMR and HRMS.

**EXPERIMENTAL**

**Synthesis of triazole compound 7a-j**

A mixture of prepared compound 5 (1 equiv.) (A. Keivanloo et al.\(^26\)), NaN\(_3\) (1.2 equiv.) was stirred with benzyl bromide (1.2 equiv.), triethylamine (2.5 equiv.) and CuI (2.5 mol%) in 2mL of t-butanol:water (1:1) mixture at normal temperature for 2 hours. The reaction was periodically monitored via TLC in order to check the reaction was completed. The mixture was further extracted with ethyl acetate and the catalyst was removed by filtration. Finally, the filtrate was concentrated and the residue was run through column chromatography with 40% ethyl acetate–hexane. The Pure triazole derivatives are obtained at better yields.

**RESULTS AND DISCUSSION**

To a mixture of 5a, Benzyl bromide 6a, NaN\(_3\), Et\(_3\)N and CuI, t-butanol:water was added in 1:1 ratio and stirring was done at normal temperature for 2 hours. The crude was extracted with ethylacetate and purified via column chromatography (40% ethyl acetate–hexane mixture) to produce triazole in excellent yields. The structure of the products obtained were characterize by using \(^1\)H and \(^13\)C nmr and HR-mass spectroscopy. The products were summarizing in Table 3.
The solvent screening was carried out using water, t-BuOH and mixture of tertiary butanol and water and it was found that, the better result were obtained with tertiary butanol and water mixture. The results were summarized in Table 2.

The ^1^H-nmr range of compound 7a displayed a two singlet at 5.06ppm and 5.19ppm clearly shows that the presence of two methylene group (-CH2). The singlet at 8.25ppm was assigned to pyrazole ring proton. The singlet at 7.41ppm was attributed to triazole ring proton. The range of 7.77-6.98ppm was assigned to aromatic proton.

The 13^C^-nmr spectra, the peak at 40.5ppm and 54.1ppm was attributed to methylene carbon. The peak at 110.9ppm was assigned to pyrazole ring carbon and a peak at 122.0ppm was assigned to triazole ring carbon. The peak appeared at 151.5ppm was imidazole ring carbon. The HR-Mass spectrum reveals the molecular ion peak [M]+ at m/z 619.

| Product | Time (h) | Water | t-BuOH | t-BuOH - Water mixture |
|---------|----------|-------|--------|-----------------------|
| 7a      | 4        | 45    | 81     | 92                    |
| 7b      | 6        | 42    | 70     | 88                    |
| 7c      | 4        | 36    | 76     | 90                    |
| 7d      | 5        | 47    | 78     | 94                    |
| 7e      | 6        | 31    | 67     | 86                    |
| 7f      | 4        | 35    | 75     | 88                    |
| 7g      | 3.5      | 44    | 77     | 90                    |
| 7h      | 4        | 48    | 80     | 96                    |
| 7i      | 4.5      | 42    | 74     | 90                    |
| 7j      | 6        | 40    | 73     | 92                    |

The scope of reaction was elaborated to other N-Propargyl Pyrazolyl benzoimidazole (5a-d) and benzylbromide 6a-c (Table 3). The reaction afforded compounds 7a-j in very good yields. The scopes of the reaction are summarized in Table 3.

| Product | Time (h) | Water | t-BuOH | t-BuOH - Water mixture |
|---------|----------|-------|--------|-----------------------|
| 7a      | 4        | 45    | 81     | 92                    |
| 7b      | 6        | 42    | 70     | 88                    |
| 7c      | 4        | 36    | 76     | 90                    |
| 7d      | 5        | 47    | 78     | 94                    |
| 7e      | 6        | 31    | 67     | 86                    |
| 7f      | 4        | 35    | 75     | 88                    |
| 7g      | 3.5      | 44    | 77     | 90                    |
| 7h      | 4        | 48    | 80     | 96                    |
| 7i      | 4.5      | 42    | 74     | 90                    |
| 7j      | 6        | 40    | 73     | 92                    |

Also isolated yield. Base Et3N and catalyst CuI was used for all the reactions.
Table 3: Synthesis of 1,2,3-triazole (7a-j)

| S.No | Reactant (5) | Benzyl bromide | Product (7)a | Time (h) | Yieldb (%) |
|------|--------------|----------------|--------------|----------|------------|
| 1    | ![image](image1.png) | ![image](image2.png) | ![image](image3.png) | 4        | 92         |
| 2    | ![image](image4.png) | ![image](image5.png) | ![image](image6.png) | 3.5      | 88         |
| 3    | ![image](image7.png) | ![image](image8.png) | ![image](image9.png) | 4        | 90         |
| 4    | ![image](image10.png) | ![image](image11.png) | ![image](image12.png) | 4.5      | 94         |
| 5    | ![image](image13.png) | ![image](image14.png) | ![image](image15.png) | 3        | 86         |
| 6    | ![image](image16.png) | ![image](image17.png) | ![image](image18.png) | 4        | 88         |
| 7    | ![image](image19.png) | ![image](image20.png) | ![image](image21.png) | 3.5      | 90         |
| 8    | ![image](image22.png) | ![image](image23.png) | ![image](image24.png) | 4.5      | 96         |
| 9    | ![image](image25.png) | ![image](image26.png) | ![image](image27.png) | 4        | 90         |
| 10   | ![image](image28.png) | ![image](image29.png) | ![image](image30.png) | 3.5      | 92         |

aAll the integrated compounds were characterized by FTIR, ¹H NMR, ¹³C NMR, HRmass spectroscopy
bIsolated yield after column chromatography
Table 4: Characterization of compounds 7a-j

| P   | FTIR            | ¹H-NMR                        | ¹³C-NMR          | HRMS [M]+ m/z   | Rf   | m.p. (°C)(cm⁻¹) |
|-----|-----------------|-------------------------------|-----------------|----------------|------|-----------------|
| 7a  | 3431, 3136, 3090, 3057, 2925, 1915, 1899, 1596, 1505, 1457, 1439, 1409, 1391, 1330, 1223 | 8.25 (s, 1H), 7.77 – 7.70 (m, 3H), 7.52 – 7.47 (m, 2H), 7.46 – 7.40 (m, 2H), 7.31 – 7.28 (m, 1H), 7.27 – 7.19 (m, 5H), 7.17 – 7.13 (m, 3H), 7.02 – 6.97 (m, 2H), 6.37 (s, 1H), 5.19 (s, 2H), 5.06 (s, 2H) | 151.6, 146.8, 143.4, 143.3, 139.6, 135.1, 134.4, 132.5, 130.4, 129.7, 129.2, 128.9, 128.7, 127.9, 127.7, 127.4, 123.3, 122.8, 122.1, 120.1, 119.5, 111.0, 54.2, 40.5 | 507.2199 | 0.47 | 164–168 |
| 7b  | 3433, 3133, 3084, 2924, 2854, 1596, 1522, 1455, 1403, 1347, 1229 | 8.37 (s, 1H), 8.01 – 7.98 (m, 1H), 7.72 (m, 2H), 7.50 – 7.46 (m, 2H), 7.44 – 7.38 (m, 5H), 7.30 – 7.27 (m, 1H), 7.27 – 7.24 (m, 1H), 7.23 (dd, J = 2.1, 1.0 Hz, 1H), 7.15 – 7.11 (m, 3H), 6.82 – 6.79 (m, 1H), 6.71 (s, 1H), 5.59 (s, 2H), 5.12 (s, 2Ha) | 151.6, 147.5, 146.7, 143.2, 142.9, 139.6, 134.9, 134.4, 132.4, 130.6, 130.4, 130.2, 129.9, 129.8, 129.0, 128.7, 127.7, 127.4, 125.5, 123.5, 123.4, 123.0, 119.9, 119.5, 111.0, 110.5, 50.8, 40.4 | 552.1998 | 0.52 | 190–194 |
| 7c  | 3430, 3133, 3066, 2923, 2851, 1935, 1868, 1665, 1581, 1508, 1441, 1405, 1388, 1230 | 8.30 (s, 1H), 7.73 (dd, J = 8.7, 1.2 Hz, 3H), 7.51 – 7.46 (m, 2H), 7.44 – 7.31 (m, 1H), 6.30 – 6.27 (m, 7H), 7.27 – 7.24 (m, 1H), 7.23 (dd, J = 2.1, 1.0 Hz, 1H), 7.15 – 7.11 (m, 3H), 6.82 – 6.79 (m, 1H), 6.71 (s, 1H), 5.59 (s, 2H), 5.12 (s, 2Ha) | 151.0, 146.8, 145.2, 143.2, 143.1, 139.6, 135.0, 133.8, 133.2, 132.9, 132.4, 130.5, 130.4, 130.1, 129.7, 129.6, 129.0, 128.9, 128.7, 128.2, 127.6, 127.3, 127.2, 123.3, 122.8, 122.5, 120.0, 119.5, 119.4, 114.2, 110.9, 110.8, 53.8, 40.4 | 585.1299 | 0.5 | 182–186 |
| 7d  | 3433, 3128, 3064, 2957, 2931, 2835, 2042, 1891, 1611, 1595, 1509, 1455, 1400, 1372, 1245 | 7.67 (d, J = 8.1 Hz, 3H), 7.37 (p, J = 7.2, 6.8 Hz, 6H), 7.26 (d, J = 7.4 Hz, 1H), 7.20 – 7.18 (m, 2H), 7.15 (d, J = 1.9 Hz, 1H), 6.98 (dd, J = 6.5, 3.0 Hz, 3H), 6.69 (dd, J = 12.1, 9.3 Hz, 3H), 6.48 (s, 1H), 5.17 (s, 2H), 5.04 (s, 2H), 3.66 (s, 2H) | 160.1, 160.0, 151.2, 143.1, 139.3, 134.3, 133.9, 129.7, 129.1, 128.9, 128.9, 128.8, 128.7, 128.6, 127.9, 127.7, 127.2, 121.7, 124.8, 124.6, 123.5, 122.9, 122.2, 119.4, 199.3, 119.2, 114.4, 55.3, 54.1 | 537.2299 | 0.45 | 196–200 |
| 7e  | 3434, 3129, 3058, 2921, 2851, 1893, 1594, 1503, 1454, 1400, 1371, 1330, 1224 | 8.42 (s, 1H), 7.69 (dt, J = 8.9, 1.9 Hz, 3H), 7.39 (dd, J = 8.8, 4.0, 2.1 Hz, 5H), 7.30 – 7.25 (m, 2H), 7.24 – 7.18 (m, 2H), 7.21 (m, 4H), 7.09 (s, 1H), 7.08 – 7.04 (m, 3H), 6.74 (s, 1H), 5.27 (s, 2H), 5.16 (s, 2H) | 150.6, 146.3, 143.1, 139.4, 134.7, 134.6, 134.4, 130.7, 129.8, 129.2, 129.0, 129.0, 128.9, 128.0, 127.5, 127.3, 123.2, 122.2, 119.7, 119.4 | 541.1799 | 0.54 | 234–238 |
| 7f | 3434, 3133, 3083, 2922, 2852, 1892, 1595, 1525, 1466, 1339, 1228 | 8.55 (d, J = 5.0 Hz, 1H), 8.11 – 8.07 (m, 1H), 7.80 – 7.77 (m, 2H), 7.56 – 7.44 (m, 8H), 7.36 – 7.32 (m, 3H), 7.16 – 7.09 (m, 3H), 7.02 – 6.98 (m, 1H), 5.75 (s, 2H), 5.28 (s, 2H) | 119.2, 110.9, 110.1, 54.3, 40.5 |
| 7g | 3427, 3131, 3079, 2923, 2852, 1892, 1594, 1506, 1441, 1336, 1230 | 8.38 (s, 1H), 7.73 – 7.69 (m, 3H), 7.47 – 7.41 (m, 5H), 7.26 – 7.20 (m, 3H), 7.16 – 7.07 (m, 5H), 6.92 (dt, J = 7.5, 1.9 Hz, 1H), 6.87 (s, 1H), 5.39 (s, 2H), 5.15 (s, 2H) | 150.5, 146.5, 145.3, 143.2, 143.1, 139.5, 135.0, 134.6, 133.8, 133.3, 130.8, 130.6, 130.5, 130.3, 129.8, 129.1, 129.0, 128.9, 128.5, 128.3, 127.5, 123.5, 123.0, 122.3, 120.1, 119.5, 110.7, 54.0, 40.3 |
| 7h | 3434, 3129, 3058, 2921, 2851, 1893, 1514, 1503, 1454, 1330, 1224 | 8.30 (s, 1H), 7.77 – 7.73 (m, 1H), 7.73 – 7.68 (m, 2H), 7.46 – 7.34 (m, 6H), 7.33 – 7.29 (m, 3H), 7.24 – 7.20 (m, 4H), 7.06 – 7.01 (m, 2H), 6.59 (s, 1H), 5.25 (s, 2H), 5.11 (s, 2H) | 150.6, 146.4, 143.3, 143.2, 139.5, 135.0, 134.4, 132.0, 131.4, 130.5, 129.8, 129.3, 129.2, 129.0, 128.9, 127.9, 127.8, 127.5, 123.5, 123.0, 122.9, 121.9, 120.2, 119.5, 110.8, 54.3, 40.4 |
| 7i | 3434, 3132, 3081, 2923, 2852, 1594, 1519, 1451, 1339, 1227 | 8.88 (s, 1H), 8.04 – 7.98 (m, 1H), 7.97 – 7.91 (m, 2H), 7.86 (d, J = 7.9 Hz, 2H), 7.78 (d, J = 2.5 Hz, 1H), 7.66 – 7.60 (m, 1H), 7.57 – 7.52 (m, 1H), 7.50 – 7.43 (m, 6H), 7.36 (d, J = 1.9 Hz, 1H), 7.25 – 7.19 (m, 2H), 6.80 – 6.73 (m, 1H), 5.79 (s, 2H), 5.36 (s, 2H) | 150.6, 147.6, 134.5, 132.4, 131.1, 130.6, 130.2, 129.8, 129.3, 128.0, 125.6, 125.5, 119.9, 51.4, 29.8 |
| 7j | 3413, 3131, 3075, 2928, 2852, 1899, 1594, 1502, 1441, 1333, 1228 | 8.32 (s, 1H), 7.76 – 7.72 (m, 2H), 7.45 (d, J = 1.7 Hz, 1H), 7.42 (dt, J = 7.2, 1.2 Hz, 3H), 7.40 (d, J = 2.0 Hz, 1H), 7.37 – 7.34 (m, 1H), 7.32 – 7.28 (m, 3H), 7.25 – 7.20 (m, 2H), 7.16 – 7.06 (m, 3H), 6.89 (dd, J = 7.4, 1.9 Hz, 1H), 6.80 (s, 1H), 5.37 (s, 2H), 5.12 (s, 2H) | 150.5, 146.5, 145.3, 143.3, 143.1, 139.5, 135.0, 133.8, 133.3, 132.0, 131.3, 130.6, 130.5, 130.3, 129.8, 129.2, 128.3, 127.5, 123.5, 123.4, 122.9, 122.9, 122.3, 120.2, 119.5, 110.7, 54.0, 40.3 |

P = Product, $^1$H NMR = 400MHz - CDCl$_3$, $^{13}$C NMR = 100MHz - CDCl$_3$, Rf = 40% EtOAc - Hexane, MP = Melting Point, 7a-7d and 7f-7j = white powder, 7e = yellow powder
CONCLUSION

We have demonstrated a one pot multicomponent reaction that offers a simple method for the synthesis of biologically important 1,2,3-triazole derivatives from substituted imidazole, NaN₃, benzyl bromide, triethylamine, and CuI in t-butanol:water (1:1). This method offers more precedence like light reaction conditions, latent period, no toxic byproducts, good yield and simple experimental and isolation procedures making it a methodical route to synthesize the derivatives of 1,2,3-triazole. For the future, our perspective is anticancer, anti-viral and antimicrobial activities studies in 1,2,3-triazole derivatives.

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