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Triazole Synthesis by using Transition Metal based Heterogenous Catalyst

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Abstract : *A novel, green and efficient synthesis has been devised for some substituted triazole derivatives by treating active methylene compounds with acyl azide using Heterogenous catalyst. The interesting features of the present synthesis over existing routes are ecofriendly synthesis, under normal reaction conditions, good yields with high selectivity, minimum reaction times, use of catalytic source, less wastage and recycling of the metal-based catalyst. The structures of the synthesized molecules were confirmed by spectral techniques like IR, Proton NMR and 13C NMR.*

Key Words: Active methylene, Heterocycles, Ferrite nano-catalyst, sustainability.

I. INTRODUCTION

Triazoles are class of 5 membered heterocyclic compounds containing 'N' atom in their structure (1-2). The synthesis of this type of molecule has received great attention due to the various medicinal properties that have been attributed to chemically distinct azoles and azines (3-4). Atorvastatin, voriconazole, Fluconazole, itraconazole, Cimetidine etc. are well known antifungal drug commercially available which contain a N-based heterocyclic ring (5-8). Celecoxi-b is a analgesic agent and NSAID belongs to triazole class (9-11). Ferrite nano catalyst plays an important role in most heterogeneous catalytic reactions that considerably growing with the development of synthetic protocol that enables fine control of morphology, size, composition and shape of transition metal-based nano-catalyst at atomic level (12-14).

II. EXPERIMENTAL

Physical constant such as M.P of all synthesized products were determined in open capillary tubes on an electro thermal apparatus and are uncorrected. The reaction was controlled by TLC with the help of visualizing agent such as U.V light. 1H NMR spectrum were recorded on 400 Mega Hertz NMR spectrophotometer by using DMSO-d6 as solvent and Tetra Methyl Silane as an internal standard (chemical shifts is determined in δ ppm).

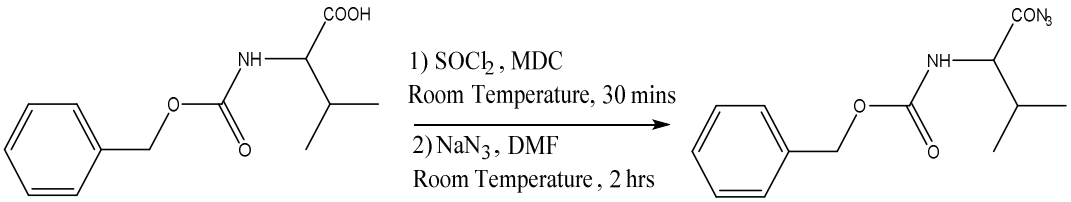
Preparation of (1-Azidocarbonyl-2-methylpropyl)carbamic acid benzyl ester (2)

In a 100 ml flask, mixture of SOCl2 (3g, 0.037 mol) and compound 1 (6.25 g, 0.025 mol) in CH2Cl2 (13mL) was transferred. The solution obtained was stirred for 4 hrs. The progress of reaction was controlled by TLC (for preparing methyl ester of acyl chloride). Upon completion of reaction, solvent was removed under vacuum distillation, the product obtained was used as such for next step. Acid Chloride obtained and sodium azide (0.015mol) in dry DMF (35mL) was stirred at 20°C for at least 6hrs. The reaction was monitored by Chromatographic technique. After completion, the reaction mixture was poured into cold water (250 mL). The product obtained, was filtered, washed with ice cold water followed by dryether to get compound 2.

Yield of the reaction: 78%, Melting point.: 268-270 °C

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Scheme 1 for acyl azide:

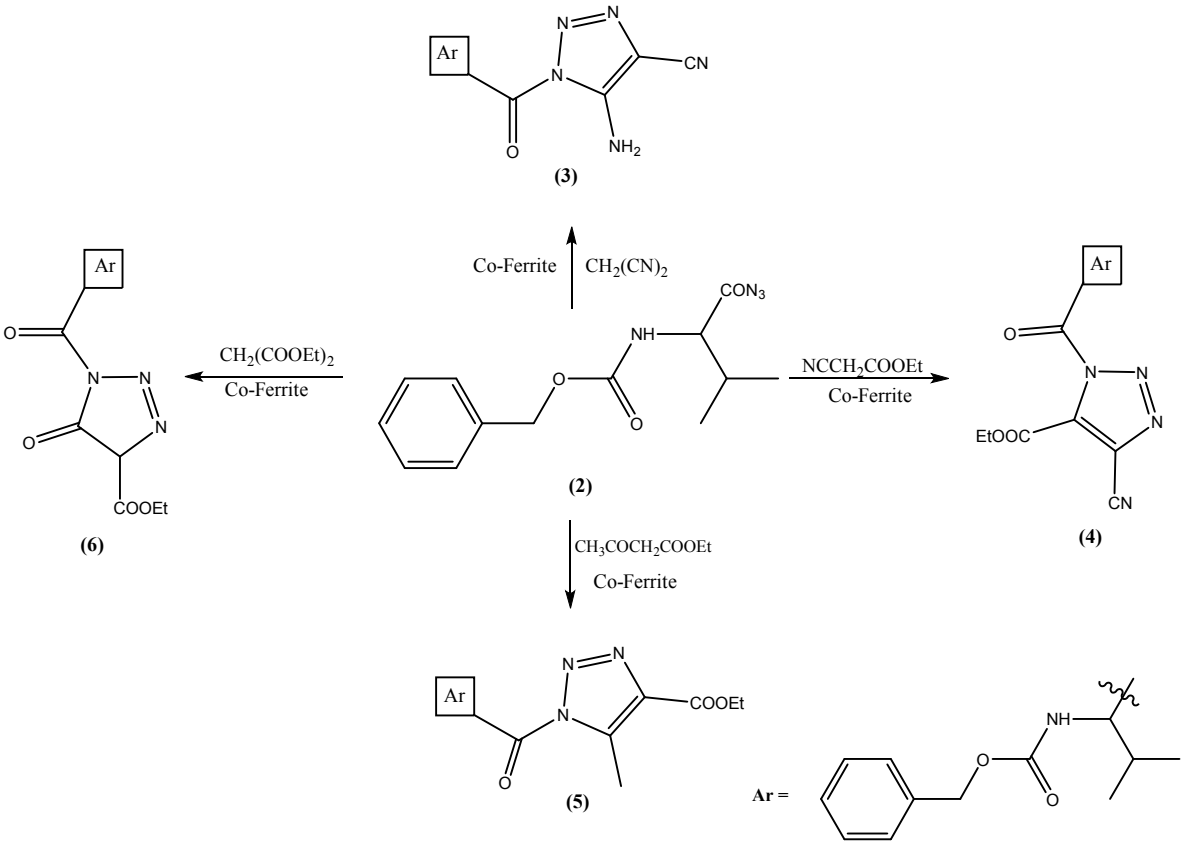


(1) (2)

Co-Ferrite catalyzed synthesis of [1(5-Amino-4-cyano-[1, 2, 3]-triazole-1-carbonyl)-2-methylpropyl]carbamic acid benzyl ester (3-6)

Ice Cold solution of compound 2 (0.690g, 0.01mol) was added to active methylene compound (0.01mol) in the presence of catalytic amount of Cobalt Ferrite Nanoparticles, the reaction mixture was stirred at room temperature in ethanol, the progress of reaction was monitored by Thin Layer Chromatography (TLC). The compound obtained was poured onto ice; solid product thus obtained was collected by filtration and recrystallized from suitable solvent to obtain compound 3, 4, 5 and 6.

Scheme 2 for synthesis of triazoles:



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Table 1: Data of synthesized products 3-6

Compounds	Molecular formula of products	Molecular mass of products	Melting points (°C)	Percentage Yield
3	C16H18N6O3	342	290	85
4	C19H21N5O5	399	297	86
5	C19H24N4O5	388	Above 300	81
6	C18H22N4O6	390	Above 300	82

Spectral Characterisation data of Compound 3-6:

Compound 3: 1(2-Benzyloxycarbonylamino-3-methyl-1-oxo-but-1-yl)4-amino-5-cyano-3H-[1, 2, 3] triazole

IR (cm-1): 3325(NH), 2155(CN), 1685(C=O).

1H NMR (δ ppm): 1.11 (d, 6H, 2 x CH3), 2.62 (m, 1H, CH), 4.02(s, 2H, NH2), 4.41 (d, 1H, CH), 5.34 (s, 2H, OCH2), 6.12 (s, 1H, NH), 7.19- 7.28 (m, 5H, Ar-H)

13C NMR (δ ppm): 16.61 (2 x CH3), 28.1 (CH), 59.6(CH), 69.6(CH2), 118.2(CN), 127.3-140.23(ArC, C=C), 161.52(C=O), 179.02(C=O).

Compound 4: 1-(2-Benzyloxycarbonylamino-3-methyl-1-oxo-but-1-yl)-5-cyano-3H-[1, 2, 3] triazole-4-carboxylic acid ethyl ester

IR (cm^{-1}): 3335(NH), 2155(CN),1685(CO).

1H NMR (δ ppm): 1.24 (d, 6H, 2 x CH3), 1.34 (t, 3H, CH3), 2.7 (m, 1H, CH), 3.5(q, 2H, CH2), 4.4 (d, 1H, CH), 5.35 (s, 2H, OCH2), 6.1 (s, 1H, NH), 7.19- 7.28 (m, 5H, Ar-H)

13C NMR (δ ppm): 13.61(CH3), 16.6(2 x CH3), 28.3(CH), 58.62(CH2), 59.6(CH), 69.6(CH2), 118.2(CN), 128.3-141.5(ArC, C=C), 161.8(C=O), 171.5(C=O), 179.5(C=O).

Compound 5: 1-(2-Benzyloxycarbonylamino-3-methyl-1-oxo-but-1-yl)-5-methyl-1H-[1, 2, 3] triazole-4-carboxylic acid ethyl ester

IR (cm^{-1}): 3329(NH), 2158(CN), 1678(CO).

1H NMR (δ ppm): 1.11 (d, 6H, 2 x CH3), 1.4 (t, 3H, CH3), 2.32 (s, 3H, CH3), 2.61 (m, 1H, CH), 3.7 (q, 2H, OCH2), 4.41 (d, 1H, CH), 5.35 (s, 2H, OCH2), 6.23 (s, 1H, NH), 7.21- 7.45 (m, 5H, Ar-H)

13C NMR (δ ppm): 13.31(CH3), 16.61(2 x CH3), 28.31(CH), 44.62(CH3), 58.12(CH), 59.62(CH2), 69.61(CH2), 128.3-141.5(ArC, C=C), 163.71(C=O), 175.41(C=O), 179.21(C=O).

Compound 6: 1(2-Benzyloxycarbonylamino-3-methyl-1-oxobut-1-yl)5-oxo-4, 5-dihydro-1H-[1, 2, 3] triazole-4-carboxylic acid ethyl ester

IR (cm^{-1}): 3323(NH), 2148(CN), 1682(CON).

1H NMR (δ ppm): 1.23 (s, 6H, 2 x CH3), 1.41(t, 3H, CH3), 2.62 (m, 1H, CH), 3.51(q, 2H, OCH2), 4.2 (s, 1H, CH), 4.41 (d, 1H, CH), 5.46 (s, 2H, OCH2), 6.12 (s, 1H, NH), 7.18- 7.28 (m, 5H, Ar-H)

13C NMR (δ ppm): 13.51(CH3), 17.5 (2 x CH3), 29.92 (CH), 61.4(CH), 63.6(CH), 70.2(CH2), 75.2(CH2), 128.3-140.2(ArC, C=C), 162.52(C=O), 172.5(C=O), 176.5(C=O), 179.33(C=O).

III. CONCLUSION

Triazole derivatives were prepared in excellent yield by using transition metal-based Nano-particles. The Co- Ferrite nano catalyst used was found to be effective to increase the reaction rate and atom economy. The nanoparticles showing magnetic behaviour facilitate easy separation, recycling and promoting valuable organic synthesis for the researcher working in this area.

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