(IJRST) 2020, Vol. No. 10, Issue No. III, Jul-Sep e-ISSN: 2249-0604, p-ISSN: 2454-180X

Development of Substituted 4-Methyl-5-nitro-2-(1-phenyl-1H-[1, 2, 3] Triazol-4-yl Methoxy) Pyridine Derivatives via Synthetic Approaches and its Characterization[1]

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DOI: 10.37648/ijrst.v10i03.009

Received: 14th July, 2020; Accepted: 22nd August, 2020; Published: 19th September, 2020

INTRODUCTION

The **1,2,3-triazole** ring is one of the most significant nitrogen-containing five-membered heterocyclic motifs due to its broad applicability in medicinal chemistry, chemical biology, organic synthesis, supramolecular chemistry, and industrial applications [1-2]. Numerous clinically important pharmaceuticals contain a **1,2,3-triazole** core, highlighting its significance in medicinal chemistry. Notable examples include the anticonvulsant drug **Rufinamide**, the broad-spectrum cephalosporin antibiotic **Cefatrizine**, the anticancer agent **Carboxyamidotriazole**, and the β-lactamase inhibitor **Tazobactam**, among others [3].The triazole ring system is present in a number of physiologically active natural compounds as well as pharmaceuticals [4], and it possesses capabilities that are anti-inflammatory [5], antimicrobial [6], anticancer [7], and antiviral [8]. In recent years, a great number of combinatorial approaches have been published [9]. Studies on the cycloaddition of different azides with various alkynes [10] have proliferated over the course of the past several decades, with a focus on "regio-control on terminal substrates in the Cu-catalysed vs uncatalyzed reaction".

We sought to explore the applicability of this **click reaction** for the synthesis of triazole derivatives. Upon photolysis, alkyl-substituted azo compounds readily eliminate molecular nitrogen (N_2), generating highly reactive intermediates that can participate in subsequent transformations [11]. There have been reports in the published research of examples of this reaction taking place in both thermal [12] and photochemical [13] condition. essential roles in heterocyclic chemistry as well as in medicinal applications. Pyridine-based ring systems constitute an important class of pharmacophores and have significantly contributed to the discovery and development of numerous therapeutic agents. Owing to their diverse biological activities and favorable physicochemical properties, pyridine derivatives are extensively employed in drug discovery. Furthermore, the pyridine scaffold readily accommodates a variety of substituents at different positions, enabling the design and optimization of clinical candidates with enhanced pharmacokinetic properties and improved biological potency.

a review of the literature shows that several pyridine analogues were effective against a variety of microorganisms, but the two main obstacles facing the researchers are drug resistance and deadly side effects. Pyridine derivatives' remarkable therapeutic effects frequently motivate efforts to be focused toward further study.

[1] *How to cite the article:* Goud G.S., Rumandla L, Development of Substituted 4-Methyl-5-nitro-2-(1-phenyl-1H-[1, 2, 3] Triazol-4-yl Methoxy) Pyridine Derivatives via Synthetic Approaches and its Characterization, IJRST, Jul-Sep 2020, Vol 10, Issue 3, 62-71, DOI: http://doi.org/10.37648/ijrst.v10i03.009

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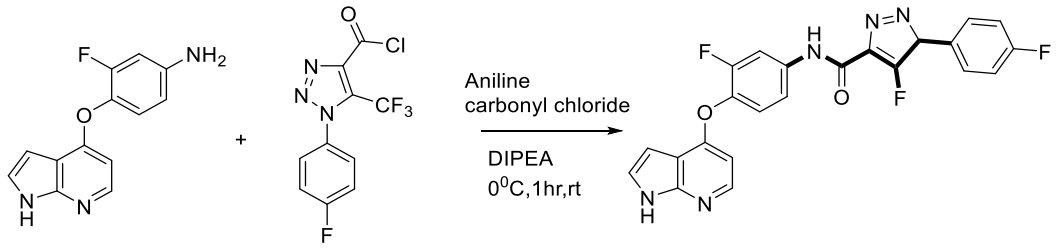
A single molecule that combines the characteristics of multiple physiologically active segments is predicted to exhibit significant pharmacological activity while maintaining a high degree of biological relevance and diversity.Owing to the above fact, we tried our best to synthesized 4-Methyl-5-nitro-2-(1-phenyl-1H-[1,2,3]triazol-4-ylmethoxy)-pyridine derivatives.

PREVIOUS APPROACHES:

A concise analysis of the procedures that were carried out by the synthesis of 1,2,3-triazol linked can be found on the following.

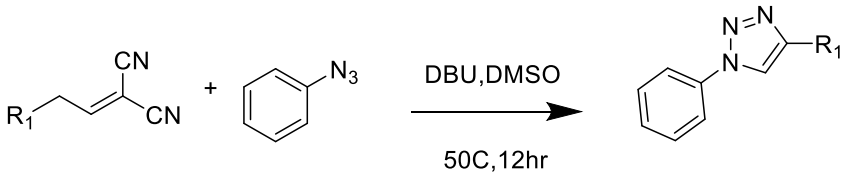
Qidong Tang et. al[14]The c-Met kinase inhibitory and antiproliferative properties of a series of new pyrrolo [2,3-b]pyridine derivatives carrying 1,2,3-triazole moiety were developed, synthesized, and assessed against four cancer cell lines (HT29, A-549, MCF7, and PC3) in vitro.

Among the compounds, most exhibited potencies ranging from moderate to excellent; the most promising compound, had a c-Met IC-50 value of 1.67 nM. Studies of the structure-activity connection revealed that in order to increase the inhibitory activity, electron-withdrawing groups (X = CF3, R1 = F, and R2 = 4-F) had to appropriately reduce the greater electron density on the 5-atom linker.(**Scheme-1)**



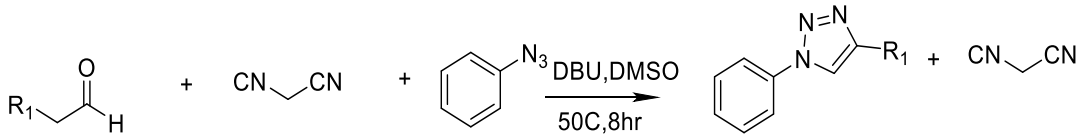
(**Scheme-1)**

Akbar Ali et.al [15]have shown effective 1,3-dipolar cyclo addition between aryl azides and alkylidene malononitrile in the presence of base for the synthesis of 1,4-disubstituted-1,2,3-triazoles.(**Scheme-2)**



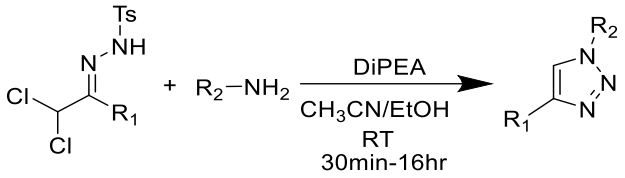
Scheme-2

They also developed a different method for producing 1,4-disubstituted 1,2,3-triazoles under standard circumstances utilizing aldehydes, malanonitrile, and aryl azides.(**Scheme-3)**



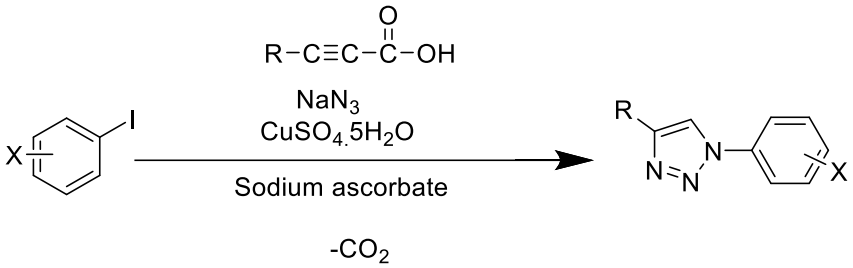
Scheme-3

Sander S et al [16]have revealed the synthesis of triazoles based on tosyl hydrazone that leave no trace. The compounds were made by combining functional, -dichlorotosyl hydrazones with primary amines in an ambient solvent mixture of 1:1 acetonitrile and ethanol, along with N,N-diisopropylethylamine (DiPEA).(**Scheme-4)**



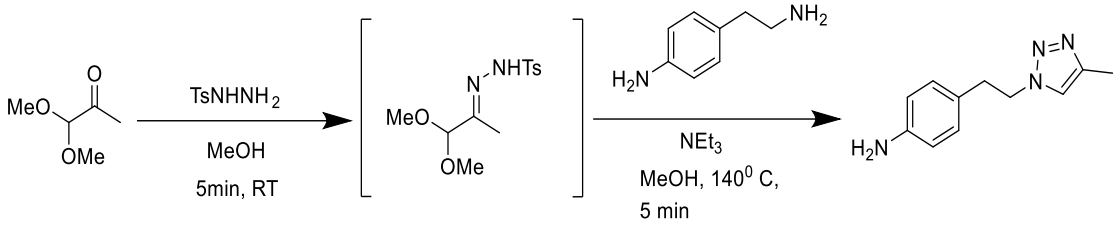
Scheme-4

Andrej et al [17]have described the three-step, one-pot synthesis of 1,4-disubstituted 1,2,3-triazoles using the decarboxylation of alkynoic acids, the 1,3-dipolar cycloaddition of azides, and terminal alkynes.(**Scheme-5)**

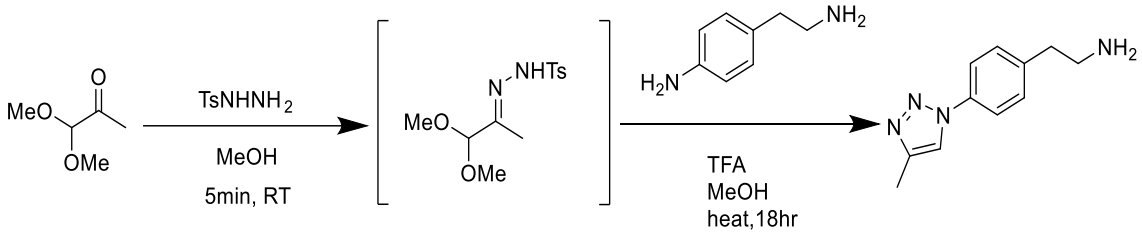


Scheme-5

Peter R. Clark et al [18]have presented a fantastic three-component coupling of -Ketoacetals, tosyl hydrazide, and a primary amine for the synthesis of 1,4-disubstituted 1,2,3-triazoles that is metal, azide, and halogen free. They have described techniques for chemo selective reactions of primary anilines and aliphatic amines that involve only a small modification of the reaction conditions.(**Scheme-6 and Scheme-7)**

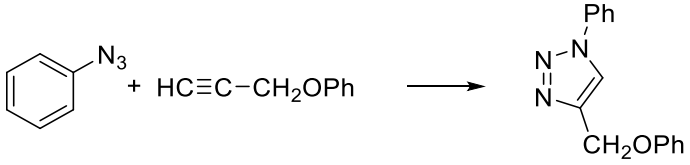


Scheme-6



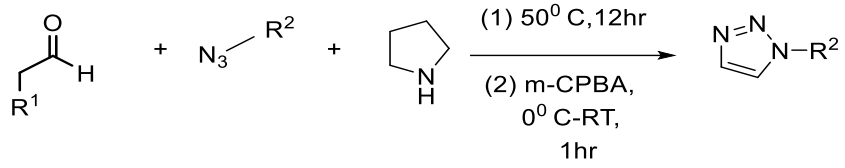
Scheme-7

Ototiko Tsuge et al [19]have described the 1,3-dipolar cycloaddition of phenyl azide and phenyl propargyl ether to produce 1-phenyl-4-phenoxymethyl-1,2,3-triazole. (**Scheme-8)**



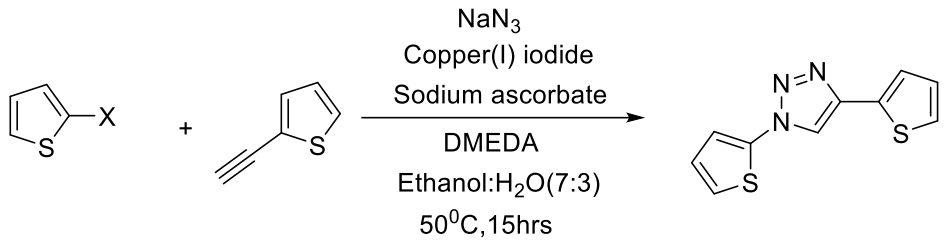
Scheme-8

Qianfa Jia et al [20]have created an effective one-pot metal-free synthesis of 1,4-disubstituted 1,2,3 - triazoles using the 1,3-dipolar cycloaddition of readily available commercial aldehydes with azides and secondary amines. (**Scheme-9)**



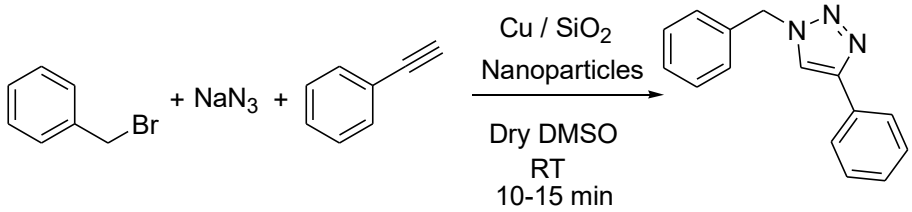
(Scheme-9)

Stefanie Potratz et al [21]have described a one-pot, three-component method for creating new thiophene-1,2,3-triazole co-oligomers. Here, the result of the cycloaddition of terminally ethylated thiophenes and halogenated thiophenes in the presence of copper(I) iodide, sodium azide, sodium ascorbate, N, NI-dimethylethylenediamine (DMEDA), and ethanol in a 7:3 water to ethanol ratio was produced. (**Scheme-10)**



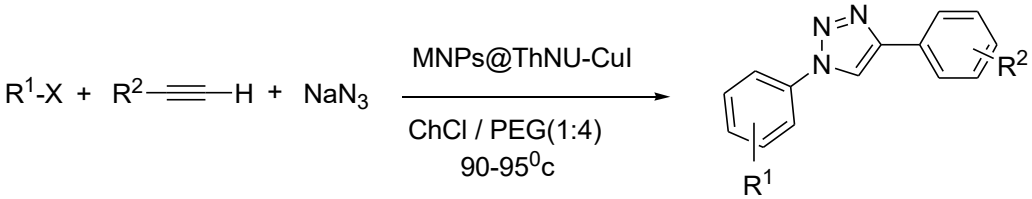
Scheme-10

Pitchaimani Veerakumar et al [22]have created a method for synthesizing 1,3 disubstituted 1,2,3-triazoles utilizing catalysts made of copper nanoparticles. Here, copper nanoparticles were used as a catalyst in the reaction of benzyl bromide with sodium azide and phenyl acetylene in DMSO medium at room temperature for 10–15 minutes to create the derivatives.(**Scheme-11)**



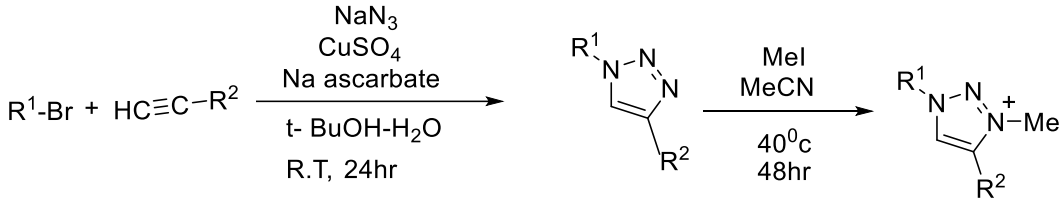
Scheme-11

Mirshafiee S et al [23] have described the one-pot, three-component synthesis of 1,4-disubstituted 1,2,3 triazoles using sodium azide, aryl halides, and terminal alkynes with MNPs@ThNU-CuInanocatalyst in choline chloride/PEG 200 (1:1) DES at 90-95⁰C.(**Scheme-12)**



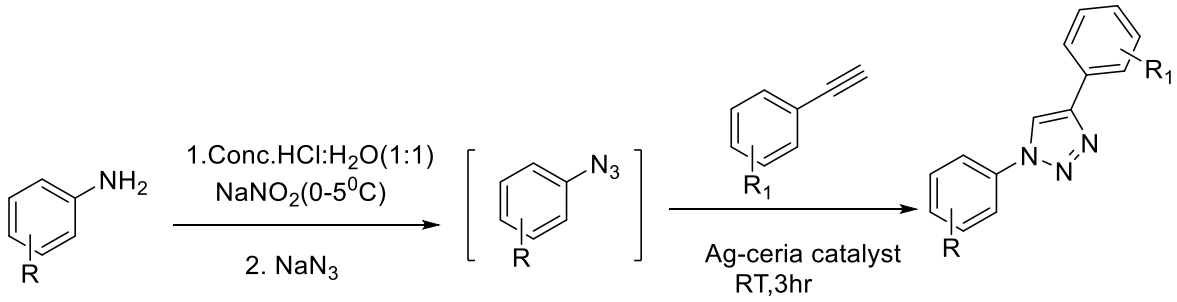
Scheme-12

J.T. Fletcher et al [24]have published a method for synthesizing 1,4-disubstituted 1,2,3-triazoles that involves a one-pot, two-step click transformation. Here, the derivatives were created by allyl or benzyl halides reacting with sodium azide to create their respective organic azides, which were then added to an alkyne in a copper-catalyzedHuisgen 1,3 - dipolar cycloaddition with methyl iodide and methyl cyanide at400 °C for 48 hours. (**Scheme-13)**



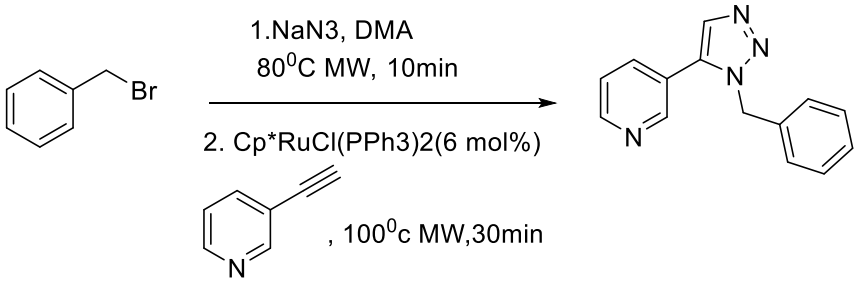
Scheme-13

Subhasis Das et al [25]have documented the use of the click reaction to produce 1,4-disubstituted 1,2,3-triazoles. Here, CeO2-Ag nano catalyst was used to catalysed the reaction between aromatic amines and phenyl acetylene to produce the products (**Scheme-14)**



Scheme-14

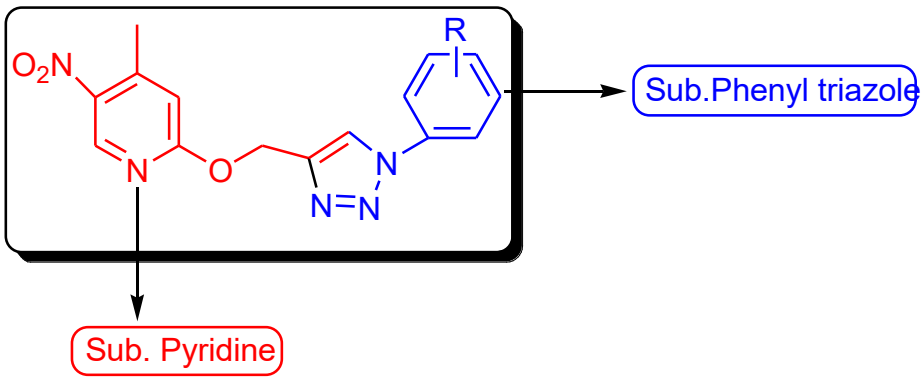
Johansson et al.[26]reported a sequential microwave-assisted one-pot process to directly manufacture triazoles from alkyl halides, which are more readily accessible than alkyl azides.While direct one-pot techniques for CuAAC have been documented,[27] when benzyl bromide, sodium azide, 3-ethynylpyridine, and Cp*RuCl(PPh3)2 in DMA were heated in a microwave for 30 minutes at 100 °C, only traces of product were produced. The most likely causes in this case are competing reactions or catalyst deactivation.[30] But by heating the alkyl halide and sodium azide together for ten minutes first, followed by the addition of the catalyst and the alkyne **(Scheme 15)**



Scheme-15

PRESENT WORK

To enhance the antibacterial capabilities of a class of hybrid compounds with various heterocyclic scaffolds, such as pyridine and triazoles. We produced them taking into account the biological profile of pyridine and triazole derivatives as part of our ongoing work on the synthesis of new pyridine and triazole molecules. In the current work, we synthesized novel pyridine-Triazole **37 (a-l)** and evaluated its inhibitory activities of the synthesized compounds



In **Scheme-20**, the synthesized triazole derivatives were shown. By nitrating 4-Methyl-pyridin-2-amine **31** with a nitration combination (HNO_3+H_2SO_4) under cooling conditions of 0-5^{0}C for 3–5 hours, the initial product **32** was produced in good yield. The Synthesized 4-Methyl-5-nitro-pyridin-2-amine **32**, reacted with $NaNO_3$, H_2SO_4, at 0-5^{0}C to form 4-Methyl-5-nitro-pyridin-2-ol **33**, this compound having active -OH functional group now this compound treated with 3-Bromo-propyne **34** in the presence of DMF and K_2CO_3 in RT condition 5-6 hrs appeared compound 4-Methyl-5-nitro-2-(prop-2-yn-1-yloxy)-pyridine **35.**

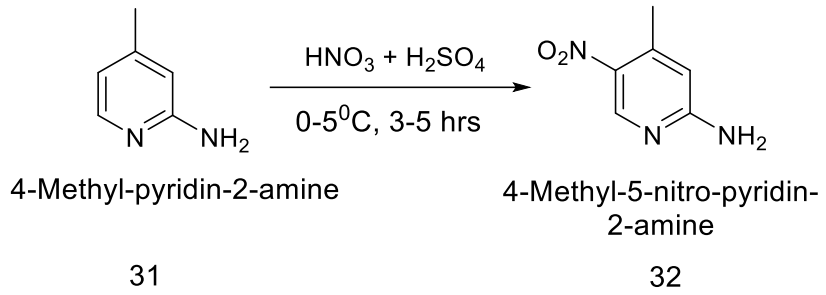
The newly synthesized compound **35** structure was verified using spectroscopic methods such as ^{1}H NMR, ^{13}C-NMR, Mass, and IR. Emergence of the target in a click reaction setting, substituted 4-Methyl-5-nitro-2-((1-phenyl-1H-[1,2,3]triazol-4-yl)methoxy)-pyridine **37 (a-l)** was produced by reacting 4-Methyl-5-nitro-2-(prop-2-yn-1-yloxy)-pyridine**35** with various substituted aromatic azides **36 (a-l).**The final compound confirmed by ^{1}H NMR, ^{13}C NMR, IR and Mass spectral analysis. In ^{1}H-NMR the proton signals observed at δ9.28ppm (s) of triazole protons and methylene (-CH_2) protons gives signals at δ 5.39 ppm respectively, methyl protons give signal at δ 2.50 ppm, remaining all protons signals are appeared at their corresponding integral values. The structures of the synthesized compounds **37(a-l**) were confirmed on the basis of absence of their -OH proton signals, and the appearance of a new signal due to the triazole ring protons group appeared at *δ* values between 9.28–9.09 ppm in all the compounds.In addition to ^{1}H-NMR remaining spectral data like ^{13}C-NMR, IR and Mass also gives their respected integral values. The remaining stretching vibration of compounds **37 (a-l**) gave rise to a band observed at their corresponding values. The mass spectra of the compounds showed (M+1) peaks, and are in agreement with their molecular formula.

GENERAL EXPERIMENTAL METHODS

Merck was utilized straight out of the container without any additional purification, and TCI provided all of the chemicals, including the organic reagents and solvents. Using spectrometers set to run at 500 and 125 MHz (Bruker Avance II 400 MHz device), 1H and 13C NMR spectra were acquired in CDCl3. Singlet (s), doublet (d), doublet of doublet (dd), triplet (t), and multiplets (m) are the spin multiplicities. The coupling constant values are displayed in hertz. Parts per million (ppm) is the unit of measurement for chemical shift values. Distilled hexane and ethyl acetate were used as solvents in the chromatography process, which used a silica gel column with a mesh size range of 60–120. The QSTAR XL GCMS and the Shimadzu FT-IR-8400s mass spectrometer were used to record the mass and infrared spectra, respectively. Melting points were measured in an exposed glass capillary tube using Dbk-Prog melting point equipment; the results were displayed without correction.

General procedure for Synthesis of 4-Methyl-5-nitro-pyridin-2-amine(32)

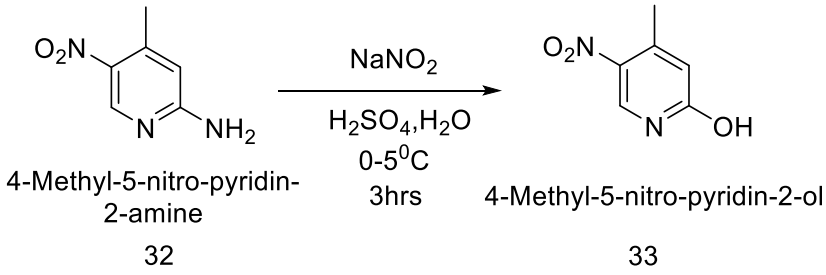
A commercially available chemical called 4-Methyl-pyridin-2-amine (0.98 mmol) is combined with a drop wise addition of nitration combination (HNO_3+H_2SO_4) in a round-bottom flask at 0–5^{0}C, the reaction mixture was agitated for two to three hours. After allowing the suspension to come to room temperature, it was diluted with EtOAc, water, and a celite bed filter. After being separated, the organic layer was cleaned with water, brine solution, and dried on Na_2SO_4. It was then filtered and concentrated. The residue was purified by flash chromatography on silica gel (100-200 mesh) using EtOAc: Pet-ether as eluant to afford pure compound **32**. yellow solid; Yield: 78%.



Scheme-16

General procedure for Synthesis of 4-Methyl-5-nitro-pyridin-2-ol (33)

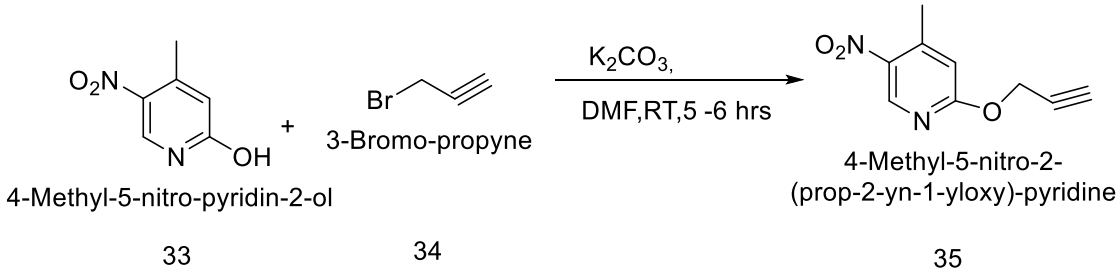
In a clean 200ml round bottom (RB) flask charged compound **32** and $NaNO_2$ in sulphuric acid at 0-5^{0}Cfor 3hrs. In this condition amino group converted hydroxy group. After that reaction mixture at room temperature work up finished in neutral condition, an off-white colour solid is appeared based on TLC (EtOAc + hexane 3:1 ratio) and ^{1}H-NMR spectra confirms the structure. Yield: 82%.



Scheme-17

General procedure for Synthesis of 4-Methyl-5-nitro-2-(prop-2-yn-1-yloxy)-pyridine (35):

In order to synthesize the 4-Methyl-5-nitro-2-(prop-2-yn-1-yloxy)-pyridine **(35)** compound, the starting material compound of 4-Methyl-5-nitro-pyridin-2-ol **(33)**, which was treated with propargyl bromide **(34)** in the presence of dry dimethyl formamide (Dry DMF) and dry potassium carbonate at room temperature for 5 to 6 hours. The Compound 4-Methyl-5-nitro-2-(prop-2-yn-1-yloxy)-pyridine **(35)** were identified by TLC in 30% Ethyl Acetate in hexane spot were observed at slight non polar region compare to starting material.

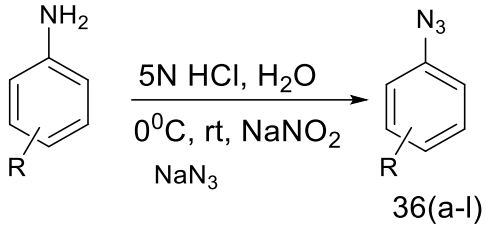


Scheme-18

Synthesis of substituted azido benzene (36 a-l):

The substituted azidobenzene**(36 a-l)** was made by adding 5N hydrochloric acid (HCl) solution to a mixture of corresponding amines in CH_2Cl_2 at 0°C, then gradually adding sodium nitrite solution ($NaNO_2$) while shaking at zero degrees Celsius for half an hour. After adding NaN_3 at zero degrees Celsius, the mixture was agitated for two hours

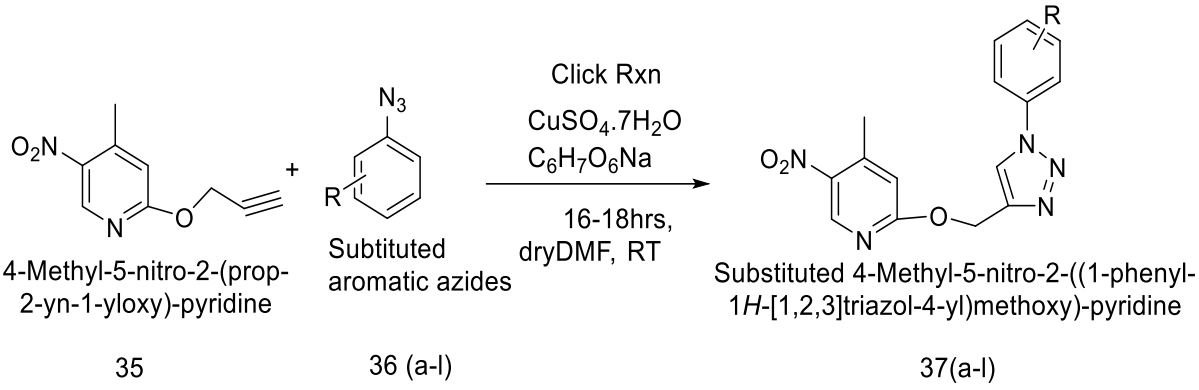
at the ambient temperature. After that, it was left alone to let the organic and aqueous layers separate. To get the needed aryl azides **(36 a-l)**, the organic layer was washed with $NaHCO_3$, then brine, and the solvent was evaporated in a vacuum. In the next step, these azides are used without any further purification.



Sheme-19

Experimental Procedure for the Synthesis of Substituted 4-Methyl-5-nitro-2-((1-phenyl-1H-[1,2,3]triazol-4-yl)methoxy)-pyridine 37(a-l):

Reaction in between 4-Methyl-5-nitro-2-(prop-2-yn-1-yloxy)-pyridine **(35)** and substituted azidobenzene **(36 a-l)** on treatment with $CuSO_4.7H_2O$ and $C_6H_7O_6Na$(Sodium ascorbate) in dry DMF at ambient temperature, (Click Condition) were converted to corresponding products Substituted 4-Methyl-5-nitro-2-((1-phenyl-1H-[1,2,3]-triazol-4-yl)methoxy)-pyridine **(37a-l)** in 80-86% Yields were obtained.Tetrazole causes absorption bands at 1533 cm-1, pyridine causes absorption bands at 1453 cm-1, and benzene causes absorption bands at 1503cm^{-1}. The ether causes these absorption bands. Its 1HNMR spectra showed that the methyl and methylene signlets occurred at 2.45 and 5.39 ppm, respectively; the triazole peak was at 8.82 ppm, and the pyridine N-CH peak was at 9.28 ppm, appearing as a singlet. Pyridine carbon was found at 159.91 ppm in its ^{13}CNMR, Tetrazole carbon at 154.80 ppm, and aromatic carbon which is linked to triazole at 136.46 ppm.



Scheme-20

If R=a=H,

EXPERIMENTAL-TABLE-1:

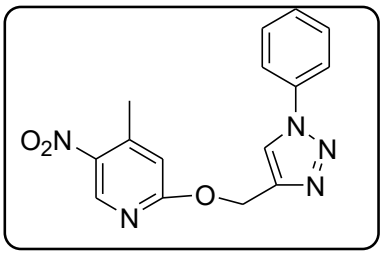
S.No	Compound	M.P. (^{O}C)	M.F. (M.Wt.)	Time(h)	Yield (%)
1	4-methyl-5-nitro-2-((1-phenyl-1H-1,2,3-triazol-4-yl)methoxy)pyridine (**37a**)	142-144	Mol. Formula: $C_{15}H_{13}N_5O_3$ (311)	8.5	83

CONCLUSION

In conclusion, we have synthesized a novel derivatives of 4-methyl-5-nitro-2-((1-phenyl-1H-1,2,3-triazol-4-yl)methoxy)pyridine **(37a–l)** and characterized by IR, ^{13}C NMR, ESI-MS, and ^{1}H NMR.

SPECTRAL DATA

Analytical data of 4-methyl-5-nitro-2-((1-phenyl-1H-1,2,3-triazol-4-yl)methoxy)pyridine (37a)



Colour	Pale yellow solid
Yield	83%
Mol. Formula	$C_{15}H_{13}N_5O_3$
M.P	142-144°C
IRData ν, cm^{-1}(KBr)	3380, 3160, 2929, 1657, 1603, 1294, 1039, 878, 754
^{1}H NMR-400 MHz (DMSO-d6)	9.28 (S, 1H), 8.82 (S, 1H), 7.89 (d, 2H, d=7.2Hz), 7.59 (t, 2H, J=7.6Hz), 7.51 (t, 1H, J=6.2Hz), 6.44 (s, 1H), 5.39 (S, 2H), 2.45 (s, 3H).
^{13}C NMR	159.91, 145.80, 142.95, 142.42, 136.46, 131.76, 129.93, 128.83, 122.12, 120.10, 119.07, 20.72
ESI-MS: m/z	311
Elemental Analysis	CAL: C, 77.87; H, 4.21; N, 22.50; O, 15.42 EXP: C, 74.50; H, 4.32; N,14.52; O, 8.64

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