J. Chemtracks 27 (1&2) 1-8, 2025

Abstracted / Indexed in
A division of the American Chemical Society
Chemical Abstract U.S.A.

ISSN 0973-239X
CODEN:JCOHF7
IMPACT FACTOR : 7.866

One Pot Protocol for the Synthesis of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole carbonitrile derivatives

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Manuscript received online 21 November 2024, accepted on 06 January 2025

Abstract : A series of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives was successfully synthesized through a one-pot, three-component reaction involving aromatic aldehydes, malononitrile, and 3-methyl-1-phenyl-2-pyrazolin-5-one. The reaction utilized NaOH: fly ash as a cost-effective and environmentally friendly catalyst in an aqueous medium. This approach demonstrated excellent catalytic efficiency, yielding products of high purity and an excellent yield. Key advantages of this methodology include a reduced environmental impact, low catalyst cost, and simple workup, making it a sustainable and practical synthetic strategy.

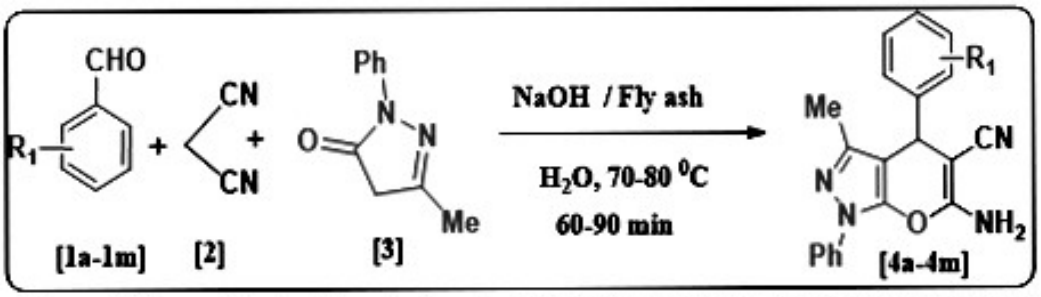
(Keywords : NaOH: fly ash, cost effective, malononitrile, one-pot, eco-friendly, catalysis)

Introduction

Multicomponent reactions (MCRs) have revolutionized modern synthetic organic chemistry, emerging as one of the field's most versatile and efficient tools. These reactions represent the principles of ideal synthesis through their rapid and straightforward procedures, exceptional atom economy, energy efficiency, and environmentally friendly nature[1-2]. Consequently, the innovation and advancement of Multicomponent reactions (MCRs) strategies for producing biomedical and industrial scaffolds have become a priority in modern research. In this context, the choice of catalyst plays a pivotal role. Among various options, fly ash, a byproduct of industrial processes and a significant environmental pollutant[3-4] has demonstrated remarkable efficacy in driving reactions with considerable industrial and pharmacological relevance. The application of fly ash highlights their catalytic strength and contributes to the development of cost effective and sustainable chemical processes, representing a promising opportunity for the chemical industry. These derivatives, which arise from multi-component reactions (MCRs), are important building blocks for a variety of biologically active compounds. They exhibit diverse pharmaceutical properties, including pharmaceutical properties such as fungicidal[5], anti-inflammatory[6], antimicrobial[7], anticancer[8], analgesic[9], and antibacterial activities[10], processing their immense biological and pharmaceutical significance, the synthesis of pyranopyrazole-containing heterocyclic compounds has attracted considerable attention.

Various synthetic approaches have been developed, employing catalysts for the synthesis of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives such as tungstate sulfuric acid[11], SnS nanoparticles[12], morpholinium glycolate[13], and cesium fluoride[14]. have also been explored for these syntheses. Notably, some other methodologies are employed as one-pot, three-component condensations involving malononitrile, aldehydes, and 3-methyl-1-phenyl-2-pyrazolin-5-one, using catalysts such as TEBAC[15], hexadecyltrimethylammonium bromide[16], DL-proline[17], p-dodecylbenzene sulfonic acid[18], and sodium fluoride[19]. Recently, focus has been a shift towards utilizing industrial waste materials, particularly fly ash, for value-

added applications. Fly ash, an abundant and low-cost resource, has demonstrated significant potential as a reusable catalyst for various chemical transformations[20-21]. While its catalytic properties have been studied extensively[22-23]. its broader application in sustainable chemistry is still an active area of exploration. In response to this, we present a highly efficient and environmentally friendly method for synthesizing 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives. This approach utilizes NaOH: fly ash as catalysts in an aqueous medium, offering a sustainable alternative for the synthesis of these valuable heterocycles. **(Scheme 1).**



Scheme 1. The synthesis of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole carbonitrile derivatives

Materials and Methods

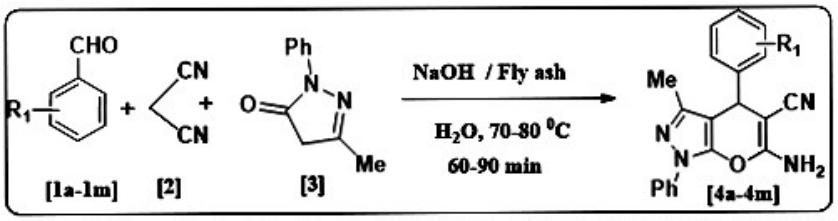
Fly ash was obtained from a local sugar factory in Faizabad, Uttar Pradesh, India. All chemicals and reagents were procured from Sigma-Aldrich and TCI Pvt. Ltd. Melting points are uncorrected and are determined in open capillary tubes. IR measurements were carried out using KBr pellets on FTIR spectrometer. ^{1}H and $^{13}CNMR$ spectra were on Bruker spectrometers. All known compounds were identified by comparison of their melting points and spectroscopic data with those reported in the literature.

Preparation of catalyst NaOH/Fly ash

The NaOH/fly ash catalyst was prepared according to the reported procedure [24]. Fly ash (15 g) was thoroughly mixed with NaOH (3 g), and the resulting mixture was fused in air and dried in an electric oven as mentioned in reference. The fused product was then calcined at 600°C for 120 minutes in a high-temperature muffle furnace under an air atmosphere. The prepared catalyst was subsequently used in further reactions.

General Procedure for the Synthesis of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives.

A mixture of different aromatic aldehydes (1 mmol), malononitrile (1 mmol), 3-methyl-1-phenyl-2-pyrazolin-5-one (1 mmol), and 0.5 gm of NaOH: fly-ash (5%weight with respect to the reactants) catalyst was added to 25 mL of water. The reaction mixture was heated at 70-80°C for 60-90 minutes with progress monitored by thin-layer chromatography (TLC). Upon completion, the reaction mixture was cooled, and the resulting residue was dissolved in ethanol. The catalyst, being insoluble, was separated by filtration from the ethanolic solution and subsequently recovered for reuse. The filtrate was evaporated under reduced pressure to yield the crude product, which was purified by recrystallization from aqueous ethanol to obtain the pure derivatives (**Scheme 1)**.



Scheme 2. The synthesis of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole carbonitrile derivatives

Table 1.

The synthesis of model compounds catalysed by NaOH: fly-ash in water[a].

Entry	Aldehyde	Compound[d]	Time (min)	Yield[b] (%)	mp (°C)[c]
1.	H-	6a	60	88	169-170
2.	4-chloro	6b	65	84	176-177
3.	4-hydroxy	6c	70	84	211-212
4.	4-fluoro	6d	65	80	166-168
5.	4-nitro	6e	60	83	194-196
6.	4-methyl	6f	60	90	160−162
7.	4-methoxy	6g	70	89	172-174
8.	4-cyano	6h	90	84	197–199
9.	2,6-dichloro	6i	85	89	179–181
10.	3-nitro	6j	75	85	191-193
11.	3,4-dimethoxy	6k	90	88	190–192
12.	2,4-dihydroxy	6l	58	80	320–321
13	5-bromo-2hydroxy	6m	68	83	313–315

Table 1. [a]Reaction condition, aromatic aldehydes (1 mmol), malononitrile (1 mmol), 3- Methyl-1-phenyl-2-pyrazolin-5-one (1 mmol) and the catalyst, NaOH: fly-ash (5%weight with respect to the reactants) in water (25mL) was heated at 70-80^{0}C temperature [b]Isolated yield., [c]All the melting point are uncorrected compared with reference[25-32] [d]All the compounds were characterized by IR, ^{1}H and ^{13}C NMR and were compared with the reference[25-32]

Results and Discussions

Based on research findings regarding the effectiveness of catalysts in facilitating various synthetic organic reactions, we hypothesized that this particular reagent could also effectively promote the synthesis of a series of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives. To test our hypothesis, we conducted a three-component reaction involving 3-methyl-1-phenyl-2-pyrazolin-5-one (1 mmol), malononitrile (1 mmol), and various aromatic aldehydes (1 mmol). The reaction was carried out in the presence of NaOH: fly ash (at 5% weight relative to the reactants) as the catalyst under aqueous conditions **(Scheme 1).** Choosing water as the solvent was a key step for the model reactions. We were pleased to observe promising results when the model reaction was performed in water at 70-80°C, yielding the desired product with a yield of 90%. Additionally, we found that the efficiency of the three-component reaction was influenced by the catalyst loading and reaction time. To optimize our results, we varied the amounts of NaOH: fly

ash used in the condensation of benzaldehyde, malononitrile, and 3-methyl-1-phenyl-2-pyrazolin-5-one. Our investigation determined that the optimal catalyst loading is 5% weight relative to the reactants. All synthesized compounds (4a–4m) were characterized using spectral analysis, and their melting points were consistent with those reported in the literature.[25-32]

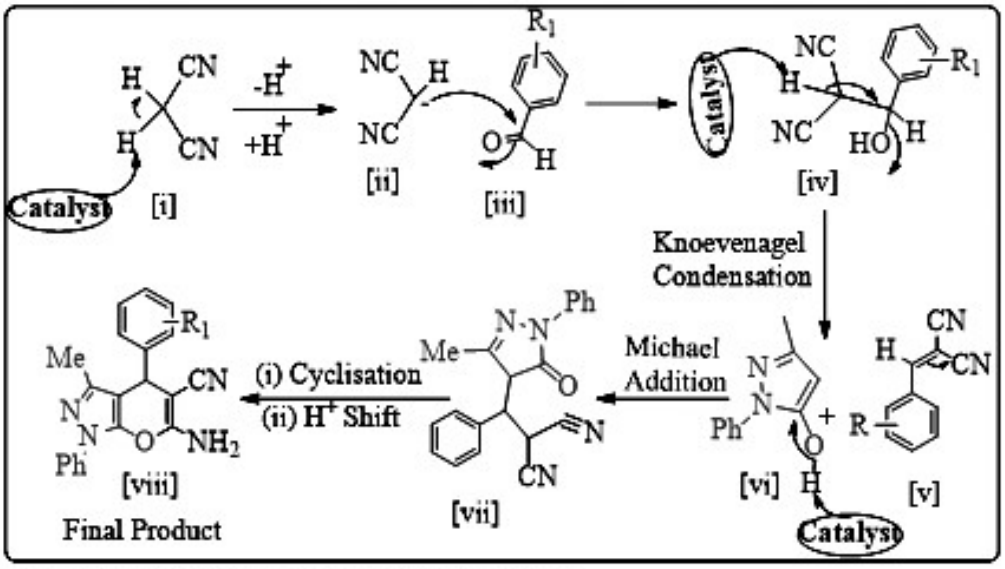


Figure 1. mechanism for the proposed synthesis

Proposed Reaction Mechanism for model synthesis: -

The proposed mechanism for the synthesis of compounds (4a–4m) is illustrated in **Figure 1**. Initially, malononitrile [i] reacts with the NaOH: fly ash catalyst, resulting in the formation of an activated intermediate anion [ii] through deprotonation and tautomerization. This intermediate then undergoes a nucleophilic attack on the carbonyl group of the aldehydes [iii], leading to the creation of a hydroxyl intermediate [iv]. Subsequently, the hydroxyl intermediate undergoes dehydration in the presence of the catalyst, resulting in the formation of a Knoevenagel condensation product [v]. The condensation product [v] then reacts with 3-methyl-1-phenyl-2-pyrazolin-5-one [vi] via a Michael addition mechanism, forming a new intermediate [vii]. This Michael addition product [vii] undergoes intramolecular cyclization, which results in the formation of a six-membered pyran ring. Finally, the cyclic intermediate tautomerizes to form the final product, 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives. To assess the advantages of the proposed protocol for synthesizing these derivatives, the efficiency and reaction conditions were compared with those reported in the literature.

Spectral Data of Representative Compounds[25-32]: -

6-Amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile[4a]

Orange crystals, mp: 169-170 °C. IR (KBr, cm^{-1}): 3415, 3320, 3105, 2180, 1655, 1595, 1260, 1060. ^1H NMR (400 MHz, DMSO-d_6): δ 1.88 (s, 3H), 4.38 (s,

1H), 5.18 (br s, 2H), 7.3-7.41 (m, 5H), 7.63-7.86 (m, 5H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 12.1, 31.3, 65.8, 104.7, 114.5, 119.8, 125.8, 128.2, 129.9, 130.2, 133.8, 138.6, 142.8, 144.2, 149.8, 152.1

6-Amino-3-methyl-4-(4-chlorophenyl)-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile[4b] White crystals, mp: 176-177 °C. IR (KBr, cm^{-1}): 3455, 3345, 3120, 2195, 1620, 1585, 1170, 760, ^{1}H NMR (500 MHz, DMSO-d_6): δ 2.20 (s, 3H), 4.79 (d, *J* = 11.0 Hz, 1H), 7.24 (m, 2H), 7.45 (m, 4H), 7.62-7.72 (m, 3H), 11.68-11.70 (s, 2H), ^{13}C NMR (125 MHz, DMSO): ´ 12.62, 34.25, 57.18, 101.48, 119.15, 120.58, 124.95, 128.08, 129.25, 129.78, 131.94, 134.08, 137.25, 138.94, 144.33, 146.12, 159.40.

6-Amino-4-(4-hydroxyphenyl)-3-methyl-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile[4c] Yellow crystals, mp: 211-212 °C. IR (KBr, cm^{-1}): 3455, 3320, 2205, 1610, 1585, 1295, 1035. ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.78 (s, 3H), 4.65 (s, 1H), 6.50 (br s, 2H), 6.85 (d, 2H, *J* = 7.8 Hz), 6.92 (d, 2H, *J* = 7.7 Hz), 7.44-7.55 (m, 2H), 7.75-7.94 (m, 4H), 8.17 (br s, 1H).^{13}C NMR (100 MHz, DMSO-d_6): ´ 13.8, 33.4, 72.5, 108.3, 113.6, 121.3, 127.6, 129.4, 132.2, 136.8, 138.3, 141.6, 145.5, 148.2, 150.4, 150.7.

6-Amino-4-(4-fluorophenyl)-3-methyl-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile[4d] Light yellow crystals, mp: 166-168 °C. IR (KBr, cm^{-1}): 3460, 3315, 3060, 2210, 1600, 1580, 1235, 1125. ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.74 (s, 3H), 4.82 (s, 1H), 4.93 (br s, 2H), 7.32-7.45 (m, 3H), 7.48-7.58 (m, 2H), 7.78 (d, 2H, *J* = 8.4 Hz), 7.90 (d, 2H, *J* = 8.5 Hz). ^{13}C NMR (100 MHz, DMSO-d_6): δ 13.1, 37.3, 57.8, 111.6, 118.7, 120.9, 121.2, 128.4, 129.7, 130.3, 133.5, 138.4, 145.5, 149.2, 156.2, 161.2.

6-Amino-4-(4-nitrophenyl)-3-methyl-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile,[4e] Yellow crystals mp: 194-196 °C. IR (KBr, cm^{-1}): 3445, 3300, 3035, 2200, 1640, 1580, 1145. ^{1}H NMR (400 MHz, DMSO-d_6): ´ 1.75 (s, 3H), 4.74 (s, 1H), 5.15 (br s, 2H), 7.45-7.55 (m, 3H), 7.63-7.77 (m, 2H), 7.80 (d, 2H, *J* = 8.2 Hz), 7.86 (d, 2H, *J* = 8.2 Hz). ^{13}C NMR (100 MHz, DMSO-d_6): δ 13.4, 37.6, 68.2, 110.9, 119.7, 119.8, 120.8, 129.8, 129.7, 130.6, 133.5, 138.7, 145.7, 149.5, 153.1, 159.3.

6-Amino-3-methyl-1-phenyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile[4f] Yellow crystals, mp: 160-162 °C. IR (KBr, cm^{-1}): 3445, 3321, 2187, 1645, 1589, 1220. ^{1}H NMR (250 MHz, DMSO-d_6): δ2.16-2.18 (s, 3H), 2.38–2.40 (s, 3H), 4.65-4.70 (d, *J* = 10.8 Hz, 1H), 7.05-7.15 (m, 3H), 7.40-7.50 (t, *J* = 8.2 Hz, 4H), 7.65-7.70 (d, *J* = 7.8 Hz, 2H), 11.50-11.60 (s, 2H). ^{13}C NMR (62.5 MHz, DMSO): δ 20.50, 26.22, 41.18, 57.52, 102.28, 114.15, 118.72, 120.40, 124.96, 127.53, 128.96, 129.25, 130.68, 136.26, 137.14, 147.80, 161.25.

6-Amino-4-(4-methoxyphenyl)-3-methyl-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile[4g]
Yellow crystals, mp: 172-174 °C. IR (KBr, cm^{-1}): 3465, 3355, 3160, 2215, 1625, 1595, 1295, 1035. ^{1}H NMR (400 MHz, DMSO-d_6): ´ 1.74 (s, 3H), 3.65 (s, 3H), 4.47 (s, 1H), 4.72 (br s, 2H), 6.85 (d, 2H, *J* = 7.4 Hz), 6.94 (d, 2H, *J* = 7.4 Hz), 7.35–7.55 (m, 2H), 7.70-7.95 (m, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ12.5, 35.9, 55.7, 77.9, 102.5, 113.7, 118.5, 121.6, 127.4, 129.2, 132.4, 136.7, 139.2, 140.4, 146.7, 149.2, 151.2.

6-Amino-4-(4-cyanophenyl)-3-methyl-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile,[4h]
White solid, mp: 197-199 °C. IR (KBr, cm^{-1}): 3405, 3300, 2235, 2180, 1640, 1490, 1260, 1025, 755. ^{1}H NMR (250 MHz, $CDCl_3$): δ 1.87 (s, 3H, CH_3), 4.75 (s, 1H, C-H), 4.78 (s, 2H, NH,), 7.26-7.70 (m, 9H, ArH). ^{13}C NMR (62.5 MHz, $CDCl_3$): δ 12.8, 37.6, 62.7, 97.3, 111.9, 118.6, 121.4, 127.7, 128.5, 128.7, 129.5, 131.8, 132.4, 132.7, 146.2, 147.1, 158.5.

6-Amino-4-(2,6-dichlorophenyl)-3-methyl-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile[4i]

White crystals, mp: 179-181 °C. IR (neat, cm^{-1}): 1385, 1555, 2190, 3325, 3455. ^{1}H NMR (300 MHz, DMSO-d_6): δ 1.76 (s, 3H), 5.13 (s, 1H), 7.23-7.56 (m, 7H), 7.62 (s, 1H), 7.76 (d, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ 12.38, 49.16, 49.24, 56.16, 97.34, 119.57, 120.05, 126.30, 128.12, 128.37, 128.99, 129.37, 132.51, 132.57, 133.08, 137.45, 139.29, 144.29, 144.86, 159.97.

6-Amino-3-methyl-4-(3-nitrophenyl)-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazol-5-carbonitrile, [4j]

Yellow solid, mp: 191-193 °C. IR (KBr, cm^{-1}): 3445, 3300, 3035, 2200, 1640, 1580, 1145.
^{1}H NMR (250 MHz, DMSO-d_6): δ 1.76 (s, 3H), 4.95 (s, 1H), 7.25-7.48 (m, 5H), 7.60-7.75 (m, 4H), 8.10-8.15 (s, 2H). ^{13}C NMR (62.5 MHz, DMSO): ′ 12.54, 36.18, 56.94, 97.68, 110.55, 119.72, 120.05, 122.23, 126.62, 129.31, 130.8, 134.75, 137.40, 143.94, 145.16, 145.90, 147.94, 159.75.

6-Amino-3-methyl-4-(3,4-dimethoxyphenyl)-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazol-5-carbonitrile [4k] Yellow crystalline solid, mp: 190-192 °C. IR (KBr, cm^{-1}): 3490, 3320, 3017, 2937, 2882, 2198, 1666, 1589, 1368, 1242, 1128, 882. ^{1}H NMR ($CDCl_3$): δ 1.92 (s, 3H, CH_3), 3.82 (s, 3H, -OCH_3), 3.81 (s, 3H, -OCH_3), 4.67 (s, 1H, ArCH), 6.33 (s, 2H, br., NH,), 6.80-6.90 (s, 1H, ArH), 6.72 (d, *J* = 8.28 Hz, 1H, ArH), 6.79 (d, *J* = 8.28 Hz, 1H, ArH), 7.29-7.37 (m, 5H, ArH). ^{13}C NMR (100 MHz, DMSO-d_6): δ 12.5, 35.9, 55.7, 77.90, 102.34, 113.77, 118.52, 121.66, 127.45, 129.29, 132.45, 136.72, 139.20, 140.14, 146.70, 149.25, 151.20.

6-Amino-3-methyl-4-(2,4-dihydroxyphenyl)-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazol-5-carbonitrile[4l] White crystals, mp: 320–321 °C. IR (KBr, cm^{-1}): 3424, 3309, 3188, 2923, 2854, 2211, 1590, 1514, 1410, 1351, 1252, 1189, 1045, 842, 800. ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.65-9.70 (s, 1H), 7.85-7.95 (s, 1H), 7.70-7.75 (s, 2H), 7.51-7.70 (m, 2H), 7.25-7.27 (m, 2H), 6.88–6.90 (s, 2H), 6.45 (d, 2H, *J* = 4 Hz), 6.33-6.38 (s, 1H), 5.50-6.0 (s, 1H), 1.80–1.85 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) : δ 166.65, 162.22, 161.98, 159.72, 155.85, 153.64, 138.32, 126.38, 123.14, 117.15, 114.33, 108.86, 107.09, 103.83, 101.34, 75.76, 42.11, 26.20.

6-Amino-3-methyl-4-(5-bromo-2-hydroxyphenyl)-1-phenyl-1,4-dihydropyrano[2,3-c]pyrazol-5-carbonitrile[4m] Yellow crystals, mp: 313–315 °C. IR (KBr, cm^{-1}): 3455, 3342, 3210, 2935, 2220, 1646, 1618, 1554, 1477, 1378, 1280, 1222, 1087, 802, 470. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.70 (s, 1H), 7.71 (d, 2H, *J* = 4 Hz), 7.52-7.54 (t, 2H, *J* = 4 Hz), 7.30-7.35 (s, 1H), 7.22-7.28 (m, 2H), 6.70-6.88 (m, 2H), 6.60-6.65 (d, 1H, *J* = 4 Hz), 5.40 (s, 1H), 2.04 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.94, 162.31, 158.20, 158.22, 151.67, 140.94, 137.25, 127.62, 121.35, 118.30, 118.04, 118.03, 117.18, 116.70, 97.66, 75.22, 47.56, 23.87.

Conclusion: -

In conclusion, we have successfully developed a novel and efficient method for synthesizing 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives. This method employs NaOH: fly ash (5% by weight relative to the reactants) as a catalyst, in aqueous medium. This approach is not only simple but also sustainable. Key advantages of this protocol include its novelty, high yields, straightforward workup process, non-toxicity, and environmentally friendly nature. As a result, it presents an attractive alternative for the organic synthesis of 6-amino-3-methyl-1,4-diphenyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives.

Conflict of Interest: -

The authors declare no conflicts of interest regarding this publication.

Acknowledgments: -

The authors thank CSIR for financial support (NET JRF, 09/001/(0409)/2017-EMR-1) and the University of Allahabad for instrumental facilities.

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An International Peer Reviewed Research Journal of Chemistry
Abstracted / Indexed in : Chemical Abstract U.S.A.
JC- 1606/2K25 (ISSN:0973-239X), INDIA
Journal Chemtracks Vol. 27 (1&2),1-8, January to December 2025