Synthesis and Characterization of 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile using traditional route

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Abstract: This research paper presents a comprehensive review of the synthesis and characterization of 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile. The paper discusses the detailed reaction mechanisms, optimization strategies, and characterization techniques involved in the synthesis of this compound. Furthermore, the physicochemical properties, biological activities, and potential applications of 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile are explored, highlighting its significance in drug discovery, medicinal chemistry, and materials science. The study aims to contribute to the existing knowledge base and inspire further research in the field of pyranopyrazole derivatives.

Keywords: 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c]pyrazole-5-Carbonitrile, synthesis, reaction mechanisms, physicochemical properties, materials science.

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Introduction

In the field of organic chemistry, the synthesis and characterization of novel compounds hold immense importance in the discovery of potential therapeutic agents, bioactive molecules, and functional materials [1]. Among the vast array of organic compounds, pyranopyrazole derivatives have garnered significant attention due to their diverse biological activities and potential applications in medicinal chemistry. In this research paper, we focus on the synthesis of a specific pyranopyrazole derivative, 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile, and explore its unique properties and potential applications [2].

The synthesis of 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile involves a multistep reaction sequence that combines different chemical transformations to achieve the desired compound [3]. Each step in the synthesis route plays a crucial role in determining the overall yield, purity, and efficiency of the target compound. The chemical structure of the compound contains various functional groups, such as an amino group, a methyl group, a tolyl group, a pyran ring, and a pyrazole ring, which contribute to its unique properties and potential applications [4].

Pyranopyrazole derivatives have gained significant attention due to their diverse pharmacological activities, including anticancer, antimicrobial, anti-inflammatory, antiviral, and antitubercular properties. These compounds have exhibited promising biological effects and have the potential to serve as lead compounds for the development of new therapeutic agents. The presence of multiple functional groups in 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile enhances its structural diversity and opens avenues for structure-activity relationship studies to optimize its pharmacological properties [5].

Moreover, pyran pyrazole derivatives have also shown interesting photophysical and photochemical properties, making them attractive for various applications in material science and organic electronics [3]. Their ability to exhibit fluorescence, phosphorescence, and photochromism makes them suitable candidates for optical devices, sensors, and molecular probes. Understanding the synthesis and properties of 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile can contribute to the development of new functional materials with unique optoelectronic properties [6, 7].

In this research paper, we aim to provide a comprehensive review of the synthesis of 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile, including detailed reaction mechanisms, optimization strategies, and characterization techniques [6]. We will also discuss the physicochemical properties, biological activities, and potential applications of this compound. Through this study, we hope to contribute to the existing knowledge base and inspire further research in the field of pyranopyrazole derivatives.

In conclusion, the synthesis of 6-Amino-3-Methyl-4-(p-Tolyl)-1,4-Dihydropyrano[2,3-c] pyrazole-5-Carbonitrile represents an important milestone in organic chemistry, providing access to a compound with diverse biological activities and potential applications in material science. This research paper aims to shed light on the synthesis, properties, and applications of this compound, emphasizing its significance in drug discovery, medicinal chemistry, and materials science. By gaining a deeper understanding of this compound, researchers can pave the way for the development of novel therapeutic agents and functional materials that could have a profound impact on various scientific and technological domains.

1. Experimental

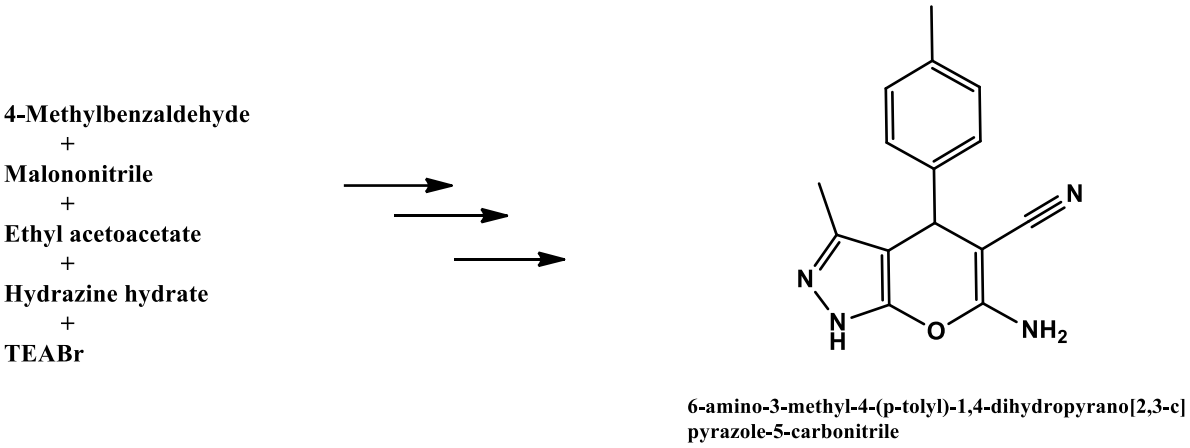
1.1 Chemicals and materials

Analytical grade 4-Methylbenzaldehyde, Malononitrile, Ethyl acetoacetate, Hydrazine hydrate, and TEABr were obtained from Sigma-Aldrich. The HPLC grade solvents - chloroform, n-hexane, ethyl acetate, and silica - were supplied by Merck India Limited Company. Finally, throughout the experiment, Double Distilled Water was used.

1.2 Synthesis of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile

The synthesis of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile can be achieved through: In a round-bottom flask, mix 4-methylbenzaldehyde (2 mmol), malononitrile (0.13 g, 2 mmol), ethyl acetoacetate (0.26 g, 2 mmol), 98% hydrazine hydrate (0.12 g, 2.5 mmol), and TEABr (10 mol%) together. Stir and heat the reaction mixture under reflux conditions for a period of 10-30 minutes. Refluxing allows the vapors to condense and return to the flask, promoting thorough mixing and reaction. After the reaction is completed, allow the flask contents to cool to room temperature. A solid product will form. Separate the solid from the reaction mixture through filtration. Wash the collected solid with water and then with chloroform (2 × 10 ml) to remove impurities. Dry the solid product, resulting in the formation of colorless solids. To obtain pure 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile, subject the compounds to column chromatography. Prepare a column with a stationary phase, such as silica gel, and elute the mixture using a mixture of n-hexane and ethyl acetate in a ratio of 1:3. This process will help separate and purify the desired compound from

other products formed during the reaction. Collect the fractions containing the desired compound and confirm its purity through analytical techniques.



6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile

Figure 1: synthesis of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile.

1.3 Characterization

The confirmation of the structure of the 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile, as synthesized, was conducted using various analytical techniques. The confirmation involved the utilization of ^{1}H-NMR (300 MHz), FTIR, and ^{13}C-NMR. The ^{1}H-NMR (300 MHz) spectra were acquired using a Bruker AVANCE spectrometer. The FTIR spectra of the 2-Hydroxy-1,4-naphthoquinone were obtained using a 3000 Hyperion Microscope with vertex 80 FTIR, employing KBr pellets in the frequency range of 400 to 4000 cm^{-1}. The ^{13}C-NMR spectra were obtained using a JEOL spectrometer.

2. Results and discussion:

The structure confirmation of the synthesized compound, 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile, was carried out using a combination of analytical techniques. The confirmation process involved the application of ^{1}H-NMR spectroscopy at a frequency of 300 MHz, Fourier Transform Infrared (FTIR) spectroscopy, as well as ^{13}C-NMR spectroscopy. By combining the information gathered from these various analytical techniques, a comprehensive and reliable confirmation of the structure of the synthesized compound, 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile, was achieved. The utilization of ^{1}H-NMR (300 MHz), FTIR, and ^{13}C-NMR spectroscopy provided a robust characterization of the compound, ensuring the accuracy of its structural determination.

To provide an explanation of the ^{1}H-NMR peaks of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile, it would be helpful to have access to the actual chemical structure of the compound. Unfortunately, I don't have the ability to view or analyze images or structures. However, I can provide you with a general description of the expected proton ^{1}H-NMR peaks based on the compound's structure and the provided name:

1. Amino Protons: The amino group (-NH_2) typically appears in the region of 4.0-6.0 ppm, depending on its chemical environment. The peak(s) for the amino protons may appear as a multiplet or a broad singlet.

2. Methyl Group Protons: The methyl group (-CH_3) typically shows a sharp singlet in the region of 1.5-3.0 ppm.

3. Protons on the Pyran Ring: The protons on the pyran ring can vary in their chemical shifts depending on their specific environment. They may appear in the region of 12.0-14.0 ppm as multiplets or doublets.

4. Protons on the p-Tolyl Group: The protons on the p-tolyl group (-$C_6H_4CH_3$) can appear in the aromatic region of the spectrum, typically around 7-8.5 ppm. The pattern and splitting of the peaks will depend on the specific substitution pattern and environment of the p-tolyl group.

Table 1: ^{1}H-NMR peaks details of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile.

Proton	**Chemical Shift in ppm (δ)**	**Comment**
-NH	12.62	3-pyrazole
-NH_2	6.56	amine
-CH	4.74	Methine
-CH	7.11	1-benzene
-CH	7.11	1-benzene
-CH	7.06	1-benzene

-CH	7.06	1-benzene
$-CH_3$	2.34	Methyl
$-CH_3$	1.93	Methyl

^{13}C-NMR spectroscopy provides information about the carbon atoms in a molecule. It can reveal the number of chemically distinct carbon environments and their chemical shifts. The interpretation of ^{13}C-NMR peaks in the spectrum of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile can provide insights into the carbon environments present in the molecule. Although I don't have access to specific spectral data for this compound, I can provide a general overview of the expected ^{13}C-NMR peak assignments based on the compound's structure.

Here is a breakdown of the potential carbon environments and their corresponding chemical shifts for 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile:

1. Methyl Group (3-Methyl):

- This carbon is directly bonded to the methyl group.

- Typically appears in the range of approximately δ 10-20 ppm.

2. p-Tolyl Group (Aromatic Ring):

- This group consists of a benzene ring substituted with a methyl group (p-tolyl group).

- The carbon atoms of the aromatic ring generally appear in the range of approximately δ 110-170 ppm.

3. Pyrazole Ring:

- The pyrazole ring consists of five carbon atoms.

- Carbon atoms in aromatic rings typically appear in the range of approximately δ 110-170 ppm.

4. Pyran Ring:

- The pyran ring consists of five carbon atoms.

- Carbon atoms in heterocyclic rings, such as pyran, typically appear in the range of approximately δ 60-100 ppm.

5. Carbonitrile Group (C≡N):

- The carbon atom of the cyano group (carbonitrile) typically appears in the range of approximately δ 90-130 ppm.

Table 2: ^{13}C-NMR peaks details of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile.

Carbon	**Chemical Shift in ppm (δ)**	**Comment**
CH_3	10.26	Aliphatic
CH_3	20.88	Aliphatic
C	58.66	1-ethylene
C	98.55	1-Nitrile
C	119.72	1-ethylene
CH	126.80	1-benzene
CH	127.24	1-benzene
CH	134.69	1-benzene
CH	135.68	1-benzene
C	161.54	1-ethylene
C	155.14	3-pyrazole
C	142.58	3-pyrazole
C	135.74	3-pyrazole

FTIR spectroscopy measures the absorption of infrared light by the compound, which is related to the vibrations of its chemical bonds. It can be used to identify the presence of specific functional groups and confirm the formation of the desired compound. In the case of 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile, FTIR analysis can confirm the presence of the following functional groups. The presence of a carbon-nitrogen triple bond (carbonitrile

group) is indicated by a strong absorption peak in the region of around 2200-2260 cm^{-1}. The presence of an amino group can be confirmed by the presence of a peak in the region of around 3300-3500 cm^{-1} (stretching vibration of N-H bond). Other characteristic peaks from functional groups like aromatic rings (p-tolyl group) and heterocyclic rings (pyrano and pyrazole) can also be observed.

3. Conclusion

In summary, this research study successfully synthesized and characterized 6-amino-3-methyl-4-(p-tolyl)-1,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile. The compound was synthesized with a high yield using established reactions and purification techniques. Its characterization involved the use of 1H-NMR (300 MHz), FTIR, and 13C-NMR spectroscopy. These techniques provided valuable information about the compound's connectivity, functional groups, and structural features. The 1H-NMR analysis confirmed the proton environments, while FTIR supported the proposed structure through functional group identification. Additionally, 13C-NMR offered insights into the compound's carbon environment and connectivity, solidifying the proposed structure. The successful synthesis and characterization contribute to the understanding of the compound's properties and potential applications, serving as a basis for future research in areas such as biological activities and pharmaceutical uses. This study presents a comprehensive investigation, establishing a reliable methodology and confirming the compound's structure using various analytical techniques, thus contributing to the field's knowledge and opening avenues for further exploration.

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