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Proceeding Paper

Synthesis and Characterization of New 3-Cyano-2-Pyridones Derivatives as Fluorescent Scaffolds †

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Abstract: 2-pyridones are among the most widely used nitrogen-containing heterocyclic derivatives in various fields due to their structural properties and biological and therapeutic activities. Therefore, their synthesis has become a major focus of organic chemistry in recent years. In this context, we are interested in the synthesis of N-alkyl-3-cyano-2-pyridones and their derivatives. This work describes a new synthetic method that allow easy and efficient access to these structures under mild conditions through a simple study of their fluorescence applications.

Keywords: organic synthesis; nitrogen heterocycles; 3-cyano-2-pyridones; Fluorescence.

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1. Introduction

Heterocyclic compounds represent the largest and most diverse family of organic compounds [1]. Today there are many heterocyclic compounds it is known that their number is increasing rapidly day by day due to extensive synthesis research and their synthetic benefits [2]. Heterocycle compounds play a role in most scientific fields such as medicinal chemistry, biochemistry, and other scientific fields [3]. There are many heterocyclic compounds, especially nitrogen heterocyclic compounds, they represent an important and unique class in the applied branch of organic chemistry [4]. A large amount of research has been devoted to the development of new molecular composite materials and has contributed to the development of a lot organic synthesis protocols to find abundant applications in the chemical sciences [5,6]. Numerous *N*-heterocyclic compounds that are generally found in nature have both physiological and pharmacological properties and are part of many important biological molecules, particularly 2-pyridone [7,8].

2-pyridone and its derivatives have attracted widespread attention due to their wide application in many fields such as fluorescence [9,10]. Moreover, the 2-pyridone core is ordinary to many natural products as well as synthetically useful compounds among which they exhibit various biological and therapeutic activities [11,12]. These wide utilities and waves of interest in the 2-pyridone as a key heterocycle, motivated us to look for new developments in the synthesis of 2-pyridone in recent years [13].

In our work, we report the synthesis of series of 3-cyano-2-pyridone derivatives (Figure 1) via a simple and convenient protocol.

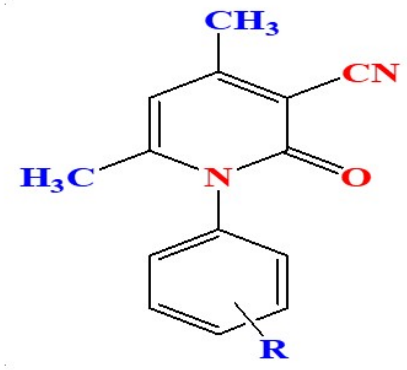


Figure 1. General structure of 3-cyano-2-pyridone derivatives.

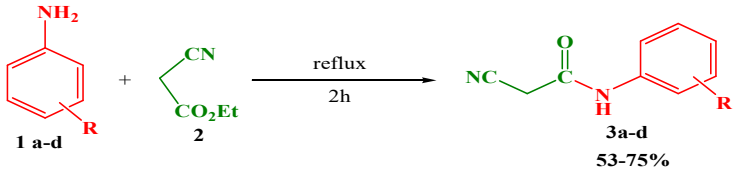
2. Results and Discussion

In this work, we reported the synthesis of 3-cyano-2-pyridones derivatives in two-step which are as follows:

2.1. Synthesis of N-alkylated-2-cyanoacetamide Derivatives

The starting point for our new synthetic approach is to synthesize N-alkylated-2-cyanoacetamide derivatives **3a-d** by the reaction of substituted anilines **1a–d** and ethyl cyanoacetate **2** refluxed at high temperature for 2h, the desired products obtained with good yields (Table 1).

Table 1. Synthesis of N-alkyled-2-cyanoacetamide derivatives.



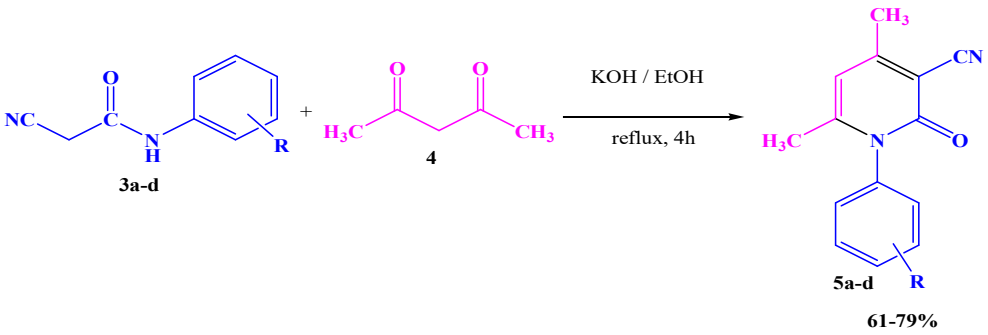
Entry	R	Yields (%)
3a	**H**	**58**
3b	**4-Cl**	**75**
3c	**4-F**	**62**
3d	**3-4diCl**	**53**

The structure of the synthesized compounds **3a–d** was confirmed by spectral analysis; the IR spectra (KBr, ν, cm^{-1}) showed the appearance of CN at 2255–2258 $cm^{-1,}$ NH at 3182–3303 cm^{-1}, and CO at 1663–1680 cm^{-1}, the ^1H NMR ($CDCl_3$, δ, ppm) showed the appearance of NH stretch at 7.90–7.95 ppm, CH_2 stretch at 3.56–3.62ppm, and H_{Ar} at 6.3–7.5 ppm.

2.2. Synthesis of 3-Cyano-2-pyridones Derivatives

We have synthesized a series of 3-cyano-2-pyridones derivatives **5a–d** using the cyanoacetamide **3a–d** previously synthesized in the first step with acetylacetone 4 at reflux in the presence of KOH as base and ethanol as a solvent for 4h. This strategy led to the 3-cyano-2-pyridones derivatives in excellent yields (Table 2).

Table 2. Synthesis of 3-cyano-2-pyridone derivatives.



Entry	R	Yields (%)
5a	**H**	**75**
5b	**4-Cl**	**78**
5c	**4-F**	**79**
5d	**3-4diCl**	**61**

The synthesized compounds **5a–d** was confirmed by spectroscopic analysis; the IR spectra (KBr, ν, cm^{-1}) showed the appearance of CN at 2215–2216 cm^{-1}, and CO at 1653–1674 cm^{-1}, the ^1H NMR ($CDCl_3$, δ, ppm) showed the appearance of CH stretch at 6.04–6.32 ppm, methyl groups at 2.19–2.20 ppm, and H_{Ar} at 7.19–7.82 ppm.

3. Experimental Procedures

*3.1. General Synthesis of N-Alkylated-2-cyanoacetamide Derivatives **3a–d***

The *N*-alkylated-2cyanoacetamide derivatives **3a–d** were obtained by the reaction between (0.02 mol) of anilines derivatives **1a–d** and (0.02 mol) of ethyl cyanoacetate 2, the reaction mixture refluxed at high temperature for 2h, the progress of the reaction is monitored by TLC. The solid obtained is filtered and washed with diethyl ether and ethanol to afford the desired product in good yields (53–75%).

*3.2. General Synthesis of 3-Cyano-2-pyridones Derivatives **5a–d***

The products **5a–d** were prepared using (0.006 mol) cyanoacetamide derivatives **3a–d** with (0.006 mol) of acetylacetone 4 in the presence of a few quantity of KOH and about 10 mL of ethanol as a solvent the reaction mixture was stirred and refluxed at 80 °C for 4h according to TLC, the formed precipitate after cooling was collected by filtration and washed with ethanol. The required products were obtained with excellent yields (61–79%).

4. Fluorescence Studies of Our 3-Cyano-2-pyridone Derivatives

In the aim to evaluate the interest of our synthesized products **5a–d** we decided to study their molecular fluorescence in three different concentrations; 10^{-4}, 10^{-5} and 10^{-6} using DCM as solvent. The fluorescence emission spectra obtained show the influence of concentration on the intensity (Figure 2).

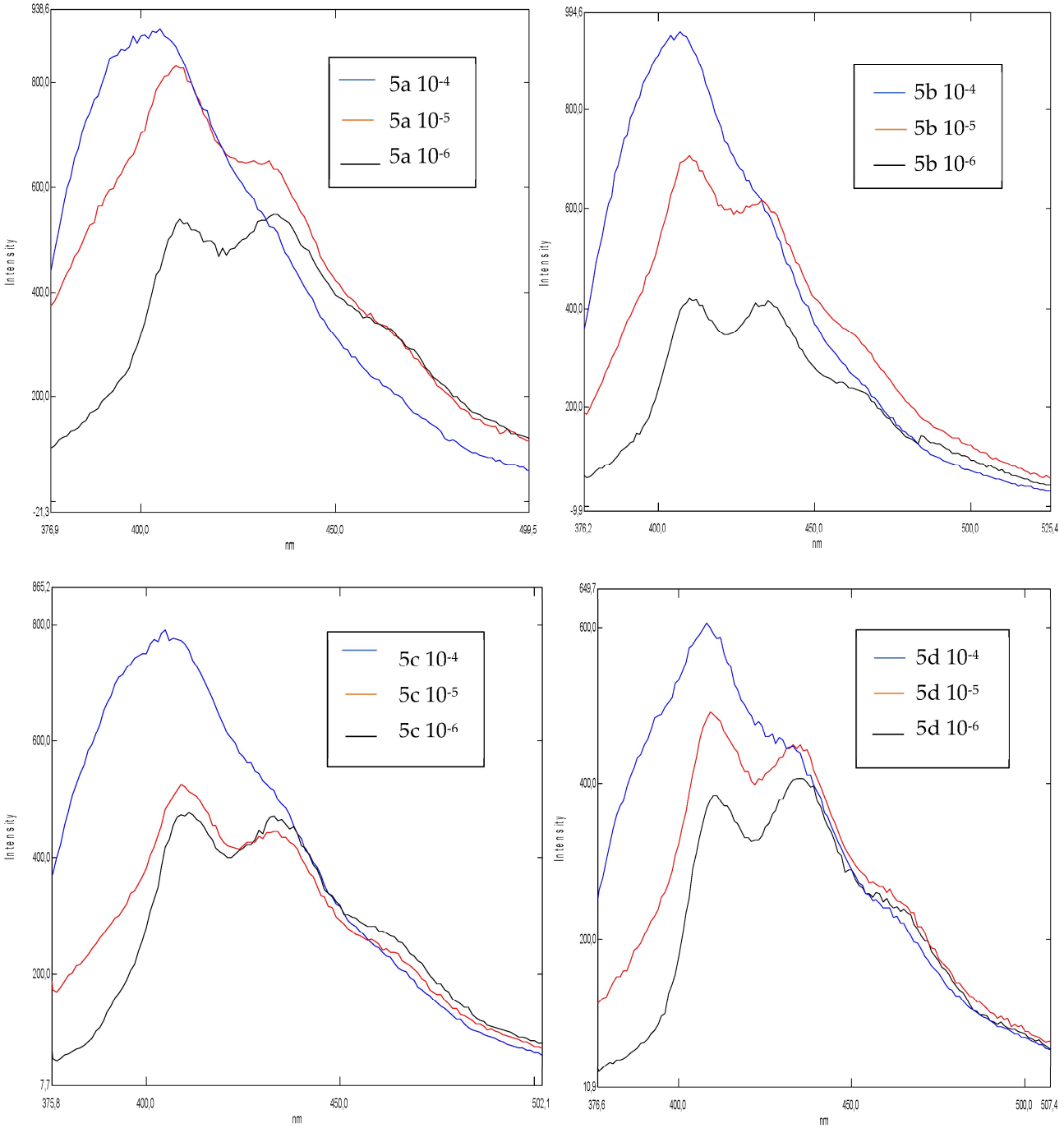


Figure 2. Emission spectra of the products **5a–d**.

We note a maximum intensity for all products at 10^{-4} M varies between 610–990 u.a and a low intensity at 10^{-6} M varies between 400–530 u.a. Therefore, we conclude that there is a positive correlation between concentration and the intensity; when the concentration is decreased the intensity is decreased with it.

5. Conclusions

we have succeeded in developing a simple, rapid, and efficient synthesis route for 3-cyano-2-pyridones derivatives this process includes some advantages such as mild operation conditions, simple reactants, and excellent yields. To evaluate the interest of our synthesized products we studied the influence of the concentration on the intensity of

their fluorescence where we noticed that the best intensity at a concentration of 10^{-4} corresponds to the product **5b**.

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Conflicts of Interest: The authors declare no conflict of interest, financial or otherwise.

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