

Effect of a Substituent in the Fourth Position on the Optical Properties of 2-Oxonicotinonitriles

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Abstract—Based on six representatives of 2-oxonicotinonitriles, the effect of the nature of the substituent in the fourth position of the pyridine system on the photophysical characteristics was studied. The role of the donor/acceptor nature of the substituent and the solvent nature in the absorbing and fluorescent properties of the compounds was shown.

Keywords: pyridines, nicotinonitrile, fluorescence, nitriles, trifluoromethyl, isonicotinic acid

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INTRODUCTION

2-Oxonicotinonitrile derivatives (2-oxo-1,2-dihydropyridine-3-carbonitrile, 3-cyanopyrid-2-one) are of considerable interest due to their diverse applications in various fields of science and technology. Among them, substances were found that are used in pharmaceuticals [1–14], agrochemistry [15], as agents of reducing steel corrosion [16], and in the creation of organic functional materials [17–28]. Substituted 2-oxonicotinonitriles are well known for their versatile biological activity. For example, they exhibit antitumor [3,4], anti-tuberculosis [5], anti-inflammatory [6], antipyretic [7], cytotoxic [8], and antimicrobial activity [9]. The possibility of their use as inhibitors of SARS-CoV-2 protease [10], aggregation of α -synuclein [11],

In addition, 2-oxonicotinonitrile derivatives are known for their unique photophysical properties and a wide range of potential applications based on them, for example, as dyes and pigments [17–21], nonlinear optical (NLO) and photorefractive materials [22], dye-sensitized solar cells (DSSC) [23, 24], an emitter in a device with a host–guest configuration in the manufacture of OLED displays [25], a fluorescent probe for visualizing lipid droplets to distinguish between dead and live zebrafish [26], a fluorescent dye for visualizing latent fingerprints and detecting nitrite ion (NO_2^-) [27], a multisensitive sensor for Ru^{3+} , Fe^{3+} , CrO_4^{2-} , $\text{Cr}_2\text{O}_7^{2-}$ and MnO_4^- ions [28]. Many

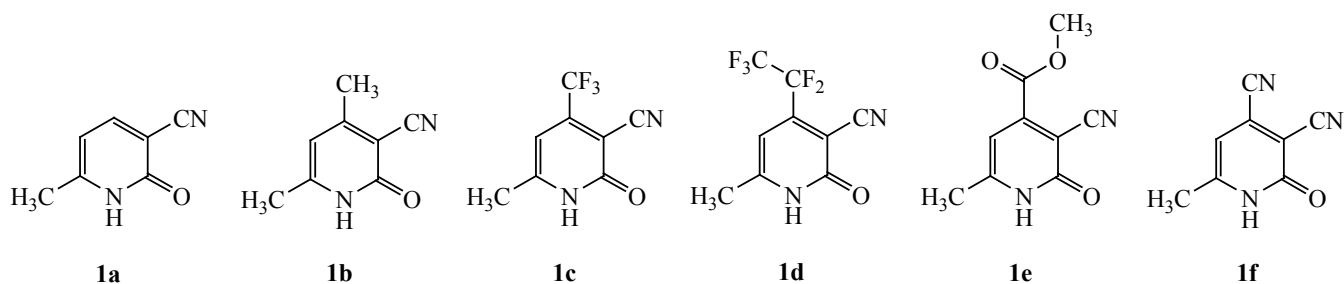
of the above properties are based on the luminescence phenomenon.

The pyridone fragment, in particular, the 2-oxonicotinonitrile one, causes the appearance of fluorescent properties in the molecule [25–36]. However, there is practically no information in the literature on the systematic study of the effect of an individual substituent on the photophysical properties of such compounds, despite the fact that the optical properties can be finely tuned by introducing individual functional groups. In this regard, this work was devoted to comparing the fluorescent properties of 6-methyl-2-oxo-1,2-dihydropyridine-3-carbonitriles **1a–1f** differing by a substituent in the fourth position of the pyridine system (Scheme 1). Pyridone **1a** was chosen as the model structure. Molecule **1b** contains an electron-donating methyl group. Perfluoroalkyl groups (compounds **1c** and **1d**) were studied as electron-withdrawing substituents with a strong negative inductive effect. The ester group (**1e**) and cyano group (**1f**) were studied as substituents with the conjugation effect.

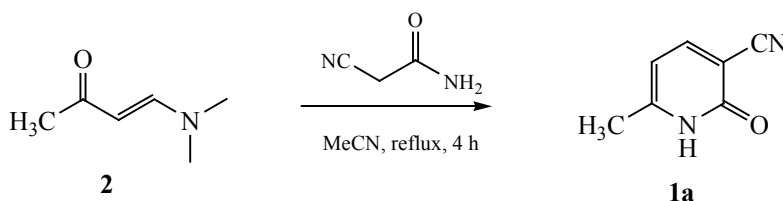
RESULTS AND DISCUSSION

Compound **1a** was synthesized by reacting enamino-ketone **2** with cyanoacetamide in acetonitrile (Scheme 2). Compounds **1b–1e** were obtained from the corresponding β -diketones **3** and cyanoacetamide in refluxing ethanol in the presence of 1,4-diazabicyclo[2.2.2]-octane (DABCO) (Scheme 3).

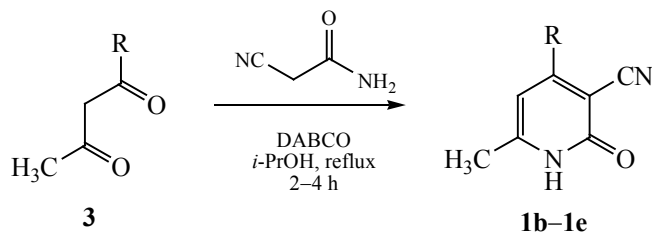
Scheme 1.



Scheme 2.

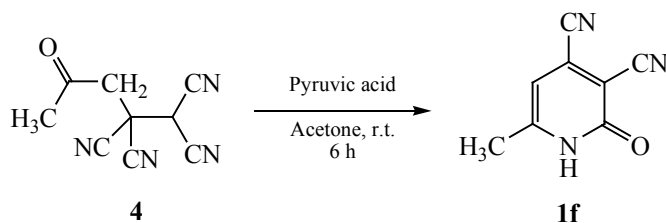


Scheme 3.



R = CH₃ (**b**), CF₃ (**c**), C₂F₅ (**d**), COOMe (**e**).

Scheme 4.



Compound **1f** was prepared according to a previously developed procedure [37] based on the intramolecular cyclization of 4-oxopentane-1,1,2,2-tetracarbonitrile **4** in the presence of pyruvic acid in acetone at room temperature (Scheme 4).

Compounds **1a–1c**, **1e**, and **1f** have been reported earlier, their structure was confirmed by IR and mass

spectrometry data, as well as by ¹H, ¹³C, and ¹⁹F NMR for pyridone **1e**, which was not previously described.

Photophysical properties of compounds **1a–1f** were studied in three solvents of different nature: acetonitrile, acetic acid and pyridine (Table 1, Figs. 1–3). It was found that, as a rule, for all the studied compounds, changing the solvent from acetonitrile to acetic acid leads to a

Table 1. Spectral data for compounds **1a–1f**

Compound	Solvent	$\lambda_{\text{abs (max)}}$, nm ^a	ϵ_{max} , L mol ⁻¹ cm ⁻¹	$\lambda_{\text{fl (max)}}$, nm ^b	Φ_{F} ^c
1a	MeCN	340	9542	385	0.18
	AcOH	329	8891	385	0.21
	Pyridine	346	9891	390	0.13
1b	MeCN	334	9254	382	0.07
	AcOH	323	5082	379	0.13
	Pyridine	338	9068	386	0.06
1c	MeCN	360	10369	410	0.66
	AcOH	347	9353	410	0.71
	Pyridine	365	8280	409	0.35
1d	MeCN	360	11116	411	0.69
	AcOH	349	9787	410	0.70
	Pyridine	365	9045	419	0.32
1e	MeCN	372	7545	439	0.71
	AcOH	360	7202	439	0.74
	Pyridine	380	4068	447	0.39
1f	MeCN	378	9171	434	0.77
	AcOH	366	7030	431	0.85
	Pyridine	384	4948	437	0.29

^a Absorption spectra of solutions with a concentration of 10^{-4} M.

^b Fluorescence spectra of solutions with a concentration of 10^{-4} M. The excitation wavelength corresponds to the absorption maximum.

^c Relative quantum yield was measured using 7-hydroxy-4-methylcoumarin in phosphate buffer pH = 10 ($\Phi_{\text{F}} = 0.7$, $\lambda_{\text{ex}} = 330$ nm).

hypsochromic shift of the absorption maxima by an average of 12 nm, but has almost no effect on the location of the emission maximum. The use of pyridine leads to a bathochromic arrangement of the absorption band maxima by 6 nm on average, and the fluorescence maxima are shifted to the red region relative to acetonitrile.

For all the compounds obtained, with the exception of **1a** and **1b**, there is a tendency to a decrease in the value of the molar light absorption coefficient when pyridine or acetic acid is used instead of acetonitrile.

The maxima of the absorption and fluorescence bands for compounds substituted with acceptor substituents

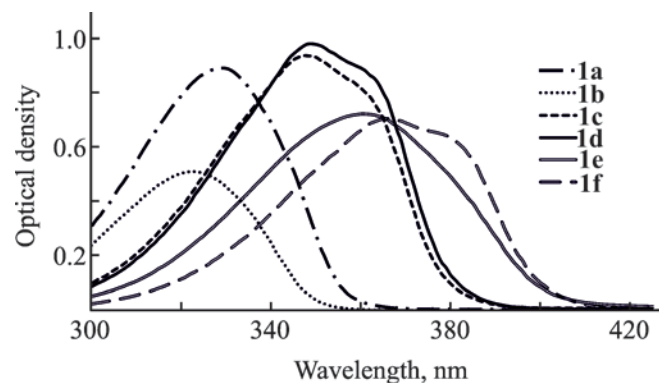


Fig. 1. Absorption spectra of compounds **1a–1f** in acetic acid solution ($c = 10^{-4}$ M).

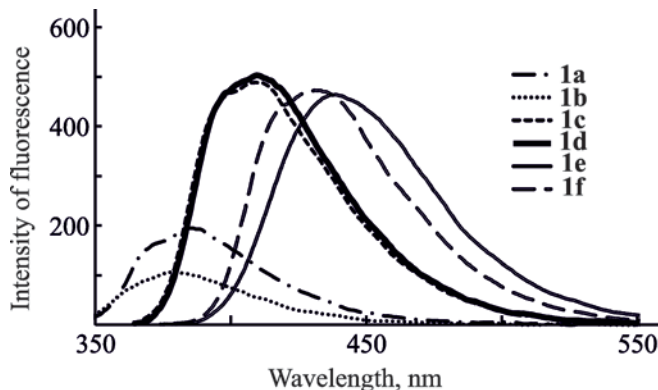


Fig. 2. Fluorescence spectra of compounds **1a–1f** in acetic acid solution ($c = 10^{-4}$ M).

with the conjugation effect in all the studied solvents are bathochromic relative to all others and are in the range of 360–380 and 439–447 nm, respectively, for compound **1e**, 366–384 and 431–437 nm for **1f**. Compounds **1a** and **1b** have the most hypsochromic shifts in the electronic spectra, with absorption and emission band maxima in the range of 329–346 and 385–390 nm, respectively, for **1a**, 323–338 and 379–386 nm for **1b**. The maxima of the absorption and photoluminescence bands for fluoroalkyl-substituted derivatives **1c** and **1d** have intermediate values relative to those described above.

The maximum quantum yield for all the tested compounds is observed in acetic acid solution. Pyridine, having a basic nature, can lead to deprotonation of the NH acid center and the process of salt formation [38]. Apparently, therefore, the minimum values of the fluorescence quantum yield are observed in it. In contrast, acetic acid suppresses the dissociation process, which affects the efficiency of the radiative process of pyridones **1** in its solution.

It was found that the introduction of an electron-withdrawing substituent into the molecule of 2-oxonicotinonitrile **1a** leads to a significant increase in the quantum yield (more than 3 times), the leader is compound **1f** containing a cyano group, $\Phi_F(\text{AcOH}) = 85\%$. On the contrary, if the fourth position of the pyridine ring contains an electron-donating substituent (compound **1b**), then a sharp decrease in the efficiency of the radiative process is observed in all the studied solvents (Table 1, Fig. 3).

EXPERIMENTAL

IR spectra were recorded in a thin layer (suspension in mineral oil) on an FSM-2201 IR Fourier spectrometer. NMR spectra were recorded on a Bruker DRX-500 spectrometer, operating frequency 500.13 (^1H), 125.76 (^{13}C), 470.59 MHz (^{19}F), using DMSO- d_6 as a solvent and TMS as an internal standard. Mass spectra were taken on a Shimadzu GCMS-QP2020 spectrometer (energy of ionizing electrons was 70 eV). Elemental analysis was performed on a FlashEA 1112 CHN analyzer. Progress of reactions and purity of the synthesized substances were monitored by TLC on Sorbfil PTSKh-AF-A-UV plates (detecting with UV irradiation, iodine vapor, thermal decomposition). Melting points of the substances were determined on an OptiMelt MPA100 instrument. Absorption spectra were recorded on an Agilent Cary 60 UV-Vis Spectrophotometer. Fluorescence spectra

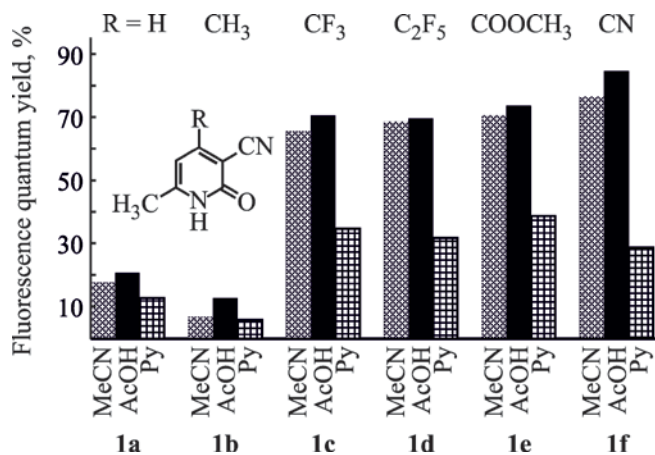


Fig. 3. Fluorescence quantum yield of compounds **1a–1f**.

were recorded on an Agilent Cary Eclipse spectrometer. Fluorescence quantum yield for all solutions was measured relative to 7-hydroxy-4-methylcoumarin in phosphate buffer with pH = 10 ($\Phi_F = 0.7$) [39]. The excitation wavelength is 330 nm.

6-Methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (1a). Cyanoacetamide (0.372 g, 4.4 mol) was added to solution of enaminoketone **2** (0.5 g, 4.4 mmol) in 15 mL of acetonitrile, and the mixture was refluxed for 4 h with stirring. The formed precipitate was filtered off and recrystallized from a mixture of ethanol–water (1 : 1). Yield 83%, mp 294–296°C. IR spectrum, ν , cm^{-1} : 3291 (N–H), 2223 (C≡N), 1666 (C=O). Mass spectrum, m/z (I_{rel} , %): 134 (100) $[M]^+$, 119 (4) $[M - \text{CH}_3]^+$, 106 (30) $[M - \text{CO}]^+$, 105 (73) $[M - \text{CO} - \text{H}]^+$. Found, %: C 62.76; H 4.55; N 20.79. $\text{C}_7\text{H}_6\text{N}_2\text{O}$. Calculated, %: C 62.68; H 4.51; N 20.88.

General procedure for the synthesis of 6-methyl-2-oxo-1,2-dihydropyridine-3-carbonitriles 1b–1e. The corresponding diketone **3** (4.4 mmol) was dissolved in 15 mL of propanol-2, then 0.372 g (4.4 mol) of cyanoacetamide and 1 g of 1,4-diazabicyclo[2.2.2]octane (8.9 mmol) were added. The mixture was stirred at reflux for 2–4 h (monitoring by TLC). After completion of the reaction, the reaction mixture was cooled to room temperature, poured into cold water (30 mL), and acidified with 2 M HCl solution until acidic. The resulting precipitate was filtered off, washed with water, recrystallized from the appropriate solvent, and dried in a vacuum desiccator over CaCl_2 to constant weight.

4,6-Dimethyl-2-oxo-1,2-dihydropyridine-3-carbonitrile (1b). Yield 90%, mp 293–295°C. IR spectrum, ν , cm^{-1} : 3291 (N–H), 2219 (C≡N), 1664 (C=O). Mass spectrum, m/z (I_{rel} , %): 148 (100) $[M]^+$, 133 (2) $[M - \text{CH}_3]^+$, 120 (35) $[M - \text{CO}]^+$, 119 (80) $[M - \text{CO} - \text{H}]^+$. Found, %: C 65.01; H 5.40; N 18.82. $\text{C}_8\text{H}_8\text{N}_2\text{O}$. Calculated, %: C 64.85; H 5.44; N 18.91.

6-Methyl-2-oxo-4-(trifluoromethyl)-1,2-dihydropyridine-3-carbonitrile (1c). Yield 92%, mp 234–236°C. IR spectrum, ν , cm^{-1} : 3324 (N–H), 2226 (C≡N), 1675 (C=O). Mass spectrum, m/z (I_{rel} , %): 202 (100) $[M]^+$, 187 (2) $[M - \text{CH}_3]^+$, 174 (45) $[M - \text{CO}]^+$, 173 (67) $[M - \text{CO} - \text{H}]^+$, 105 (26) $[M - \text{CF}_3 - \text{CO}]^+$. Found, %: C 47.47; H 2.50; N 13.79. $\text{C}_8\text{H}_5\text{F}_3\text{N}_2\text{O}$. Calculated, %: C 47.54; H 2.49; N 13.86.

6-Methyl-2-oxo-4-(pentafluoroethyl)-1,2-dihydropyridine-3-carbonitrile (1d). Yield 84%, mp 241–243°C. IR spectrum, ν , cm^{-1} : 3306 (N–H), 2233 (C≡N), 1677 (C=O). ^1H NMR spectrum (DMSO- d_6), δ , ppm: 2.37 s (3H, CH_3), 6.56 s (1H, Py), 13.38 br. s (1H, NH). ^{13}C NMR spectrum (DMSO- d_6), δ_{C} , ppm: 19.9 (CH_3), 85.7 (β -Pyr), 103.1 (β -Pyr), 112.1 t. q (CF , $^1J_{\text{CF}}$ 285, 39 Hz), 113.9 (CN), 118.6 q. t (CF , $^1J_{\text{CF}}$ 287, 37 Hz), 144.8 q (γ -Pyr, $\underline{\text{C}}-\text{CF}_3$, $^2J_{\text{CF}}$ 23 Hz), 156.7 (α -Pyr), 160.9 (α -Pyr). ^{19}F NMR spectrum (DMSO- d_6), δ_{F} , ppm: –82.2, –112.7. Mass spectrum, m/z (I_{rel} , %): 252 (100) $[M]^+$, 225 (5) $[M - \text{HCN}]^+$, 224 (39) $[M - \text{CO}]^+$, 223 (12) $[M - \text{CO} - \text{H}]^+$, 155 (94) $[M - \text{CF}_3 - \text{CO}]^+$. Found, %: C 42.96; H 1.98; N 11.16. $\text{C}_9\text{H}_5\text{F}_5\text{N}_2\text{O}$. Calculated, %: C 42.87; H 2.00; N 11.11.

Methyl 3-cyano-6-methyl-2-oxo-1,2-dihydropyridine-4-carboxylate (1e). Yield 88%, mp 242–244°C. IR spectrum, ν , cm^{-1} : 3306 (N–H), 2221 (C≡N), 1746 (C=O). Mass spectrum, m/z (I_{rel} , %): 192 (100) $[M]^+$, 161 (52) $[M - \text{OCH}_3]^+$, 134 (58) $[M - \text{OCH}_3 - \text{HCN}]^+$, 133 (73) $[M - \text{COOCH}_3]^+$, 132 (18) $[M - \text{COOCH}_3 - \text{H}]^+$. Found, %: C 56.36; H 4.17; N 14.66. $\text{C}_9\text{H}_8\text{N}_2\text{O}_3$. Calculated, %: C 56.25; H 4.20; N 14.58.

6-Methyl-2-oxo-1,2-dihydropyridine-3,4-dicarbonitrile (1f). To a solution of 1 g (5.4 mmol) of 4-oxopentane-1,1,2,2-tetracarbonitrile **4** in 5 mL of acetone was added a solution of 0.47 g (5.4 mmol) of pyruvic acid in 3 mL of water. The mixture was stirred at room temperature for 6 h, the formed precipitate was filtered off, washed with water, and dried in a vacuum desiccator over CaCl_2 to constant weight. Yield 40%, mp 247–249°C. IR spectrum, ν , cm^{-1} : 3329 (N–H), 2225 (C≡N), 1661 (C=O). Mass spectrum, m/z (I_{rel} , %): 159

(83) $[M]^+$, 144 (4) $[M - \text{CH}_3]^+$, 132 (6) $[M - \text{HCN}]^+$, 131 (50) $[M - \text{CO}]^+$, 130 (100) $[M - \text{CO} - \text{H}]^+$. Found, %: C 60.29; H 3.14; N 26.46. $\text{C}_8\text{H}_5\text{N}_3\text{O}$. Calculated, %: C 60.38; H 3.17; N 26.40.

CONCLUSIONS

In conclusion, six derivatives of 2-oxonicotinonitrile were obtained. The effect of the nature of the substituent in the fourth position of the pyridine system on the photophysical properties was studied. It was shown that the introduction of a cyano group leads to the maximum values of the fluorescence quantum yield among the studied functional groups and reaches 85%.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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