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Three-Component synthesis and characterization of nicotinamide derivatives containing a buta-1,3-diene-1,1,3-tricarbonitrile fragment

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ABSTRACT

The synthesis of nicotinamide derivatives containing a buta-1,3diene-1,1,3-tricarbonitrile fragment through the base-promoted three-component reaction of an aromatic aldehyde, an acetoacetamide and malononitrile dimer in the presence of an oxidant is reported. The HOMO-LUMO energy levels were estimated by cyclic voltammetry and UV-Vis measurements. The influence of aryl and carbomoyl substituents on the optical and electrochemical properties was also investigated.

GRAPHICAL ABSTRACT



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KEYWORDS

Cyclic voltammetry; malononitrile dimer; multicomponent reaction; optical properties; TCPy

Introduction

Nicotinamide derivatives widely occur in various natural products, functional materials, and pharmaceutical agents.^[1-15] Nicotinamide, also known as niacinamide, is a form of vitamin B³ and it is essential for the human health.^[1] Various nicotinamide derivatives possess antitumor,^[2-6] anti-HIV,^[7] and anti-diabetogenic properties.^[8] Several nicotinamides were used for treating acquired inflammatory,^[9,10] heart^[11-13] and skin^[14,15] diseases.

At the same time, the spectral-luminescent properties of nicotinamides and their complexes have not been studied practically. Although the presence of an amide group in the beta position of substituted heterocyclic compounds favored the appearance of optical properties.^[16–19]

A convenient approach to the synthesis of highly functionalized nicotinamides is the use of multicomponent reactions (MCR).^[20–22] Herein, we report a three-component synthesis of nicotinamide derivatives containing a buta-1,3-diene-1,1,3-tricarbonitrile

• Supplemental data for this article can be accessed on the publisher's website.

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Scheme 1. Synthesis of 4-aryl-5-cyano-6-(dicyanomethylene)-2-methyl-1,6-dihydropyridine-3-carboxa-mides 1a–i.

(BDTC) fragment. The importance of this fragment is that it acts as a powerful acceptor in push-pool chromophores and compounds with practically useful properties.^[23-27]

Results and discussion

The synthesis of 4-aryl-5-cyano-6-(dicyanomethylene)-2-methyl-1,6-dihydropyridine-3carboxamides 1 (TCPy) is based on the three-component reaction between an acetoacetamide, an aromatic aldehyde and malononitrile dimer in the presence of sodium nitrite working as a base as well as an oxidant (Scheme 1).

Various organic bases (piperidine, DABCO, ammonium acetate, sodium ethoxide and potassium tert-butoxide) were tested and they led to the isolation of the mixture of compounds 1 and 2. Prolonged heating at reflux with air bubbling also did not ensure a complete reaction. The isolation of compounds 2 with a subsequent oxidation step significantly decreased the yield of compounds 1. It was found that the use of sodium nitrite as a catalyst led to the formation of compounds 1 with good yields of up to 84%. Apparently, sodium nitrite works as the basic catalyst as well as the oxidant.^[28]

The reaction includes the tandem Knoevenagel condensation–Michael addition steps, which lead to the formation of intermediate A (Scheme 1). Subsequent cyclization of the amino group to the carbonyl group led to the formation of hexahydropyridine derivatives **B**. Elimination of water forms 3-cyano-2-(dicyanomethylene)-1,2,3,4-tetra-hydropyridines **2** and the further oxidation results in the pyridines **1** with 60-84% yield.

TCPy 1 are yellow or orange crystalline powders and were found to be fluorescent both in the solution and the solid state. Using compound 1b as a model the solvatochromic properties were investigated (Table 1). It was observed that the maxima of emission and absorption bands were almost constant in most of the solvents. The only exception is the "blue shift" of the fluorescence maximum in acetic acid. The most intensive emission was observed in acetonitrile and DMSO solutions.

Furthermore, the spectral-luminescent properties of compounds 1 with various aryl and carbomoyl substituents were studied using acetonitrile as the solvent (Fig. 1,

Solvent	λ_{\max}^{abs} (nm)	A _{max} ^{abs}	$\varepsilon \text{ (mol} \times L^{-1} \times \text{cm}^{-1})$	$\lambda_{\max}^{ \ \ flu}$ (nm)
CH ₃ CN	326, 398	0.3183, 0.0327	31965, 3284	513
AcOH	327, 414	0.4403, 0.0766	40846, 7106	413
DMSO	328	0.4488	65407	509
AcOEt	328, 429	0.7455, 0.1248	70102, 11735	516
1,4-Dioxane	325	0.0813	7508	507

Table 1. Solvatochromic properties of compound 1b.



Figure 1. Absorption spectra of compounds 1a-i in acetonitrile.

Compound	λ_{abs}^{max} (nm)	$A_{\rm abs}^{\rm max}$	$\epsilon \text{ (mol} \times L^{-1} \times \text{cm}^{-1})$	$\lambda 1_{flu}^{max}$ (nm) ¹	$\lambda_{flu}^{2}^{max}$ (nm) ¹
1a	325, 389	0.4379, 0.0491	39894, 4473	519	528
1b	326, 398	0.3183, 0.0327	31965, 3284	513	511
1c	325	0.2143	20430	509	536
1d	325	0.4935	48731	-	545
1e	328, 389	0.4688, 0.0721	44733, 6880	518	522
1f	327, 396	0.4111, 0.0636	42178, 6525	516	535
1g	325, 382	0.1823, 0.0157	15991, 1377	517	525
1h	327, 386	0.4759, 0.0487	47969, 4909	523	518
1i	329, 386	0.4255, 0.0483	42663, 4843	521	516

Table 2. Absorption and fluorescent properties of compounds 1.

 $1 \lambda_{flu}^{1}$ max – fluorescence maximum in acetonitrile, excited at 325 nm; λ_{flu}^{2} max – solid-state emission maximum of compounds 1, excited at 365 nm.

Table 1). Two peaks characterize the absorption spectra: an intense band at 325–329 nm and a broad band with low intensity at 382–398 nm, presumably corresponding to π – π * and n– π * electronic transitions, respectively.

A fluorescence study showed that the most intense fluorescence is observed for compounds 1b,c,e,f with halo- and methoxy-groups. In contrast, for the nitro-substituted compound 1d the fluorescence was practically not observed. The substituents in the carboxamide group of compounds 1h,i had literally no influence on the spectral properties. The solid-state emission of compounds 1 is in the range of 511-545 nm (Table 2).

	E _{ox} ^{onset} vs. Ag/AgCl (V)	HOMO (eV)		LUMO (eV)		<i>E</i> _g (eV)		
Compound		found	calc.	found	calc.	found	calc.	$\lambda_{ m abs}^{ m onset}$ (nm)
1a	0.713	-5.06	-6.29	-2.32	-3.11	2.74	3.18	452
1b	0.688	-5.04	-6.19	-2.27	-2.99	2.77	3.2	447
1c	0.690	-5.04	-6.16	-2.18	-2.91	2.86	3.25	433
1d	0.750	-5.10	-6.74	-2.42	-4.31	2.68	2.43	463
1e	0.732	-5.08	-6.36	-2.31	-3.19	2.77	3.17	448
1f	0.738	-5.09	-6.35	-2.33	-3.15	2.76	3.2	449
1g	0.712	-5.06	-6.23	-2.32	-3.04	2.74	3.19	453
1h	0.692	-5.04	-6.12	-2.31	-3.02	2.73	3.1	453
1i	0.738	-5.09	-6.25	-2.30	-3.11	2.79	3.14	444

 Table 3. Electrochemical characteristics of compounds 1, obtained by using cyclic voltammetry and electronic spectroscopy.

The HOMO energy levels of compound 1 were estimated from the onset oxidation potential (E_{ox}^{onset}) (Table 3). Cyclic voltammetry (CV) was performed in acetonitrile solution containing 0.1 M tetrabutylammonium hexafluorophosphate (NBu₄PF₆) as supporting electrolyte. All potentials have been calibrated with ferrocene/ferrocenium (Fc/Fc⁺) couple as the internal standard and are reported versus saturated silver chloride electrode (Ag/AgCl). The CV of the compounds 1 shows a reversible oxidation process with the onset points of oxidation in the 0.692–0.750 V region. The width of the band gap (E_g) was refined using electronic spectra of compound 1. The energy of the LUMO was found from the values of HOMO and E_g because of the irreversibility of the reduction process. It was found that all the compounds synthesized had a small band gap (E_g) of 2.68–2.86 eV, and their LUMO energies were in the range from -2.42 to -2.18 eV, which is higher than the edge of the conduction band of titanium dioxide (-4.4 eV). This indicates the possibility of an efficient electron transfer from the HOMO of a dye to the conduction band of the semiconductor.

Figure 2 shows the computational calculations to expect the HOMO/LUMO energy potentials of compound 1a. The computational theoretical calculation was carried out using TDDFT(B3LYP)/6-31 + G(d,p). It showed that electron density of the highest occupied molecular orbital (HOMO) is localized on the dicyanomethylene moiety and the lowest unoccupied molecular orbital (LUMO) involved pyridine and phenyl ring (Fig. 2). Table 3 shows the HOMO/LUMO energy levels for the compounds 1, where the potential energy values for the computationally calculated levels and the determined levels are shown. For each compounds, the potential energy levels showed the similar characteristic patterns from the values of computational calculation and electrochemical measurement.

It was found that all the compounds synthesized have a small band gap (E_g) of 2.68–2.86 eV, and their LUMO energies are in the range from -2.18 to -2.42 eV, which is higher than the edge of the conduction band of titanium dioxide (-4.4 eV). This indicates the possibility of an efficient electron transfer from the HOMO of a dye to the conduction band of the semiconductor. Moreover, the oxidation potentials of the test compounds were higher than the standard redox potential of the couple triiodide/iodide in acetonitrile, which demonstrates the possibility of dye regeneration by the electrolyte solution containing this redox couple.



Figure 2. HOMO–1, HOMO, LUMO and LUMO +1 for compound 1a, computed with TDDFT(B3LYP)/ 6-31 + G(d,p).

Conclusions

Thus, several pyridine derivatives 1 containing the buta-1,3-diene-1,1,3-tricarbonitrile (BDTC) fragment have been synthesized by the one-pot reaction between an aromatic aldehyde, an acetoacetamide and malononitrile dimer in the presence of sodium nitrite working as a base as well as an oxidant. Compounds 1 are yellow or orange crystalline powders and were found to be fluorescent, both in the solution and the solid state. Also, HOMO, LUMO levels and energy band gap of synthesized compounds were estimated by the combination of cyclic voltammetry and UV-Vis measurements.

Experimental

The progress of the reactions as well as purity of compounds was monitored by TLC with F254 silica-gel precoated sheets (Sorbfil, Russia) using ethyl acetate as eluent (spots were visualized under UV light, by treatment with iodine vapor, or by heating). The melting points were measured on an OptiMelt MPA100 (Stanford Research Systems, USA) melting point apparatus. IR spectra were recorded on an FSM-1202 spectrometer with Fourier transform (Infraspek, Russia) from samples dispersed in mineral oil. The NMR spectra were recorded on Bruker DRX-500 spectrometers (Bruker Corporation, Germany) using DMSO- d_6 as solvent and TMS as internal standard, chemical shifts are expressed in ppm. Elemental analysis was performed on a Carlo-Erba model EA1108 (Isomass Scientific Inc., Canada) and the values are $\pm 0.4\%$ of the theoretical ones. Mass spectra were recorded on a Finnigan MAT INCOS-50 (EI, 70 eV) (GenTech Scientific, Inc., USA). The UV spectra were recorded on an SF-2000 spectrophotometer (OKB Spectr, Russia). Fluorescence spectra were recorded on a Fluorat®-02-Panorama spectrofluorimeter (Lumex, Russia). Solid-state emission was registered in powders at room temperature on the Cary Eclipse fluorescence spectrophotometer (Agilent, USA). The electrochemical measurements were carried out using a Keithley 2450-EC Graphical Potentiostat (Tektronix, USA) and a voltammetric three-electrode VC-2 cell (working electrode - platinum, reference electrode - silver chloride, auxiliary electrode - platinum).

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General procedure for the synthesis of a series of 4-aryl-5-cyano-6-(dicyanomethylene)-2-methyl-1,6-dihydropyridine-3-carboxamides 1

A mixture of aromatic aldehydes (1 mmol), acetoacetamide (1 mmol), malononitrile dimer (1 mmol) and sodium nitrite (1.1 mmol) in EtOH (5 mL) was stirred at 70 °C for 6 h. After reaction completion (TLC), the solution was neutralized with 1% HCl to pH = 4. The precipitate was triturated with H₂O (10 mL), filtered and washed with H₂O (10 mL), and then recrystallized from a mixture of 1,4-dioxane: *i*-PrOH.

5 -Cyano-6-(dicyanomethylene)-4-(4-methoxyphenyl)-2-methyl-1,6-dihydropyridine-3carboxamide 1b

mp 269-270°C (dec.); ¹H NMR (500.13 MHz, DMSO- d_6): δ 2.42 (3H, s, CH₃), 3.80 (3H, s, OCH₃), 4.08 (1H, br s, NH), 7.01 (2H, d, J=8.8 Hz, C_6H_4), 7.32 (2H, d, J=8.7 Hz, C_6H_4), 7.37 (1H, NH₂), 7.59 (1H, NH₂). ¹³C NMR (125.76 MHz, DMSO- d_6): δ 19.28, 40.57, 55.15, 96.84, 113.57, 114.21, 118.21, 124.59, 126.64, 129.91, 152.49, 154.50, 156.69, 160.27, 165.96. IR (mineral oil, cm⁻¹): 3388-3107 (NH₂, NH), 2226, 2199 (CN), 1659 (C=O), 1603 (C=C). MS (EI, 70 eV): m/z (%) 331 [M]⁺ (15). Anal. Calcd for $C_{18}H_{13}N_5O_2$: C, 65.25; H, 3.95; N, 21.14. Found: C, 65.41; H, 4.11; N, 20.98.

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