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AN EFFICIENT SYNTHETIC ROUTE TO SUBSTITUTED XANTHENE ANALOGUES

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The reactions of N-[(9-chloro-1,2-dihydrocyclopenta[b]chromen-3-yl)methylene]-N-methylmethanaminium and N-[(11-chloro-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)methylene]-N-methylmethanaminium perchlorates with (1-phenylethylidene)malononitrile were studied in boiling acetonitrile in the presence of piperidine. These reactions led to the formation of new organic dyes, [(2E)-1-phenyl-3-(9-piperidin-1-yl-1,2-dihydrocyclopenta[b]chromen-3-yl)prop-2-en-1-ylidene]malononitrile and [(2E)-1-phenyl-3-(11-piperidin-1-yl-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)prop-2-en-1-ylidene]malononitrile, obtained in high yields. The structures of the products were confirmed by ^{1}H NMR spectroscopy, mass spectrometry, and elemental analysis. The observed spin-spin coupling constants indicated that the products adopted the E-configurations of the double bonds. The reaction mechanism was determined to proceed via a Knoevenagel condensation followed by nucleophilic substitution of chlorine with a piperidine fragment. Spectroscopic investigations of the dyes were performed in organic solvents, in particular acetonitrile and chloroform. For [(2E)-1-phenyl-3-(9-piperidin-1-yl-1,2-dihydrocyclopenta[b]chromen-3-yl)prop-2-en-1-ylidene]malononitrile, the absorption maximum was ~605 nm, and the emission maximum was ~685 nm, with a quantum yield of 34.62% in acetonitrile and 16.84% in chloroform. In contrast, [(2E)-1-phenyl-3-(11-piperidin-1-yl-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)prop-2-en-1-ylidene]malononitrile exhibited an absorption maximum at 607 nm with negligible fluorescence. The synthesized dyes may be of interest to researchers in the field of dye chemistry and related areas.

Keywords: xanthene-like dyes, fluorophore, Stokes shift, Knoevenagel reaction, quantum yield.

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Introduction

The xanthene framework is considered to be one of the most prevalent scaffolds in organic chemistry. Dyes based on the xanthene skeleton have found widespread application in diverse scientific and technological domains. These dyes have been utilized as reference dyes for determining the relative quantum yield of fluorescence [1], for the treatment and diagnosis of various diseases [2], for the detection of small molecules and radiotoxic compounds in various environments [3–6], and so on. This compendium is not an exhaustive enumeration of applications for xanthene dyes; rather, it is a focused analysis of the most significant domains in the authors' estimation. A review of the extant literature reveals that several of the most frequently used techniques for constructing xanthene frameworks have been described. In our opinion, these techniques are well illustrated in the work [7]. In all of these methods, the 9-position of the xanthene scaffold remains unfunctionalized, which significantly reduces the synthetic potential of such precursors. In previous studies, we developed fundamentally new approaches to the synthesis of functionalized derivatives of xanthene-like compounds

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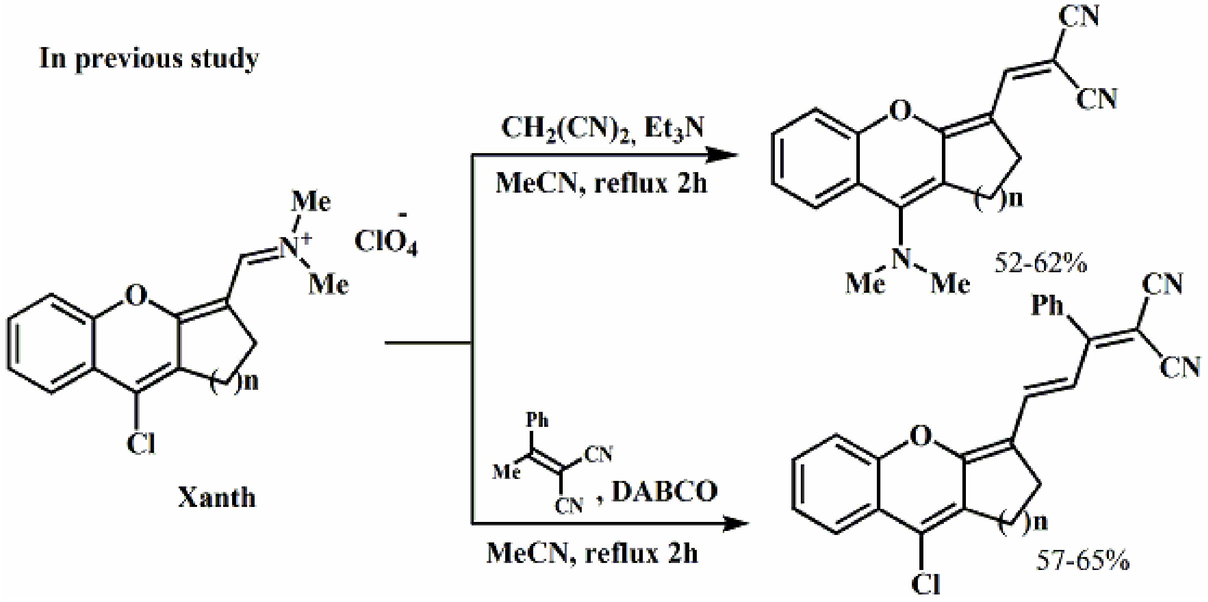
based on electrophilic rearrangements of geminal (O–C–O, O–C–N) heterocycles [8,9]. Consequently, when 1,3-dioxanes interact with the Vilsmeier-Haack reagent, the generation of promising building blocks is observed [10]. Subsequent to the identification of these precursors, a series of xanthene dyes were synthesized and their optical properties were examined [11–13]. The cited work demonstrates that when Xanth salts interact with specific active CH-acids, two distinct outcomes occur. Firstly, the dimethylimine substituent in the original xanthene dye is replaced by a CH-acid fragment. Secondly, the chlorine atom in position 9 is replaced by the released dimethylamine. Alternatively, a classic Knoevenagel reaction product is formed (Scheme 1).

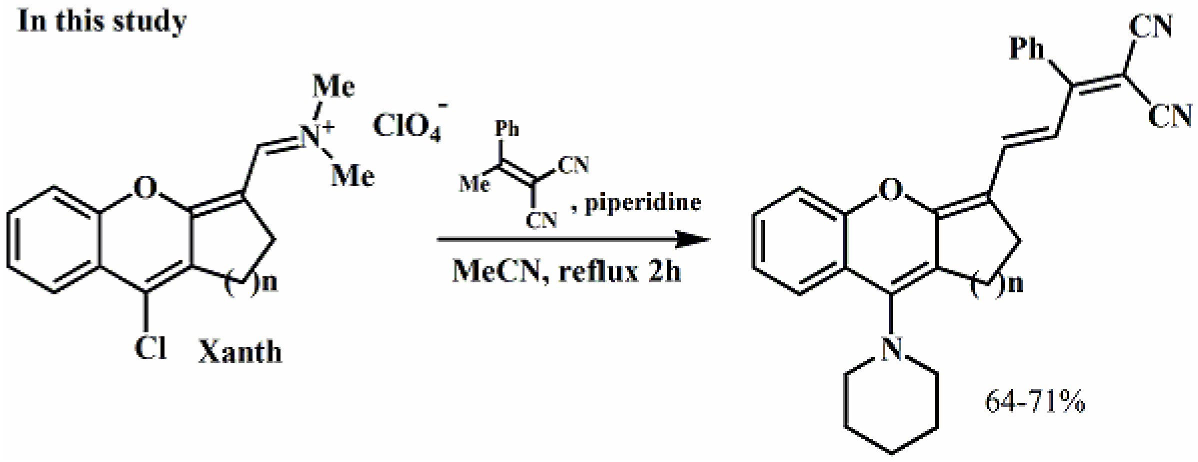
In the course of this research, a novel method for synthesizing xanthene-like dyes was developed. This method involves substituting the dimethyliminium substituent with a CH acid and the chlorine atom with a piperidine fragment, a process that occurs under the conditions of the Knoevenagel reaction. Additionally, the spectral characteristics of the obtained dyes were investigated (Scheme 2).

Experimental

Unless otherwise stated, all reagents of analytical grade were purchased from commercial suppliers and used without any further purification. The ^{1}H NMR spectra were recorded on a Bruker Avance II 400 spectrometer (400.13 MHz) in DMSO-d_6 using residual solvent peak as internal standard. The



Scheme 1. Xanth salts interact with certain active CH-acids



Scheme 2. Novel method for synthesizing xanthene-like dyes

EI mass spectra were recorded on a MX1321 instrument with direct sample inlet system, ionization voltage 70 V and ion source temperature of 220⁰C. Elemental analysis was performed on a LECO CHN-900 instrument. Melting points were determined using an Electrothermal 9100 Digital Melting Point apparatus and were uncorrected. The control of reactions and the purity of the synthesized compounds were monitored by TLC on Merck Silica gel 60 F-254 plates with 10:1, v/v $CHCl_3$/MeOH as eluent.

Absorption spectra were recorded in 1-cm quartz cells at 25⁰C using UV-2401 PC spectrophotometer (Shimadzu, Japan). To measure the extinction coefficients, each dye (7–10 mg) was dissolved in 50 cm^3 of the solvent. The resulting solution was diluted by 25–50 times and the absorption spectra were recorded in 5 cm quartz cuvettes. The extinction coefficients were calculated according to the Beer-Lambert law. Measurements were performed three to four times and the obtained data were averaged. The fluorescence measurements were performed in 1-cm standard quartz cells at 25⁰C using a Varian Cary Eclipse spectrofluorometer. The emission spectra were corrected for wavelength-dependent instrument sensitivity. Emission maxima were determined with an accuracy of ±1.0 nm. The standard 4-(dicyanomethylene)-2-methyl-6-(*p*-dimethylaminostyryl)-4*H*-pyran [14] was used for the measurement of fluorescence quantum yield.

General method

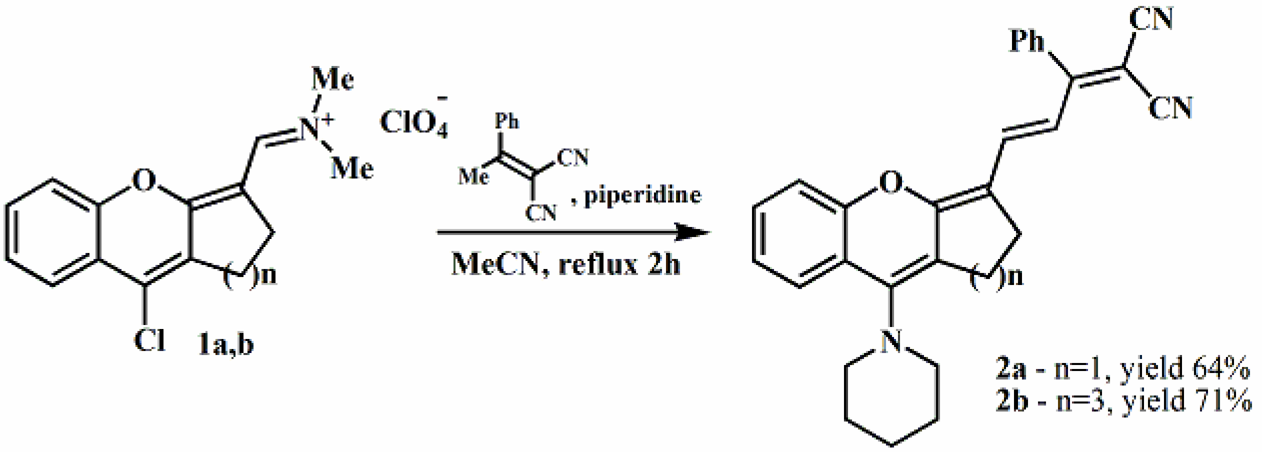
The corresponding perchlorate **1a,b** (1.3 mmol) was dissolved in 5 cm^3 boiling acetonitrile, then (1-phenylethylidene)malononitrile (0.22 g, 1.3 mmol) and piperidine (0.11 g, 1.3 mmol) were added. The reaction mixture was heated under reflux for 2 h. The solution was cooled to room temperature. The precipitate was collected by filtration and purified by recrystallization from MeCN.

[(2*E*)-1-phenyl-3-(9-piperidin-1-yl-1,2-dihydrocyclopenta[*b*]chromen-3-yl)prop-2-en-1-ylidene]malononitrile (2a). Yield 0.36 g (64%), green crystals, mp 235–237⁰C. ^{1}H NMR, δ, ppm (*J, Hz*): 7.56–7.57 (3H, m, H Ar); 7.49–7.50 (1H, m, H Ar); 7.40–7.37 (4H, m, H Ar); 7.25 (1H, t, ^3J=7.3, H Ar); 7.09 (1H, d, ^3J=8.3, H Ar); 6.91 (1H, d, ^3J=13.7, CH); 3.22 (4H, m, $2CH_2$); 3.08–3.11 (2H, m, CH_2); 2.70 (2H, m, CH_2); 1.67 (6H, m, $3CH_2$). Mass spectrum (EI), m/z (I_{rel}, %): 431 $[M]^+$ (91). Found, %: C 80.86; H 5.93; N 9.83. $C_{29}H_{25}N_3O$. Calculated, %: C 80.72; H 5.84; N 9.74.

[(2*E*)-1-phenyl-3-(11-piperidin-1-yl-7,8,9,10-tetrahydrocyclohepta[*b*]chromen-6-yl)prop-2-en-1-ylidene]malononitrile (2b). Yield 0.42 g (71%), green crystals, mp 203–204⁰C. ^{1}H NMR, δ, ppm (*J, Hz*): 7.59–7.64 (3H, m, H Ar); 7.51 (1H, d, ^3J=7.8, H Ar); 7.43–7.45 (3H, m, H Ar); 7.15–7.20 (1H, m, H Ar); 6.93 (1H, d, ^3J=14.7, CH); 6.59–6.67 (1H, m, H Ar); 6.52 (1H, d, ^3J=7.8, H Ar); 3.25 (2H, m, CH_2); 3.05 (2H, m, CH_2) 2.83 (2H, m, CH_2); 2.57 (4H, m, $2CH_2$); 1.76 (6H, m, $3CH_2$); 1.65 (2H, m, CH_2). Mass spectrum (EI), m/z (I_{rel}, %): 459 $[M]^+$ (11). Found, %: C 81.16; H 6.26; N 9.03. $C_{31}H_{29}N_3O$. Calculated, %: C 81.02; H 6.36; N 9.14.

Results and discussion

The reaction of perchlorates **1a,b** with (1-phenylethylidene)malononitrile in boiling acetonitrile, in the presence of an equimolar amount of piperidine, for a period of two hours resulted in the formation of dyes **2a,b** in satisfactory yields (Scheme 3).



Scheme 3. Preparation of piperidine-substituted dyes **2a,b**

The structures of products **2a** and **2b** were ascertained through the utilization of ^{1}H NMR spectroscopy and mass spectrometry. The molecular formula was confirmed by means of elemental analysis. The formation of the E-configuration of double bonds is indicated by the coupling constant values, which are 13.7 and 14.7 Hz for compounds **2a** and **2b**, respectively.

As demonstrated previously [12], the implementation of a comparable interaction, accompanied by the substitution of piperidine with DABCO, results in the formation of a xanthene dye, while preserving the chlorine atom within the heterocyclic fragment. In the same work, it was found that carrying out a reaction with malononitrile (malononitrile dimer) in the presence of triethylamine leads not only to a condensation product, but also to the replacement of the chlorine atom by a dimethylamino group. A thorough analysis of the obtained data indicates that the initial stage of the reaction is the Knoevenagel condensation, followed by the replacement of the chlorine atom by a piperidine fragment through the addition-elimination mechanism.

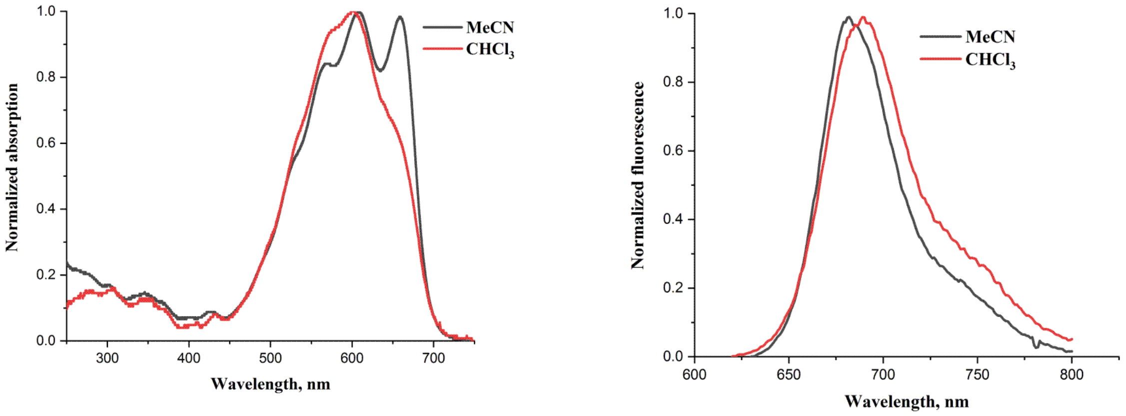
Spectral properties

The optical spectra of the obtained dyes were recorded in various organic solvents with a concentration of $5 \cdot 10^{-6}$ mol/L (Figure and Table). It was observed that dyes **2a,b** exhibited an absorption maximum of approximately 605 nm in acetonitrile and chloroform. Compound **2a** has an emission maximum at 682 nm and 689 nm in acetonitrile and chloroform, respectively, with relative fluorescence quantum yields of 34.62% and 16.84%. Compound **2b** exhibits virtually no fluorescence, a finding that aligns with our earlier observations on analogous molecular systems [12].

Dye **2a** exhibits substantial Stokes shifts of 1810 cm^{-1} and 2070 cm^{-1} in acetonitrile and chloroform, respectively. Furthermore, both dyes exhibit high absorption coefficients, as demonstrated in Table.

Conclusions

Thus, it was shown that the interaction of N-[(9-chloro-1,2-dihydrocyclopenta[b]chromen-3-yl)methylene]-N-methylmethanaminium and N-[(11-chloro-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)methylene]-N-methylmethanaminium perchlorates



a

b

Normalized spectra of **2a** absorption (a) and emission (b) in different organic solution

The spectral properties^a of dyes 2a,b

Compound	Solvent	λ_{max} (Abs), nm	λ_{max} (Em), nm	ε, M^{-1}·cm^{-1}	E$_g^{opt}$, eV^b	Φ_F, %	Stokes shift	
							nm	cm^{-1}
2a	MeCN	607	682	26819	1.79	34.62	75	1810
	CHCl$_3$	603	689	50371	1.77	16.84	86	2070
2b	MeCN	605	–	27350	1.75	–	–	–
	CHCl$_3$	608	–	47232	1.77	–	–	–

Notes: ^{a} – absorption ($\lambda_{Abs\text{-}max}$), emission ($\lambda_{Em\text{-}max}$) maxima and extinction coefficients (ε_{max}) were determined experimentally;
^{b} – E_g^{opt} is the optical energy gap of the HOMO-LUMO transition calculated on the basis of the wavelength of the red limit of intrinsic absorption [15].

with (1-phenylethylidene)malononitrile in boiling acetonitrile in the presence of an equimolar amount of piperidine yields [(2E)-1-phenyl-3-(9-piperidin-1-yl-1,2-dihydrocyclopenta[b]chromen-3-yl)prop-2-en-1-ylidene]malononitrile and [(2E)-1-phenyl-3-(11-piperidin-1-yl-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)prop-2-en-1-ylidene]malononitrile, respectively. The synthesized dyes were found to have an absorption maximum of ~605 nm in acetonitrile and chloroform. Only the dye with a five-membered partially saturated cycle, moderate quantum yields, and large Stokes shifts exhibited fluorescence.

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ДОЦІЛЬНИЙ ШЛЯХ СИНТЕЗУ ЗАМІЩЕНИХ АНАЛОГІВ КСАНТЕНУ

С.А. Варениченко, А.В. Ковтун, В.К. Фарат, О.К. Фарат

Здійснено дослідження реакцій *N*-[(9-хлоро-1,2-дигідроциклопента[*b*]хромен-3-іл)метилен]-*N*-метилметанамініум і *N*-[(11-хлоро-7,8,9,10-тетрагідроциклогепта[*b*]хромен-6-іл)метилен]-*N*- метилметанамініум перхлоратов з (1-фенілетиліден)малононітрилом у киплячому ацетонітрилі в присутності піперидину, що приводить до утворення нових органічних барвників, [(2*E*)-1-феніл-3-(9-піперидин-1-іл-1,2-дигідроциклопента[*b*]хромен-3-іл)проп-2-єн-1-іліден]малононітрил і [(2*E*)-1-феніл-3-(11-піперидин-1-іл-7,8,9,10-тетрагідроциклогепта[*b*]хромен-6-іл)проп-2-єн-1-іліден]малононітрил, з високими виходами. Структура продуктів була підтверджена за допомогою ЯМР ^{1}Н спектроскопії та мас-спектрометрії. За допомогою значень констант спін -спинового взаємодії встановлено, що у продуктах утворюються Е-конфігурації подвійних зв'язків. Аналіз даних показав, що реакція проходить через конденсацію Кньовенагеля, з подальшим заміщенням атома хлору піперидиновим фрагментом. Для отриманих барвників було проведено спектроскопічне дослідження в органічних розчинниках, зокрема ацетонітрилі та хлороформі. Максимум поглинання сполуки [(2*E*)-1-феніл-3-(9-піперидин-1-іл-1,2-дигідроциклопента[*b*]хромен-3-іл)проп-2-єн-1-іліден]малононітрилу становить ~605 нм, а випромінювання ~685 нм з квантовим виходом 34,62% в ацетонітрилі та 16,84% в хлороформі. Натомість [(2*E*)-1-феніл-3-(11-піперидин-1-іл-7,8,9,10-тетрагідроциклогепта[*b*]хро-мен-6-іл)проп-2-єн-1-іліден]малононітрил має максимум поглинання при ~607 нм в органічних розчинниках з незначною флуоресценцією. Запропоновані барвники можуть становити інтерес для фахівців в галузі барвників і суміжних галузей.

Ключові слова: ксантеноподібні барвники, флуорофори, зсув Стокса, реакція Кньовенагеля, квантовий вихід.

AN EFFICIENT SYNTHETIC ROUTE TO SUBSTITUTED XANTHENE ANALOGUES

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The reactions of N-[(9-chloro-1,2-dihydrocyclopenta[b]chromen-3-yl)methylene]-N-methylmethanaminium and N-[(11-chloro-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)methylene]-N-methylmethanaminium perchlorates with (1-phenylethylidene)malononitrile were studied in boiling acetonitrile in the presence of piperidine. These reactions led to the formation of new organic dyes, [(2E)-1-phenyl-3-(9-piperidin-1-yl-1,2-dihydrocyclopenta[b]chromen-3-yl)prop-2-en-1-ylidene]malononitrile and [(2E)-1-phenyl-3-(11-piperidin-1-yl-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)prop-2-en-1-ylidene]malononitrile, obtained in high yields. The structures of the products were confirmed by ^{1}H NMR spectroscopy, mass spectrometry, and elemental analysis. The observed spin-spin coupling constants indicated that the products adopted the E-configurations of the double bonds. The reaction mechanism was determined to proceed via a Knoevenagel condensation followed by nucleophilic substitution of chlorine with a piperidine fragment. Spectroscopic investigations of the dyes were performed in organic solvents, in particular acetonitrile and chloroform. For [(2E)-1-phenyl-3-(9-piperidin-1-yl-1,2-dihydrocyclopenta[b]chromen-3-yl)prop-2-en-1-ylidene]malononitrile, the absorption maximum was ~605 nm, and the emission maximum was ~685 nm, with a quantum yield of 34.62% in acetonitrile and 16.84% in chloroform. In contrast, [(2E)-1-phenyl-3-(11-piperidin-1-yl-7,8,9,10-tetrahydrocyclohepta[b]chromen-6-yl)prop-2-en-1-ylidene]malononitrile exhibited an absorption maximum at 607 nm with negligible fluorescence. The synthesized dyes may be of interest to researchers in the field of dye chemistry and related areas.

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