



# Thermosolvatochromic Behavior of Quinolinium and Pyridinium 4-Hydroxystyryl Dyes in Solutions

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## Abstract

In the current study a number of 4-hydroxystyryl dyes (4-HSD), 4-[-2-(4-hydroxy-3-methoxyphenyl)vinyl]-1-octylpyridinium bromide (4OP-MS) and 2-[-2-(3,5-dimethyl-4-hydroxystyryl)]-1-methylquinoline iodide (2MQ-DMS), were synthesized and characterized using NMR, IR and Raman spectroscopy methods. The solvatochromic and thermochromic properties of their merocyanine form in solutions (water, ethanol, propanol-2, butanol-1) were observed and studied via UV–Vis spectrophotometry and tristimulus colorimetry methods. The sensitivity of the studied representatives to temperature changes in the range of 20.0–70.0 °C showed opposite thermochromic effects in solutions. Thus, the 4OP-MS shows a bathochromic shift of the spectrum (up to 17 nm) with increasing temperature and a significant increase in intensity in alcohol media (up to 3.5%—without taking into account the thermal expansion of the solvent). Under similar conditions, the 2MQ-DMS shows a significant decrease in intensity, which reaches 44% in the case of butanol, with practically no shifts in the light absorption maximum with temperature. When passing from aqueous to propanol-2 solutions, the thermosolvatochromic effect in the case of 4OP-MS increases slightly from 108 to 113 nm with an increase in temperature by 50.0 °C, and for 2MQ-DMS, on the contrary, it decreases from 64 to 48 nm. These effects indicate, on the one hand, the possibility of using such dyes to create liquid thermochromic systems, and on the other hand, the need to take them into account when creating and using molecular optical sensors.

**Keywords** 4-hydroxystyryl dyes · Merocyanine dyes · Thermochromic properties in solution · Tristimulus colorimetry

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## 1 Introduction

Thermochromic compounds have significant potential for functional use in different areas as smart materials [1]. Some organic substances, which show reversibly thermochromic color changes, have different direct or indirect mechanisms. Heat is a trigger that can change the molecular structure and color, but after elimination of the source of heat, the system reverts to the thermally more stable form [2]. Studies have shown that 4-(*N*-alkyl)-pyridinium, 4-(*N*-alkyl)-quinolinium are sensitive to temperature changes. Usually, the development of thermochromic materials is aimed at creating solid or polymeric materials and much less attention is devoted to the study of thermochromic behavior in solutions [3–5]. Thermosolvatochromism of 7H-indolo[1,2-*a*]quinolinium dyes derivatives in pure solvents was studied in work [6]. The latter may come in handy in creating molecular probes and other means of analysis. Thermochromic properties materials used in food, textile, paint, cosmetic, pharmaceutical industry, medicine, etc. For example, in the production of indicator films for foodstuff, safety markers for banknotes, documents, sensors temperature to ensure safety and product quality control, biosensing systems [7–14].

4-Hydroxystyryl dyes (4-HSD) and related compounds have unique spectral-luminescent, solvatochromic and physicochemical properties [15–20]. Based on these properties of 4-HSD, highly sensitive methods for the determination of cyanides and biosensors for the detection of endogenes in living cells have been developed [21, 22]. Also, the spectrophotometric and tristimulus colorimetric methods have been proposed for the water impurities determination in organic solvents [23–25]. On the other hand, considerable attention is paid to the study of acid–base properties of 4-HSD [26–28]. The thermochromism of 4-HSD has not been studied in detail enough. Recently, much attention has been paid to finding new thermochromic materials, as well as the study of the dyes thermochromic properties in different nature solvents. The future looks bright for the development of novel microencapsulated thermochromic compounds possessing nanostructural derived properties that can be effectively used in inks, paints, and coating agents for sustainable energy efficiency and other applications. Thermochromic materials have received great attention by researchers in the past few years for their wide applications in modernization, urbanization, and also in the improvement of health care systems. However, several strategies can be applied in the improvement of such compounds properties to make them more effective. Several processes have been developed for the synthesis and microencapsulation in recent years, but further investigation is required to overcome the limitations of the methods in the pursuit of making them more efficient with increasing stability and durability, particularly from photodegradation and thermal degradation [9]. At the same time, no need to stop the study of new compounds that have thermochromic properties.

It should be noted that the tristimulus colorimetric method, which has successfully proven itself in the study of the acid–base properties of dyes, the development of quantitative and semi-quantitative methods for the different analytes determination, was not used to study the thermochromic properties of dyes [29–32].

This work is devoted to the study of the thermochromic properties of a number of new 4-hydroxystyryl dyes by spectrophotometry and tristimulus colorimetry methods.

## 2 Experimental Section

### 2.1 Chemicals Used

For thermochromic properties study, the  $10^{-3}$  mol·L<sup>-1</sup> solutions of merocyanine form of 4-HSD with organic solvent or distilled water were used. Some organic solvents, such as ethanol, butan-1-ol, propanol-2, were purchased from Sigma Aldrich and used without any additional purification. The following heterocyclic bases as 2-methylquinoline and 4-methylpyridine and alkyl halides as 1-octylbromide and iodomethane (Ukrorgsyntez) with the major compound content at least 95% were used as received. All other chemicals, used in the present study, were analytically pure grade.

### 2.2 Instrumentations

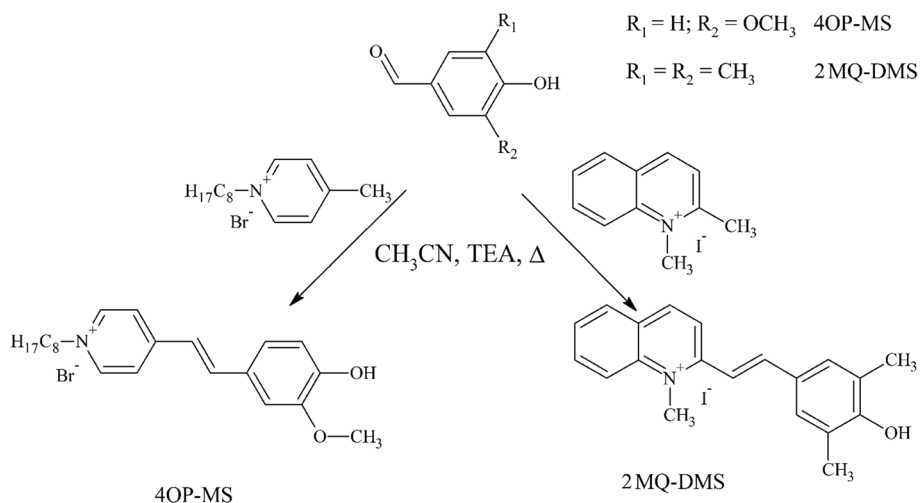
UV–Vis spectra were recorded in the range of 300–700 nm and for tristimulus colorimetry calculation in the visible range (380–780 nm). The light absorption spectra were recorded on Shimadzu UV-2600 spectrophotometer with TCC-100 temperature cell with temperature control accuracy of  $\pm 0.1$  °C. The measurements were performed at temperatures ranging from 20.0 to 70.0 °C with step of  $\Delta = 10.0$  °C. All measurements were repeated three times.

The color coordinates (CIE XYZ and CIE Lab) were calculated by the selected ordinates method based on the recorded absorption spectra [33]. All other color characteristics used in the work (saturation, yellowness index, CIE color difference) were calculated according to the recommendations set out in the work [34].

<sup>1</sup>H Nuclear magnetic resonance (NMR) spectra for synthesized dyes were obtained on Bruker Avance III 400 MHz NMR-spectrometer. Attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectra were recorded on an FT-IR-8400S IR spectrometer (Shimadzu) in the range 400–4000 cm<sup>-1</sup> on the set-top device Quest Specac with baseline correction. Raman spectra were obtained on DXR Raman Microscope (Thermo Scientific) under the following conditions: laser wavelength 780 nm, laser power 24 mW, full-band grating, Stokes range 200–3500 cm<sup>-1</sup>.

### 2.3 The 4-Hydroxystyryl Dyes Synthesis Protocols

The HSD, used in the work, were synthesized by reacting equivalent amounts of suitable 4-hydroxyaldehydes with corresponding quaternary ammonium salts of heterocyclic bases (4-methyl-1-octylpyridinium bromide and 1,4-dimethylquinolinium iodide) by boiling in acetonitrile in the presence of triethylamine according to the scheme:



The purity of the synthesized dyes was checked by thin-layer chromatography as described in the literature [25, 26]. The structures of these dyes were confirmed by  $^1H$  NMR-spectroscopy.

### 2.3.1 2-[2-(3,5-dimethyl-4-hydroxystyryl)]-1-methylquinoline iodide (2MQ-DMS)

$^1H$  NMR (500 MHz, Chloroform-d)  $\delta$ , ppm: 2.22 (s, 6 H), 4.50 (s, 3 H), 7.61 (s, 2 H), 7.66 (d,  $J=15.6$  Hz, 1 H), 7.89 (t,  $J=7.4$  Hz, 1H), 8,12 (d+t 15.6, 7.0 Hz, 2H), 8.29 (d,  $J=8.0$  Hz, 1 H), 8.51 (dd,  $J=9.2, 9.2$  Hz, 2 H), 8.93 (d,  $J=8.8$  Hz, 1 H), 9.22 (br. s., 1 H).

IR spectrum: 3356.3 (O–H), 3016.8 (C–H Ar), 1624.1 (C–C, Ht), 1585.5, 1570.1, 1523.8, 1485.2, 1419.7, 1315.5, 1296.2, 1230.6, 1188.2, 1153.5, 1024.2, 972.2, 943.2, 844.9, 746.5, 569.0, 495.7, 462.9, 449.3, 422.4  $cm^{-1}$ .

Raman spectrum: 1618.3 (C–C, Ht), 1580.7, 1518.0, 1487.1, 1469.8 (C–H, Alk), 1420.6 (C–C, Ar), 1383.9 (C–H, Alk), 1357.9, 1311.7, 1237.4, 1182.4, 1153.5, 1130.4, 1111.1, 799.6, 540.3, 523.9, 472.8, 458.3, 445.8, 249.1, 236.5  $cm^{-1}$ .

### 2.3.2 4-[2-(4-hydroxy-3-methoxyphenyl)vinyl]-1-octylpyridinium bromide (4OP-MS)

$^1H$  NMR (400 MHz, DMSO-d<sub>6</sub>)  $\delta$ , ppm: 0.84 (t,  $J=7$  Hz, 3H), 1.14–1.34 (m, 10H), 1.88 (qu, 2H), 3.85 (s, 3H), 4.45 (t,  $J=7$  Hz, 2H), 6.86 (d,  $J=8$  Hz, 1H), 7.18 (d,  $J=7$  Hz, 1H), 7.34(d,  $J=16.1$  Hz, 1H), 7.36 (s, 1H), 7.94 (d,  $J=16.2$  Hz, 1H), 8.13 (d,  $J=6.5$  Hz, 2H), 8.87 (d,  $J=6$  Hz, 2H).

IR spectrum: 3336.5 (O–H), 2924.2 (C–H, Ar), 1643.4, 1612.5 (C–C, Ht), 1585.5, 1516.1, 1465.9, 1383.0, 1280.8, 1207.5, 1157.3, 1124.5, 1033.9, 979.9, 850.6, 815.9, 690.5, 617.2, 601.8, 561.3, 528.5, 520.8, 486.1, 455.2, 418.6  $cm^{-1}$ .

Raman spectrum: 1611.5 (C–C, Ht), 1583.6, 1557.5, 1514.1, 1467.9 (C–H, Alk), 1451.5 (C–C, Ar), 1384.9 (C–H, Alk), 1325.1, 1310.7, 1277.9, 1257.7, 1238.4, 1206.6, 1181.5, 1158.3, 1123.6 (C–O–C), 1046.5, 979.9, 601.9  $cm^{-1}$ .

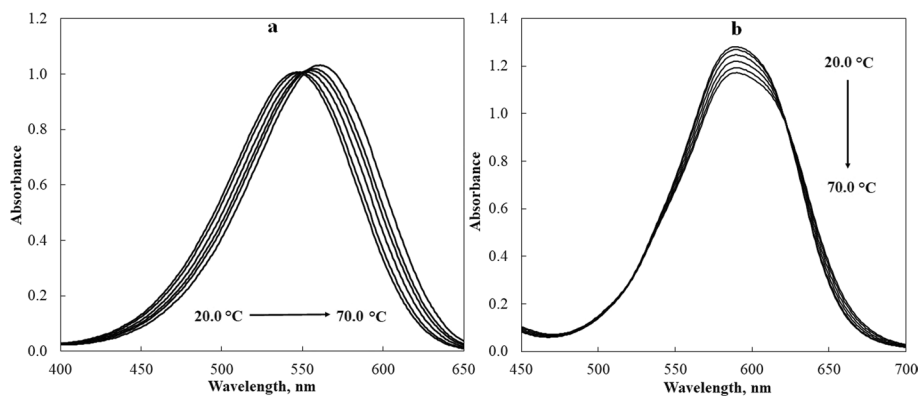
## 2.4 Thermosolvatochromism Study

0.2 mL of a solution of merocyanine form of 4-HSD with a concentration of  $10^{-3}$  mol·L<sup>-1</sup> prepared in ethanol was added to the test tube, mixed with 10 mL of lower alcohol or water. The solution transferred to the quartz cuvette closed Teflon cap, thermostated to a certain temperature in the range from 20.0 to 70.0 °C and light absorption spectra were recorded. For thermosolvatochromic studies, water and lower alcohols were chosen as solvents since they are less toxic.

## 3 Results and Discussion

It is known that 4-HSD are capable of reversible color change with changing pH and solvent polarity [35]. In this work, the main attention is paid to the thermochemical properties of some 4-HSD. The influence of temperature on the absorbance spectra of 4OP-MS and 2MQ-DMS dyes was studied at temperatures ranging from 20.0 to 70.0 °C (Fig. 1).

As can be seen from the UV–Vis spectra (Fig. 1), the compounds 4OP-MS and 2MQ-DMS are sensitive to temperature changes. It can be noted that in the case of manifestation of only thermal expansion of the solvent for most dissolved substances, one would expect to obtain of a hypochromic effect with increasing temperature. However, the nature of changes in the absorption intensity at the maximum for both dyes is opposite in direction and weakly related to the thermal expansion of the solvent. Thus, with increasing temperature, an increase in the light absorption intensity is observed for the 4OP-MS dye with a simultaneous bathochromic shift of the light absorption maximum (bathochromic-hyperchromic effect). And for 2MQ-DMS solutions, with a similar change in temperature, there is almost no bathochromic shift of the maximum of the spectrum (with the exception of water), however, there is an opposite decrease in the intensity of light absorption, which significantly outweighs the effects of changes in volume due to thermal expansion of the solvent, which for of alcohols is 9–11% when the temperature increases by 100 °C, while the corresponding value of deviations increases in the series: "water–ethanol–propanol-2–butanol-1".



**Fig. 1** UV–Vis absorption spectra of 4OP-MS (a) and 2MQ-DMS (b) in ethanol solution measured at different temperatures with the step of 10.0 °C;  $C_{\text{Dye}} = 0.2 \times 10^{-5}$  mol·L<sup>-1</sup>;  $l = 1$  cm

The positions of the long-wave absorption band maximum and molar absorptivity of these dyes measured in studied solvents are summarized in Table 1.

In the absence of thermochromic effects of substances, with an increase in temperature, one should expect a decrease of absorbance solutions due to thermal expansion, and the corresponding calculated values for the selected temperature interval ( $\Delta t = 50.0\text{ }^\circ\text{C}$ ) would be about 2% for water and 6–7.5% for the studied alcohols.

The influence of temperature on "solute–solvent" systems is usually complex in nature, firstly, when the temperature changes, there is a change in the viscosity, density, dielectric constant and other integral and specific properties of the solvent (e.g. acid–base), and secondly, the temperature affects ground and excited energy states of the solute and structure of the molecule/solvates. Which processes play a dominant role in relation to changes in the light absorption spectra of a solute is caused by the change in the structure of solvates or the solute in general. In the case of 4OP-MS, most likely, thermochromic effects are caused by changes in the dielectric constant of the medium, which causes a redistribution of the electron density in the molecule (solvate), and as a result of which a bathochromic-hyperchromic effect is observed, and the hypochromic effect of the 2MQ-DMS dye is most likely, associated with structural changes.

For a more detailed study of the thermochromic properties of 4-HSD, colorimetric approaches were used. In general, the dependences of the value of studied colorimetric functions on temperature are similar for both studied dyes. For example, Fig. 2 a, b shows the dependencies of changing the color coordinates (CIELab) for 4OP-MS,  $l = 1\text{ cm}$ .

It should be concluded that on the presented dependences one feature should be noted. In the case of a highly polar solvent (water), the dependence of the color coordinate on temperature changes direction: an increase in temperature leads to an increase in the value of the color parameter (Fig. 2a). When considering the second color coordinate of the CIELab space, a sharp change in the polarity of the medium also leads to a different course of dependences: in a highly polar solvent, the value of the color coordinate  $b$  takes positive values.

For a more detailed study of the thermochromic properties of 4-HSD, other color parameters should be used. Previously, to study the effect of pH on the state of dyes, including 4-HSD, the parameter specific color discrimination ( $SCD$ ) was proposed (Eq. 1). In this paper, it was decided to calculate this parameter in the form (Eq. 2), and the corresponding graphical dependences of the change in color saturation  $S$  and the  $SCD$  parameter are shown in Fig. 3.

$$SCD = \frac{\Delta S}{\Delta pH} \quad (1)$$

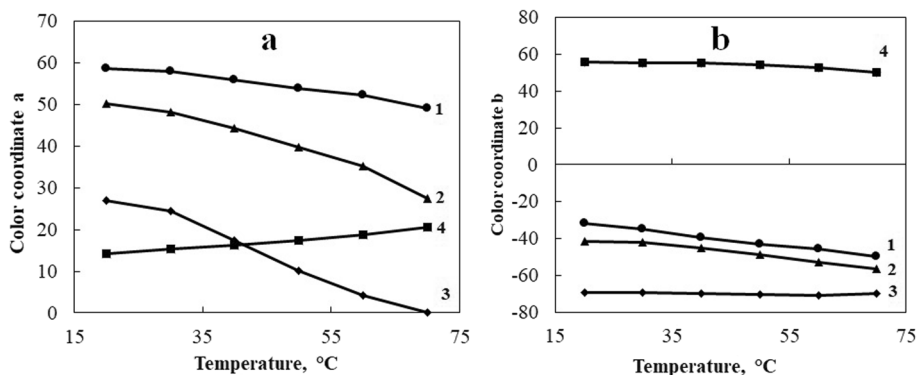
$$SCD = \frac{\Delta S}{\Delta T} \quad (2)$$

where  $SCD$ —specific colour discrimination;  $S'$ ,  $S''$ —colour saturation of the studied solutions at  $pH'$  and  $pH''$  (or at temperatures  $T'$  and  $T''$ ) in accordance,  $\Delta pH = pH' - pH''$ ;  $\Delta T = T' - T''$ ;  $\Delta S = |S' - S''|$ .

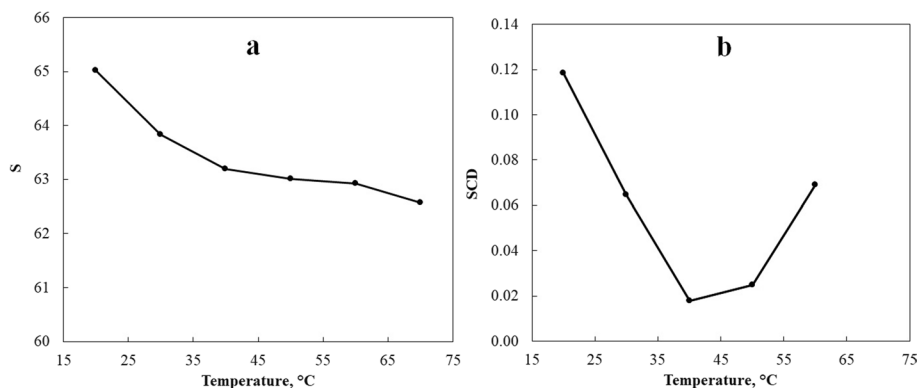
As can be seen from Fig. 3a, the color saturation curve from  $pH$  is similar to other colorimetric functions (Fig. 2), but one can note some change in the course of the curves around  $40\text{ }^\circ\text{C}$ . In its turn, its derivative (Fig. 3b) has a clear break at about  $40\text{ }^\circ\text{C}$ . This behavior of 4-HSD can be explained as follows. It is known that 4-HSD and related compounds are capable of forming J and H-aggregates in solutions, and the formation of the latter was

**Table 1** Positions ( $\lambda$ , nm) of absorption band maximum and molar absorptivity ( $\epsilon$ ) of 4OP-MS and 2MQ-DMS dyes in different solvents

Solvents	20.0 °C		30.0 °C		40.0 °C		50.0 °C		60.0 °C		70.0 °C	
	$\epsilon \times 10^4$	$\lambda$ , nm	$\epsilon \times 10^4$	$\lambda$ , nm	$\epsilon \times 10^4$	$\lambda$ , nm	$\epsilon \times 10^4$	$\lambda$ , nm	$\epsilon \times 10^4$	$\lambda$ , nm	$\epsilon \times 10^4$	$\lambda$ , nm
<b>4OP-MS</b>												
Water	3.38	471	3.34	472	3.31	473	3.28	474	3.25	475	3.20	477
Ethanol	5.04	547	5.04	549	5.04	551	5.07	555	5.09	557	5.17	561
Butanol	4.93	557	4.82	560	4.84	563	4.91	566	5.02	570	5.10	574
Propanol-2	6.74	579	6.70	580	6.72	583	6.75	586	6.77	586	6.62	590
<b>2MQ-DMS</b>												
Water	3.82	524	3.81	526	3.80	529	3.79	531	3.77	535	3.73	539
Ethanol	6.40	589	6.35	589	6.23	590	6.10	590	5.97	590	5.86	590
Butanol	6.01	592	5.73	592	5.14	592	4.51	591	3.78	591	3.36	591
Propanol-2	5.79	588	5.61	588	4.89	591	5.22	587	4.88	587	4.63	587



**Fig. 2** Changes in CIELab color coordinates *a* (a) and *b* (b) of the 4OP-MS dye in ethanol (1), butanol (2), 2-propanol (3) and water (4) at different temperatures;  $C_{\text{Dye}} = 0.2 \times 10^{-5} \text{ mol}\cdot\text{L}^{-1}$ ;



**Fig. 3** Changes in saturation parameters (a) and specific color discrimination (b) of the 4OP-MS dye in butanol at different temperatures;  $C_{\text{Dye}} = 0.2 \times 10^{-5} \text{ mol}\cdot\text{L}^{-1}$ ;  $l = 1 \text{ cm}$

noted by us earlier [36]. Since H-aggregates are formed due to the formation of hydrogen bonds between dye molecules, an increase in temperature should lead to the breaking of such bonds, and a bathochromic shift of the absorption band should be expected in the absorption spectra, which is observed in the systems under study (Fig. 1a). Tristimulus colorimetry method allows fixing the temperature at which H-aggregates are destroyed. It is interesting to note that in more polar solvents (for example, in water), sharp changes in colorimetric characteristics are shifted to higher temperatures (50.0 °C), which is probably due to the possibility of solvation due to the competitive formation of hydrogen bonds and the redistribution of electron density in the 4-HSD molecule and smoother change of colorimetric parameters. In less polar solvents (butanol), H-aggregates are destroyed at lower temperatures (of the order of 30.0 °C), and fewer opportunities for the formation of hydrogen bonds with the solvent leads to a sharper change in colorimetric characteristics. On the other hand, possible reasons for this behavior can include both, photoinduced *cis/trans* isomerization of the dye or probe structure- and solvent-dependent contributions of the quinonoid and zwitterionic limiting formulas to the ground and excited states of the probe [37].



## 4 Conclusion

4-HSD have unique protolytic, solvatochromic, thermochromic, colorimetric and other properties. The influence of temperature on the absorbance spectra of 4OP-MS and 2MQ-DMS dyes was studied in the temperatures ranging from 20.0 to 70.0 °C by spectrophotometry and tristimulus colorimetry methods. A bathochromic shift of the maximum light absorption at 6/11/14/17 nm in aqueous/isopropanol/ethanol/butanol solutions, respectively dye (OP-MS) was observed with the increasing temperature for octylpyridinium-3-methoxy-4-hydroxystyryl. At the same time expressed by increasing the intensity of color. In contrast to OP-MS for methylquinolinium-3,5-dimethyl-4-hydroxystyryl dye (2MQ-DMS) hypochromic effect was observed with the increasing temperature. For 2MQ-DMS dye with the increasing solution temperature by 50.0 °C, the absorbance of butanol dye solutions decreased almost twice. The dependences of the value of studied colorimetric functions on temperature are similar for both studied dyes. As follows from the studies, such multi-informative dyes can be used as chemosensors.

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**Author contributions** Yuliya Zhukova - spectrophotometric studies of dyes; Yaroslav Studenyak - synthesis of dyes; Ruslan Marychuk - IR and Raman spectroscopic study of dyes; Denys Snigur - studies by tristimulus colorimetry. All authors wrote the main manuscript text and reviewed the manuscript.

## Declarations

**Conflict of Interest** The authors declare no conflict of interest.

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