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Solvatochromism of dyes. Part IV—Energetic characteristics of merocyanine derivatives of 1-phenyl-2-[2-(3-X-4-hydroxy-5-R-phenyl)ethenyl]-3,3-dimethyl-3*H*-indolium cation

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ABSTRACT: The fundamental energetic parameters (intercepts and slopes of straight lines approximating energetic levels) of solvatochromic betaines generated by deprotonation of 2-(4-hydroxystyryl)-3*H*-indolium salts were determined from solvatochromic data in one-component solvents. It was demonstrated that the solvatochromism of the investigated dyes can be described by the VBHB (vinylogous amide–betaine–hydrogen-bonded betaine) physical model. In comparison with similar dyes, the ability to form hydrogen-bonded solvates is weaker owing to the higher acidity of the dye precursor used. The type of observed solvatochromism was determined. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: solvatochromic dyes; VBHB model of solvatochromism; 3*H*-indoliummerocyanines

INTRODUCTION

Observable changes in the shape and position of absorption bands result from energy differences between the ground and excited states of the chromogene.[1] They are caused by one or more types of intermolecular interactions.[2] The interactions between the dissolved substance (solute) and the solvent and their intensity are determined by the polarity of the solvent, which can be expressed in terms of various scales, depending on the type of phenomena considered.[2–6] So far, the most often used is the $E_T(30)$ solvent scale proposed by Dimroth and Reichardt.[7,8] $E_T(30)$ polarity values have been determined for more than 360 single solvents.[9]

Absence of linearity in the plots of the position of the maximum absorption band versus solvent polarity has been observed for many types of solvatochromic dyes and especially for 7*H*-indolo[1,2-a]quinolinium oxymerocyanines.[10–12] The phenomenon was explained by the Sorokas' physical VBHB (vinylogous amide–betaine–hydrogen-bonded betaine) model of solvatochromism.[13] This well-working model assumed that solvated merocyanines exist in the two or three electronic ground states whereas there is only one lowest electronic excited state, common to those ground states. Theoretically, in solution these structures exist in equilibrium, but in fact one is dominant. Both structures are observed only at intersection points of terms (energetic levels, see Fig. 1). The solvatochromism is taken into account in the model as the dependence of the equilibria between these ground states on the solvent polarity.

EXPERIMENTAL

Instruments and laboratory equipment

A Specord M40 computer-controlled spectrophotometer (Carl Zeiss, Jena, Germany) modified by Medson Electronics (Poznań-Paczkowo, Poland), Bruker DPX400 NMR spectrometer (400 MHz, ^{1}H; 100.58 MHz, ^{13}C), a Corning Checkmate deluxe pH-meter, 1 cm gas-tight quartz cells, automatic 250, 500 and 2000 μl pipettes from Plastomed and a liquid 10 μl syringe from Hamilton (Bonaduz, Switzerland) were used.

Software

The Medson M48 set of programs for spectra processing (Medson Electronics) and home-written software (Turbo

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Table 1. Locations of maximum of the solvatochromic bands of merocyanine dyes obtained in selected solvents

Solvent	Polarity ($E_T(30)$) (kJ mol^{-1})	$\bar{\nu}_{max}$ (1000 cm^{-1})					
		b1	**b2**	**b3**	**b4**	**b5**	**b6**
n-Hexane	129.5	19.931	20.024	20.146	20.110	—[b]	—[b]
Cyclohexane	130.8	19.891	19.838	19.966	19.937	18.124	18.197
Carbon tetrachloride	136.2	19.490	19.449	19.544	19.524	17.838	17.847
Benzene	144.5	19.196	19.188	19.206	19.257	17.503	17.542
Anisole	155.8	18.075	18.130	18.793	18.790	17.230	17.234
Dichloromethane	172.2	17.611	17.623	17.634	17.630	17.151	17.137
Acetone	176.8	17.959	17.954	18.003	18.001	17.457	17.453
N,*N*-Dimethylformamide	183.5	17.662	17.644	17.676	17.663	17.329	17.318
Acetonitrile	192.8	17.744	17.733	17.757	17.751	17.421	17.400
2-Propanol	203.7	17.499	17.513	17.492	17.513	17.463	17.412
1-Propanol	212.5	17.364	17.371	17.351	17.362	17.455	17.425
Ethanol (95%)	224.8[a]	17.365	17.364	17.348	17.354	17.538	17.500
Di(ethylene glycol)	225.4	17.427	17.437	17.421	17.428	17.380	17.357
Methanol	232.6	17.415	17.398	17.400	17.383	17.728	17.681
Ethylene glycol	235.9	17.255	17.231	17.240	17.218	17.676	17.622
3-Hydroxypropionitrile	249.5	17.330	17.296	17.315	17.284	17.787	17.746
Water-d_2	262.9	17.772	17.738	17.756	17.733	18.650	18.618
2,2,2-Trifluoroethanol	263.6[a]	17.742	17.378	17.717	17.694	18.410	18.349
Water	264.74	17.753	17.724	17.748	17.719	18.533	18.483
1,1,1,3,3,3-Hexafluoro-2-propanol	273.4	18.649	18.595	18.651	18.660	19.357	19.336

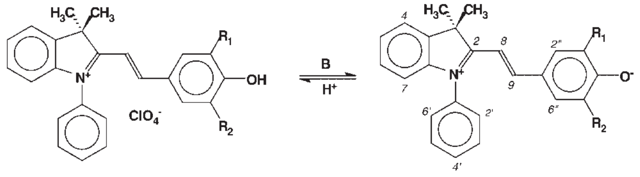
[a] Experimental value.
[b] Insufficient solubility.

Basic) for data processing according to the VBHB model were used.

Chemicals

The following were used: distilled water, Merck Uvasol grade solvents for spectroscopy or spectrofluorimetry (see Table 1), dimethyl sulfoxide for spectroscopy (Merck Uvasol), pure anhydrous potassium carbonate (POCh, Gliwice, Poland) and precursors **a** of solvatochromic dyes **b** (Scheme 1), 1-phenyl-2-[2-(3-R_1-4-hydroxy-5-R_2-phenyl)ethenyl]-3,3-dimethyl-3*H*-indolium perchlorates (prepared in our laboratory[15]). All new compounds had the expected NMR spectra and satisfactory elemental analyses.

a, **b**	R_1	R_2
1 [11]	Br	OCH_3
2	Br	OC_2H_5
3	Cl	OCH_3
4	Cl	OC_2H_5
5 [11]	NO_2	OCH_3
6	NO_2	OC_2H_5



a *(precursor)* **b** *(solvatochromic dye)*

Scheme 1

Dye **a1**: $C_{25}H_{23}NClBrO_6 = 548.82$; %C calc./exp. 54.71/54.65; %H calc./exp. 4.22/4.29; m.p. 216–219 °C; pK_a 4.47. ^{1}H NMR ($CDCl_3$/TMS; δ, ppm; J, Hz): 2.02 (s, 6H, CH_3), 3.94 (s, 3H, R_2:CH_3), 6.72 (d, 1H, $J = 16.05$, H8), 6.99 (d, 1H, $J = 7.95$, H4), 7.31 (s, 1H, H6″), 7.37 (s, 1H, H2″), 7.46 (t, 1H, $J = 7.64$, H6), 7.58 (t, 1H, $J = 7.41$, H5), 7.62 (d, 1H, $J = 6.01$, H7), 7.62–7.85 (m, 5H, H2′–H6′), 8.26 (d, 1H, $J = 16.08$, H9). ^{13}C NMR (δ, ppm): 26.87, 52.83, 56.87, 109.69, 110.28, 113.42, 114.93, 122.97, 126.42, 126.45, 128.16, 129.43, 129.78, 131.33, 132.22, 132.89, 142.31, 142.69, 148.5, 150.98, 155.98, 183.32.

Dye **a2**: $C_{26}H_{25}NClBrO_6 = 562.84$; %C calc./exp. 55.48/55.51; %H calc./exp. 4.48/4.50; m.p. 236–238 °C; pK_a 4.55. ^{1}H NMR ($CDCl_3$/TMS; δ, ppm; J, Hz): 1.46 (t, 3H, $J = 6.57$, R_2:CH_3), 2.02 (s, 6H, CH_3), 4.17 (q, 2H, $J = 6.25$, R_2:CH_2), 6.71 (d, 1H, $J = 16.01$, H8), 7.01 (d, 1H, $J = 7.89$, H4), 7.42 (s, 1H, H6″), 7.48 (t, 1H, $J = 7.19$, H6), 7.49 (t, 1H, $J = 7.58$, H5), 7.57–7.85 (m, 5H, H2′–H6′), 7.61 (s, 1H, H2″), 7.7 (d, 1H, $J = 7.3$, H7), 8.25 (d, 1H, $J = 15.97$, H9). ^{13}C NMR (δ, ppm) 14.54, 26.85, 52.71, 65.51, 109.48, 110.39, 113.84, 114.90, 122.95, 126.31, 126.35, 128.44, 129.45, 129.76, 131.30, 132.21, 132.83, 142.27, 142.53, 147.81, 151.38, 155.90, 183.10.

Dye **a3**: $C_{25}H_{23}NCl_2O_6 = 504.37$; %C calc./exp. 59.54/59.44; %H calc./exp. 4.60/4.66; m.p. 210–212 °C. pK_a 4.22. ^{1}H NMR ($CDCl_3$/TMS; δ, ppm; J, Hz): 2.01 (s, 6H, CH_3), 3.94 (s, 3H, R_2:CH_3), 6.73 (d, 1H, $J = 16.11$, H8), 6.99 (d, 1H, $J = 8.01$, H4), 7.19 (d, 1H, $J = 1.66$, H6″), 7.31 (d, 1H, $J = 1.69$, H2″), 7.45 (t, 1H, $J = 7.45$, H6), 7.57 (t, 1H, $J = 7.42$, H5), 7.61

(d, 1H, $J = 7.09$, H7), 7.63–7.84 (m, 5H, H2′–H6′), 8.27 (d, 1H, $J = 16.12$, H9). ^{13}C NMR (δ, ppm): 26.84, 52.89, 56.91, 109.89, 113.13, 114.94, 121.33, 122.96, 124.82, 125.86, 126.43, 129.40, 129.78, 131.32, 132.19, 132.90, 142.30, 142.77, 148.70, 149.64, 156.17, 183.46.

Dye **a4**: $C_{26}H_{25}NCl_2O_6 = 518.39$; %C calc./exp. 60.24/60.21; %H calc./exp. 4.86/4.88; m.p. 242–243 °C; pK_a 4.36. ^{1}H NMR (CDCl$_3$/TMS; δ, ppm; J, Hz): 1.45 (t, 3H, $J = 6.78$, R$_2$:CH$_3$), 2.01 (s, 6H, CH$_3$), 4.19 (q, 2H, $J = 6.7$, R$_2$:CH$_2$), 6.72 (d, 1H, $J = 16.08$, H8), 7.0 (d, 1H, $J = 7.95$, H4), 7.23 (s, 1H, H6″), 7.27 (s, 1H, H2″), 7.47 (t, 1H, $J = 7.75$, H6), 7.58 (t, 1H, $J = 7.48$, H5), 7.61 (d, 1H, $J = 6.06$, H7), 7.62–7.85 (m, 5H, H2′–H6′), 8.26 (d, 1H, $J = 16.11$, H9). ^{13}C NMR (δ, ppm): 14.60, 26.87, 52.83, 65.62, 109.72, 113.82, 114.95, 121.50, 122.98, 124.93, 125.76, 126.39, 129.45, 129.81, 131.34, 132.23, 132.88, 142.29, 142.67, 148.07, 150.05, 156.16, 183.31.

Dye **a5**: $C_{25}H_{23}N_2ClO_8 = 514.92$; %C calc./exp. 58.31/58.37; %H calc./exp. 4.50/4.55; m.p. 204–207 °C; pK_a 3.61. ^{1}H NMR (CDCl$_3$/TMS; δ, ppm; J, Hz): 2.04 (s, 6H, CH$_3$), 3.94 (s, 3H, R$_2$:CH$_3$), 6.84 (d, 1H, $J = 16.36$, H8), 7.08 (d, 1H, $J = 7.75$, H4), 7.44 (d, 1H, $J = 1.55$, H6″), 7.54 (t, 1H, $J = 7.65$, H6), 7.66 (t, 1H, $J = 7.26$, H5), 7.67–7.87 (m, 5H, H2′–H6′), 7.78 (d, 1H, $J = 7.49$, H7), 8.10 (d, 1H, $J = 1.61$, H2″), 8.47 (d, 1H, $J = 16.24$, H9). ^{13}C NMR (δ, ppm): 25.56, 52.12, 56.11, 110.44, 114.46, 120.54, 122.18, 123.57, 124.07, 125.52, 128.69, 129.28, 130.35, 131.47, 131.89, 135.35, 141.51, 141.96, 148.91, 149.79, 153.79, 182.57.

Dye **a6**: $C_{26}H_{25}N_2ClO_8 = 528.95$; %C calc./exp. 59.04/58.98; %H calc./exp. 4.76/4.82; m.p. 223–225 °C; pK_a 3.52. ^{1}H NMR (CDCl$_3$/TMS; δ, ppm; J, Hz): 1.43 (t, 3H, $J = 6.91$, R$_2$:CH$_3$), 2.0 (s, 6H, CH$_3$), 4.13 (q, 2H, $J = 6.9$, R$_2$:CH$_2$), 6.8 (d, 1H, $J = 16.32$, H8), 7.03 (d, 1H, $J = 8.05$, H4), 7.43 (d, 1H, $J = 1.45$, H6″), 7.49 (t, 1H, $J = 7.71$, H6), 7.61 (t, 1H, $J = 8.35$, H5), 7.64 (d, 1H, $J = 6.19$, *7*), 7.69–7.83 (m, 5H, H2′–H6′), 7.99 (d, 1H, $J = 1.54$, H2″), 8.36 (d, 1H, $J = 16.32$, H9). ^{13}C NMR (δ, ppm): 14.43, 26.41, 53.12, 65.82, 111.79, 115.38, 116.99, 120.42, 123.03, 124.79, 126.32, 129.55, 130.26, 131.22, 132.35, 132.69, 135.52, 142.26, 142.88, 149.82, 149.96, 154.52, 183.63.

Measurement procedure

The solutions were prepared directly in spectrophotometric cells by adding 4 μl of liquid concentrate of dye (10% by weight in anhydrous DMSO) to 2 ml of solvent. The betaine form of dye **b** was generated *in situ* by alkalizing solution of dye **a** with anhydrous potassium carbonate. Spectra were recorded in range 12 000–27 000 cm^{-1} at 25 ± 1 °C using 20 solvents of polarities from 129.5 to 273.4 kJ mol^{-1} according to the Dimroth–Reichard $E_T(30)$ scale. Values of pK_a were determined in aqueous solution at 20 °C, according to our previously described procedure.[11]

RESULTS AND DISCUSSION

VBHB model

Solvatochromism described by VBHB model is illustrated in Fig. 1.

The physical VBHB model of solvatochromism[13] leads to a mathematical equation describing relation between the position of the maximum of the absorption band $\bar{\nu}_i$ (cm^{-1}) and the solvent polarity π (kJ mol^{-1}) as follows:

$$\bar{\nu}_i = \frac{f_1\chi_1 \exp\left(\frac{\Delta E_{21}}{kT}\right) + f_2\chi_2 + f_3\chi_3 \exp\left(\frac{\Delta E_{23}}{kT}\right)}{\chi_1 \exp\left(\frac{\Delta E_{21}}{kT}\right) + \chi_2 + \chi_3 \exp\left(\frac{\Delta E_{23}}{kT}\right)} \qquad (1)$$

where

$$\begin{aligned} f_1 &= a_1 + b_1\pi \\ f_2 &= a_1 - a_{02} + (b_1 - b_{02})\pi \\ f_3 &= a_1 - a_{03} + (b_1 - b_{03})\pi \\ \Delta E_{21} &= [a_{02} - a_{01} + (b_{02} - b_{01})\pi]hc \\ \Delta E_{23} &= [a_{02} - a_{03} + (b_{02} - b_{03})\pi]hc \end{aligned}$$

a_1, a_{01}, a_{02}, a_{03} are intercepts (see Fig. 1, E_1, E_{01}, E_{02}, E_{03}) expressed in cm^{-1} and b_1, b_{01}, b_{02}, b_{03} are slopes (E_1, E_{01}, E_{02}, E_{03}), expressed in mol cm^{-1} (kJ)$^{-1}$. $\chi_i = \varepsilon_i/\sigma_i^2$ is a band shape parameter, in which ε_i denotes the decimal coefficient of molar extinction (l mol^{-1} cm^{-1}) and σ_i is the band half-width (cm^{-1}).

Owing to the mathematical properties of the VBHB model equation [Eqn (1)] χ_i can be expressed as their relative values χ_i, i.e. $\chi_i' = \chi_i/\chi_1$ or $\chi_i/\sum\chi_i$, which means that values of dye concentrations are unnecessary.

The type of solvatochromism can be defined with theoretical curves generated by Eqn (1). It is possible to find 32 types of solvatochromism. Nine of them are the most common (Fig. 2).[14,15]

If N, P and Z denote negative, positive or no (zero) solvatochromism, the most common can be named N, P, PP, PPN, PNN, NN, PZN, PZ and ZN. Positive solvato-

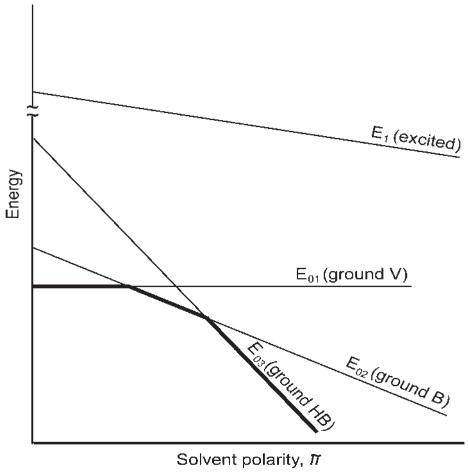


Figure 1. Illustration of fundamental assumption in the VBHB model of merocyanine solvation

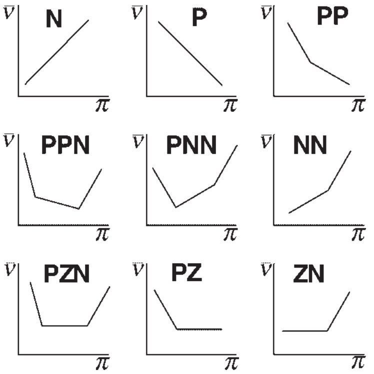


Figure 2. Common types of solvatochromism illustrated by location of the last absorption band, expressed as wavenumber $\bar{\nu}$ versus solvent polarity π

chromism (type P) is exhibited by dyes for which the low-energy absorption band shifts towards longer wavelengths (batochromic or red shift) with increase in solvent polarity. In type N solvatochromism, the trend of changes is the opposite (hypsochromic or blue shift). A stable location of the last absorption band during solvent polarity changes means zero solvatochromism (type Z).

Determination of energetic parameters of 3*H*-indoliummerocyanines

In order to determine fundamental energetic parameters describing series of merocyanine-like dyes, investigations in one-component solvents were carried out using the theoretical VBHB approach. As a dye, 1-phenyl-2-[2-(3-X-5-R-phenolates-4)ethenyl]-3,3-dimethyl-3*H*-indolium, generated by deprotonation of corresponding hydroxyhemicyanines, was used.

The spectrum shape and location of long-wavelength absorption band changes of merocyanine/betaine dye **b1** in solvents with different polarity are presented in Fig. 3.

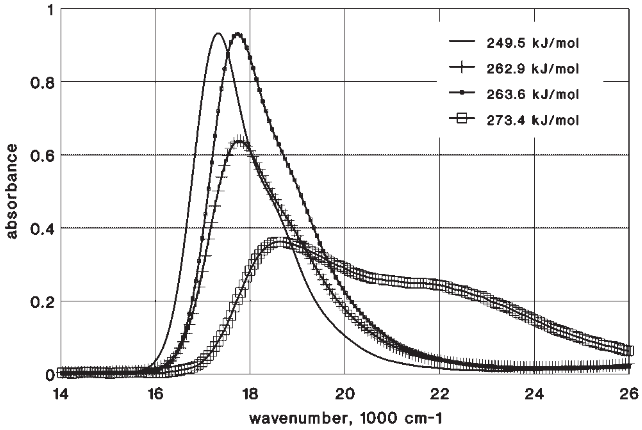


Figure 3. Influence of solvent polarity on UV–visable absorption spectra of betaine **b1**. Solvents used: 3-hydroxypropionitrile (249.5 kJ mol^{-1}), water-d_2 (262.9 kJ mol^{-1}), 2,2,2-trifluoroethanol (263.6 kJ mol^{-1}) and 1,1,1,3,3,3-hexafluoro-2-propanol (273.4 kJ mol^{-1}) (see key)

Table 2. Evaluated intercepts a_i and slopes b_i of terms according to the VBHB model[a]

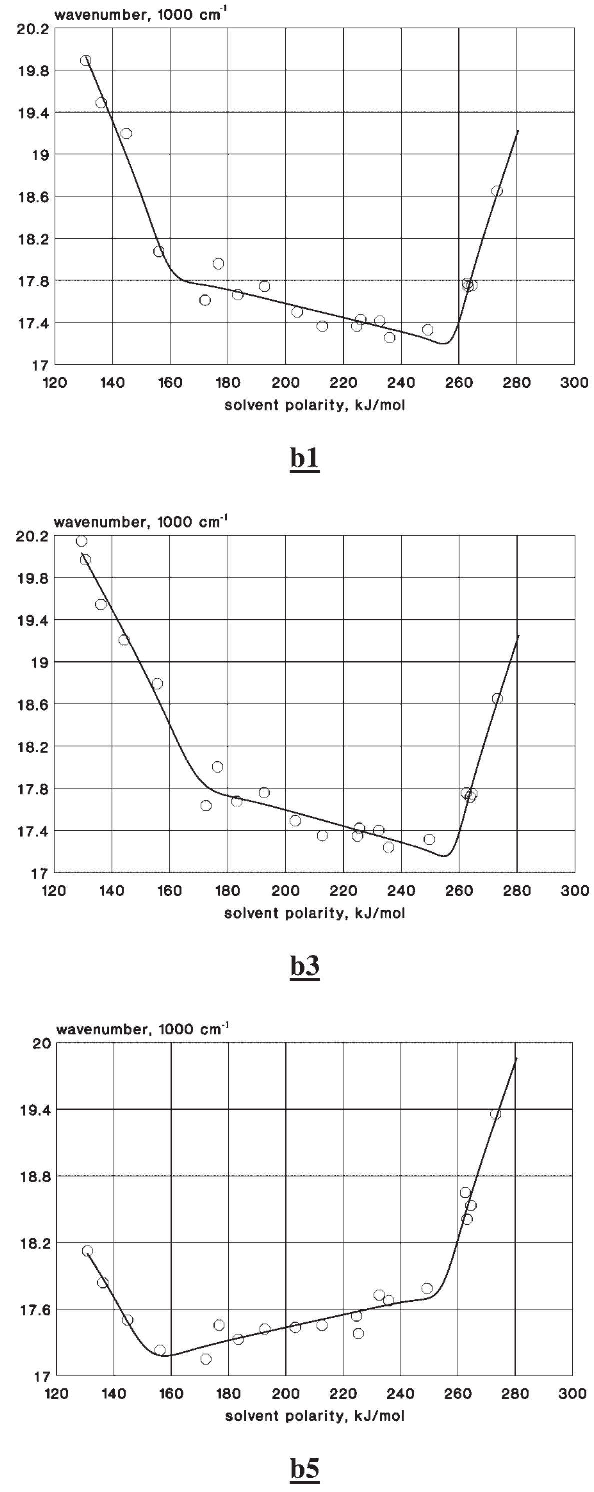
Dye	a_1[b]	b_1[c]	a_{02}[b]	b_{02}[c]	a_{03}[b]	b_{03}[c]	s[b]	r	Randomness[d] (%)
b1	28512.84 ± 0.23	$-65.651 \pm 5.2 \times 10^{-4}$	9591.88 ± 0.07	$-58.947 \pm 4.7 \times 10^{-3}$	32789.99 ± 0.26	$-148.371 \pm 1.2 \times 10^{-3}$	122	0.9910	55.0
b2	28142.41 ± 0.22	$-63.049 \pm 4.9 \times 10^{-4}$	9152.87 ± 0.07	$-55.995 \pm 4.4 \times 10^{-4}$	31812.85 ± 0.25	$-144.454 \pm 1.1 \times 10^{-3}$	114	0.9938	57.1
b3	26587.17 ± 0.25	$-50.596 \pm 4.8 \times 10^{-4}$	7445.94 ± 0.07	$-42.287 \pm 4.1 \times 10^{-4}$	31186.65 ± 0.29	$-135.599 \pm 1.3 \times 10^{-3}$	131	0.9927	57.1
b4	26425.19 ± 0.23	$-49.499 \pm 4.6 \times 10^{-4}$	7247.00 ± 0.06	$-41.574 \pm 3.7 \times 10^{-4}$	31945.99 ± 0.28	$-137.908 \pm 1.2 \times 10^{-3}$	123	0.9931	57.1
b5	22989.41 ± 0.29	$-37.246 \pm 4.6 \times 10^{-4}$	6699.41 ± 0.08	$-42.975 \pm 5.4 \times 10^{-4}$	24188.38 ± 0.31	$-112.323 \pm 1.4 \times 10^{-3}$	111	0.9863	50.0
b6	23397.61 ± 0.28	$-39.949 \pm 4.7 \times 10^{-4}$	7015.71 ± 0.08	$-45.111 \pm 5.3 \times 10^{-4}$	25381.08 ± 0.31	$-117.816 \pm 1.4 \times 10^{-3}$	116	0.9848	50.0

[a] Calculated for $\chi_1, \chi_3 = 1$ and $\chi_2 = 3$.

[b] Expressed in cm^{-1}; these values are apparent, because zero polarity means that $E_T(30) = 125$ kJ mol^{-1}.

[c] Slopes of terms, expressed in mol cm^{-1} kJ^{-1}.

[d] Measure of randomness in scattering of experimental points around estimated line.



b1

b3

b5

Figure 4. Changes in locations of the longwave length absorption bands of betaines **b1**, **b3** and **b5** as a function of solvent polarity, expressed on the $E_T(30)$ scale. Solvents used are presented in Table 1

Subtraction of background absorption (spectrophotometric cell and solvent used) from the spectrum of the dye solution and precise location of the absorption maximum were executed using Medson M48 software. Locations of the maximum of the solvatochromic bands ($\bar{\nu}_{\max}$) obtained in this way are given in Table 1.

The numerical values in Table 1 were then processed according to the VBHB theoretical model using home-written software. First the VBHB non-linear equation [Eqn(1)], describing the $\bar{\nu}$–solvent polarity relationship was fitted to the experimental data. In this equation, intercepts and slopes of terms are expressed as fitted parameters. Arbitrarily, intercept a_{01} and slope b_{01} of the betaine term were taken as zero.

Comparison of the experimental data (circles) with the theoretical curve fitted with the VBHB model (curves) is shown in Fig. 4.

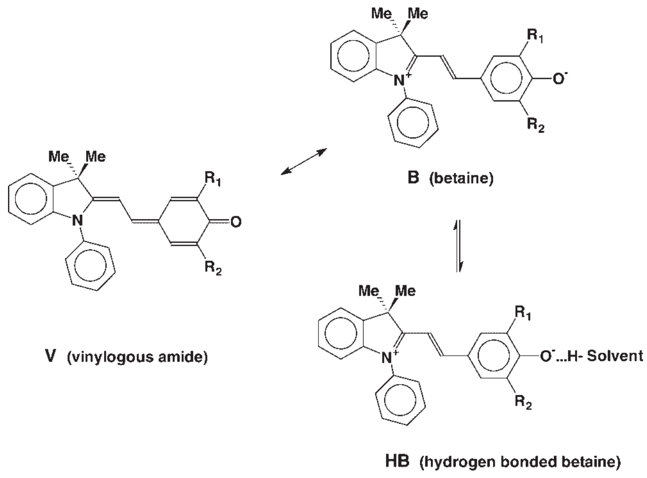
Evaluated by the non-linear least-squares method, regression coefficients of the theoretical VBHB equation applied in the figures are given in Table 2. The set of data for each dye consists of 20 experimental points, which means that 14 degrees of freedom giving credible statistical characteristics are saved.

High values of the correlation coefficients and very low standard deviations with relatively high number of degrees of freedom indicate that the experimental data comply with the VBHB model. Dyes **b5** and **b6** represent solvatochromism of the PNN type. In this case, when the aprotic solvent polarity increases, a bathochromic shift is observed. This means that the electronic excited state of dye is more strongly solvated than the ground state.

In protic solvents of high polarity or medium polarity aprotic solvents a hypsochromic shift is observed. In this case a dye in its electronic ground state is more strongly solvated and better stabilized than in the excited state.

In contrast, dyes **b1**–**b4** exhibit solvatochromism of the PPN type. This means that in the region of medium polarities an increase in polarity makes the solvation of the excited-state dye more effective.

With the use of the most polar solvent (higher than water), 1,1,1,3,3,3-hexafluoro-2-propanol, it was demonstrated that it complies with the same mechanism of solvatochromism as described for earlier derivatives of



Scheme 2

the system.[13] Three structures of merocyanines are presented on Scheme 2.

Because 3*H*-indoliummerocyanine precursors are about 100 times more acidic than the corresponding 7*H*-indolo[1,2-a]quinolinium compounds,[11] the ability to undergo hydrogen bond formation is limited in this former case. The result is that the betaine form (**B**) is stable over wide range of solvent polarity, so that the third structure (**HB**) appears only in highly polar solvents.

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