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Tuning reverse ring closure in the photochromic and thermochromic transformation of 1′,3′,3′-trimethyl-6-nitrospiro[2*H*-1-benzopyran-2,2′-indoline] analogues by ionic moieties

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Abstract

The photochromism and thermochromism of eight 1′,3′,3′-trimethyl-6-nitrospiro[2*H*-1-benzopyran-2,2′-indoline] analogues bearing cationic, neutral and anionic moieties were examined in acetonitrile at 318 K. The rate constants of forward colouration (k_1) and reverse ring closure (k_{-1}) were evaluated. The transition energy (E_T) of the merocyanines and k_{-1} are strongly correlated with the ionic charge of the substituent moiety and with the solvent polarity. A linear correlation ($\ln k_{-1} = \alpha E_T + \beta$) was observed for the series of merocyanines in acetonitrile (similar to that obtained on changing the solvent). The slope α obtained in the present case was more than threefold steeper than that observed for the conventional solvent effect; k_1 was almost constant regardless of the solvent or ionic charge. The energy gap between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) in the coloured form and the formation energy of the transition state in the reverse reaction were calculated. Both increased in the same order of ionic charge: $-1 < +1$. The influence of the ionic substituent is significant in the reverse ring closure reaction due to its ionic recombination character, but small in the forward electrocyclic C–O bond rupture. © 1997 Elsevier Science S.A.

Keywords: Coulombic effect; Merocyanine; Photochromism; Thermochromism; Spiropyran

1. Introduction

The chemistry of spiropyrans has been studied extensively, with special regard to the remarkable photochromic properties associated with photochemical C–O bond rupture at the spiro position. Spiropyrans exhibit two discrete, interconvertible spectra in the UV–visible region corresponding to the closed spiro form (**I**) and the open merocyanine form (**II**) (Fig. 1). The open form exists as the zwitterionic structure (**II**) rather than the quinoid structure (**II′**) even in nonpolar solvents [1], so that the reverse ring closure follows an ionic recombination scheme. A correlation of the reaction rate parameters with the solvent polarity [1] and the Hammett constants of the substituents [2] has been demonstrated. Coulombic interaction in structure **II** contributes to the stability and interconversion of photomerocyanine isomers [3]. In this paper, we report the photochromic and thermochromic processes of eight 1′,3′,3′-trimethyl-6-nitrospiro[2*H*-1-benzopyran-2,2′-indoline] analogues possessing various ionic substituents. Strong inhibition of the backward reaction is demonstrated when a cationic moiety is attached to the ortho position (8*H*) to phenolate O^-. The ionic substituent effect is three times more effective than the known solvent effect as described below, and provides an active method of tuning and designing new chromic molecules.

2. Experimental details

UV–visible spectra were obtained using an HP8452A diode array spectrophotometer at a regulated temperature of 318 ± 0.1 K. One of the emission lines from an Hg lamp (254 nm, 365 nm and 440 nm) was used to generate the open merocyanines. All solvents were of spectrograde and were used as supplied. MM2, ZINDO and MOPAC/PM3 calculations were carried out with the CAChe satellite program. Spiropyrans **2**–**7** were synthesized as described below, and their structures were confirmed by ^{1}H NMR spectroscopy and elemental analysis (EA). Spiropyrans **1** (1′,3′,3′-trimethyl-6-nitrospiro[2*H*-1-benzopyran-2,2′-indoline], Tokyo Kasei Co., SU grade) and **8** (1′-carboxyethyl-3′,3′-trimethyl-6-

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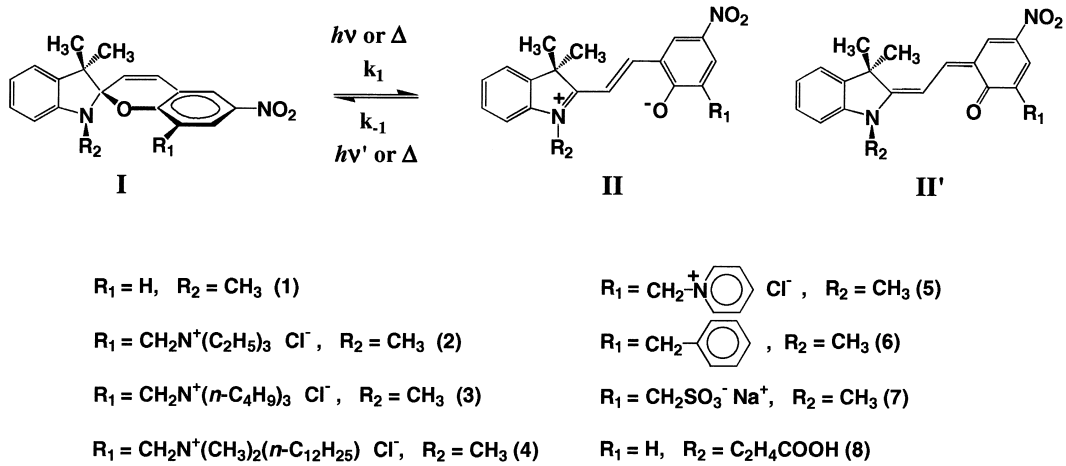

Fig. 1. Photochemical and thermal transformation of 1′,3′,3′-trimethyl-6-nitrospiro[2*H*-1-benzopyran-2,2′-indoline] analogues.

nitrospiro[2*H*-1-benzopyran-2,2′-indoline], Nihon Chemics) were recrystallized before use.

*2.1. 1′,3′,3′-Trimethyl-6-nitro-8-trimethylammoniomethylspiro[2H-1-benzopyran-2,2′-indoline] chloride (**2**)*

A solution of 500 mg (1.30 mmol) of 1′,3′,3′-trimethyl-6-nitro-8-chloromethylspiro-[2*H*-1-benzopyran-2,2′-indoline] (**9**) [4] and 2.0 ml of triethylamine (14 mmol) in 20 ml of chloroform was heated at reflux for 6 h under a nitrogen atmosphere. After the solvent had been removed, the residual solid was reprecipitated repeatedly from a cyclohexane–dichloromethane mixture, chromatographed on TOYOPEARL® HW40C (Toso) with ethanol as eluent and vacuum dried. Yield based on **9**, 16%. Melting point (m.p.), 393 K. EA: found (calculated for $C_{26}H_{34}O_3N_3Cl\cdot1.5H_2O$): C, 62.7 (62.6); N, 7.80 (8.42); H, 7.23 (7.47); Cl, 6.16 (7.10). ^{1}H NMR ($CDCl_3$, ppm) δ: 1.32 (m, 15H, $N^+(CH_2CH_3)_3$ and indoline $C(CH_3)_2$), 2.69 (d, 3H, N–CH_3), 3.10–3.36 (m, 6H, $N^+(CH_2CH_3)_3$), 4.39–4.85 (d, 2H, Ph–CH_2–N^+), 6.01 (d, 1H, NO_2Ph–C*H*=CH), 6.53 (d, 1H, NO_2Ph–CH=C*H*), 6.92–7.21 (m, 4H, indoline Ph*H*), 8.14–8.38 (d, 2H, NO_2Ph*H*).

*2.2. 1′,3′,3′-Trimethyl-6-nitro-8-tributylammoniomethylspiro[2H-1-benzopyran-2,2′-indoline] chloride (**3**)*

Compound **9** (500 mg; 1.30 mmol) and tri-*n*-butylamine (300 mg; 1.60 mmol) were dissolved in 5 ml of methanol, and the solution was heated at reflux for 6 h under a nitrogen atmosphere. The solvent was removed and the residual solid was reprecipitated repeatedly from a cyclohexane–dichloromethane mixture. The solid was purified by TOYOPEARL® HW40C gel chromatography and dried in vacuum. Yield based on **9**, 17%. M.p., 378 K. EA: found (calculated for $C_{32}H_{46}O_3N_3Cl\cdot H_2O$): C, 66.9 (66.9); N, 7.28 (7.32); H, 8.07 (8.43); Cl, 6.17 (6.17). ^{1}H NMR ($CDCl_3$, ppm) δ: 0.81–1.25 (m, 27H, $N^+(CH_2CH_2CH_2CH_3)_3$ and indoline $C(CH_3)_2$), 2.69 (s, 3H, N–CH_3), 3.32 (m, 6H, $N^+(CH_2)_3$), 4.53–5.04 (d, 2H, Ph–CH_2–N^+, 6.02 (d, 1H, NO_2Ph–C*H*=CH), 6.57 (d, 1H, NO_2Ph–CH=C*H*), 6.92–7.24 (m, 4H, indoline Ph*H*), 8.15–8.40 (d, 1H, NO_2Ph*H*).

*2.3. 1′,3′,3′-Trimethyl-6-nitro-8-dimethyldodecylammoniomethylspiro[2H-1-benzopyran-2,2′-indoline] chloride (**4**)*

Compound **9** (500 mg; 1.30 mmol) and dimethyl-*n*-dodecylamine (340 mg; 1.60 mmol) were dissolved in 5.0 ml of methanol and refluxed for 6 h under a nitrogen atmosphere. After the methanol had been removed, the residue was reprecipitated repeatedly from a cyclohexane–dichloromethane mixture. The precipitate was purified by TOYOPEARL® HW40C gel chromatography with ethanol as eluent and vacuum dried. Yield based on **9**, 45%. M.p., 376 K. EA: found (calculated for $C_{34}H_{50}O_3N_3Cl\cdot0.5H_2O$): C, 68.5 (68.8); N, 6.96 (7.08); H, 8.70 (8.66); Cl, 5.59 (5.98). ^{1}H NMR ($CDCl_3$, ppm) δ: 0.89–1.24 (m, 29H, N^+–$CH_2C_{11}H_{23}$ and indoline $C(CH_3)_2$), 2.71 (s, 3H, N–CH_3), 2.87–3.26 (m, 8H, N^+–$CH_2C_{11}H_{23}$ and $N^+(CH_3)_2$), 4.37–5.05 (d, 2H, Ph–CH_2–N^+), 6.03 (d, 1H, NO_2Ph–C*H*=CH), 6.53 (d, 1H, NO_2Ph–CH=C*H*), 6.91–7.21 (m, 4H, indoline Ph*H*), 8.15–8.56 (d, 2H, NO_2Ph*H*).

*2.4. 1′,3′,3′-Trimethyl-6-nitro-8-pyridiniomethylspiro-[2H-1-benzopyran-2,2′-indoline] chloride (**5**)*

Compound **9** (150 mg; 0.40 mmol) and 1.0 ml of pyridine were added to 5.0 ml of chloroform, and the mixture was heated at reflux for 6 h under a nitrogen atmosphere. The solvent was evaporated, and the residue was reprecipitated repeatedly from a cyclohexane–dichloromethane mixture. The precipitated solid was filtered, washed with diethylether and dried in vacuum. Yield based on **9**, 83%. EA: found (calculated for $C_{25}H_{24}O_3N_3Cl\cdot H_2O$): C, 64.5 (64.2); N, 8.89 (8.98); H, 5.34 (5.60); Cl, 8.85 (7.58). ^{1}H NMR ($CDCl_3$, ppm) δ: 1.05 (d, 6H, indoline C–CH_3), 2.51 (s, 3H, N–CH_3), 4.56–5.30 (d, 2H, Ph–CH_2–N^+), 5.90 (d, 1H, NO_2Ph–C*H*=CH), 6.57 (d, 1H, NO_2Ph–CH=C*H*), 6.95 (m,

4H, indoline Ph*H*), 7.60–8.32 (m, 5H, pyridinium *H*), 8.73–8.75 (d, 2H, NO_2Ph*H*).

2.5. 1′,3′,3′-Trimethyl-6-nitro-8-phenoxymethylspiro-[2H-1-benzopyran-2,2′-indoline] (6)

To a mixture of 1.33 g of phenol (13.0 mmol) and 0.56 g of 25% tetramethylammonium hydroxide methanol solution (1.5 mmol), a solution of **9** (500 mg in 10 ml of tetrahydrofuran (THF)) was added dropwise. The solution was refluxed for 3 h under a nitrogen atmosphere. After removing the solvent, the residue was dissolved in ethylacetate, worked up with 5% aqueous NH_4Cl and saturated aqueous NaCl solutions and dried by $MgSO_4$. The organic layer was concentrated, chromatographed on silica gel and dried in vacuum. Yield based on **9**, 16%. EA: found (calculated for $C_{26}H_{24}O_4N_2 \cdot 0.5H_2O$): C, 71.1 (71.4); N, 6.17 (6.40); H, 5.75 (5.76). ^{1}H NMR ($CDCl_3$, ppm) δ: 1.25 (m, 6H, indoline C–C*H*$_3$), 2.73 (s, 3H, N–C*H*$_3$), 4.80 (s, 2H, NO_2Ph–C*H*$_2$), 5.90 (d, 1H, NO_2Ph–C*H*=CH), 6.53 (d, 1H, NO_2Ph–CH=C*H*), 7.00 (m, 9H, indoline Ph*H* and phenoxy *H*), 8.00–8.26 (d, 2H, NO_2Ph*H*).

2.6. 1′,3′,3′-Trimethyl-6-nitrospiro[2H-1-benzopyran-2,2′-indoline]-8-methanesulphonate sodium salt (7)

A solution of 500 mg of **9** (1.30 mmol) and 270 mg of $NaHSO_3$ (2.60 mmol) in 10 ml of 60% aqueous ethanol was refluxed for 7 h. After evaporation, the residue was chromatographed on TOYOPEARL® HW40C with 60% aqueous ethanol as eluent and dried in vacuum. Yield based on **9**, 44%. EA: found (calculated for $C_{20}H_{19}O_6N_2SNa \cdot 4H_2O$): C, 47.3 (47.1); N, 5.41 (5.49); H, 4.30 (5.33).

3. Results and discussion

Thermally equilibrated solutions of **1** exhibit colourless UV absorption corresponding to the closed form **I**. Excitation of the UV absorption band leads to photochemical C–O bond rupture of **I**, followed by rapid transformation to **II**, with a characteristic absorption increase in the visible region. The coloured zwitterionic state **II** represents a fluxional molecule composed of eight conformational isomers. They are abbreviated as CCC, CCT, CTC, CTT, TCC, TCT, TTC and TTT, where T and C represent trans and cis with regard to the two C=C double bonds in the chromophoric conjugation from indoline N to phenolate O. Photochemically generated merocyanine **1-II** undergoes a thermal ring closure reaction to produce the original colourless state. Non-ionic **6** and anionic **7** and **8** show the same trend. MM2 calculations and low-temperature experiments have revealed that the stability of the merocyanine isomers decreases in the order CTT > TTT > CTC > TTC > CCT > TCT > CCC > TCC [5]. In homogeneous dilute solution at room temperature, interconversion between the merocyanine isomers is very fast and the rate-determining process will be the final C–O bond formation. The merocyanine form of spiropyrans **1**, **6**, **7** and **8** is only generated by appropriate UV light irradiation. In contrast, the cationic spiropyrans **2**, **3**, **4** and **5** are thermochromic, forming coloured open species at room temperature in the dark. As a working hypothesis, we assume that such a thermochromic nature is intrinsic in all the spiropyrans investigated. When thermal equilibrium is attained between **I** and **II**, and interconversion between the merocyanine isomers is sufficiently rapid, we obtain the following equation for the thermochromic process

$$K_{eq} = k_1/k_{-1} = c_{\mathbf{II}}/c_{\mathbf{I}} = [(\epsilon_{\mathbf{II}} c_0 l/A_{\mathbf{II}}) - 1]^{-1} \quad (1)$$

where K_{eq} is the equilibrium constant, k_1 is the forward reaction rate constant, k_{-1} is the reverse reaction rate constant, c_0 is the analytical concentration of spiropyran, $c_{\mathbf{I}}$ is the equilibrium concentration of **I**, $c_{\mathbf{II}}$ is the equilibrium concentration of **II**, $\epsilon_{\mathbf{II}}$ and $A_{\mathbf{II}}$ are the molar absorption coefficient and observed optical density at the visible absorption maximum (λ_{max}) respectively and l is the optical path length. The equilibrium can be shifted to a new state by appropriate photoirradiation. The direction of the shift depends on the compound and the light wavelength. When irradiation is stopped, the solution returns thermally to the original state. For this type of relaxation process, Eq. (2) can be applied

$$A_t = (A_0 - A_\infty)\ \exp[-(k_1 + k_{-1})t] + A_\infty \quad (2)$$

where A_t, A_0 and A_∞ are the optical densities at time t, zero and infinity respectively. In Fig. 2(a) and Fig. 2(b), the observed relaxation processes and generated non-linear least-squares fitting results are shown. All data can be reasonably expressed by a monoexponential fit and the values of $k_1 + k_{-1}$ can be obtained. Assuming that $\epsilon_{\mathbf{II}} = 35\,000\ M^{-1}\ cm^{-1}$ for all the spiropyrans [1], we obtained K_{eq}, k_1 and k_{-1}.

In Table 1, the reaction parameters and spectroscopic properties are shown. The λ_{max} value of the coloured form varies depending on the molecular structure, particularly on the ionic charge; the transition energy (E_T) decreases in the order of the charge of the substituents (+1 > 0 > −1). A linear correlation between E_T and Dimroth's $E_T(30)$ parameter has been obtained for many spiropyrans. The most plausible electronic structure of merocyanines is the zwitterionic structure, which is easily perturbed by solute–solvent interactions. Since the E_T values of the merocyanines reflect their electronic states, charge distribution and solvation, they can be used as an index of the change in the ring closure reactivity of the merocyanine vs. the solvent polarity. A linear free energy correlation

$$\ln k_{-1} = \alpha E_T + \beta \quad (3)$$

has been proposed [6] as an expression of the solvent effect. In Fig. 3, $\ln k_{-1}$ is plotted vs. E_T for the various spiropyrans in acetonitrile. The plots follow Eq. (3), although a variation in E_T occurs due to the changing ionic charge. From least-squares fitting, $\alpha = -0.59\ (kJ/mol)^{-1}$ and $\beta = 1.2 \times 10^2$ were obtained for the ionic substituent effect. We also exam-

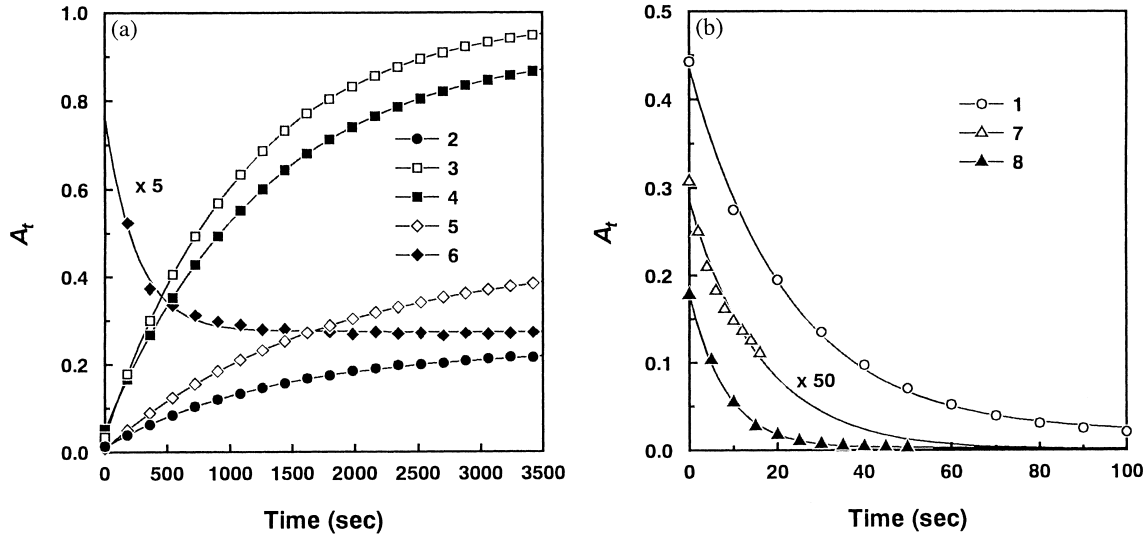

Fig. 2. Thermal decay/growth profiles of open merocyanines **1**–**8**. Lines were obtained by non-linear, least-squares fitting based on Eq. (2) (see text).

Table 1
Reaction parameters of reverse ring closure for various spiropyrans [a]

Compound	$10^2 K_{eq}$	k_1+k_{-1} (s^{-1})	k_{-1} (s^{-1})	k_1 (s^{-1})	λ_{max} (nm)	E_T (kJ mol^{-1})	E_{LUMO} (eV) [b]	E_{HOMO} (eV) [b]	ΔE [c] (eV)	H [d] (kJ mol^{-1})
1	0.82	4.2×10^{-2}	4.2×10^{-2}	3.4×10^{-4}	558	214.4	−1.76	−7.09	5.34	203
2	33	7.3×10^{-4}	5.5×10^{-4}	1.8×10^{-4}	540	221.5	−3.85	−9.83	5.98	747
3	160	9.1×10^{-4}	3.5×10^{-4}	5.6×10^{-4}	542	220.7	−3.83	−9.83	6.00	693
4	120	7.9×10^{-4}	3.5×10^{-4}	4.4×10^{-4}	540	221.5	−3.68	−9.67	5.99	550
5	37	5.6×10^{-4}	4.1×10^{-4}	1.5×10^{-4}	538	222.3	−3.74	−9.67	5.93	954
6	3.6	1.2×10^{-2}	1.2×10^{-2}	4.1×10^{-4}	554	215.9	−1.76	−7.05	5.28	195
7	≤0.001[e]	1.5×10^{-1}	1.5×10^{-1}	– [e]	565	211.2	−0.15	−5.01	4.86	−425
8	≤0.001[e]	1.2×10^{-1}	1.2×10^{-1}	– [e]	564	212.1	−1.81	−6.95	5.14	−318

[a] All the experimental values were obtained in acetonitrile at 318 K. In the evaluation of K_{eq}, the molar absorption coefficient at λ_{max} was assumed to be 3.5×10^4 M^{-1} cm^{-1} for all merocyanines.
[b] Orbital energies of HOMOs and LUMOs. These were calculated for the optimized structures of the CTT forms by MM2/ZINDO. For **5**, the indoline-centred LUMO + 1 was used instead of the pyridine-centred LUMO.
[c] Energy gap between HOMO and LUMO.
[d] Heat of formation of the transition state of the reaction CCC→**I**. Calculated by MOPAC/PM3.
[e] Less than the detection limit in spectrophotometric analysis.

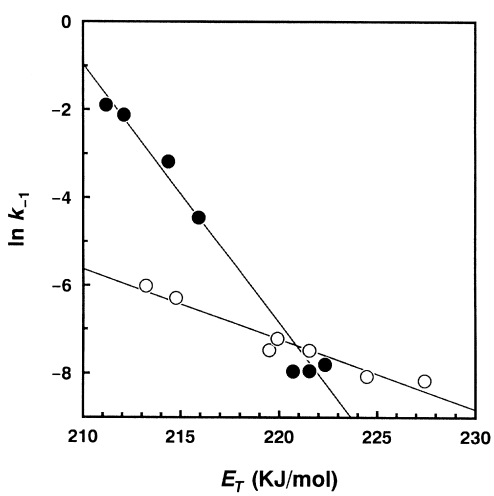

Fig. 3. Plots of ln k_{-1} vs. E_T: ●, for spiropyrans **1**–**8** in acetonitrile; ○, for spiropyran **2** in various organic solvents.

ined the solvent effect for the cationic spiropyran **2**, and obtained a less E_T-dependent correlation: $\alpha = -0.16$ (kJ/mol)$^{-1}$ and $\beta = 28$ (Fig. 3). Song et al. [7] reported $\alpha = -0.178$ (kJ/mol)$^{-1}$ and $\beta = 32.6$ for 1′-hexadecyl-3′,3′-trimethyl-6-nitrospiro[2*H*-1-benzopyran-2,2′-indoline] as the solvent effect at 304 K. Keum et al. [8] reported a slope α in the range -0.055–-0.092 (kJ/mol)$^{-1}$ for analogous spiropyrans with $R_1 \equiv$ Br, I and OCH_3 as the solvent effect. Our observed dependence of ln k_{-1} on the ionic moiety has a slope α 3–10 times larger than those due to the solvent effect. Ionic moieties have little influence on the solute–solvent interaction, but affect the intrinsic nature of spiropyrans.

According to MM2/ZINDO calculations, the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) of the CTT isomer are separately located on phenolate O and indoline 1′N-2′C respectively,

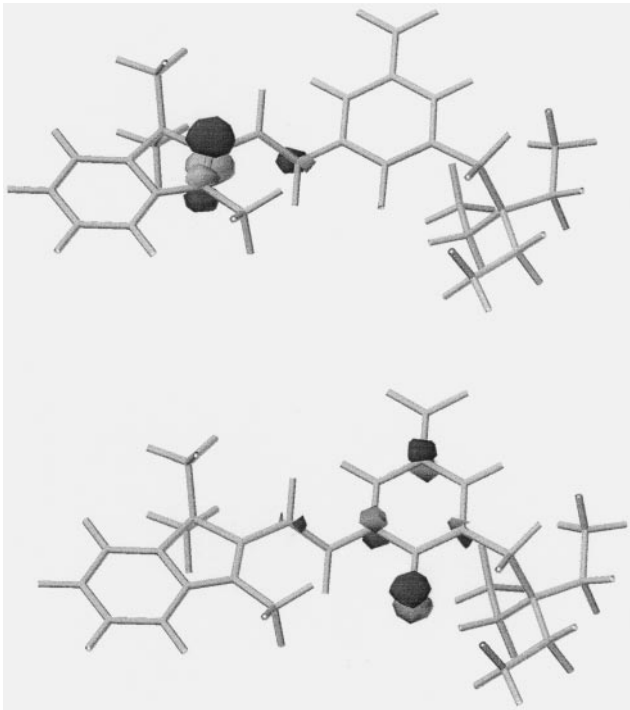

Fig. 4. Surfaces of HOMO (bottom) and LUMO (top) of cationic merocyanine **2** calculated by MM2/ZINDO.

as shown in Fig. 4. E_T and $\ln k_{-1}$ correlate with the energy gap ΔE between the two MOs, suggesting that electronic interaction between phenolate (donor) and indoline (acceptor) should be substantial. When ionic substituents are covalently bound to spiropyran, the electronic structure of the open merocyanine form will be perturbed; the HOMO and LUMO energies are influenced by the ionic charge in the vicinity.

The ionic substituents also modify the Coulombic interaction between phenolate and indoline in the ionic recombination process. We calculated the heat of formation H of the transition state of ring closure (CCC→**I**) using MOPAC with PM3 parameters, and found a strong dependence of H on the ionic substituent. The rate constants correlate well with H as shown in Fig. 5. The ionic charge markedly influences the calculated H values of our merocyanines. H is changed by more than 500 kJ mol^{-1} on changing the charge on R_1 from 0 to +1 or from 0 to −1. The steric influence is smaller than the charge effect. The H values (kJ mol^{-1}) of cationic spiropyrans with $R_1 \equiv CH_2N(C_nH_{2n+1})_3$ are 777 ($n=1$), 747 ($n=2$) and 693 ($n=3$) respectively. For neutral $R_1 \equiv CH_2C(CH_3)_3$, H is calculated to be 108 kJ mol^{-1} resembling H of other merocyanines with neutral R_1. The dependence of H on the charge may be exaggerated since solvation was not considered in our calculation. However, the present results are indicative of a Coulombic interaction on formation of the transition state controlled by the neighbouring ionic moieties.

For reaction between two ions in solution, the rate constant can be expressed by the following simplified equation

$$\ln k = \ln k_0 - (Z_A Z_B e^2 / D_s d_{AB} k_B T) \quad (4)$$

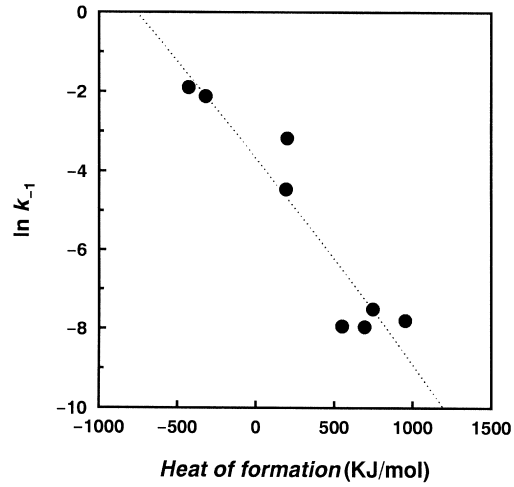

Fig. 5. Plots of $\ln k_{-1}$ vs. heat of formation (H) of the transition state in CCC→**I** reactions for spiropyrans **1**–**8**.

where $Z_{A(B)}$ is the charge of the ion A (B), e is the elementary electric charge, D_s is the static dielectric constant, d_{AB} is the distance between the charges in the activated complex and k_B is Boltzmann's constant. The second term was derived from the Coulombic work to bring ions into the reaction distance. For the reaction of $Z_A Z_B = 1$ in acetonitrile ($D_s = 37.5$) at 318 K, the term was calculated to be $14.0/d_{AB}$, where d_{AB} is in angstroms. According to our calculation, d_{AB} between the charged atom of R_1 and indoline N^+ should be 5–8 Å at the transition state. We estimate that $\ln k_{-1}$ will decrease 2.8 units (when $d_{AB}=5$) ~ 1.8 units (when $d_{AB}=8$), by increasing $Z_A Z_B$ by one unit. The observed dependence of $\ln k_{-1}$ on $Z_A Z_B$ is not far from this estimation.

On the other hand, the forward reaction rate does not show any noticeable dependence on the ionic charge, E_T or H, but remains almost constant at $\ln k_1 \sim -8$ for all of the spiropyrans. It has been pointed out that k_1 is also solvent independent [7]. The forward C–O bond rupture does not produce zwitterionic **II** directly, but first quinoid **II′** through the electrocyclic mechanism. The charge and solvent have no influence on this process.

4. Conclusions

By incorporating an ionic moiety in the molecular skeleton of spiropyran, the suppression or acceleration of the reverse ring closure becomes possible. The ionic moiety has both an electronic (modification of HOMO–LUMO energies) and Coulombic (charge interaction between phenolate and indoline) influence on the merocyanine form **II**. Although it is not possible to elucidate which factor governs the reaction, since both modify the rate in the same direction in the present experiment, the coulombic work in the simple ionic recombination scheme apparently accounts for the effect of the ionic moiety.

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