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Synthesis, structure and photochromic properties of 4-acyl pyrazolone derivants

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Abstract

A new organic photochromic compound containing pyrazolone-ring photochromic functional unit: 1-phenyl-3-methyl-4-benzoylpyrazole-5-one methyl thioesmicarbazone (1A), and some analogous non-photochromic derivatives were synthesized and characterized by elemental analyses, MS, IR spectra, ^{1}H NMR spectra. The photochromic properties and photochemical kinetics of 1A have been studied by UV reflectance spectra under irradiation of 365 nm light. The crystal structure analyses of photocolored product (1A) show the photochromism is due to the photoisomerization from enol form to keto form. According to the results of structure analyses, an intermolecular proton transfer mechanism of the photochemical process is proposed.

Keywords: Synthesis; Pyrazolone; Photochromism; Intermolecular proton transfer mechanism

1. Introduction

Photochromism of solid materials has attracted much attention because of the scientific interest and potential commercial applications such as high-density optical storage media, photo switching devices, eye-protecting glasses, etc. [1–6]. Developing new photochromic system and determining the mechanistic aspects of the photochemical process are the power to speed the progress of this field, though numerous photochromic compounds on spiropyrans, spiroxazine, diarylethenes, fulgides and Schiff bases [7,8] have been extensively studied.

Schiff bases from salicylaldehyde and its derivatives are well-known examples, which show either photo- or thermochromism in the solid state. All of the compounds are photochromism in solutions, but one can not measure them in room temperature by the steady equipment [9]. X-ray structural data [10,11] have shown that in the thermochromic crystals the molecules are essentially planar and the packing consists of stacks of parallel molecules with an interplanar distance close to 3.3 Å. In the photochromic crystals, the molecules are not planar since the aniline ring is twisted about the exocyclic N–C bond by 55° and the packing is consequently more ‘open’. The difference in chromobehavior is attributed to differences in the molecular geometry.

Cohen and Schmidt undertook a more systematic study of crystalline anils of salicylaldehydes and proposed an intramolecular H-transfer mechanism as shown in Scheme 1 [10].

Two photochromic compounds on thiosecarbazone have been synthesized [12]. An intermolecular H-transfer mechanism was proposed on the basis of their crystal structure. A series of derivants containing pyrazolone-ring have been synthesized in our laboratory [13] in order to interpret further the phenomena of photochromism and confirm further the mechanism proposed above. In this paper, five hydrazone compounds containing pyrazolone-ring were synthesized and characterized (shown in Scheme 2). It was found that 1A exhibited reversible color changes induced by 365 nm ultraviolet irradiation or temperature, respectively. So it is interesting as potential candidates for practical applications. However, the crystal structure of 1A* showed that the photocoloration of 1A involved in an intermolecular proton transfer from *O*-hydroxyl group to the nitrogen atom of adjoining molecular pyrazolone-ring as shown in Scheme 3.

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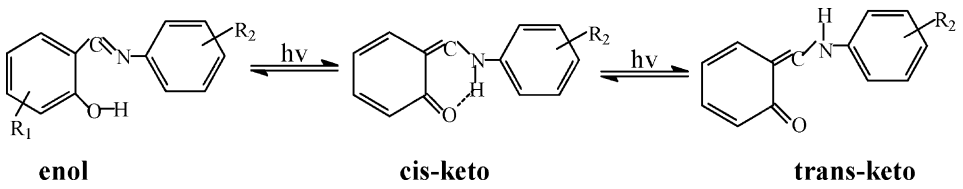
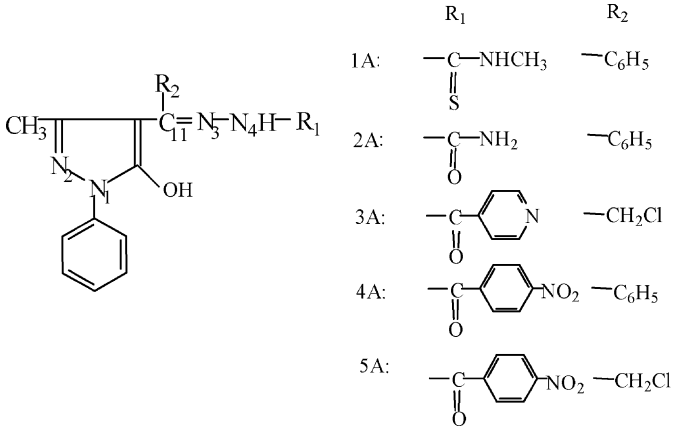
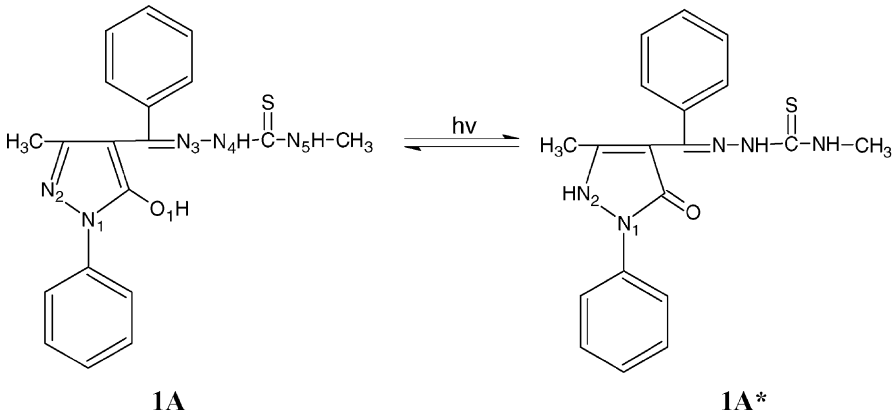


Scheme 1.



Scheme 2.



Scheme 3.

2. Experimental details

2.1. Physical measurements

The elemental analyses were performed on a PE-2400C model elemental analyzer. IR spectra were recorded as KBr disc on Bio-RAD FTS-40 spectrometer. ^{1}H NMR spectra were determined on an AC-80 spectrometer with $CDCl_3$ and DNSO-d_6 as mixing solvent. Mass spectra were obtained on HP-5988 GC/MS. UV reflectance spectra of 1A were recorded on an UV-3010 spectrophotometer. All melting points were measured with a TECH XT-5 melting point apparatus and were uncorrected.

2.2. Materials

Semicarbazide was synthesized by refluxing the mixture of urea (15 g), hydrazine (20 ml) and water (30 ml) for 8 h at 95–105 °C. Then water was removed by evaporation, and the remains were cooled. The white product was precipitated, the precipitates were suction filtered, washed three times with ether, and dried in vacuum. Elemental analyses for CH_5N_3O requires 16.00% C, 6.71% H, 55.97% N; found: 16.39% C, 6.60% H, 56.06% N.

Isonicotinoylhydrazine was synthesized by refluxing the mixture of isonicotinic acid (10 g), ethanol (40 ml), benzene (40 ml) and sulfuric acid (98%, 6 ml) for 5 h at refluxing

temperature. Then benzene and ethanol were removed by evaporation, and the remains were poured into 200 ml of cooling water. After being neutralized to pH 7.0 by using $NaHCO_3$, the solution was extracted three times by ether (30–60 ml). The yellow liquid was mixed with ethanol (60 ml) and hydrazine (20 ml). The mixture was heated to reflux for 4 h, then cooled. The cream product was precipitated, the precipitate was filtered, washed with water, and dried in vacuum. Elemental analyses for $C_6H_7N_3O$ requires 52.55% C, 5.14% H, 30.64% N; found: 52.71% C, 5.00% H, 31.00% N.

1-Phenyl-3-methyl-4-chloroacetyl-pyrazolone-5 was synthesized by the literature [14]. $C_{12}H_{11}O_2N_2Cl{\cdot}H_2O$ requires 53.64% C, 4.88% H, 10.43% N; found: 54.13% C, 4.84% H, 10.14% N.

1-Phenyl-3-methyl-4-benzoyl-5-one-pyrazole methyl thiosemicarbazone (1A) was prepared by mixing 1-phenyl-3-methyl-4-benzoyl-pyrazolone-5 (5 mmol) and N(4)-methyl-3-thiosemicarbazide (5 mmol) in 50 ml of MeOH solution containing five to six drops of glacial acetic acid at 70 °C for ca. 3 h by stirring. The mixture was cooled in the dark at room temperature. The colorless crystal suitable for X-ray analysis was obtained by allowing MeOH to evaporate for a few days in the dark. The other new compounds were prepared with the same method as 1A.

Table 1
Summary of crystal data, experimental details and refinement parameters for 1A*

Empirical formula	$C_{19}H_{19}N_5OS$
Formula weight	365.45
Temperature	295(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	C_c
Unit cell dimensions	$a = 7.8420(10)$ Å, $\alpha = 90°$ $b = 20.768(4)$ Å, $\beta = 106.810(1)°$ $c = 11.805(2)$ Å, $\gamma = 90°$
Volume (Z)	1840.4(5) Å^3, 4
Density (calculated)	1.319 g/cm^3
Absorption coefficient	0.194 mm^{-1}
$F(0\,0\,0)$	768
Crystal size	0.48 mm × 0.48 mm × 0.16 mm
θ range for data collection	1.96–28.50°
Limiting indices	$-10 \le h = 10$, $-26 \le k \le 27$, $-15 \le l \le 5$
Reflections collected	5088
Independent reflections	4452 ($R_{int} = 0.0111$)
Absorption correction	Empirical
Maximum and minimum transmission	0.9793 and 0.9038
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	4452/2/238
Goodness-of-fit on F^2	0.997
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0371$, w$R_2 = 0.0896$
R indices (all data)	$R_1 = 0.0470$, w$R_2 = 0.0938$
Absolute structure parameter	0.07(7)
Extinction coefficient	0.0072(7)
Largest difference peak and hole	0.169 and 0.160 e. Å^{-3}

Table 2
Non-hydrogen atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å^2 $\times 10^3$) of 1A*

Atoms	x	y	z	U(eq)
S	5158(1)	5526(1)	7051(1)	62(1)
O	41(2)	5332(1)	4519(1)	43(1)
N(1)	258(2)	5243(1)	2513(1)	35(1)
N(2)	88(2)	4750(1)	1777(1)	38(1)
N(3)	2279(2)	4043(1)	5630(1)	37(1)
N(4)	3140(2)	4625(1)	5773(1)	40(1)
N(5)	4458(2)	4352(1)	7688(1)	50(1)
C(1)	2432(3)	5799(1)	930(2)	42(1)
C(2)	3447(3)	6344(1)	513(2)	52(1)
C(3)	3279(4)	6879(1)	1204(2)	61(1)
C(4)	2089(4)	6883(1)	2317(2)	67(1)
C(5)	1051(3)	6351(1)	2748(2)	52(1)
C(6)	1257(2)	5803(1)	2052(2)	34(1)
C(7)	160(2)	5005(1)	3654(1)	34(1)
C(8)	690(2)	4352(1)	3594(2)	33(1)
C(9)	505(2)	4225(1)	2410(2)	35(1)
C(10)	931(3)	3637(1)	1812(2)	49(1)
C(11)	1188(2)	3908(1)	4600(1)	33(1)
C(12)	366(3)	3259(1)	4503(2)	37(1)
C(13)	1172(3)	2766(1)	5277(2)	52(1)
C(14)	350(4)	2173(1)	5230(2)	67(1)
C(15)	1264(4)	2058(1)	4407(2)	61(1)
C(16)	2068(3)	2534(1)	3629(2)	54(1)
C(17)	1263(3)	3132(1)	3682(2)	45(1)
C(18)	4220(2)	4793(1)	6863(2)	40(1)
C(19)	5604(4)	4430(2)	8882(2)	76(1)

U(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Dioxane was purified as standard method. N(4)-methyl-3-thiosemicarbazine was purchased from Aldrich chemicals, USA. The other materials were AR grade, obtained from commercial sources and used without further purification.

2.3. Determination of the crystal structure of 1A*

Data were collected at 294 K by a Siemens P4 diffractermeter with Mo Kα radiation ($\lambda = 0.71073$ Å). The structure was solved by direct methods. All calculations and drawing were performed by the Siemins SHELXTL crystallographic software package of molecular structure. The intensities were measured by ω-scans. Cell constants and orientation matrix for data collection were obtained by least-squares refinement of the diffraction data from reflections in the range 1.96–28.50°. Non-H atoms were located in successive ΔF maps and refined anisotropically by full-matrix least-squares on F^2. Hydrogen atoms were found from the difference Fourier map and refined isotropically. The crystal data and structure refinement details are given in Table 1. Non-hydrogen atomic coordinates and thermal parameters are listed in Table 2. The bond lengths and bond angles are listed in Table 3. The molecular structure and the packing arrangement in the unit cell are shown in Figs. 1 and 2, respectively.

Table 3
The data of bond lengths (Å) and bond angles (°) of 1A*

S–C(18)	1.679(2)	N(2)–N(1)–C(7)	108.14(14)
O–C(7)	1.253(2)	N(2)–N(1)–C(6)	120.78(13)
N(1)–N(2)	1.374(2)	C(7)–N(1)–C(6)	127.94(15)
N(1)–C(7)	1.383(2)	C(9)–N(2)–N(1)	109.75(13)
N(1)–C(6)	1.420(2)	C(11)–N(3)–N(4)	118.24(15)
N(2)–C(9)	1.328(2)	C(18)–N(4)–N(3)	119.37(15)
N(3)–C(11)	1.298(2)	C(18)–N(5)–C(19)	124.20(19)
N(3)–N(4)	1.370(2)	C(6)–C(1)–C(2)	119.39(19)
N(4)–C(18)	1.366(2)	C(3)–C(2)–C(1)	120.4(2)
N(5)–C(18)	1.309(3)	C(2)–C(3)–C(4)	119.9(2)
N(5)–C(19)	1.445(3)	C(3)–C(4)–C(5)	120.8(2)
C(1)–C(6)	1.378(3)	C(4)–C(5)–C(6)	119.0(2)
C(1)–C(2)	1.390(3)	C(1)–C(6)–C(5)	120.44(17)
C(2)–C(3)	1.363(3)	C(1)–C(6)–N(1)	120.29(16)
C(3)–C(4)	1.374(4)	C(5)–C(6)–N(1)	119.26(16)
C(4)–C(5)	1.380(3)	O–C(7)–N(1)	123.23(16)
C(5)–C(6)	1.385(3)	O–C(7)–C(8)	130.41(15)
C(7)–C(8)	1.427(2)	N(1)–C(7)–C(8)	106.35(14)
C(8)–C(9)	1.388(2)	C(9)–C(8)–C(7)	106.44(15)
C(8)–C(11)	1.464(2)	C(9)–C(8)–C(11)	128.56(16)
C(9)–C(10)	1.496(3)	C(7)–C(8)–C(11)	124.84(15)
C(11)–C(12)	1.484(3)	N(2)–C(9)–C(8)	109.21(15)
C(12)–C(17)	1.387(3)	N(2)–C(9)–C(10)	119.60(15)
C(12)–C(13)	1.397(3)	C(8)–C(9)–C(10)	131.11(17)
C(13)–C(14)	1.384(3)	C(15)–C(16)–C(17)	120.0(2)
C(14)–C(15)	1.375(4)	C(16)–C(17)–C(12)	121.1(2)
C(15)–C(16)	1.373(4)	N(5)–C(18)–N(4)	116.01(17)
C(16)–C(17)	1.387(3)	N(5)–C(18)–S	124.97(15)
N(3)–C(11)–C(8)	124.92(17)	N(4)–C(18)–S	119.02(15)
N(3)–C(11)–C(12)	114.99(15)	N(5)–C(19)–H(19C)	109.5
C(8)–C(11)–C(12)	120.04(15)	C(15)–C(14)–C(13)	120.4(2)
C(17)–C(12)–C(13)	117.96(18)	C(16)–C(15)–C(14)	119.9(2)
C(17)–C(12)–C(11)	121.23(17)	C(14)–C(13)–C(12)	120.6(2)
C(13)–C(12)–C(11)	120.74(17)		

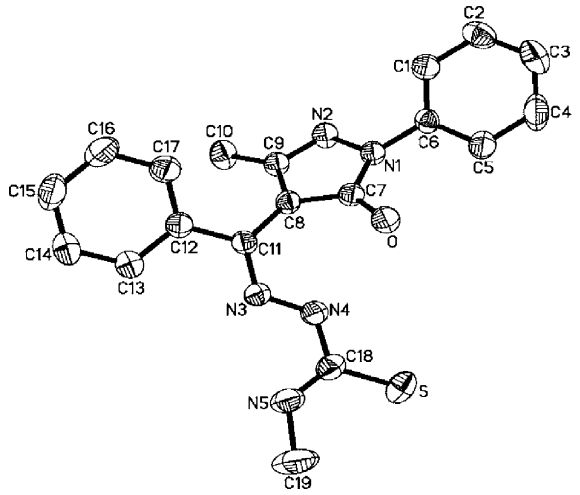


Fig. 1. The crystal structure of 1A*.

3. Results and discussion

3.1. Characterization for new compounds

(1A) Yield: 75.0%; mp: 195–196 °C. Elemental analyses: formula $C_{19}H_{19}N_5OS$ requires 62.45% C, 5.24% H, 19.16% N; found: 62.36% C, 5.21% H, 18.94% N. The spectroscopic data are as follows. IR (cm^{-1}): 3371 (m, υ_{NH}), 2597 (br, $\upsilon_{O–H}$), 1610 (s, $\upsilon_{C11=N3}$), 1546, 1513, 1406 (s,$\upsilon_{pyrazolone\text{-}ring}$), 835 (m, $\upsilon_{C=S}$). ^1H NMR ($CHCl_3$, δ): 1.56 (s, 3H, CH_3), 1.65 (s, 1H, N5–H), 3.15 (s, 3H, N4–CH_3), 6.32 (w, 1H, N4–H), 7.22–7.78 (m, 10H, $2C_6H_5$), 8.25 (w, 0.7H, N2–H), 11.31 (w, 0.3H, OH). MS: $M^+ = 366.0$ (formula weight: 365.45).

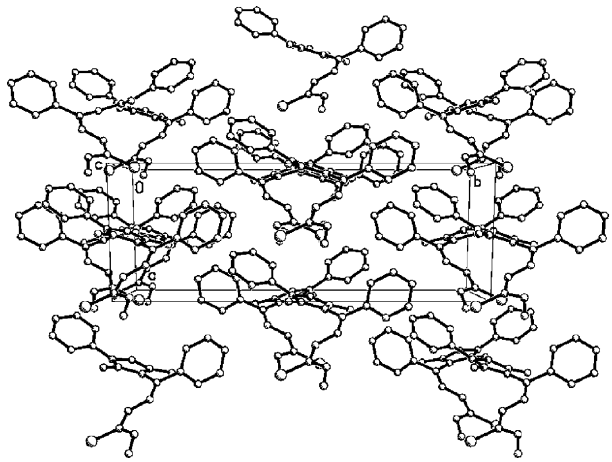


Fig. 2. Packing in unit cell of 1A*.

(2A) Yield: 87.0%; mp: 186–188 °C. Elemental analysis: formula $C_{18}H_{17}N_5O_2$ requires 64.47% C, 5.11% H, 20.88% N; found: 64.48% C, 5.13% H, 20.60% N. The spectroscopic data are as follows. IR (cm^{-1}): 3363 (m, υ_{NH_2}), 3143 (m, $\upsilon_{O–H}$), 1718 (s, $\upsilon_{C=O\,hydrazide}$), 1626 (s, $\upsilon_{C=N}$), 1592, 1548, 1499 (s, $\upsilon_{pyrazolone\text{-}ring}$). ^1H NMR ($CHCl_3$, δ): 1.54 (s, 3H, CH_3), 5.09 (w, 1H, N4–H), 6.92 (s, 1H, N2–H), 7.25–7.64 (m, 10H, $2C_6H_5$), 7.93 (s, 2H, NH_2). MS: $M^+ = 336$ (formula weight: 335.37).

(3A) Yield: 90%. Elemental analysis: formula $C_{18}H_{16}N_5O_2Cl$ requires 58.46% C, 4.36% H, 18.94% N; found: 57.95% C, 4.25% H, 18.98% N. The spectroscopic data are as follows. IR (cm^{-1}): 3442 (m, υ_{NH}), 2602 (br, $\upsilon_{O–H}$), 1619 ($\upsilon_{C=O\,hydrazide}$), 1592 (s, $\upsilon_{C=N}$), 1525, 1495(s, $\upsilon_{pyrazolone\text{-}ring}$). ^1H NMR (DMSO-d_6, δ): 2.45 (s, 3H, CH_3), 4.81 (s, 2H, CH_2Cl), 7.14–7.51 (m, 5H, C_6H_5), 7.9–8.0 (m, 4H, C_6H_4), 8.80 (s, 1H, N4–H), 8.90 (m, 1H, N2–H). MS: $M^+ = 370.0$ (formula weight: 369.81).

(4A) Yield: 92%. Elemental analysis: formula $C_{24}H_{19}N_5O_4$ requires 65.30% C, 4.34% H, 15.86% N; found: 65.07% C, 4.07% H, 15.91% N. The spectroscopic data are as follows. IR (cm^{-1}): 3180 (m, υ_{NH}), 3002 (m, υ_{OH}), 1658 (s, $\upsilon_{C=O\,hydrazine}$), 1599 (s, $\upsilon_{C=N}$), 1522, 1480 (s, $\upsilon_{pyrazolone\text{-}ring}$). ^1H NMR ($CDCl_3$ + DMSO-d_6, δ): 1.56 (s, 3H, CH_3), 1.77 (s, 1H, N4–H), 7.25–7.63 (m, 10H, $2C_6H_5$), 7.93–8.25 (m, 4H, $C_6H_4NO_2$), 8.43 (s, 1H, N2–H). MS: $M^+ = 442$ (formula weight: 441.45).

(5A) Yield: 85%. Elemental analysis for $C_{19}H_{16}N_5O_4Cl$; found: 55.20% C, 3.79% H, 16.81% N; Cala. 55.15% C, 3.90% H, 16.92% N. The spectra data are as follows.

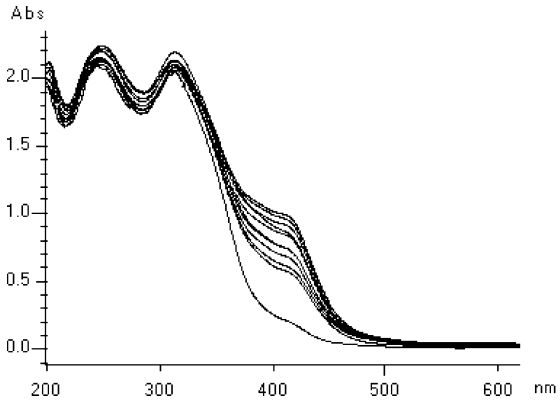


Fig. 3. UV reflectance spectra of photochemical coloration of 1A with 365 nm light at 298 K.

IR (cm^{-1}): 3110 (m, υ_{NH}), 2500 (br, $\upsilon_{O–H}$), 1634 (s, $\upsilon_{C=O\,hydrazine}$), 1600 (s, $\upsilon_{C=N}$), 1565, 535, 1490 (s, $\upsilon_{pyrazolone\text{-}ring}$), 1406 (s, υ_{NO_2}). ^{1}H NMR ($CDCl_3$ + DMSO-d_6, δ): 2.50 (s, 3H, CH_3), 2.68 (m, 1H, N4–H), 4.55 (s, 2H, CH_2Cl), 7.15–7.46 (m,5 H, C_6H_5), 7.62–8.28 (m, 4H, C_5H_4N), 8.40 (w, 0.8H, N2–H), 11.28 (w, 0.2H, OH). MS: $M^+ = 414$ (formula weight: 413.82).

3.2. Photochromism and thermochromism

The colorless of 1A (enol form) changes to yellow under irradiation of 365 nm ultraviolet light. The color turns back to the original colorless when it is placed in the dark. Thus, 1A has photochromic properties in the solid state. The powder-UV reflectance spectra at 298 K with 365 nm UV light irradiated at regular time intervals are shown in Fig. 3. A new absorption band appears around 415 nm.

On the other hand, the yellow photocolored product at room temperature changes to the red color at 100 °C, and returns to the starting colorless after being placed in the refrigerator. Its thermochromism behavior is confirmed by Fig. 4. A new absorption band appeared between 400 and 600 nm.

So the compound 1A exhibits photochromic and thermochromism only in the solid state. However, it does not exhibit these properties in the solution.

3.3. Kinetics of the photochromic reaction

The first-order rate constant of 1A was determined essentially as described in the literature [15]. The experimental data were treated with the standard integrated expression:

$$\ln\frac{A_\infty - A_0}{A_\infty - A_t} = kt \qquad (1)$$

where A_0, A_∞, A_t are the observed absorption data corresponding to 415 nm wavelength at the beginning, at time t, and at the end of the reaction, respectively.

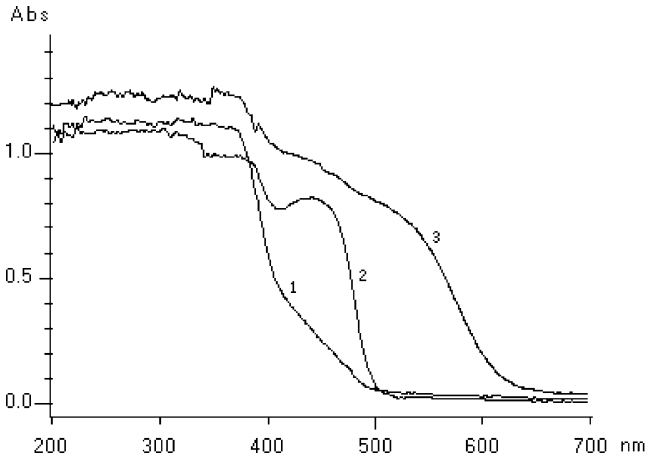


Fig. 4. Thermobleaching spectra of 1A: (1) before heated; (2) after irradiated with 365 nm ring; (3) after heated.

The kinetic curve of 1A is plotted according to the Eq. (1) and shown in Fig. 5. The wavelength at which the maximum change in absorption data occurred was used for the kinetic measurements. A good linear fit was found for 1A in the kinetic plot from which the first-order or pseudo-first-order rate constant was obtained as $k = 3.91 \times 10^{-4}\,s^{-1}$, the result shows that the photocoloration of 1A is slower than the analogous compound reported previously [12].

3.4. Mechanism of photochromism

The IR spectra analysis in the previous section show that the colorless compound (1A) is the enol form before the photocoloration takes place. But the reliable structure data of 1A are not obtained because the crystal of 1A is easily turned into 1A* during measurement. Therefore, the crystal structure data of photocolored product are only reported in this paper. The C7–O band distance is 1.253(2) Å, which is indicative of significant double character. So the yellow

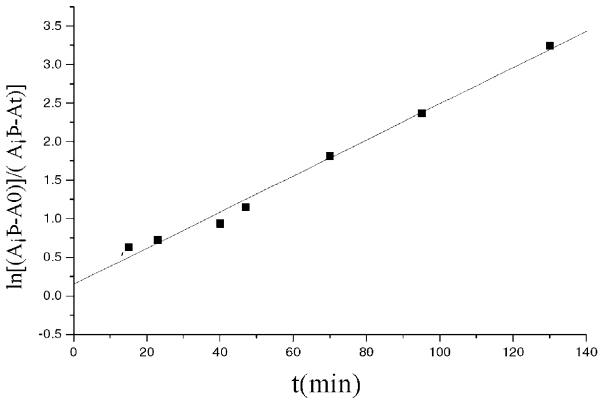


Fig. 5. First-order kinetic plot of photoisomerization (enol → keto) reaction of 1A induced by 365 nm light.

compound is the keto form. The structure of 1A* shows that an intermolecular hydrogen bond [N(2)–H · · · O] (2.7010 Å) exists between neighboring molecules. It suggests that the change of color is due to the photoisomerization from the enol form to the keto form. It is different from the photochromic processes of Schiff base compounds proposed by Cohen et al. The photocoloration processes of Schiff bases such as salicylaldehyde and its derivatives was suggested as the mechanism of intramolecular proton transfer from the hydroxylic group to the imine (–CH=N–) nitrogen atom (shown in Scheme 3). However, it is almost impossible for 1A to transfer hydroxyl proton (O–H) to N_2 atom of pyrazolone-ring in molecule itself because the proton must surmount not only the bonding effort with N_2 but also potential barrier, especially when it pass through pyrazolone-ring with great electron cloud density. According to the results of structure analyses, the photochromic process of 1A is considered to be similar to that of analogous compounds [12] with the same environment reported previously. So, the intermolecular proton transfer mechanism of the photochromism postulated in [12] is further supported by parallel studies on PMBP methyl-thiosemicarbazone carried out in this paper.

Although 1A belongs to Schiff base compound, its photoisomerization does not involve the imine (–C11–N3–) of Schiff base. Therefore, this kind of compound does not belong to Schiff base photochromism compound proposed by Cohen et al.

It has been reported HPMBP has two tautomer, but it has no photochromism. However, the thiosemicarbazone group are incorporated, PMBP-thiosemicarbazones have photochromic properties. It suggests that the incorporation of the thiosemicarbazone group improve greatly the systematical conjugacy, which could be advantageous to transform the photon energy into the hydrogen bond. It can be expected that photochromism is observed when N(4) substituted thiosecarbazone group is incorporated. However, as R_1 is replaced by semicarbazide or benzoyl hydrazine, the compounds formed have no photochromism. There are due to the systematical conjugacy is reduced. In order to synthesis further new photochromic compounds, so we should make effort to add some thiosemicarbazone derivatives to in the side chain or R_2 is replaced by the group with larger conjugacy as possibly in the future.

4. Conclusion

A novel photochromic compound containing pyrazolone-ring has been prepared. Structure analysis suggests the photochromic properties are the result of isomerization from enol form to keto form and the photochromic mechanism is suggested intermolecular proton transfer by the way of intermolecular hydrogen bond.

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