

Photoreaction in two-component molecular crystals of α -hydroxycarboxylic acids and aza aromatic compounds

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Abstract

A series of two-component molecular crystals of α -hydroxycarboxylic acids such as benzoic acid (**1**) and *S*-(+)-mandelic acid (**2**) with aza aromatic compounds such as acridine (**3**), phenanthridine (**4**) and phenazine (**5**) were prepared by crystallization from the solutions of both components. Irradiation of the crystals caused photodecarboxylation and dehydrogenation to give benzophenone (**6**) or benzaldehyde (**7**) as a main product. ©1999 Elsevier Science S.A. All rights reserved.

Keywords: Solid-state photoreaction; Photodecarboxylative dehydrogenation; Two-component molecular crystals; α -Hydroxycarboxylic acids; Aza aromatic compounds

1. Introduction

Solid-state organic photoreactions have attracted much attention, since there is a growing number of reaction in the crystals [1–4]. We have developed various solid-state bimolecular photoreactions in two-component molecular crystals self-assembled from two different compounds [5–14]. An important advantage for utilizing such two-component crystals as reaction media is that reactivity can be induced via photoinduced electron transfer by combining an electron donor and acceptor species. Further, most of solid-state photoreactions give higher selectivity due to the restricted freedom of molecules in the crystal lattice. For instance, although selectivity of photodecarboxylation in solution has been reported to be low [15–20], we have achieved highly selective photodecarboxylation by utilizing various two-component crystals of aralkyl carboxylic acids as electron donors and aza aromatic compounds as electron acceptors [8–13]. Herein, photodecarboxylative dehydrogenation in two-component crystals of α -hydroxycarboxylic acids with aza aromatic compounds was examined.

2. Experimental details

2.1. General procedures

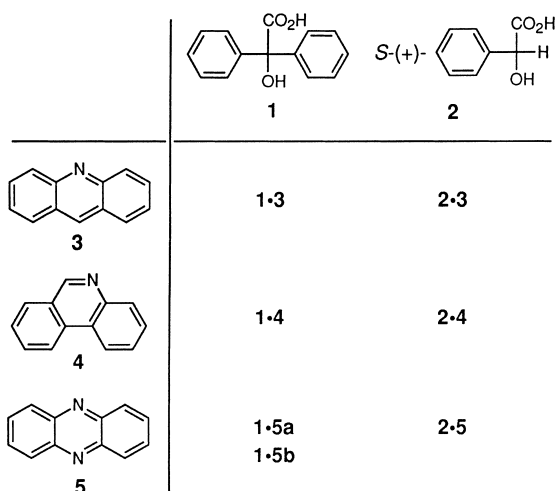
¹H-NMR spectra were measured on a 60-MHz JEOL spectrometer with tetramethylsilane as an internal standard. IR spectra were recorded on a JASCO FT/IR-8300 spectrophotometer. Differential scanning calorimetry (DSC) was done on a Rigaku Thermoflex TAS-2000 DSC8230D and melting points were not corrected. Elemental analysis was carried out with a Yanaco CHN Corder MT-5. HPLC with a photodiode-array detector were used for determining the products on a Waters HPLC system. All the reagents were commercially available.

2.2. Preparation of two-component molecular crystals

The two-component molecular crystals investigated here incorporate benzoic acid (**1**) and *S*-(+)-mandelic acid (**2**) as α -hydroxycarboxylic acids and acridine (**3**), phenanthridine (**4**) and phenazine (**5**) as electron acceptors (Scheme 1). Seven two-component molecular crystals were prepared by crystallization from the solutions of both components. The crystals were characterized by DSC, IR, and elemental analyses.

1·3: yellow prisms; mp 135°C (from 1 : 1 of acetone and AcOEt); IR (KBr) 3384, 3058, 2402, 1964, 1645 cm⁻¹.

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Scheme 1. Two-component molecular crystals.

Analytically calculated for $C_{27}H_{21}NO_3$ (1 : 1): C, 79.59; H, 5.19; N, 3.44. Found: C, 80.04; H, 5.39; N, 3.45.

1-4: colorless prisms; mp 137°C (from 2 : 1 : 1 of acetone, MeCN and benzene); IR (KBr) 3435, 3066, 2360, 1713 cm^{-1} . Analytically calculated for $C_{27}H_{21}NO_3$ (1 : 1): C, 79.59; H, 5.19; N, 3.44. Found: C, 79.81; H, 5.30; N, 3.38.

1-5a: yellow prisms; mp 125°C (from 1 : 1 of acetone and benzene); IR (KBr) 3600–2000, 1705 cm^{-1} . Analytically calculated for $C_{40}H_{24}N_2O_6$ (2 : 1): C, 75.45; H, 5.08; N, 4.40. Found: C, 75.89; H, 5.23; N, 4.86.

1-5b: yellow prisms; mp 90°C (from 1 : 1 of acetone and benzene); IR (KBr) 3600–2700, 1732 cm^{-1} . Analytically calculated for $C_{26}H_{22}N_2O_4 \cdot \{1 : 1 : 1(\text{H}_2\text{O})\}$: C, 73.23; H, 5.20; N, 6.57. Found: C, 73.37; H, 5.27; N, 6.56.

2-3: yellow prisms; mp 107°C (from 1 : 1 of acetone and AcOEt); IR (KBr) 3367, 3056, 1958, 1661 cm^{-1} . Analytically calculated for $C_{21}H_{17}NO_3$ (1 : 1): C, 76.12; H, 5.17; N, 4.23. Found: C, 76.31; H, 5.10; N, 4.15.

2-4: colorless prisms; mp 115°C (from 1 : 1 : 1 of acetone, AcOEt and MeCN); IR (KBr) 3417, 2438, 1956, 1709 cm^{-1} . Analytically calculated for $C_{21}H_{17}NO_3$ (1 : 1): C, 76.12; H, 5.12; N, 4.23. Found: C, 76.40; H, 5.36; N, 4.29.

2-5: yellow prisms; mp 148°C (from 1 : 1 of acetone and benzene); IR (KBr) 3336, 2908, 2560, 1738 cm^{-1} . Analytically calculated for $C_{28}H_{24}N_2O_6$ (2 : 1): C, 69.40; H, 5.00; N, 5.78. Found: C, 69.40; H, 5.16; N, 5.78.

2.3. Preparative photoreaction in solution

A solution (100 ml) of 0.05 M α -hydroxycarboxylic acid of **1** or **2** and 0.05 M aza aromatic compound of **3**, **4** or **5** in acetonitrile was internally irradiated with a 100-W high-pressure mercury lamp under argon bubbling for 2 h at room temperature. After the reaction, the precipitation of biacridane (**8**), biphenanthridane (**9**) or charge transfer (CT) complex **10** of phenazine **5** and dihydrophenazine (**11**)

was removed by filtration. The solution was submitted to silica–gel column chromatography to give benzophenone (**6**) or benzaldehyde (**7**). The melting points, IR spectra, and NMR patterns of **6–9** were consistent with those of the authentic samples [12]. The results are shown in Scheme 2 and Table 2.

10: dark blue prisms; decolorization at 180°C , sublimation at 219°C ; IR (KBr) 3259 cm^{-1} . Analytically calculated for $C_{24}H_{18}N_4$: C, 79.53; H, 5.01; N, 15.46. Found: C, 79.74; H, 5.08; N, 15.43. The NMR spectrum could not be measured due to the oxidation of **11** to **5** in the solution.

2.4. Solid-state photoreaction

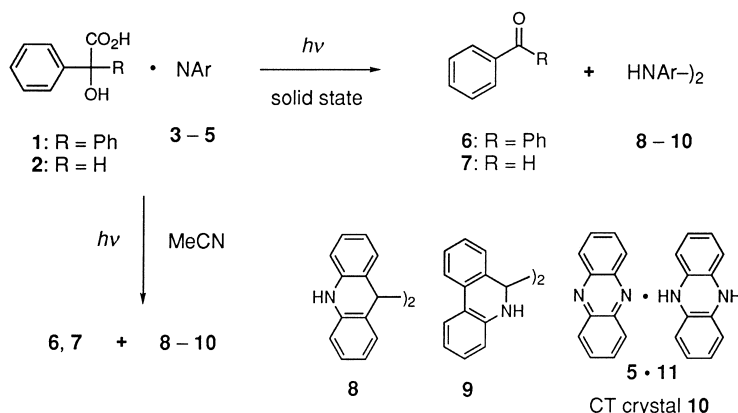
A crystal (20 mg) was pulverized in a mortar, placed between two Pyrex glass plates, and irradiated with a 500-W xenon short arc lamp with a UV transparent filter (290–400 nm irradiation) or a UV cut filter ($>400\text{ nm}$ irradiation) under argon at 15°C for 2 h. The irradiated sample was filtered and the solution was treated with CH_2N_2 followed by HPLC analysis. The results are shown in Scheme 2 and Table 3.

2.5. X-Ray crystallographic analysis

Four crystals of **1-3**, **1-4** and **1-5** were submitted to X-ray structure analysis. Data collections were performed on a Rigaku AFC7R automatic four-circle X-ray diffractometer with a graphite-monochromated Mo $K\alpha$ ($\lambda = 0.71069\text{ \AA}$) radiation. Absorption corrections were applied. No degradation of the crystal by X-ray was ascertained in all cases by repeated monitoring of the three representative reflections every 150 reflections. These structures were solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Atomic parameters were refined by the full matrix least-squares method at the final stage. All the calculations were carried out on teXsan crystallographic software package, Molecular Structure Corporation. The crystal data are listed in Table 1.

3. Results and discussion

Crystallization of the solutions of α -hydroxycarboxylic acids and aza aromatic compounds gave totally seven two-component molecular crystals (Scheme 1). Two kinds of yellow crystals **1-5a** and **1-5b** were simultaneously obtained from an equimolar solution of **1** and **5** in 1 : 1 of acetone and benzene. The yellow colors of **1-5a** and **1-5b** are slightly very different. The molar ratio of **1** and **5** is 2 : 1 for **1-5a**, while **1-5b** includes a water molecule in 1 : 1 : 1. By seeding the desired crystals they can be prepared on a large scale. The molar ratios are 1 : 1 for **1-3**, **1-4**, **2-3** and **2-4**, and 2 : 1 for **2-5**. The solvents, melting points, IR spectral patterns, and elemental analyses are summarized



Scheme 2. Photoreactions in solid state and in solution.

Table 1
Crystal data of the two-component crystals

	1-3	1-4	1-5a	1-5b
<i>Crystal data</i>				
Formula	C ₂₇ H ₂₁ NO ₃	C ₂₇ H ₂₁ NO ₃	C ₄₀ H ₃₂ N ₂ O ₆	C ₂₆ H ₂₂ N ₂ O ₄
<i>M_w</i>	407.47	407.47	636.70	426.47
Crystal system	triclinic	monoclinic	triclinic	triclinic
Space group	<i>P</i> $\bar{1}$	<i>C</i> 2/ <i>c</i>	<i>P</i> $\bar{1}$	<i>P</i> $\bar{1}$
<i>a</i> (Å)	17.052 (4)	23.783 (1)	9.0374 (8)	12.013 (2)
<i>b</i> (Å)	18.194 (2)	7.400 (2)	12.676 (1)	12.979 (2)
<i>c</i> (Å)	6.841 (2)	25.012 (6)	7.9426 (8)	7.360 (2)
α (°)	95.09 (1)	90.0	102.979 (8)	105.90 (1)
β (°)	100.29 (2)	109.28 (1)	113.243 (7)	103.78 (2)
γ (°)	94.06 (1)	90.0	80.901 (8)	94.95 (2)
<i>V</i> (Å ³)	2072.9 (7)	4155 (1)	812.2 (1)	1057.6 (4)
<i>Z</i>	4	8	1	2
<i>D_c</i> (g cm ⁻³)	1.306	1.303	1.302	1.339
<i>F</i> (000)	856.00	1712.00	334.00	448.00
μ /cm ⁻³	0.85	0.85	0.88	0.91
<i>Data collection</i>				
Crystal size (mm)	0.20 × 0.10 × 0.80	0.40 × 0.80 × 0.90	0.40 × 0.40 × 0.60	0.50 × 0.30 × 0.40
$2\theta_{\text{max}}$ (°)	50.0	50.0	50.0	50.0
No. of reflections measured	7561	4072	3055	5089
<i>Final refinement</i>				
No. of observations, <i>I</i> > 3σ(<i>I</i>)	3165	3016	2460	3231
No. of variables	576	287	224	302
<i>R</i>	0.047	0.036	0.037	0.045
<i>R_w</i>	0.061	0.063	0.070	0.066

in Section 2. The crystals **1-3**, **1-4**, **1-5a** and **1-5b** were submitted to X-ray crystallographic analysis to belong to the achiral space groups of *P* $\bar{1}$, *C*2/*c*, *P* $\bar{1}$ and *P* $\bar{1}$, respectively. Table 1 summarizes the X-ray crystal data for the crystals.

Fig. 1 shows the molecular arrangement of **1-3**. Two independent molecular pairs (A and B in Fig. 1(a)) of **1** and **3** co-exist in a centrosymmetrical arrangement within the lattice (*P* $\bar{1}$, *Z*=4). The each carboxylic acid proton of **1** in A and B is transferred to the each N atom of acridine molecule to form C–O[−]...⁺H–N salt bridges with the distances of 1.50 and 1.64 Å, respectively. Intramolecular hydrogen bonding is also formed between the α-hydroxyl group and the carboxylic acid group in each molecule **1** is also formed; the

O–H...O–C distances in A and B are 1.93 and 2.00 Å, respectively. The antipodal molecular pairs of A are arranged in a head-to-tail manner to form a column structure along the *c*-axis; the acridine molecules are almost parallel with the plane-to-plane distance of 3.4 Å (Fig. 1(b)). Similarly a column of B is formed along the *c*-axis. Further, the columns of A and B are separately arranged along the *b*-axis to form a row structure.

The crystal **1-4** (Fig. 2) has a different packing arrangement from that of **1-3**. Two molecules of **1** form a dimer structure between the α-hydroxyl group and the carboxylic acid group through O–H...O–C hydrogen bonding (2.10 Å). Further each molecule of **1** forms intramolecular

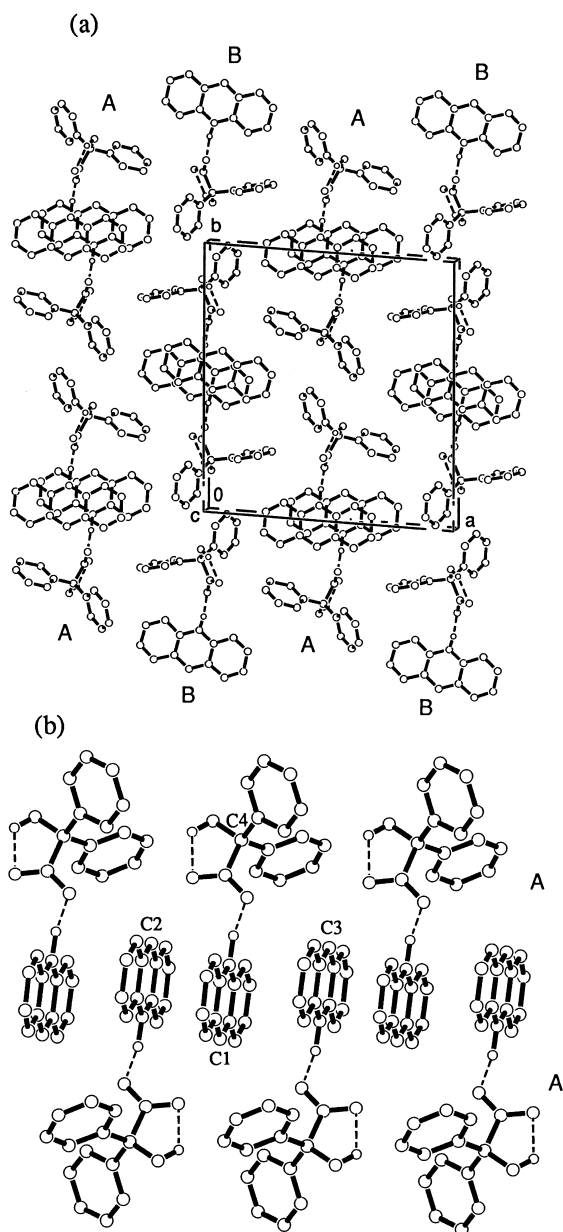


Fig. 1. Molecular arrangement of the crystal 1-3.

O–H...O=C hydrogen bonding (2.24 Å) to give a hydrogen bonded four-member ring. Two molecules of **4** connect to each molecule **1** of the dimer between the N atom and the carboxylic acid group through N...H–O hydrogen bonding (1.38 Å) to complete a 2:2 molecular unit (Fig. 2(b)). The units are arranged in a head-to-tail manner along the *b*-axis to form a parallel stacking structure of **4** with the plane-to-plane distance of 3.8 Å (Fig. 2(b)).

In the 2:1 crystal **1-5a** (Fig. 3), a carboxy dimer of **1** is formed with the O–H...O–C distance of 1.63 Å. The two α -hydroxyl groups of dimer also connect to each N atom of next molecules of **5** with the distance of 2.04 Å to form a 2:1 molecular unit. The unit is centrosymmetrically arranged in the lattice ($P\bar{1}$, $Z=1$). Further, the units connect continuously

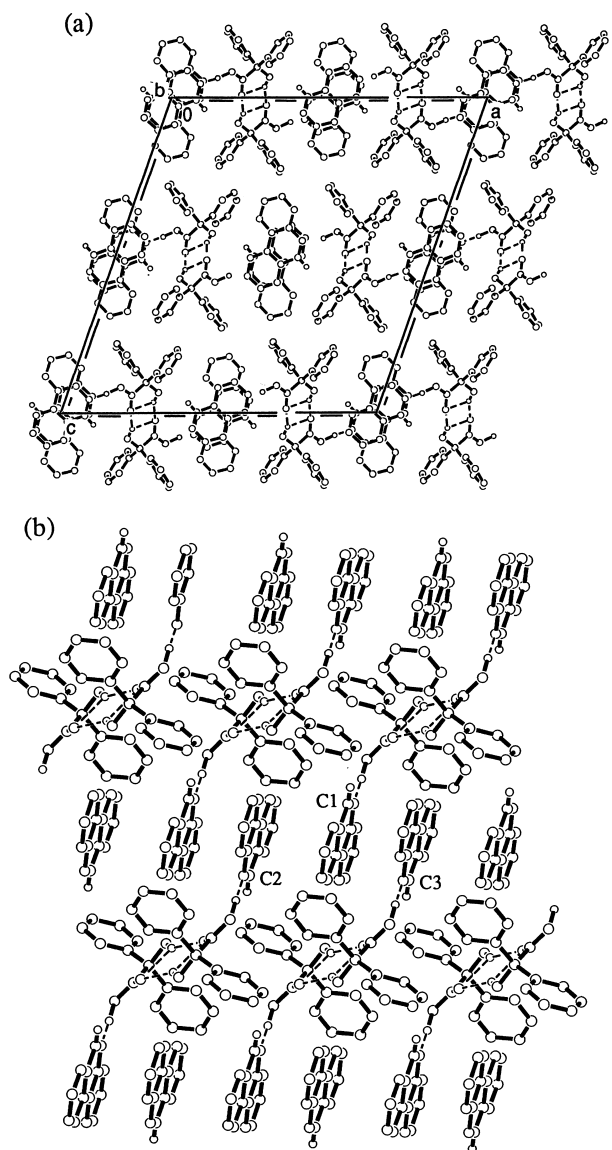


Fig. 2. Molecular arrangement of the crystal 1-4.

through the α -O–H...N hydrogen bonding to form a linear chain along the *b*-axis (Fig. 3(b)).

On the other hand, 1:1:1 crystal **1-5b** (Fig. 4) of **1**, **5** and water has a different packing arrangement from that of 2:1 crystal **1-5a**. The molecules of **5**, **1** and water are continuously connected through the N...HOOC (1.85 Å), α -OH...OH₂ (1.93 Å) and HOH...N (2.13 Å) hydrogen bondings to form a linear chain along the *a*-axis (Fig. 4(b)). Further, the α -HO...HOH (2.24 Å) hydrogen bonded bridges are formed between the molecules of **1** and water in the neighboring antipodal chain to give eight-member rings, leading to the formation of a pair of chains (Fig. 4(a) and (b)). The molecules of **5** are arranged in a parallel manner with the plane-to-plane distance of 4.6 Å in the chain pair. Finally, the molecular chains are stacked to form a column structure along the *c*-axis (Fig. 4(b)). Thus, the small water molecules

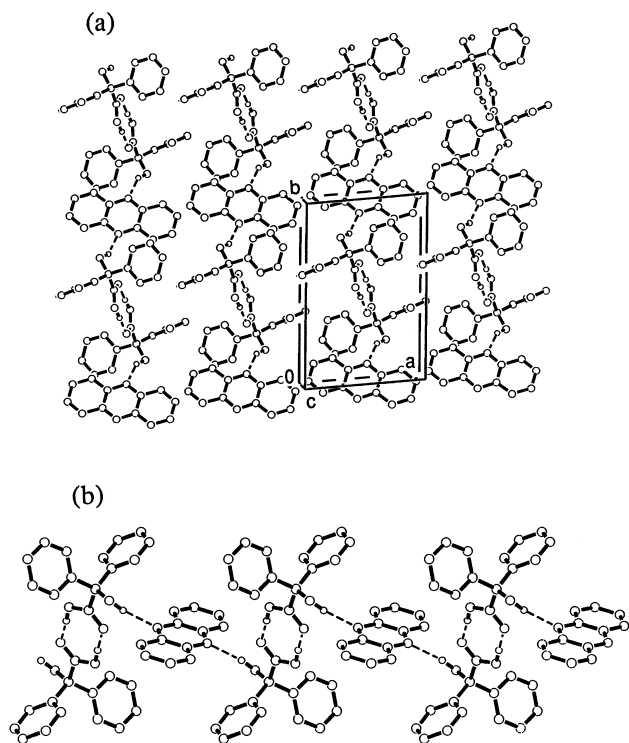
Fig. 3. Molecular arrangement of the crystal **1-5a**.

Table 2

Photoreaction of aza aromatic compounds and α -hydroxycarboxylic acids in acetonitrile

Components	Conversion (%)		Yield (%)	
	Acid	NAr	Ketone	HNAr- ₂
1+3	73	98	86 (6)	100 (8)
1+4	78	91	56 (6)	92 (9)
1+5	90	57	84 (6)	74 (10)
2+3	68	100	26 (7)	98 (8)
2+4	45	57	16 (7)	98 (9)
2+5	90	44	10 (7)	82 (10)

play an important role in the formation of three-component crystal **1-5b**.

Irradiation of α -hydroxycarboxylic acid and aza aromatic compound in acetonitrile under argon bubbling caused decarboxylation and dehydrogenation to give two products as the main products (Scheme 2 and Table 2). They are the corresponding decarboxylative dehydrogenation product benzophenone **6** or benzaldehyde **7**, and the corresponding dimer biacridane **8** or biphenanthridane **9**. Table 2 shows the corrected yields of **6** or **7**, and **8** or **9** based on the consumed acid and aza aromatic compound, respectively. Fairly good yields of **6** were obtained with every aza aromatic compound, while the yields of **7** were low due to the vaporization during separation processes. The high yields of **8** and **9** are due to the high mobility of intermediate radical species in the solution. In the reactions of **1** and **5**, and **2** and **5**, dark blue crystals of charge transfer (CT) complex

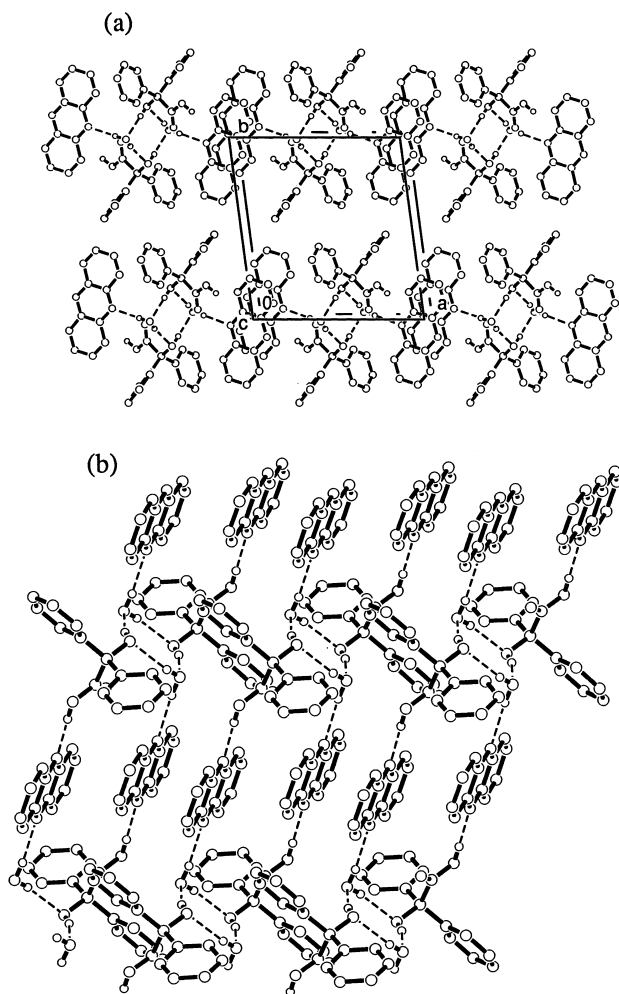
Fig. 4. Molecular arrangement of the crystal **1-5b**.

Table 3

Photoreaction in the two-component molecular crystals from aza aromatic compounds and α -hydroxycarboxylic acids

Entry	Crystal	Irradiation		Conversion (%)		Yield (%)		
		(°C)	gas (nm)	Acid	NAr	Ketone	HNAr- ₂	
1	1-3	15	Ar	290–400	40	36	48 (6)	9 (8)
2	1-3	15	O ₂	290–400	40	24	48 (6)	3 (8)
3	1-3	15	Ar	>400	52	56	60 (6)	11 (8)
4	1-3	-50	Ar	>400	20	23	57 (6)	5 (8)
5	1-4	15	Ar	290–400	42	41	55 (6)	24 (9)
6	1-5a	15	Ar	290–400	42	16	63 (6)	–
7	1-5b	15	Ar	290–400	59	14	90 (6)	–
8	1-5b^a	15	Ar	290–400	100	19	96 (6)	–
9	2-3	15	Ar	290–400	56	42	34 (7)	10 (8)
10	2-4	15	Ar	290–400	51	40	38 (7)	5 (9)
11	2-5	15	Ar	290–400	61	12	33 (7)	–

^a Irradiation for 3 h. Others are 2 h.

10 of phenazine **5** and dihydrophenazine **11** were obtained as the precipitates.

Solid-state irradiation of the two-component crystals caused similar decarboxylative dehydrogenation to give **6** or **7** as the main product (Scheme 2 and Table 3). The yields

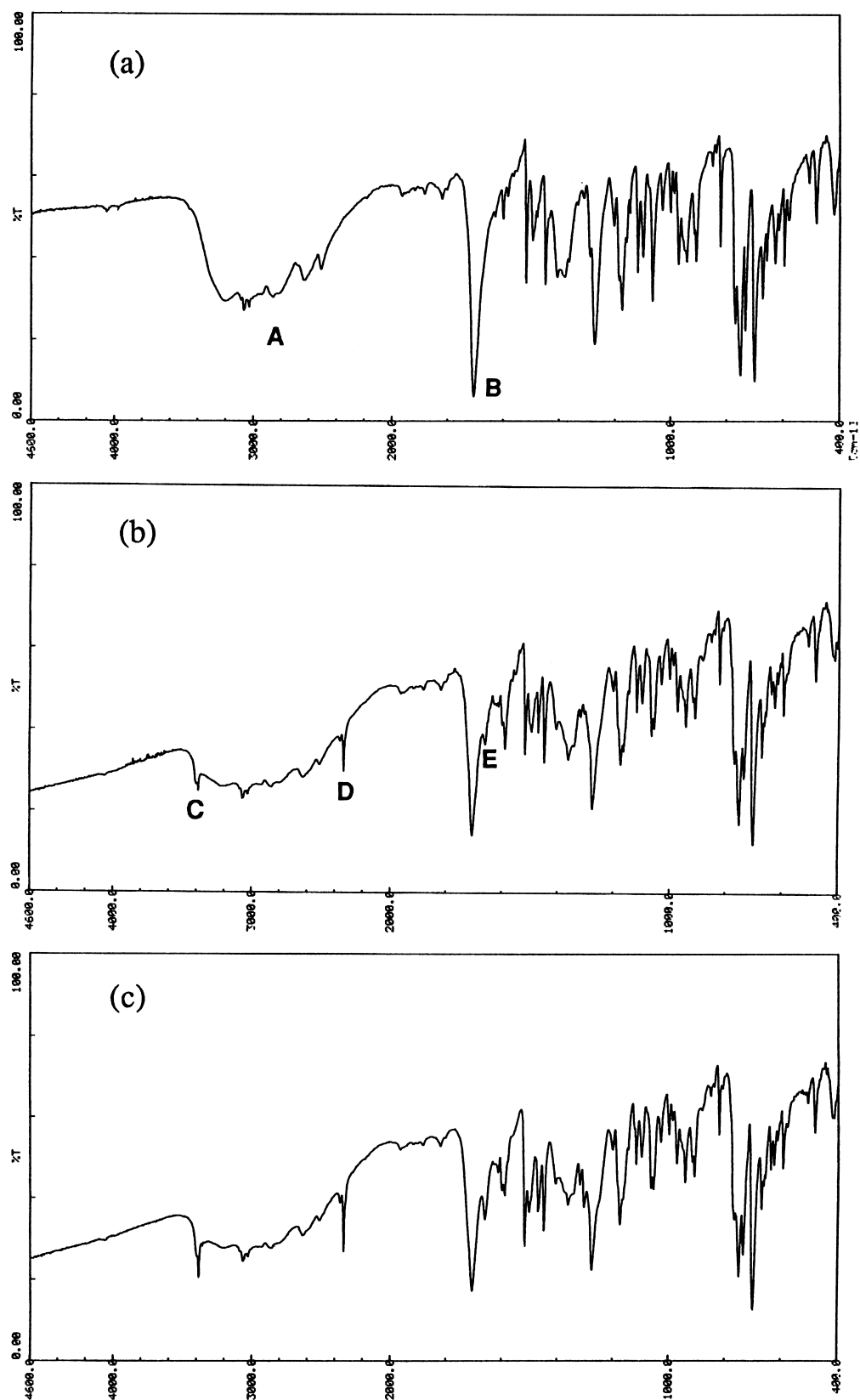
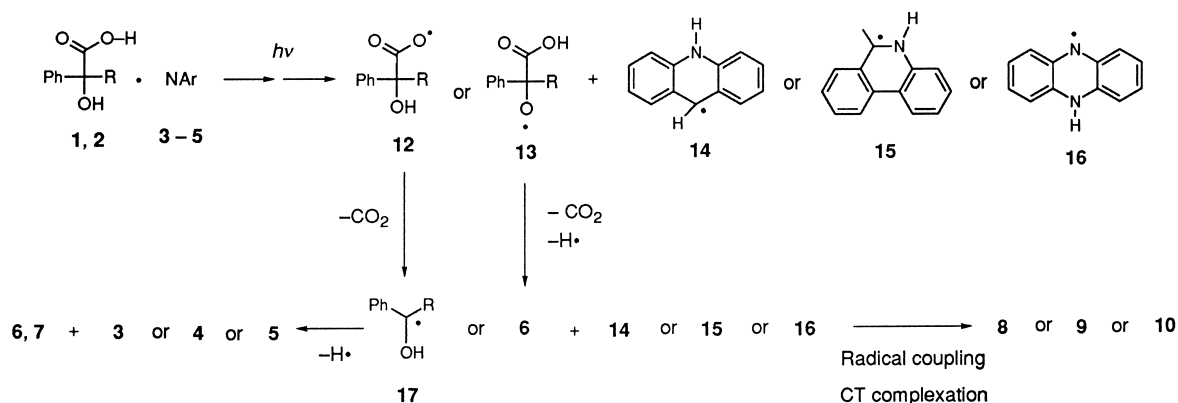


Fig. 5. IR spectra of the crystal **1-5a** after UV irradiation for (a) 0 min, (b) 10 min, and (c) 60 min. A, broad hydrogen bonding, 3600–2000 cm^{-1} ; B, C=O stretch, 1705 cm^{-1} ; C, N–H stretch, 3384 cm^{-1} ; D, CO_2 asymmetric stretch, 2336 cm^{-1} ; E, C=O stretch, 1660 cm^{-1} .



Scheme 3. Possible reaction mechanism.

of corresponding dimer **8** or **9** were however low, different from those obtained by the solution reactions (Table 2). Several biproducts were formed but could not be separated due to the small amounts to result in the low material balances. After irradiation of **1-5a**, **1-5b** and **2-5**, the crystal color changed to dark green, showing the formation of crystalline CT complex **10** of **5** and **11**. However, the yield of **10** could not be determined due to the air oxidation of **11** to **5** when the irradiated sample was dissolved in methanol for HPLC analysis (entries 6–8 and 10).

Several experimental conditions were changed during irradiation of **1-3**. The reaction occurred in not only argon but also oxygen to give similar yields of **6** and **8**, suggesting no participation of oxygen in the reaction (entries 1 and 2). The photoreactivity was retained even at -50°C with accompanying slight decrease of the reaction rate (entries 3 and 4). In addition, the reaction was brought about by irradiation using not only UV light (290–400 nm) but also visible light ($>400\text{ nm}$) (entries 3 and 4). Therefore, this reaction can be induced by the excitation of the acridine molecule **3** at $>400\text{ nm}$, and **3** acts as the excited species in the crystal **1-3**.

The reaction process was traced by the measurement of IR spectra of **1-5** as KBr film (Fig. 5). Before irradiation (a), the crystal has the broad hydrogen bonding band A ($3600\text{--}2000\text{ cm}^{-1}$) and the C=O stretching band B (1705 cm^{-1}). Upon irradiation for 10 min (b), the CO_2 asymmetrical stretching band D appeared at 2336 cm^{-1} , giving the evidence for decarboxylation. The small N–H stretching band C (3384 cm^{-1}) of product **11** and C=O stretching E (1660 cm^{-1}) of product **6** were also observed. Prolonged irradiation for 60 min (c) increased these peaks C, D and E.

Scheme 3 shows the possible reaction mechanism. Irradiation of crystal causes electron transfer from acid to aza aromatic compound followed by proton transfer, or conversely proton transfer followed by electron transfer to afford α -hydroxycarboxylate radical (**12**), and hydroacridine radical (**14**), hydrophenanthridine radical (**15**) or phenazine

semiquinone radical (**16**). Next, decarboxylation of **12** produces ketyl radical (**17**) followed by dehydrogenation to lead to the formation of **6** or **7**. Radical coupling of **14** or **15** also occurs to give the dimer **8** or **9**, while in the crystals **1-5a**, **1-5b** and **2-5**, disproportionation of semiquinone radical **16** into **5** and **11** followed by CT complexation affords **10**.

Although these reaction processes inevitably lead to the alteration of the crystal lattice, the reaction paths at the initial reaction stage can be discussed on the basis of the distances estimated from the crystallographic data. In the crystal **1-3**, the $\text{C-O}^{\ominus}\cdots\text{H-N}^{\oplus}$ salt structures are initially formed (Fig. 1); therefore the irradiation causes electron transfer from **1** to **3** through the salt bridges of A pair (1.50 \AA) and B pair (1.64 \AA) to give the radical species **12** and **14** as the canonical forms. Next decarboxylation of **12** followed by dehydrogenation of **17** in highest priority to give **6** as the main product. Dimerization of **14** occurs in a less extent due to the longer coupling distance of C1–C2 (4.96 \AA) and C1–C3 (5.33 \AA) in the column A (Fig. 1(b)), C1–C2 (5.22 \AA) and C1–C3 (5.43 \AA) in the column B to give **8** as the minor product. Radical couplings between **17** and **14** may occur over the longer distances, e.g., C4–C2 (5.23 \AA) and C4–C3 (5.22 \AA) to give minor cross-coupling products, but the separation was unsuccessful due to the very small amounts.

Similar decarboxylative dehydrogenation occurs within the 2:2 molecular unit of **1** and **4** of the crystal **1-4** to give **6** as the main product. The dimerization of **15** occurring in a less extent with the longer distance of C1–C2 (4.57 \AA) and C1–C3 (5.63 \AA) (Fig. 2(b)) affords **9** as the minor product.

In the crystal **1-5a**, the α -hydroxyl group of **1** is connected to the N atom of **5** to form the $\alpha\text{-O-H}\cdots\text{N}$ hydrogen bonding (Fig. 3), which is different from the $\text{CO}_2\text{H}\cdots\text{N}$ hydrogen bonding in the other crystals **1-3**, **1-4** and **1-5b**. Therefore, upon irradiation of **1-5a**, electron transfer may occur from **1** to **5** through the $\alpha\text{-O-H}\cdots\text{N}$ hydrogen bonding (2.04 \AA) followed by proton transfer to give α -oxycarboxylic acid radical **13** and phenazine semiquinone radical **16**. Next, cleavage of **13** gives **6** as the main product and CO_2H radical, which

is immediately decomposed to CO₂ and H⁺. The occurrence of decarboxylation was confirmed by the appearance of CO₂ band in the IR spectrum (peak D in Fig. 5(b)). Furthermore, disproportionation of **16** into **5** and **11** followed by CT complexation results in the formation of crystalline CT complex **10**, which was also confirmed by the color change from yellow to dark green after irradiation. However, when the irradiated mixture was worked up to the solution state for HPLC analysis, **11** in **10** was immediately air-oxidized to **5** to lead to obtain the low conversion (16%) of **5** (entry 6 in Table 3).

On the other hand, the three-component crystal **1·5b** of **1**, **5** and water, has the usual CO₂H...N hydrogen bonding between **1** and **5** (Fig. 4(b)). Therefore, photoinduced electron transfer and proton transfer can occur more smoothly through the CO₂H...N hydrogen bonding (1.85 Å) than those through the α-O-H...N hydrogen bonding in **1·5a**. One of the reasons why such the high yields (90 and 96%) of **6** was obtained by the reaction of **1·5b** (entries 7 and 8 in Table 3) is due to the clean reaction without significant side reactions, understood from the result of HPLC analysis. The parallel stacking structure of **5** with the relatively short plane-to-plane distance of 4.6 Å (Fig. 4(b)) may also contribute to the smooth disproportionation of **11** followed by CT complexation to **10**.

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