

# Preparation and photoreaction of two-component molecular crystals between aza-aromatic compounds and *N*-phenylglycine<sup>1</sup>

Hideko Koshima<sup>a,b</sup>, Kuiling Ding<sup>b,2</sup>, Takashi Miura<sup>b</sup>, Teruo Matsuura<sup>b,\*</sup>,<sup>3</sup>

<sup>a</sup> PRESTO, Research Development Research Corporation of Japan, Faculty of Science and Technology, Ryukoku University, Seta, Otsu 520, Japan

<sup>b</sup> Department of Materials Chemistry, Faculty of Science and Technology, Ryukoku University, Seta, Otsu 520, Japan

Received 9 September 1996; accepted 25 November 1996

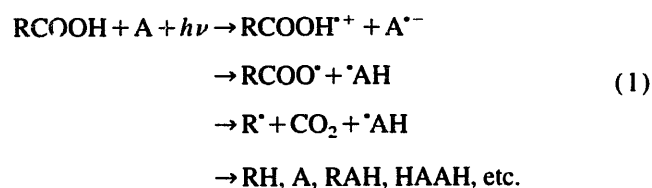
## Abstract

Crystalline 1 : 1 two-component molecular compounds ("two-component molecular crystals") crystallized from a solution of a mixture of an aza-aromatic compound (acridine or phenanthridine) and *N*-phenylglycine. These two-component molecular crystals were characterized by various physical methods, including X-ray crystallographic analysis. UV irradiation of the crystals was carried out in the solid and solution phases to give aniline, *N*-methylaniline, formanilide and decarboxylating condensation products. The product ratio was dependent on the reaction conditions. Particular attention is focused on the selectivity of the photoreactions in the solid state compared with those in the solution phase and the factors controlling the photoreactions. © Elsevier Science S.A.

**Keywords:** Aza-aromatic compounds; *N*-Phenylglycine; Solid state bimolecular photoreactions; Two-component molecular crystals

## 1. Introduction

A large number of photoinduced electron transfer (PET) reactions are known [1]. Carboxylic acids (RCOOH) undergo a PET reaction, followed by decarboxylation and decarboxylating condensation, when aza-aromatic compounds [2–5], dye sensitizers [3] and polycyanoaromatics [6,7] are used as electron acceptors (A) as shown below



During the course of studies on the bimolecular photoreactions between two different organic molecules in the solid state [8,9], we utilized this type of PET reaction for the mixed crystals formed between aralkyl carboxylic acids and electron

acceptors, and found that the solid state photoreactions usually occur with a remarkable specificity compared with the solution phase photoreactions. For example, the following photoreactions occur in the crystal lattice of a molecular compound formed between two components: stoichiometrically sensitized decarboxylation in a two-component molecular crystal formed between 3-indoleacetic acid and phenanthridine to give 3-methylindole quantitatively [10]; retention of the chirality of (S)-(+)-2-(6-methoxy-2-naphthyl)propanoic acid during decarboxylating condensation with 1,2,4,5-tetracyanobenzene in a crystalline charge transfer (CT) complex [11]; photodecarboxylation and decarboxylating condensation of arylacetic acids by excitation of the CT bands of the complexes formed with 1,2,4,5-tetracyanobenzene in the solid state [12]; absolute asymmetric synthesis via photodecarboxylating condensation in a chiral two-component molecular crystal crystallized from a solution of acridine and diphenylacetic acid [13]. A similar type of decarboxylating condensation was also found to occur at the interface between polycrystalline mixtures of certain arylacetic acids and 1,2,4,5-tetracyanobenzene [14].

This paper deals with the preparation of the two-component molecular crystals acridine–*N*-phenylglycine and phenanthridine–*N*-phenylglycine and their solid state and solution phase photoreactions.

\* Corresponding author.

<sup>1</sup> Dedicated to Professor Pill-Soon Song on the occasion of his 60th birthday.

<sup>2</sup> Guest researcher from Zhengzhou University, People's Republic of China.

<sup>3</sup> Present address: 21-26 Kawashima-Gondencho, Saikyoku, Kyoto 615, Japan.

## 2. Experimental details

### 2.1. General procedure

<sup>1</sup>H Nuclear magnetic resonance (NMR) spectra were measured on a 60 MHz JEOL spectrometer with tetramethylsilane as internal standard. IR spectra were measured on a JASCO FT-IR 8300 with KBr discs. Differential scanning calorimetry (DSC) was performed on a Rigaku Thermoflex TAS-200 DSC8230D and melting points (m.p.) were uncorrected. Elemental analyses were carried out with a Yanaco CHN Corder MT-5. High performance liquid chromatography (HPLC) was used to determine the products on a Waters HPLC system with a photodiode array detector. All the reagents were commercially available.

### 2.2. Preparation of two-component molecular crystals

A solution of an equimolar mixture of acridine (**1**) or phenanthridine (**2**) and *N*-phenylglycine (**3**) in acetonitrile was slowly evaporated at room temperature to deposit the corresponding two-component molecular crystals **4** and **5** respectively.

The acridine–*N*-phenylglycine two-component molecular crystal (**4**) was obtained as yellow prisms from acetonitrile (m.p., 135.0–137.0 °C). DSC showed a peak at 131.4 °C. Analysis: calculated for C<sub>21</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>: C, 76.34%; H, 5.49%; N, 8.48%; found: C, 76.38%; H, 5.57%; N, 8.54%.

The phenanthridine–*N*-phenylglycine two-component molecular crystal (**5**) was obtained as pale yellow prisms from acetonitrile (m.p., 112.0–114.0 °C). DSC showed a peak at 109.8 °C, whereas phenanthridine (**2**) and *N*-phenylglycine (**1**) showed peaks at 124.8 and 107.1 °C respectively. IR (KBr): 3421, 3061, 3048, 2360, 1612, 1599, 1578, 1509, 1448, 1352, 1231, 922, 754, 742, 690, 456 cm<sup>-1</sup>. Analysis: calculated for C<sub>21</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>: C, 76.34%; H, 5.49%; N, 8.48%; found: C, 76.39%; H, 5.65%; N, 8.46%.

### 2.3. Solid state photoreactions of two-component molecular crystals **4** and **5**

The two-component molecular crystal **4** was pulverized in a mortar to a fine powder, placed between two Pyrex glass plates and irradiated with a high-pressure mercury lamp with Pyrex glass housing or a 500 W xenon short arc lamp with a UV filter under argon at a given temperature in an irradiation apparatus [15].

#### 2.3.1. Two-component molecular crystal **4** (preparative scale)

In a preparative experiment, two-component molecular crystal **4** (800 mg, 2.42 mmol) was irradiated as above for 4 h at 15 °C. The irradiated solid was mixed with acetonitrile (20 ml) and the insoluble polymeric material (60 mg) was separated by filtration. The filtrate was applied to silica gel thin layer chromatography (TLC) plates and eluted with 5 : 1 benzene–ethyl acetate to give **6** (85 mg, 12.3% yield) and **7** (7 mg, 1% yield). Compound **6**: m.p., 155.0–157.0 °C (from 2 : 1 methanol–water); UV λ<sub>max</sub> (CH<sub>3</sub>CN): 200 nm (log ε = 4.81), 250.3 (4.32), 283.5 (4.23); IR (KBr): 3400, 3390, 2920, 1598, 1580, 1508, 1478, 1304, 750, 740, 694 cm<sup>-1</sup>; <sup>1</sup>H NMR (CD<sub>3</sub>CN) δ: 6.47–7.27 (m, 13H), 4.17 (t, 1H, *J* = 7 Hz), 3.17 (q, 2H, *J* = 7 Hz); analysis: calculated for C<sub>20</sub>H<sub>18</sub>N<sub>2</sub>: C, 83.88%; H, 6.34%; N, 9.78%; found: C, 84.22%; H, 6.48%; N, 9.80%. Compound **7**: m.p., 168–171 °C (from CH<sub>3</sub>CN); IR (KBr): 3380, 3045, 2810, 1605, 1582, 1476, 1295, 760, 715, 608 cm<sup>-1</sup>; <sup>1</sup>H NMR (CD<sub>3</sub>CN) δ: 6.50–7.15 (m, 14H), 3.93 (broad s, 4H, non-splitting CH and CH<sub>3</sub> protons).

#### 2.3.2. Two-component molecular crystal **4** (analytical scale)

Pulverized **4** (20 mg) was placed between two Pyrex glass plates and irradiated for 2 h at a given temperature (15, –30 and –70 °C) with a 500 W xenon short arc lamp using a UV filter (Toshiba UV-D36C filter or L-39 filter). The irradiated solid was submitted to quantitative HPLC analysis (C<sub>18</sub> col-

Table 1  
Photoreactions of two-component molecular crystal **4** (acridine–*N*-phenylglycine) in the solid state and solution phase

Irradiation conditions		Conversion (%)		Product yields based on consumed <b>3</b> (%)					
Temperature (°C)	Lamp and filter	<b>1</b>	<b>3</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>
Solid state									
–70	<sup>a</sup>	0	0	0	0	0	0	0	0
–30	<sup>a</sup>	6	2	60	0	30	0	0	0
15	<sup>a</sup>	13	13	37	2	16	0	0	0
15	<sup>b</sup>	15	21	38	1	19	0	0	Trace
15	<sup>c</sup>	44	53	38	3	17	0	0	30
Solution phase									
15	<sup>c</sup>	94	91	7	2	80	10	92 <sup>d</sup>	0

<sup>a</sup>Xenon short arc lamp (500 W) with Toshiba UV-D36C filter.

<sup>b</sup>Xenon short arc lamp (500 W) with Toshiba L-39 filter.

<sup>c</sup>High-pressure mercury arc lamp (400 W) with Pyrex housing.

<sup>d</sup>This yield is based on consumed **1**.

Table 2  
Photoreactions of two-component molecular crystal **5** (phenanthridine-*N*-phenylglycine) in the solid state and solution phase

Irradiation conditions		Conversion (%)		Product yields based on consumed <b>3</b> (%)		
Temperature (°C)	Lamp and filter	<b>2</b>	<b>3</b>	<b>8</b>	<b>9</b>	<b>11</b>
<b>Solid state</b>						
-70	a	43	0	0	0	0
-30	a	44	0	0	0	0
-15	a	32	0	0	0	0
15	b	54	76	3	0	2
<b>Solution phase</b>						
-70	a	50	50	13	4	0
-30	a	42	63	21	7	0
-15	a	43	73	24	2	0
15	b	62	60	22	3	2

<sup>a</sup>Xenon short arc lamp (500 W) with Toshiba UV-D36C filter.

<sup>b</sup>High-pressure mercury arc lamp (400 W) with Pyrex housing.

umn, eluting with gradient solvents, 50% (v/v) methanol-water to 100% methanol). The results are summarized in Table 1.

### 2.3.3. Two-component molecular crystal **5**

Pulverized **5** (20 mg) was placed between two Pyrex glass plates and irradiated with a 400 W high-pressure mercury lamp for 1 h at a given temperature under argon. The irradiated solid was submitted to quantitative HPLC analysis using a C<sub>18</sub> column and eluting with gradient solvents (50% (v/v) methanol-water to 100% methanol). The results are shown in Table 2.

## 2.4. Solution phase photoreactions

### 2.4.1. Acridine (**1**) and *N*-phenylglycine (**3**)

Two-component molecular crystal **4** (165 mg, 0.50 mmol) was dissolved in acetonitrile (10 ml) and irradiated for 3 h with a 400 W high-pressure mercury lamp at 15 °C with argon bubbling. The colourless crystals deposited were collected by filtration, and were identified as 9,9'-biacridane (**10**) [3] (77.4 mg, 92% yield). The filtrate was evaporated and the residue was submitted to quantitative HPLC analysis as in Section 2.3.2. The products (yields based on the consumed starting materials) were found to be **6** (7%), **7** (2%), **8** (80%) and **9** (10%) with 94% and 91% conversion of **1** and **3** respectively. These results are listed in Table 1 for comparison.

### 2.4.2. Phenanthridine (**2**) and *N*-phenylglycine (**3**)

Two-component molecular crystal **5** (165 mg, 0.50 mmol) was dissolved in acetonitrile (10 ml) and irradiated with a 400 W high-pressure mercury lamp for 4 h at 15 °C. The irradiated mixture was filtered from a trace of solid materials and the filtrate was evaporated. The residue was submitted to HPLC analysis as in Section 2.3.2 for identification of the products and conversion of the starting materials. Under similar conditions, acetonitrile solutions of **5** were irradiated at

temperatures lower than 0 °C with a 500 W xenon short arc lamp filtered with a UV-D36C filter. The results are summarized in Table 2.

### 2.4.3. X-Ray crystallographic analysis of two-component molecular crystals **4** and **5**

Crystals of **4** and **5** having approximate dimensions of 0.40 mm × 0.49 mm × 0.70 mm and 0.10 mm × 0.30 mm × 0.50 mm respectively were mounted on a glass fibre. Data collections were performed on a Rigaku AFC7R automatic four-circle X-ray diffractometer equipped with graphite monochromated Mo K $\alpha$  ( $\lambda = 0.71069 \text{ \AA}$ ) radiation and a rotating anode. Absorption correction was applied. No degradation of the crystal by X-rays was ascertained in both cases by repeated monitoring of the three representative reflections every 150 reflections. The 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 using the Molecular Structure Corporation teXan crystallographic software package. Detailed crystal data are summarized in Table 3. The details of these X-ray crystallographic structural analyses will be deposited at the Cambridge Crystallographic Data Centre.

## 3. Results and discussion

In contrast with the previous report on the photolysis of an acetonitrile solution of acridine (**1**) and *N*-phenylglycine (**3**) by Brimage et al. [3], who identified carbon dioxide (61% yield), aniline (**8**) (55%) and formanilide (**9**) (30%), we isolated the decarboxylating condensation products **6** (7%) and **7** (2%) and 9,9'-biacridane (**10**) (92%), in addition to **8** (80%) and **9** (10%), in the photolysis of an acetonitrile solution of **1** and **3** (Scheme 1). In our case, the material balance of the photoreaction was quite good.

Table 3  
X-Ray crystallographic data for two-component molecular crystals **4** (acridine-*N*-phenylglycine) and **5** (phenanthridine-*N*-phenylglycine)

Parameter	4	5
Formula	C <sub>21</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub>	C <sub>21</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub>
<i>f</i> <sub>w</sub>	330.39	330.39
Crystal system	Monoclinic	Monoclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i> (#14)	<i>C</i> 2/ <i>c</i> (#15)
<i>Z</i>	4	8
Cell constants		
<i>a</i> (Å)	9.58(2)	29.884(7)
<i>b</i> (Å)	12.01(2)	5.397(5)
<i>c</i> (Å)	15.50(2)	24.915(8)
β (°)	102.4(1)	123.94(1)
<i>V</i> (Å <sup>3</sup> )	1741(4)	3333(3)
<i>D</i> <sub>calcd</sub> (g cm <sup>-3</sup> )	1.260	1.316
μ (cm <sup>-1</sup> )	0.82	0.86
Radiation	Mo Kα	Mo Kα
2θ <sub>max</sub> (°)	50.0	50.0
Scan mode	ω-2θ	ω-2θ
No. of reflections measured	3441	3358
No. of unique reflections	3240	3286
No. with <i>I</i> > 3.00σ( <i>I</i> )	2047	1350
No. of variables	227	298
<i>R</i> (%)	3.8	5.1
<i>R</i> <sub>w</sub> (%)	5.7	6.6

In order to compare the solution phase photoreaction of **1** and **3** with the solid state photoreaction, we tried to obtain a two-component molecular crystal between acridine (**1**) and *N*-phenylglycine (**3**). Correlations between the crystal structures of the substrates and the photoproducts have been demonstrated to be very important for the diagnosis of solid state bimolecular photoreactions of two-component molecular crystals [8,10–13], as well as for the diagnosis of solid state monomolecular and bimolecular photoreactions of single crystalline compounds (originally reported by Cohen and Schmidt [16,17] and by other investigators [18,19]).

A 1 : 1 two-component molecular crystal **4** was successfully obtained by the slow evaporation of an equimolar solution of **1** and **3** in acetonitrile as yellow prisms (m.p., 131.4 °C (a single DSC peak), which is higher than those of **1**

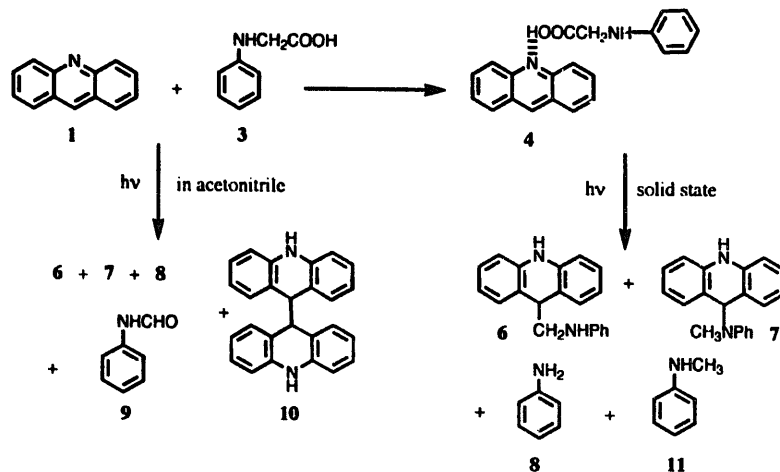
(108.6 °C) and **3** (124.8 °C) indicating the formation of a molecular compound). This was confirmed by X-ray crystallographic analysis of a single crystal of **4**. The molecular structure and crystal packing are shown in Figs. 1 and 2 respectively. Selected crystal data are listed in Table 3. The important features of the crystal structure of the molecular compound **4** are that the *N*-phenylglycine molecule has a nearly coplanar geometry due to an internal –NH···O=C < hydrogen bond (2.32 Å), the molecules **1** and **3** are connected through a –COOH···N≡ hydrogen bond (1.48 Å) and the distance between C14 (CH<sub>2</sub>) and C7' of the acridine molecule of an adjacent molecular compound **4** is 4.20 Å.

The solid state UV irradiation of the two-component molecular crystal **4** was carried out under various conditions (Table 1). Photoreaction occurred at a lower rate as the irradiation temperature was decreased. Compared with the solution phase photoreaction, the solid state reaction showed several different features in the product ratio.

1. The decarboxylating condensation product **6** became the major product, whereas the yield of aniline (**8**) was decreased.
2. Unlike the solution phase photoreaction, no formation of formanilide (**9**) and 9,9'-biacridane (**10**) was observed.
3. The formation of *N*-methylaniline (**11**) was observed when a high-pressure mercury arc lamp was employed, but we can offer no reasonable interpretation for this particular case.

The photoreaction of **1** and **3** in both the solid state and solution phase is interpreted to be initiated by a PET reaction between **1** and **3**, followed by decarboxylation and successive pathways as reported previously [2–7]. A possible mechanism is illustrated in Scheme 2. Thus a PET reaction takes place between acridine (**1**) and *N*-phenylglycine (**3**) to give a radical ion pair (**1**<sup>·-</sup>–**3**<sup>·+</sup>), which undergoes proton transfer to form a radical pair **12**–**13**. Decarboxylation occurs within this radical pair to give a key radical pair **12**–**14** which can account for the formation of all the products.

Two hydroacridyl radicals (**12**) couple to 9,9'-biacridane (**10**). This homocoupling reaction occurs only in the solution



Scheme 1.

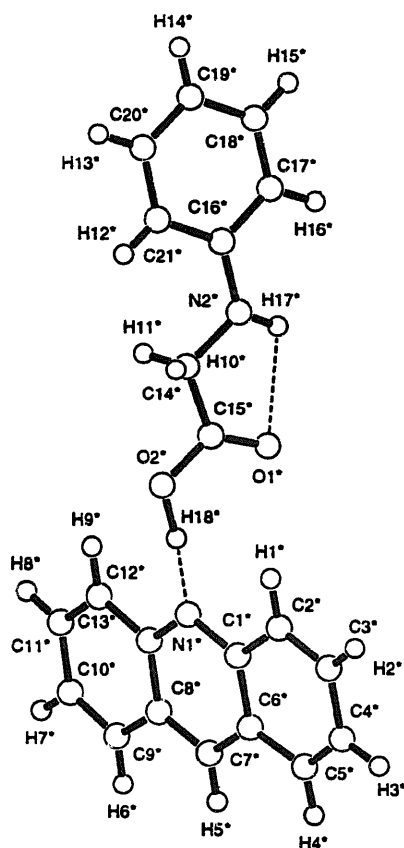
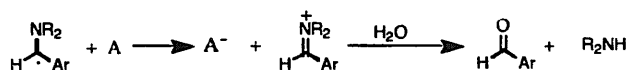


Fig. 1. Molecular structure of two-component molecular crystal 4.

phase, since in the solid state the distance between the C7 and C7' carbon atoms of the two adjacent radicals is too far to undergo homocoupling; this distance is estimated to be close to 6.82 Å assuming that molecular motions are small in the crystal lattice during photolysis. In contrast, heterocoupling within the radical pair 12–14 should occur more easily in the solid state, since the distance between the radical carbon

atoms of 12 and 14 is estimated to be close to 4.20 Å based on the distance between C14 and C7' of two adjacent molecules of 4 in the crystal lattice as described above. Another heterocoupling product 7 should arise from 12 and the *N*-methylanilino radical 15 which is the 1,2-hydrogen migration product of 14. The distance between the radical centres in 15 and 12 is estimated to be sufficiently close for radical coupling based on the distance (3.81 Å) between N2 and C7' in the molecular crystal 5. The formation of *N*-methylaniline (11) is simply interpreted as a result of hydrogen transfer from 12 to 14. Aniline (8) and formanilide (9) may be secondary products from 14. The radical 14 undergoes electron transfer to give a methyleneanilinium cation 16 which can be hydrolysed to give aniline (8). A similar type of electron transfer of a certain  $\alpha$ -amino radical, leading to amine and carbonyl compounds as shown in the following equation, has been proposed [20].



The formation of 8 is a minor pathway compared with solution phase photolysis, indicating that in the solid state the heterocoupling reaction leading to 6 is more favourable due to the molecular arrangement in the crystal lattice. Finally, the formation of formanilide (9), which occurs as a minor pathway only in the solution phase, may be caused via the oxygenation of 14 with a trace of oxygen contamination in argon.

The two-component molecular crystal 5 was similarly obtained by the slow evaporation of an equimolar solution of phenanthridine (2) and *N*-phenylglycine (3) in acetonitrile. The crystal 5 crystallizes as pale yellow prisms (m.p., 109.8 °C (a single DSC peak), which is between those of 2 (107.1 °C) and 3 (124.8 °C)). X-Ray crystallographic analysis (Figs. 3 and 4) confirms that 5 is a 1 : 1 molecular compound.

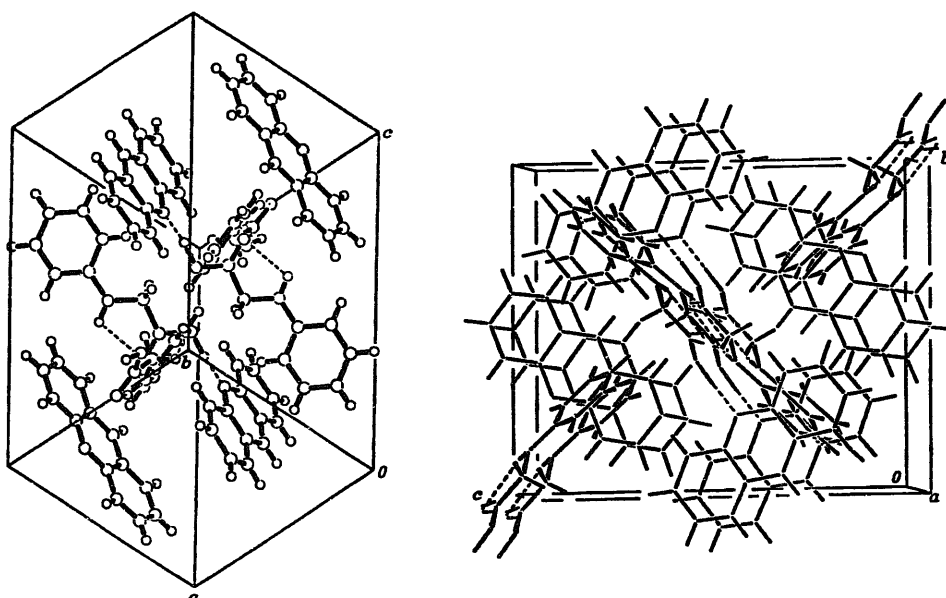


Fig. 2. Molecular packing in two-component molecular crystal 4.

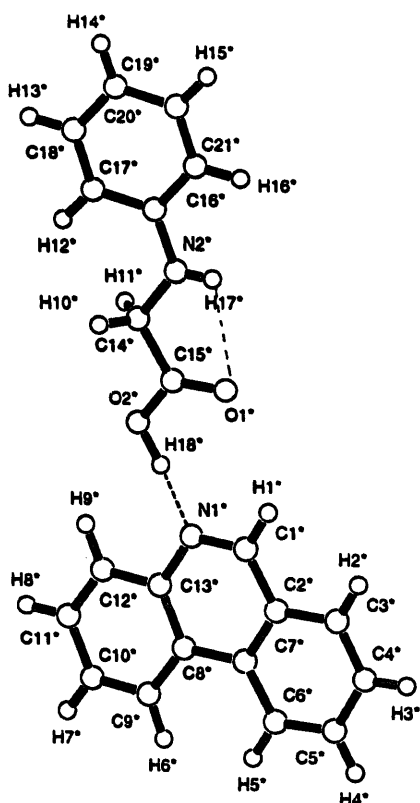
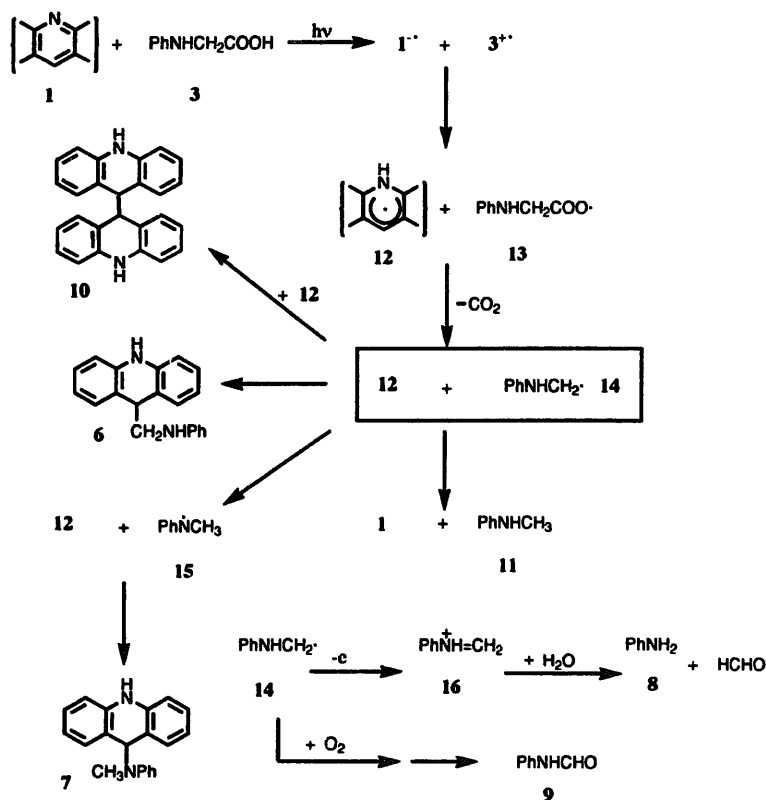


Fig. 3. Molecular structure of two-component molecular crystal 5.

Selected crystal data are listed in Table 3. In the crystal lattice of 5, the two component molecules are connected through a  $-\text{COOH}\cdots\text{N}\equiv$  hydrogen bond (1.63 Å) and the *N*-phenylglycine molecule has a nearly coplanar geometry due to an internal  $-\text{NH}\cdots\text{O}=\text{C}$  hydrogen bond (2.39 Å) as shown in Fig. 3. Another structural feature is shown in Fig. 4: a number of molecules of 5 stack with an intermolecular separation of 5.40 Å in a head-to-head manner, and this stacking column intersects with another stacking column of 5 at approximately right angles.

The solid state and solution phase irradiations of two-component molecular crystal 5 were carried out under various conditions. The results are summarized in Table 2. We were able to identify only products 8, 9 and 11 originating from *N*-phenylglycine (3) as in Scheme 3. However, we could not isolate any product originating from phenanthridine (2), such as coupling products analogous to 6, 7 and 10 in Scheme 1, although the estimated distances between C14 and C1' for the heterocoupling of radical 14 and the 9-(1-hydrophenanthridyl) radical and between C1 and C1' for the homocoupling of radical 14 are small enough for the formation of such coupling products on the basis of the C14–C1' (4.43 Å) and C1–C1' (5.40 Å) distances in the crystal lattice of 5 (see Fig. 3 for numbering).

The material balance was low in both solid state and solution phase reactions. At low temperatures (below 0 °C), no

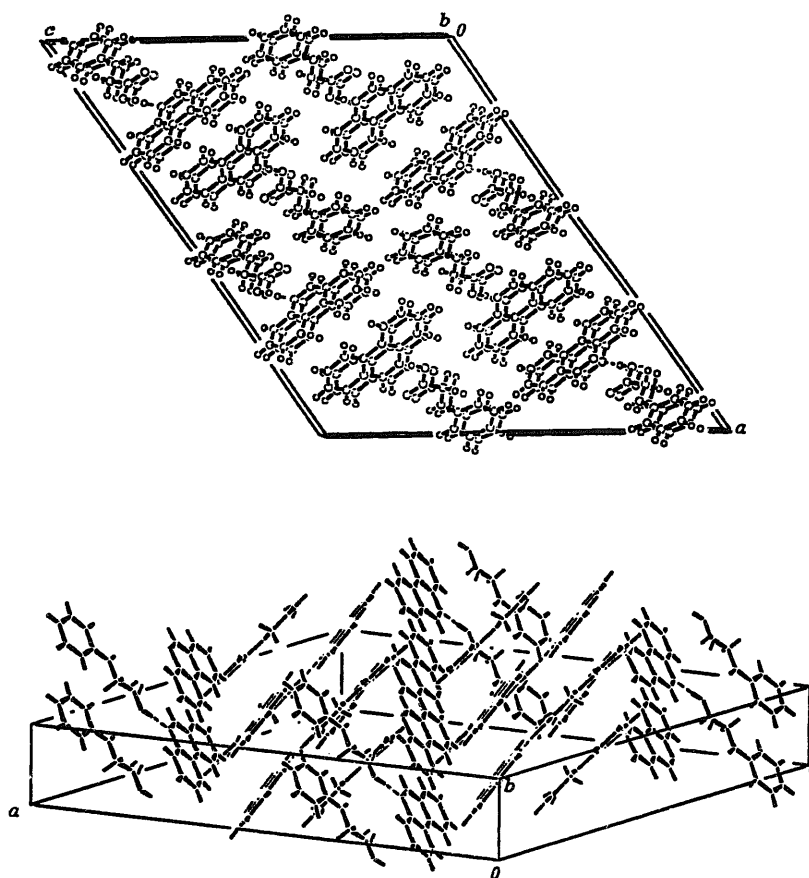


Fig. 4. Molecular packing in two-component molecular crystal 5.

consumption of **3** was observed in the solid state photolysis, whereas at room temperature both **2** and **3** were consumed to give aniline (**8**) and *N*-methylaniline (**11**) in low yield. Attempts made to isolate radical coupling products, such as **6**, **7** and **10**, in the photolysis of the phenanthridine–*N*-phenylglycine system were unsuccessful, resulting in the formation of many unstable products. The solution phase irradiations gave better yields of aniline (**8**), in addition to a small amount of formanilide (**9**), but *N*-methylaniline (**11**) was obtained in a small yield only at room temperature. We can offer no reasonable interpretation for the messy photo-reactions of the phenanthridine–*N*-phenylglycine system. In contrast, the solid state and solution phase irradiations of the two-component molecular crystal formed between phenan-

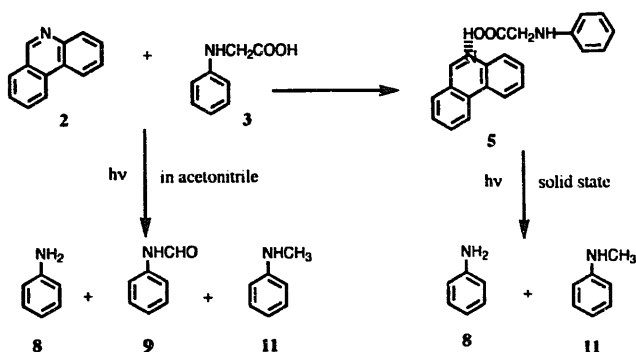
thridine and 3-indolepropionic acid have been shown to give radical coupling products in moderate yields [10].

### Acknowledgements

This work was partially supported by a Grant-in-Aid for Scientific Research from the Ministry of Education, Science and Culture of Japan.

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Scheme 3.

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