[2+2]Photocycloaddition of 2-(dibenzylamino)ethyl 3-benzoylacrylate in a crystalline state: weak intermolecular interactions as important factors determining efficiency of the solid-state photoreaction

Tadashi Hasegawa,* Kaori Ikeda and Yuko Yamazaki

Department of Chemistry, Tokyo Gakugei University, 4-1-1 Nukuikitamachi, Koganei, Tokyo 184-8501, Japan
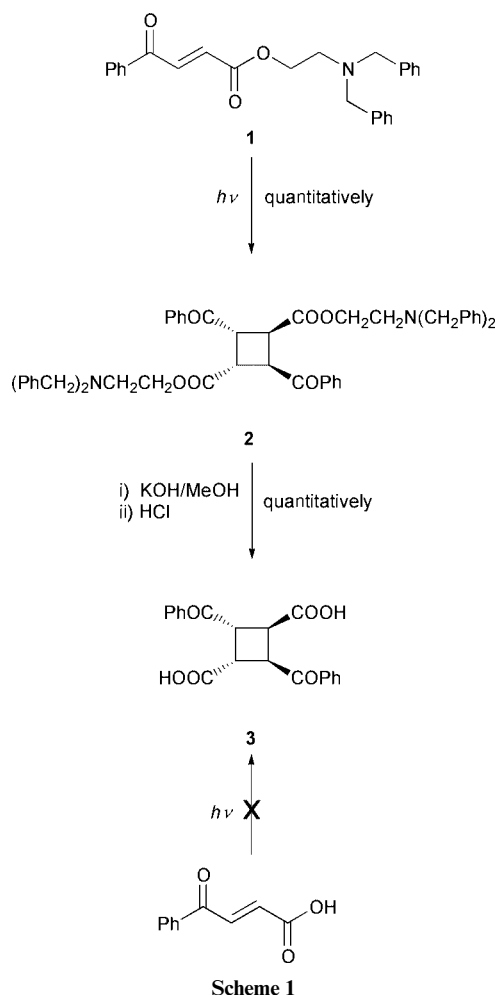
Upon UV irradiation 2-(dibenzylamino)ethyl 3-benzoylacrylate in a crystalline state undergoes efficient [2+2]photocycloaddition to give a head-to-tail dimer, bis[2-(dibenzylamino)ethyl] *c*-2,*t*-4-dibenzoylcyclobutane-*r*-1,*t*-3-dicarboxylate, quantitatively. The X-ray structure analysis shows CH/π interactions between aliphatic and aromatic C–H bonds and benzene rings make the orientation of the benzoylacrylate molecules in a crystalline state suitable for the [2+2]photocycloaddition. The aliphatic CH/π interaction is responsible for formation of the *cis*-configuration between the 2-(dibenzylamino)ethoxycarbonyl and benzoyl groups in the cyclobutanedicarboxylate. The cyclobutanedicarboxylate is hydrolyzed quantitatively to 2,4-dibenzoylcyclobutane-1,3-dicarboxylic acid, which is not directly produced from irradiation of crystals of 3-benzoylacrylic acid.

One of the useful and extensively studied photochemical reactions is the photocycloaddition of one unsaturated system to another. The [2+2]photocycloaddition is particularly important in organic synthesis since cyclobutane rings, not easily prepared by classical ground-state methods,[1] are quite commonly formed.[2] The photocycloaddition has been applied to synthesis of natural products such as caryophyllene and isocaryophyllene.[3] The photoreaction has been often performed in a crystalline state[4,5] because of restriction of molecular motion causing competitive reactions such as *cis–trans* isomerization although most organic crystals are thermally and photochemically stable. Recently, a great deal of attention has been focused on solventless reactions because of social demands for clean or 'green' chemistry.[6] Reactions that occur under the influence and control of crystalline media are known as topochemical reactions.[4] The topochemical postulate suggests that the photocycloaddition may occur with a minimum of atomic and molecular motion. Schmidt *et al.* suggested that the [2+2]photocycloaddition may occur when double bonds of neighbouring molecules in a crystalline state are parallel to each other and the center-to-center distance between the bonds is shorter than or equal to 4.1 Å.[4b] The crystalline environment is capable of producing high chemo-, regio-, and stereoselectivity. However, the synthetic potential of solid-state photoreactions does not only depend on highly selective transformations but also requires high chemical efficiency. We report here on the efficient [2+2]photocycloaddition of 2-(dibenzylamino)ethyl 3-benzoylacrylate **1**. Weak CH/π interactions were found to be important factors for determination of crystal packing and molecular arrangements suitable for the photocycloaddition.

Results and discussion

Pale yellow crystals of 2-(dibenzylamino)ethyl (*E*)-3-benzoylacrylate[7] **1** were obtained by recrystallization from a mixture of ethyl acetate and hexane. The crystals were placed between Pyrex glass plates and irradiated with a 400 W high-pressure mercury lamp. After *ca.* 1 h of irradiation the colour of the crystals (1.6 mmol) had disappeared, the starting material was consumed completely, and the [2+2]cycloadduct **2** was formed quantitatively (Scheme 1). The structure of **2** was elucidated
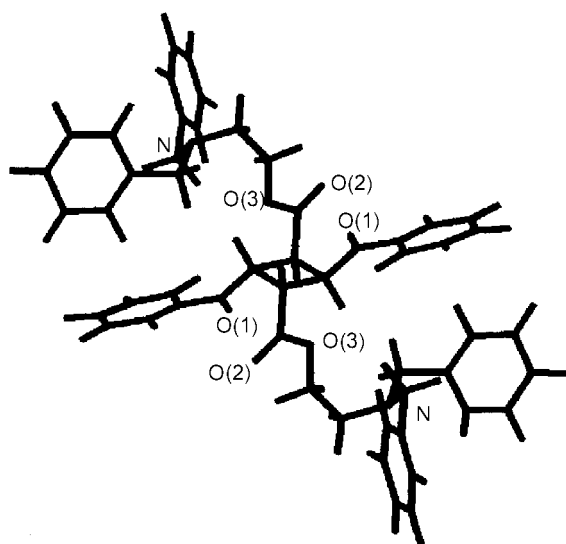

Scheme 1

Fig. 1 The structure of the cycloadduct **2**.

Fig. 2 The structure of **1**. The distance between the ester carbonyl carbon and amine nitrogen is 3.521 Å.

Fig. 3 The crystal structure of **1**. The center-to-center distance of the nearest double bond is 3.673 Å.

on the basis of spectral data and by elemental analysis. The ¹H-NMR spectrum showed two doublet of doublet signals at δ 4.06 and 4.66 attributable to methine protons on the cyclobutane ring. The IR spectrum showed strong absorptions at 1670 and 1730 cm⁻¹ attributable to the benzoyl and ester carbonyl groups, respectively. The absolute configuration of **2** was determined to be bis[2-(dibenzylamino)ethyl] *c*-2,*t*-4-dibenzoylcyclobutane-*r*-1,*t*-3-dicarboxylate on the basis of X-ray structure analysis (Fig. 1). The cycloadduct **2** is a head-to-tail dimer. So, the photoreaction proceeded highly chemo-, regio- and stereoselectively to give only one head-to-tail adduct quantitatively. This can be explained in terms of the topochemical preference. It is noteworthy that the 1-alkoxycarbonyl group is *trans* to the 2-benzoyl group and *cis* to the 4-benzoyl group though the most stable stereoisomer should have all-*trans* substituents. This suggests the presence of a sort of attractive interaction between the 2-(dibenzylamino)ethoxycarbonyl group in a molecule and the benzoyl group in a partner molecule required for the [2+2]photocycloaddition in crystals.

Other benzoylacrylates, 2-(dimethylamino)ethyl 3-benzoylacrylate, 2-(dibenzylamino)ethyl 3-benzoylacrylates having a *p*-chloro, *p*-methyl, *p*-phenyl or *p*-methoxy substituent on the benzoyl group, and 3-(dibenzylamino)propyl 3-benzoylacrylate were prepared in order to clarify the interaction. However, all these compounds were viscous liquids and could not be crystallized at room temperature. No [2+2]cycloadducts were obtained from the irradiation of these liquid compounds. As crystals of the 3-benzoylacrylate **1** were easily obtained and underwent the [2+2]photocycloaddition quite efficiently, there must be a strong driving force for crystal packing which is peculiar to **1** and the molecular arrangement in the crystalline state must meet the topochemical requirements. Thus, the X-ray crystallographic structure analysis of **1** was performed.

The X-ray analysis showed that a molecule of **1** has a bent 'C-shaped' configuration and the amine nitrogen stands at a distance of 3.521 Å from the ester carbonyl carbon (Fig. 2). This value is nearly equal to the sum of the van der Waals radii of nitrogen and carbon (*ca.* 3.4 Å).[8] Electrostatic attractive interaction between the nitrogen and the ester carbonyl carbon in **1** must allow their approach. Fig. 3 shows the crystal structure of **1**. The crystal structure is characterized by the presence of 'strings' consisting of a range of unit pairs of **1**. Double bonds of the benzoylacrylate molecules are forced to be the outer side of the strings and the center-to-center distance between the nearest double bonds in different strings is 3.673 Å (Figs. 3 and 4-a). This value is smaller than the limiting value of 4.1 Å in the topochemical postulate for the solid-state [2+2]photocyclo-
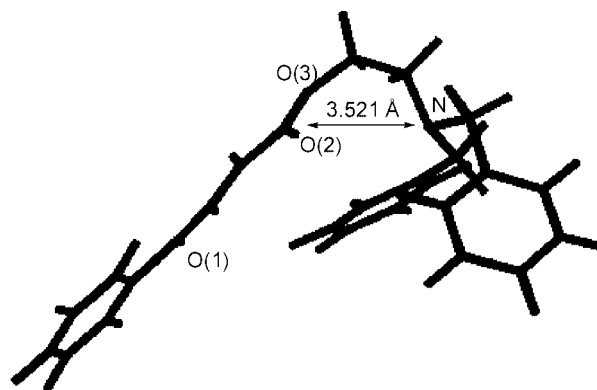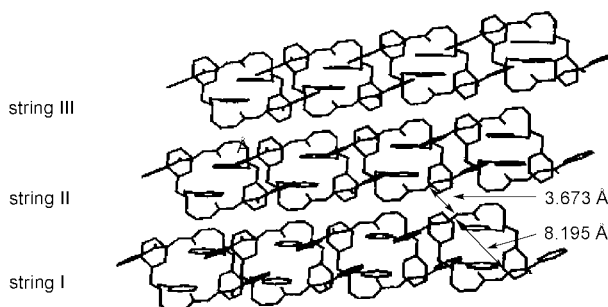
addition.[4b] This must be the reason for the efficient [2+2]photocycloaddition in **1**. The aliphatic CH/π interactions[9] between one of the *O*-methylene hydrogens of a benzoylacrylate molecule **A′** in a string I in Fig. 4-a and a benzoyl benzene ring of a molecule **B** in a different string II is probably responsible for the propinquity of the two strings because the distance between the hydrogen and C-3 of the benzene ring is 2.859 Å. Recently the aliphatic CH/π interactions[9,10] have been observed in large numbers of organic crystals,[11] known to play a dominant role in supramolecular chemistry,[9,12] and gained much attention in the consideration of a variety of molecular phenomena, such as molecular recognition,[13] chiral discrimination,[14] coordination chemistry,[15] chiroptical properties,[16] and crystal packing.[11] The aliphatic CH/π interaction in the crystals of **1** must cause configuration of the benzoyl group *cis* to the (dibenzylamino)ethoxycarbonyl group in the head-to-tail cycloadduct **2**. The CH/π interaction should be quite sensitive to substitution and length of the alkyl chain. Absence of the [2+2]photoreactivity in 3-benzoylacrylates other than **1** supports this idea.

Additional interactions probably contribute to the proximity of strings resulting in the propinquity of double bonds to react with each other. The plane of a benzyl benzene ring of a benzoylacrylate molecule **A′** in Fig. 4-b in a string meets the plane of one of the benzyl benzene rings of a different molecule **B** in a neighbouring string at an angle of 70° (α in Fig. 4-b). The distance between hydrogen on the benzyl benzene ring in **A′** and the benzene ring in **B** is 3.08 Å (d in Fig. 4-b). The attractive interactions between benzene rings have been studied both experimentally and theoretically. The theoretical studies showed that the interaction between benzene rings is attractive when the benzene rings are stacked vertically (α = 90°), and repulsive when they are parallel to each other (α = 0°).[17] The attractive interaction results from the CH/π interaction between aromatic C–H and the π-electron cloud. Although a rotation of 90° to 0° leads to repulsion caused by the π–π electronic interaction, attraction should dominate over repulsion in a geometry with α = 70°.[17] The molecules of **1** in neighbouring strings are, therefore, bound by the aromatic CH/π interactions. The
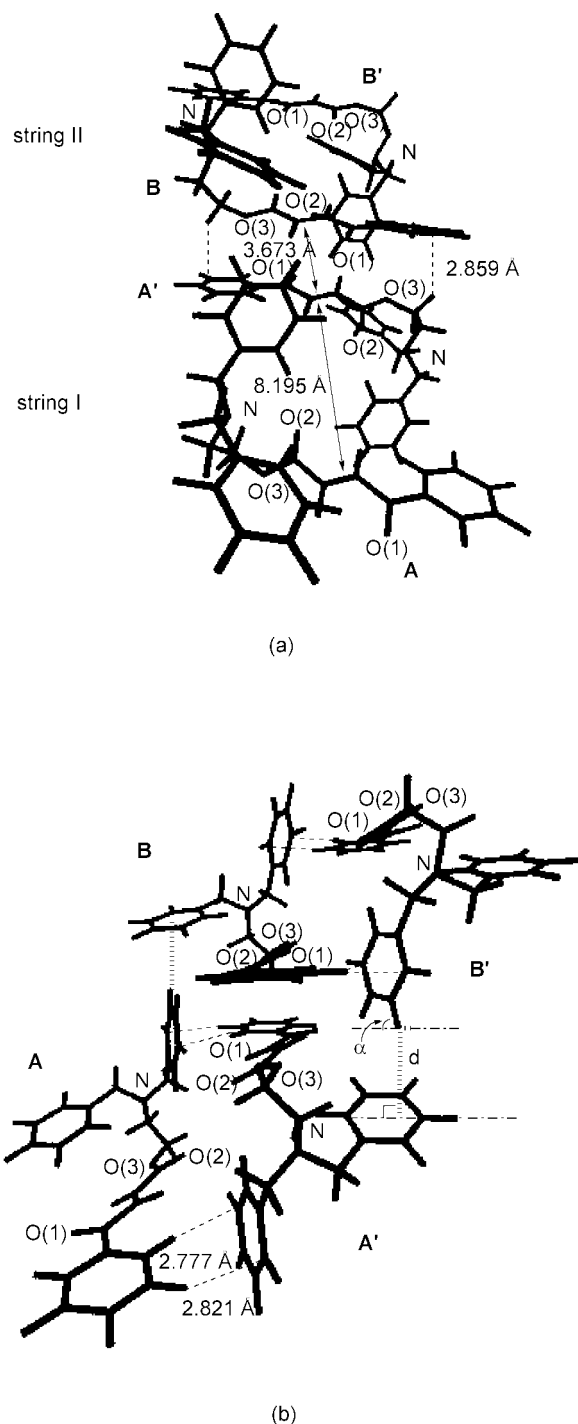
(a)

(b)

Fig. 4 Perspective views of **1**. **A–A′** and **B–B′** are unit pairs of **1** in the different strings I and II, respectively. The center-to-center distance between the nearest double bonds in different strings is 3.673 Å and that between double bonds in a unit pair is 8.195 Å. The dotted lines in (a) show the presence of the aliphatic CH/π interaction and those in (b) indicate the aromatic CH/π interaction between the benzyl benzene ring and hydrogen on the different benzyl ring. The thick dotted lines in (b) show the aromatic CH/π interaction between the benzyl benzene ring and hydrogens on the benzoyl benzene ring.

aromatic CH/π interactions, diverse phenomena as that in π–π repulsive interactions, are known to contribute to the crystal packing of aromatic molecules[18] and appearance of a variety of supramolecular properties.[5b,19]

The aromatic CH/π interaction must also contribute to formation of the unit pairs of **1** in a string. Each of the benzoyl benzene rings in a pair of the benzoylacrylate molecules is perpendicular to a benzyl benzene ring of the partner molecule in the pair as shown in Fig. 4-b and the distances between hydrogen on the benzoyl group and the benzyl benzene ring are

ca. 2.8 Å. As the distance between the double bonds of the component molecules in the pair is 8.195 Å (Figs. 3 and 4-a) the [2+2]photocycloaddition cannot occur in the pair.

The cycloadduct **2** was easily hydrolyzed to form 2,4-dibenzoylcyclobutane-1,3-dicarboxylic acid **3** quantitatively. The cyclobutanedicarboxylic acid may be expected to be produced directly from irradiation of 3-benzoylacrylic acid **4**. However, no reaction was observed when crystals of **4** were irradiated for 24 h under the same conditions (Scheme 1). Therefore, the [2+2]photocycloaddition of **1** is a useful and convenient method for the synthesis of 2,4-dibenzoylcyclobutane-1,3-dicarboxylic acid.

In conclusion the 3-benzoylacrylate **1** in a crystalline state underwent the [2+2]photocycloaddition efficiently. The electrostatic interaction between the ester carbonyl carbon and amine nitrogen and the weak aliphatic and aromatic CH/π interactions in **1** are crucial driving forces of the crystal packing and of the suitable orientation of molecules for the efficient [2+2]photocycloaddition. The aliphatic CH/π interaction is responsible for formation of the *cis*-configuration between the 2-(dibenzylamino)ethoxycarbonyl and benzoyl groups in the [2+2]cycloadduct. The weak intermolecular interactions should be important factors for controlling solid-state organic photoreactions.

Experimental

The IR spectra were recorded with a JASCO IR Report-100 spectrometer. The ^{1}H- and ^{13}C-NMR spectra were measured with a Bruker AM400 or JEOL JNM-LA-500 spectrometer using tetramethylsilane as internal standard. A Toshiba 400 W high-pressure mercury lamp was used as the irradiation source. The 3-benzoylacrylate **1** was prepared according to the reported method.[7]

Photolysis of 2-(dibenzylamino)ethyl (*E*)-3-benzoylacrylate 1 in a crystalline state

The crystals of **1** (0.635 g, 1.6 mmol) were placed between Pyrex glass plates and irradiated for 75 min with a 400 W high-pressure mercury lamp. After the irradiation the colour of the crystals had changed from pale yellow to white and bis[2-(dibenzylamino)ethyl] *c*-2,*t*-4-dibenzoylcyclobutane-*r*-1,*t*-3-dicarboxylate **2** was obtained quantitatively: mp 143.0 °C (from a mixture of dichloromethane and hexane); ν_{max}(KBr)/cm^{-1} 1730 and 1670; δ_{H}(CDCl$_3$) 2.42 (4H, t, *J* 6.3, 2 × CH$_2$N), 3.45 (4H, d, *J* 13.8, 2 × C*H*HPh), 3.50 (4H, d, *J* 13.8, 2 × CH*H*Ph), 3.77 (2H, dt, *J* 11.6 and 6.3, 2 × OC*H*H), 4.02 (2H, dt, *J* 11.6 and 6.3, 2 × OCH*H*), 4.06 (2H, dd, *J* 10.2 and 6.7, 2 × C*H*COPh), 4.66 (2H, dd, *J* 10.2 and 6.7, 2 × CHCOO), 7.2–7.5 (26H, m, aromatic) and 7.8–7.9 (4H, m, aromatic); δ_{C}(CDCl$_3$) 40.9 (2d), 43.3 (2d), 51.2 (2t), 58.5 (4t), 68.0 (2t), 126.9 (4d), 128.2 (8d), 128.5 (4d), 128.6 (10d), 133.5 (4d), 135.4 (2s), 139.2 (4s), 170.8 (2s) and 196.6 (2s) (Found: C, 78.25; H, 6.39; N, 3.47. C$_{52}$H$_{50}$N$_2$O$_6$ requires C, 78.17; H, 6.31; N, 3.51%).

X-Ray structure determination of 1 and 2

A Rigaku AFC6S diffractometer with graphite-monochromated Mo-K$_\alpha$ radiation (0.710 69 Å) was used. The unit-cell parameters were determined from 22 reflections with $26.04° \leqq 2\theta \leqq 35.32°$ for **1** and from 24 reflections with $6.58° \leqq 2\theta \leqq 13.43°$ for **2**. Intensity data with $2\theta \leqq 55.1°$ were collected with the ω–2θ scan technique (scan speed 8.0° min^{-1} for **1** and 4.0° min^{-1} for **2**) at 4566 reflections for **1** and 4870 for **2**. The intensities of three representative reflections which were measured after every 150 reflections remained constant throughout data collection indicating crystal and electronic stability. No decay correction was applied. The intensities were corrected for Lorentz and polarization factors, but not for absorption. The structure was solved by direct methods[20] and expanded using

Fourier techniques.[21] The full-matrix least-squares refinement for non-hydrogen atoms was carried out for $\Sigma w(|F_o| - |F_c|)^2$, where the weight $w = 4F_o^2/\sigma^2(F_o^2)$, for 4261 independent reflection with $I > -10.00\sigma(I)$ for **1** and 4729 for **2**. The final discrepancy factors were $R = 0.080$ and $R_w = 0.033$ for **1** and $R = 0.139$ and $R_w = 0.089$ for **2**. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.51 and -0.54 e Å^{-3}, respectively, for **1** and 2.61 and -3.05 for **2**. Neutral atom-scattering factors were taken from Cromer and Waber.[22] Anomalous dispersion effects were included in F_c;[23] the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley.[24] The values for the mass attenuation coefficients are those of Creagh and Hubbell.[25] All calculations were performed using the CrystalStructure[26,27] crystallographic software package.

Crystal data of 1.† $C_{26}H_{25}NO_3$, $M = 399.49$, triclinic, $a = 9.556(2)$, $b = 14.124(8)$, $c = 8.881(1)$ Å, $\alpha = 90.30(4)$, $\beta = 108.2273$, $\gamma = 105.3744°$, $V = 1092.6(6)$ Å^3, $T = 293 \pm 1$ K, space group $P\text{-}1$, $Z = 2$, μ(Mo-Kα) = 0.8 cm^{-1}, 4569 reflections measured, 4566 unique ($R_{int} = 0.005$).

Crystal data of 2. $C_{52}H_{50}N_2O_6$, $M = 798.98$, monoclinic, $a = 18.467(7)$, $b = 5.519(9)$, $c = 20.166(7)$ Å, $\beta = 90.2445°$, $V = 2055.3(2)$ Å^3, $T = 296 \pm 1$ K, space group $P2_1/c$, $Z = 2$, μ(Mo-Kα) = 0.8 cm^{-1}, 5388 reflections measured, 4870 unique ($R_{int} = 0.382$).

2,4-Dibenzoylcyclobutane-1,3-dicarboxylic acid 3

The [2+2]cycloadduct **1** (0.497 g, 0.6 mmol) was dissolved in 10 ml of methanol containing 10% potassium hydroxide and heated at 50 °C for 4 h. The mixture was acidified with hydrochloric acid. The solid formed was collected, and washed with water. After drying, diacid **3** was obtained quantitatively: mp 178.0–179.0 °C (from 95% EtOH); ν_{max}(KBr)/cm^{-1} 2950br, 1720 and 1680; δ_H(DMSO) 2.49 (2H, t, J 8.9, 2 × C*H*COPh), 3.51 (2H, t, J 8.9, 2 × C*H*COOH), 6.44–6.47 (4H, m, aromatic), 6.56–6.59 (2H, m, aromatic) and 6.85–6.89 (4H, m, aromatic); δ_C(DMSO) 42.1 (2d), 52.1 (2d), 128.5 (4d), 128.8 (4d), 134.0 (2d), 134.6 (2s), 171.7 (2s) and 196.4 (2s) (Found: C, 67.86; H, 4.88. $C_{20}H_{16}O_6$ requires C, 68.18; H, 4.58%).

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† CCDC reference numbers 167182 and 167183. See http://www.rsc.org/suppdata/p1/b1/b105970j/ for crystallographic files in .cif or other electronic format.

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