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Photochemical *cis–trans* isomerization of 1,2-dibenzoyl-3-substituted cyclopropanes

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Abstract

Irradiation of *cis*, *trans*-l,2-dibenzoyl-3-methylcyclopropane (*cis*-1a) produced *trans*, *trans*-1,2-dibenzoyl-3-methylcyclopropane (*trans*-1a). This *cis–trans* isomerization was shown to be reversible through cleavage of the bond beta to both carbonyl (1,2-bond fission) while irradiation of *cis*, *trans*-l,2-dibenzoyl-3-phenylcyclopropane (*cis*-1b) gave *trans*, *trans*-1,2-dibenzoyl-3-phenylcyclopropane (*trans*-1b) irreversibly through cleavage of the bond beta to both carbonyl groups (1,2-bond fission) and the bond beta to phenyl group (1,3-bond fission). 1,2-bond fission of optically active *trans*-1b showed racemization only under irradiation. This racemization of *trans*-1b was considered to be responsible for irreversible *cis–trans* isomerization of *cis*, *trans*-1,2-dibenzoyl-3-phenylcyclopropane (*cis*-1b).

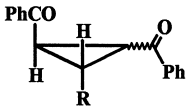
Keywords: *Cis–trans* isomerization; Racemization; π-π interaction

1. Introduction

In the course of over last decade the photochemistry of disubstituted cyclopropane was expanded with study of ring opening reaction mechanism [1] including 1,3-diradical stabilization [2], kinetics for isomerization [3–6] and radical intermediate trapping by enzyme catalyzed reactions [7–9].

These investigations were performed with mostly 1,2-disubstituted cyclopropanes and a few papers [9–11] discussed the photochemistry of 1,2,3-trisubstituted cyclopropanes. Greenberg [12] and Trost [13] synthesized 1,2-dibenzoyl-3-phenylcyclopropane (1b), which would show more variable ways of bond fission than disubstituted cyclopropane.

Unlike disubstituted cyclopropane, *cis*, *trans*-l,2-dibenzoyl-3-phenylcyclopropane (1b) revealed irreversible *cis–trans* isomerization in our preliminary experiment [14]. This result led us to investigate a detailed mechanism for the ring opening reaction of 1,2-dibenzoyl-3-phenylcyclopropane (1b) and 1,2-dibenzoyl-3-methylcyclopropane(1a).



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a, R=Me
b, R=Ph

2. Experimental details

2.1. Materials and general methods

All solvents were freshly distilled and dried before use according to standard procedures. Benzene was shaken with cold concentrated sulphuric acid until the acid layer was colorless and then distilled from sodium. All other reagents were used as received unless otherwise specified.

Absorption spectra were measured on a Shimadzu UV-2600 spectrometer. Gas chromatography-mass spectrometry (GC-MS) measurements were made on a Hewlett–Packard 5980 gas chromatograph with a Hewlett–Packard 5988 mass spectrometer (EI 70 eV) using an HP-1 (11 m), SPB-5 (30 m) or Ultra-2 (50 m) capillary column. Elemental analyses were performed using a Carlo Erba 1108 instrument. ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AMX-500 or

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Varian VXR 200S spectrometer. IR spectra were recorded on a Jasco IR-810 instrument. HPLC analyses were carried out on a Spectra-Physics P100 using a Versapack Si 10U analytical column. Optical rotations were recorded on a A-Kruss optronic P-3001 polarimeter.

Irradiations were carried out in Pyrex cells with a Rayonet photoreactor equipped with 16 RPR 300 nm broad band lamps.

2.2. Synthesis

2.2.1. 1,2-dibenzoyl-3-methylcyclopropane (1a) [16]

The mixture of 1,3-dibenzoyl-2-methylpropane (3a) (6.66 g, 0.025 mol) and a solution of sodium hydroxide (2 g) in methanol (72 ml) was warmed to 45°C with stirring. A solution of iodine(6.35 g, 0.025 mol) in methanol (36 ml) was slowly added to the stirred solution from dropping funnel. After the addition had been completed, stirring was continued for 1.5 h at room temperature.

During this period, a white solid was precipitated. The solid was filtered and washed with water and dried at reduced pressure to obtain 5.54 g (84%) of *trans*-1a and trace of *cis*-1a. Spectral data for *trans*, *trans*-1,2-dibenzoyl-3-methylcyclopropane (*trans*-1a). ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.3 (d, 3H), 2.4 (m, 1H), 3.5 (m, 2H), 7.5–8.1 (m, 10H). ^{13}C NMR (80 MHz, $CDCl_3$): δ 11.161, 29.066, 32.380, 35.580, 128.244, 128.568, 133.122,137.242, 137.799, 195.693, 197.708. IR (neat): 3060, 2950, 1650 cm^{-1}. HRMS (EI^+) Calc. for $C_{18}H_{16}O_2$: 264.1151. Found 264.1151. Anal. Calc. for $C_{18}H_{16}O_2$: C, 81.78;H, 6.11. Found: C, 81.77. H, 5.91.

1,2-dibenzoyl-3-methylcyclopropane (1a) was also synthesized by the procedures of Greenberg [12] and Trost [13]. A solution of dimethylsulfonium phenacylide (0.85 g, 4.71 mmol) and 1-benzoylpropene (0.69 g, 4.71 mmol) in 300 ml of benzene was stirred for 15 h at 25°C under nitrogen. The benzene was removed under reduced pressure to afford a yellow oil, which was purified by column chromatography to obtain *trans*-1a (0.86 g, 69.4%) and *cis*-1a (0.043 g, 3.5%). Spectral data for *cis*, *trans*-1,2-dibenzoyl-3-methylcyclopropane (*cis*-1a): ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.4 (d, 3H), 2.5 (m, 1H), 2.9 (d, 2H), 7.4–8.0 (m, 10H). ^{13}C NMR (200 MHz, $CDCl_3$): δ 17.5390, 22.3889, 36.2526, 128.1650, 128.4250, 132.8570, 137.3840. IR (neat): 3060, 2950, 1680 cm^{-1}. HRMS (EI^+) Calc. For $C_{18}H_{16}O_2$: 264.1151. Found 264.1155. Anal. Calc. for $C_{18}H_{16}O_2$: C, 81.78. H, 6.11. Found: C, 81.81. H, 5.98.

2.2.2. 1,3-dibenzoyl-2-methylpropane (3a) [15]

3-methylglutaric acid (7.3 g, 0.05 mol) was placed in 500 ml round-bottomed flask equipped with a condenser, and 17.8 g (0.15 mol) of thionyl chloride was added at once. The mixture was heated gently on a water bath held at 50–60°C After about 4 h, solution was complete and evolution of hydrogen chloride was ceased. Excess thionyl chloride was removed under reduced pressure to give light yellow residue, 3-methylglutaryl chloride. A mixture of anhydrous aluminum chloride (15 g, 0.11 mol) and benzene (75 ml, 0.85 mol) was cooled in ice bath with rapid stirring, 3-methylglutaryl chloride was added dropwise at even rate. After the glutarylchloride had been added, the ice bath was removed and stirring was continued for 2 h at room temperature. The solution was poured into a mixture of 50 g cracked ice and concentrated hydrochloric acid. The benzene layer was separated and washed with an equal volume of dilute sodium carbonate solution, and then with water. The benzene solution was dried over magnesium sulphate and removed by distillation. The residual oil was crystallized from ethanol to obtain 5.4 g (40%) of 1,3-dibenzoyl-2-methylpropane (3a): ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.1 (d, 3H), 2.8–3.2 (m, 5H), 7.5–8.0 (m, 10H). IR (neat): 2950, 1680, 1460, 1365 cm^{-1}. GC/MS: *m*/*e* 266, 161, 147, 105, 77, 51.

2.2.3. 1,2-dibenzoyl-3-phenylcyclopropane (1b) [16]

1,3-dibenzoyl-2-phenylpropane (3b) (5.9 g, 18.0 mmol) was placed in 250 ml round-bottomed flask and a solution of sodium hydroxide (1.44 g) in methanol (72 ml) was added with stirring at 45°C After dissolution of the diketone 3b, a solution of iodine (4.57 g, 18.0 mmol) in 26 ml of methanol was slowly added to the stirred solution from dropping funnel. After the addition had been completed, stirring was continued for 2 h at room temperature. During this period, a white solid was precipitated. The solid was filtered and purified by column chromatography to obtain 2.57 g (43%) of *trans*, *trans*-1,2-dibenzoyl-3-phenylcyclopropane (*trans*-1b) and 0.43 g (7.2%) of *cis*, *trans*-1,2-dibenzoyl-3-phenylcyclopropane (*cis*-1b).

Trans, *trans*-1,2-dibenzoyl-3-phenylcyclopropane (*trans*-1b): ^{1}H NMR: (300 MHz, $CDCl_3$): δ 3.5–3.6 (dd, 1H), 3.7–3.8 (dd, IH), 4.2–4.3 (dd, 1H), 7.2–8.2 (m, 15H). UV (benzene): λ_{max} 277 nm (*e* 4300). *Cis*, trans-1,2-dibenzoyl-3-phenyl cyclopropane: ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.4 (d, 2H), 3.5 (t, 1H), 7.3–8.0 (m, 15H). UV (benzene) λ_{max} 277 nm (*e* 4200). These spectral data for *cis*-and *trans*-1b are the same as those of Trost. [13].

2.2.4. 1,3-dibenzoyl-2-phenylpropane (3b)

The procedures of Fusion et al. [15] was employed for the synthesis of 1,3-dibenzoyl-2-phenylpropane (3b) without modification. The compound 3b was synthesized by an acylation of benzene with corresponding acyl halide, 3b: ^{1}H NMR (200 MHz, $CDCl_3$) δ 3.2–3.5 (m, 4H), 4.1 (quin, 1H), 7.1–8.0 (m, 15H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 37.007, 44.777, 126.554, 127.358, 128.005, 128.463, 132.951, 136.769,143.718, 198.415. IR (neat): 3060, 2900, 1680 cm^{-1}. GC/MS: *m*/*e* 328, 209, 105, 77, 51.

2.2.5. 1,2-dibenzoyl-3-phenylpropane (4)

1,4-diphenylbutane-1,4-dione as a starting material for the compound 4 was synthesized according to the procedures of Drewes et al. [17] Acetophenone trimethylsilyl enol ether was oxidatively coupled at 90°C. Column chromatographic separation yielded the desired 1,4-diketone (1.0 g, 61.6%).

Lithium diisopropylamide (9.0 mmol) in THF was cooled to −78°C The 1,4-diketone (1.0 g, 4.2 mmol) was dissolved in dry THF (17 ml) and added to the LDA solution during 20 min. The mixture was stirred at −78°C for 1 h and then a solution of benzylbromide (0.92 g, 4.2 mmol) in THF was added dropwise. After 1 h at −78°C the mixture was quenched with 3 M hydrochloric acid, and extracted with chloroform. After removal of chloroform, column chromatographic separation afforded 0.5 g (36%) of 1,2-dibenzoyl-3-phenylpropane (4): ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.7 (dd, 1H), 3.1 (dd, 1H), 3.2 (dd, 1H), 3.7 (dd, 1H), 4.4 (m, 1H), 7.2–8.0 (m, 15H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 38.625, 40.663, 43.785, 127.067, 128.499, 128.727, 128.954, 129.047, 129.083, 129.465, 133.411, 133.625, 136.950. 137.028, 139.079, 198.862, 203.104. IR (neat): 3060, 2950, 1690 cm^{-1}. HRMS (EI$^+$) Calc. for $C_{23}H_{20}O_2$: 328.1464. Found: 328.1463.

2.3. Photochemistry

2.3.1. Irradiation of l,2-dibenzoyl-3-methylcyclopropane (1a)

A solution of 350 mg of *trans*-1a in 200 ml of benzene was transferred into 10 Pyrex cells and degassed with purified nitrogen. The samples were irradiated with 16 RPR 300 nm lamps for 4 h. The solvent was then evaporated in vacuo to give a pale yellow liquid. Silica gel chromatography gave 65 mg (21%) of *cis*-1a, 63 mg (20%) of 1,2-dibenzoylvinylethane (2) and 34 mg of recovered *trans*-1a. Spectral data for 1,2-dibenzoylvinylethane (2): ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.2 (dd, 1H), 3.9 (dd, 1H), 4.8 (m, 1H), 5.3 (dd, 2H), 5.9 (m, 1H), 7.4–8.0 (m, 10H). ^{13}C NMR (200 MHz, $CDCl_3$): δ 41.103, 46.528, 118.631, 128.012, 128.526, 128.548, 128.709, 132.983, 133.204, 135.523, 136.453, 136.493, 197.862, 199.589. IR (neat): 3060, 2980, 2900, 1685 cm^{-1}. HRMS (EI$^+$) Calc. for $C_{18}H_{16}O_2$: 264.1151. Found: 264.1145.

Irradiation of *cis*-1a gave *trans*-1a and 2 as a secondary product.

Trans-1a was irradiated under presence of thiophenol. A solution of 52.5 mg (0.19 mmol) of *trans*-1a and 1-5 ml of thiophenol (8–42 mmol) in 15 ml of benzene was degassed with purified nitrogen. The sample was irradiated with 16 RPR 300 nm lamps for 3 h. Silica gel chromatography gave 20 mg (48%) of 1,3-dibenzoyl-2-methylpropane (3a) and 11.6 mg of starting material (*trans*-1a) was recovered. Under presence of thiophenol, *cis–trans* isomerization was completely quenched. The spectral data for 3a are identical with those of authentic compound.

2.3.2. Irradiation of l,2-dibenzoyl-3-phenylcyclopropane (1b)

A solution of 49.0 mg (0.15 mmol) of *cis*-1b in 30 ml of benzene was degassed with nitrogen for 30 mm. The sample was irradiated with 16 RPR 300 nm lamps for 2 h and then the solvent was removed in vacuo. Silica gel chromatography gave 45 mg (92%) of *trans*-1b. GC-analysis of the irradiation mixture showed no other product except *trans*-1b. Irradiation of *trans*-1b (50 mg, 0.15 mmol) under the same condition as *cis*-1b did not produce any product and the starting *trans*-1b was almost completely recovered.

A solution of *trans*-1b (50.6 mg, 0.16 mmol) in 15 ml of benzene and 1–5 ml of thiophenol was irradiated with 16 RPR 300 nm lamps for 20 h. The solvent was then evaporated to give a yellow liquid. Silica gel chromatography afforded 1,2-dibenzoyl-3-phenylpropane (4) (14.0 mg, 28%) and 1,3-dibenzoyl-2-phenylpropane (3b) (16.7 mg, 33%).

Irradiation of *cis*-1b (48 mg, 0.147 mmol) with 5 ml of thiophenol under the same condition as *trans*-1b gave *trans*-1b (9.3 mg, 19%), 3b (3.5 mg, 7.2%) and 4 (4.2 mg, 9%). The spectral data for 3b and 4 are the same as those for authentic compounds.

2.3.3. Photo-racemization of trans-1b

A solution of 37.4 mg of (+)-*trans*-1b ($[\alpha]_D = +22.5$) in 8 ml of benzene was placed in modified polarimeter cell and carefully degassed with nitrogen at 0°C for 20 min. The sample was irradiated with 4 RPR 300 nm lamps for 3 min Polarimetric determination showed complete racemization of (+)-*trans*-1b and no other reactions such as isomerization and decomposition were detected by GC-analysis. Irradiation of (–)-*trans*-1b ($[\alpha]_D = -26.0$) also showed racemization only.

2.3.4. Determination of quantum yield

A solution of 17.1 mg of *cis*-1b in 10 ml of ether in Pyrex tube was degassed with purified nitrogen at 0°C for 20 min. Irradiation was carried out with 16 RPR 300 nm lamps. Light output was monitored by potassium ferrioxalate actinometry according to the method of Hatchard et al. [18]. After irradiation with several intervals, the solutions were analyzed by gas chromatography using *o*-xylene as an internal standard.

2.4. Optical resolution of (±)-trans, trans-1,2-dibenzoyl-3-phenylcyclopropane ((±)-trans-1b) [19]

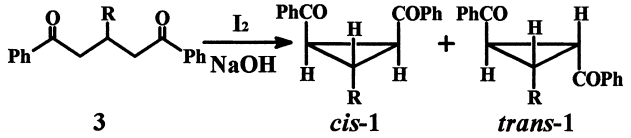
To a solution of 1.53 g (4.7 mmol) of (±)-*trans*-1b and 0.43 g (4.7 mmol) of (2R, 3R)-(–)-2,3-butanediol in 80 ml of benzene was added 10 mg of *p*-toluenesulfonic acid. The mixture was vigorously refluxed for 15 h with a fitted Dean–Stark trap. The solution was then washed with water and dried over magnesium sulphate. After evaporation of benzene, the residue was purified by silica gel chromatography to obtain 0.79 g (42%) of diastereomeric ketal. The ketal

was separated into two portion, the first crop and the second crop, by recrystalization from hexane. Each of the crops was dissolved, respectively, in the mixture of 10 ml of THF and 1.0 ml of hydrochloric acid. Each of the solutions was refluxed for 16 h and extracted with benzene three times. The extracts were washed with water and l0% $NaHCO_3$ solution. After chromatographic purification, 26.9 mg (13%) of (–)-*trans*-1b was obtained from the first crop and 37.4 mg (20.5%) of (+)-*trans*-1b was obtained from the second crop. Specific optical rotations for each enantiomers were +22.48 and −20.04, respectively. Optical purities and the absolute configurations for the enantiomers were not determined.

3. Results and discussion

3.1. Synthesis of 1,2-dibenzoyl-3-methylcyclopropane (1a) and 1,2-dibenzoyl-3-phenylcyclopropane (1b)

l,3-dibenzoyl-2-methylpropane (3a) was prepared by acylation of benzene with corresponding acylhalide from 3-methylglutaric acid. 1,3-dibenzoyl-2-methylpropane (3a) was treated with iodine and sodium hydroxide to obtain 1a. 1,2-dibenzoyl-3-phenylcyclopropane (1b) was synthesized from 1,3-dibenzoyl-2-phenylpropane (3b) treating with iodine and sodium hydroxide (Eq. (1)) [16].



3 *cis*-**1** *trans*-**1**

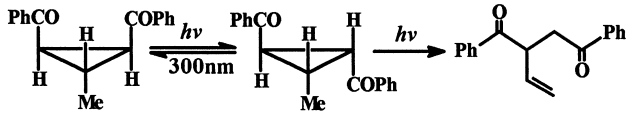
a, R=Me
b, R=Ph

(1)

l,3-dibenzoyl-3-methylcyclopropane (1a) was also synthesized by the addition of dimethylsulfonium phenacylide to 1-benzoylpropane in benzene [12,13].

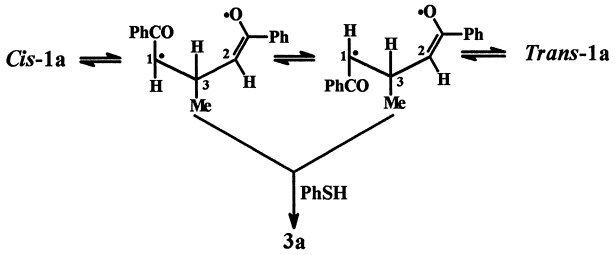
3.2. Photochemistry of l,2-dibenzoyl-3-methylcyclopropane (1a) and 1,2-dibenzoyl-3-phenylcyclopropane (1b)

Irradiation of *cis*-1a in benzene at 300 nm produced *trans*-1a and 1,2-dibenzoyl-l-vinylethane (2) as a secondary product. The *cis–trans* isomerization of 1a was shown to be reversible (Eq. (2)).



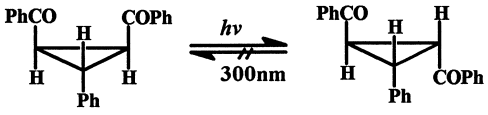
***cis*-1a** ***trans*-1a** **2**

(2)



Scheme 1.

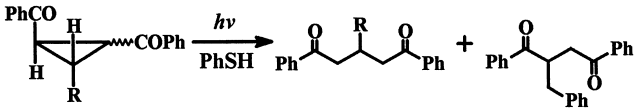
Irradiation of *cis*-1b, however, produced *trans*-1b irreversibly and shown to be a clean conversion to *trans*-1b (91–96%) with a quantum yield of $\phi = 0.27$ at low or high conversion (Eq. (3)).



***cis*-1b** ***trans*-1b** (3)

To explain the difference in reversibility of *cis–trans* isomerization of 1a and 1b, these compounds were irradiated, respectively, under presence of thiophenol, radical trapping reagent [20] which would show the position of bond cleavage.

Irradiation of 1a under the presence of thiophenol produced a reduction product 3a and irradiation of 1b under same condition produced 3b and 4 (Eq. (4)).



1 **3** **4**

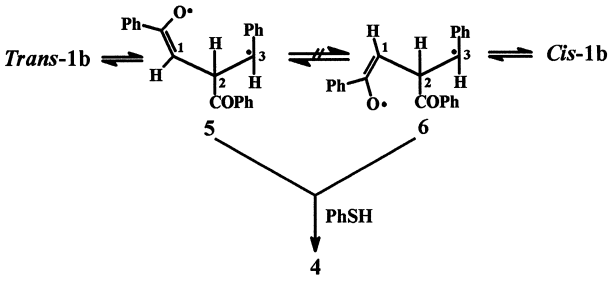
a, R=Me
b, R=Ph

(4)

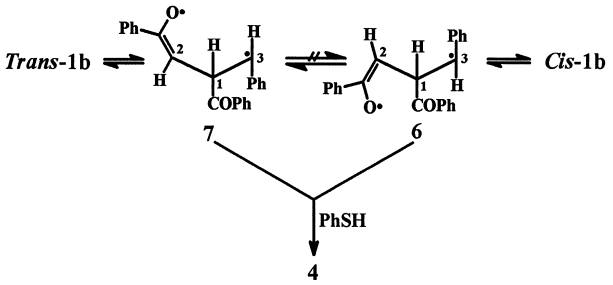
Excited state of 1 react with thiophenol to give a cyclopropylcarbinyl radicals and these radicals are expected to undergo facile ring opening reaction followed by hydrogen abstraction and ketonization to give 3 and 4. The product 3a can be formed by 1,2-bond cleavage of a cyclopropylcarbinyl radical followed by hydrogen abstaction.

After 1,2-bond cleavage of *cis*-1a, 1,3- or 2,3-bond rotation is not prevented by rotational energy barriers (methyl–bonzoyl interaction) and *cis–trans* isomerization of 1a is reversible (Scheme 1).

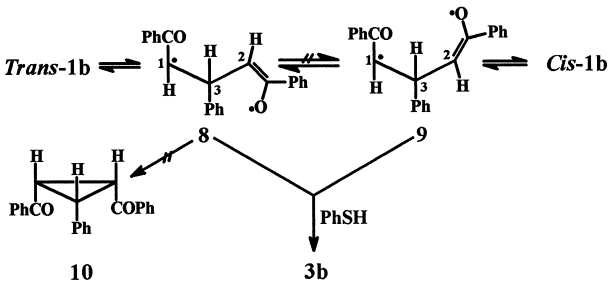
The product 2 from *trans*-1a can be rationalized by intramolecular hydrogen abstraction ([1,5] hydrogen shift of methyl hydrogen) followed by ring cleavage. The product 4 from *trans*-1b can be formed by 1,3- or 2,3-bond cleavage of a cyclopropylcarbinyl radical followed by hydrogen abstraction. Since 1,3- and 2,3-bond in *trans*-1b are not equivalent, 1,3- and 2,3-bond were considered separately.



Scheme 2.



Scheme 3.



Scheme 4.

After 1,3-bond cleavage of *trans*-1b, 1,2-bond rotation of 5 to 6 is unfavorable because of repulsive interaction between two benzoyl groups and this high rotation energy barriers of 1,2-bond is considered to be responsible for irreversible *cis–trans* isomerization of *cis*-1b. 2,3-bond rotation is possible, however, this rotation results the product same as starting compound (Scheme 2).

After 2,3-bond cleavage in *trans*-1b, double rotation, 1,2- and 1,3-bond of 7 to 6 would give *cis*-1b, however, 1,2-bond rotation of 7 is also prevented due to repulsive interaction between two benzoyl groups (Scheme 3). 1,3-bond rotation of 7 is possible, however, this rotation results the product same as starting compound.

Ring opening of 1b occurs not only by 1,3- and 2,3-bond cleavage but also by 1,2-bond cleavage which results formation of 3b under presence of thiophenol (Scheme 4). In Scheme 4, 2,3-bond rotation of 8 to 9 would produce *cis*-1b, however, according to the experimental results, 2,3-bond rotation of 8 to 9 is prohibited.

To explain this irreversible *cis–trans* isomerization of *cis*-1b concerning 2,3-bond rotation of 8, optically active *trans*-1b was irradiated. Irradiation of (–)-*trans*-1b ($[\alpha] = -20.0$ optical purity and absolute configuration were not determined) showed racemization only and no other products were produced during the irradiation. Irradiation of (+)-*trans*-1b ($[\alpha] = +22.5$) also showed racemization only.

Quantitative data for the rotational energy barriers for the interaction between phenyl and benzoyl group in biradical 8 are not available at present, however, Sanders' work [21] for the nature of π–π interaction between two aromatic rings demonstrated that an attractive interaction exists between nonpolarized phenyl ring and polarized ring (electron acceptor) depending upon a proper orientation of the rings. Since Sanders' work can be applicable to *trans*-1b, an attractive interaction is considered to exist between phenyl and benzoyl group.

This attractive π–π interaction in biradical 8 is thought to prevent 2,3-bond rotation to 9 which gives *cis*-1b. *Cis–trans* isomerization of 1a and 1,2-dibenzoylcyclopropane (11) [22] are reversible because this kind of π–π interaction does not exist in 1a and 11. Since 1,3-bond rotational energy barrier cannot overcome the sterric congestion in 10, 1,3-bond rotation is always accompanied by 2,3-bond rotation, which leads to racemization of *trans*-1b.

As a result, photo-isomerization of *cis*-1b to *trans*-1b is almost quantitative (91–96% conversion) and quantum yield for disappearance of *cis*-1b is 0.27 and for appearance of *trans*-1b is also 0.27 even at high conversion.

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