

Taehee Noh,* Youngmee Jeong and Daekyun Kim

Department of Chemistry Education, Seoul National University, Seoul, 151-742, Korea

An unprecedented photodimerization of 1-substituted naphthalenes is reported. Upon irradiation of naphthalene-1-carbonitrile or methyl 1-naphthoate through a Pyrex filter at $-78\text{ }^{\circ}\text{C}$, the corresponding *syn*-[2+2] cyclodimer was isolated as a single product. However, no cyclodimer was found in the irradiation of 1-methylnaphthalene, 1-methoxynaphthalene, and naphthalene under the same irradiation conditions. By a triplet-sensitized photoreaction and low-temperature NMR study of the reaction mixture irradiated at low temperature, the *syn*-[2+2] cyclodimer was found to be produced through a facile Cope rearrangement of the *exo*-[4+4] cyclodimer which was the primary product formed by a singlet-state 1,4-4',1' dimerization of the substituted rings.

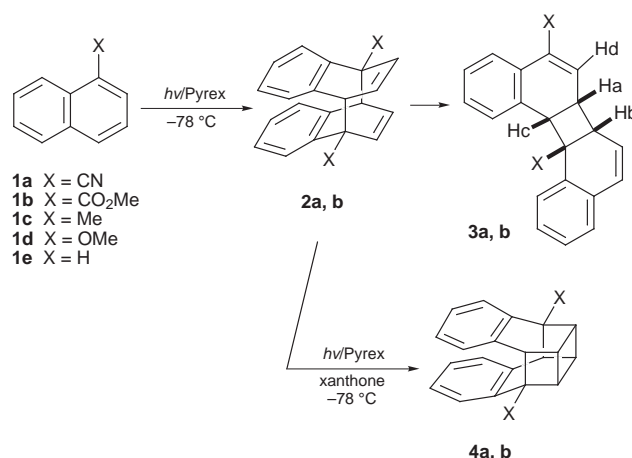
Introduction

Although synthetic and mechanistic studies on the [4+4] photodimerization of anthracenes have been carried out for over a century, it is still an area of substantial interest.^{1,2} In contrast, naphthalenes are much less photoreactive than anthracenes, thus less is known about the photodimerization of naphthalenes. The first photodimer in the naphthalene series was prepared from 2-methoxynaphthalene in 1963.³ Later, several groups reported [4+4]- and cage-cyclodimers formed in the irradiation of certain naphthalenes, in particular, 2-alkoxy-naphthalenes,⁴ naphthalene-2-carbonitrile,^{5,6} and naphthalene-2-carboxylic acid derivatives.⁷ Cross photodimerization of naphthalene-2-carbonitrile and naphthalene has been also observed.⁶ However, neither 1-substituted naphthalene nor naphthalene itself has been known to undergo photodimerization.

There has been substantial interest in the chemistry of energy-rich aromatic cyclodimers.⁸ Accordingly, the photo-reactions of substituted naphthalenes and anthracenes have been reinvestigated recently resulting in intriguing observations which do not agree with the previous reports.^{2,9} In the photocycloaddition reaction of naphthalene-1-carbonitrile (**1a**) to cyclohexa-1,3-diene or furan at ambient temperature, the primary [4+4] adduct was found to undergo a facile rearrangement to give a *syn*-[2+2] adduct which was dissociated to regenerate the starting materials under the irradiation conditions. Although [4+4] photodimerization of 1-substituted naphthalene is expected,¹⁰ no cyclodimer has been found in the irradiation of 1-substituted naphthalenes,^{1,7a} which might be due to the same dissociation mechanism. Since the photochemistry of 1-substituted naphthalenes is of current interest in several laboratories,¹¹ we investigated the photodimerization of 1-substituted naphthalenes at low temperature.

Results and discussion

In order to prevent secondary thermal processes during irradiation, low-temperature irradiation of naphthalene itself and some 1-substituted naphthalenes was conducted. A dichloromethane solution of **1a** (0.065 M) at $-78\text{ }^{\circ}\text{C}$ was irradiated through a Pyrex filter for 9 h. No precipitates were observed during the irradiation. The reaction mixture was separated by chromatography to yield a single product (**3a**) along with unreacted **1a**. The isolated yield of **3a** based on consumed **1a** was 99% in 41% conversion (Scheme 1). Unlike the photodimerization of naphthalene-2-carbonitrile,^{5,6} no cage compound was found. In the photodimerization of methyl 1-naphthoate (**1b**) under the same conditions, the conversion was about 17% and



Scheme 1 Photodimerization of naphthalenes

3b was isolated in 89% yield based on consumed **1b**. However, no cyclodimer was obtained in the low-temperature irradiations of 1-methylnaphthalene (**1c**), 1-methoxynaphthalene (**1d**) and naphthalene (**1e**). No cross dimerization was observed in the irradiation of 1:1 mixture of **1a** and **1e** under similar conditions.

Upon heating, both products (**3a** and **3b**) decomposed into **1a** and **1b**, respectively. The mass spectrum (CI) of **3a** exhibits a molecular ion peak (MH^+) at m/z 307 and a base peak at m/z 154, and that of **3b** shows a similar pattern. These indicated that the products were the cyclodimers of **1a** and **1b**. The structures were elucidated on the basis of their spectroscopic properties. Eight aromatic protons, three olefinic protons, and three cyclobutyl protons in the ^1H NMR spectrum of **3a** indicated that the cyclodimer was fused at the 1,2 position of one naphthalene skeleton and the 3,4 position of the other. The connectivity of the cyclodimer is consistent with the coupling patterns of its COSY spectrum. The doublet peak at δ 4.72, which was assigned to Hc, exhibits strong coupling with Ha (δ 3.75). Coupling of Ha with Hb (δ 4.24) and Hd (δ 6.58) was also observed (Scheme 1). The large coupling constant (9.1 Hz) between Ha and Hb suggested the cyclodimer has the *syn* orientation.^{4c,9} Cyclodimer **3b** was also characterized by the same spectroscopic data analyses.

The UV spectra of **1a** and **3a** are shown in Fig. 1. Cyclodimer **3a** has an absorption maximum at 271 nm and a tail which reaches beyond 330 nm. The irradiation of isolated **3a** and **3b** at $-78\text{ }^{\circ}\text{C}$ or room temperature through a Pyrex filter ($>290\text{ nm}$) resulted in quantitative dissociation to their corresponding

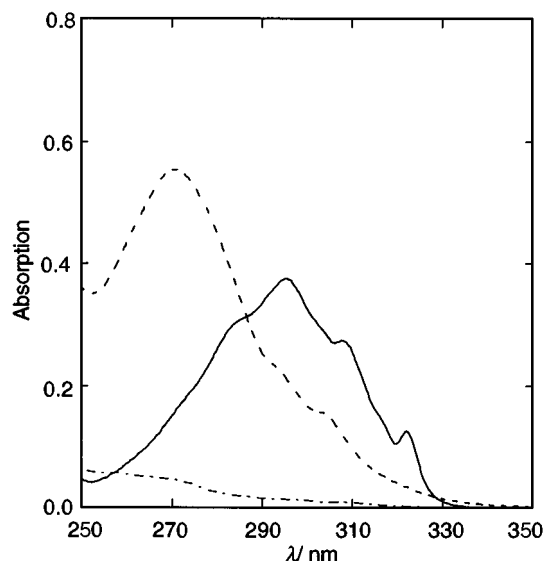


Fig. 1 UV spectra of **1a** (—), **3a** (---) and **4a** (-·-) in acetonitrile (conc.: 5.0×10^{-5} M)

naphthalenes, respectively. Since excellent yields of **3a** and **3b** were obtained in the low-temperature irradiations, **3a** and **3b** may be formed from **2a** and **2b** which are likely to be less labile under the irradiation conditions (chromophore of **2a** and **2b**: *o*-xylene). In order to study this, a sensitization experiment with xanthone^{9,12} was conducted. After **1a** was irradiated at -78°C for 9 h, xanthone was added. The resulting mixture was irradiated through a Pyrex filter for 4 h. Similar separatory methods resolved a new product (**4a**) in 49% yield along with **3a** in 30% yield. Photoexcitation of xanthone with an aqueous filter solution of sodium bromide and lead acetate¹³ (>335 nm) also resulted in the formation of **4a**. In the case of **1b** with a Pyrex filter, **4b** was also isolated in 44% yield on the basis of consumed starting material. The formation of **4a** and **4b** suggested the presence of *exo*-[4+4] cyclodimers, which are eventually transformed to **3a** and **3b** by the thermal Cope rearrangement (Scheme 1). This explains the previous result that no cyclodimer was isolated in the irradiation of **1b** at ambient temperature.^{7a}

In order to obtain conclusive evidence for the existence of **2a**, a low-temperature NMR study was performed. A solution of **1a** in CDCl_3 in an NMR tube was irradiated through a Pyrex filter for 5 h at -60°C and the ^1H NMR spectrum was taken at -50°C . After allowing the reaction mixture to warm to room temperature over 30 min, another ^1H NMR spectrum was taken (Fig. 2). The first low-temperature NMR spectrum exhibits four peaks of δ 7.29, 6.98, 6.76 and 4.22 with a ratio of 1:3:2:1 in addition to those of **1a** at δ 8.31–7.58. The ^1H NMR spectrum of the product is in agreement with the structure of **2a**. Bridgehead protons show a doublet peak at δ 4.22 and olefinic protons at δ 6.76, which are in the expected region when compared to those of the *exo*-[4+4] cyclodimer of **1a** and cyclohexa-1,3-diene.⁹ Aromatic protons (δ 7.29–6.98) are deshielded as one would expect from the anisotropy of the aromatic ring. When the solution was warmed up to room temperature, the NMR peaks of **2a** disappeared while a new set of peaks corresponding to **3a** appeared. Since the half-life for the thermal Cope rearrangement of the *exo*-[4+4] cyclodimer of naphthalene in chloroform was estimated to be about 120 min at 40°C ,¹⁴ the facile rearrangement of **2a** to **3a** should be related to the weakness of the bridgehead linkage carrying the 1-cyano group,¹⁵ which was also observed with the *exo*-[4+4] cyclodimer of **1a** with furan or cyclohexa-1,3-diene.⁹

Photoexcitation of xanthone through the filter solution (>335 nm) in the presence of **1a** or **1b** at -78°C did not yield any product, although energy transfer from xanthone (triplet energy = $74.0 \text{ kcal mol}^{-1}$)¹⁶ to naphthalenes (triplet energy of

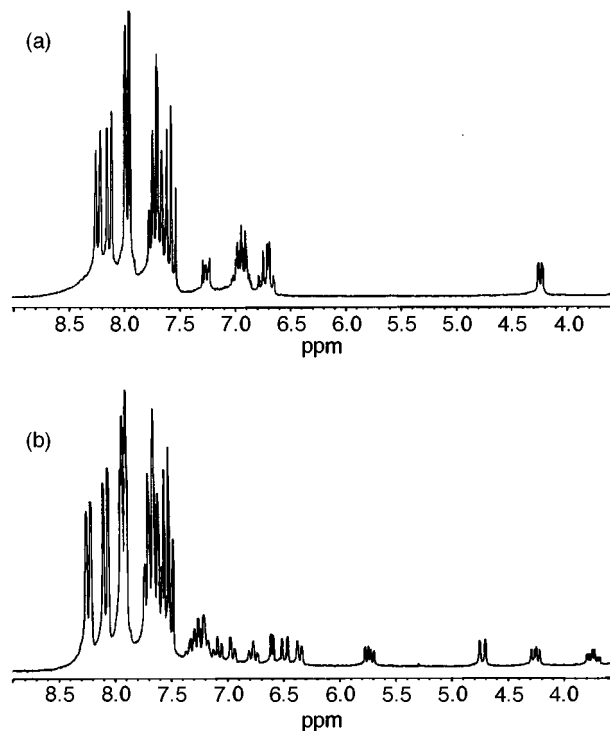


Fig. 2 Low-temperature NMR study for the photodimerization of **1a**. (a) ^1H NMR spectrum at -50°C for the irradiated solution of **1a** at -60°C ; (b) ^1H NMR spectrum at 25°C for the resulting solution after standing at room temperature.

1a = $57.4 \text{ kcal mol}^{-1}$,¹⁷ triplet energy of **1b** = $57.2 \text{ kcal mol}^{-1}$)¹⁸ is expected to be effective. This indicated that the primary photoreaction could be attributed to the excited singlet state. The observed products indicated that bonding took place exclusively between the substituted rings to result in the formation of cyclodimers with the head-to-tail structure (1,4-4',1' dimerization). Although consideration of the electronic and steric effects of the substituents reveals no very obvious reason for the unreactivity of **1c**–**1e**, the regiochemistry in the photodimerization of **1a** and **1b** is consistent with that of most naphthalenes and anthracenes.¹ In the photoreactions of bis(1-naphthylmethyl) ether¹⁹ and naphthalene-2-carbonitrile⁶ to give the corresponding cage compounds, it was argued that *syn*-[2+2] cyclodimers were the precursors for the intramolecular cyclization reactions. However, irradiation of xanthone through the filter solution (>335 nm) in the presence of **3a** resulted in quantitative dissociation to **1a**. This suggested that the intramolecular cyclization to **4a** exclusively occurred from **2a**.

Experimental

Compound **1b** was prepared from **1a** according to the literature method²⁰ and purified by chromatography on silica. Most reagents and solvents were purified by the literature procedures.²¹

Mps were determined on a MEL-TEMP II melting point apparatus and are reported uncorrected. UV spectra were recorded on a Cary 300 Bio UV/VIS spectrophotometer. IR spectra were recorded on a Perkin-Elmer Spectrum 2000 Explorer FT-IR spectrometer. ^1H and ^{13}C NMR spectra were routinely recorded at 300 MHz on an AMX Bruker spectrometer. Chemical shifts (δ) are reported as ppm downfield from TMS, and coupling constants (J) are given in Hz. Low-temperature NMR spectra were obtained at 200 MHz on a Varian VXR 200S spectrometer. Mass spectra (MS) were obtained on a Hewlett-Packard 5890-JMS AX505WA double focusing magnetic sector mass spectrometer.

All the irradiations were performed with a 450 W Hanovia medium-pressure mercury lamp irradiating through a Pyrex

filter or a filter solution. The filter solution was an aqueous solution of sodium bromide (0.54 g cm⁻³) and lead acetate (0.007 g cm⁻³)¹³ that blocked wavelengths below 335 nm. A triple walled quartz immersion well with the outer two walls permanently sealed together and dry ice–solvent baths were used for low-temperature irradiations. For the preparative reaction, the solutions were purged with nitrogen for more than 20 min before and then during the irradiation. The reactions were followed by either TLC or ¹H NMR spectrum analysis.

Low-temperature irradiation of naphthalenes

A solution of **1a** (1.49 g, 9.74 mmol) in dichloromethane (150 cm³) was irradiated at -78 °C for 9 h. The solvent was evaporated off under reduced pressure, and the resulting mixture was separated by flash silica gel chromatography eluting with *n*-hexane–dichloromethane mixtures of increasing polarity to afford **3a** (0.61 g, 99% based on consumed **1a**) and unreacted **1a** (0.88 g, 59%). **3a**: mp 173.0–175.0 °C (from dichloromethane and *n*-hexane); λ_{max}(CH₃CN)/nm 270.9 (ε/dm³ mol⁻¹ cm⁻¹ 11 100); ν_{max}/cm⁻¹ (CHCl₃) 3017, 2942, 2231 (CN), 1492 and 1452; δ_H(300 MHz; CDCl₃; Me₄Si) 7.32 (1 H, ddd, *J* 1.3, 7.4, 7.4, ArH), 7.25 (1 H, ddd, *J* 1.3, 7.5, 7.5, ArH), 7.20–7.19 (2 H, m, ArH), 7.08 (1 H, ddd, *J* 1.2, 7.6, 7.6, ArH), 6.96 (1 H, dd, *J* 1.0, 7.6, ArH), 6.77 (1 H, ddd, *J* 1.3, 7.6, 7.6, ArH), 6.58 (1 H, d, *J* 3.9, Hd), 6.49 (1 H, d, *J* 9.9, Ar-CH=C), 6.37 (1 H, d, *J* 7.7, ArH), 5.72 (1 H, dd, *J* 5.8, 9.9, Ar-C=CH), 4.72 (1 H, d, *J* 9.7, Hc), 4.24 (1 H, dd, *J* 5.8, 9.1, Hb) and 3.75 (1 H, ddd, *J* 3.9, Ha); δ_C(75 MHz; CDCl₃; Me₄Si) 141.88, 132.70, 130.65, 130.23, 129.60, 129.47, 129.38, 129.24, 128.53, 128.32, 128.24, 127.69, 126.70, 126.08, 122.63, 120.05, 116.50, 115.21, 50.14, 45.43, 41.62 and 40.62; *m/z* (CI) 307 (MH⁺, 6%), 182 (20), 155 (12), 154 (100) and 153 (23); HRMS calcd for C₂₂H₁₄N₂ (MH⁺) *m/z* 307.1236, found 307.1237.

In the case of **1b**, 1.18 g (6.32 mmol) was used. A similar separatory method afforded **3b** (180 mg, 89% based on consumed **1b**) and unreacted **1b** (979 mg, 83%). **3b**: mp 103 °C (from dichloromethane and *n*-hexane); λ_{max}(CH₃CN)/nm 275.5 (ε/dm³ mol⁻¹ cm⁻¹ 7300); ν_{max}/cm⁻¹ (CHCl₃) 3020, 2953, 2844, 1721 (C=O), 1435, 1275 (C–O) and 1215; δ_H(300 MHz; CDCl₃; Me₄Si) 7.48 (1 H, dd, *J* 0.8, 7.8, ArH), 7.31 (1 H, dd, *J* 1.5, 7.4, ArH), 7.21 (1 H, ddd, *J* 1.3, 7.4, 7.4, ArH), 7.14 (1 H, ddd, *J* 1.6, 7.6, 7.6, ArH), 6.95 (1 H, ddd, *J* 1.2, 7.4, 7.4, ArH), 6.85 (1 H, dd, *J* 1.3, 7.5, ArH), 6.77 (1 H, d, *J* 3.4, Hd), 6.62 (1 H, ddd, *J* 1.5, 7.6, 7.6, ArH), 6.44 (1 H, d, *J* 9.8, Ar-CH=C), 6.25 (1 H, d, *J* 7.8, ArH), 5.74 (1 H, dd, *J* 5.9, 9.8, Ar-C=CH), 4.57 (1 H, d, *J* 9.5, Hc), 4.06 (1 H, dd, *J* 5.9, 8.8, Hb), 3.67 (3 H, s, OMe), 3.64 (3 H, s, OMe) and 3.42 (1 H, ddd, *J* 3.4, Ha); δ_C(75 MHz; CDCl₃; Me₄Si) 175.02, 167.20, 137.86, 133.55, 131.28, 131.20, 130.64, 130.02, 129.78, 129.54, 128.66, 128.49, 128.05, 127.88, 127.78, 127.17, 126.37, 121.70, 54.33, 52.80, 51.96, 48.13, 44.47 and 38.47; *m/z* (CI) 373 (MH⁺, 14%), 215 (10), 188 (13), 187 (100), 186 (25), 183 (11) and 155 (33); HRMS calcd for C₂₄H₂₀O₄ (MH⁺) *m/z* 373.1440, found 373.1438.

Irradiation of the irradiated mixture of naphthalenes in the presence of xanthone at -78 °C

After 9 h of irradiation of **1a** (1.25 g, 8.16 mmol) in dichloromethane (140 cm³) at -78 °C, xanthone (300 mg) was added. The resulting mixture was irradiated at the same temperature for 4 h. After evaporation of the solvent, the reaction mixture was subjected to flash column chromatography (silica gel) eluting with *n*-hexane–dichloromethane mixtures of increasing polarity to afford unreacted **1a** (0.89 g, 71%), **4a** (178 mg, 49% based on consumed **1a**) and **3a** (110 mg, 30% based on consumed **1a**). **4a**: mp 176.0–177.0 °C (from dichloromethane and *n*-hexane); λ_{max}(CH₃CN)/nm 258.2 and 296.5 (ε/dm³ mol⁻¹ cm⁻¹ 1150 and 280); ν_{max}/cm⁻¹ (CHCl₃) 3019, 2235 (CN), 1520, 1492 and 1211; δ_H(300 MHz; CDCl₃; Me₄Si) 7.43 (2 H, dd, *J* 1.6, 7.3, ArH), 7.19–7.09 (4 H, m, ArH), 7.02 (2 H, dd, *J* 1.7, 7.2, ArH), 4.70 (2 H, d, *J* 7.6, Ar-CH-C) and 3.65–3.55 (4 H, m,

cyclobutyl); δ_C(75 MHz; CDCl₃; Me₄Si) 130.48, 129.53, 128.44, 127.69, 127.61, 125.44, 122.01, 51.01, 43.69, 37.09 and 30.20; *m/z* (CI) 307 (MH⁺, 10%), 182 (19), 155 (12), 154 (100) and 153 (21); HRMS calcd for C₂₂H₁₄N₂ (MH⁺) *m/z* 307.1236, found 307.1237.

In the case of **1b**, 1.47 g (7.90 mmol) of **1b** and 265 mg of xanthone were used. A similar separatory method afforded unreacted **1b** (1.12 g, 76%), **4b** (155 mg, 44% based on consumed **1b**) and **3b** (70 mg, 20% based on consumed **1b**). **4b**: mp 165.0 °C (from dichloromethane and *n*-hexane); λ_{max}(CH₃CN)/nm 260.2 (ε/dm³ mol⁻¹ cm⁻¹ 2594); ν_{max}/cm⁻¹ (CHCl₃) 3019, 1724 (C=O) and 1219 (C–O); δ_H(300 MHz; CDCl₃; Me₄Si) 7.04 (2 H, m, ArH), 6.99–6.93 (4 H, m, ArH), 6.67 (2 H, m, ArH), 4.90 (2 H, d, *J* 8.7, Ar-CH-C), 3.85 (6 H, s, OMe), 3.42 (2 H, t, *J* 7.3, cyclobutyl) and 3.23 (2 H, m, cyclobutyl); δ_C(75 MHz; CDCl₃; Me₄Si) 177.17, 135.31, 135.03, 129.69, 127.49, 127.37, 125.87, 58.38, 52.87, 48.44, 38.77 and 30.05; *m/z* (CI) 373 (MH⁺, 2%), 215 (8), 188 (13), 187 (100), 186 (33), 183 (10), 156 (6), 155 (51) and 143 (7); HRMS calcd for C₂₄H₂₀O₄ (MH⁺) *m/z* 373.1440, found 373.1431.

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