

# Effects of oligooxyethylene linkage on intramolecular [2 + 2] photocycloaddition of styrene derivatives

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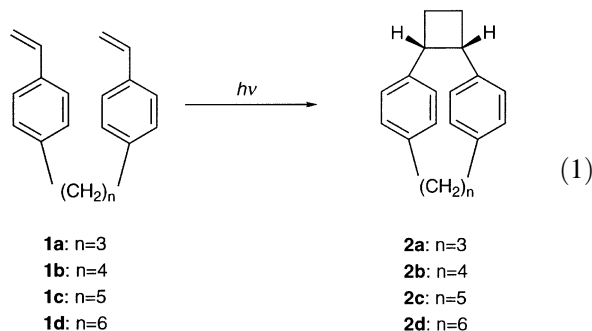
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**ABSTRACT:** There are two possible explanations for the high yields in the intramolecular [2 + 2] photocycloaddition of compounds **3a–d** in which two styrene moieties are bridged by an oligooxyethylene linkage: one is the electronic effects of phenoxy oxygen atoms at the *para*-position of the vinyl group and the other is the steric effects due to the flexibility of oligooxyethylene linkages. In order to clarify the contributions of the two effects, a study was made of the photoreaction of similar compounds in which oxygen atoms are present only at the ends of the chain, and therefore only the electronic effects are involved. In these compounds, the quantum yields of the photocycloaddition were found to be low. Hence the high yields in the photocycloaddition of **3a–d** are concluded to be ascribable to the flexibility of oligooxyethylene linkages. © 1998 John Wiley & Sons, Ltd.

**KEYWORDS:** [2 + 2]; photocycloaddition; quantum yield; oligooxyethylene linkage; cyclophane; styrene derivatives

## INTRODUCTION

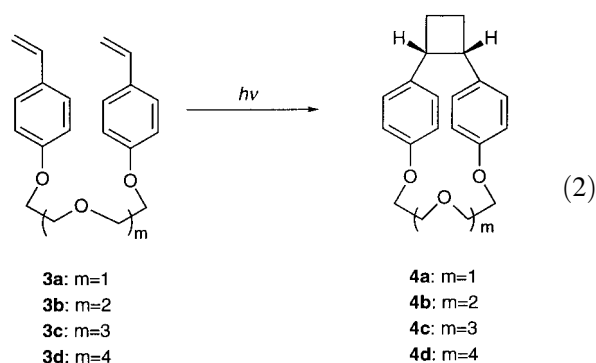
We have reported that the intramolecular [2 + 2] photocycloaddition of  $\alpha,\omega$ -bis(*p*-vinylphenyl)alkanes (**1**) readily afforded [2.*n*]paracyclophanes (**2**) having a cyclobutane ring [equation (1)].<sup>1</sup> The product yields were



found to depend sensitively on the length (*n*) of the oligomethylene chain; they were fairly high for *n* = 4 and 5 (61 and 49%, respectively), but much lower when *n* ≤ 3 (14% for *n* = 3) and *n* ≥ 6 (trace). For *n* = 2 and 3, the

desired cyclophanes are considered to be very strained, according to MM2 calculations. On the other hand, when *n* ≥ 6, the interaction between the two styrene moieties seems to be small owing to the long oligomethylene chain. Detailed photophysical investigations also revealed that this photoreaction is efficient with high quantum yields ( $\phi_d$ ) of ca 0.4 when *n* = 4 and 5.<sup>2</sup>

Furthermore, we have successfully applied this photoreaction to the synthesis of [2.*n*]paracyclophanes (**4**) possessing a crown ether subunit [equation (2)].<sup>3</sup>

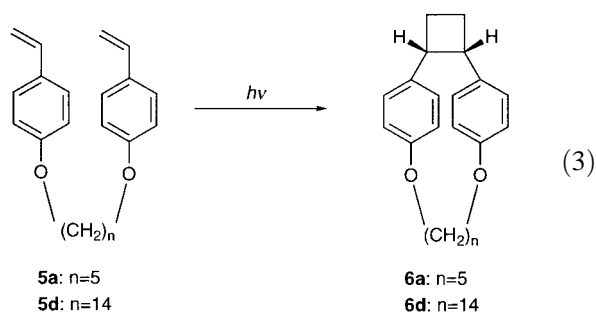


Surprisingly, high isolated yields could be attained for all of **3a–d**, although their chain length is comparable to *n* = 7, 10, 13 and 16, respectively, in **1**. Hence it is worthwhile investigating the photoreaction of **3** in detail and clarifying the reason for the high yields. There seem to be two possible explanations for the high yields. One is the electronic effects of the phenoxy oxygen atoms at the

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*para*-position of the vinyl group; these substituents may influence the properties of the excited state of **3**, such as the lifetime. The fluorescence of *p*-methoxystyrene is too weak to measure its lifetime, but several qualitative studies on the dimerization of styrene and *p*-methoxystyrene suggest these differences.<sup>4</sup> The other is the steric effects due to the flexibility of oligooxyethylene linkages, which may bring the two styrene units close together, in contrast with the oligomethylene chain in **1**. The difference in flexibility between C—C and C—O bonds is apparent, because according to Benson *et al.*'s increments, alkane *gauche* has a 0.8 kcal mol<sup>-1</sup> correction, whereas ether *gauche* has only a 0.3 kcal mol<sup>-1</sup> correction<sup>5</sup> (1 kcal = 4.184 kJ).

In order to clarify the contributions of the two effects, we have designed and synthesized two styrene derivatives, **5a** and **d**, in which oxygen atoms are present only at the ends of the chain and their total chain length is equal to that of **3a** and **3d**, respectively [equation (3)]. The



steric effects expected for the oligooxyethylene linkage of **3** can no longer be involved in **5**, while the electronic effects of alkoxy groups should still be effective. We have examined the intramolecular [2 + 2] photocycloaddition of **5a** and **d** and measured the quantum yields, in comparison with those of **1c** and **3a** and **d**. In this paper,

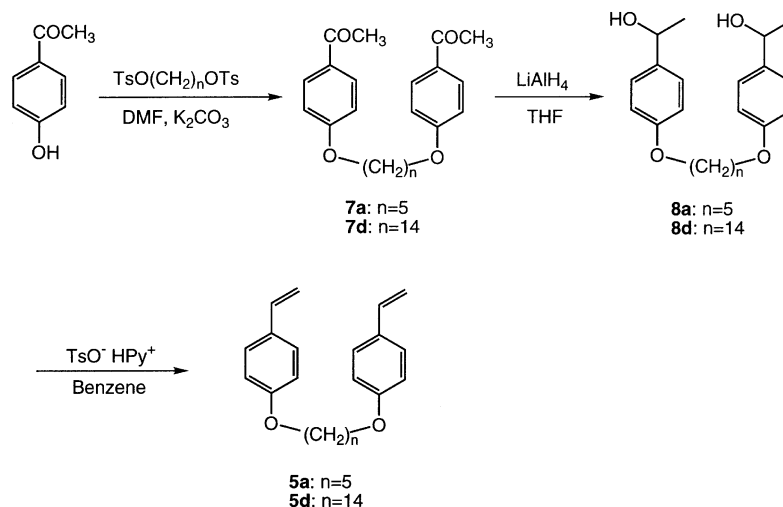
the role of the oligooxyethylene linkage in the photo-reaction is considered.

## RESULTS AND DISCUSSION

Styrene derivatives **5a** and **d** were synthesized as shown in Scheme 1. The intramolecular [2 + 2] photocycloaddition of **5** towards **6** was carried out with a 400 W high-pressure mercury lamp through a Pyrex filter in dry benzene under a nitrogen atmosphere, in a manner similar to that for **1** and **3**.<sup>1,3</sup> The yields of **6a** and **d** from **5a** and **d** were much lower (50 and 25%, respectively) than those of **4a** and **d** from **3a** and **d** (88 and 95%, respectively), and some unknown by-products were detected by TLC, HPLC and NMR spectroscopy after long reaction periods.

The quantum yields of the intramolecular [2 + 2] photocycloaddition of **3a** and **d** and **5a** and **d** were indirectly determined by using the value for **1c** ( $\phi_d = 0.39$ ) upon photoirradiation with  $\lambda = 253$  nm in non-polar isoctane as reported previously.<sup>2</sup> In practice, the  $\phi_d$  values were determined on the basis of the changes in the absorbance ( $D$ ) of the solution of styrene derivatives **3a** and **d** or **5a** and **d** at 253 nm. Since  $\epsilon_{\text{styrene}}$  for **3a** and **d** and **5a** and **d** (*ca*  $4 \times 10^4$ ) is much larger than  $\epsilon_{\text{cyclophane}}$  for **4a** and **d** and **6a** and **d** ( $10^2$ – $10^3$ ) at 253 nm, the  $D$  values gradually decreased with progress of photocycloaddition in all cases, corresponding to the consumption of the styrene derivatives.

The relationship between  $\Phi_d$  and  $D$  can be expressed as shown in equation (4) on the assumption that **3a** and **d** and **5a** and **d** are completely converted into **4a** and **d** and **6a** and **d**, respectively, at an early stage of the reaction. The derivation of equation (4) is described in the Experimental section in detail.



Scheme 1.

$$-\ln(D - D_0 \varepsilon_{\text{cyclophane}} / \varepsilon_{\text{styrene}}) + \ln(\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}}) \\ = \Phi_d I_0 \varepsilon_{\text{styrene}} \int_0^t (1 - 10^{-D}) / D dt, \quad (4)$$

where  $D_0$  denotes the initial absorbance. Since  $\ln(\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}})$  is constant, the plot of  $-\ln(D - D_0 \varepsilon_{\text{cyclophane}} / \varepsilon_{\text{styrene}}) + \ln(\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}})$  vs  $\int_0^t (1 - 10^{-D}) / D dt$  gives  $\Phi_d I_0 \varepsilon_{\text{styrene}}$  as the slope of the straight line. By determining  $\Phi_d I_0 \varepsilon_{\text{styrene}}$  for **1c** and other styrene derivatives, **3a** and **d** or **5a** and **d**, and using 0.39 as  $\Phi_d$  for **1c**, the  $\Phi_d$  values can be determined for **3a** and **d** and **5a** and **d**.

This method is sufficiently reliable in the case of **3a** and **d**; their consumption is considered to be virtually equal to the formation of cyclophanes **4a** and **d**, since the isolated yields are high. In contrast, the consumption of **5a** and **d** does not correspond exactly with the formation of **6a** and **d**, but is slightly higher than expected. However, the upper limit of  $\Phi_d$  for the photocycloaddition of **5a** and **d** can be reasonably estimated.

The quantum yields were determined for **3a** and **d** and **5a** and **d** in isoctane and acetonitrile. In all cases, the plot of  $-\ln(D - D_0 \varepsilon_{\text{cyclophane}} / \varepsilon_{\text{styrene}}) + \ln(\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}})$  vs  $\int_0^t (1 - 10^{-D}) / D dt$  was found to be sufficiently linear. The quantum yields determined on the basis of the slope  $\Phi_d I_0 \varepsilon_{\text{styrene}}$  are summarized in Table 1, along with the values reported previously.<sup>2</sup>

The quantum yields for the photocycloaddition of **3a** and **d** in isoctane ( $\Phi_d = 0.35$ ) are as high as that for **1c**. Thus, in this reaction, not only the yield but also the efficiency are high. The quantum yields seem hardly to be influenced by the length of the oligoxyethylene chain, although the values have not been measured for **3b** and **c** because they are easily expected to be the same. This tendency is in remarkable contrast with that in **1** having an oligomethylene chain.

On the other hand, the quantum yields for **5a** and **d**, especially **5d**, in isoctane are apparently lower than

those for **1c** and **3a** and **d**, as expected from the isolated yields, although the total chain length is equal to that of **3a** and **d**. Since the values ( $\Phi_d = 0.22$  and 0.12) in Table 1 are the upper limit of  $\Phi_d$  for **5a** and **d**, the true values should be smaller.

These results apparently indicate that the high yields in the photocycloaddition of **3a–d** are obviously ascribable to the steric effects of the oligoxyethylene chain rather than the electronic effects of the phenoxy oxygen atoms. The oligoxyethylene linkage is expected to bring the two styrene units close together in non-polar isoctane. Such steric effects of oligoxyethylene linkages are apparently derived from the presence of the ether linkages. These are expected to make the overall linkage more flexible than methylene chains, as mentioned in the Introduction. Such steric factors facilitate the proximity of the two styrene moieties almost independent of the chain length. Unfortunately, we could obtain no spectroscopic evidence supporting the proximity of the two styrene moieties. The UV spectra of **3d** and **5d** were similar to each other; the position of the absorption maximum was 260 nm ( $\varepsilon = 4 \times 10^4$ ) in both compounds. The fluorescence of both **3d** and **5d** was extremely weak, and no intramolecular excimer formation could be recognized, although the interaction between the two pyrene moieties at both ends of various chains has been thoroughly investigated by excimer formation.<sup>6</sup>

Although the phenoxy oxygen atoms at the *para*-position of the vinyl group seem somehow to influence also the behavior of the excited state of **3**,<sup>5</sup> the weak fluorescence prevents a deeper insight from being revealed.

The quantum yields in acetonitrile are almost comparable to those in isoctane for both **3a** and **d**, indicating that the steric effect of the oligoxyethylene chain is similarly operative also in a polar solvent for this reaction. The determination of the values for **5a** and **d** in acetonitrile was unsuccessful owing to the extremely low solubility. The isolated yields of cyclized products in polar solvents such as acetonitrile were often observed to be lower than those in non-polar solvents. It is concluded again from this study that the low efficiency in polar media is due to the decomposition of products due to the prolonged irradiation.<sup>3</sup>

**Table 1.** Quantum yields of intramolecular [2 + 2] photocycloaddition of styrene derivatives

Styrene derivative	Solvent		Ref.
	Isoctane	Acetonitrile	
<b>1a</b>	0.031	—	2
<b>1b</b>	0.38	—	2
<b>1c</b>	0.39	0.39	2, this work
<b>1d</b>	0.25	—	2
<b>3a</b>	0.35	0.34	This work
<b>3d</b>	0.35	0.33	This work
<b>5a</b>	0.22	— <sup>b</sup>	This work
<b>5d</b>	0.12 <sup>a</sup>	— <sup>b</sup>	This work

<sup>a</sup> Upper limit of the quantum yield.

<sup>b</sup> Measurements were impossible owing to the extremely low solubility of **5a** and **d** in acetonitrile.

## EXPERIMENTAL

*General.* Elemental analysis was performed at the Technical Research Center of Instrumental Analysis, Gunma University. NMR spectra were recorded on a Jeol Alpha-500 FT NMR spectrometer in CDCl<sub>3</sub> with tetramethylsilane (TMS) as an internal standard. Fast atom bombardment (FAB) mass spectra were measured on a JEOL JMS-HX110A mass spectrometer. UV spectra were recorded on a Hitachi U-3210 spectrophotometer in iso-

octane or acetonitrile. Melting points are not corrected. Benzene and tetrahydrofuran (THF) were distilled over sodium under a nitrogen atmosphere. *N,N*-Dimethylformamide (DMF) was dried over molecular sieves 4A 1/16. Other commercially available reagents were used without further purification.

**Preparation of 1,14-bis(4-acetylphenoxy)tetradecane (7d).** A mixture of *p*'-hydroxyacetophenone (3.30 g, 24.2 mmol), 1,14-tetradecaneditosylate (3.30 g, 6.1 mmol) and  $K_2CO_3$  (6.80 g, 49.2 mmol) was stirred in DMF (300 ml) at 30–35°C under a nitrogen atmosphere for 3 days. After the mixture had cooled to room temperature, excess  $K_2CO_3$  was filtered off. The filtrate was poured into water (400 ml) and extracted with dichloromethane (300 ml) three times. The combined extracts were washed with water and dried over anhydrous  $MgSO_4$ . The solution was concentrated under reduced pressure to give **7d** (1.63 g, 3.49 mmol) as a pale yellow solid in 57% yield; m.p. 115–117°C.  $^1H$  NMR (500 MHz,  $CDCl_3$ ),  $\delta$  7.93 (4H, d,  $J = 8.8$  Hz), 6.92 (4H, d,  $J = 8.9$  Hz), 4.01 (4H, t,  $J = 6.3$  Hz), 2.55 (4H, s), 1.80 (4H, m), 1.46 (4H, m), 1.24 (16H, m). HRMS (FAB): calculated for  $C_{30}H_{43}O_4$  ( $MH^+$ ) 467.3161; found, 467.3143.

**Preparation of 1,14-bis[4-(1-hydroxyethyl)phenoxy]tetradecane (8d).** A solution of **7d** (1.63 g, 3.49 mmol) in THF (50 ml) was added dropwise a suspension of  $LiAlH_4$  (0.76 g, 20.0 mmol) in THF (90 ml) at room temperature under a nitrogen atmosphere. After the mixture had been stirred for 19 h under reflux, water (10 ml) and THF (100 ml) were slowly added dropwise to the reaction mixture. The resulting suspension was filtered and the filtrate was concentrated under reduced pressure to give **8d** (1.59 g, 3.38 mmol) as a white solid in 96% yield; m.p. 106–109°C.  $^1H$  NMR (500 MHz,  $CDCl_3$ ),  $\delta$  7.29 (4H, d,  $J = 8.6$  Hz), 6.87 (4H, d,  $J = 8.6$  Hz), 4.85 (2H, q,  $J = 6.7$  Hz), 3.94 (4H, t,  $J = 6.6$  Hz), 1.77 (4H, m), 1.48 (6H, d,  $J = 6.4$  Hz), 1.45 (4H, m), 1.27 (16H, m). HRMS (FAB): calculated for  $C_{30}H_{45}O_3$  ( $MH^+ - H_2O$ ), 453.3369; found, 453.3350.

**Preparation of 1,14-bis(4-vinylphenoxy)tetradecane (5d).** A solution of **8d** (1.59 g, 3.38 mmol) and pyridinium *p*-toluenesulfonate (0.32 g, 1.27 mmol) in benzene (120 ml) was stirred under reflux overnight with a Dean–Stark trap. After the mixture had cooled to room temperature, the precipitate was filtered off. The filtrate was washed with saturated aqueous NaCl solution twice, dried over anhydrous  $MgSO_4$  and concentrated under reduced pressure. The residue was purified by column chromatography ( $SiO_2$ , hexane–benzene) to give **5d** (0.54 g, 1.24 mmol) as a white solid in 36% yield; m.p. 110–113°C.  $^1H$  NMR (500 MHz,  $CDCl_3$ ),  $\delta$  7.33 (4H, d,  $J = 8.6$  Hz), 6.85 (4H, d,  $J = 8.6$  Hz), 6.66 (2H, dd,  $J = 11.0$  & 17.7 Hz), 5.60 (2H, d,  $J = 17.4$  Hz), 5.11 (2H,

d,  $J = 11.0$  Hz), 3.95 (4H, t,  $J = 6.5$  Hz), 1.77 (4H, m), 1.44 (4H, m), 1.27 (16H, m). HRMS (FAB): calculated for  $C_{30}H_{43}O_2$  ( $MH^+$ ), 435.3263; found, 435.3285.

**Photoreaction of 5d towards 6d.** Photoirradiation of **5d** (0.16 g, 0.368 mmol) was carried out with a 400 W high-pressure mercury lamp through a Pyrex filter in benzene (185 ml) under a nitrogen atmosphere for 2 h. Insoluble material was filtered off and the filtrate was concentrated under reduced pressure. The residue (yellow oil) was stirred with borane–THF complex (0.75 ml) in a small amount of THF under a nitrogen atmosphere for 3 days. Water was added dropwise to the mixture in order to decompose the borane complex, until evolution of gas stopped. The solvent was evaporated under reduced pressure and the resulting residue was purified by column chromatography ( $SiO_2$ , hexane–benzene) to give **6d** (0.04 g, 0.09 mmol) as a pale yellow solid in 25% yield; m.p. 49–52°C.  $^1H$  NMR (500 MHz,  $CDCl_3$ ),  $\delta$  6.82 (4H, d,  $J = 8.5$  Hz), 6.65 (4H, d,  $J = 8.9$  Hz), 3.91 (2H, m), 3.85 (4H, t,  $J = 6.5$  Hz), 2.40 (4H, m), 1.72 (4H, m), 1.43 (4H, m), 1.27 (16H, m). HRMS (FAB): calculated for  $C_{30}H_{43}O_2$  ( $MH^+$ ), 435.3263; found, 435.3257.

**Spectroscopic data for 5a.** M.p. 84–85°C.  $^1H$  NMR (500 MHz,  $CDCl_3$ ),  $\delta$  7.33 (4H, d,  $J = 8.6$  Hz), 6.85 (4H, d,  $J = 8.6$  Hz), 6.66 (2H, dd,  $J = 10.9$  and 17.7 Hz), 5.60 (2H, d,  $J = 17.7$  Hz), 5.12 (2H, d,  $J = 10.7$  Hz), 3.99 (4H, t,  $J = 6.4$  Hz), 1.86 (4H, m), 1.65 (2H, m). Analysis: calculated for  $C_{21}H_{24}O_2$ , C 81.78, H 7.84; found, C 81.75, H, 7.84%.

**Spectroscopic data for 6a.** M.p. 118–122°C.  $^1H$  NMR (500 MHz,  $CDCl_3$ ),  $\delta$  6.90 (2H, dd,  $J = 2.3$  and 8.4 Hz), 6.60 (2H, dd,  $J = 2.6$  and 8.4 Hz), 6.56 (2H, dd,  $J = 2.3$  and 8.4 Hz), 6.51 (2H, dd,  $J = 2.6$  and 8.4 Hz), 4.07 (4H, t,  $J = 6.0$  Hz), 4.00 (2H, m), 2.47 (4H, m), 1.51 (4H, m), 1.15 (2H, m). HRMS (FAB): calculated for  $C_{21}H_{25}O_2$  ( $MH^+$ ), 309.1855; found, 309.1876.

**Derivation of equation (4).** On the assumption that **3a** and **d** and **5a** and **d** are completely converted into **4a** and **d** and **6a** and **d**, respectively, at an early stage of the reaction, the  $\Phi_d$  value is defined as follows:

$$\Phi_d I_{\text{styrene}} = -dC_{\text{styrene}}/dt \quad (5)$$

where  $I_{\text{styrene}}$  is the intensity of the light absorbed by styrene derivatives **3a** and **d** and **5a** and **d** and  $C_{\text{styrene}}$  is their concentration.  $I_{\text{styrene}}$  is expressed as

$$I_{\text{styrene}} = I_{\text{total}} D_{\text{styrene}} / (D_{\text{styrene}} + D_{\text{cyclophane}}) \quad (6)$$

where  $I_{\text{total}}$  is the total intensity of the light absorbed by both styrene derivatives and cyclophanes and  $D_{\text{styrene}}$  and  $D_{\text{cyclophane}}$  are the absorbances of the styrene derivatives and cyclophanes, respectively.  $I_{\text{total}}$  is expressed as

$$I_{\text{total}} = I_0[1 - 10^{-(D_{\text{styrene}} + D_{\text{cyclophane}})}] \quad (7)$$

Equations (5), (6) and (7) lead to

$$-dC_{\text{styrene}}/dt = \Phi_d I_0 [1 - 10^{-(D_{\text{styrene}} + D_{\text{cyclophane}})}] D_{\text{styrene}} / (D_{\text{styrene}} + D_{\text{cyclophane}}) \quad (8)$$

By substituting  $D$  for  $D_{\text{styrene}} + D_{\text{cyclophane}}$ , equation (8) becomes

$$-dC_{\text{styrene}}/dt = \Phi_d I_0 (1 - 10^{-D}) D_{\text{styrene}} / D \quad (9)$$

Since  $D_{\text{styrene}} = \varepsilon_{\text{styrene}} C_{\text{styrene}} d$ , where  $d$  is the optical path length [=1 cm (constant)], equation (9) is transformed into

$$-dC_{\text{styrene}}/dt = \Phi_d I_0 (1 - 10^{-D}) \varepsilon_{\text{styrene}} C_{\text{styrene}} / D \quad (10)$$

$$(-dC_{\text{styrene}}/dt)(1/C_{\text{styrene}}) = \Phi_d I_0 (1 - 10^{-D}) \varepsilon_{\text{styrene}} / D \quad (11)$$

By integrating both sides of equation (11) with respect to time  $t$ , we obtain

$$-\ln C_{\text{styrene}} = \Phi_d I_0 \int_0^t (1 - 10^{-D}) \varepsilon_{\text{styrene}} / D dt \quad (12)$$

When  $C_0$  is defined as the initial concentration of styrene derivatives and  $D_0$  as the initial absorbance, we obtain

$$D_0 = \varepsilon_{\text{styrene}} C_0 d \quad (13)$$

On the assumption that styrene derivatives are completely converted into cyclophanes, we obtain

$$C_0 = C_{\text{styrene}} + C_{\text{cyclophane}} \text{ (constant)} \quad (14)$$

By using equations (13) and (14),  $D$  is expressed as follows:

$$\begin{aligned} D &= (\varepsilon_{\text{styrene}} C_{\text{styrene}} + \varepsilon_{\text{cyclophane}} C_{\text{cyclophane}}) d \\ &= (\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}}) C_{\text{styrene}} d + \varepsilon_{\text{cyclophane}} C_0 d \\ &= (\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}}) C_{\text{styrene}} d + \varepsilon_{\text{cyclophane}} C_0 d \\ &= (\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}}) C_{\text{styrene}} d + \varepsilon_{\text{cyclophane}} D_0 / \varepsilon_{\text{styrene}} \end{aligned} \quad (15)$$

Equation (15) is transformed into

$$C_{\text{styrene}} = (D - D_0 \varepsilon_{\text{cyclophane}} / \varepsilon_{\text{styrene}}) / (\varepsilon_{\text{styrene}} - \varepsilon_{\text{cyclophane}}) \quad (16)$$

Equations (12) and (16) result in equation (4).

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