

# Electropolymerization of an EDOT-modified diarylethene

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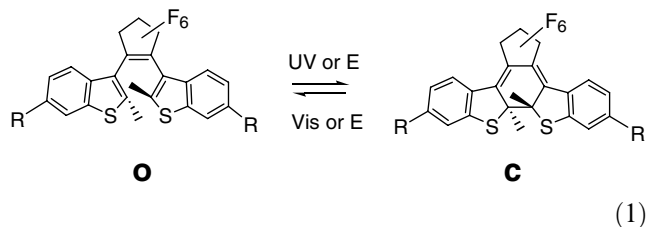
**Abstract**—A diarylethene substituted with 3,4-ethylenedioxythiophene (EDOT) was synthesized to induce electrochemical anodic polymerization. Upon electrochemical oxidation of 1,2-bis(2-methylbenzo[*b*]thiophene-3-yl)perfluorocyclopentene (BTF)-substituted EDOT at the 6,6'-position (BTFTT), a red-purple polymeric film (PBTFTT) was deposited on a working electrode. A similar film was deposited on an electrode from the solution exposed to UV light through electrochemical oxidation. The film growth was controlled by the cycle numbers in cyclic voltammetry during the electropolymerization. The film thickness was linearly correlated to the potential cycle numbers, with a slope of 17.9 nm/cycle. The IR spectrum of the electrodeposited polymer showed characteristic C=C stretching frequency at 1630 and 1481  $\text{cm}^{-1}$  indicating that the BTF units in the polymer are closed. © 2006 Published by Elsevier Ltd.

## 1. Introduction

Diarylethenes have attracted much interest as a switching material.<sup>1–7</sup> Stimulating them by irradiation with light at an appropriate wavelength or electrochemically, it is possible to switch their properties not only in electronic absorption but also for various physical and chemical properties,<sup>7–11</sup> which are important for electronic and photonic applications. To be able to employ diarylethenes in such devices, diarylethenes should be processed in a solid form such as a film or a crystal. Thus, the direct electrodeposition of a diarylethene film on a substrate is a convenient solution to their application.

The most remarkable difference between diarylethene isomers formed by photoexcitation<sup>12,13</sup> (or electrochemical reaction for some specific derivatives) is that the  $\pi$ -system of two aryl rings are separated in an open-ring isomer (o), whereas they are connected throughout the molecule in a closed-ring isomer (c).

Thus, the oxidation potential of the c isomer is lower than that of the open-form (o) isomer,<sup>14</sup> and there is a high possibility of oxidative coupling in the c isomer to lead anodic polymerization, which can offer a convenient method for the preparation of a photochromic



diarylethene film. Although electrochemical reactions of diarylethenes have been studied extensively,<sup>15–17</sup> there is no report yet of their anodic polymerization leading to the electrodeposition of a diarylethene polymer film, possibly due to the formation of stable dication, which leads to dimers.

To investigate the anodic polymerization of diarylethene, a new BTF derivative was synthesized with redox-active EDOT groups at the 6,6'-position (R = EDOT in Eq. 1). It has been well known that EDOT polymerizes to yield a PEDOT film through its electrochemical oxidation.<sup>18</sup> Thus EDOT-modified diarylethene is a promising material to stimulate electropolymerization of the closed isomer, which could allow for the direct electrodeposition of diarylethenes as thin films for organic devices.

This letter reports on the synthesis and electropolymerization of the 3,4-ethylene-dioxythiophene (EDOT)-modified diarylethene (BTFTT).

**Keywords:** Diarylethene; Electrodeposition; Thin film; Photocurrent; Switching; Photovoltaic.

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## 2. Results and discussion

### 2.1. Synthesis and photochromic properties of BTFTT

1,2-Bis(2-methyl-1-benzothiophen-3-yl)perfluorocyclopentene (**1**, BTF) was prepared from the reactions of perfluorocycloalkene with an organolithium compound.<sup>8a</sup> Compound **1** was subjected to iodination with iodine and periodic acid in an acetic acid solution to yield **2**. EDOT-modified diarylethene (BTFTT) was synthesized as a pale yellow powder through palladium-catalyzed Stille coupling of **2** with **3**<sup>19</sup> in the presence of Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> as a catalyst (Scheme 1).

Figure 1 shows the UV/vis spectral change upon the photochromic conversion of BTFTT in methylene chloride (1.0 × 10<sup>-5</sup> M) through their exposure to 365 nm light. A colorless solution that contained the open-ring isomer (o-BTFTT) showed an absorption tail that reached 390 nm. Upon its irradiation with UV light, new absorption bands appeared at longer wavelengths [ $\lambda_{\text{max}}$  = 406 nm (3.05 eV) and 570 nm (2.17 eV)] within 3 min, which were ascribed to the absorption bands of the closed-ring isomer (c-BTFTT). Compared to the unsubstituted diarylethene (**1**), the absorption bands of BTFTT were red shifted in both the open and closed isomers, indicating that the EDOT groups affected the electronic transition of the diarylethenes. The color of the solution was completely bleached upon irradiation with a visible light source (532 nm laser) as shown in Figure 1.

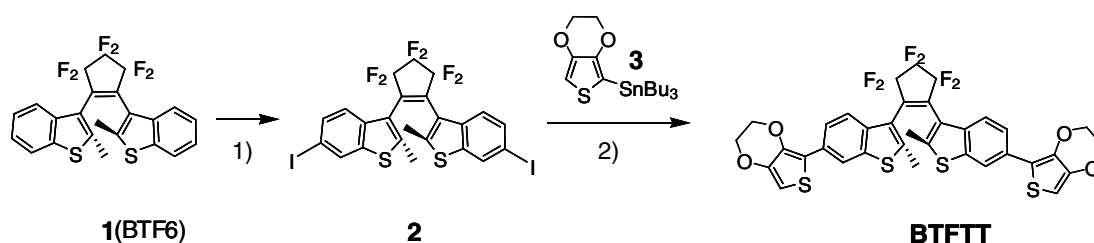
Accompanied by the color change, the solution of BTFTT in CDCl<sub>3</sub> showed a new peak at 2.04 ppm upon its excitation with UV light, indicating the formation of

a closed isomer with UV excitation (Supporting information). The integrated intensity increase was linearly correlated to the irradiation time and reached photostationary state after 30 min of irradiation.

### 2.2. Electrodeposition of diarylethene polymer film

Figure 2 shows cyclic voltammograms (CVs) of BTFTT (0.001 M) in methylene chloride that contained 0.1 M tetra-*n*-butylammonium perchlorate (TBAP), by cycling the potential between -0.7 and +1.5 V, in the dark. The first cycle shows an anodic peak at 1.45 V, which is ~0.1 V lower than the oxidation of the unsubstituted derivative (**1**),<sup>10,14</sup> implying that the electron donating EDOT group lowers the oxidation potential.

Application of repetitive scans between -0.7 and +1.5 V resulted in the increase in the intensity of anodic waves at lower potentials with a dramatic decrease of the peak at ~1.45 V and at -0.28 V. The irreversibility of the redox peak at 1.45 V indicates that there are fast chemical reaction followed by the redox reaction. Surprisingly, the electrode was coated with a red-purple film, and the film color was intensified as the potential cycles were continued. This indicates that polymer is deposited on the working electrode by the electropolymerization of the BTFTT, as observed in the electropolymerization of other EDOT derivatives. Since the colorless solution containing o-BTFTT (Fig. 2a) showed a peak at ~1.0 V, which could be assigned as the oxidation peak for the closed isomer,<sup>14</sup> we can ascribe the polymerization to the oxidative polymerization of the closed isomer formed by the oxidation of the open isomer.



Scheme 1. Synthesis of BTFTT. Reagents and conditions: (1) I<sub>2</sub>, H<sub>5</sub>IO<sub>6</sub>/acetic acid, H<sub>2</sub>SO<sub>4</sub>, 70 °C. (2) PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>/toluene, reflux, 24 h.

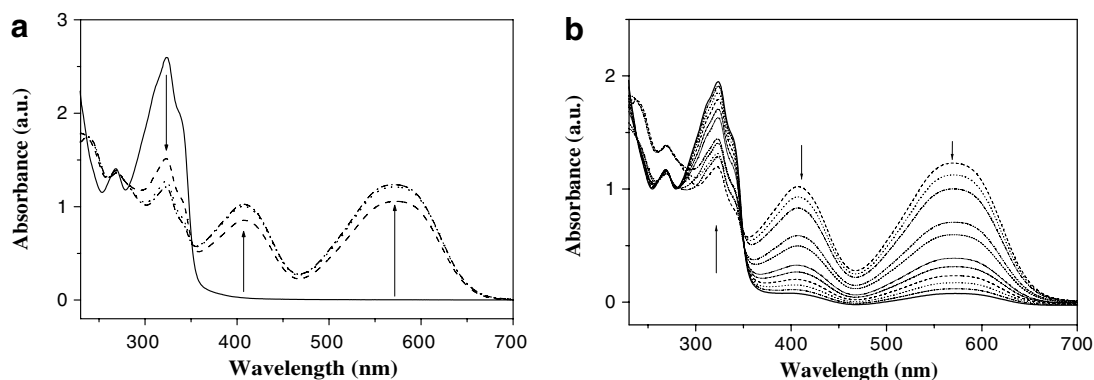
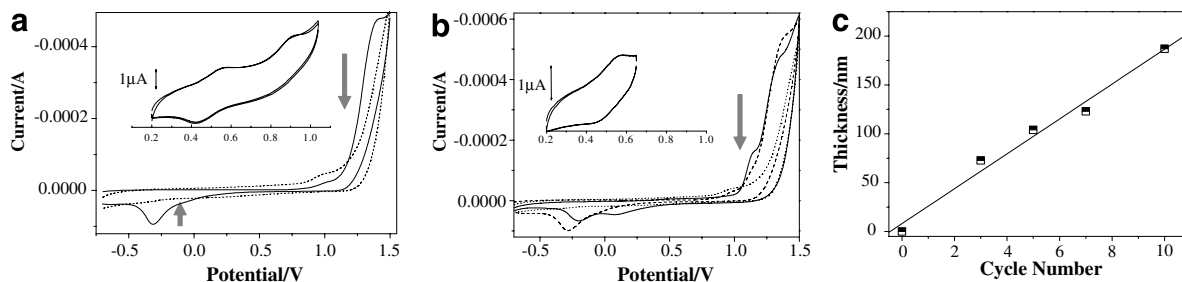


Figure 1. Spectral change of BTFTT (1.0 × 10<sup>-5</sup> M) in methylene chloride through UV/vis irradiation. (a) UV exposure for 0, 60, 120, and 180 s. (b) Room light exposure for 0, 60, 120, 180, 240, 300, 360, 420, 480, and 600 s.



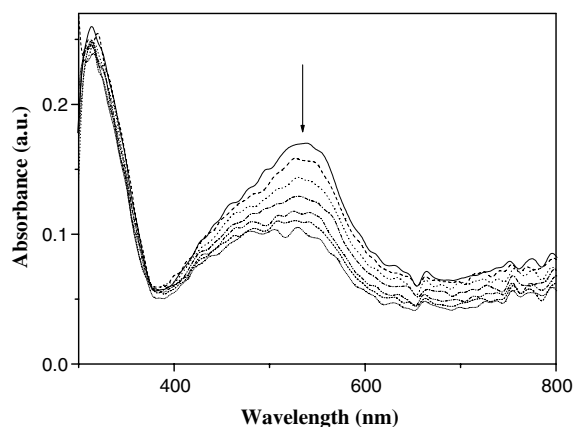
**Figure 2.** Cyclic voltammograms of BTFTT ( $1 \times 10^{-3}$  M) in dichloromethane containing 0.1 M of *n*-Bu<sub>4</sub>NClO<sub>4</sub> (a) before and (b) after irradiation with a UV light (365 nm), for the first cycle (solid line) and the fifth cycle (dotted line) with a scan rate of 50 mV/s (Ag/AgCl reference). The dashed line in Figure 2b shows the CV of the first cycle before UV irradiation. Insets show the CV of the electrodeposited film in the same electrolyte solution without BTFTT (scan rate: 10 mV/s). (c) Film thickness at different cycle numbers in (b).

Figure 2b shows the CV for a purple solution of BTFTT that was exposed to UV light for 3 min. The first cycle CV in Figure 2b showed two irreversible peaks at  $\sim 1.45$  V and  $\sim 1.1$  V, which can be assigned as the oxidation process of open and closed isomer, respectively. It was noticeable that the intensity of the peak at  $\sim 1.1$  V (oxidation of the closed BTF unit) was reduced in the CV of the fifth cycle, indicating that the closed isomer was converted to the polymers by electrochemical polymerization. The electrode was also coated with a red-purple film and the film color was intensified as the potential cycles were increased, indicating the growth of a film similar to that in Figure 2a. After five potential cycles between  $-0.7$  and  $+1.5$  V, the thickness of the film reached to 110 nm, as determined by an alpha step.

The film growth could be controlled by the cycle numbers in cyclic voltammetry during the electropolymerization. The film thickness determined from an alpha step was linearly correlated to the number of potential cycles, with a correlation factor of 0.9911, as shown in Figure 2c. The slope indicated that the growth of film in each cycle corresponded to the film growth of 17.9 nm.

The red-purple films deposited onto the electrode from the solution of BTFTT in the dark and exposed to UV light showed similar redox peaks (reversible at  $E_{1/2} = 0.48$  V; quasi-reversible at  $E_{1/2} = 0.82$  V), as compared in the inset of Figure 2a and b, respectively. The film was insoluble in common solvents and showed absorption band centered at 534 nm (Fig. 3), which were different from that of *c*-BTFTT (Fig. 1b). Its color was bleached slowly upon its exposure to a 532 nm laser, as shown in Figure 3.

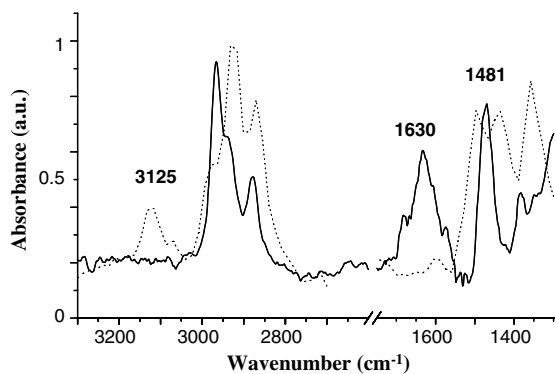
Many diarylethene derivatives are known to undergo electrocyclic ring cyclization induced by electrochemical reduction or oxidation.<sup>15–17</sup> The diarylethene derivatives undergo oxidative cyclization reactions when the radical cations of the closed-ring isomers are more stable than the open-ring isomers and also that the diarylethenes undergo oxidative cycloreversion reactions when the radical cations of the open-ring isomers are more stable than the closed-ring isomers.<sup>17b</sup> The electrochemically produced closed isomers, dication, and dimers of diarylethene are generally soluble in organic solvents. The



**Figure 3.** Spectral change of the electrodeposited film, prepared from the BTFTT solution exposed to UV light, by the visible light exposure (532 nm laser,  $5 \text{ mW cm}^{-2}$ ) for 0, 15, 30, 45, 60, 75, and 90 min (top to bottom).

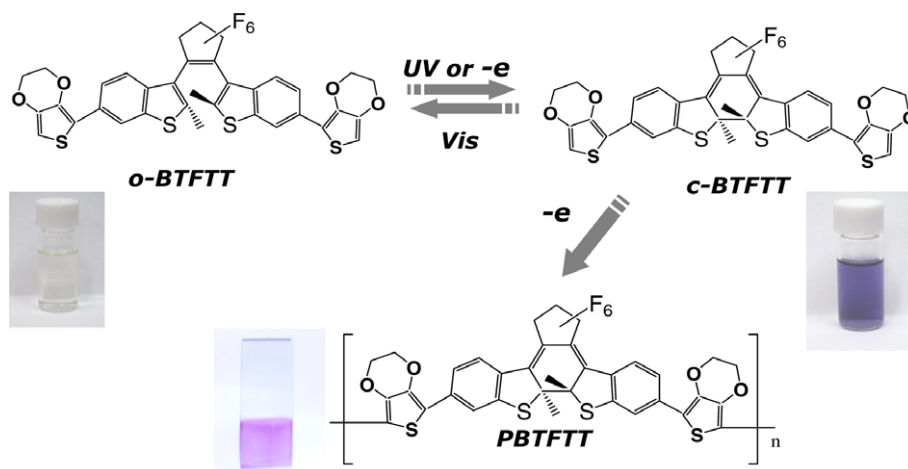
closed isomers produced electrochemically have same spectral/physical properties as those produced from photoisomerization reaction.<sup>15</sup> The electrodeposited red-purple colored film from the solution of BTFTT (both unexposed and exposed to UV light) in our study is insoluble in an organic solvent and water. Thus it could be high molecular products having BTFTT unit. Since it is deposited under oxidative condition, it can be assigned as the polymer of *c*-BTFTT that is produced by either electrochemical oxidation or UV exposure.

The structure of the polymer was characterized by FT-IR. The aromatic C–H stretching observed at  $3125 \text{ cm}^{-1}$  for the monomer (BTFTT) was significantly reduced in the polymer film as shown in Figure 4. This result indicates that the polymerization consumes aromatic hydrogen, which could be replaced by BTFTT units in the polymer. The closed form of diarylethene has been reported to show characteristic C=C stretching frequency for the internal C=C bond of the hexatriene in the BTF unit in the range of  $1650\text{--}1400 \text{ cm}^{-1}$ .<sup>11a</sup> Indeed we observed new bands for the polymer sample at  $1630$  and  $1481 \text{ cm}^{-1}$  which could be assigned as the C=C stretching frequency of the newly formed C=C bond in the closed BTF unit upon electropolymerization. Thus the structure of BTF unit in the polymer could be assigned as closed.

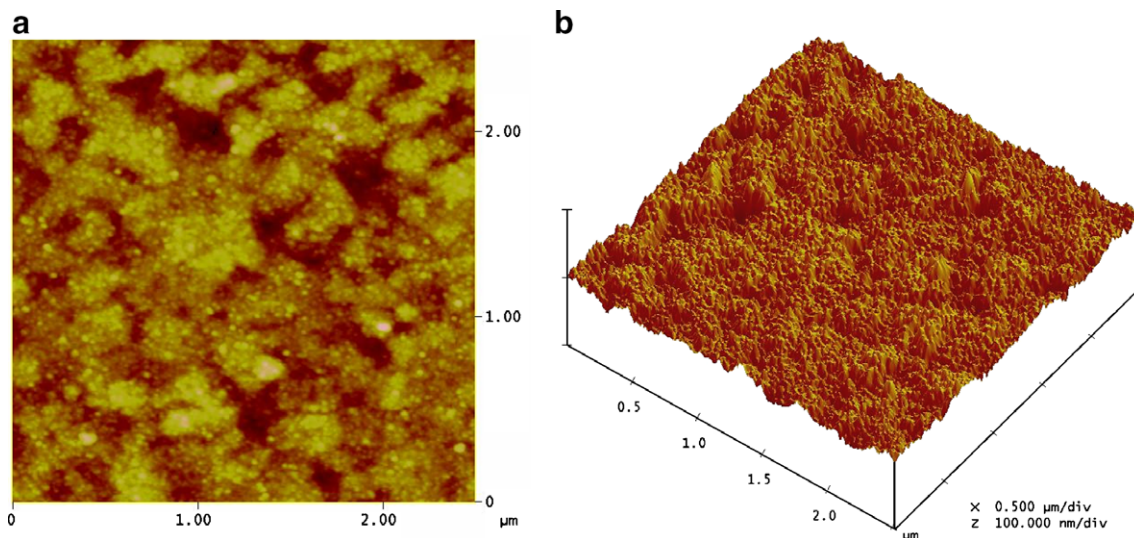


**Figure 4.** FT-IR spectra of PBTFTT (solid line) and BTFTT (dotted line) solids.

The driving force for the polymerization of the closed isomer is the formation of redox stable insoluble polymer having lower oxidation potential by the extended  $\pi$ -electron conjugation pathways through the main chain. **Scheme 2** summarizes the electrochemical polymerization processes.



**Scheme 2.** Electrochemical deposition of BTFTT.



**Figure 5.** Morphology of the electrodeposited film examined through AFM: (a) top view and (b) side view.

Although electrochemical reactions of dithienylcyclopentenes (DTEs) have been reported,<sup>15–17</sup> their anodic polymerization leading to the electrodeposition of a diarylethene polymer film has not yet been reported. Only oxidation polymerization of a hydroxyl-functionalized diarylethene has been reported to give insoluble polymers in an organic solvent.<sup>20</sup> Thus, the electrochemical deposition of a BTF derivative substituted with an electropolymerizable group (EDOT) in this study is unique, and it offers a convenient deposition method of a diarylethene polymer film.

The morphology of the film (dried, 50 nm thickness) electrodeposited onto an ITO substrate from the UV-exposed solution of BTFTT was examined through Tapping-mode atomic force microscopy (AFM). A flat film of a rather homogeneous structure with an average roughness of 5 nm was observed (**Fig. 5a** and **b**). The films consisted of small grains with an average size of 20 nm.

In summary a new diarylethene derivative, BTFTT was synthesized by coupling BTF and EDOT. BTFTT

showed reversible photochromic properties by alternative irradiations with a UV and vis light source. BTFTT underwent electrochemical oxidation to afford polymeric films deposited on an ITO glass. The electrochemical deposition of a BTF derivative substituted with an electropolymerizable group (EDOT) in this study is unique, and it offers a convenient deposition method of a diarylethene polymer film. Further characterizations of the electrodeposited film from BTFTT and its derivatives are in progress, to understand the mechanism of the electrodeposition and to explore their photoelectric properties.

### 3. Experimental

#### 3.1. General methodology

Methylene chloride and toluene were distilled from phosphorus pentoxide prior to use, and tetra-*n*-butylammonium perchlorate (TBAP) was purchased from TCI. Compounds **2** and **3** were synthesized according to Refs. **21** and **19**, respectively. <sup>1</sup>H NMR spectra was recorded in a CDCl<sub>3</sub> solution that contained Me<sub>4</sub>Si as the internal standard with Bruker (300 MHz). The high resolution mass spectrum, HRMS, (FAB mode) was obtained from the Korea Basic Science Institute (KBSI). FT-IR spectra were obtained from a powdered neat samples using a Bruker Vector 22 FT-IR spectrometer. Column chromatography was performed using silica gel (200–400 mesh, Merck). All reactions were monitored for completion using thin-layer chromatography (TLC), which was performed using a pre-coated silica gel plate (Merck 60F<sub>245</sub>), and detection was performed with the aid of UV light. The UV–vis spectra were recorded for chloroform solutions with a AvaSpec (AvaSpec-2048, light source: AvaLight-DHS). The electrochemical and photoelectrochemical properties were studied using an electrochemical analyzer (CH Instruments Inc, CHI624B). The liquid electrolyte solution for the redox properties of BTFTT was a methylene chloride solution that contained tetra-*n*-butylammonium perchlorate (TBAP, 0.1 M) as a supporting electrolyte. The condition was composed of a three-electrode assembly equipped with a platinum or ITO working electrode, a platinum coil as the counter electrode, and an Ag/AgCl electrode as the reference electrode. Measurements were examined in a glass cell at room temperature. The cell was measured before and after it was irradiated with UV light (PowerArc UV100, 100 mW/cm<sup>2</sup>) and after it was bleached with visible light. The scan rate in the CV experiment was 100 mV/s and the voltage range was –0.7 to +1.5 V.

#### 3.2. Synthesis of 1,2-bis[6-(3,4-ethylenedioxythienyl)-2-methyl-1-benzothiophen-3-yl]perfluorocyclopentene (BTFTT)

Under an argon atmosphere, **2** (100 mg, 0.138 mmol) and **3** (119 mg, 0.277 mmol) were dissolved in toluene (40 mL). To this was added a catalytic amount of dichlorobis(triphenyl-phosphine)palladium(II) (PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>) (7 mg, 0.01 mmol) and the mixture was refluxed for

24 h. During the reaction, the color changed from yellow to black as Pd<sup>0</sup> was formed. After the reaction mixture was cooled, it was poured into a saturated sodium chloride solution (50 mL). Then benzene (20 mL) was added to it. The organic layer was extracted with benzene (3 × 60 mL), dried over MgSO<sub>4</sub>, filtered, and evaporated. The residue was purified using column chromatography on a silica gel with hexane/ethyl acetate (5:1) as the eluent to produce BTFTT [yield = 87%; parallel (p):anti-parallel (ap) ratio = 65:35; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) = 2.04 (s, 2H, apMe), 2.19 (s, 2H, apMe), 2.47 (s, 1H, pMe), 4.25–4.27 (m, 2H, –OCH<sub>2</sub>), 4.32 (m, 2H, –OCH<sub>2</sub>), 6.27–6.32 (s, 1H, –CH–), 7.2–7.7 (m, 6H, ArH), 7.9 (s, 0.4H, apArH), and 8.00 (s, 0.6 H, pArH); HRMS (FAB) *m/z* (MH<sup>+</sup>) calcd for C<sub>35</sub>H<sub>22</sub>O<sub>4</sub>F<sub>6</sub>S<sub>4</sub>, 748.0305; obsd 748.0311 (100%, M<sup>+</sup>).

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#### Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tetlet.2006.11.039.

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