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Visible-Light-Dependent Photocyclization: Design, Synthesis, and Properties of a Cyanine-Based Dithienylethene

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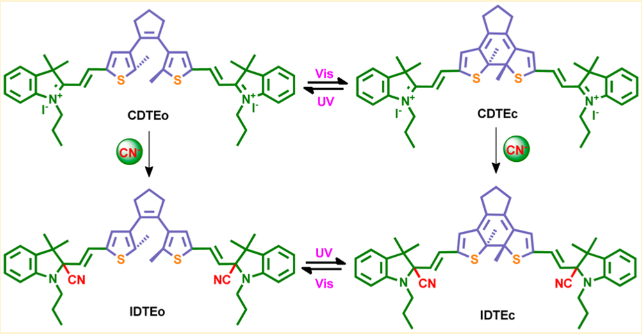
Supporting Information

ABSTRACT: A cyanine-based dithienylethene is developed. Its photoisomerization shows that (1) the irradiation of visible light can induce cyclization while the cycloreversion takes place upon irradiation with UV light and (2) the addition of CN⁻ can lead to cyclization and cycloreversion upon irradiation of UV light and visible light, respectively.



Photochromic materials have attracted considerable attention because of their significant potential applications in many fields such as electro-optical functional materials and novel biomaterials.[1] Photochromic dithienylethene derivatives with heterocyclic aryl groups are one of the most attractive families among photochromic compounds, due to their remarkable fatigue resistance, excellent thermally irreversible properties, and high sensitivity.[2] Over the past decades, numerous dithienylethenes with excellent photochromism have been widely developed. With regard to photocyclization, most of them usually rely on the irradiation of ultraviolet light with a high energy.[2]

It is well-known that visible or near-infrared light has deeper penetration and weaker energy than ultraviolet light and is more suitable for application in biological systems.[3] Therefore, developing photochromic materials that undergo reversible isomerization reactions upon visible or near-infrared light irradiation is significant. At present, several main strategies have been employed to design visible or near-infrared light-induced photochromic materials, which include intramolecular energy transfer via triplet states, multiphoton processes, and upconversion luminescence.[4] In addition, another alternative approach is to extend the π-conjugation length by introducing a conjugated aromatic dye into a dithienylethene backbone.[5] A representative example has been reported recently by Fukaminato and Irie.[5d] In this example, the introduction of perylene diimide (PDI) or perylene monoimide (PMI) dyes linking the central reactive carbon atoms (such as the R_1 site in Scheme 1a) by an acetylene bridge leads to the photocyclization of dithienylethene upon irradiation with visible light. In comparison with the central reactive carbon atoms, the two side positions of the dithienylethene backbone are easier to modify by chemical reactions.[2] The same group found that no photocyclization takes place when the PMI dye unit is located in the R_2 position of the 1,2-bis(thiophen-3-yl)cyclopent-1-ene backbone, despite the fact that the conjugated system has been extended, as shown in Scheme 1a.[5d] Accordingly, finding a suitable conjugated block is still a challenging project for developing ditheinylethenes with visible light cyclization. On the basis of our previous work,[6] herein we present an unprecedented example of cyanine-based dithienylethene by introducing acceptor−π−acceptor (A−π−A) dye moieties[7] into the two sides of the dithienylethene backbone. As shown in Scheme 1b, the cyanine-based dithienylethene (**CDTEo**) undergoes photocyclization on irradiation of visible light, affording the ring-closed isomer (**CDTEc**). It is worth mentioning that **CDTEo** displays a highly selective response for cyanide anion due to the formation of indole-based dithienylethene (**IDTEo**). In comparison to **CDTEo**, UV light irradiation can induce the photocyclization of **IDTEo** to generate the corresponding ring-closed isomer (**IDTEc**).

The dithienylethene **CDTEo** described above was synthesized in 70% yield by refluxing 2,3,3-trimethyl-1-propyl-3H-indol-1-ium iodide[8] with 1,2-bis(5-formyl-2-methylthien-3-yl)-cyclopentene[9] in anhydrous ethanol (Scheme 2). Next, studies were carried out to explore the photoisomerization behavior of **CDTEo**. The photochromism of **CDTEo** induced by photoirradiation in CH_3CN was investigated at room temperature. It underwent photoisomerization between the ring-opened

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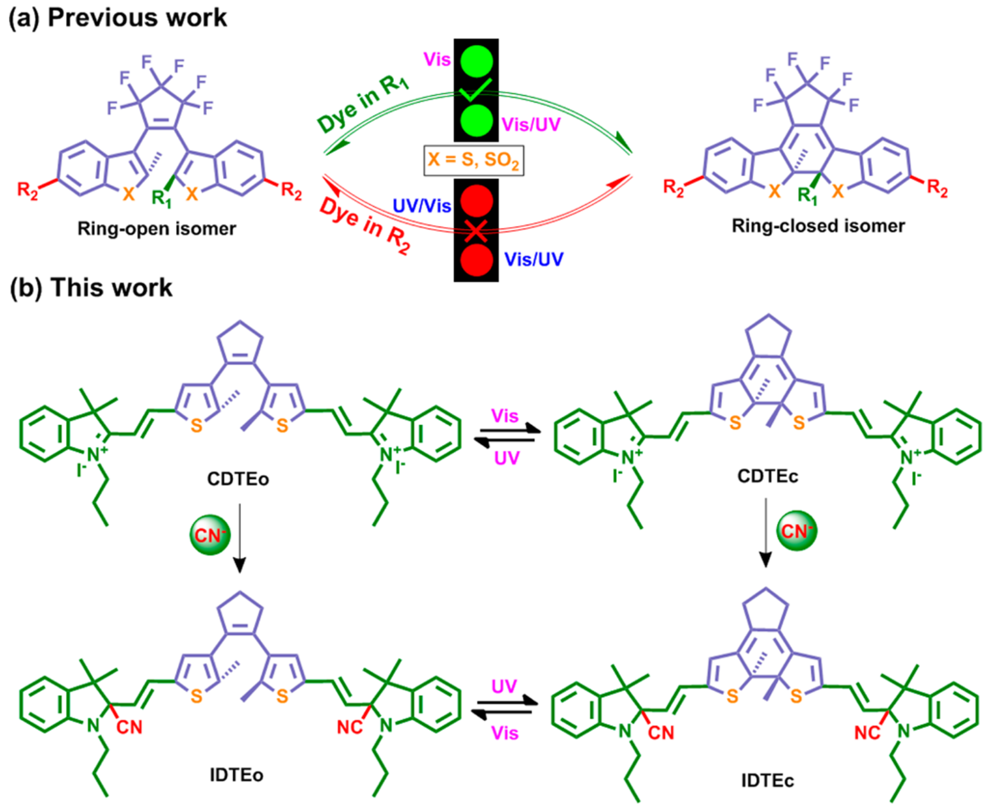
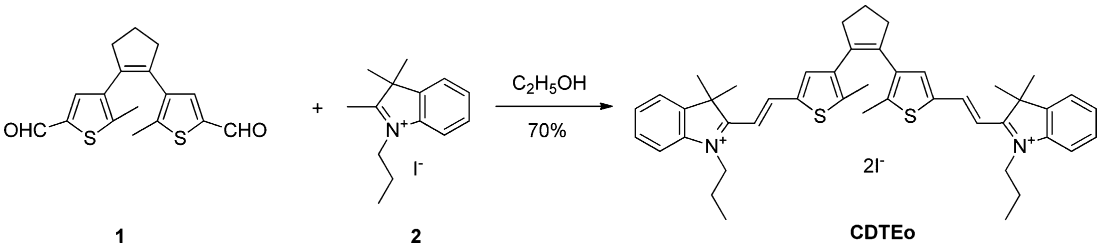
Scheme 1. Photochromism of (a) Perylene Dye Based Dithienylethenes and (b) Cyanine Based Dithienylethene



Scheme 2. Synthesis of Cyanine Based Dithienylethene CDTEo



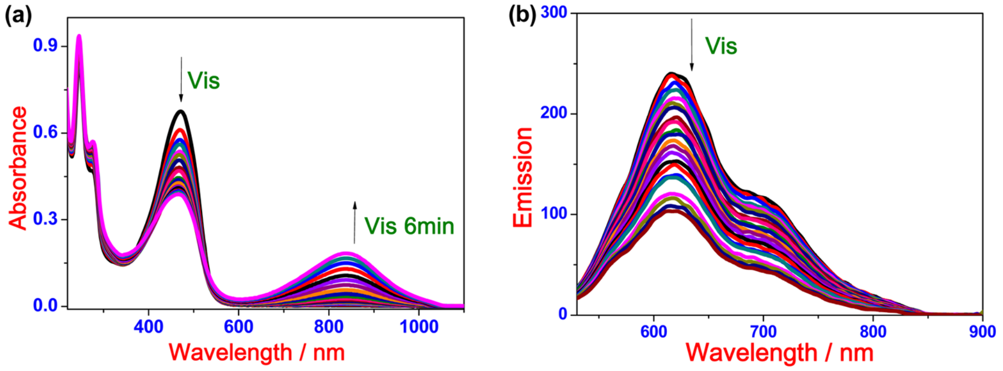


Figure 1. Changes in absorption spectra (a) and emission intensity (λ_{ex} 500 nm) (b) of cyanine based dithienylethene **CDTEo** in CH_3CN (2.0×10^{-5} mol/L) under light irradiation at 402 nm.

isomer and the ring-closed isomer upon alternating irradiation with visible light (λ >402 nm) and UV light (λ 302 nm), as illustrated in Scheme 1b. As shown in Figure 1a, the absorption maximum of **CDTEo** in CH_3CN was observed at 470 nm (ε = 3.37×10^4 L mol^{-1} cm^{-1}) as a result of a π–π* transition.[10] In addition, a new absorption band centered at 840 nm (ε = 0.92 $\times$ 10^4 L mol^{-1} cm^{-1}) appeared when it was irradiated with visible light (λ >402 nm), as a result of a ring-closure reaction to give the ring-closed isomer **CDTEc**. Upon irradiation with UV light (λ 302 nm), the colored ring-closed isomer **CDTEc** underwent a cycloreversion reaction to form the initial ring-opened isomer, and the investigation on fatigue resistance

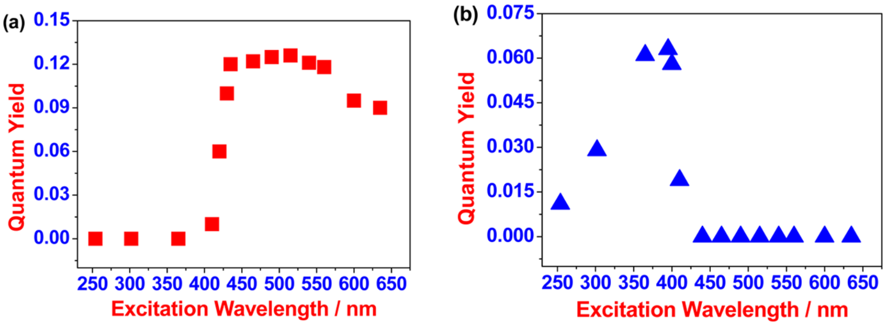


Figure 2. Excitation wavelength dependence of the photocyclization (a) and photocycloreversion (b) reactions of **CDTE** in CH_3CN.

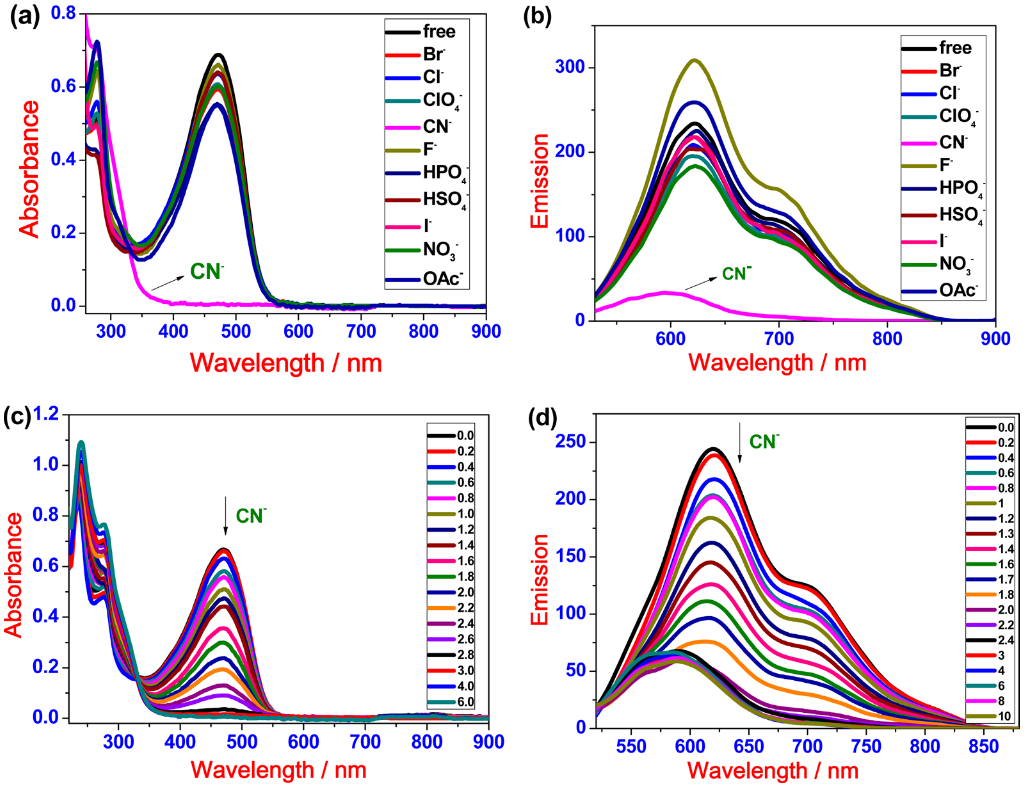


Figure 3. Changes in absorbance spectra (a) and fluorescence intensity (b) of **CDTEo** upon the addition of different anions (10 equiv) and the titration of UV−vis absorption (c) and fluorescence (d) upon the addition of cyanide in CH_3CN/H_2O (95/5, v/v).

indicates that **CDTEo** has good reversibility (Figure S1 in the Supporting Information). The cyclization and cycloreversion quantum yields of **CDTEo** are 0.151 and 0.029, respectively. Simultaneously, we also find that there were no obvious changes when the ring-opened isomer **CDTEo** underwent irradiation with UV light, and no decrease of intensity was found when the **CDTEc** underwent irradiation with visible light (Figure S2 in the Supporting Information). This result further confirms that the photocyclization of **CDTEo** is visible light dependent. The results indicated that the photocyclization reaction takes place upon irradiation with visible light (λ >402 nm), which is different from the case for normal photochromic molecules, possibly owing to introducing acceptor−π−acceptor (A−π−A) dye moieties at the two sides of the dithienylethene backbone. To further evaluate the photochromic reactivity, HPLC was used to analyze the conversion ratio (Figure S3 in the Supporting Information). The chromatograms of **CDTEo** indicated that the conversion ratio is 79% from the ring-opened to the ring-closed isomer at the photostationary state (PSS) under visible light irradiation and the ratio is 73% from the ring-closed to the ring-opened isomer at the PSS under UV light irradiation. Next, we investigated the excitation wavelength dependence for photocyclization and photocycloreversion, as shown in Figure 2, which shows that only visible light can excite the photocyclization of **CDTEo**, while only UV light can induce the photocycloreversion of **CDTEc**. The excitation wavelength dependence of the photocycloreversion reaction of **CDTEc** showed that the quantum yield decreases to almost zero upon excitation with >435 nm light. The photochromic parameters are summarized in Table S1 in the Supporting Information.

The strongly electron deficient indolium group is usually used as a nucleophilic addition reaction block for anions, especially for those of strong nucleophilic activity such as cyanide, in the field of molecular recognition and fluorescent probes.[11] Subsequent investigations of anion response were performed. As shown in Figure 3a, this yellow solution turns colorless upon the addition of CN^- ions (10 equiv) in a

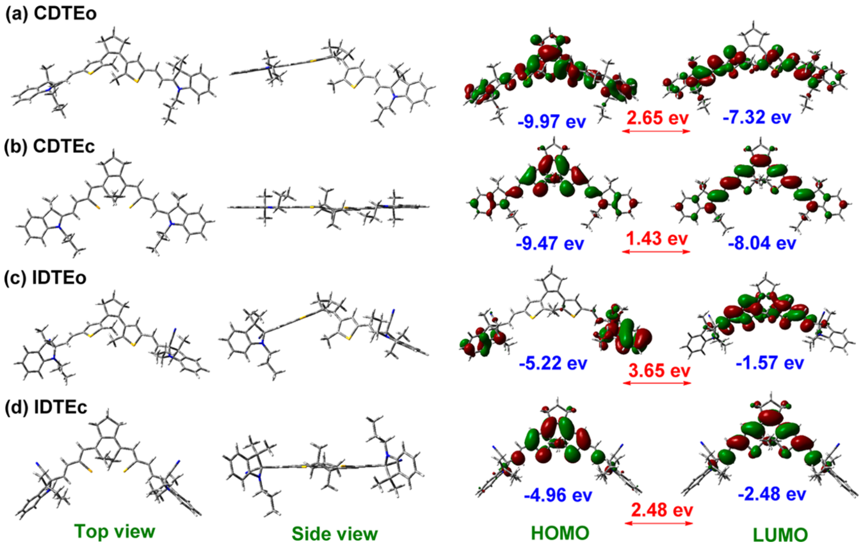


Figure 4. Frontier molecular orbital profiles of **CDTE** and **IDTE** based on TD-DFT calculations at the B3LYP/6-31G* level by using the Gaussian 09 program.

CH_3CN/H_2O solution mixture (95/5, v/v); however, no obvious colorimetric changes were observed by the naked eye when other anions ($CH_3CO_2^-$, F^-, Cl^-, NO_3^-, Br^-, I^-, $H_2PO_4^-$, ClO_4^-, and HSO_4^-) were added. Similarly, an obvious fluorescence decrease was found upon addition with CN^- ions, as presented in Figure 3b. The results suggest that **CDTEo** can be used as a colorimetric and fluorescent probe for detecting cyanide in aqueous solution (Figure S4 in the Supporting Information). Next, the titration of **CDTEo** upon addition of cyanide in a CH_3CN/H_2O solution mixture (95/5, v/v) was investigated. As described in Figure 3c, a gradual decrease at 450 nm of UV−vis absorption is observed along with the addition of cyanide due to the breaking of the conjugated system. Similar fluorescence titration in Figure 3d indicated that cyanide led to decreasing fluorescent emission accompanied by a blue shift of around 30 nm. In addition, a well-defined isosbestic point at 330 nm was observed and the Job plot confirms a product with ratio of 1/2 (Figure S5 in the Supporting Information). Accordingly, we guess that the nucleophilic addition reaction takes place on the indolium sites, generating corresponding cyano-based **IDTEo**. Further proof provided by ^{1}H NMR confirmed the formation of the product **IDTEo** (Figure S7 in the Supporting Information). The partial ^{1}H NMR spectra of **CDTEo** in CD_3CN/D_2O (95/5, v/v) were obtained (as shown in Figure S7). Upon addition of CN^- ions, the resonance signals of the protons on aromatic cycles and vinylene units of **CDTEo** displayed remarkable upfield shifts in comparison with **IDTEo** as a result of the transfer from positively charged indolium to the neutral indole moiety. The results indicate that the nucleophilic attack of cyanide anions takes place in indolium, forming the corresponding adduct **IDTEo.**[11b] Moreover, cyanide also breaks the acceptor−π−acceptor (A−π−A) system and as a consequence the photoreactivity of cyanine-based dithienylethene obviously decreases upon irradiation from visible light with low energy to UV light with high energy.

The isomerization behavior of **CDTEo** in the presence of CN^- in CH_3CN-H_2O (95/5, v/v) was also investigated at room temperature. The result showed that it underwent changes between the ring-opened isomer **IDTEo** and the ring-closed isomer **IDTEc** upon alternating irradiation with UV light (λ 302 nm) and visible light (λ >402 nm), respectively, as illustrated in Scheme 1b. As observed (Figure S8 in the Supporting Information), **IDTEo** reveals an absorption maximum at 280 nm (ε = 1.61 × 10^4 L mol^{-1} cm^{-1}) in CH_3CN/H_2O (95/5, v/v). This colorless solution turned purple and a new absorption band centered at 542 nm (ε = 0.374 × 10^4 L mol^{-1} cm^{-1}) appeared when it was irradiated with 302 nm UV light, as a result of a ring-closure reaction to give the ring-closed isomer of **IDTEc**. Upon irradiation with visible light (λ >402 nm), the colored ring-closed isomer underwent a cycloreversion reaction to give the initial colorless ring-opened isomer. Similarly, the fluorescent property of **CDTEo** in the presence of CN^- induced in CH_3CN/H_2O (95/5, v/v) was studied at room temperature. The result exhibited that the fluorescent emission intensity at 590 nm gradually decreased upon irradiation with UV light (λ 302 nm) and the structure changed from the ring-opened state to the ring-closed state. The result implies that the cyanide can induce the cyclization reaction with irradiation with UV light in comparison to the visible-light-dependent cyclization of **CDTEo.**

In order to further investigate the photoreactivity of **CDTEo** and **IDTEo**, time-dependent density functional theory (TD-DFT) calculations at the B3LYP/6-31G* level using the Gaussian 09 program were performed. Details of the optimized structures and molecular orbital correlation diagrams of ring-opened and ring-closed isomers are shown in Figure 4. Despite the fact that the cyclopentene ring is noncoplanar with the two thiophene rings, thiophene rings with indolium linked by a vinylene unit are on the same plane with each other, implying that the two sides of **CDTEo** have a good conjugation. Its HOMO orbital energy is almost distributed over the whole molecule, while the orbital energy of the LUMO is largely distributed over the vinylene and indolium moieties due to the strong influence of positively charged indolium. The closed-ring form **CDTEc** presents a fully planar conjugated backbone. As a consequence, its HOMO and LUMO orbital energies are delocalized along the whole conjugated molecule, with the HOMO coefficient mainly on the switch center while the LUMO coefficient is nearly on the whole molecular backbone. Such extended π conjugation leads to a low optical energy gap of 1.43 eV, in comparison to 2.65 eV for the ring-opened isomer **CDTEo**. In contrast, the addition of cyanide results in distortion between the thiophene rings and indole units of **IDTEo** owing to the break in conjugation. As shown in Figure 4, the HOMO coefficient is mainly distributed on the two indole cycles due to the large influence of the electron-deficient cyano group, while the LUMO coefficient mainly focuses on the vinylene and thiophene units. The good coplanarity and conjugation of ring-closed form **IDTEc** induces the orbital energies of the HOMO and LUMO to be delocalized nearly along the vinylene-linked switch center. As expected, **IDTEc** exhibits a narrower energy band gap (2.48 eV) in comparison to **IDTEo** (3.65 eV). It is worth mentioning that the calculated electronic absorption spectrum is similar to the experimental data (Table S2 and Figure S9 in the Supporting Information).

CONCLUSION

In summary, an unprecedented example of cyanine-based dithienylethene by introduction of acceptor−π−acceptor (A−π−A) dye moieties into the two sides of the dithienylethene backbone has been reported. Its photoswitching behavior shows that the photocyclization is induced by visible light irradiation, affording the ring-closed isomer. Specially, **CDTEo** displays a highly selective response for cyanide anion due to the formation of indole-based dithienylethene (**IDTEo**). In comparison to **CDTEo**, the UV light irradiation can induce the photocyclization of **IDTEo** to generate the corresponding ring-closed isomer (**IDTEc**). This work provides an alternative strategy for designing a dithienylethene system with photocyclization by visible light irradiation.

EXPERIMENTAL SECTION

General Methods. All manipulations were carried out under an argon atmosphere by using standard Schlenk techniques, unless stated otherwise. Ethanol was freshly distilled from magnesium/iodine. 1,2-Bis(5-formyl-2-methylthien-3-yl)cyclopentene (**1**) was prepared by literature methods.[9] Compound **2** was synthesized by a reported method. All other starting materials were obtained commercially as analytical grade and were used without further purification. The relative quantum yields were determined by comparing the reaction yield with the known yield of the compound 1,2-bis(2-methyl-5-phenyl-3-thienyl)perfluorocyclopentene.[12] Elemental analyses were performed by investigation of C, H, and N. ^{1}H and ^{13}C NMR spectra were collected on a 400 or 600 MHz spectrometer. ^{1}H and ^{13}C NMR chemical shifts are relative to TMS. UV−vis spectra were obtained on a UV spectrophotometer. UV irradiation was performed with a UV lamp (302 nm) and visible light irradiation (λ >420 nm) with a tungsten lamp with cutoff filters. The samples were injected (10 μL) into a reversed-phase monolithic column, connected to an HPLC system coupled to an ultraviolet detector (DAD, λ_{exc} 250 nm). Chromatographic separation was achieved by isocratic mode consisting of 60/40 (v/v) solution of water and methanol at a flow rate of 1.0 mL min^{-1}.

Synthesis of CDTE. To a solution of **1** (94.92 mg, 0.3 mmol) in anhydrous EtOH (40 mL) was added 2,3,3-trimethyl-1-propylindolenine (**2**; 207.40 mg, 0.63 mmol) under an argon atmosphere. The mixture was refluxed for 24 h. After the solvent was removed, the formed precipitate was collected, the crude product was washed with ethyl ether, and the dried solid was recrystallized from ethyl ether to give the brown solid **CDTEo** in a yield of 70%. ^{1}H NMR (400 MHz, CD_3CN): δ ppm 1.02−1.07 (m, 10H), 1.55 (s, 9H), 1.75 (s, 2H), 1.77 (s, 6H), 2.74 (s, 3H), 2.85−2.91 (m, 4H), 4.32−4.43 (m, 4H), 6.96 (d, J = 16 Hz, 2H), 7.60−7.76 (m, 8H), 7.88 (s, 2H), 8.39 (d, J = 16 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ ppm 11.3, 17.0, 21.4, 23.1, 27.2, 51.2, 52.2, 54.6, 115.3, 123.3, 129.5, 130.0, 135.7, 136.8, 139.4, 140.6, 140.7, 141.5, 143.1, 149.3, 180.9, 195.7. EI MS: m/z 685.12 [M − 2I$^-$ + H$^+$]; calculated exact mass 938.16. Anal. Calcd for $C_{45}H_{52}I_2N_2S_2$: C, 57.57; H, 5.58; N, 2.98. Found: C, 57.37; H, 5.45; N, 2.89.

ASSOCIATED CONTENT

Supporting Information

Figures and tables giving UV−vis absorption data, data for theoretical calculations, and ^{1}H NMR, ^{13}C NMR, and MS spectra of all new compounds. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.joc.5b01466.

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Notes

The authors declare no competing financial interest.

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