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Synthesis and photochromic reactivity of a new class of photochromic diarylethene dimer-containing dithieno[3,2-*b*:2′3′-*d*]thiophene

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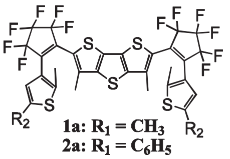
ABSTRACT

Novel photochromic diarylethene dimers containing dithieno[3,2-*b*:2′3′-*d*]thiophene were synthesized and their photochromic properties were studied in solution as well as in the crystalline phase. Only the isomer with the diarylethene units one in open-form and one in closed-form was produced upon irradiation with ultraviolet light because of the intramolecular excitation energy transfer in this isomer. Their electrochemical properties were also investigated associating with computational studies.

1. Introduction

Fused oligothiophene derivatives have received increasing attention in the last decade due to their fundamental optoelectronic properties and their potential applications in the fabrication of semiconductor devices.[1–4] Amongst them, dithieno[3,2-*b*:2′,3′-*d*] thiophene (**DTT**) is a fused oligomer of thiophene units with extended π-conjugation, interesting electric and optical properties, which have been widely employed as building block for the development of organic semiconductors as active materials in organic field effect transistors (OFETs) and organic photovoltaics (OPVs).[5–10]

Photochromic diarylethenes have attracted remarkable attention because of their potential ability for studies of optical memory media and switching devices.[11] Among them diarylperfluorocyclopentenes are the most promising candidate due to their superior photochromic properties with high thermal stability, high fatigue resistance, and fast response time.[11,12] Aiming to develop organic semiconductors with photoswitchable electrical transport properties, we have tried to incorporate diarylethene units with **DTT**.[13] Recently, there is also a similar report on diarylethene compound containing **DTT** moiety for the development of multi-functional material.[14,15] However, up to date, reversible photochromic reaction in solid state is still unsuccessful in such molecular system, which is critical for achieving photoswitchable electrical transport induced by reversible photochromic reaction. Herein, a new class of photochromic diarylethene dimer with a **DTT** linkage, compounds **1a** and **2a** (Scheme 1) are designed and synthesized. The two compounds show favorable photochromic performance in solution as well as in the crystalline phase. To the best of our knowledge, this is the first report on diarylethene dimers, which display reversible photochromic reaction in the crystalline state.



Scheme 1. Chemical structures of diarylethenes **1a** and **2a**.

2. Discussion and results

2.1. Synthesis

Diarylethenes **1a** and **2a** containing **DTT** units were synthesized according to the route illustrated in Scheme 2. The key starting material, 3,5-dimethyldithieno[3,2-*b*:2′3′-*d*]thiophene, was synthesized with an efficient method developed in our group.[13]

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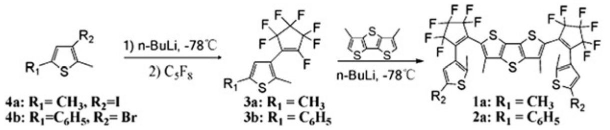
3,5-Dimethyldithieno[3,2-*b*:2′3′-*d*]thiophene has two reaction sites in 2- and 6-positions, so coupling of lithiated 3,5-dimethyldithieno[3,2-*b*:2′3′-*d*]thiophene with compound **3a**/**3b** afforded **1a** and **2a** with yields of 46% and 60% by controlling the ratio of starting materials, respectively. The molecular structures of diarylethenes **1a** and **2a** were confirmed by ^{1}H NMR, ^{13}C NMR, EIMS, elemental analysis, and X-ray crystallography.



Scheme 2. Synthetic route of diarylethenes **1a** and **2a**.

2.2. Photochromic reactions in solution

The photochromic behavior of diarylethenes **1a** and **2a** induced by photoirradiation at room temperature was measured in hexane. The absorption spectral changes of **1a** and **2a** induced by alternating irradiation with UV light and visible light at appropriate wavelength are shown in Fig. 1. As shown in Fig. 1a, the open-ring isomer **1a** exhibited an absorption maximum at 386 nm as a result of a π–π* transition.[16] Upon irradiation with 380 nm, the absorption band at 386 nm decreased along with the appearance of a new absorption band at 468 nm, indicating the formation of a closed-ring isomer. The original absorption spectrum was recovered by irradiation with visible light. Two well-defined isosbestic points at about 338 and 426 nm were observed, suggesting that the photocyclization could only take place at one of the diarylethene units (Scheme 3) due to the intramolecular excitation energy transfer from the excited open-ring unit to the closed-ring unit prohibits the formation of the two closed-ring form dimer **1c**.[14,15] This could be further verified by ^{1}H NMR spectra analysis. The absence of the isomers having two closed-ring forms **1c** in the photoirradiated dimer agrees to the result observed in the dimers reported by Branda.[17] Similar inactivity of the second photocyclization has also been reported for molecules containing two diarylethene units.[14,15,18–20] In the photostationary state, the conversion ratio from **1a** to **1b** was 49.01% under irradiation with a light of 380 nm as estimated by HPLC analysis (Fig. S1).

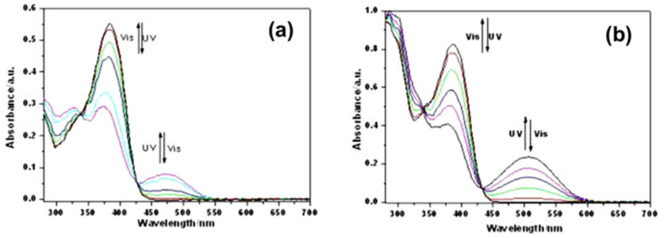
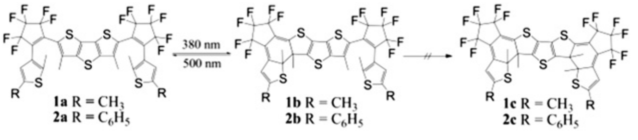


Fig. 1. Absorption spectral changes of **1a** and **2a** in hexane upon irradiation with 380 and 500 nm (C=1.5×10^{-5} mol/L): (a) **1a**, (b) **2a**.



Scheme 3. Photochromic reaction of **1** and **2**.

The absorption characteristic of **2a** was similar to that of **1a** (Fig. 1b). Upon irradiation with UV light, a new absorption band in the visible region appeared as a result of the cyclization reactions, which results in the formation of isomer **2b**. In the photostationary state, the conversion ratio from **2a** to **2b** was 87.91% under irradiation with a light of 380 nm as estimated by HPLC analysis (Fig. S3).

The closed-ring isomers **1b** and **2b** were isolated by HPLC from the photogenerated colored solution and characterized by ^{1}H NMR. For diarylethene **1a** in the photostationary state upon irradiation with 380 nm, the photogenerated colored solution gave two peaks with retention time of 31 and 39 min in HPLC. The first one is assigned to **1a**, and the second peak is due to **1b** according to their absorption spectra and ^{1}H NMR spectroscopy. Fig. 2a shows ^{1}H NMR spectra of the isomer **1b** in the range of 2.6–1.6 ppm. Six different protons with the same integration corresponding to six methyls could be observed for protons at 2.44, 2.31, 1.93, 1.89, 1.72, and 1.64 ppm. The assignment of these signals was illustrated in inset molecular structure in Fig. 2a. It is expected that if the isomer **1c** with two closed-ring form of dithienylethene was generated upon UV irradiation, three methyl protons should be observed due to the symmetric molecular structure of **1c**.

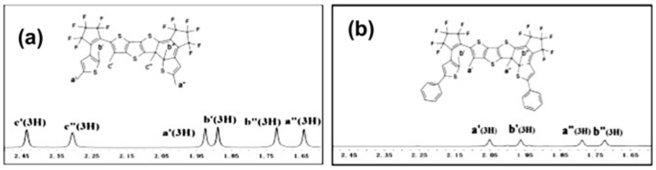


Fig. 2. ^{1}H NMR spectra of **1b** (a) and **2b** (b) in the range of 2.6–1.6 ppm.

Similar to that of **1b**, four methyl protons could be observed in the ^{1}H NMR spectra of isolated **2b** with chemical shifts of 2.05, 1.96, 1.79, and 1.72 ppm (Fig. 2b) corresponding to the formation of the isomer with one closed-ring form dithienylethene. The signals at 1.96 ppm (b′) and 1.72 ppm (b″) are assigned to the 2-methyl protons of the open-ring form unit and the closed-ring form unit, respectively. The signals at 2.05 ppm (a′) and 1.79 ppm (a″) are assigned to the methyl protons of the **DTT**. The ratio of the signals at 1.96, 1.72, 2.05, and 1.79 ppm is 1:1:1:1. So, the formation **2b** can be verified by this ^{1}H NMR data.

Fig. 3 shows absorption spectra of **1a**, **1b**, **2a**, and **2b**. As diarylethenes **1a** and **2a** possess similar molecular structures, it is interesting to compare their absorption bands. The absorption maxima of the open isomers **1a** and **2a** are at 386 and 388 nm. The absorption maximum of closed-ring isomer **2b** (508 nm) showed red shift about 40 nm in comparison with **1b** (468 nm) because of the conjugation extending induced by the introduction of the phenyl group.

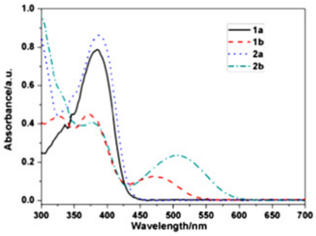
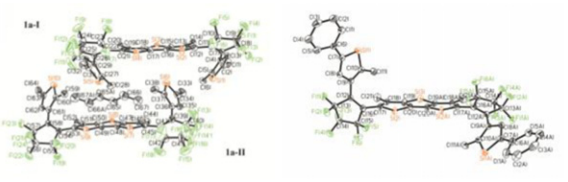


Fig. 3. Absorption spectrum of **1a** (solid line), **1b** (dash line), **2a** (dot line), **2b** (dash dot line).

2.3. X-ray crystallographic analysis

Crystals suitable for single-crystal X-ray diffraction analysis were obtained by slow evaporation of the solution of corresponding compounds in hexane at room temperature. The X-ray crystallographic analysis data are collected in Table S1. The ORTEP drawing of the molecule structures in single crystals of **1a** and **2a** are shown in Fig. 4 and their packing diagrams are shown in Fig. S5.



(a) (b)

Fig. 4. ORTEP drawing of crystals **1a** (a) and **2a** (b), showing 50% probability displacement ellipsoids.

It can be observed that compounds **1a** and **2a** adopted photoactive antiparallel conformation in the crystalline phase. For **1a**, there were two independent molecules in the nonsymmetrical unit and all of them were in photoactive antiparallel conformation. The distance between the photoactive carbons were 3.464 Å (C5···C13) and 3.593 Å (C19···C29) in molecule **1a-I**, and 3.550 Å (C37···C45) and 3.535 Å (C51···C59) in molecule **1a-II**. For diarylethene **2a**, the distance between the photoactive carbons were 3.616 Å (C10···C18) and 3.616 Å (C10A···C18A), respectively. These values are all less than 4.2 Å the distances required for a photochromic reaction in the solid state.[21]

In fact, the crystals **1a** and **2a** showed good photochromism in accordance with the expected analysis. Their color changes upon photoirradiation are shown in Fig. 5. For diarylethene dimer, in most cases the intramolecular excited energy transfer from the open-form unit to the closed-form one prohibits the formation of the two closed-ring form dimer. Crystals **1a** and **2a** could only take place at one of the diarylethene units even upon prolonged irradiation and this could be confirmed by the HPLC chromatograms (Figs. S2 and S4). Upon irradiation with 380 nm light, the pale-yellow crystals of **1a** and **2a** turned orange quickly, as a result of the formation of the closed-ring isomers **1b** and **2b**. Alternatively, the orange crystals returned to pale-yellow and reproduced **1a** and **2a** upon irradiation with visible light (>500 nm). The photochromic conversion ratios of **1a** and **2a** crystals in the photostationary state under UV irradiation were 8.08% for **1a** to **1b** conversion and 15.01% for **2a** to **2b** as evaluated by HPLC (Figs. S2 and S4). Those crystals exhibited coloration/decoloration cycles by alternate irradiation with UV and visible light and their closed-ring forms **1b**/**2b** remained stable for more than four months in the dark at room temperature. This indicated that they could be potentially used for the construction of certain optoelectronic devices.[22,23]

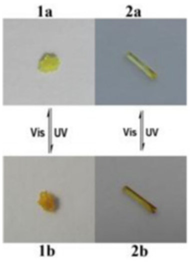


Fig. 5. Photographs of photochromic processes for diarylethenes **1a** and **2a** in the crystalline phase.

2.4. Electrochemical properties of diarylethenes 1a/1b and 2a/2b

Switching behaviors triggered by electrochemical reactions have been employed for the development of molecular switches. It has already been demonstrated that ring opening and closing reaction of diarylethenes could also be induced by electrochemically redox reactions. Therefore, it should be interesting to investigate the electrochemical behaviors of diarylethenes synthesized here. Fig. 6 shows the cyclic voltammetry (CV) curves of diarylethenes in acetonitrile. The oxidation onsets of the open-ring isomers **1a** and **2a** were at 1.61 and 1.58 V, and those of the closed-ring isomers **1b** and **2b** were at 1.13 and 1.10 V, respectively. The result showed that the oxidation process for the open-ring isomers **1a** and **2a** occurs at higher potentials than the corresponding closed-ring isomers **1b** and **2b**, indicating the extending of the π-conjugation in the closed-ring isomers. This is in accordance with the theory that the longer conjugation length of the closed-ring isomer generally leads to a less positive oxidation potential. Because the π-electrons delocalize throughout the two connected aryl moieties and extend to the substituents after cyclization reaction, the π-conjugation length of the closed-ring isomers **1b** and **2b** was much longer than those of their open-ring isomers **1a** and **2a**, resulting in the larger oxidation onsets. However, no ring closing or opening reaction was observed during the electrochemical characterizations.

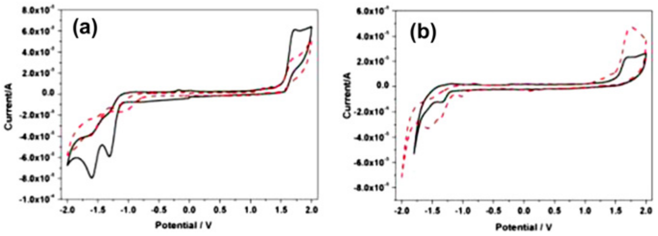


Fig. 6. Cyclic voltammetry of **1a** and **2a** (solid line) and **1b** and **2b** (dash line): (a) **1a**/**1b**, (b) **2a**/**2b**. Measurements were performed in acetonitrile (1.0×10^{-3} M) on a platinum electrode, scan rates were 50 mV/s.

The HOMO, LUMO energy levels derived from the CV data, and HOMO–LUMO energy gap (HLG) of each compound were summarized in Table 1. The data showed that the HLG of close-ring isomers **1b** and **2b** (2.02 and 2.03 eV) was smaller than those of their corresponding open-ring isomers **1a** and **2a** (2.82 and 2.83 eV). This was in well agreement with the bathochromic shift observed by UV–vis spectroscopy and also can be mainly ascribed to the extending of conjugation length for the closed-ring isomers **1b** and **2b** than that of the open-ring isomers **1a** and **2a**.

Table 1
Electrochemical properties of diarylethenes **1a**/**1b** and **2a**/**2b** in acetonitrile

Compounds	Oxidation		Reduction		HLG (eV)	Optical band gap (eV)
	E_{onset} (V)	HOMO (eV)	E_{onset} (V)	LUMO (eV)		
1a	1.61	−6.01	−1.21	−3.19	2.82	2.88
1b	1.13	−5.53	−0.90	−3.50	2.03	2.34
2a	1.58	−5.98	−1.25	−3.15	2.83	2.86
2b	1.10	−5.50	−0.92	−3.48	2.02	2.11

2.5. Theoretical investigations

To gain insight into nonsymmetrical structure and electronic absorption properties of diarylethenes **1a**/**1b** and **2a**/**2b**, we carried out molecular orbital calculation on the open- and closed-ring isomers of diarylethenes. The geometry optimization was calculated at the density functional theory (DFT) level using the B3LYP functional with 6-31G* basis sets. To reduce the computational cost, only the photoactive antiparallel conformation in the open-form was considered.

The HOMO–LUMO gaps (HLG) of these isomers were summarized in Table S2. The HLG of the open-ring isomers **1a** and **2a** was 3.37 and 3.30 eV, while a significant decrease in energy gap is found in the corresponding closed-ring isomers **1b** and **2b** (3.01 and 2.77 eV). This was in agreement with data extracted from the electrochemical studies.

Fig. 7 illustrates the calculated HOMO and LUMO of the geometry optimized structures in the ground states for the diaryelthenes **1a**/**1b** and **2a**/**2b**. The electron densities of both HOMO and LUMO with ring-opening isomers are different from those with closed-ring isomers. For open-ring isomers **1a** and **2a**, the HOMO orbital distribution of the electron density is mainly composed of the π orbital of **DTT** core and spread partially on the peripheral thiophene units. For closed-ring isomers **1b** and **2b**, the electron densities of HOMO and LUMO both localize on the one side of the closed diarylethene units.

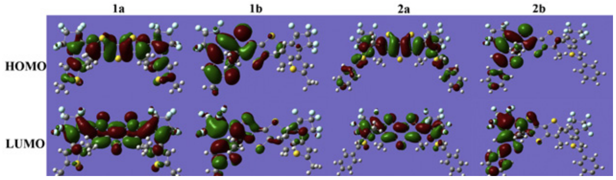


Fig. 7. Frontier molecular orbital densities for diarylethenes **1a**/**1b** and **2a**/**2b** at the B3LYP/6-31G* level of theory.

3. Conclusion

In conclusion, a new class of photochromic diarylethene dimer with a **DTT** linkage **1a** and **2a** were designed and synthesized. Their electronic structures and computational studies have been carried out. Their photochromic behavior was examined in solution as well as in the crystalline phase. However, only the isomer with the diarylethene units one in open-form and one in closed-form **1b**/**2b** was produced upon irradiation with ultraviolet light because of the intramolecular excitation energy transfer between the closed-ring and open-ring dithienylethene unit. The molecular structures of isomers **1b** and **2b** were confirmed by absorption spectra and ^{1}H NMR spectroscopy. In the photostationary state, the conversion ratios for **1a** to **1b** and **2a** to **2b** were 49.01% and 87.91% in solution under irradiation with a light of 380 nm, which are higher than that (8.08% for **1a** to **1b** and 15.01% for **2a** to **2b**) in the crystalline phase. Although similar compounds have been reported previously, no photochromic reaction in the solid state has been studied for such molecules. It should be useful for the development of molecular materials based on **DTT** units. Further investigations on their possible applications are in progress.

4. Experiment section

4.1. General

THF was distilled over sodium benzophenone ketyl before use. All other reagents and solvents were purchased from commercial suppliers and used directly as received. ^{1}H NMR and ^{13}C NMR spectra were performed in $CDCl_3$ with TMS as internal standard on Bruker Advance 400 MHz spectrometers. MS (EI) measurements were performed on SHIMADZU GCMSQP2010 or UK GCT-Micromass spectrometers. MALDI-TOF mass spectrometric measurements were performed with a Bruker Biflex III 45 MALDI-TOF spectrometer. Elemental analyses were measured on a Carlo Erba 1106 elemental analyzer. HRMS measurements were performed on a Bruker APEXIIFT-ICRMS spectrometer. UV–vis spectra were recorded on a JASCO V-570 spectrometer. Photoirradiation was carried out using a PLS-SXE300UV xenon arc lamp (Beijing Trust-tech Co. Ltd). Appropriate wavelength was isolated by passing through a bandfilter.

4.2. (2,5-Dimethyl-3-thienyl)perfluorocyclopentene (3a)

n-BuLi (4.2 mL, 2.5 M solution in hexanes) was added dropwise to a stirred solution of 3-iodo-2,5-dimethylthiophene (2.38 g, 10.0 mmol) in THF at −78 °C under nitrogen atmosphere. After stirring at this low temperature for 2 h, perfluorocyclopentene (1.43 mL, 10.5 mmol) was added to the reaction mixture at −78 °C, and the mixture was stirred for another 2 h at this temperature. The reaction mixture was warmed to room temperature spontaneously and then stopped by the addition of water. The product was extracted with ether. The organic layer was washed with water and dried over magnesium sulfate, filtrated, and evaporated. Compound **3a** was obtained after purification by column chromatography using silica (hexane) (2.23 g, 73.4%) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 6.73 (s, 1H), 2.43 (s, 3H), 2.39(s, 3H). HRMS (*m*/*z*): calcd for $C_{11}H_7F_7S$: 304.0157, found: 304.0162.

4.3. (2-Methyl-5-phenyl-3-thienyl)perfluorocyclopentene (3b)

*Compound **3b*** was prepared by a method similar to that used of **3a**. The crude product was purified by column chromatography using silica (hexane) to afford 4.91 g (71.5%) of compound **3a** as white powder. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.51 (d, *J*=7.2 Hz, 2H), 7.38 (t, *J*=7.3 Hz, 2H), 7.27 (t, *J*=8.0 Hz, 1H), 7.10(s, 1H), 2.42(s, 3H). Anal. Calcd for $C_{16}H_9F_7S$: C, 52.46; H, 2.48. Found: C, 52.58; H, 2.60.

4.4. 2,6-Bis[1-(2,5-dimethyl-3-thienyl)-2-perfluorocyclopentene]-3,5-dimethyldithieno[3,2-*b*:2′3′-*d*]thiophene (1a)

n-BuLi (2.5 M solution in hexanes, 0.176 mL) was added dropwise to a stirred solution of 3,5-dimethyldithieno[3,2-*b*:2′,3′-*d*]thiophene (0.0472 g; 0.21 mmol) in THF at −78 °C under nitrogen atmosphere. Stirring was continued for 2 h at the low temperature. Compound **3a** (0.134 g; 0.44 mmol) in THF was slowly added to the reaction mixture at −78 °C and the mixture was stirred for 2 h at this low temperature. Then, the reaction mixture was quenched with water. The product was extracted with ether. The organic layer was washed with water, dried with anhydrous magnesium sulfate, and concentrated under reduced pressure. The crude product was purified by column chromatography using silica (hexane) to afford 0.101 g (60 %) compound **1a** as yellow powder. Mp: 138–139 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 6.75(s, 2H), 2.43 (s, 6H), 1.89 (d, *J*=4.2 Hz, 12H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 144.1, 140.5, 138.4, 133.8, 132.2, 124.9, 124.5, 15.31, 14.6, 14.5; MS (*m*/*z*): calcd 792.8, found: 792.7. Anal. Calcd for $C_{32}H_{20}F_{12}S_5$: C, 48.48; H, 2.54. Found: C, 48.65; H, 2.81.

4.5. 2,6-Bis[1-(2-methyl-5-phenyl-3-thienyl)-2-perfluorocyclopentenyl]-3,5-dimethyldithieno[3,2-*b*:2′3′-*d*]thiophene (2a)

Compound ***2a*** was prepared by a method similar to that used for **1a**. The crude product was purified by column chromatography using silica (hexane) to afford 0.089 g (63%) of compound **2a** as yellow powder. Mp: 191–192 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.54 (d, J=7.4 Hz, 4H), 7.37–7.39 (t, J=7.5 Hz, 4H), 7.32–7.33 (t, J=8.9 Hz, 4H), 1.99(s, 6H), 1.93 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 144.2, 142.8, 141.9, 134.0, 133.3, 132.4, 129.2, 128.2, 125.8, 124.8, 122.6, 14.8, 14.6; MS (m/z): calcd 916.9, found: 916.1. Anal. Calcd for $C_{42}H_{24}F_{12}S_5$: C, 55.01; H, 2.64. Found: C, 55.16; H, 2.93.

CCDC-884600 (**1a**) and CCDC-884601 (**2a**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgements

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Supplementary data

These data include HPLC chromatograms for compounds **1a** and **2a** in solution and in the crystalline phase, packing view of crystals **1a** and **2a**, the calculated energies of frontier orbitals, HOMO–LUMO gap (HLG) of diarylethenes **1a**/**1b** and **2a**/**2b**. Supplementary data associated with this article can be found in the online version, at http://dx.doi.org/10.1016/j.tet.2012.08.026. These data include MOL files and InChiKeys of the most important compounds described in this article.

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