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Synthesis, photochromic mechanism and properties of a novel biindenylidenedione compound containing ferrocene units

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A novel biindenylidenedione compound, [2,2′-bi-1*H*-indene]-3,3′-bis [(4-oxo-4-ferrocenylbutanoyloxy)propyl]-3,3′-dihydroxy-1,1′-dione was synthesized and characterized fully by ^{1}H NMR, IR, MS and elemental analysis. The photochromic and photo-induced radical properties were investigated by means of UV–vis and electron spin resonance spectroscopy, respectively. The results show that the title compound simultaneously exhibits photochromism in the solid state as well as generating stable free radicals under irradiation with 330 nm light. This fact provides evidence for the photochromic mechanism of this kind of compounds.

Keywords: photochromism; biindenylidenedione; ferrocene; free radical; mechanism

Introduction

Photochromic materials have attracted a great deal of interest due to their potential application in optical memories and switching devices.[1,2] To date, many types of photochromic compounds have been reported. Among them, the biindenylidene derivatives first investigated by Toda *et al*. in 1992 are unusual in that they simultaneously exhibit photochromism in the solid state as well as the generation of stable radicals.[3] Toda also reported that the *trans-syn*-3,3′-diaryl-2,2′- biindenylidene-1,1′-diones undergo photochromism based on the formation of a triplet biradical.[4] In recent years, we have extensively studied the relationship between the photochromic properties and structures of the 3,3′-diaryl/alkyl-3,3′-dihydroxyl- [2,2′-bi-1*H*-indene]-1,1′-diones.[5–10] Our results suggest that their ESR signal intensity shows characteristic temperature dependence: it decays with the decrease of temperature, and disappears completely on cooling down to liquid nitrogen temperature; the signal is regenerated gradually with the increase of temperature, and is restored to its original shape at room temperature.[6,8] The observation above reveals that the biradical derived from the irradiation of these types of compounds is not a triplet biradical but a singlet biradical.[6] This conclusion demonstrates their photo-induced radical behaviors and provides a better understanding of the observed photochemical outcome.

Ferrocenes have been used with great success in many areas of material and medicinal chemistry.[11–14] They are characterized by remarkably robust stability, possessing electron transfer properties and ease of chemical modification.[15] Ferrocene moieties were also appended on diverse photochromic compounds such as spironaphthoxazine,[16] tetraazaporphyrin,[17] spiropyran,[18] ethynylethene[19] and dithienylmaleimide.[20] The incorporation of the ferrocene moiety greatly improves the thermal stability of the photochromic states; it can also adjust the fluorescent, electrochemical and magnetic properties of photochromic materials. In addition, ferrocene moieties have a low-lying triplet excited state and are known to be effective triplet quenchers.[21–23] In this paper, two ferrocene units were appended to the biindenylidenedione backbone to obtain a new photochromic compound. The synthetic route is shown in Scheme 1. The effect of the ferrocene units on the photochromic properties and photo-induced radical behavior were investigated and the photochromic mechanism is discussed.

Experimental

Materials

2,2′-Biindanylidene-1,1′,3,3′-tetraone[24] and β-ferrocenoylpropionic acid[25] were prepared according to the literature procedures. All other chemicals were of analytical grade and used as purchased from Aldrich. Benzene and THF were distilled from sodium–benzophenone. Dichloromethane used for photophysical measures was dried with calcium chloride and distilled from calcium hydride prior to use.[26]

Melting point measurements

Melting points were measured on a X4 Micro-melting point apparatus without correction.

NMR analyses

^{1}H NMR and ^{13}C NMR spectra were performed in $CDCl_3$ and recorded on a Bruker Avance 300 at 300 and 75 MHz, respectively. ^{1}H NMR spectra were collected at 300 MHz using a 6000 Hz spectral width, a relaxation delay of 3.5 s, 64 K data points, a pulse width of 90° and $CHCl_3$ (7.26 ppm) as the internal reference.

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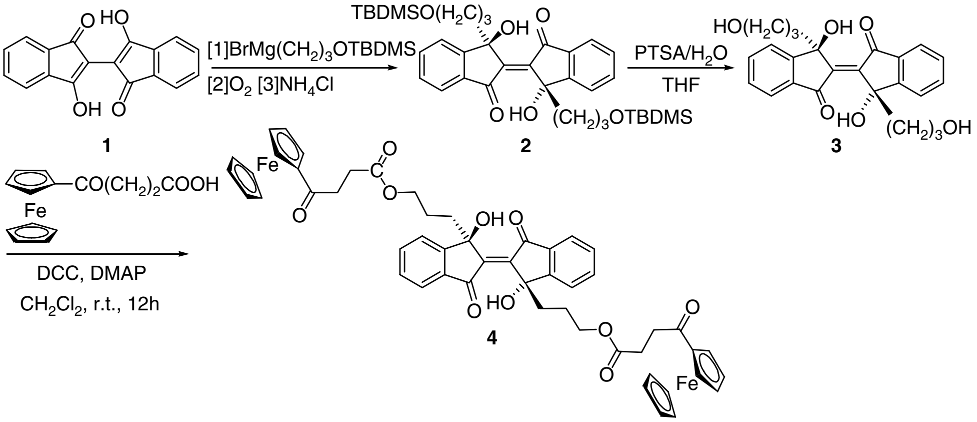


Scheme 1. The synthetic route of compound **4**.

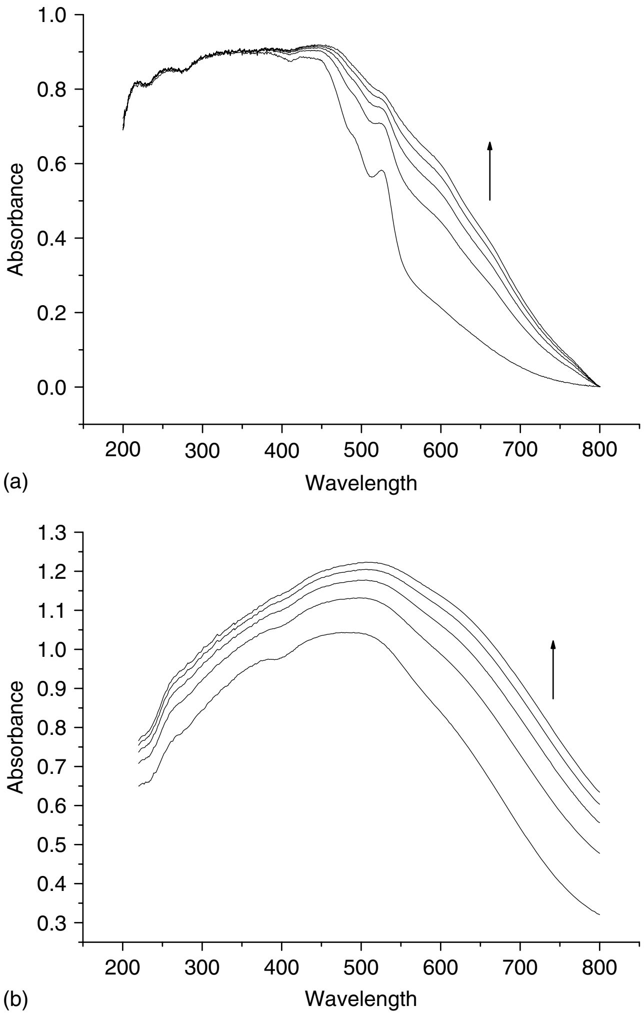


Figure 1. UV–vis spectral changes of compounds **3** and **4** in the solid state on irradiation. Measurement was made every 3.75 min from the bottom to the top. (a) Compound **3**; (b) compound **4**.

IR analyses

Infrared spectral analysis was performed on a Jasco FT/IR-620v spectrophotometer using KBr pellet. Here, the concentration of the samples in KBr was 5% by weight. Measurement conditions: resolution 4 cm^{-1}; scanning times 20; spectral range 4000–400 cm^{-1}.

MS analyses

The mass spectra were recorded on Thermo Finnigan LCQ Advantage spectrometer in ESI mode-I. Measurement conditions: spray voltage, 4.55 kV; sheath gas flow rate, 16.60 L/min; capillary voltage, 44.79 V; capillary temperature, 250 °C.

Elemental analyses

All elemental analysis were performed on a Yamaco CHN corder MT-3 apparatus equipped with two heaters: heater 1 (combustion tube), CHN operation, 950 °C (maximum is 1000 °C); heater 2 (reduction tube), CHN operation, 500 °C (maximum is 550 °C). The reduction performance decreases at higher temperatures.

UV–vis spectroscopic measurements

UV–vis spectra were recorded on a Jasco V-570 UV–vis–nIR spectrophotometer equipped with an integrating sphere. Spectra were measured from thick layers (3 mm) of each sample pressed against a suitable holder. Spectra were acquired using the commercial instrumental setup.

UV irradiation

The samples for photochromism observation were UV irradiated with a 200 W high-pressure mercury lamp equipped with a water filter and a set of optical filters transmitting the 330 nm spectral line only. The irradiation lasted for 15 min at room temperature.

ESR

The irradiated samples (about 2 mg for each sample) were powdered and sealed with a torch into melting point capillaries which were housed within a quartz ESR tube for the measurements. ESR spectra were taken at X-band with a Bruker EMX-6/1 EPR spectrometer. A program within Bruker's software tuned the bridge, obtained a spectrum, and wrote it to the hard disk. This process was repeated five times in a row for each sample. Measurement conditions: microwave power 0.2 mW, sweep width 150.0 G, time constant 10 ms, and sweep time 84 s.

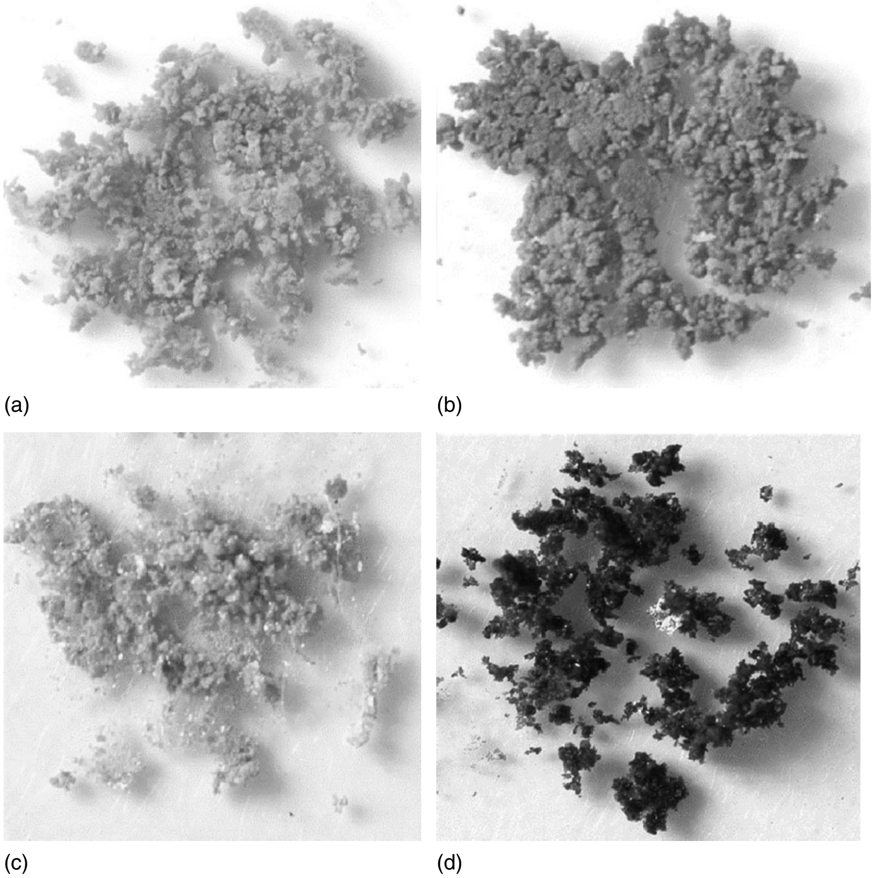


Figure 2. The color changes of compounds **3** and **4** before and after irradiation in the solid state. (a) Compound **3**, before irradiation; (b) compound **3**, after irradiation for 15 min; (c) compound **4**, before irradiation; (d) compound **4**, after irradiation for 15 min.

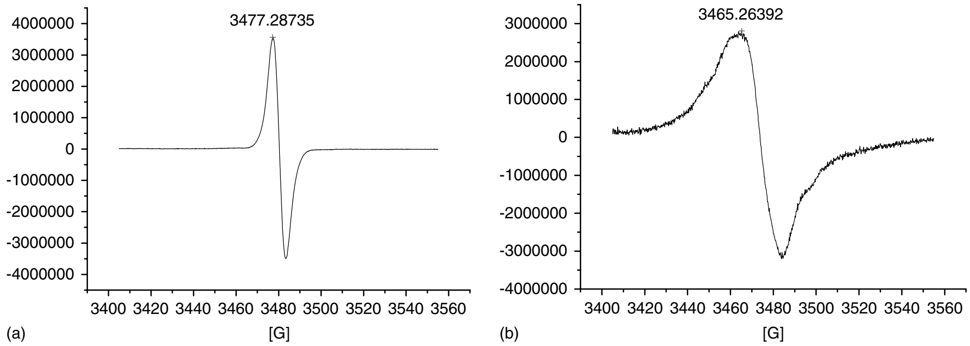


Figure 3. ESR spectra of compounds **3** and **4** upon irradiation in the solid state at room temperature. (a) Compound **3**; (b) compound **4**. Measurement conditions: for compound **3**, center field, 3480.0 G; sweep width, 150.0 G; modulation, 100.0 kHz; amplitude, 6.0 G. For compound **4**, center field, 3480.0 G; sweep width, 150.0 G; modulation, 100.0 kHz; amplitude, 10.0 G.

Preparation of [2,2′-bi-1H-indene]-3,3′-di[(tert-butyldimethylsiloxy) propyl]-3,3′-dihydroxy-1,1′-dione (2)

To a three-necked 250 ml round-bottom flask equipped with magnetic stirring bar was added 1.8 g of magnesium turning and 10 ml of anhydrous THF under N_2. To the as-formed mixture, 1-bromo-3-(*tert*-butyldimethylsiloxy)propane (13 g, 51 mmol) in THF (50 ml) was added dropwise over the course of 20 min. A water bath was used to control the exothermic reaction. Then the reaction mixture was stirred at room temperature for another 1 h. A suspension of **1** (1.9 g, 6.5 mmol) in dry benzene (40 ml) was added dropwise to the resulting Grignard reagent over a period of 20 min. The dark green reaction mixture was stirred vigorously for 24 h under nitrogen atmosphere, and then exposed to the air for an additional 3 h. Finally, the reaction mixture became a dark purple color, and was quenched with saturated NH_4Cl (2.5 g) aqueous solution. The organic phase was collected and dried over anhydrous $MgSO_4$. After filtration and evaporation of solvent (50 ml) on a rotary evaporator, the residue was eluted with petroleum ether–ethyl acetate (8:1) through a silica gel column (2 × 30 cm) to afford product **2** as yellow plates (0.68 g, 31%); m.p. 173–175 °C. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.07 [s, 12H, $-Si(CH_3)_2$], 0.90 [s, 18H, $-C(CH_3)_3$], 1.56 (m, 4H, $-CH_2CH_2CH_2O-$),

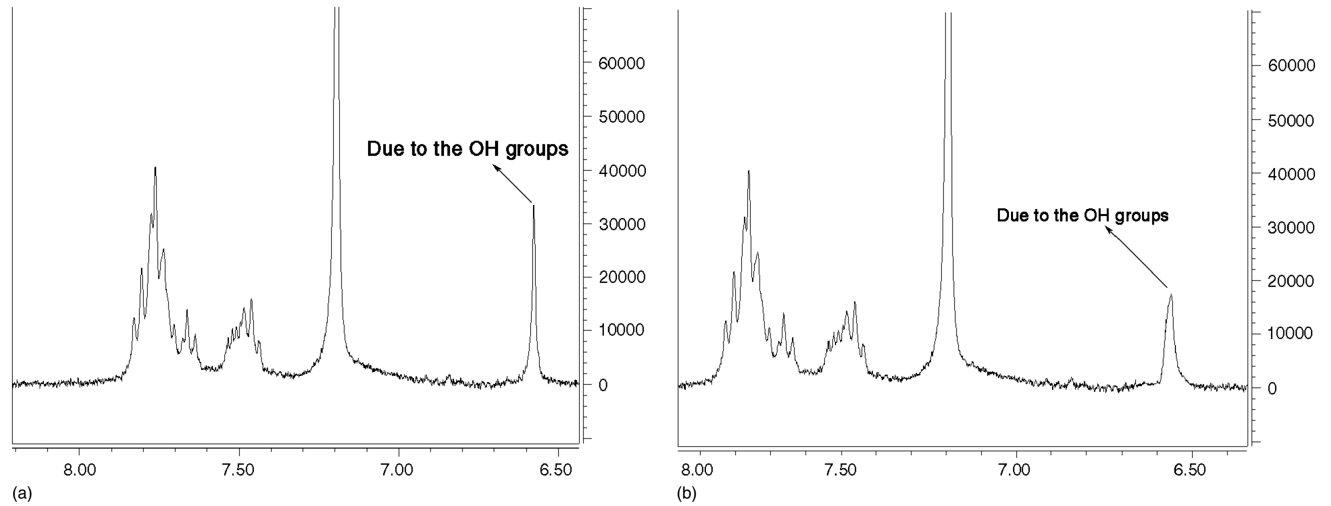
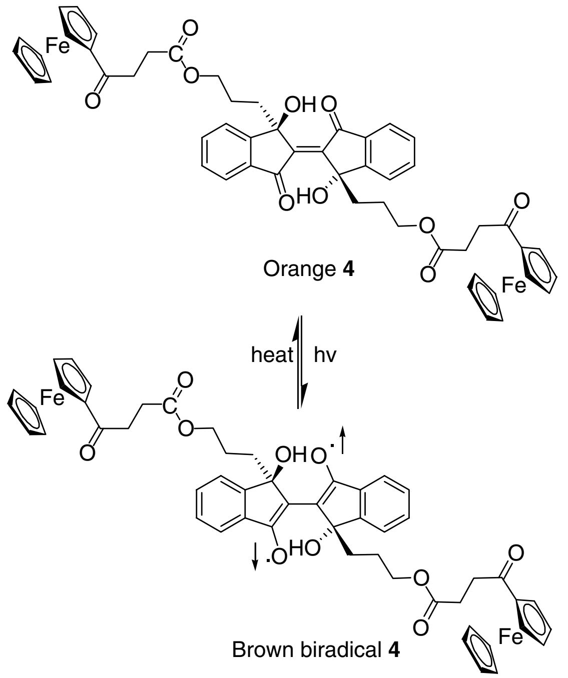


Figure 4. ^{1}H NMR spectra of **4**. (a) ^{1}H NMR spectra of **4**, before irradiation (b) ^{1}H NMR spectra of **4**, after irradiation.



Scheme 2. The photochromic mechanism of compound **4**.

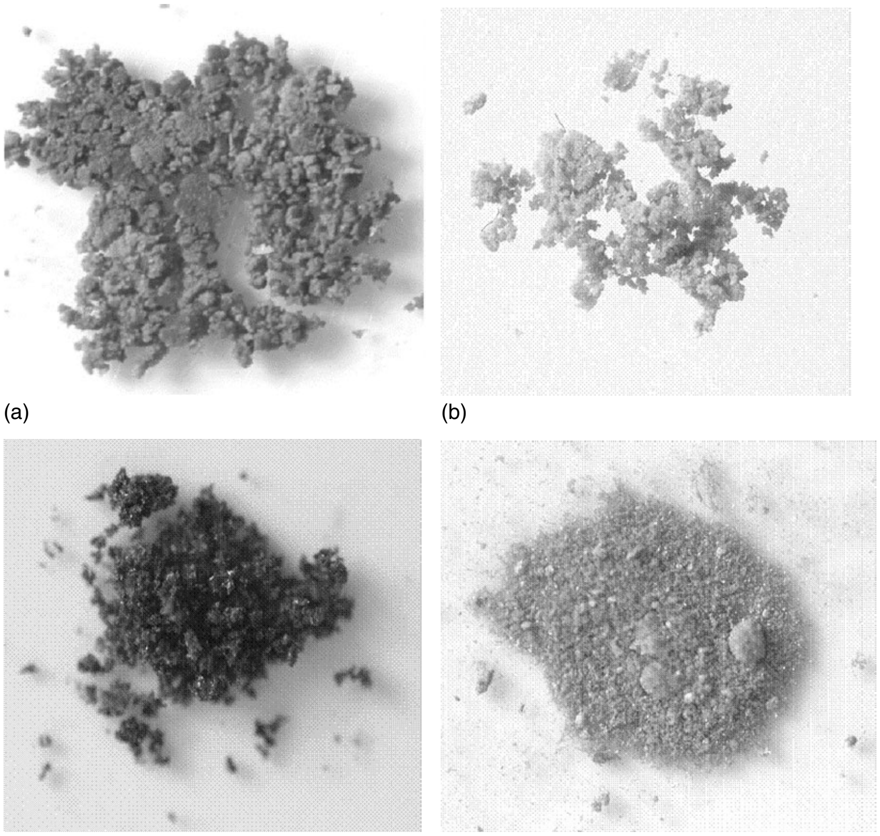
2.17 (t, 4H, $-CH_2CH_2CH_2O-$), 3.37 (t, 4H, $-CH_2CH_2CH_2O-$), 6.65 (s, 2H, $-OH$), 7.54 (m, 2H, $-Ar$), 7.81 (m, 6H, $-Ar$). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 195.6, 153.9, 149.5, 136.4, 128.5, 123.8, 122.7, 77.6, 76.4, 76.0, 75.6, 62.2, 61.4, 36.2, 31.9, 27.4, 24.8, 17.1, −6.4. MS (ESI, *m/z*): 637.34 $[M + 1]^+$. IR (cm^{-1}): 3396, 2952, 1660, 1649. Anal. calcd for $C_{36}H_{52}O_6Si_2$: C, 67.88, H, 8.23. Found: C, 67.85, H, 8.26.

Preparation of [2,2′-bi-1H-indene]-3,3′-bis(3-hydroxypropyl)-3,3′-dihydroxy-1,1′-dione (3)

To a 250 ml round-bottom flask equipped with magnetic stirring bar was added **2** (0.64 g, 1 mmol), PTSA (*p*-toluenesulfonic acid, *ca* 30 mg), water (2 ml), and THF (40 ml). The solution was stirred at room temperature for 12 h, when TLC showed no starting material **2** remaining. Water (10 ml) was added, and the resulting mixture was extracted with CH_2Cl_2 (2 × 60 ml). The organic extracts were combined and dried over $MgSO_4$. After filtration and evaporation on a rotary evaporator, the crude product was purified by column chromatography (1 × 25 cm column, 35% petroleum ether in ethyl acetate) to give a yellow powder **3** (0.39 g, 95%); m.p. 203–206 °C. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.59 (m, 4H, $-CH_2CH_2CH_2O-$), 2.17 (t, 4H, $-CH_2CH_2CH_2O-$), 3.37 (t, 4H, $-CH_2CH_2CH_2O-$), 6.67 (s, 2H, $-OH$), 7.53 (m, 2H, $-Ar$), 7.82 (m, 6H, $-Ar$). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 195.5, 153.5, 149.4, 136.6, 128.8, 123.9, 122.9, 77.6, 76.4, 76.0, 75.6, 61.3, 36.1, 29.3, 28.7, 27.3, 24.6. MS (ESI, *m/z*): 409.37 $[M + 1]^+$. IR (cm^{-1}): 3456, 2962, 1663, 1601. Anal. calcd for $C_{24}H_{24}O_6$: C, 70.57, H, 5.92. Found: C, 70.55, H, 5.98.

Preparation of [2,2′-bi-1H-indene]-3,3′-bis[(4-oxo-4-ferrocenyl butanoyloxy)propyl]-3,3′-dihydroxy-1,1′-dione (4)

To a 100 ml round-bottom flask equipped with a magnetic stirring bar was added **3** (102 mg, 0.25 mmol), β-ferrocenoylpropionic acid (157 mg, 0.55 mmol), *N,N*′-dicyclohexylcarbodiimide (113 mg, 0.55 mmol), 4-dimethylaminopyridine (34 mg, 0.275 mmol) and CH_2Cl_2 (40 ml). The solution was stirred under N_2 at room temperature for 12 h, and was then filtered through a pad of Celite to remove *N,N*-dicyclohexylurea. The filtrate was concentrated under reduced pressure on a rotary evaporator and the residue was chromatographed on a silica gel column (2 × 30 cm) with petroleum ether–ethyl acetate (2 : 1) as eluant. The pure product **4** was collected from the second fraction as bright orange solid. Yield: 661 mg (70%). m.p. 174–176 °C. ^{1}H NMR ($CDCl_3$, 300MHz): δ 1.85 (m, 4H, $-CH_2CH_2CH_2O-$), 2.47 (t, 4H, $-CH_2CH_2CH_2O-$) 2.89 (t, 4H, $-CH_2CH_2CH_2O-$), 3.84 (t, 4H, $-OCOCH_2CH_2CO-$), 4.00 (t, 4H, $-OCOCH_2CH_2CO-$), 4.14 (s, 10H, Fc−H), 4.42 (t, 4H, Fc−H), 4.70 (t, 4H, Fc−H), 6.58 (s, 2H, $-OH$), 7.46 (m, 2H, $-Ar$), 7.76 (m, 6H, $-Ar$). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 200.9, 195.4, 171.8, 153.5, 149.4, 136.7, 136.2, 128.9, 123.9, 122.9, 77.4, 77.3, 76.4, 76.0, 75.6, 71.1, 68.9, 68.2, 62.9, 36.0 33.1, 26.8, 23.3. MS (ESI, *m/z*): 967.34 $[M + Na]$. IR (cm^{-1}): 3332, 2959, 1731, 1630. Anal. calcd for $C_{52}H_{48}Fe_2O_{10}$: C, 66.12, H, 5.12. Found: C, 66.19, H, 5.11.



(a) (b) (c) (d)

Figure 5. The color changes of irradiated **3** and **4** before and after heating in the solid state. (a) Irradiated **3**, before heating; (b) irradiated **3**, after heating for 10 min; (c) irradiated **4**, before heating; (d) irradiated **4**, after heating for 10 min.

Results and Discussion

Synthesis

The synthesis of the biindenylidenedione compound **2** was first attempted by the reaction of 2,2′-biindanylidene-1,1′,3,3′-tetraone with the Grignard reagent derived from THP-protected 3-bromo-1-propanol, but failed because the addition reaction produced at least four different products, which were inseparable by means of recrystallization and column chromatography. Subsequently, the use of *tert*-butyldimethylsilyl (TBDMS) as protecting group instead of THP successfully gave the desired **2** in a 31% yield from the otherwise same addition reaction. Under mild conditions, **2** was converted into compound **3** by treatment with PTSA in the solution of THF–H_2O (20: 1, v/v) at room temperature in good yield. The title compound **4** was then readily prepared by the esterification reaction using *N,N*′- dicyclohexylcarbodiimide and 4-dimethylaminopyridine as a catalyst.

Photochromic properties of compounds 3 and 4

In order to evaluate the effect of the ferrocene groups on the photochromic properties of **4**, the intermediate compound **3** was also selected for the purpose of comparison. As expected, compound **3** undergoes photochromism in the solid state upon irradiation with 330 nm light. Its photo-color reaction was monitored by UV–vis absorption [Fig. 1(a)], which clearly indicates that compound **3** has an apparent increase in UV absorption in the wavelength range of 400–800 nm after UV light irradiation. The photochromic properties of compound **4** were also tested by means of UV–vis spectroscopy, and the results are shown in Fig. 1(b). By comparison of Fig. 1(b) with Fig. 1(a), the major differences in the UV absorptions of **3** and **4** are as follows: first, the increase in UV absorption range (200–800 nm) of compound **4** is wider than that of compound **3**; second, the intensity of the absorption change of **4** is also stronger than that of **3**; third, there is no isosbestic point observed because the absorbance increases in the whole spectral region in Fig. 1(b). These results indicate that the incorporation of electron-rich ferrocene units into the biindenylidenediones may accordingly affect their electronic structures, as well as their UV spectra. From the viewpoint of the application to the high-density optical memory media, it is desired to develop photochromic compounds that have a light sensitivity in region between 407 and 532 nm.[27] As seen in Fig. 1(b), compound **4** exhibits the maximum absorption of photocolored isomers in the region of 450–550 nm. It might become a promising candidate for practical applications.

The changes in the absorption of **3** and **4** are accompanied by their color changes (Fig. 2), which are visible to the naked eye. Figure 2 clearly shows the obvious color changes after irradiation with UV light. It is worth noting that, either before or after irradiation, the color of compound **4** has a remarkable difference from those of compound **3**. The apparent color changes are from bright yellow to red for **3**, and from orange to deep red for **4**. The typical color change from **3** to **4** is characterized as the diminishing of the yellow color and increase in the orange color, which should be attributed to the ferrocene moieties incorporated into the

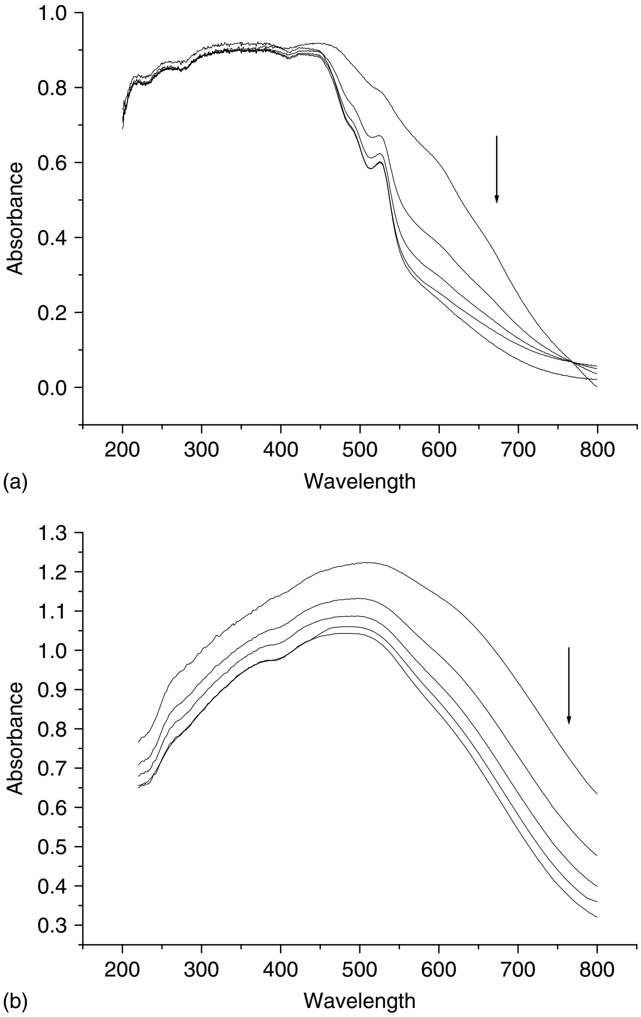


Figure 6. UV–vis spectral changes of irradiated **3** and **4** in the solid state upon heating at 70 °C. Measurement was made every 2.5 min from the top to the bottom. (a) Compound **3**; (b) compound **4**.

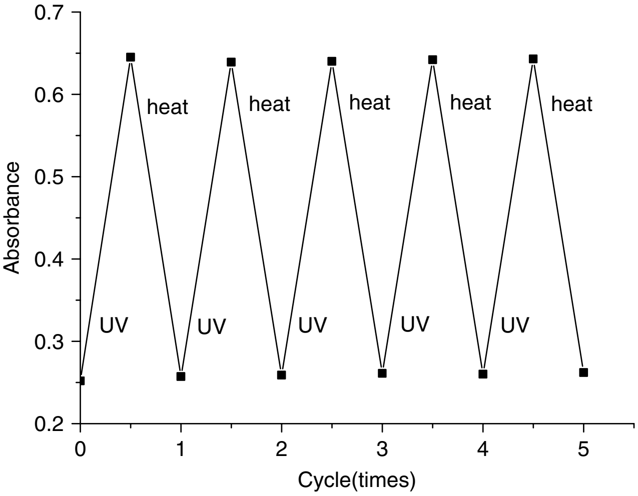


Figure 7. The reversible absorbance variation at 580 nm for compound **3** after alternating UV light irradiation (15 min) and heating (10 min).

biindenylidenedione. A difference in the color, especially in red intensity, is also observable for the irradiated samples **3** and **4**. The color differences between **3** and **4** indicate that the introduction of ferrocene groups on the side chains of biindenylidenediones significantly affects the photochromic properties of this class of compounds. These observations are in agreement with the fact that the ferrocene functions as an efficient sensitizer in photochemical reaction due to its high absorption of ultraviolet irradiation.[28]

The photo-induced radical behaviors of **3** and **4** were studied by ESR measurements in air at room temperature. The *g*-value was calculated by the known parameters of g_3 and g_4 of Mn-mark. Neither **3** nor **4** showed any ESR signals before irradiation; however, the corresponding irradiated samples of **3** and **4** gave distinctive ESR signals, as shown in Fig. 3. The characteristic values of ESR signals are as follows: **3** ($g = 2.006$, $\Delta Hpp = 25.3717$ G), **4** ($g = 2.001$, $\Delta Hpp = 100.6747$ G), respectively. These data indicate that the ferrocene groups incorporated into the biindenylidenediones have considerable influence on their photo-induced radical behavior. It is known that the ferrocene moiety has a low-lying triplet excited state and is an effective triplet quencher.[21–23] If the photo-induced biradical generated from the irradiation of compound **4** is a triplet, the biradical would be easily quenched by the excited ferrocene moieties. However, Fig. 3(b) clearly shows that the irradiated **4** exhibits a distinctive ESR signal. The biradical has not been quenched at all. A possible explanation for this observation is that the photo-induced biradical of **4** is a singlet one. The results obtained in this study are also consistent with the previously proposed photochromic mechanism for biindenylidenediones.[6] After photoirradiation, two unpaired radicals generate, then the π-conjugation extends throughout the whole molecule and the UV–vis absorption spectrum subsequently displays drastic changes [Fig. 1(b)]. The photo-induced singlet biradicals could be stabilized by the conjugation of the whole molecular system while the oxygen atoms of carbonyls contribute a lot to the spin density distribution owing to their electronegativity (Scheme 2). The change in ^{1}H NMR spectra of **4** (Fig. 4) also supports this claim. The peak at δ_H 6.58 ppm loses its sharpness after photoirradiation. This phenomenon is consistent with the decrease in the hydrogen-bonding strength because of the generation of a radical on oxygen atoms of carbonyls. The classical intermolecular hydrogen-bonding interactions between the hydroxy proton and the carbonyl oxygen in biindenylidenedione derivatives had been determined by X-ray analysis in our previous reports.[5–10] Unfortunately, despite numerous efforts, single crystals of **4** suitable for a structure determination by X-ray diffraction were not obtained.

It is worth noting that the photochromic biindenylidenedione derivatives are thermally labile. Their photochromic state may revert to the original state gradually in the dark or quickly upon heating under nitrogen atmosphere.[6–10] Hence, the photochromic process of the compounds in this work is reversible too. Figure 5 shows that the photochromic state of **3** and **4** can be turned back to the original state upon heating at 70 °C for 10 min under nitrogen atmosphere. The changes in UV absorption spectra of **3** and **4** during heating are shown in Fig. 6. Clearly, heating results in a return to the original absorption spectrum of irradiated samples of **3** and **4**, confirming the thermochromism of biindenylidenediones. The fatigue resistance of **3** and **4** was also investigated in this work. As seen from Fig. 7, the photochemical–thermal behavior of **3** is fairly reversible and little fatigue has been observed after five cycles. In comparison with

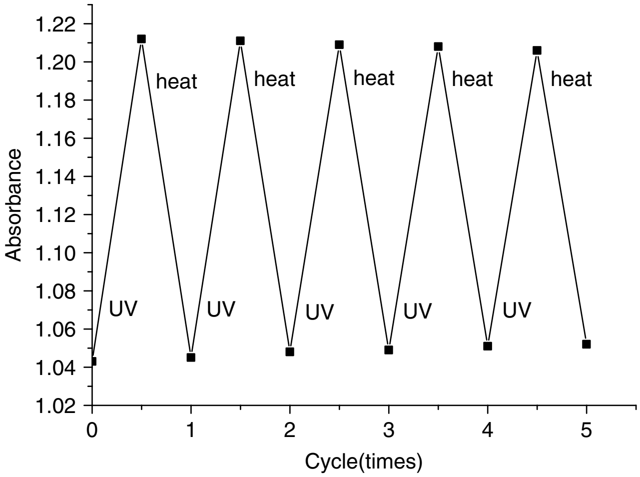


Figure 8. The reversible absorbance variation at 482 nm for compound **4** after alternating UV light irradiation (15 min) and heating (10 min).

3, the photochemical–thermal process of **4** exhibits little fatigue since the relative intensity of absorption at 482 nm decreases from 0.169 for the first cycle to 0.154 for the fifth cycle (Fig. 8).

Conclusions

A novel biindenylidenedione compound containing two ferrocene units was successfully prepared in high yield. It simultaneously exhibits photochromism in the solid state, as well as generating stable radicals after irradiation with 330 nm light. The free radical of the photochromic state for the title compound could not be quenched by the ferrocene groups. This fact provides solid evidence for the photochromic mechanism, and also suggests that the photo-induced radicals of biindenylidenediones, including those in this work, are not triplet, but singlet biradical.

Acknowledgments

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