

Synthesis and properties of novel photochromic biindenylidenedione derivative bearing TEMPO radical

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Abstract

Stable nitroxide radical TEMPO was introduced to the benzene rings of novel photochromic 7,7'-dimethyl-[2,2'-bi-1*H*-indene]-3,3'-diethyl-3,3'-dihydroxy-1,1'-dione (compound **1**), leading to the synthesis of novel multifunctional title compound **4**. The photochromic property, magnetic property and ESR spectroscopy of the title compound were investigated. Compound **4** possesses visible photochromism upon photoirradiation, and its ESR signal undergoes secular broadening after photoirradiation. The magnetic susceptibility measurement shows that the antiferromagnetic interaction for **4** becomes stronger after photoirradiation. In compound **4** there are two kinds of spin center after photoirradiation: one is TEMPO radical, and another is photo-generated radical of two indanone moieties. Our results demonstrated that the colour and magnetic property of compound **4** could be controlled by photoirradiation.

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Keywords: Dimethyl-[2,2'-bi-1*H*-indene]-diethyl-3,3'-dihydroxy-1,1'-dione; Synthesis; Photo-controlled magnetism; Photochromism; Organic solid material

1. Introduction

The development of organic multifunctional materials is one of the most attractive fields of chemistry and material science [1]. Nowadays considerable interest is focused on the development of multifunctional spin systems with synergetic properties, and successful studies have been carried out toward the photofunctional magnetic materials derived from some organic compounds [2–5]. Regarding organic photoresponsive spin system, Iwamura, Matsuda, et al. reported azobenzene derivatives carrying two nitronyl nitroxide radicals, in which UV and ESR changes upon irradiation were observed in solution [6]. Matsuda and Irie have succeeded in developing a fascinating series of diarylethene derivatives carrying two nitronyl nitroxides, in which a photo-switching behavior of the intramolecular magnetic interactions has been found under irradiation [7–16]. Nitroxide radicals have been most often used as spin sources in the development of molecular-based magnetic materials. Among them, TEMPO radical has been widely used due to its versatility and good

stability as building blocks [17–19]. As disclosed in recent reports from our group and others, the biindenylidene derivatives are a unique class of photochromic organic compounds, which undergo photochromism upon irradiation with light, and simultaneously generate radicals in the crystalline state as exemplified in Scheme 1 [20–25].

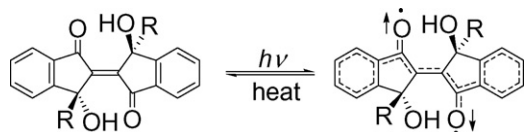
This promising property should be useful for the development of new photoswitching unit possessing inter- and/or intra-molecular magnetic interactions. It is already known that molecular magnetism can be photo-controlled by using spin crossover phenomena [26–29]. In addition to spin crossover systems, several other systems bearing photochromic units were also reported [30–33]. Herein, we have successfully synthesized a new photo-control unit for magnetic interaction, which is based on photochromic biindenylidenedione bearing TEMPO radical (compound **4**, Scheme 2).

The starting material **1** was prepared according to our recent reported literature [25], and its structure was confirmed by the X-ray crystallography (Fig. 1).¹

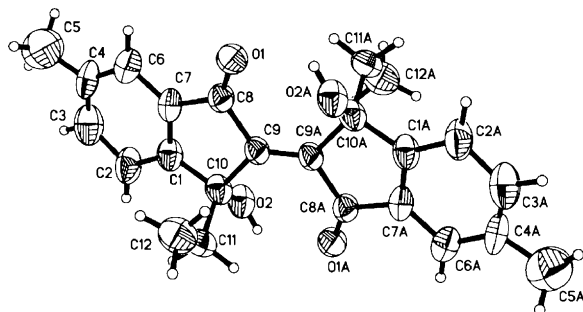
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¹ X-ray crystal and molecular structure analysis. Crystal data for **1**: empirical formula C₄₈H₄₈O₈, formula weight = 752.86, monoclinic, space group P2(1)/n, *a* = 9.577(5) Å, *b* = 7.548(4) Å, *c* = 14.708(8) Å, α = 90°, β = 107.290(9)°, γ = 90°.



Scheme 1. Photochromism of biindenylidenedione derivatives.

Fig. 1. Molecular structure of **1**.

The title compound **4** was synthesized via three steps reaction, starting from compound **1**. In compound **4**, stable nitroxide radical (TEMPO) moiety is incorporated photochromic biindenylidene moiety, i.e. compound **4** is a new multi-functional compound, which does not only possess photochromic and photomagnetic properties, but also has photomagnetic moiety and magnetic moiety of TEMPO radical of possible interaction under photoirradiation. Herein, we report the results of our investigation.

2. Experimental

2.1. Materials and instruments

All chemicals were purchased from commercial sources, and solvents were dried by refluxing over an appropriate drying agent and distilled prior to use. Melting points were determined with Yanagimoto MP-35 melting point apparatus and uncorrected. ^1H NMR spectra were recorded on BRUKER AC-P300. UV–vis spectra were recorded on Shimadzu V-2101PC

$\gamma = 90^\circ$, $V = 1015.2(9) \text{ \AA}^3$, $Z = 2$, $d(\text{calc}) = 1.231 \text{ g cm}^{-3}$, reflection collected/unique 4774/1790 [$R(\text{int}) = 0.0720$], final R indices [$I > 2\sigma(I)$] $R_1 = 0.144$, $wR_2 = 0.3816$, R indices (all data) $R_1 = 0.2355$, $wR = 0.4357$, GOF = 1.275, CCDC 627738.

UV–vis spectrophotometer. The mass spectra were recorded on Thermo Finnigan LCQ advantage spectrometer in ESI mode-I with spray voltage 4.8 kV. Infrared spectra were recorded on a NICOLET-380-FT-IR spectrophotometer. A Yamaco CHN CORDER MT-3 apparatus was used for elemental analysis. The X-ray diffraction data were collected using Mo $K\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$) on a BRUKER SMART 1000 diffractometer. The powder ESR spectra were recorded using a BRUKER A-320 EPR spectrometer. Variable-temperature magnetic susceptibilities were measured on a MPMS XL-7 SQUID magnetometer in the temperature range 2–300 K, with an applied field of 2000 G.

2.2. Synthesis

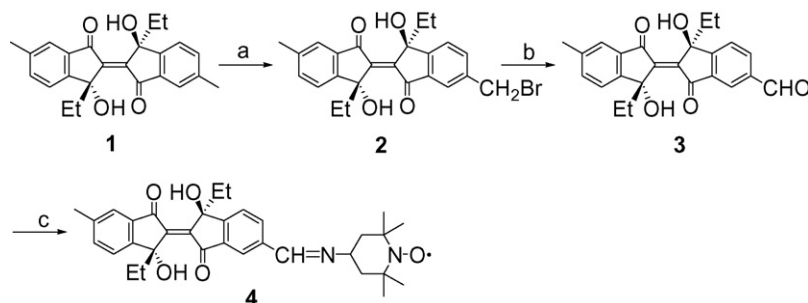
Synthesis of the title compound **4** was successfully carried out as shown in Scheme 2.

2.2.1. 7-Bromomethyl-7'-methyl-[2,2'-bi-1H-indene]-3,3'-diethyl-3,3'-dihydroxy-1,1'-dione (**2**)

Compound **1** (1.000 g, 2.66 mmol) and NBS (1.000 g, 5.62 mmol) were added 80 mL dry benzene, and then the reaction mixture was heated to reflux for 12 h under irradiation by light with stirring. The solvent was then removed under reduced pressure, the solid residue was obtained, which purification was performed by column chromatography (silica, hexane/ether (v/v) = 4:1). **2** was obtained as yellow solid (0.592 g, 49%): Mp 145–146 $^\circ\text{C}$; ^1H NMR (CDCl_3 , 300 MHz): δ 0.55–0.60 (t, $J = 7.8 \text{ Hz}$, 6H), 2.16–2.30 (m, 4H), 2.49 (s, 3H), 4.56 (s, 2H), 6.52 (br s, 1H), 6.67 (br s, 1H), 7.66–7.86 (m, 6H). MS (ESI): (m/z) 455 (M^+). IR (KBr) (cm^{-1}): 3368, 2960, 2926, 1668, 1601, 1400, 1283, 1160. Anal. Calcd for $\text{C}_{24}\text{H}_{23}\text{O}_4\text{Br}$: C, 63.31; H, 5.09. Found: C, 63.35; H, 5.09.

2.2.2. 7-Formyl-7'-methyl-[2,2'-bi-1H-indene]-3,3'-diethyl-3,3'-dihydroxy-1,1'-dione (**3**)

Sodium (20 mg, 0.87 mmol) was dissolved in 25 mL of absolute ethanol, then 2-nitropropane (80 mg, 0.90 mmol) and **2** (300 mg, 0.8 mmol) were added. The reaction mixture was left at 55–60 $^\circ\text{C}$ for 12 h with stirring. The precipitated sodium bromide was filtered and filtrate was concentrated under reduced pressure to remove ethanol, the solid residue was obtained. Purification was performed by column chromatography (silica, hexane/ether (v/v) = 1:1). **3** was obtained as orange-yellow solid (153 mg,

Scheme 2. Reagents and conditions: (a) NBS/ C_6H_6 , 49%; (b) $(\text{CH}_3)_2\text{CHNO}_2$, $\text{C}_2\text{H}_5\text{ONa}/\text{C}_2\text{H}_5\text{OH}$, 60%; (c) TEMPO/ C_6H_6 , 35%.

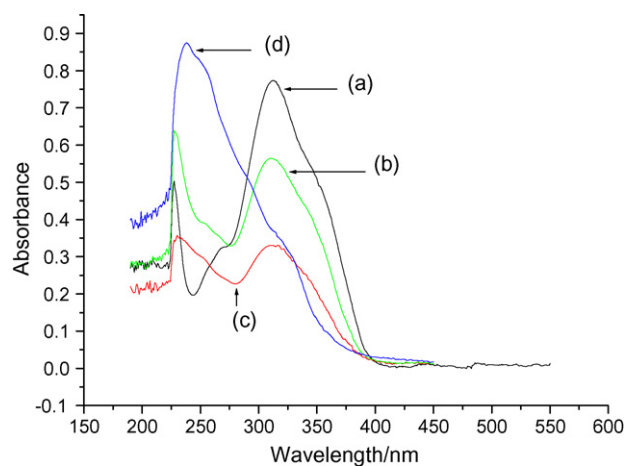


Fig. 2. UV-vis absorption spectra of **1–4** in CH_2Cl_2 solution: (a) **1**; (b) **2**; (c) **3**; (d) **4**.

60%). mp 165–167 °C; $^1\text{H NMR}$ (CDCl_3 , 300 MHz) δ 0.56–0.61 (t, $J=6.0$ Hz, 6H), 2.17–2.31 (m, 4H), 2.50 (s, 3H), 6.34 (br s, 1H), 6.73 (br s, 1H), 7.67–8.32 (m, 6H), 10.22 (s, 1H). MS (ESI): (m/z) 389 ($M-1$). IR (KBr) (cm^{-1}): 3373, 2971, 2915, 1714, 1663, 1613, 1411, 1283, 1160, 1093. Anal. Calcd for $\text{C}_{24}\text{H}_{22}\text{O}_5$: C, 73.83; H, 5.68. Found: C, 73.78; H, 5.58.

2.2.3. 7-Formyl-7'-methyl-[2,2'-bi-1H-indene]-3,3'-diethyl-3,3'-dihydroxy-1,1'-dione with TEMPO radical (**4**)

A benzene solution (10 mL) of **3** (100 mg, 0.26 mmol) and 4-amino-TEMPO (44 mg, 0.26 mmol) with two drops acetic acid was stirred under nitrogen at 45 °C for 10 h. The reaction mixture was concentrated under reduced pressure and then recrystallized from the mixed solvent of hexane/benzene to give the corresponding imine derivative (**4**) as orange-yellow solid (49 mg, 35%). mp 138–139 °C; HRMS (ESI): (m/z) M^+ Calcd for $\text{C}_{33}\text{H}_{39}\text{N}_2\text{O}_5$: 543.2853. Found: 543.2862. IR (KBr) (cm^{-1}): 3444, 2947, 2930, 1711, 1611, 1590, 1453, 1337, 1279, 1176, 1084. Anal. Calcd for $\text{C}_{33}\text{H}_{39}\text{N}_2\text{O}_5$: C, 72.90; H, 7.23; N, 5.15. Found: C, 72.83; H, 7.07; N, 5.25.

3. Results and discussion

3.1. UV-vis absorption spectra in solution

UV-vis absorption peaks and coefficients of compounds **1–4** in CH_2Cl_2 solution are listed in Table 1 and Fig. 2. Compounds **1–3** have two absorption peaks, as shown, respectively, from Table 1 and Fig. 2: one is at 227.5–230 nm,

another is at 310–312.5 nm. The absorption maximum of compound **1** is stronger at long-wavelength band ($\lambda_{\text{max}} = 312.5$ nm) than at short-wavelength band ($\lambda_{\text{max}} = 227.5$ nm), but the absorption maximum of compounds **2** and **3** are stronger at short-wavelength band ($\lambda_{\text{max}} = 227.5$ and 230 nm) than at long-wavelength band ($\lambda_{\text{max}} = 310$ and 310.8 nm). It is interesting that compound **4** has only one strong absorption peak at $\lambda_{\text{max}} = 238.3$ nm, with the strongest intensity $\epsilon = 33,222$ among compounds **1–4**. The absorption peak at short-wavelength band of compound **3** shows a little bathochromic shift (2.5 nm) in comparison with compounds **1** and **2**. However, the absorption peak at short-wavelength band of compound **4** shows considerably bathochromic shift (10.8 nm) in comparison with compounds **1** and **2**, and the absorption peak at long-wavelength band of **4** submerges in its strong absorption peak at short-wavelength band, leading to a strong and broadening absorption peak forming.

3.2. Photochromic property in solid state

All compounds **1–4** possess visible photochromism upon irradiation with high pressure Hg lamp. Their color changes after photoirradiation are listed in Table 2. In consistency with our previous observation [20–25], their UV-vis absorption considerably increases in the wavelength range of 500–800 nm after irradiation with high pressure Hg lamp as illustrated in Fig. 3 for compounds **1–4**. It is interesting that the absorbance of compound **1** is stronger at short-wavelength band (350–450 nm), as well as at long-wavelength band (500–800 nm) after photoirradiation. However, the absorbance of compounds **2–4** become weaker at short-wavelength band (350–450 nm) after photoirradiation. These observations confirm that the compound **4** is photochromic as expected. The photochromic mechanism of **4** is proposed in Scheme 3. The orange solid powder of **4a** turns to dark-brown **4b** upon irradiation with high Hg lamp for 10 min; **4b** is thermally labile, and the dark-brown solid powder fades and returns to the original orange gradually in dark or quickly upon heating under nitrogen atmosphere.

3.3. ESR spectra

As identified previously [20–25], biindenylidenedione derivatives simultaneously generate radicals while undergoing photochromism. Hence, ESR technique could provide solid evidence for the existence of radical species upon photochromism. For the comparison with compound **4**, the precursor **1–3** were tested by ESR technique before and after photoirradiation, respectively. Before photoirradiation, **1–3** do not show ESR signals in solid state at room

Table 1
UV-vis absorption maxima and coefficients of **1–4** in CH_2Cl_2 solution

Compound	λ_{max} (nm) (ϵ)	c ($\times 10^{-5}$ M)
1	227.5 (15,608), 312.5 (23,996)	3.2
2	227.5 (20,554), 310 (18,145)	3.1
3	230 (15,255), 310.8 (14,095)	2.3
4	238.3 (33,222)	2.6

Table 2
The colour changes of compounds **1–4** before and after upon irradiation with high pressure Hg lamp in the solid state

	Compound			
	1	2	3	4
Before photoirradiation	Yellow	Yellow	Orange	Orange
After photoirradiation	Coffee	Brick-red	Brown	Dark-brown

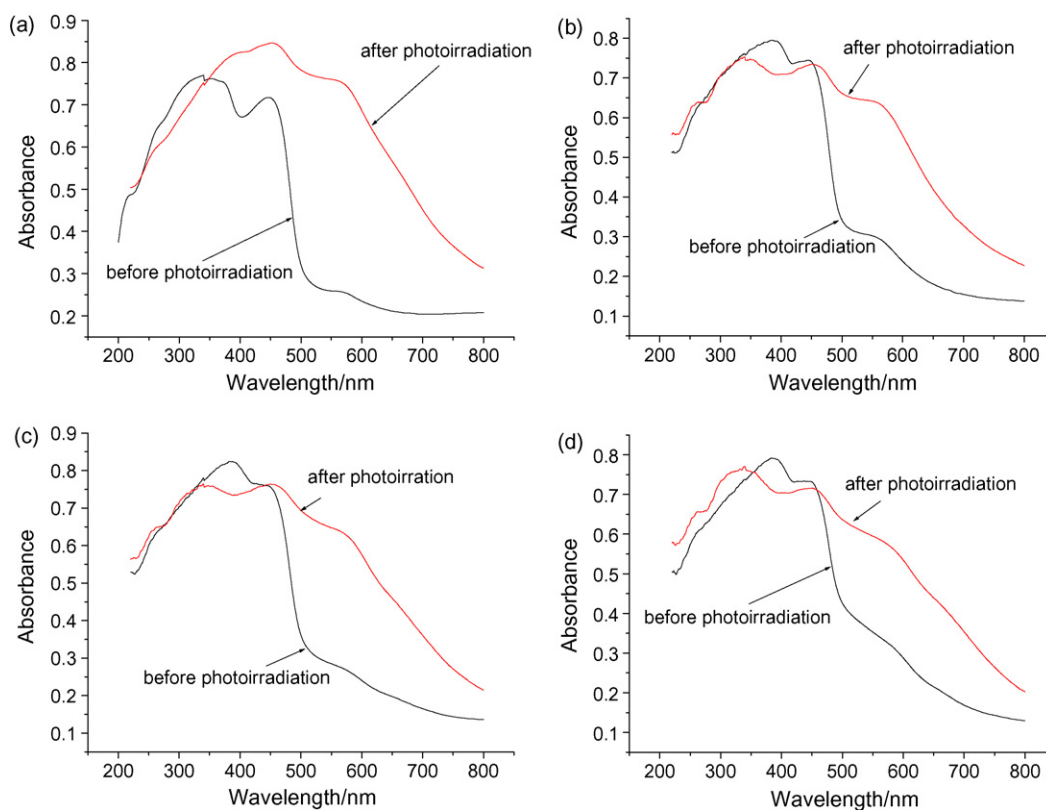
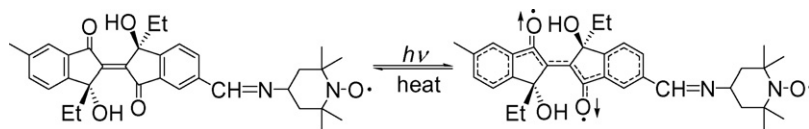


Fig. 3. The changes of UV-vis spectral of compounds **1–4** before and after upon irradiation with high pressure Hg lamp in the solid state: (a) **1**; (b) **2**; (c) **3**; (d) **4**.



Scheme 3. The photochromic mechanism of **4**.

temperature; after photoirradiation, **1–3** show ESR signals in solid state at room temperature (Fig. 4). The characteristic values of ESR signals are as follows: **1** ($g = 2.0029$, $\Delta H_{pp} = 6.45166$ G), **2** ($g = 2.0058$, $\Delta H_{pp} = 6.45141$ G), **3** ($g = 2.0023$, $\Delta H_{pp} = 5.27856$ G), respectively. While the irradiated **1–3** are dissolved in dichloromethane, the resulting solutions do not show any ESR signals. The results clearly indicated that the radicals generated from two indanone moieties are quenched in the solution.

Compound **4** was then monitored with ESR measurement. Fig. 5 shows the corresponding ESR spectra of **4** in different states of its photochromic reaction before and after irradiation with high-pressure mercury lamp in solid state. Before photoirradiation, **4** has a one-line ESR spectrum, showing only ESR signal of TEMPO radical ($g = 2.0055$, $\Delta H_{pp} = 25.0244$) (Fig. 5a). By comparison with its precursors **1–3**, the characteristic values of ESR signals of **4** show distinctive differences with **1–3**, for example, ΔH_{pp} of compound **4** is especially larger than its precursors. It indicates that the ESR signal of TEMPO radical is different from the ESR signal of photo-generated radical originating from two indanone moieties. After irradiation, the ESR spectrum ($g = 2.0072$, $\Delta H_{pp} = 25.8064$) of **4** is very similar to

that of unirradiated **4**, but with considerable broadening of the signal (Fig. 5b). Why does the ESR signal broadening occur after photoirradiation of **4**? Usually, the magnitude of the intermolecular interaction depends on the distance between the spins, and their exchange interaction becomes stronger as the distance of the spin centres decreases [34,35]. Before photoirradiation, in compound **4** there is only one spin centre from TEMPO radical, and after photoirradiation a new spin centre is formed from photo-generated radical of two indanone moieties. Thus, in compound **4** there are two kinds of spin centre after irradiation. The increasing number of spin centres will result in the inter- and/or intra-molecular distances between the spins decreasing, and then the interactions between the spins become stronger, leading to ESR signal broadening, i.e. secular broadening. Presumably, this secular broadening comes from the weak spin–spin interactions between the generated radicals resulting from photochromism and TEMPO radical. Also, this result reflects that the generated radicals and TEMPO radical spatially locate in a distant way, and there are only weak interactions among them.

At room temperature, as the irradiated dark-brown **4** was dissolved in dichloromethane, there is only a typical TEMPO radical signal in the ESR spectrum of the resultant solution,

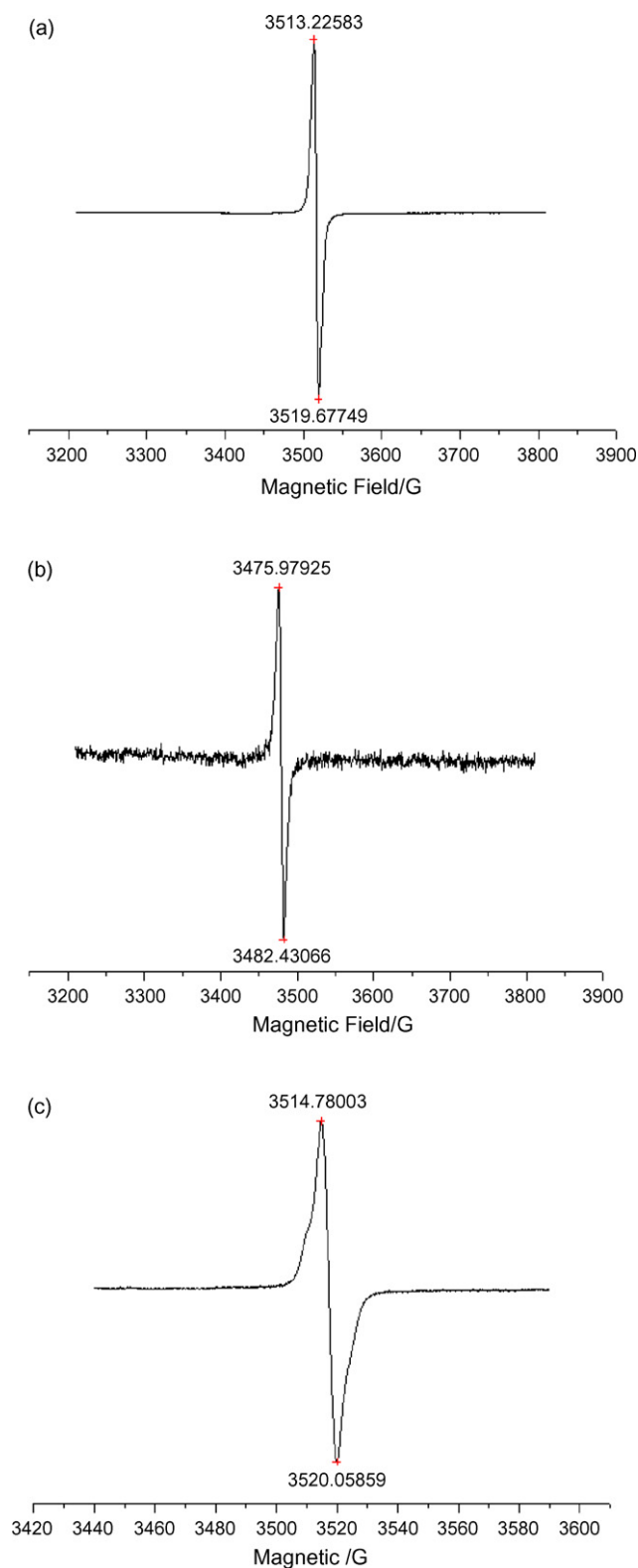


Fig. 4. ESR spectra of **1–3** in solid state at room temperature: (a) **1**; (b) **2**; (c) **3**. (a) Measurement condition: center field, 3510.000 G; sweep width, 600.000 G; working frequency, 9.858 GHz; power, 2.002 mW; modulation frequency, 100 kHz; modulation amplitude, 1.00 G. (b) Measurement condition: center field, 3510.290 G; sweep width, 600.000 G; working frequency, 9.768 GHz; power, 1.992 mW; modulation frequency, 100 kHz; modulation amplitude, 1.00 G. (c) Measurement condition: center field, 3515.000 G; sweep width, 150.000 G; working frequency, 9.858 GHz; power, 2.002 mW; modulation frequency, 100 kHz; modulation amplitude, 1.00 G.

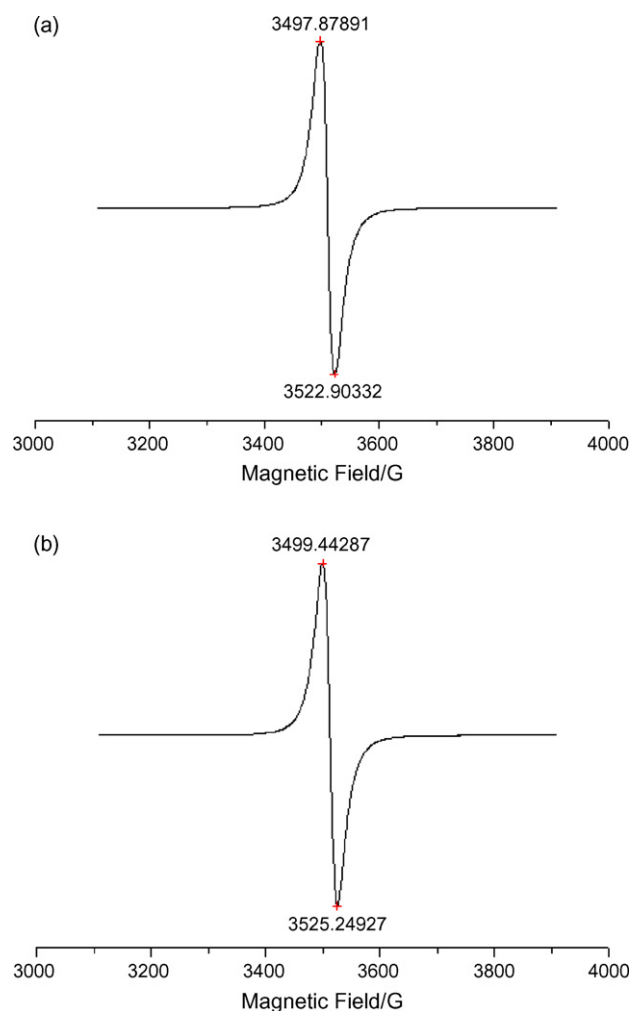


Fig. 5. ESR spectra of **4** in solid state at room temperature: (a) Before photoirradiation; (b) after irradiation with a high pressure Hg lamp. (a) Measurement condition: center field, 3510.000 G; sweep width, 800.000 G; working frequency, 9.854 GHz; power, 2.002 mW; modulation frequency, 100 kHz; modulation amplitude, 1.00 G. (b) Measurement condition: center field, 3510.000 G; sweep width, 800.000 G; working frequency, 9.868 GHz; power, 2.002 mW; modulation frequency, 100 kHz; modulation amplitude, 1.00 G.

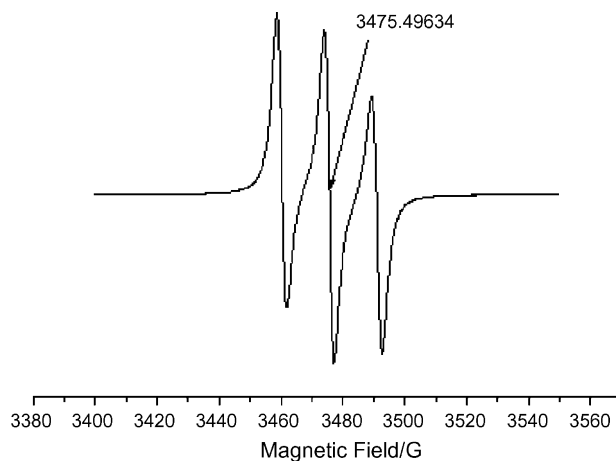


Fig. 6. ESR spectra of **4** in dichloromethane solution. Measurement conditions: center field, 3475.000 G; sweep width, 150.000 G; working frequency, 9.771 GHz; Power, 1.996 mW; modulation frequency, 100.00 kHz; modulation amplitude, 1.00 G.

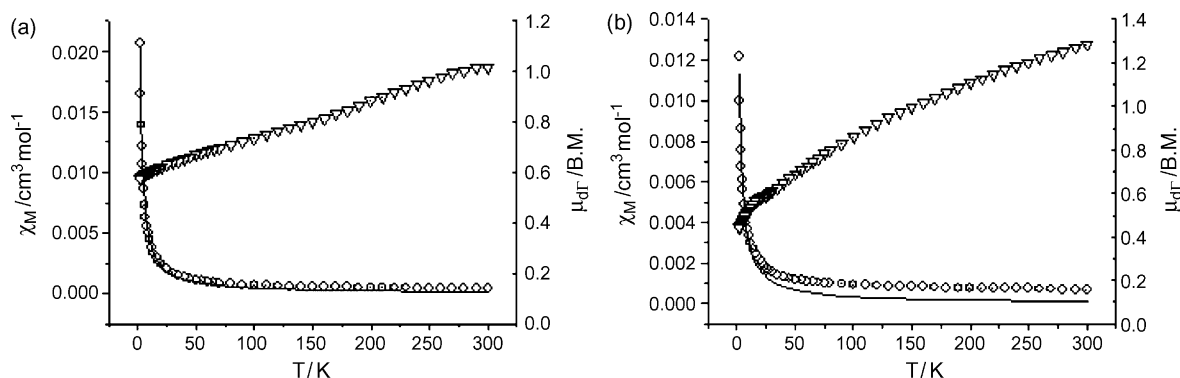


Fig. 7. Temperature dependence of the magnetic susceptibility (χ) of **4**: (a) before photoirradiation; (b) after photoirradiation.

the spectrum consists of a well-resolved symmetrical three-line pattern at $g_{\text{iso}} = 2.0086$ with intensities close to 5:5:3 ratio, and a hyperfine coupling, $a_N = 15$ G (Fig. 6). ESR spectra of compound **4** do not change in solution under photoirradiation. This indicates that the radicals originated from the indanone moieties are quenched in the solution. Further research discovered that the family of compounds in this work display photochromism and simultaneously generate radicals in solid state, however, the radicals generated from two indanone moieties can be easily quenched in solution and the photochromism also does not appear in solution.

3.4. Solid-state magnetic susceptibility

The magnetic susceptibility measurement for **4** was carried out on powder samples by a MPMS XL-7 SQUID susceptometer in the temperature range of 2–300 K, at a magnetic field of 2000 G. The results are shown in Fig. 7 as a plot of μ_{eff} and χ_M versus T . The magnetic behavior of **4** was observed before and after photoirradiation, respectively. Before photoirradiation, the antiferromagnetic spin–spin interaction originating from TEMPO radical was found in **4** with Curies constant ($C = 0.046$ emu K mol $^{-1}$) and Weiss temperature ($\theta = -0.257$ K) (Fig. 7a). In contrast, after irradiation, a considerably different Curie–Weiss behavior of antiferromagnetic interaction was observed for the spins of the corresponding **4** with Curies constant ($C = 0.034$ emu K mol $^{-1}$) and Weiss temperature ($\theta = -1.003$ K) (Fig. 7b). This result clearly shows that the antiferromagnetic interaction for **4** becomes stronger after irradiation. This enhanced antiferromagnetism supposedly results from spin–spin interaction both TEMPO radical and photo-generated radicals of two indanone moieties. Thus, the magnetism of **4** is indeed photo-controlled. Further investigation on this type of compounds is under way in our laboratory.

4. Conclusion

In this work, stable nitroxide radical TEMPO was successfully introduced into the benzene rings of novel photochromic biindenylidenedione derivatives, leading to a novel multifunctional compound **4**. Its photochromic property, the change

of ESR spectra and magnetic property upon photoirradiation was investigated. The experiments display that compound **4** possesses visible photochromism upon photoirradiation. ESR spectral analysis demonstrated that secular broadening of ESR signals occurs after photoirradiation in solid state of compound **4**. The magnetic susceptibility measurement shows that the antiferromagnetic interaction for **4** becomes stronger after photoirradiation. These results demonstrated that, in compound **4** there are two kinds of spin centres after photoirradiation: one is TEMPO radical, and another is photo-generated radical of two indanone moieties. Also, between the two kinds of spin centres supposedly exists inter- and/or intra-molecular spin–spin interaction in **4**. Thus, the colour and magnetic properties of compound **4** could be controlled by photoirradiation.

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Appendix A

(1) Calculate of g value for compounds **1–4**.

- (i) For **1**: $g = h\nu/H\beta = (6.625 \times 10^{-27}) \times (9.858 \times 10^9) / (3516.45166 \times (0.9273 \times 10^{-20})) = 2.0028562 \approx 2.0029$.
- (ii) For **2**: $g = h\nu/H\beta = (6.625 \times 10^{-27}) \times (9.768 \times 10^9) / (3479.204955 \times (0.9273 \times 10^{-20})) = 2.0058167 \approx 2.0058$.
- (iii) For **3**: $g = h\nu/H\beta = (6.625 \times 10^{-27}) \times (9.858 \times 10^9) / (3517.41931 \times (0.9273 \times 10^{-20})) = 2.00231 \approx 2.0023$.
- (iv) For **4**: before photoirradiation: $g = h\nu/H\beta = (6.625 \times 10^{-27}) \times (9.854 \times 10^9) / (3510.391115 \times (0.9273 \times 10^{-20})) = 2.0055$.
- (v) For **4**: after photoirradiation: $g = h\nu/H\beta = (6.625 \times 10^{-27}) \times (9.868 \times 10^9) / (3512.34607 \times (0.9273 \times 10^{-20})) = 2.0072314 \approx 2.0072$.

(vi) For **4**: in CH_2Cl_2 solution: $g = h\nu/H\beta = (6.625 \times 10^{-27}) \times (9.771 \times 10^9) / (3475.49634 \times (0.9273 \times 10^{-20})) = 2.00857 \approx 2.0086$.

(2) The HRMS spectrum of compound **4**.

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