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Dyes and Pigments 75 (2007) 111–115
DYES and PIGMENTS
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Synthesis and photochromic properties of 9-(4-substituted)phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-diones derived from phenoxynaphthacenequinones

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Received 11 April 2006; received in revised form 22 May 2006; accepted 22 May 2006
Available online 27 June 2006

Abstract

A series of novel 9-(4-substituted)phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-diones were synthesized and their structures were confirmed using elemental analysis and the IR, ^{1}H NMR and MS spectra. The photochromism of these compounds was observed in DMSO, DMF, CH_2Cl_2, $CHCl_3$, benzene and toluene. The *trans* forms had an absorption maximum in the range of 419–422 nm, while the *ana* forms had two large absorption maxima in the ranges 489–492 nm and 524–526 nm. The λ_{max} and isosbestic points were affected little by the electronic properties of the substituents on the phenoxy group. However, the formation of the photostationary state was strongly influenced by the polarity of the substituents.

Keywords: 9-Phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-dione derivative; Phenoxynaphthacenequinones; Synthesis; Photochromism

1. Introduction

One promising class of photochromic compounds is the phenoxynaphthacenequinones which were shown to be photochromic in the early 1970s [1]. Their photochromism is based on the photoinduced interconversion of the yellow *trans* form into the orange-coloured *ana* form (Scheme 1). The *trans* form has an absorption peak at around 400 nm, while the *ana* form is characterized by a double peak in the range of 440–480 nm. The important features of these materials are their relatively low fatigue and negligible thermal *ana* to *trans* form reaction. Another important feature is that the hologram obtained by UV irradiation can be fixed by storing in NH_3 vapor over ammonia solution, which coverts the *ana* form into the amino-*ana*-phenoxynaphthacenequinone [2–4]. These properties suggest that the compounds may be suitable for use as materials for molecular switches, 3D optical memory, and hologram recording [3,5–7].

However, another photochromic compound 9-phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-dione, which is derived from phenoxynaphthacenequinone and has similar photoisomerization behaviour [8], has been rarely studied. To the best our knowledge, no literature has appeared on the photochromic properties of its derivatives. We report herein the synthesis and absorption spectra of the derivatives 9-(4-substituted)phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-diones (**4**), which were prepared from the corresponding phenoxynaphthacenequinones (Scheme 2). Effects of the substituent on the photochromic behaviour of these compounds are also reported.

2. Experimental

Melting points were measured on an XT-4 micromelting point apparatus and were uncorrected. IR spectra were recorded with a Nicolet 5SXC instrument using KBr tabulating.

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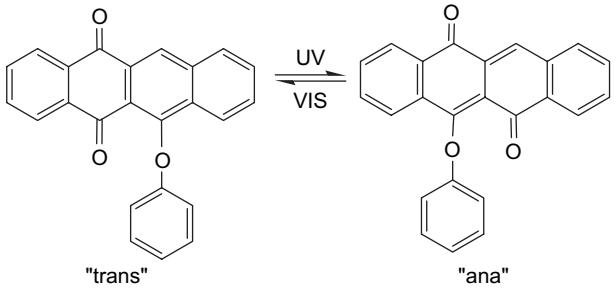
doi:10.1016/j.dyepig.2006.05.026



Scheme 1. Photoisomerization of phenoxynaphthacenequinone.

The ^{1}H NMR spectra were recorded with a Brucker AVANCE 500 at 500 MHz in deuterium solvent with TMS as an internal reference. EI mass spectra (70 eV) were obtained with Micromass LCT spectrometer. An element vario EL III analyzer (made by Elementar Analysem Systeme, Germany) was used for elemental analysis. Absorption spectra were recorded with a Varian-Cary 100 spectrophotometer. Photoreactions were carried out in solvents, such as DMSO (1×10^{-4} mol dm^{-3}), at an ambient temperature and irradiation at 365 nm from an ultraviolet lamp in a ZF-20D ultraviolet analyzer. 6-Chloro-5,12-naphthacenequinone (**1**) was synthesized and purified according to the method described by Buchholtz et al. [2], starting from phthalic anhydride and naphthol. All other solvents and chemicals were purchased from commercial suppliers.

*2.1. Synthesis of 6-(4-substituted)phenoxy-5,12-naphthacenequinones (**2a–e**)*

*2.1.1. 6-Phenoxy-5,12-naphthacenequinone (**2a**) [1]*

A mixture of 6-chloro-5,12-naphthacenequinone (1.0 g, 0.00342 mol), phenol (9.0 g, 0.107 mol) and potassium hydroxide (0.28 g, 0.005 mol) was heated at 165 °C for 1 h and then was poured over aqueous potassium hydroxide solution. The precipitate was filtered, rinsed with boiled water several times and dried. The product was recrystallized from DMSO.

Yield of compound **2a**: 58.8%, (0.68 g); m.p. 224–224.5 °C; Anal. Calcd. for $C_{24}H_{14}O_3$: C, 82.28; H, 4.00. Found: C, 81.96; H, 3.84. IR/cm^{-1}: ∼*ν*: 3060 (w, Ar–H), 1672 (s, C=O), 1580, 1488, 1429, 1344, 1280, 1235, 1217, 981, 748, 714. ^{1}H NMR (500 MHz, $CDCl_3$): *δ*/ppm = 6.89 (d, *J* = 7.92 Hz, 2H, Ar–H), 7.02 (t, *J* = 7.37 Hz, 1H, Ar–H), 7.29 (t, *J* = 7.52 Hz, 2H, Ar–H), 7.66 (t, *J* = 7.22 Hz, 1H, Ar–H), 7.69–7.81 (m, 3H, Ar–H), 8.14 (d, *J* = 8.21 Hz, 1H, Ar–H), 8.20–8.27 (m, 2H, Ar–H), 8.32–8.38 (m, 1H, Ar–H), 8.86 (s, 1H, Ar–H). MS: *m*/*z* (%): 350 (M^+, 100).

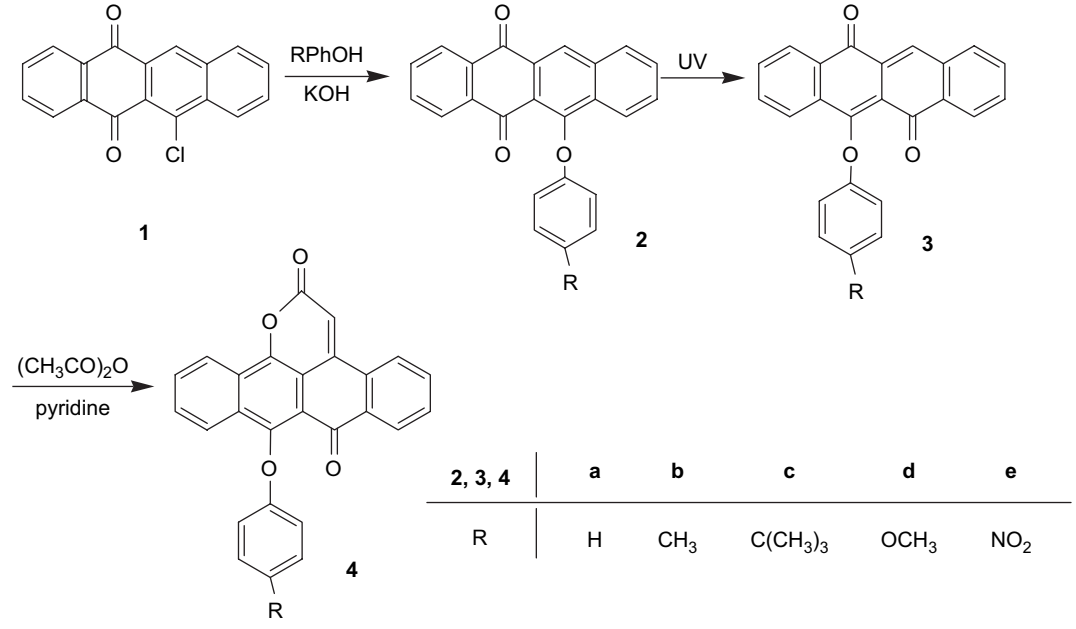
*2.1.2. 6-(4-Methylphenoxy)-5,12-naphthacenequinone (**2b**)*

The product was synthesized and purified as described for compound **2a**.

Yield of compound **2b**: 68.2%; m.p. 211–212 °C; Anal. Calcd. for $C_{25}H_{16}O_3$: C, 82.42; H, 4.40. Found: C, 82.09; H, 4.31. IR/cm^{-1}: ∼*ν*: 3064 (w, Ar–H), 2916 (w, CH_3), 2857, 1675 (s, C=O), 1612, 1580, 1504, 1424, 1396, 1343, 1273, 1213, 982, 814, 754, 714. ^{1}H NMR (500 MHz, $CDCl_3$): *δ*/ppm = 2.27 (s, 3H, CH_3), 6.78 (d, *J* = 8.53 Hz, 2H, Ar–H), 7.07 (d, *J* = 8.17 Hz, 2H, Ar–H), 7,65 (t, *J* = 7.10 Hz, 1H, Ar–H), 7.70–7.80 (m, 3H, Ar–H), 8.14 (d, *J* = 8.18 Hz, 1H, Ar–H), 8.24 (d, *J* = 7.68 Hz, 2H, Ar–H), 8.31–8.37 (m, 1H, Ar–H), 8.86 (s, 1H, Ar–H). MS: *m*/*z* (%): 364 (M^+, 100).

*2.1.3. 6-(4-tert-Butylphenoxy)-5,12-naphthacenequinone (**2c**)*

The product was synthesized and purified as described for compound **2a**.



Scheme 2. Synthesis of 9-(4-substituted)phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-diones (**4**).

Yield of compound **2c**: 63.1%; m.p. 251.5–252 °C; Anal. Calcd. for $C_{28}H_{22}O_3$: C, 82.76; H, 5.42. Found: C, 82.28; H, 5.32. IR/cm^{-1}: ∼ν: 3066 (w, Ar–H), 2959 (m, $C(CH_3)_3$), 2899, 2862, 1672 (s, C=O), 1614, 1579, 1507, 1425, 1395, 1363, 1343, 1277, 1235, 1176, 981, 830, 748, 714. ^{1}H NMR (500 MHz, DMSO-d_6): δ/ppm = 1.20 (s, 9H, $C(CH_3)_3$), 6.77 (d, J = 8.88 Hz, 2H, Ar–H), 7.26 (d, J = 8.93 Hz, 2H, Ar–H), 7.76 (t, J = 8.22 Hz, 1H, Ar–H), 7.83 (t, J = 7.92 Hz, 1H, Ar–H), 7.86–7.93 (m, 2H, Ar–H), 8.03–8.13 (m, 2H, Ar–H), 8.20–8.27 (m, 1H, Ar–H), 8.39 (d, J = 8.13 Hz, 1H, Ar–H), 8.85 (s, 1H, Ar–H). MS: *m/z* (%): 391 (100), 406 (M^+, 39).

2.1.4. 6-(4-Methoxyphenoxy)-5,12-naphthacenequinone (**2d**)

The product was obtained as described for compound **2a** and further purified on a silica column with toluene as eluent.

Yield of compound **2d**: 44.0%; m.p. 210.5–211.5 °C; Anal. Calcd. for $C_{25}H_{16}O_4$: C, 78.95; H, 4.21. Found: C, 78.86; H, 4.05. IR/cm^{-1}: ∼ν: 3057 (w, Ar–H), 2929 (w, CH_3), 2902, 2831, 1672 (s, C=O), 1613, 1580, 1503, 1426, 1398, 1345, 1279, 1228, 1212, 983, 824, 757, 714. ^{1}H NMR (500 MHz, $CDCl_3$): δ/ppm = 3.75 (s, 3H, OCH_3), 6.7–6.9 (m, 4H, Ar–H), 7.59–7.69 (m, 1H, Ar–H), 7.69–7.83 (m, 3H, Ar–H), 8.13 (d, J = 8.15 Hz, 1H, Ar–H), 8.19–8.31 (m, 2H, Ar–H), 8.31–8.40 (m, 1H, Ar–H), 8.85 (s, 1H, Ar–H). MS: *m/z* (%): 380 (M^+, 100).

2.1.5. 6-(4-Nitrophenoxy)-5,12-naphthacenequinone (**2e**)

The product was obtained as described for compound **2a** and further purified on a silica column with toluene as eluent.

Yield of compound **2e**: 52.8%; m.p. 285–286 °C; Anal. Calcd. for $C_{24}H_{13}NO_5$: C, 72.91; H, 3.29; N, 3.54. Found: C, 72.93; H, 3.15; N, 3.30. IR/cm^{-1}: ∼ν: 3058 (w, Ar–H), 1671 (s, C=O), 1655, 1592, 1580, 1509, 1342 (s, NO_2),1283, 1245, 1168, 1110, 843, 757, 718. ^{1}H NMR (500 MHz, $CDCl_3$): δ/ppm = 6.98 (d, J = 9.11 Hz, 2H, Ar–H), 7.72 (t, J = 8.41 Hz, 1H, Ar–H), 7.75–7.85 (m, 3H, Ar–H), 8.14 (d, J = 8.18 Hz, 1H, Ar–H), 8.18–8.25 (m, 4H, Ar–H), 8.37 (d, J = 7.21 Hz, 1H, Ar–H), 8.94 (s, 1H, Ar–H). MS: *m/z* (%): 395 (M^+, 100).

2.2. Synthesis of 9-(4-substituted)phenoxy-naphthaceno-[5,6-bc]pyran-2,8-diones (**4a–e**)

2.2.1. 9-Phenoxy-naphthaceno[5,6-bc]-pyran-2,8-dione (**4a**) [1,8]

A solution of compound **2a** (0.28 g, 0.0008 mol) in 280 ml of toluene was exposed to UV light for 20 h in a round-bottomed glass flask. The colour of the reaction mixture changed from light yellow to deep yellow gradually during the reaction at ambient temperature. The solvent was removed under reduced pressure and the solid product, consisting mainly of compound **3a**, was obtained. This material was dissolved in pyridine (20 cm^3), and acetic anhydride (10 cm^3) was added. The reaction mixture was stirred for 36 h at room temperature and then poured into ice water. The precipitate was filtered, washed with water, and dried. The product was chromatographed using silica as sorbent and toluene as eluent and then recrystallized from DMSO.

Yield of compound **4a**: 57.7% (0.18 g); m.p. 319–321 °C; Anal. Calcd. for $C_{26}H_{14}O_4$: C, 80.00; H, 3.59. Found: C, 79.33; H, 3.60. IR/cm^{-1}: ∼ν: 3450, 3070 (w, Ar–H), 1707 (vs, C=O), 1657 (s, C=O), 1594, 1490, 1426, 1363, 1287, 1251, 1212, 1027, 884, 743, 715. ^{1}H NMR ($CDCl_3$): δ/ppm = 6.88 (d, J = 7.87 Hz, 2H, Ar–H), 7.03 (t, J = 7.38 Hz, 1H, Ar–H), 7.29 (d, J = 7.55 Hz, 2H, Ar–H), 7.35 (s, 1H), 7.65–7.76 (m, 2H, Ar–H), 7.79 (t, J = 7.28 Hz, 1H, Ar–H), 7.87 (t, J = 8.28 Hz, 1H, Ar–H), 8.18 (d, J = 8.14 Hz, 1H, Ar–H), 8.30 (d, J = 7.25 Hz, 1H, Ar–H), 8.40 (d, J = 5.20 Hz, 1H, Ar–H), 8.73 (d, J = 8.42 Hz, 1H, Ar–H). MS: *m/z* (%): 390 (M^+, 100).

2.2.2. 9-(4-Methylphenoxy)-naphthaceno[5,6-bc]-pyran-2,8-dione (**4b**)

The product was synthesized and purified as described for compound **4a**.

Yield of compound **4b**: 51.8%; m.p. 307–308 °C; Anal. Calcd. for $C_{27}H_{16}O_4$: C, 80.20; H, 3.97. Found: C, 80.12; H, 3.78. IR/cm^{-1}: ∼ν: 3432, 3071 (w, Ar–H), 2919 (w, CH_3), 1708 (vs, C=O), 1658 (s, C=O), 1595, 1505, 1427, 1363, 1287, 1252, 1214, 1027, 884, 780, 717. ^{1}H NMR ($CDCl_3$): δ/ppm = 2.28 (s, 3H, CH_3), 6.78 (d, J = 8.63 Hz, 2H, Ar–H), 7.07 (d, J = 7.46 Hz, 2H, Ar–H), 7.33 (s, 1H), 7.71 (q, 2H, Ar–H), 7.79 (t, J = 7.20 Hz, 1H, Ar–H), 7.87 (t, J = 7.04 Hz, 1H, Ar–H), 8.18 (d, J = 7.82 Hz, 1H, Ar–H), 8.29 (d, J = 8.14 Hz, 1H, Ar–H), 8.40 (d, J = 8.34 Hz, 1H, Ar–H), 8.73 (d, J = 8.25 Hz, 1H, Ar–H). MS: *m/z* (%): 404 (M^+, 100).

2.2.3. 9-(4-tert-Butylphenoxy)-naphthaceno[5,6-bc]-pyran-2,8-dione (**4c**)

The product was synthesized and purified as described for compound **4a**.

Yield of compound **4c**: 40.9%; m.p. 320–322 °C; Anal. Calcd. for $C_{30}H_{22}O_4$: C, 80.72; H, 4.93. Found: C, 80.13; H, 4.95. IR/cm^{-1}: ∼ν: 3431, 3073 (w, Ar–H), 2956 (ms, CH_3), 2901, 2864, 1712 (vs, C=O), 1661 (s, C=O), 1598, 1507, 1427, 1363, 1287, 1253, 1233, 1177, 1027, 885, 824, 773, 719. ^{1}H NMR (DMSO-d_6): δ/ppm = 1.25 (s, 9H, $C(CH_3)_3$), 6.79 (d, J = 8.85 Hz, 2H, Ar–H), 7.27 (d, J = 8.88 Hz, 2H, Ar–H), 7.76 (s, 1H, Ar–H), 7.80 (d, J = 7.48 Hz, 1H, Ar–H), 7.84 (t, J = 8.17 Hz, 1H, Ar–H), 7.90 (t, J = 8.33 Hz, 1H, Ar–H), 7.97 (t, J = 7.30 Hz, 1H, Ar–H), 8.15 (d, J = 8.45 Hz, 1H, Ar–H), 8.20 (d, J = 7.84 Hz, 1H, Ar–H), 8.58 (d, J = 8.47 Hz, 1H, Ar–H), 8.66 (d, J = 8.28 Hz, 1H, Ar–H). MS: *m/z* (%): 431 (100), 446 (M^+, 19).

2.2.4. 9-(4-Methoxyphenoxy)-naphthaceno[5,6-bc]-pyran-2,8-dione (**4d**)

The product was obtained as described for compound **4a**. It was further purified on a silica column with toluene as eluent and then recrystallized from DMSO/H_2O (3:1 v/v) mixed solvent.

Yield of compound **4d**: 33.6%; m.p. 302–303.5 °C; Anal. Calcd. for $C_{27}H_{16}O_5$: C, 77.14; H, 3.81. Found: C, 76.57; H, 3.69. IR/cm^{-1}: ∼ν: 3448, 3070 (w, Ar–H), 2950 (w, CH_3), 2833, 1708 (vs, C=O), 1656 (s, C=O), 1596, 1505, 1427, 1364, 1286, 1241, 1206, 1181, 1031, 885, 818, 780, 746, 716. ^{1}H NMR ($CDCl_3$): δ/ppm = 3.75 (s, 3H, OCH_3), 6.72–6.90 (m, 4H, Ar–H), 7.35 (s, 1H), 7.67–7.76 (m, 2H, Ar–H), 7.79 (t, J = 7.20 Hz, 1H, Ar–H), 7.87 (t, J = 7.06 Hz, 1H, Ar–H), 8.17 (d, J = 7.73 Hz, 1H, Ar–H), 8.32 (d, J = 8.98 Hz, 1H, Ar–H), 8.40 (d, J = 7.84 Hz, 1H, Ar–H), 8.73 (d, J = 8.34 Hz, 1H, Ar–H). MS: *m/z* (%): 420 (M^+, 100).

*2.2.5. 9-(4-Nitrophenoxy)-naphthaceno[5,6-bc]-pyran-2,8-dione (**4e**)*

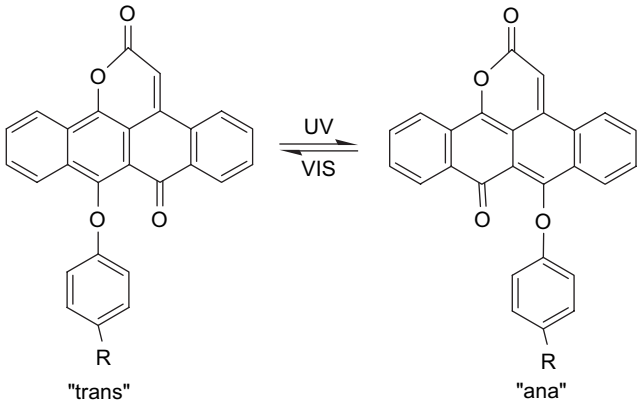
The product was obtained as described for compound **4a**. It was further purified on a silica column with toluene as eluent and then recrystallized from DMSO/H_2O (4:1 v/v) mixed solvent.

Yield of compound **4e**: 22.7%; m.p. >322 °C; Anal. Calcd. for $C_{26}H_{13}NO_6$: C, 71.72; H, 2.99; N, 3.22. Found: C, 71.67; H, 2.84; N, 3.15. IR/cm^{-1}: ∼ν: 3430, 3074 (w, Ar–H), 1720 (vs, C=O), 1666 (s, C=O), 1593, 1511, 1488, 1431, 1343 (vs, NO_2), 1258, 1223, 1109, 1025, 721. ^{1}H NMR ($CDCl_3$): δ/ppm = 6.99 (d, J = 9.04 Hz, 2H, Ar–H), 7.38 (s, 1H, Ar–H), 7.72 (t, J = 7.54 Hz, 1H, Ar–H), 7.75–7.87 (m, 2H, Ar–H), 7.92 (t, J = 7.64 Hz, 1H, Ar–H), 8.21 (t, J = 7.80 Hz, 4H, Ar–H), 8.36 (d, J = 7.40 Hz, 1H, Ar–H), 8.78 (d, J = 8.37 Hz, 1H, Ar–H). MS: *m/z* (%): 407 (100), 435 (M^+, 76).

3. Results and discussion

3.1. Synthesis

In recent years, the usual synthetic method for the preparation of 6-(4-substituted)phenoxy-5,12-naphthacenequinones is via the reaction of 6-chloro-5,12-naphthacenequinone with 4-substituted phenols and potassium carbonate in dry DMF; a reaction time of 3–4 h at about 110 °C is common [2,5,9]. We also used this synthetic method to prepare compounds **2a–e** and found that compounds **2b** and **2c** were difficult to purify by recrystallization and compound **2e**, prepared in low yield, was not the main product after purification. In comparison, the synthetic method described in Section 2 was simple and gave good results.



Scheme 3. Photoisomerization of compound **4**.

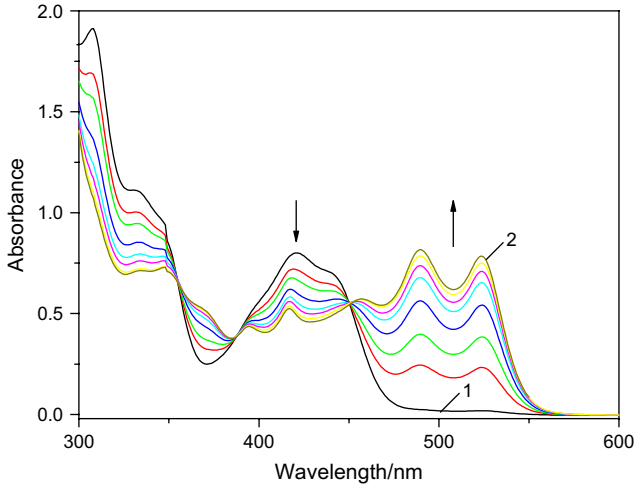


Fig. 1. Spectral changes of compound **4a** in DMSO (1×10^{-4} mol dm^{-3}) due to light irradiation (λ = 365 nm). Irradiation time (min): 0 (curve 1), 5, 10, 20, 30, 40, 60, and 80 (curve 2, PSS).

Compound **3** was obtained by the photoisomerization of compound **2**; it was used without further purification in the next step as a little unreacted compound **2** could not be reacted with acetic anhydride, and furthermore, it was not easily separated from, the similar, compound **3**.

According to thin layer chromatography (TLC) in toluene the reaction of compound **3a** and acetic anhydride was complete after 36 h, whereas 48 h was required for the same reaction under the experimental conditions reported in literature [8].

*3.2. Photochromic properties of 9-(4-substituted)-phenoxy-naphthaceno[5,6-bc]pyran-2,8-diones (**4**)*

Each of the syhthesized 9-(4-substituted)phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-diones (**4a–e**) readily dissolved in DMSO, DMF, CH_2Cl_2 and $CHCl_3$, sparingly soluble in benzene and toluene and insoluble in less polar solvents, such as carbon tetrachloride. Photochromism of these compounds was observed in DMSO, DMF, CH_2Cl_2, $CHCl_3$, benzene and toluene, which can be attributed to the photoinduced

Table 1
Spectral characteristics of compounds **4a–e** in DMSO (1×10^{-4} mol dm^{-3})

Compound	R	λ_{max} (nm)		Isosbestic points (nm)
		Trans	*Ana*	
4a	H	421	490, 524	354, 388, 450
4b	CH_3	422	490, 524	354, 388, 450
4c	$C(CH_3)_3$	421	490, 524	354, 386, 450
4d	OCH_3	422	489, 524	356, 381, 450
4e	NO_2	419	492, 526	359, 382, 454

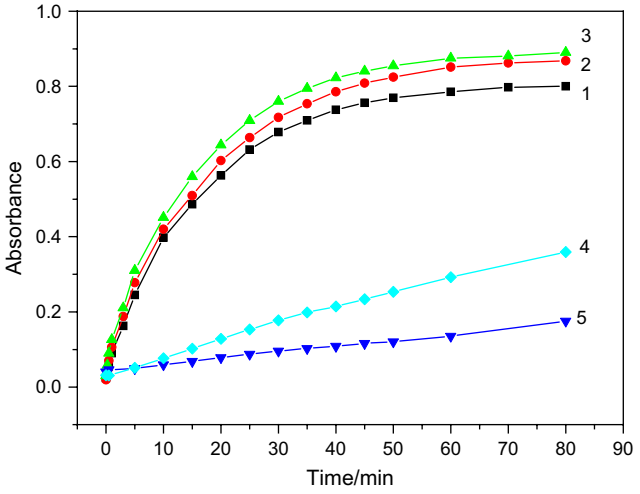


Fig. 2. Variation of the absorbance of compounds **4a**–**e** in DMSO with irradiation time: curve 1, compound **4a** at 490 nm; curve 2, compound **4b** at 490 nm; curve 3, compound **4c** at 490 nm; curve 4, compound **4d** at 489 nm; curve 5, compound **4e** at 492 nm.

isomerization of the yellow *trans* form to the *ana* orange-coloured form (Scheme 3).

Fig. 1 shows the photoinduced spectral changes of a DMSO solution of compound **4a** induced by irradiation at 365 nm. The *trans* form has an absorption peak at 421 nm, while the *ana* form displayed typical double peaks at 490 and 524 nm. The photoreaction involves only the *trans* and *ana* forms was proven by the isosbestic points observed at 354, 388 and 450 nm. Compared with the absorption spectrum of compound **2a** [4], the corresponding peaks and isosbestic points of compound **4a** were shifted 16–45 nm to longer wavelengths.

Table 1 shows that the *trans* forms of **4a**–**e** had an absorption maximum in the range of 419–422 nm and the *ana* forms exhibited two large absorption maxima in the ranges 489–492 nm and 524–526 nm. The λ_{max} and isosbestic points were little affected by the electronic properties of the substituents on the phenoxy group, which is very similar to the photochromic behaviour of compound **2** [10]. This is because the electronic effects of the substituent are buffered by the oxygen atom between the naphthacenepyrone and the phenyl group.

Fig. 2 shows that the photostationary states for compounds **4a**, **4b**, and **4c** were achieved using 80 min of UV irradiation at 365 nm, whereas compounds **2d** and **2e** could not apparently reach their photostationary states under the same experimental conditions; similar results were observed in other solvents such as benzene and toluene. This finding indicates that the polarity of the substituent on the phenoxy group has a strong effect on the photochromic behaviour of 9-(4-substituted)phenoxy-naphthaceno[5,6-*bc*]pyran-2,8-dione.

4. Conclusions

The absorption data revealed that while the nature of the substituent on the phenoxy group has little effect on the λ_{max} of both the *trans* and *ana* forms of the colourant, it did exert a strong effect on the formation of the stationary state.

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