

3,3-Bis(3,5-dimethylphenyl)-3*H*-benzo[*f*]chromeneHua-Ming Huang,* Qiao-Yun
Du, Hong-Bing Yang and Jian-Ge
WangDepartment of Chemistry, Luoyang Normal
University, Luoyang, 471022, People's Republic
of China

Correspondence e-mail: lyhbm@lynu.edu.cn

Key indicators

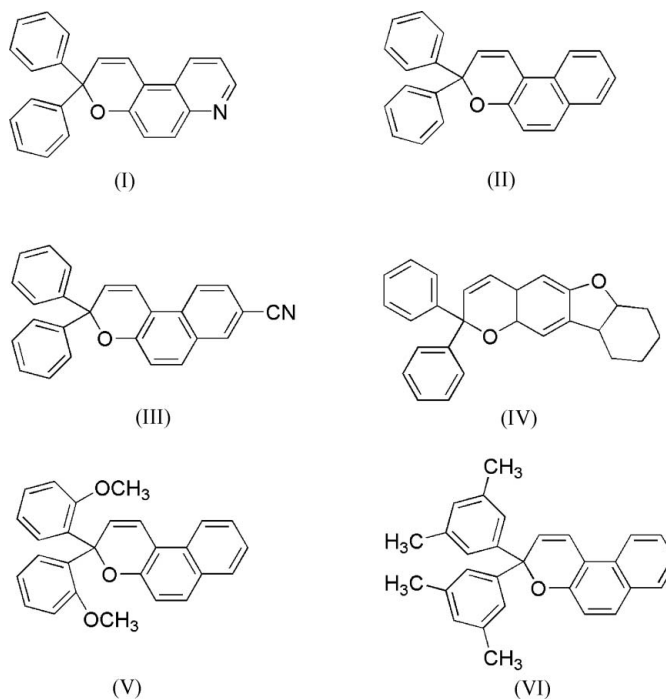
Single-crystal X-ray study
 $T = 291\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.049
 wR factor = 0.146
Data-to-parameter ratio = 18.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title molecule, $\text{C}_{29}\text{H}_{26}\text{O}$, the two methyl substituents on the benzene rings are sterically hindered and influence the structure of the molecule and its photochemical properties. The dihedral angle between the two benzene ring planes is $80.2(1)^\circ$.

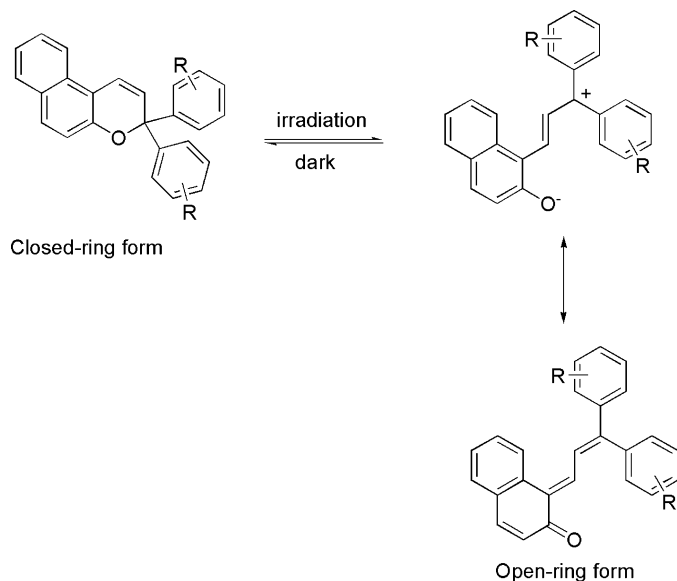
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Comment

2,2-Diphenylchromene derivatives are a special kind of photochromic compound. Even though 2*H*-chromenes do not contain a spiroheterocyclic fragment, some of them have excellent photochromic properties and have found commercial applications (Malatesta, 1999). Aldoshin *et al.* (1995, 1996) and Aldoshin, Chuev, Filipenko, Pozzo *et al.* (1998) have examined a number of 2,2-diphenylchromene derivatives, *viz.* (I), (II), (III) and (IV) (scheme 1). In our previous work, we have found that in compound (V) the substituent methoxy groups at the benzene *ortho*-positions have dramatically enhanced the optical density and reduced its fade rate (Huang *et al.*, 2004). In order to understand how the two substituents at the benzene 3- and 5-positions affect the photochemical properties, we have prepared the title compound, (VI), and its structure has been determined by X-ray crystallography.



steric interactions. The C13—O1 bond [1.461 (2) Å] is longer than a typical Csp^2 —O bond in a six-membered heterocycle [1.41 (2)–1.43 (2) Å; Birukov & Unkovskij, 1976], but is indistinguishable from those in (I)–(IV) [1.441 (2)–1.468 (2) Å].



The pyran ring in (VI) adopts a slightly distorted screw boat conformation. The dihedral angles between the naphthyl unit (C1–C10) and the benzene rings (C14–C19) and (C22–C27) are 75.3 (1)° and 94.3 (2)°, respectively. The mutual arrangement of the benzene rings in (VI) is different from that in (V); the dihedral angle between these fragments is 80.2 (1)°, smaller than the values in (V) and (II) [93.3 (2)° and 96.6 (2)°, respectively]. However, the two benzene rings become almost coplanar when the molecule is in the open-ring form (see

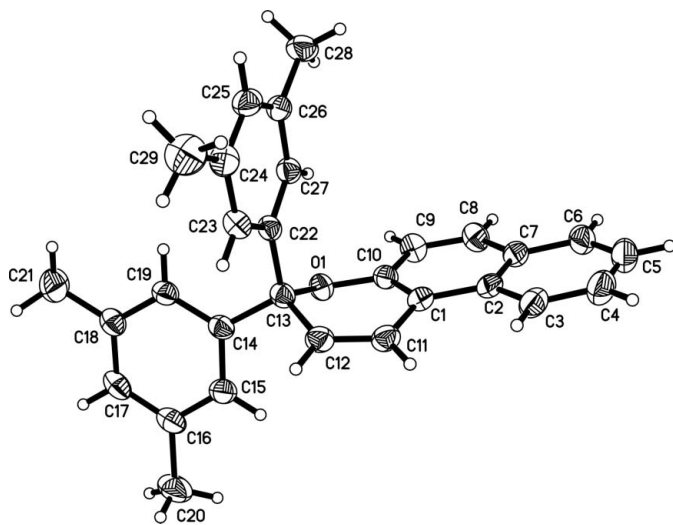


Figure 1
The molecular structure of (VI), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

scheme). Apparently, the two benzene rings have to rotate by some angle about the C13—C14 and C13—C22 bonds to revert to the closed-ring form. The electron-donating substituents on the benzene rings hinder this rotation, but stabilize the pyramidal carbocation at atom C13. As a result, the optical density is enhanced and the fade rate is reduced.

Experimental

All starting materials were commercially available and of analytical grade. The title compound was synthesized by the reaction of 7,8-benzocoumarin and phenylmagnesium bromide as described by Cottam & Livingstone (1964). Colourless, plate-like crystals suitable for X-ray measurements were obtained by slow evaporation of an ethanol solution at room temperature over a period of one week.

Crystal data

$C_{29}H_{26}O$	$Z = 4$
$M_r = 390.50$	$D_x = 1.174 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 11.5290 (8) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$b = 11.3445 (8) \text{ \AA}$	$T = 291 (2) \text{ K}$
$c = 16.9415 (12) \text{ \AA}$	Block, colourless
$\beta = 94.124 (1)^\circ$	$0.35 \times 0.18 \times 0.09 \text{ mm}$
$V = 2210.1 (3) \text{ \AA}^3$	

Data collection

Bruker ApexII CCD area-detector diffractometer	13103 measured reflections
φ and ω scans	5053 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3024 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.976$, $T_{\max} = 0.994$	$R_{\text{int}} = 0.026$
	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.2364P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.146$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
5053 reflections	$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
275 parameters	
H-atom parameters constrained	

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for aromatic H and 0.96 Å, $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$ for methyl H.

Data collection: APEX2, (Bruker 2004); cell refinement: APEX2; data reduction: SAINT, (Bruker 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker 2004); software used to prepare material for publication: SHELXTL.

References

- Aldoshin, S., Chuev, I., Filipenko, O., Pozzo, J. L., Lokshin, V. & Pèpe, G. (1998). *Acta Cryst.* **C54**, 1720–1722.
 Aldoshin, S., Chuev, I., Utenyshev, A., Lokshin, V., Pozzo, J. L., Pèpe, G. & Guglielmetti, R. (1995). *Acta Cryst.* **C51**, 141–143.
 Aldoshin, S., Chuev, I., Utenyshev, A., Filipenko, O., Pozzo, J. L., Lokshin, V. & Guglielmetti, R. (1996). *Acta Cryst.* **C52**, 1834–1838.
 Birukov, B. P. & Unkovskij, B. V. (1976). *Kristalloghimiya*, **11**, 132–177. (In Russian.)

- Bruker (2004). *APEX2* (Version 1.027), *SAINT* (Version 7.12a) and *SHELXTL* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
- Cottam, J. & Livingstone, R. (1964). *J. Chem. Soc.* pp. 5228–5231.
- Huang, H.-M., Ding, Y., Chen, P.-L. & Meng, J.-B. (2004). *Acta Cryst.* **C60**, o709–o710.
- Malatesta, V. (1999). *Photochromic and Thermochromic Compounds*. Edited by J. C. Crano and R. Guglielmetti. Vol. 2, ch. 3, pp. 65–165. New York: Kluwer Academic Publishers.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.