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14-(4-Fluorophenyl)-14*H*-dibenzo[*a*,*j*]xanthene

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.099
Data-to-parameter ratio = 8.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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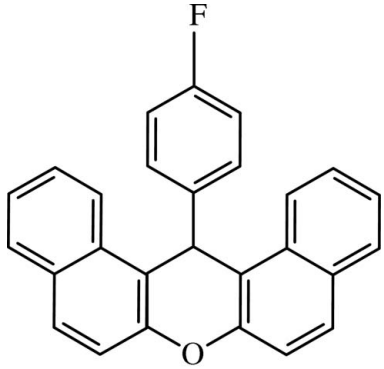
In the title compound, $C_{27}H_{17}FO$, the mean plane of the xanthene core is almost perpendicular to that of the the 4-fluorophenyl substituent, the dihedral angle being 87.69 (6)°. The xanthene core has a boat conformation, the folding angle between the naphthyl units being 22.54 (4)°.

Comment

Considerable interest has been focused on the synthesis of xanthenes. In particular, benzoxanthenes have attracted much attention in recent years because of their wide range of biological and pharmacological applications, such as antiviral, antibacterial and anti-inflammatory properties (Lambert *et al.*, 1997; Hideo, 1981; Poupelin *et al.*, 1978). They also exhibit efficacy in photodynamic therapy and are used as antagonists for the paralyzing action of zoxazolamine (Ion *et al.*, 1998). Xanthenes are also available from natural sources. The well known Santalin pigments have been isolated from a number of plant species (Kinjo *et al.*, 1995). Furthermore, due to their useful spectroscopic properties, they are used as dyes, in laser technologies and in fluorescent materials for the visualization of biomolecules (Menchen *et al.*, 2003). The present study of the title compound, (I), is part of the structural investigation of a series of xanthene derivatives which is aimed at an analysis of conformational changes.



(I)

Fig. 1 shows the molecular structure of (I) with the atomic numbering scheme. The bond lengths and angles in (I) are normal (Allen *et al.*, 1987). The central ring of the xanthene core adopts a boat conformation [puckering parameters (Cremer & Pople, 1975) Q = 0.316 (2) Å, q_2 = 0.312 (2) Å, q_3 = −0.056 (2) Å, θ = 100.3 (4)° and φ = 359.6 (4)° for the atom sequence O10/C11/C12/C9/C13/C14]. The mean plane of the xanthene core, defined by the atoms C11, C12, C13 and C14, is almost perpendicular to that of the 4-fluorophenyl substituent

at C9, the dihedral angle being 87.69 (6)°. The xanthene core of the molecule is V-shaped, with atoms C1–C4/C13/C14/C9/O10 defining one plane and atoms C5–C8/C11/C12/C9/O10 defining the other. The mean distances of these atoms from their respective least-squares planes are 0.032 and 0.043 Å. Atoms C9 and O10 lie on the line of intersection of the planes (Blackburn *et al.*, 1996) and the dihedral angle (folding angle) between the planes is 22.54 (4)°. A similar observation has been reported for the structure of 9-isopropyl-xanthene (Chu & Yang, 1977), where the folding angle was found to be 21.9°.

Experimental

To a stirred solution of 4-fluorobenzaldehyde (0.5 g, 1 mmol) in 1,2-dichloroethane (15 ml) was added β-naphthol (1.16 g, 2 mmol) with silica sulfuric acid (1.5 mol%) as catalyst. The reaction mixture was heated to reflux for 3 h. The progress of the reaction was monitored by thin-layer chromatography. After completion of the reaction, the catalyst was removed by filtration and washed with 1,2-dichloroethane. The organic solvent was evaporated from the filtrate to produce the crude product (1.46 g), which was crystallized from ethanol (m.p. 238 K).

Crystal data

$C_{27}H_{17}FO$
M_r = 376.41
Orthorhombic, $Pna2_1$
a = 13.7042 (4) Å
b = 17.1650 (5) Å
c = 7.7369 (2) Å
V = 1819.97 (9) Å^3
Z = 4
D_x = 1.374 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 160 (1) K
Prism, colourless
0.25 × 0.18 × 0.13 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans with κ offsets
Absorption correction: none
24581 measured reflections
2230 independent reflections
1743 reflections with $I > 2\sigma(I)$
R_{int} = 0.083
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.099
S = 1.05
2230 reflections
263 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.082 (5)

The H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). Although the molecule is achiral, the structure possesses a polar axis. Because of the absence of any significant anomalous scatterers in the compound, the absolute direction of the polar axis was assigned arbitrarily and the Friedel pairs were merged before the final refinement.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

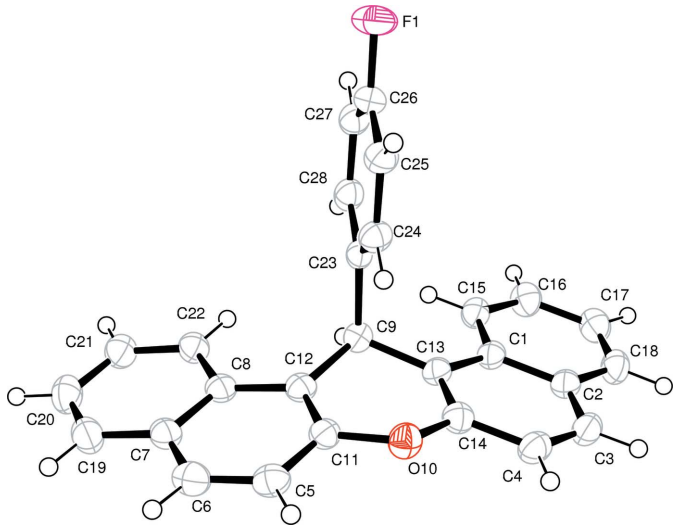


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary radii.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Blackburn, A. C., Dobson, A. J. & Gerkin, R. E. (1996). *Acta Cryst.* C**52**, 1486–1488.
Chu, S. S. C. & Yang, H. T. (1977). *Acta Cryst.* B**33**, 2291–2293.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hideo, T. (1981). Jpn Patent No. 56005480 (Kokai Tokkyo Koho); *Chem. Abstr.* **95**, 80922b.
Ion, R. M., Frackowiak, D., Planner, A. & Wiktorowicz, K. (1998). *Acta Biochim. Pol.* **45**, 833–845.
Kinjo, J., Uemura, H., Nohara, T., Yamashita, M., Marubayashi, N. & Yoshihira, K. (1995). *Tetrahedron Lett.* **36**, 5599–5602.
Lambert, R. W., Martin, J. A., Merrett, J. H., Parkes, K. E. B. & Thomas, G. J. (1997). PCT Int. Appl. WO 9706178; *Chem. Abstr.* **126**, 212377y.
Menchen, S. M., Benson, S. C., Lam, J. Y. L., Zhen, W., Sun, D., Rosenblum, B. B., Khan, S. H. & Taing, M. (2003). *Chem. Abstr.* **139**, p54287f, US Patent US6583168.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Poupelin, J. P., Saint-Rut, G., Foussard-Blanpin, O., Narcisse, G., Uchida Ernouf, G. & Lacroix, R. (1978). *Eur. J. Med. Chem.* **13**, 67–71.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.