Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2′,7′-Bis(4-methoxyphenyl)spiro[cyclopropane-1,9′-9*H*-fluorene]

Zi-Xing Wang, Hai-Yao Lin and Ping Lu*

Department of Chemistry, Zhejiang University, Hangzhou 310027, People's Republic of China

Correspondence e-mail: pinglu@zju.edu.cn

Received 9 February 2006
Accepted 13 February 2006

Key indicators

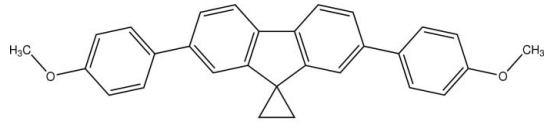
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.119
Data-to-parameter ratio = 9.0

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

The title compound, $C_{29}H_{24}O_2$, has mirror symmetry with the cyclopropane C atoms lying on the mirror plane. The fluorene system is essentially planar and makes dihedral angles of 91.43 (4) and 23.50 (6)° with the cyclopropane and substituted benzene rings, respectively.

Comment

Spirobifluorene derivatives have attracted much attention due to their potential utility in organic light-emitting diodes (Muller *et al.*, 2003). Spiro-linked molecules exhibit greater morphological stability and more intense fluorescence than the corresponding non-spiro-linked compounds. These enhanced properties occur without a significant change in their absorption and fluorescence spectra (Yu *et al.*, 2000). Meanwhile, steric factors can lead to enhanced rigidity in the spiro center, thereby preventing rotation of the adjacent aryl groups, which reduces close packing and intermolecular interaction between chromophores in the solid state (Lee *et al.*, 2005). The title compound, (I), is a useful model from which many spiro[cyclopropane-1,9′-fluorene] derivatives with extended aromatic systems can be investigated.



(I)

The title molecule has mirror symmetry, with the cyclopropane C atoms located on the mirror plane (Fig. 1). The fluorene system is essentially planar and makes a dihedral angle of 91.43 (4)° with the cyclopropane plane. The C—C bond distances within the cyclopropane ring (Table 1) show an equilateral triangle structure. The benzene ring of the methoxyphenyl group is inclined to the fluorene plane with a dihedral angle of 23.50 (6)°, indicating there is no conjugation between them. A weak C—H···O hydrogen bond is observed between neighboring molecules [C8—H8*A* = 0.97 Å, H8*A*···O1^{ii} = 2.58 Å, C8···O1^{ii} = 3.447 (3) Å and C8—H8*A*···O1^{ii} = 148°; symmetry code: (ii) $\frac{3}{2} - x, \frac{1}{2} + y, z$].

Experimental

A mixture of 2′,7′-diiodospiro[cyclopropane-1,9′-fluorene] (444 mg, 1.0 mmol), 4-methoxyphenylboronic acid (456 mg, 3 mmol), K_2CO_3 (1.4 g, 10 mmol), and $Pd(PPh_3)_4$ (10 mg, 0.08 mmol) in 50 ml toluene/degassed water (100:1) was refluxed for 40 h. After cooling and filtering, the filtrate was evaporated under reduced pressure. The crude product was purified by column chromatography (silica gel)

using *n*-hexane/dichloromethane (6:1 *v*/*v*) as eluant. In this way, 331 mg (82% yield) of (I) was obtained as a pale yellow solid. ^{1}H NMR (500 MHz, δ in p.p.m., $CDCl_3$): 1.77 (*s*, 4H), 3.84 (*s*, 6H), 6.97 (*d*, J = 9.0 Hz, 4H), 7.19 (*d*, J = 1.5 Hz, 2H), 7.53 (*dd*, J_1 = 8.0 Hz, J_2 = 1.5 Hz, 2H), 7.55 (*d*, J = 9.0 Hz, 4H), 7.83 (*d*, J = 8.0 Hz, 2H). ^{13}C NMR (125 MHz, δ in p.p.m., $CDCl_3$): 18.86, 29.85, 55.59, 114.45, 117.12, 120.44, 125.19, 128.44, 134.43, 138.56, 139.80, 149.24, 159.33; HRMS (MALDI–TOF): calculated for $C_{29}H_{24}O_2$: 404.2, found: 404.3 (M^+). The crystal used for the data collection was obtained by slow evaporation of a saturated hexane–dichloromethane (1:4 *v*/*v*) solution of (I) at room temperature.

Crystal data

$C_{29}H_{24}O_2$
M_r = 404.48
Orthorhombic, $Cmc2_1$
a = 23.307 (12) Å
b = 9.841 (4) Å
c = 9.211 (4) Å
V = 2112.7 (17) Å^3
Z = 4
D_x = 1.272 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8306 reflections
θ = 3.2–27.5°
μ = 0.08 mm^{-1}
T = 295 (2) K
Block, yellow
0.32 × 0.28 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
10172 measured reflections
1322 independent reflections
1077 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.5°
$h = -30 \rightarrow 30$
$k = -12 \rightarrow 12$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.119
S = 1.04
1322 reflections
147 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.078P)^2 + 0.1214P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0040 (11)

Table 1
Selected bond lengths (Å).

C2—C2^i	1.452 (4)	C8—C9	1.515 (4)
C5—C10	1.487 (3)	C13—O1	1.368 (3)
C7—C8	1.490 (6)	C16—O1	1.409 (5)
C7—C9	1.512 (5)		

Symmetry code: (i) $-x+1, y, z$.

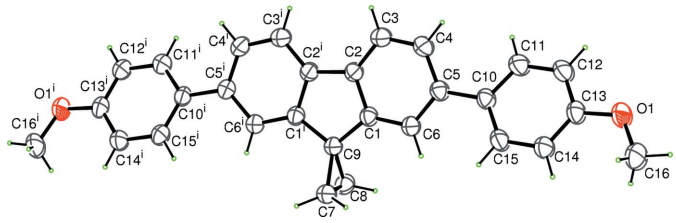


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (i) $1-x, y, z$].

In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. Methyl H atoms were placed in calculated positions (C—H = 0.96 Å) and the torsion angles were refined to fit the electron density [U_{iso}(H) = 1.5U_{eq}(C)]. Other H atoms were placed in calculated positions [C—H = 0.93 (aromatic) or 0.97 Å (methylene)] and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure*; software used to prepare material for publication: *CrystalStructure*.

PL thanks the National Natural Science Foundation of China (No. 20374045) and the Natural Science Foundation of Zhejiang Province (No. R404109).

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Lee, S. H., Jang, B. B. & Kafafi, Z. H. (2005). *J. Am. Chem. Soc.* **127**, 9071–9078.
Muller, C. D., Falcou, A., Reckefuss, N., Rojahn, M., Wiederhirn, V., Rudati, P., Frohne, H., Nuyken, O., Becker, H. & Meerholz, K. (2003). *Nature (London)*, **421**, 829–833.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.7.0. Rigaku/MSC Corporation, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yu, W.-L., Pei, J., Huang, W. & Heeger, A. J. (2000). *Adv. Mater.* **12**, 828–831.