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rac-3,3′-Bis(4-fluorophenyl)-3,3′-dihydroxy-2,3,2′,3′-tetrahydro-[2,2′]biindenyl-1,1′-dione

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.190
Data-to-parameter ratio = 16.0

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

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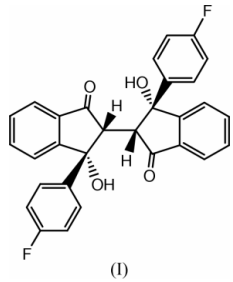
The title compound, $C_{30}H_{20}F_2O_4$, has been obtained as a by-product in the preparation of biindenylidene compounds. The molecule has a twofold axis. There are intramolecular O—H···O hydrogen bonds.

Comment

Photochromism of the inclusion crystals of a hydroperoxy-biindenylidene has been reported recently by Tanaka *et al.* (2003). The title compound, (I), was obtained as a by-product in the preparation of the biindenylidene compounds.



(I)

The molecule (I) has a twofold axis, which is parallel to *c* (Figs. 1 and 2). The central C11—C11^{i} bond [symmetry code: (i) $\frac{1}{2}-x,\frac{1}{2}-y,z$] is a single bond (Table 1), which corresponds to no photochromic property of (I), since the photochromism of these crystals seems to be due to the biradical caused by breaking the π-bond at the center of the biindenylidene moiety (Ohba *et al.*, 2003). The indene ring system is not planar, as the five-membered ring has an envelope conformation, with atom C11 in the flap position. The angle between the planes composed of atoms C12–C18/C10 and C10–C12 is 14.45 (4)°. The angle between the C10—C7 and its symmetry-related bond vector is 25.8 (1)°, and the dihedral angle between the two fluorophenyl rings is 12.15 (10)°. However, the shortest interatomic distance between the fluorophenyl rings is 4.597 (6) Å for C6···C6^{i}, and there is no intramolecular π–π interaction.

There are intramolecular O2—H2···O3^{i} hydrogen bonds (Table 2), but no intermolecular hydrogen bonds. This situation contrasts with the crystal structure of the hydroperoxy-biindenylidene compound, which forms a ladder structure through the O—H···O intermolecular hydrogen bonds, accommodating several guest molecules (Tanaka *et al.*, 2003).

Experimental

Compound (I) was obtained in trace amounts as a by-product in the treatment of 3,3′-bis(4-fluorophenyl)-[2,2′]biindenyl-1,1′-dione with Zn and $ZnCl_2$ in aqueous tetrahydrofuran to obtain 3,3′-bis(4-fluorophenyl)-3H,3′H-[2,2′]biindenylidene-1,1′-dione (22% yield) and 3,1′-bis(4-fluorophenyl)-1′-hydroperoxy-3′-hydroxy-1′,3′-dihydro-3*H*-[2,2′]biindenyliden-1-one (7% yield; Tanaka *et al.*, 2003). The

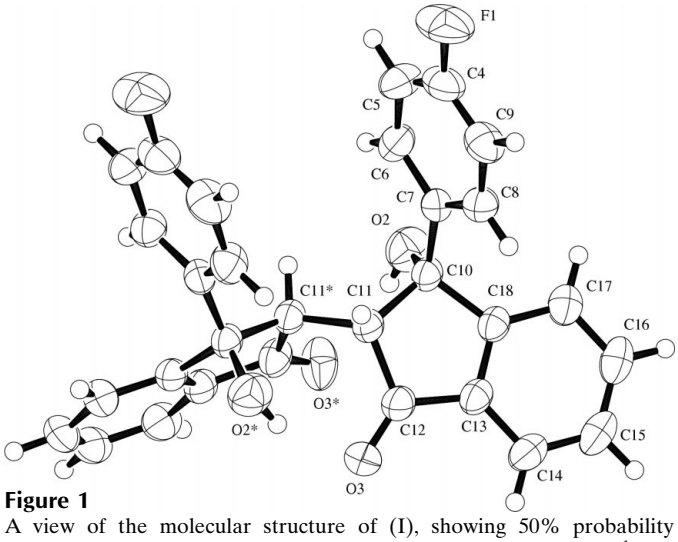


Figure 1
A view of the molecular structure of (I), showing 50% probability displacement ellipsoids for non-H atoms. [Symmetry code: (*) $\frac{1}{2}-x$, $\frac{1}{2}-y$, z.]

reaction mechanism which gave (I) is expected to be similar to that reported by Xu *et al.* (2002). Colourless crystals of (I) were grown from an ethyl acetate solution by slow evaporation (m.p. 482–485 K).

Crystal data

$C_{30}H_{20}F_2O_4$
M_r = 482.48
Orthorhombic, *Pccn*
a = 8.303 (2) Å
b = 12.011 (3) Å
c = 22.868 (6) Å
V = 2280.6 (10) Å^3
Z = 4
D_x = 1.405 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20 reflections
θ = 13.0–14.5°
μ = 0.10 mm^{-1}
T = 298 K
Plate, colourless
0.5 × 0.4 × 0.3 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω scans
Absorption correction: none
3523 measured reflections
2622 independent reflections
1644 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 27.5°
$h = -4 \rightarrow 10$
$k = -15 \rightarrow 6$
$l = 0 \rightarrow 29$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma F^2]$ = 0.054
$wR(F^2)$ = 0.190
S = 1.03
2622 reflections
164 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.093P)^2 + 1.0454P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

F1—C4	1.359 (3)	C11—C11^{i}	1.538 (4)
O2—C10	1.430 (3)	C11—C12	1.533 (3)
O3—C12	1.214 (4)	C12—C13	1.461 (4)
C10—C11	1.563 (4)	C13—C18	1.382 (4)
C10—C18	1.529 (3)		
C10—C11—C11^{i}—C10^{i}	−154.8 (3)	C10—C11—C11^{i}—C12^{i}	82.6 (2)

Symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}-y, z$.

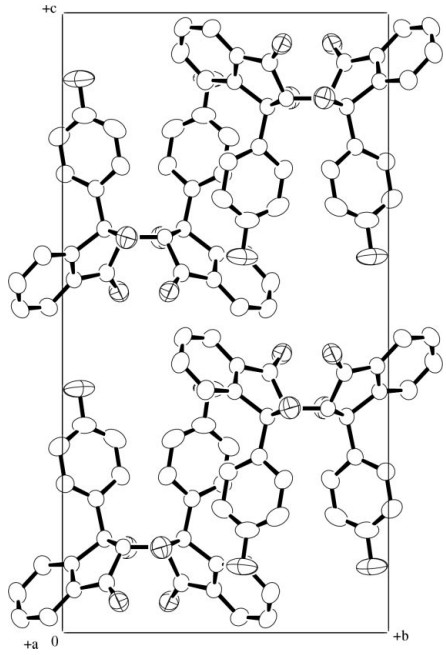


Figure 2
The crystal structure of (I), projected along *a*. H atoms have been omitted for clarity.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.82	1.99	2.766 (3)	158

Symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}-y, z$.

The hydroxyl H atom was located in a difference synthesis and was allowed to ride on the parent atom. The other H atoms were positioned geometrically and fixed, with U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *WinAFC Diffractometer Control Software* (Rigaku, 1999); cell refinement: *WinAFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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