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Key indicators

Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
 R factor = 0.041
 wR factor = 0.079
 Data-to-parameter ratio = 17.0

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

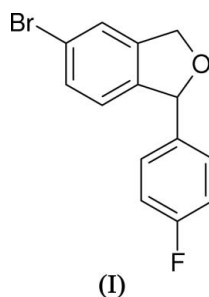
5-Bromo-1-(4-fluorophenyl)-1,3-dihydro-isobenzofuran

The title compound, $\text{C}_{14}\text{H}_{10}\text{BrFO}$, possesses normal geometrical parameters. The dihedral angle between the two ring systems is $71.50(9)^\circ$. An unusually short intermolecular $\text{Br} \cdots \text{Br}$ contact of $3.4311(5) \text{ \AA}$ occurs.

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Comment

The title compound, (I), is an intermediate in the synthesis of the antidepressant drug citalopram (Liechti *et al.*, 2000). More generally, phthalans show distinctive redox chemistry (Azzena *et al.*, 1996). We have previously deposited (CSD-260624; Cambridge Structural Database; Allen, 2002) data for a poor quality structure from a twinned crystal of (I).



The geometrical parameters for (I) are normal. Each molecule of (I) is chiral (the arbitrarily chosen asymmetric unit has an *S* conformation at C7), but crystal symmetry generates a racemic mixture of the two enantiomers. The nine-membered isobenzofuran ring system (C7–C14/O1) is almost planar [r.m.s. deviation from the mean plane = 0.018 \AA ; maximum = $0.038(3) \text{ \AA}$ for C14] and the dihedral angle between the two ring systems (C7–C14/O1 and C1–C6) is $71.50(9)^\circ$.

A PLATON (Spek, 2003) analysis of (I) identified two possible C–H \cdots F interactions (Table 1) that may help to stabilize the crystal packing (Fig. 2). There are no significant π – π stacking interactions in (I).

Inversion symmetry generates a short intermolecular $\text{Br1} \cdots \text{Br1}^i$ [symmetry code: (i) $2 - x, -y, 1 - z$] separation of $3.4311(5) \text{ \AA}$ which is significantly less than the van der Waals contact distance of 3.70 \AA for two Br atoms (Bondi, 1964). Some workers have ascribed specific attractive forces to such short intermolecular halogen–halogen contacts (Desiraju & Parthasarathy, 1989). A database survey of such contacts by Eriksson & Hu (2001) shows that the present separation lies at the lower end of the observed range of intermolecular $\text{Br} \cdots \text{Br}$ distances. However, these workers are less certain of the nature of such contacts, and suggest that they may be the consequence – rather than the cause – of the crystal packing.

In the related 1-(4-fluorophenyl)-1,3-dihydroisobenzofuran-5-carbonitrile [*i.e.* where a cyanide group replaces the Br atom in (I)], there are two molecules in the asymmetric unit with distinctly different degrees of twist between their ring systems (Yathirajan *et al.*, 2004).

Experimental

5-Bromo-3*H*-isobenzofuran-1-one (2.13 g, 10 mmol) was subjected to a Grignard reaction with 4-fluorophenyl magnesium bromide (2.4 g, 12 mmol) in tetrahydrofuran (10 ml) at 273 K. The resulting product was treated with sodium borohydride (0.37 g, 10 mmol) in methanol (10 ml) to obtain the diol, which was cyclized with *p*-toluene sulfonic acid (1 g, 5.81 mmol) in toluene (10 ml) at 353 K, yielding crude (I). Diffraction-quality crystals were obtained by recrystallization from *n*-hexane (Bigler *et al.*, 1977) (m.p. 318 K).

Crystal data

$C_{14}H_{10}BrFO$	$D_x = 1.689 \text{ Mg m}^{-3}$
$M_r = 293.13$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2707 reflections
$a = 6.0560$ (3) Å	$\theta = 1.0\text{--}27.5^\circ$
$b = 7.8659$ (4) Å	$\mu = 3.56 \text{ mm}^{-1}$
$c = 24.2289$ (14) Å	$T = 120$ (2) K
$\beta = 92.542$ (3)°	Block, yellow
$V = 1153.03$ (11) Å ³	$0.24 \times 0.15 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	1742 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.072$
Absorption correction: multi-scan (SADABS; Bruker, 2003)	$\theta_{max} = 27.6^\circ$
$T_{min} = 0.482$, $T_{max} = 0.718$	$h = -7 \rightarrow 7$
12389 measured reflections	$k = -10 \rightarrow 10$
2630 independent reflections	$l = -31 \rightarrow 27$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 0.4294P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.079$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.56 \text{ e \AA}^{-3}$
2630 reflections	$\Delta\rho_{min} = -0.40 \text{ e \AA}^{-3}$
155 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.0023 (6)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C9-H9\cdots F1^i$	0.95	2.54	3.324 (4)	140
$C14-H14B\cdots F1^{ii}$	0.99	2.52	3.265 (4)	132

Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$.

The H atoms were positioned geometrically, with $C-H = 0.95\text{--}0.99$ Å, and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$.

Data collection: COLLECT (Nonius, 1998); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997), and SORTAV (Blessing, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure:

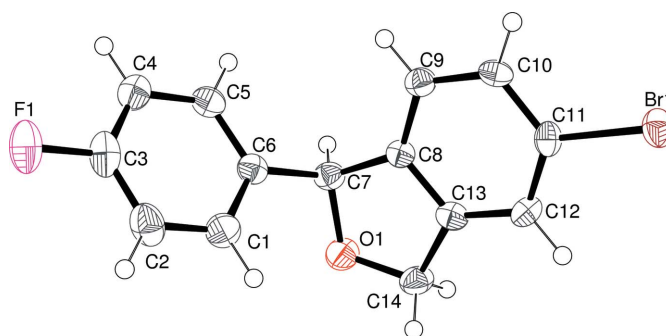


Figure 1
View of (I), showing 50% displacement ellipsoids and arbitrary spheres for the H atoms.

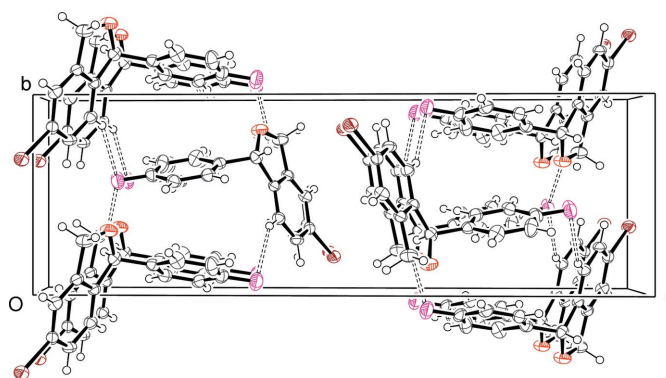


Figure 2
Unit-cell packing in (I), viewed down [100], showing 50% displacement ellipsoids and arbitrary spheres for the H atoms, with short $C-H\cdots F$ interactions shown as dashed lines.

SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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