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1-(4-Fluorophenyl)-1,3-dihydroisobenzofuran-5-carbonitrile

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.097
Data-to-parameter ratio = 11.9

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

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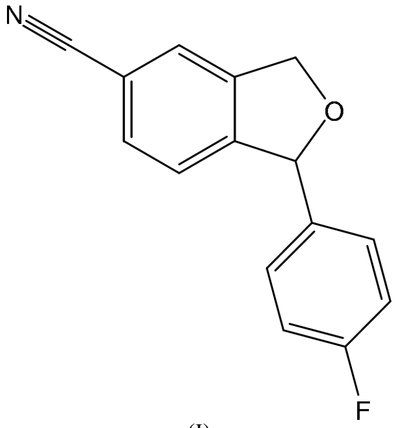
The title compound, $C_{15}H_{10}FNO$, crystallizes with two molecules in the asymmetric unit, which differ only in the orientation of the fluorophenyl ring with respect to the isobenzofuran system.

Comment

1,3-Dihydroisobenzofuran (or phthalan) is used for the preparation of 1,2-di(lithiomethyl)benzene (Almena *et al.*, 1995). Electron-transfer-induced reductive cleavage of phthalans has been reported (Azzena *et al.*, 1996). The title compound, (I), 4-fluorophenyl-5-phthalan carbonitrile, is a key intermediate in the synthesis of citalopram, which is a versatile antidepressant (Liechti *et al.*, 2000). In order to examine the conformation of the phthalan moiety and to study the influence of fluorophenyl and cyano groups, the crystal structure determination of (I) has been carried out and the results are presented here.



(I)

Compound (I) crystallizes with two molecules in the asymmetric unit, and perspective views of these are shown in Figs. 1 and 2. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *MOGUL* Version 1.0; Allen, 2002). The isobenzofuran moiety is essentially planar (r.m.s. deviations 0.020 and 0.022 Å for the two molecules). Bond lengths and angles in the two different molecules are essentially equal. The two molecules differ only in the orientation of the fluorophenyl ring with respect to the isobenzofuran system. This difference can be expressed by the corresponding torsion angles (Table 1), which differ by approximately 50°. A least-squares fit of the isobenzofuran moieties including the cyano group (Fig. 3) gives an r.m.s. deviation of 0.015 Å.

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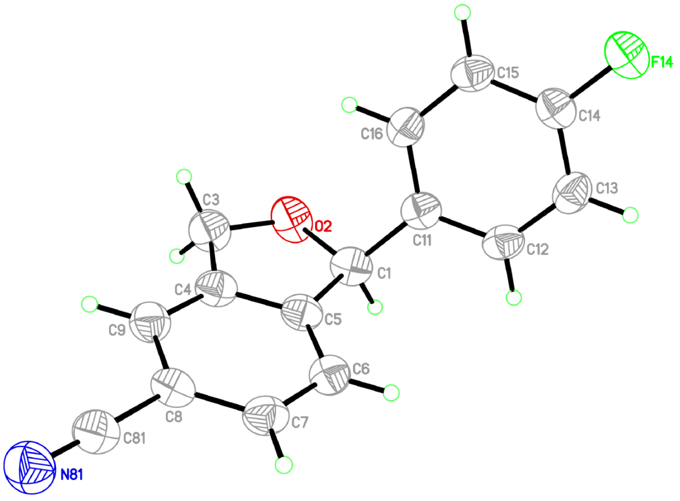

Figure 1
A perspective view of molecule 1 of the two molecules in the asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

There are no classical hydrogen bonds in the crystal structure of (I), but several short C—H···N and C—H···F contacts are observed (Table 2).

Experimental

5-Bromo-3*H*-isobenzofuran-1-one (1.0 g ,4.7 mmol) was subjected to a Grignard reaction with 4-fluorophenyl magnesium bromide (1.12 g, 5.6 mmol) in tetrahydrofuran (20 ml) at 273–278 K. The resulting product was treated with sodium borohydride (0.19 g ,5.2 mmol) in methanol (5 ml) to obtain the diol, which was cyclized with *p*-toluene sulfonic acid monohydrate (0.1 g, 0.5 mmol) in toluene (10 ml) to obtain the cyclized product. The cyclized product was refluxed with CuCN (0.5 g, 5.6 mmol) in dimethylformamide (5 ml) to obtain the title compound (Bigler *et al.*, 1977). X-ray quality crystals of (I) were obtained after recrystallization from solution in acetonitrile.

Crystal data

$C_{15}H_{10}FNO$
$M_r = 239.24$
Monoclinic, $P2_1/n$
$a = 14.606$ (2) Å
$b = 5.8056$ (6) Å
$c = 27.037$ (4) Å
$\beta = 99.032$ (11)°
$V = 2264.2$ (5) Å^3
$Z = 8$

$D_x = 1.404$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8495 reflections
$\theta = 2.8$–25.1°
$\mu = 0.10$ mm^{-1}
$T = 173$ (2) K
Block, light yellow
0.47 × 0.42 × 0.36 mm

Data collection

Stoe IPDS II two-circle diffractometer
ω scans
Absorption correction: none
10 504 measured reflections
3878 independent reflections

2571 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.045$
$\theta_{\rm max} = 25.2°$
$h = -17 \rightarrow 15$
$k = -6 \rightarrow 6$
$l = -30 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.097$
$S = 0.88$
3878 reflections
325 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.14$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

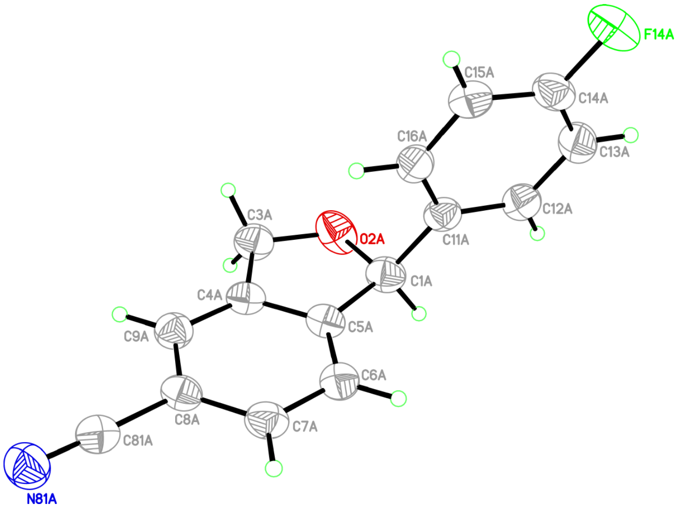

Figure 2
A perspective view of molecule 2 of the two molecules in the asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

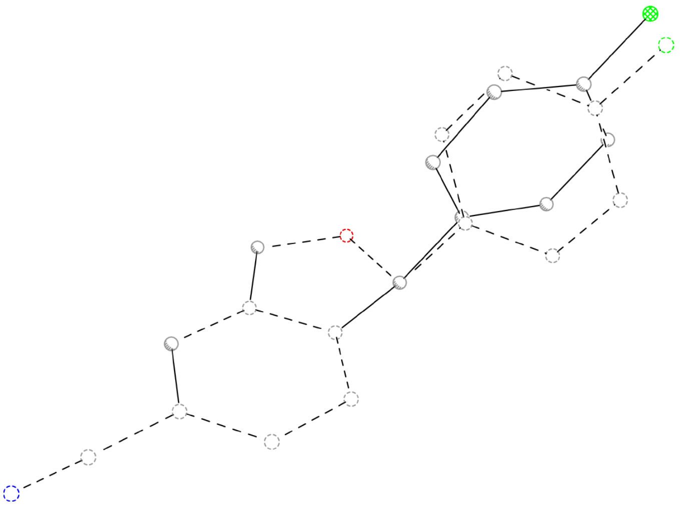

Figure 3
A least-squares fit of the isobenzofuran moieties of the asymmetric unit of (I).

Table 1
Selected geometric parameters (Å, °).

C1—O2	1.432 (2)	C1*A*—O2*A*	1.441 (2)
O2—C3	1.436 (2)	O2*A*—C3*A*	1.430 (2)
C8—C81	1.451 (3)	C8*A*—C81*A*	1.453 (3)
C81—N81	1.131 (2)	C81*A*—N81*A*	1.129 (2)
C14—F14	1.361 (2)	C14*A*—F14*A*	1.359 (2)
O2—C1—C11—C12	−149.46 (15)	O2*A*—C1*A*—C11*A*—C12*A*	−96.76 (18)
O2—C1—C11—C16	33.5 (2)	O2*A*—C1*A*—C11*A*—C16*A*	80.41 (19)

Table 2
Geometry of hydrogen bonds and weak C—H···F interactions (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···N81*A*$^{\rm i}$	0.95	2.60	3.472 (3)	152
C7*A*—H7*A*···N81$^{\rm i}$	0.95	2.62	3.511 (3)	156
C9—H9···N81$^{\rm ii}$	0.95	2.56	3.484 (2)	166
C9*A*—H9*A*···N81*A*$^{\rm iii}$	0.95	2.63	3.479 (2)	148
C6—H6···F14*A*$^{\rm iv}$	0.95	2.80	3.750 (2)	177
C3—H3*B*···F14$^{\rm v}$	0.99	2.69	3.223 (2)	114
C6*A*—H6*A*···F14$^{\rm iv}$	0.95	2.55	3.491 (2)	170
C16*A*—H16*A*···F14$^{\rm vi}$	0.95	2.80	3.611 (2)	144

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $1 - x, 2 - y, 1 - z$; (iii) $1 - x, 2 - y, -z$; (iv) $1 - x, -y, 1 - z$; (v) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (vi) $1 - x, 1 - y, 1 - z$.

H atoms were refined with fixed individual isotropic displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$], using a riding model, with C—H = 1.00, 0.99 and 0.95 Å for tertiary CH, secondary CH and aromatic CH, respectively.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 1990).

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Almena, J., Foubelo, F. & Yus, M. (1995). *Tetrahedron*, **51**, 3351–3364.

Azzena, U., Demartis, S. & Melloni, G. (1996). *J. Org. Chem.* **61**, 4913–4919.

Bigler, A. J., Bogeso, K. B. & Toft, A. (1977). *Eur. J. Med. Chem.* **12**, 289–295.

Liechti, M. E., Baumann, C., Gamma, A. & Vollenweider, F. X. (2000). *Neuropsychopharmacology*, **22**, 513–521.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Stoe & Cie (2001). *X-AREA*. MainMenu Version 1.15. Stoe & Cie, Darmstadt, Germany.