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4-[4-Dimethylamino-1-(4-fluorophenyl)-1-hydroxybutyl]-3-(hydroxymethyl)benzonitrile

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.169
Data-to-parameter ratio = 15.2

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

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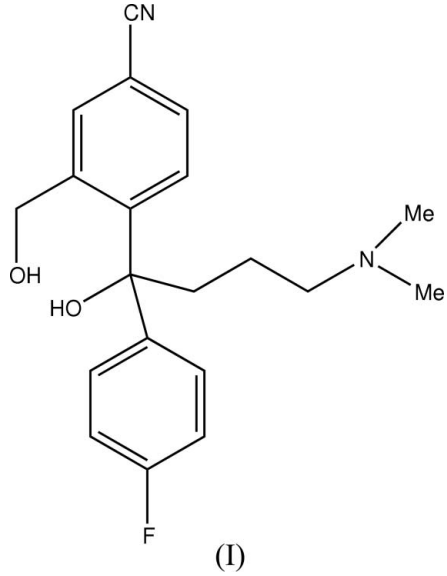
In the structure of the title compound, $C_{20}H_{23}FN_2O_2$, there are two independent molecules showing different conformations. The molecules form centrosymmetric dimers *via* O—H···N or O—H···O hydrogen bonds.

Comment

The title compound, (I), was prepared as an intermediate in the synthesis of fluoxetine, a known antidepressant (Hervas *et al.*, 1998). There are two independent molecules in the asymmetric unit, showing different conformations (Fig. 1). In molecule A, there is an O1—H45···N2 hydrogen bond (Table 2), and atom O2 of the hydroxy group is almost coplanar with the benzene ring (C2/C3/C5/C6/C8/C9). In molecule B, there is an O3—H46···O4 hydrogen bond, and atom O4 of the hydroxy group deviates from the plane of the benzene ring (C22/C23/C25/C26/C28/C29). The aminobutyl chain shows different conformations in the two molecules corresponding to the different hydrogen-bond interactions. The dihedral angles between the two benzene ring planes in molecules A and B are 86.03 (8) and 83.08 (9)°, respectively.



(I)

In the crystal structure of (I), each independent molecule forms a hydrogen-bonded dimer (Fig. 2). For molecule A, atom O2 acts as a hydrogen-bond donor to atom O1 at $(1-x, 2-y, 1-z)$, generating a centrosymmetric $R_2^2(8)$ dimer (Bernstein *et al.*, 1995) centered at $(\frac{1}{2}, 1, \frac{1}{2})$. For molecule B, atom O4 acts as a donor to atom N4 at $(1-x, 1-y, -z)$, forming an $R_2^2(8)$ dimer centered at $(\frac{1}{2}, \frac{1}{2}, 0)$.

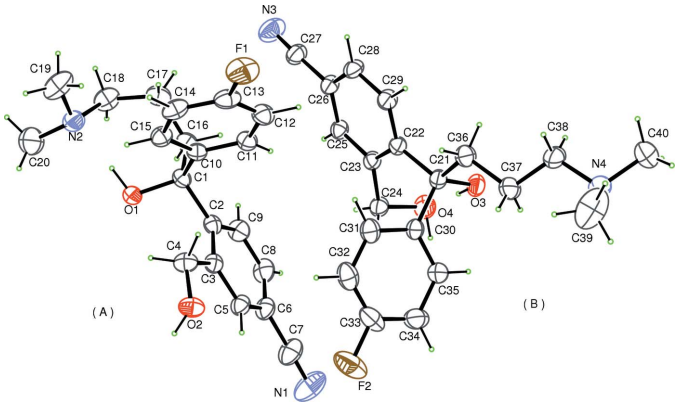


Figure 1
The two independent molecules, A and B, in (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

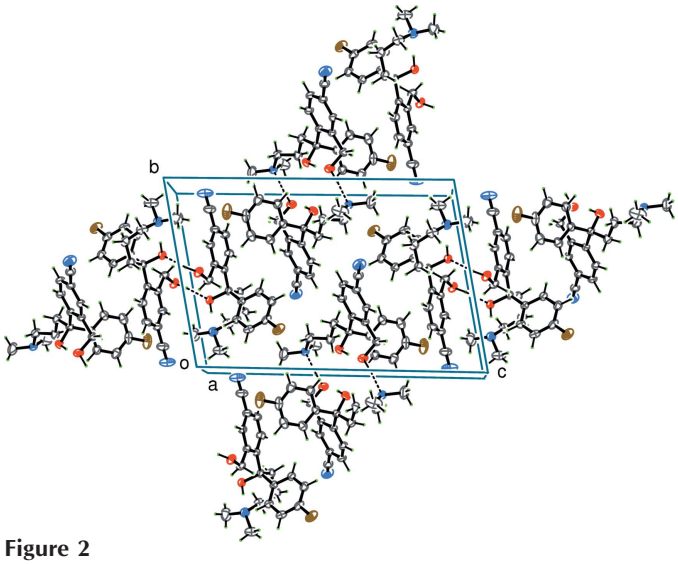


Figure 2
The two independent hydrogen-bonded centrosymmetric dimers in the crystal structure of (I). Hydrogen bonds are shown as dashed lines.

Experimental

1-Bromo-4-fluorobenzene (1.6 g, 9 mmol) in tetrahydrofuran (THF) was converted to the Grignard reagent, with ethyl magnesium bromide (1.3 g, 9.9 mmol), and added to 5-cyanophthalide (1.4 g, 8.8 mmol) in THF over a period of 3 h at 273–276 K. The mixture was stirred overnight at room temperature, and a THF solution of $Me_2N(CH_2)_3MgCl$ [from 8.8 mmol (1.1 g) $Me_2N(CH_2)_3Cl$] was added over a period of 6 h at 283–285 K. The mixture was stirred overnight and worked up to give a toluene solution of (I). After removal of the solvent, a colorless crystalline solid was obtained in 70% yield.

Crystal data

$C_{20}H_{23}FN_2O_2$
M_r = 342.41
Triclinic, $P\overline{1}$
a = 8.1512 (5) Å
b = 12.4991 (6) Å
c = 18.928 (1) Å
α = 100.708 (1)°
β = 96.0057 (8)°
γ = 94.896 (3)°
V = 1873.6 (2) Å^3
Z = 4
D_x = 1.214 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6362 reflections
θ = 2.2–27.4°
μ = 0.09 mm^{-1}
T = 295 (1) K
Block, colorless
0.41 × 0.28 × 0.24 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.947, T_{max} = 0.980
16267 measured reflections
8398 independent reflections
4412 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.023
θ_{max} = 27.4°
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.169
S = 1.00
6842 reflections
451 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[0.0028F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

F1—C13	1.360 (3)	O4—C24	1.421 (3)
F2—C33	1.366 (3)	N1—C7	1.136 (4)
O2—C4	1.405 (3)	N3—C27	1.140 (4)
O1—C1—C2—C3	−61.7 (3)	O3—C21—C22—C29	−115.5 (2)
O1—C1—C16—C17	64.0 (3)	O3—C21—C36—C37	−58.2 (2)
C5—C3—C4—O2	0.6 (3)	C22—C23—C24—O4	−66.8 (3)
C2—C3—C5—C6	0.1 (3)	C21—C36—C37—C38	144.1 (2)
C1—C16—C17—C18	−81.6 (3)	C36—C37—C38—N4	179.6 (2)
C16—C17—C18—N2	75.9 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H23···N2	0.94	1.74	2.671 (3)	172
O2—H22···O1^{i}	0.93	1.87	2.795 (2)	172
O3—H46···O4	1.04	1.61	2.634 (3)	171
O4—H45···N4^{ii}	0.99	1.70	2.686 (3)	171

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

The high-angle reflections ($\theta > 26°$) were not used in the refinement, because they were weak. The hydroxy H atoms were located in difference Fourier maps and included in the refinement based on the as-found O—H bond lengths, but their isotropic displacement parameters were refined and fixed in the final stage. The other H atoms were placed in calculated positions, with C—H = 0.95 Å, and included in the refinement in a riding model, with U_{iso}(H) = 1.2U_{eq}(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

References

Altomare, A., Burla, M., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hervas, I. & Artigas, F. (1998). *Eur. J. Pharmacol.* **358**, 9–18.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, 3-9-12 Akishima, Tokyo 196-8666, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.60. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.