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N-(2-Fluorophenyl)-2,6-dimethyl-1,3-dioxan-4-amine

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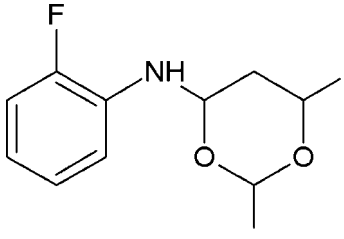
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.137; data-to-parameter ratio = 19.7.

In the title compound, $C_{12}H_{16}FNO_3$, the dioxane ring adopts a chair conformation with the methyl groups and amine N atom in equatorial positions. The best plane through the dioxane ring makes a dihedral angle of 43.16 (8)° with the phenyl ring. In the crystal, pairs of C—H···O hydrogen bonds link the molecules into centrosymmetric $R_2^2(8)$ dimers, which are linked into [100] chains by further C—H···O hydrogen bonds. The N—H group does not participate in hydrogen bonding.

Related literature

Dioxane rings are frequently encountered in structural motifs in many bioactive molecules such as cytotoxic agents (Aubele *et al.*, 2005) and antimuscarinic agents (Marucci *et al.*, 2005). For applications of this class of compounds, see: Wang, Yuan, Liu *et al.* (1996); Wang, Yuan, Lei & Liu (1996); Yuan *et al.* (2005). For related crystal structures, see: Chuprunov *et al.* (1981).



Experimental

Crystal data

$C_{12}H_{16}FNO_2$
M_r = 225.26
Monoclinic, $C2/c$
a = 19.6219 (13) Å
b = 8.1603 (6) Å
c = 15.2396 (10) Å
β = 95.950 (3)°
V = 2427.0 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.617, T_{max} = 0.746
11540 measured reflections
3020 independent reflections
1973 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.137
S = 1.03
3020 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O2^{i}	0.97	2.60	3.5563 (17)	169
C11—H11···O1^{ii}	0.93	2.54	3.473 (2)	177

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection. ZF and DV acknowledge the UGC (SAP–CAS) for the departmental facilties. ZF also thanks the UGC for a meritorious fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7132).

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supplementary materials

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N-(2-Fluorophenyl)-2,6-dimethyl-1,3-dioxan-4-amine

Zeenat Fatima, Gottimukkala Rambabu, Bandapalli Palakshi Reddy, Vijayaparthasarathi Vijayakumar and Devadasan Velmurugan

1. Comment

Oxygen heterocycles play a vital role as basic building blocks in pharmaceutical preparations. This class of compounds has useful insecticidal as well as anti-foaming properties (Yuan *et al.*, 2005). Dioxane rings are frequently encountered in structural motifs in many bioactive molecules such as cytotoxic agents (Aubele *et al.*, 2005) and antimuscarinic agents (Marucci *et al.*, 2005). As part of our studies in this area, we have undertaken a single-crystal structure determination of the title compound.

In the title compound, $C_{12}H_{16}F_1N_1O_3$ (Fig.1), pairs of C—H···O hydrogen bonds link the molecules into centrosymmetric $R_2^2(8)$ dimers (Fig.2). The dimers are linked into an infinite chain propagating along the 'a′ axis by further C—H···O hydrogen bonds. The dioxane ring adopts a *chair* conformation and the best plane through the dioxane ring makes a dihedral angle of 43.16 (8)° with the phenyl ring.

2. Experimental

To 2-fluoroaniline (1 mmol), acetaldehyde (3 mmol) was added dropwise and stirred for about 4 h at 0 °C. The progress of the reaction was monitored through TLC. The reaction mixture was washed with petroleum ether. Resultant was dissolved in diethylether and allowed to evaporate. Solid product obtained was recrystallized for diethylether solution to yield colourless blocks.

3. Refinement

The hydrogen atoms were placed in calculated positions with C—H = 0.93 Å to 0.98 Å refined in the riding model with fixed isotropic displacement parameters:U_{iso}(H) = 1.5U_{eq}(C) for methyl group and U_{iso}(H) = 1.2U_{eq}(C) for other groups.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

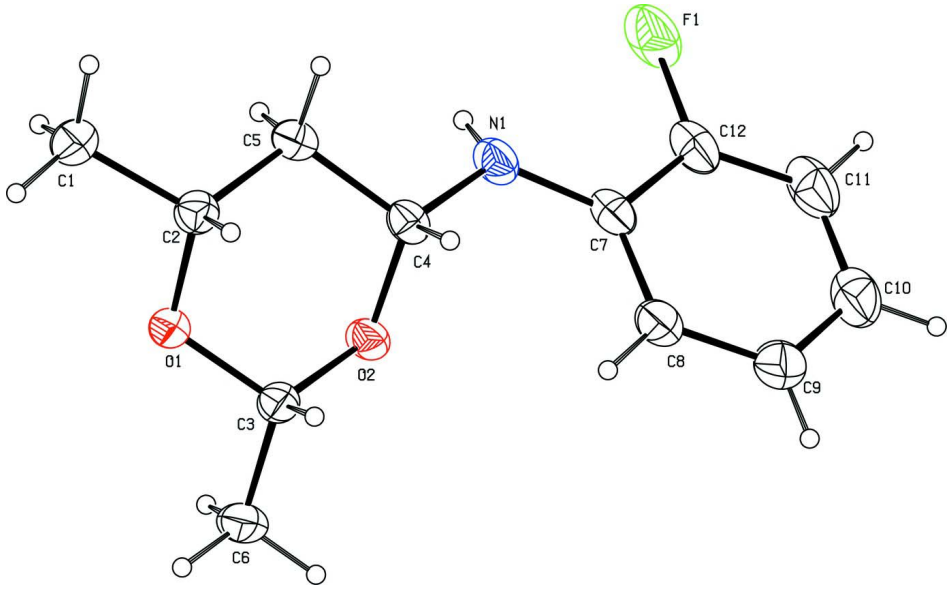

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

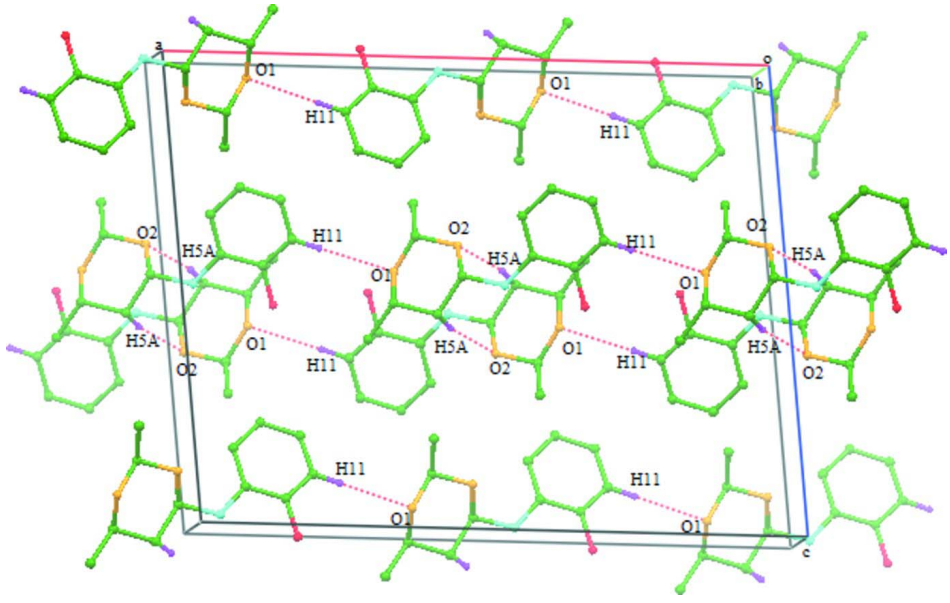

Figure 2

The crystal packing of the title compound viewed down *b* axis. H-atoms not involved in H-bonds have been excluded for clarity.

N-(2-Fluorophenyl)-2,6-dimethyl-1,3-dioxan-4-amine

Crystal data

$C_{12}H_{16}FNO_2$
M_r = 225.26
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 19.6219 (13) Å
b = 8.1603 (6) Å
c = 15.2396 (10) Å
β = 95.950 (3)°
V = 2427.0 (3) Å^3
Z = 8

$F(000) = 960$
$D_x = 1.233$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3020 reflections
$\theta = 2.7–28.4°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.617$, $T_{max} = 0.746$
11540 measured reflections
3020 independent reflections
1973 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.7°$
$h = -26\rightarrow25$
$k = -10\rightarrow10$
$l = -17\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.137$
$S = 1.03$
3020 reflections
153 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.5739P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
H1	−0.0550 (9)	0.220 (2)	1.0096 (12)	0.074 (5)*
H3	0.1172 (8)	0.1912 (19)	0.8533 (10)	0.058 (4)*
O1	0.13369 (5)	0.02783 (12)	0.94558 (6)	0.0592 (3)
O2	0.02809 (4)	0.13036 (11)	0.88947 (6)	0.0535 (3)
C4	0.02808 (7)	0.25397 (16)	0.95733 (9)	0.0526 (3)
H4	0.0476	0.3552	0.9360	0.063*
C5	0.07197 (7)	0.19735 (17)	1.03878 (9)	0.0545 (3)
H5A	0.0500	0.1056	1.0649	0.065*
H5B	0.0767	0.2854	1.0817	0.065*
N1	−0.04050 (7)	0.28505 (17)	0.97514 (9)	0.0617 (3)
C2	0.14205 (7)	0.14617 (18)	1.01572 (9)	0.0561 (3)

H2	0.1657	0.2425	0.9953	0.067*
C3	0.09501 (7)	0.0917 (2)	0.87025 (9)	0.0562 (4)
C7	−0.08824 (7)	0.35496 (17)	0.91321 (10)	0.0598 (4)
F1	−0.16772 (6)	0.33920 (15)	1.01790 (9)	0.1024 (4)
C6	0.09070 (10)	−0.0353 (3)	0.79920 (11)	0.0828 (6)
H6A	0.1360	−0.0620	0.7852	0.124*
H6B	0.0642	0.0064	0.7475	0.124*
H6C	0.0691	−0.1320	0.8192	0.124*
C12	−0.15451 (8)	0.3829 (2)	0.93551 (13)	0.0723 (5)
C1	0.18616 (9)	0.0692 (2)	1.09141 (10)	0.0745 (5)
H1A	0.2296	0.0394	1.0725	0.112*
H1B	0.1639	−0.0270	1.1108	0.112*
H1C	0.1931	0.1460	1.1392	0.112*
C8	−0.07505 (9)	0.4054 (2)	0.83014 (11)	0.0733 (5)
H8	−0.0318	0.3885	0.8120	0.088*
C9	−0.12519 (11)	0.4808 (2)	0.77326 (12)	0.0899 (6)
H9	−0.1151	0.5158	0.7180	0.108*
C11	−0.20465 (10)	0.4542 (3)	0.88042 (17)	0.0926 (7)
H11	−0.2482	0.4692	0.8979	0.111*
C10	−0.18988 (12)	0.5039 (3)	0.79822 (16)	0.0989 (7)
H10	−0.2236	0.5532	0.7596	0.119*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0587 (6)	0.0740 (6)	0.0451 (5)	0.0204 (5)	0.0063 (4)	−0.0013 (4)
O2	0.0435 (5)	0.0652 (5)	0.0531 (5)	0.0060 (4)	0.0112 (4)	−0.0052 (4)
C4	0.0477 (8)	0.0510 (7)	0.0608 (8)	0.0028 (6)	0.0144 (6)	−0.0025 (6)
C5	0.0596 (9)	0.0530 (7)	0.0523 (8)	0.0005 (6)	0.0128 (6)	−0.0070 (6)
N1	0.0521 (7)	0.0665 (7)	0.0695 (8)	0.0106 (6)	0.0208 (6)	−0.0006 (7)
C2	0.0533 (8)	0.0627 (8)	0.0526 (8)	0.0015 (6)	0.0068 (6)	0.0016 (6)
C3	0.0474 (8)	0.0767 (9)	0.0461 (7)	0.0128 (7)	0.0113 (6)	0.0061 (7)
C7	0.0516 (8)	0.0563 (7)	0.0721 (10)	0.0061 (6)	0.0093 (7)	−0.0185 (7)
F1	0.0756 (7)	0.1085 (8)	0.1316 (10)	0.0213 (6)	0.0513 (7)	0.0091 (7)
C6	0.0734 (11)	0.1247 (15)	0.0507 (8)	0.0338 (10)	0.0076 (8)	−0.0165 (9)
C12	0.0542 (9)	0.0690 (9)	0.0948 (12)	0.0079 (7)	0.0129 (9)	−0.0202 (9)
C1	0.0746 (11)	0.0911 (11)	0.0554 (9)	0.0164 (9)	−0.0043 (8)	−0.0047 (8)
C8	0.0689 (10)	0.0814 (10)	0.0697 (10)	0.0192 (8)	0.0081 (8)	−0.0148 (8)
C9	0.1005 (16)	0.0957 (13)	0.0703 (11)	0.0248 (11)	−0.0069 (10)	−0.0226 (10)
C11	0.0544 (10)	0.0973 (13)	0.1236 (18)	0.0145 (9)	−0.0033 (11)	−0.0348 (13)
C10	0.0798 (14)	0.1034 (14)	0.1047 (16)	0.0290 (11)	−0.0316 (12)	−0.0404 (13)

Geometric parameters (Å, º)

O1—C3	1.4087 (16)	C7—C12	1.396 (2)
O1—C2	1.4373 (17)	F1—C12	1.356 (2)
O2—C3	1.4105 (16)	C6—H6A	0.9600
O2—C4	1.4446 (16)	C6—H6B	0.9600
C4—N1	1.4228 (17)	C6—H6C	0.9600
C4—C5	1.508 (2)	C12—C11	1.357 (3)

C4—H4	0.9800	C1—H1A	0.9600
C5—C2	1.513 (2)	C1—H1B	0.9600
C5—H5A	0.9700	C1—H1C	0.9600
C5—H5B	0.9700	C8—C9	1.386 (2)
N1—C7	1.382 (2)	C8—H8	0.9300
N1—H1	0.820 (18)	C9—C10	1.375 (3)
C2—C1	1.506 (2)	C9—H9	0.9300
C2—H2	0.9800	C11—C10	1.376 (3)
C3—C6	1.495 (2)	C11—H11	0.9300
C3—H3	0.969 (16)	C10—H10	0.9300
C7—C8	1.381 (2)		

C3—O1—C2	111.59 (11)	C8—C7—C12	116.24 (16)
C3—O2—C4	111.98 (10)	N1—C7—C12	118.92 (15)
N1—C4—O2	109.35 (11)	C3—C6—H6A	109.5
N1—C4—C5	111.61 (11)	C3—C6—H6B	109.5
O2—C4—C5	109.44 (10)	H6A—C6—H6B	109.5
N1—C4—H4	108.8	C3—C6—H6C	109.5
O2—C4—H4	108.8	H6A—C6—H6C	109.5
C5—C4—H4	108.8	H6B—C6—H6C	109.5
C4—C5—C2	110.40 (11)	F1—C12—C11	119.33 (17)
C4—C5—H5A	109.6	F1—C12—C7	117.05 (16)
C2—C5—H5A	109.6	C11—C12—C7	123.6 (2)
C4—C5—H5B	109.6	C2—C1—H1A	109.5
C2—C5—H5B	109.6	C2—C1—H1B	109.5
H5A—C5—H5B	108.1	H1A—C1—H1B	109.5
C7—N1—C4	122.05 (13)	C2—C1—H1C	109.5
C7—N1—H1	116.8 (13)	H1A—C1—H1C	109.5
C4—N1—H1	113.9 (13)	H1B—C1—H1C	109.5
O1—C2—C1	107.55 (12)	C7—C8—C9	121.15 (18)
O1—C2—C5	108.78 (11)	C7—C8—H8	119.4
C1—C2—C5	113.66 (12)	C9—C8—H8	119.4
O1—C2—H2	108.9	C10—C9—C8	120.2 (2)
C1—C2—H2	108.9	C10—C9—H9	119.9
C5—C2—H2	108.9	C8—C9—H9	119.9
O1—C3—O2	110.36 (10)	C12—C11—C10	118.84 (19)
O1—C3—C6	108.61 (13)	C12—C11—H11	120.6
O2—C3—C6	108.76 (13)	C10—C11—H11	120.6
O1—C3—H3	107.9 (9)	C9—C10—C11	119.91 (19)
O2—C3—H3	108.7 (9)	C9—C10—H10	120.0
C6—C3—H3	112.5 (9)	C11—C10—H10	120.0
C8—C7—N1	124.81 (14)		

C3—O2—C4—N1	−178.72 (11)	C4—N1—C7—C8	−1.8 (2)
C3—O2—C4—C5	−56.18 (14)	C4—N1—C7—C12	−179.59 (13)
N1—C4—C5—C2	173.31 (11)	C8—C7—C12—F1	−177.83 (14)
O2—C4—C5—C2	52.12 (15)	N1—C7—C12—F1	0.2 (2)
O2—C4—N1—C7	−65.93 (17)	C8—C7—C12—C11	0.4 (2)
C5—C4—N1—C7	172.83 (12)	N1—C7—C12—C11	178.40 (16)

C3—O1—C2—C1	−177.76 (12)	N1—C7—C8—C9	−177.28 (16)
C3—O1—C2—C5	58.73 (14)	C12—C7—C8—C9	0.6 (2)
C4—C5—C2—O1	−53.33 (14)	C7—C8—C9—C10	−1.2 (3)
C4—C5—C2—C1	−173.11 (12)	F1—C12—C11—C10	177.44 (17)
C2—O1—C3—O2	−62.98 (16)	C7—C12—C11—C10	−0.7 (3)
C2—O1—C3—C6	177.87 (13)	C8—C9—C10—C11	0.9 (3)
C4—O2—C3—O1	61.46 (15)	C12—C11—C10—C9	0.1 (3)
C4—O2—C3—C6	−179.49 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O2^{i}	0.97	2.60	3.5563 (17)	169
C11—H11···O1^{ii}	0.93	2.54	3.473 (2)	177

Symmetry codes: (i) −*x*, −*y*, −*z*+2; (ii) *x*−1/2, *y*+1/2, *z*.