

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

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

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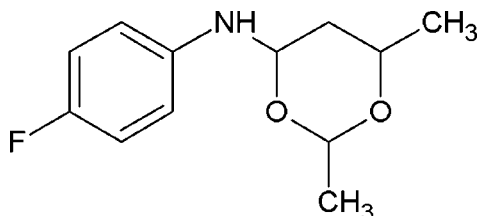
Received 10 September 2013; accepted 26 September 2013

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.129; data-to-parameter ratio = 19.8.

In the title compound,  $\text{C}_{12}\text{H}_{16}\text{FNO}_2$ , the dioxane ring adopts a chair conformation with the methyl substituents and the C—N bond in equatorial orientations. Its mean plane subtends a dihedral angle of  $40.17(6)^\circ$  with the benzene ring. In the crystal, weak N—H...F hydrogen bonds link the molecules into  $C(7)$  chains propagating in  $[100]$ .

### Related literature

For a related structure and background to dioxanes, see: Fatima *et al.* (2013).



### Experimental

#### Crystal data

 $\text{C}_{12}\text{H}_{16}\text{FNO}_2$  $M_r = 225.26$ 

Monoclinic,  $P2_1/n$   
 $a = 10.4924(10)$  Å  
 $b = 10.0614(10)$  Å  
 $c = 11.0379(11)$  Å  
 $\beta = 90.136(2)^\circ$   
 $V = 1165.2(2)$  Å<sup>3</sup>

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.25 \times 0.20 \times 0.15$  mm

#### Data collection

Bruker SMART APEXII area-detector diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 2008)  
 $T_{\min} = 0.649$ ,  $T_{\max} = 0.746$

10977 measured reflections  
2910 independent reflections  
2179 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.129$   
 $S = 1.01$   
2910 reflections

147 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.15$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{F1}^i$	0.86	2.48	3.1556 (14)	136

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

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 facilities. ZF also thanks the UGC for a meritorious fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7138).

### References

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## supporting information

*Acta Cryst.* (2013). E69, o1602 [doi:10.1107/S1600536813026561]

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

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

### S1. Comment

As part of our ongoing studies of dioxane derivatives with possible biological activity (Fatima *et al.*, 2013), we now describe the single-crystal structure determination of the title compound.

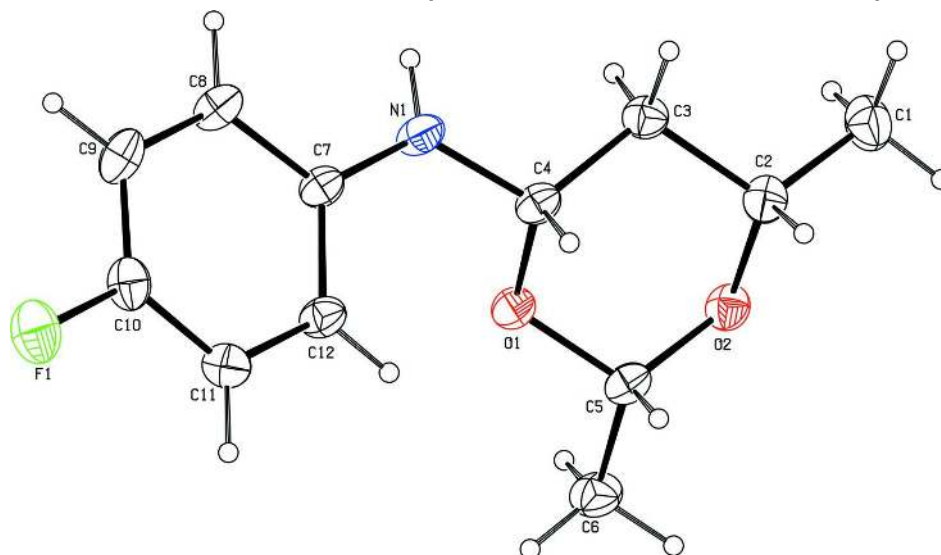
Molecules of the title compound, C<sub>12</sub>H<sub>16</sub>N<sub>1</sub>O<sub>2</sub>F<sub>1</sub>, (Fig.1) are linked by intermolecular N—H...F hydrogen bonds into infinite chains propagating along 'a' axis (Fig. 2). The dioxane ring (O1/O2/C2—C5) adopts a *chair* conformation and the best plane through the dioxane ring makes a dihedral angle of 40.17 (6)° with the phenyl ring (C7—C12).

### S2. Experimental

To 4-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 with diethylether.

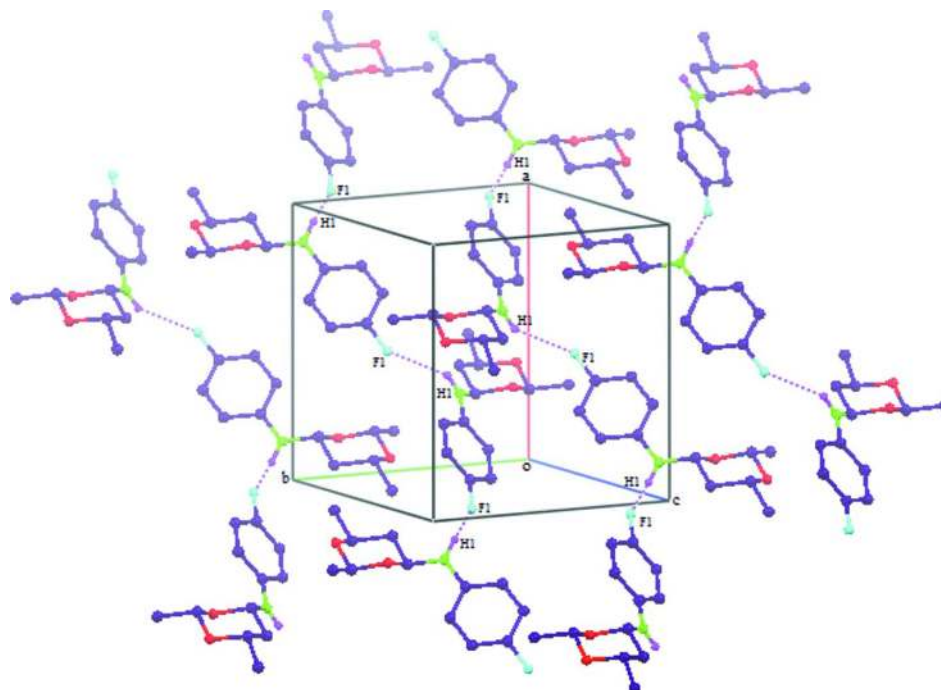
### S3. Refinement

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:  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl group and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for other groups.



**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 *a* axis. H-atoms not involved in H-bonds have been excluded for clarity.

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

#### *Crystal data*

$C_{12}H_{16}FNO_2$

$M_r = 225.26$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 10.4924$  (10) Å

$b = 10.0614$  (10) Å

$c = 11.0379$  (11) Å

$\beta = 90.136$  (2)°

$V = 1165.2$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 480$

$D_x = 1.284$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2910 reflections

$\theta = 2.7$ – $28.4$ °

$\mu = 0.10$  mm<sup>-1</sup>

$T = 293$  K

Block, colourless

$0.25 \times 0.20 \times 0.15$  mm

#### *Data collection*

Bruker SMART APEXII area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  and  $\phi$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.649$ ,  $T_{\max} = 0.746$

10977 measured reflections

2910 independent reflections

2179 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 28.4$ °,  $\theta_{\min} = 2.7$ °

$h = -13 \rightarrow 13$

$k = -13 \rightarrow 13$

$l = -14 \rightarrow 14$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.129$   
 $S = 1.01$   
 2910 reflections  
 147 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-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.1851P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.85420 (8)	0.16432 (8)	0.95248 (8)	0.0469 (2)
O2	0.91002 (9)	0.28191 (9)	0.77911 (8)	0.0509 (2)
C3	0.91619 (12)	0.04131 (13)	0.77606 (11)	0.0491 (3)
H3A	0.8970	-0.0356	0.7263	0.059*
H3B	1.0045	0.0351	0.8018	0.059*
C5	0.82700 (12)	0.27629 (12)	0.87937 (12)	0.0482 (3)
H5	0.7386	0.2718	0.8509	0.058*
N1	0.84979 (10)	-0.06797 (11)	0.96310 (10)	0.0525 (3)
H1	0.9177	-0.1148	0.9546	0.063*
C4	0.83024 (11)	0.04228 (11)	0.88605 (11)	0.0453 (3)
H4	0.7413	0.0421	0.8587	0.054*
C2	0.89697 (13)	0.16759 (13)	0.70224 (12)	0.0514 (3)
H2	0.8111	0.1669	0.6672	0.062*
F1	0.49573 (9)	-0.21970 (11)	1.30521 (8)	0.0788 (3)
C7	0.76261 (11)	-0.10329 (11)	1.05237 (11)	0.0442 (3)
C8	0.77421 (13)	-0.22492 (13)	1.11186 (14)	0.0556 (3)
H8	0.8424	-0.2806	1.0939	0.067*
C9	0.68551 (15)	-0.26374 (15)	1.19728 (14)	0.0622 (4)
H9	0.6935	-0.3450	1.2367	0.075*
C12	0.66199 (11)	-0.02028 (12)	1.08440 (12)	0.0497 (3)
H12	0.6546	0.0628	1.0481	0.060*
C11	0.57272 (13)	-0.05951 (14)	1.16951 (12)	0.0539 (3)
H11	0.5051	-0.0041	1.1898	0.065*
C6	0.84563 (15)	0.39947 (14)	0.95358 (14)	0.0630 (4)
H6A	0.9318	0.4026	0.9830	0.095*

H6B	0.7879	0.3985	1.0209	0.095*
H6C	0.8292	0.4763	0.9044	0.095*
C10	0.58580 (13)	-0.18076 (15)	1.22298 (12)	0.0553 (3)
C1	0.99285 (17)	0.18267 (17)	0.60184 (14)	0.0693 (4)
H1A	0.9765	0.2637	0.5585	0.104*
H1B	0.9860	0.1086	0.5474	0.104*
H1C	1.0772	0.1854	0.6357	0.104*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0494 (5)	0.0386 (4)	0.0529 (5)	-0.0015 (3)	-0.0005 (4)	0.0021 (3)
O2	0.0567 (5)	0.0407 (5)	0.0551 (5)	-0.0081 (4)	-0.0008 (4)	0.0033 (4)
C3	0.0510 (7)	0.0415 (6)	0.0550 (7)	-0.0041 (5)	0.0010 (5)	-0.0027 (5)
C5	0.0448 (6)	0.0382 (6)	0.0615 (7)	0.0007 (5)	-0.0030 (5)	0.0043 (5)
N1	0.0438 (5)	0.0426 (5)	0.0712 (7)	0.0076 (4)	0.0087 (5)	0.0123 (5)
C4	0.0402 (6)	0.0372 (6)	0.0584 (7)	-0.0023 (4)	-0.0015 (5)	0.0035 (5)
C2	0.0558 (7)	0.0469 (7)	0.0514 (7)	-0.0082 (5)	-0.0057 (6)	0.0004 (5)
F1	0.0799 (6)	0.0875 (7)	0.0691 (6)	-0.0231 (5)	0.0156 (5)	0.0142 (5)
C7	0.0412 (6)	0.0360 (6)	0.0555 (7)	-0.0029 (4)	-0.0031 (5)	0.0024 (5)
C8	0.0480 (7)	0.0442 (7)	0.0747 (9)	0.0025 (5)	-0.0051 (6)	0.0114 (6)
C9	0.0643 (8)	0.0516 (7)	0.0705 (9)	-0.0076 (6)	-0.0097 (7)	0.0214 (7)
C12	0.0499 (6)	0.0372 (6)	0.0621 (7)	0.0001 (5)	0.0030 (5)	0.0039 (5)
C11	0.0503 (7)	0.0510 (7)	0.0603 (8)	-0.0029 (5)	0.0048 (6)	-0.0042 (6)
C6	0.0729 (9)	0.0416 (7)	0.0746 (9)	-0.0004 (6)	0.0029 (7)	-0.0027 (6)
C10	0.0554 (7)	0.0602 (8)	0.0502 (7)	-0.0158 (6)	0.0003 (6)	0.0050 (6)
C1	0.0872 (11)	0.0654 (9)	0.0554 (8)	-0.0120 (8)	0.0075 (8)	0.0026 (7)

*Geometric parameters (Å, °)*

O1—C5	1.4145 (14)	C7—C8	1.3940 (17)
O1—C4	1.4517 (14)	C7—C12	1.3924 (17)
O2—C5	1.4111 (16)	C8—C9	1.383 (2)
O2—C2	1.4357 (15)	C8—H8	0.9300
C3—C4	1.5141 (17)	C9—C10	1.369 (2)
C3—C2	1.5226 (18)	C9—H9	0.9300
C3—H3A	0.9700	C12—C11	1.3856 (17)
C3—H3B	0.9700	C12—H12	0.9300
C5—C6	1.4981 (19)	C11—C10	1.362 (2)
C5—H5	0.9800	C11—H11	0.9300
N1—C7	1.3922 (15)	C6—H6A	0.9600
N1—C4	1.4125 (15)	C6—H6B	0.9600
N1—H1	0.8600	C6—H6C	0.9600
C4—H4	0.9800	C1—H1A	0.9600
C2—C1	1.5061 (19)	C1—H1B	0.9600
C2—H2	0.9800	C1—H1C	0.9600
F1—C10	1.3691 (15)		

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C5—O1—C4	110.55 (9)	C8—C7—C12	118.19 (12)
C5—O2—C2	111.92 (9)	N1—C7—C12	121.77 (11)
C4—C3—C2	110.20 (10)	C9—C8—C7	120.73 (13)
C4—C3—H3A	109.6	C9—C8—H8	119.6
C2—C3—H3A	109.6	C7—C8—H8	119.6
C4—C3—H3B	109.6	C10—C9—C8	119.02 (13)
C2—C3—H3B	109.6	C10—C9—H9	120.5
H3A—C3—H3B	108.1	C8—C9—H9	120.5
O2—C5—O1	110.80 (9)	C11—C12—C7	121.03 (12)
O2—C5—C6	108.40 (10)	C11—C12—H12	119.5
O1—C5—C6	108.73 (11)	C7—C12—H12	119.5
O2—C5—H5	109.6	C10—C11—C12	118.78 (13)
O1—C5—H5	109.6	C10—C11—H11	120.6
C6—C5—H5	109.6	C12—C11—H11	120.6
C7—N1—C4	122.13 (10)	C5—C6—H6A	109.5
C7—N1—H1	118.9	C5—C6—H6B	109.5
C4—N1—H1	118.9	H6A—C6—H6B	109.5
N1—C4—O1	109.60 (10)	C5—C6—H6C	109.5
N1—C4—C3	113.06 (10)	H6A—C6—H6C	109.5
O1—C4—C3	107.92 (9)	H6B—C6—H6C	109.5
N1—C4—H4	108.7	C11—C10—C9	122.19 (12)
O1—C4—H4	108.7	C11—C10—F1	118.33 (13)
C3—C4—H4	108.7	C9—C10—F1	119.47 (13)
O2—C2—C1	106.93 (10)	C2—C1—H1A	109.5
O2—C2—C3	109.87 (10)	C2—C1—H1B	109.5
C1—C2—C3	112.96 (12)	H1A—C1—H1B	109.5
O2—C2—H2	109.0	C2—C1—H1C	109.5
C1—C2—H2	109.0	H1A—C1—H1C	109.5
C3—C2—H2	109.0	H1B—C1—H1C	109.5
C8—C7—N1	120.04 (11)		
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C2—O2—C5—O1	61.43 (13)	C4—C3—C2—C1	171.55 (11)
C2—O2—C5—C6	-179.33 (10)	C4—N1—C7—C8	168.84 (12)
C4—O1—C5—O2	-64.23 (12)	C4—N1—C7—C12	-10.76 (19)
C4—O1—C5—C6	176.73 (10)	N1—C7—C8—C9	-177.40 (13)
C7—N1—C4—O1	74.98 (14)	C12—C7—C8—C9	2.2 (2)
C7—N1—C4—C3	-164.58 (11)	C7—C8—C9—C10	-0.1 (2)
C5—O1—C4—N1	-176.11 (9)	C8—C7—C12—C11	-2.58 (19)
C5—O1—C4—C3	60.37 (12)	N1—C7—C12—C11	177.03 (12)
C2—C3—C4—N1	-175.84 (10)	C7—C12—C11—C10	0.8 (2)
C2—C3—C4—O1	-54.45 (12)	C12—C11—C10—C9	1.4 (2)
C5—O2—C2—C1	-178.06 (10)	C12—C11—C10—F1	-178.74 (12)
C5—O2—C2—C3	-55.13 (13)	C8—C9—C10—C11	-1.7 (2)
C4—C3—C2—O2	52.25 (14)	C8—C9—C10—F1	178.39 (12)

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*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ F1 <sup>i</sup>	0.86	2.48	3.1556 (14)	136

Symmetry code: (i)  $x+1/2, -y-1/2, z-1/2$ .