Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(4-Fluorophenyl)-2,2-dimethylpropanamide

Zheng Fang,[a] Feng Zhang,[a] Bao-hua Zou[a] and Kai Guo[b]*

[a]School of Pharmaceutical Sciences, Nanjing University of Technology, Puzhu South Road No. 30 Nanjing, Nanjing 210009, People's Republic of China, and [b]College of Life Science and Pharmaceutical Engineering, Nanjing University of Technology, Puzhu South Road No. 30 Nanjing, Nanjing 210009, People's Republic of China
Correspondence e-mail: kaiguo@njut.edu.cn

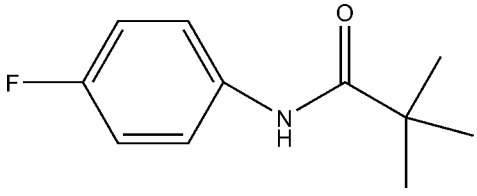
Received 13 April 2012; accepted 7 May 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.062; wR factor = 0.155; data-to-parameter ratio = 15.8.

The crystal packing in the title compound, $C_{11}H_{14}FNO$, features N—H···O hydrogen bonds, resulting in chains of molecules running parallel to the c axis. The dihedral angle between the ring and the amide group is 39.1 (3)°.

Related literature

The title compound is an intermediate in the synthesis of ezetimibe, which inhibits the absorption of cholesterol from the intestine, see: Rosenblum *et al.* (1998). For the synthesis, see: Wang *et al.* (2008). For a related structure, see: Gowda *et al.* (2007).



Experimental

Crystal data

$C_{11}H_{14}FNO$
M_r = 195.23
Monoclinic, $P2_1/c$
a = 9.5750 (19) Å
b = 13.027 (3) Å
c = 8.8340 (18) Å
β = 92.07 (3)°
V = 1101.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.974, T_{max} = 0.991
4219 measured reflections
2025 independent reflections
1091 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.155
S = 1.00
2025 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O^{i}	0.86	2.17	2.990 (3)	159

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

This research was supported financially by the College of Life Science and Pharmaceutical Engineering, Nanjing University of Technology, the 973 project (2012CB725204) and the Key Basic Research Program of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2534).

References

Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Gowda, B. T., Foro, S. & Fuess, H. (2007). *Acta Cryst.* E**63**, o2329–o2330.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Rosenblum, S. B., Huynh, T., Afonso, A., Davis, H. R., Yumibe, N., Clader, J. W. & Burnett, D. A. (1998). *J. Med. Chem.* **41**, 973–980.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Wang, Y., Zhang, H., Huang, W., Kong, J., Zhou, J. & Zhang, B. (2008). *Eur. J. Med. Chem.* **44**, 1638–1643.

supplementary materials

Acta Cryst. (2012). E**68**, o1757 [doi:10.1107/S1600536812020570]

N-(4-Fluorophenyl)-2,2-dimethylpropanamide

Zheng Fang, Feng Zhang, Bao-hua Zou and Kai Guo

Comment

Ezetimibe is a biologically active molecule and reasearch has shown it to have the useful property of inhibiting the absorption of cholesterol from the intestine (Rosenblum *et al.*, 1998). As a part of our studies on the synthesis of Ezetimibe, the title compound (fIG. 1) which is one of the derivates of an intermediate, has been synthesized and its crystal structure is reported in this paper. The crystal structure of the title compound is stabilized by N—H0A···O hydrogen bonds resulting in chains of molecules running parallel to the *c*-axis (Fig. 1 and Tab. 1). The bond distances and angles in the title molecule are in excellent agreement with the corresponding bond distances and angles reported for its chloro-analogue (Gowda *et al.*, 2007).

Experimental

To a solution of 4-fluoroaniline (13.32 g, 0.12 mol) in CH_2Cl_2(20 ml) were added 4-dimethylaminopyridine (1.2 g, 0.01 mol) and Et_3N (42.3 ml, 0.31 mol) and cooled the reaction mixture to 273 K. A solution of pivaloyl chloride (14.4 g, 0.12 mol) in CH_2Cl_2 (150 ml) was added dropwise over 1 h and the mixture was heated to reflux. After 12 h, H_2O and H_2SO_4 (2 N, 75 ml) were added, separated the layers and washed the organic layer sequentially with NaOH (10%), NaCl (satd.) and water. The organic layer was dried over $MgSO_4$ and concentrated to obtain the title compound as a yellow solid product in pure form following the procedure reported earlier (Wang *et al.*, 2008). Crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with N—H = 0.86 Å and C—H = 0.93 and 0.96 Å, for aryl and methyl H-atoms, respectively. The U_{iso}(H) were allowed at 1.2U_{eq}(N/C).

Computing details

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software* (Enraf–Nonius, 1989); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

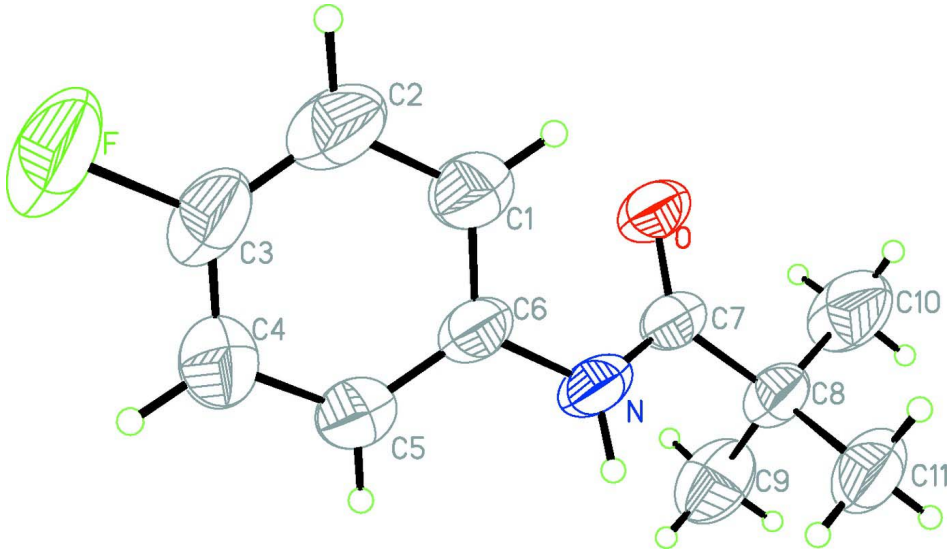


Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

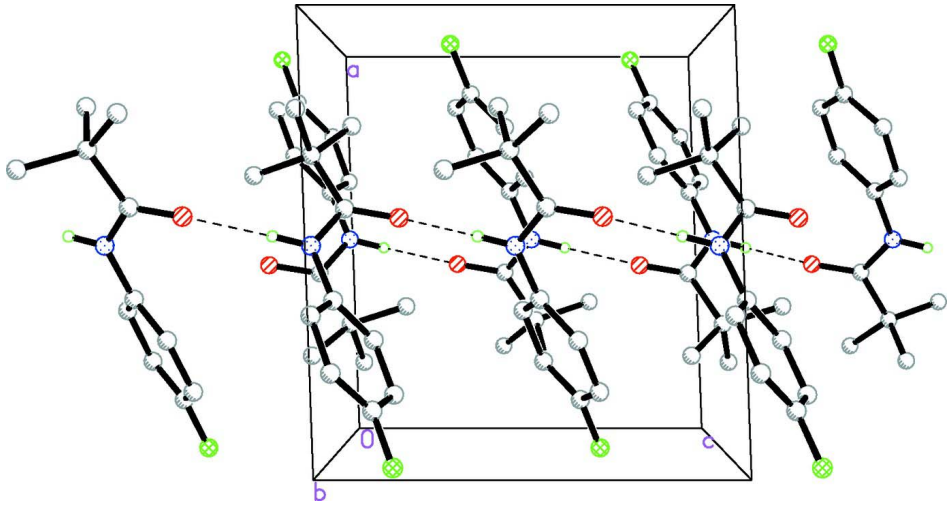


Figure 2

A view of the N—H···O hydrogen bonds (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen- bonding were omitted for clarity.

N-(4-Fluorophenyl)-2,2-dimethylpropanamide

Crystal data

$C_{11}H_{14}FNO$
$M_r = 195.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.5750\ (19)$ Å
$b = 13.027\ (3)$ Å
$c = 8.8340\ (18)$ Å
$\beta = 92.07\ (3)°$
$V = 1101.2\ (4)$ Å^3
$Z = 4$
$F(000) = 416$
$D_x = 1.178$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–13°$
$\mu = 0.09$ mm^{-1}

T = 293 K
Block, yellow

0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.974, T_{max} = 0.991
4219 measured reflections

2025 independent reflections
1091 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 25.4°, θ_{min} = 2.1°
h = −11→11
k = −15→15
l = 0→10
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.155
S = 1.00
2025 reflections
128 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
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.020 (5)

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}
O	0.55665 (18)	0.65320 (14)	0.18951 (19)	0.0619 (6)
F	0.01604 (17)	0.92797 (16)	0.1759 (2)	0.1032 (7)
N	0.4924 (2)	0.74067 (17)	−0.0211 (2)	0.0588 (7)
H0A	0.5136	0.7547	−0.1126	0.071*
C1	0.2795 (3)	0.7359 (2)	0.1236 (3)	0.0638 (8)
H1A	0.2982	0.6683	0.1514	0.077*
C2	0.1603 (3)	0.7837 (3)	0.1726 (4)	0.0728 (9)
H2A	0.0995	0.7493	0.2349	0.087*
C3	0.1340 (3)	0.8806 (3)	0.1284 (3)	0.0693 (9)
C4	0.2195 (3)	0.9346 (2)	0.0395 (3)	0.0731 (9)
H4A	0.1981	1.0016	0.0110	0.088*
C5	0.3409 (3)	0.8869 (2)	−0.0081 (3)	0.0640 (8)

H5A	0.4019	0.9225	−0.0687	0.077*
C6	0.3705 (3)	0.7878 (2)	0.0339 (3)	0.0507 (7)
C7	0.5775 (3)	0.6763 (2)	0.0568 (3)	0.0496 (7)
C8	0.7023 (3)	0.6356 (2)	−0.0266 (3)	0.0560 (7)
C9	0.8029 (3)	0.7255 (3)	−0.0500 (4)	0.0864 (11)
H9A	0.8316	0.7537	0.0465	0.130*
H9B	0.7565	0.7775	−0.1102	0.130*
H9C	0.8834	0.7014	−0.1011	0.130*
C10	0.7752 (4)	0.5529 (3)	0.0688 (4)	0.1008 (13)
H10A	0.7121	0.4967	0.0829	0.151*
H10B	0.8036	0.5809	0.1657	0.151*
H10C	0.8560	0.5289	0.0181	0.151*
C11	0.6568 (3)	0.5904 (2)	−0.1808 (3)	0.0759 (10)
H11A	0.5945	0.5338	−0.1662	0.114*
H11B	0.7375	0.5669	−0.2321	0.114*
H11C	0.6097	0.6422	−0.2408	0.114*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.0662 (12)	0.0774 (14)	0.0430 (11)	0.0127 (10)	0.0146 (9)	0.0049 (10)
F	0.0656 (12)	0.1364 (17)	0.1092 (15)	0.0362 (11)	0.0246 (11)	−0.0114 (13)
N	0.0603 (14)	0.0756 (15)	0.0418 (12)	0.0164 (13)	0.0204 (11)	0.0052 (12)
C1	0.0579 (17)	0.071 (2)	0.0637 (18)	0.0048 (15)	0.0160 (15)	0.0065 (15)
C2	0.0509 (17)	0.099 (3)	0.070 (2)	0.0022 (18)	0.0194 (15)	0.0051 (19)
C3	0.0507 (17)	0.095 (2)	0.0631 (18)	0.0197 (18)	0.0119 (15)	−0.0072 (18)
C4	0.073 (2)	0.073 (2)	0.073 (2)	0.0218 (18)	0.0069 (17)	0.0059 (17)
C5	0.0660 (19)	0.071 (2)	0.0564 (17)	0.0073 (16)	0.0177 (14)	0.0078 (15)
C6	0.0519 (15)	0.0640 (18)	0.0370 (13)	0.0053 (14)	0.0113 (12)	−0.0002 (13)
C7	0.0537 (16)	0.0574 (17)	0.0384 (14)	0.0016 (13)	0.0114 (12)	−0.0005 (13)
C8	0.0548 (16)	0.0679 (19)	0.0461 (15)	0.0087 (14)	0.0119 (12)	−0.0050 (14)
C9	0.0548 (18)	0.112 (3)	0.093 (3)	−0.0102 (18)	0.0193 (18)	−0.023 (2)
C10	0.097 (2)	0.125 (3)	0.082 (2)	0.059 (2)	0.022 (2)	0.014 (2)
C11	0.080 (2)	0.082 (2)	0.067 (2)	0.0074 (18)	0.0224 (16)	−0.0181 (17)

Geometric parameters (Å, º)

O—C7	1.233 (3)	C5—H5A	0.9300
F—C3	1.366 (3)	C7—C8	1.522 (3)
N—C7	1.342 (3)	C8—C10	1.522 (4)
N—C6	1.420 (3)	C8—C11	1.533 (4)
N—H0A	0.8600	C8—C9	1.535 (4)
C1—C6	1.376 (4)	C9—H9A	0.9600
C1—C2	1.383 (4)	C9—H9B	0.9600
C1—H1A	0.9300	C9—H9C	0.9600
C2—C3	1.343 (4)	C10—H10A	0.9600
C2—H2A	0.9300	C10—H10B	0.9600
C3—C4	1.352 (4)	C10—H10C	0.9600
C4—C5	1.397 (4)	C11—H11A	0.9600
C4—H4A	0.9300	C11—H11B	0.9600

C5—C6	1.371 (4)	C11—H11C	0.9600
C7—N—C6	125.9 (2)	C7—C8—C10	109.3 (2)
C7—N—H0A	117.1	C7—C8—C11	111.2 (2)
C6—N—H0A	117.1	C10—C8—C11	109.3 (2)
C6—C1—C2	120.4 (3)	C7—C8—C9	107.9 (2)
C6—C1—H1A	119.8	C10—C8—C9	109.7 (3)
C2—C1—H1A	119.8	C11—C8—C9	109.4 (2)
C3—C2—C1	118.7 (3)	C8—C9—H9A	109.5
C3—C2—H2A	120.6	C8—C9—H9B	109.5
C1—C2—H2A	120.6	H9A—C9—H9B	109.5
C2—C3—C4	123.1 (3)	C8—C9—H9C	109.5
C2—C3—F	118.9 (3)	H9A—C9—H9C	109.5
C4—C3—F	117.9 (3)	H9B—C9—H9C	109.5
C3—C4—C5	118.2 (3)	C8—C10—H10A	109.5
C3—C4—H4A	120.9	C8—C10—H10B	109.5
C5—C4—H4A	120.9	H10A—C10—H10B	109.5
C6—C5—C4	120.2 (3)	C8—C10—H10C	109.5
C6—C5—H5A	119.9	H10A—C10—H10C	109.5
C4—C5—H5A	119.9	H10B—C10—H10C	109.5
C5—C6—C1	119.3 (2)	C8—C11—H11A	109.5
C5—C6—N	118.6 (2)	C8—C11—H11B	109.5
C1—C6—N	122.0 (3)	H11A—C11—H11B	109.5
O—C7—N	121.6 (2)	C8—C11—H11C	109.5
O—C7—C8	122.1 (2)	H11A—C11—H11C	109.5
N—C7—C8	116.3 (2)	H11B—C11—H11C	109.5
C6—C1—C2—C3	1.4 (5)	C7—N—C6—C5	142.3 (3)
C1—C2—C3—C4	−0.9 (5)	C7—N—C6—C1	−39.9 (4)
C1—C2—C3—F	179.7 (3)	C6—N—C7—O	−1.2 (4)
C2—C3—C4—C5	0.0 (5)	C6—N—C7—C8	−179.9 (2)
F—C3—C4—C5	179.4 (2)	O—C7—C8—C10	9.3 (4)
C3—C4—C5—C6	0.5 (4)	N—C7—C8—C10	−172.0 (3)
C4—C5—C6—C1	0.0 (4)	O—C7—C8—C11	130.1 (3)
C4—C5—C6—N	177.8 (2)	N—C7—C8—C11	−51.3 (3)
C2—C1—C6—C5	−0.9 (4)	O—C7—C8—C9	−110.0 (3)
C2—C1—C6—N	−178.7 (2)	N—C7—C8—C9	68.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0*A*···O^{i}	0.86	2.17	2.990 (3)	159
C1—H1*A*···O	0.93	2.49	2.904 (3)	107

Symmetry code: (i) *x*, −*y*+3/2, *z*−1/2.