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2,2-Dibromo-*N*-(4-fluorophenyl)-acetamide

Xiangjun Qian,[a] Zheng Fang,[a] Shuxin Bao,[a] Kai Guo[b]* and Ping Wei[b]

[a]School of Pharmaceutical Sciences, Nanjing University of Technology, Xinmofan Road No. 5 Nanjing, Nanjing 210009, People's Republic of China, and [b]College of Life Science and Pharmaceutical Engineering, Nanjing University of Technology, Xinmofan Road No. 5 Nanjing, Nanjing 210009, People's Republic of China
Correspondence e-mail: fzcpu@163.com

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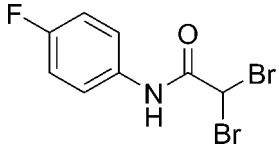
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.011 Å; R factor = 0.058; wR factor = 0.094; data-to-parameter ratio = 15.5.

In the crystal structure of the title compound, $C_8H_6Br_2FNO$, C—H···O and N—H···O hydrogen bonding results in six-membered rings and links the molecules into chains running parallel to the c axis. The dihedral angle between the fluorophenyl ring and the acetamide group is 29.5 (5)°.

Related literature

For background information, see: Fang *et al.* (2012). For related crystal structures, see: Gowda *et al.* (2009); Feng *et al.* (2012).



Experimental

Crystal data

$C_8H_6Br_2FNO$
M_r = 310.96
Monoclinic, $P2_1/c$
a = 9.746 (2) Å
b = 10.980 (2) Å
c = 9.426 (2) Å
β = 96.33 (3)°
V = 1002.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.06 mm^{-1}
T = 293 K
0.10 × 0.10 × 0.10 mm

Data collection

Enraf–Nonious CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.975, T_{max} = 0.991
1937 measured reflections
1827 independent reflections
900 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.094
S = 1.00
1827 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 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	2.06	2.868 (7)	156
C1—H1A···O^{i}	0.98	2.37	3.178 (9)	140

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) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2533).

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supporting information

Acta Cryst. (2012). E**68**, o1824 [doi:10.1107/S1600536812021174]

2,2-Dibromo-*N*-(4-fluorophenyl)acetamide

Xiangjun Qian, Zheng Fang, Shuxin Bao, Kai Guo and Ping Wei

S1. Comment

As a part of our studies on the synthesis of Ezetimibe (Fang *et al.*, 2012), the title compound which is one of the derivates of an intermediate, has been synthesized and its crystal structure is reported in this paper.

In the title molecule (Fig. 1), the dihedral angle between fluorophenyl ring (F/C3–C8) and acetamide group (O/N/C1/C2) group is 29.5 (5)°. The carbonyl O atom is hydrogen bonded to hydrogen atoms at N and C1, resulting in six membered rings linking the molecules into chains running parallel to the *c*-axis (Fig. 2 and Tab. 1). The bond distances and angles in the title molecule are in excellent agreement with the corresponding bond distances and angles reported in closely related structures (Gowda *et al.*, 2009; Feng *et al.*, 2012).

S2. Experimental

To 3-ethoxy-*N*-(4-fluorophenyl)acrylamide (1 g) was added 1,4-dioxane (20 ml) and water (20 ml) in a 50 ml flask. The solution was cooled to 273 K in an ice bath and *N*-bromosuccinimide (1.6 g) was added after 30 minutes. The solution was stirred at room temperature for 3 h. Then, the solution was heated to 353 K, after 40 minutes, the resulting mixture was concentrated under vacuum, the solid was collected by vacuum filtration, washed with cold water. Finally, the product was separated by silica gel column (yield = 59%). Crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

S3. 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 0.98 Å, for aryl and methyne H-atoms, respectively. The U_{iso}(H) were allowed at 1.2U_{eq}(N/C).

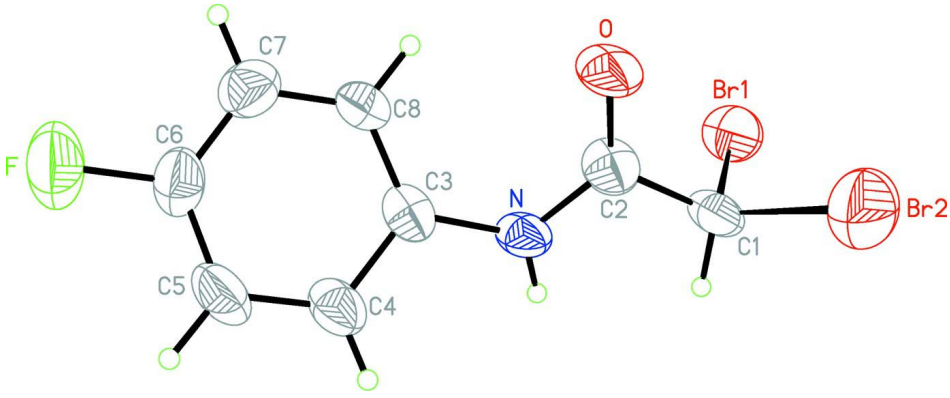

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radius.

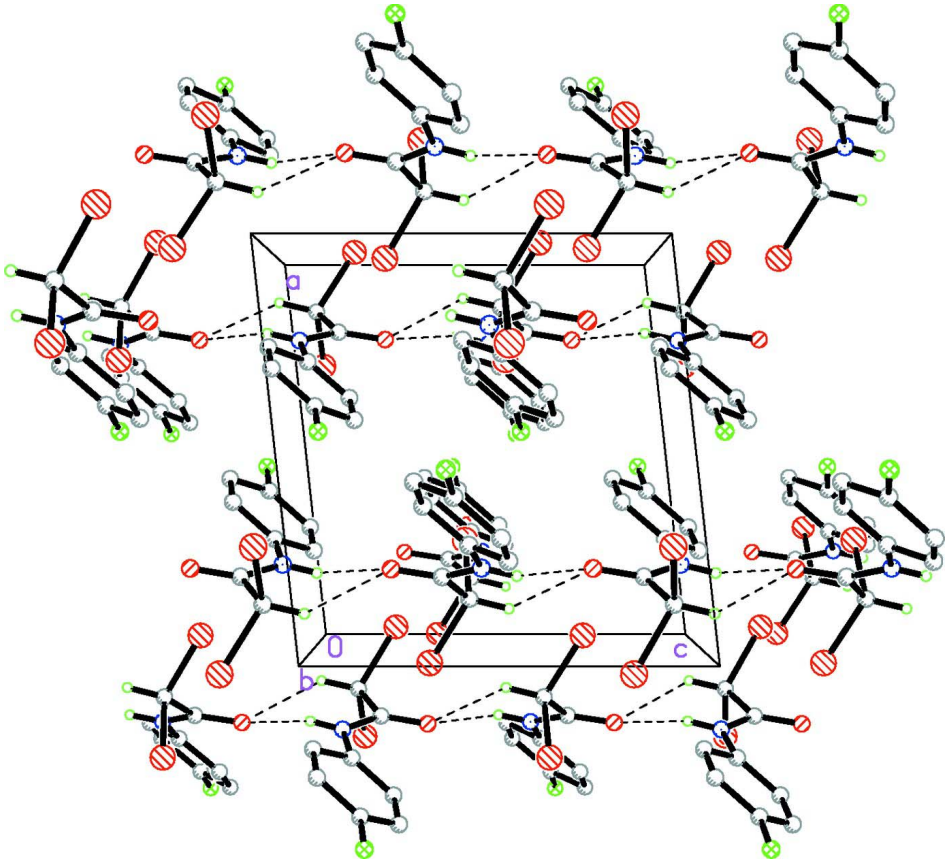

Figure 2

A view of the N—H···O and C—H···Ohydrogen bonds (dotted lines) in the crystal structure of the title compound.

2,2-Dibromo-*N*-(4-fluorophenyl)acetamide

Crystal data

$C_8H_6Br_2FNO$
$M_r = 310.96$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.746$ (2) Å
$b = 10.980$ (2) Å
$c = 9.426$ (2) Å
$\beta = 96.33$ (3)°
$V = 1002.5$ (3) Å^3
$Z = 4$
$F(000) = 592$
$D_x = 2.060$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–12°
$\mu = 8.06$ mm^{-1}
$T = 293$ K
Block, colorless
0.10 × 0.10 × 0.10 mm

Data collection

Enraf–Nonious CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.975$, $T_{max} = 0.991$
1937 measured reflections
1827 independent reflections
900 reflections with $I > 2\sigma(I)$
$R_{int} = 0.068$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 0$
$k = 0 \rightarrow 13$
$l = -11 \rightarrow 11$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.094$
$S = 1.00$
1827 reflections
118 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
$w = 1/[\sigma^2(F_o^2) + (0.024P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ 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}
F	0.4558 (5)	0.2056 (4)	0.4060 (6)	0.0868 (17)
Br1	0.27410 (10)	0.98435 (8)	0.42192 (10)	0.0675 (3)
Br2	−0.02715 (10)	0.90635 (10)	0.29891 (11)	0.0840 (4)
O	0.2133 (6)	0.7340 (5)	0.2350 (5)	0.0635 (17)
N	0.2193 (6)	0.6579 (5)	0.4588 (6)	0.0439 (17)
H0A	0.1995	0.6743	0.5434	0.053*
C1	0.1342 (8)	0.8605 (6)	0.4178 (8)	0.048 (2)
H1A	0.1111	0.8455	0.5149	0.058*
C2	0.1936 (8)	0.7438 (7)	0.3585 (8)	0.046 (2)
C3	0.2775 (8)	0.5405 (6)	0.4353 (8)	0.0390 (19)
C4	0.2452 (8)	0.4467 (7)	0.5254 (8)	0.052 (2)
H4A	0.1850	0.4610	0.5936	0.062*
C5	0.3007 (9)	0.3355 (8)	0.5140 (9)	0.059 (3)
H5A	0.2770	0.2713	0.5708	0.070*
C6	0.3943 (8)	0.3195 (8)	0.4150 (10)	0.054 (2)
C7	0.4260 (8)	0.4084 (8)	0.3246 (8)	0.056 (2)
H7A	0.4856	0.3933	0.2561	0.067*
C8	0.3694 (8)	0.5186 (7)	0.3364 (7)	0.046 (2)
H8A	0.3923	0.5815	0.2771	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F	0.081 (4)	0.047 (3)	0.135 (5)	0.008 (3)	0.025 (3)	−0.004 (3)
Br1	0.0902 (7)	0.0504 (6)	0.0640 (6)	−0.0033 (6)	0.0179 (5)	−0.0068 (5)

Br2	0.0681 (7)	0.1079 (10)	0.0749 (8)	0.0201 (7)	0.0034 (5)	0.0095 (7)
O	0.116 (5)	0.052 (4)	0.026 (3)	0.013 (4)	0.020 (3)	−0.002 (3)
N	0.067 (5)	0.037 (4)	0.030 (4)	−0.005 (4)	0.016 (3)	−0.003 (3)
C1	0.075 (6)	0.037 (5)	0.035 (5)	−0.001 (4)	0.017 (4)	0.007 (4)
C2	0.063 (6)	0.046 (5)	0.029 (5)	0.001 (5)	−0.002 (4)	−0.007 (5)
C3	0.050 (5)	0.036 (5)	0.028 (5)	−0.006 (4)	−0.010 (4)	−0.001 (4)
C4	0.079 (7)	0.042 (6)	0.036 (5)	−0.004 (5)	0.009 (5)	0.004 (4)
C5	0.063 (6)	0.039 (6)	0.075 (7)	−0.014 (5)	0.012 (5)	0.014 (5)
C6	0.043 (5)	0.040 (6)	0.078 (7)	0.004 (5)	0.007 (5)	−0.004 (5)
C7	0.064 (6)	0.054 (6)	0.051 (6)	−0.005 (5)	0.014 (5)	−0.011 (5)
C8	0.056 (5)	0.044 (5)	0.037 (5)	0.000 (5)	0.009 (4)	0.010 (4)

Geometric parameters (Å, º)

F—C6	1.394 (8)	C3—C4	1.392 (9)
Br1—C1	1.923 (7)	C4—C5	1.344 (9)
Br2—C1	1.896 (7)	C4—H4A	0.9300
O—C2	1.205 (7)	C5—C6	1.386 (10)
N—C2	1.339 (8)	C5—H5A	0.9300
N—C3	1.436 (8)	C6—C7	1.354 (10)
N—H0A	0.8600	C7—C8	1.340 (9)
C1—C2	1.536 (10)	C7—H7A	0.9300
C1—H1A	0.9800	C8—H8A	0.9300
C3—C8	1.384 (9)		
C2—N—C3	124.8 (6)	C5—C4—C3	120.2 (8)
C2—N—H0A	117.6	C5—C4—H4A	119.9
C3—N—H0A	117.6	C3—C4—H4A	119.9
C2—C1—Br2	109.1 (5)	C4—C5—C6	118.0 (8)
C2—C1—Br1	107.6 (5)	C4—C5—H5A	121.0
Br2—C1—Br1	111.3 (3)	C6—C5—H5A	121.0
C2—C1—H1A	109.6	C7—C6—C5	123.1 (8)
Br2—C1—H1A	109.6	C7—C6—F	118.6 (8)
Br1—C1—H1A	109.6	C5—C6—F	118.3 (8)
O—C2—N	125.6 (8)	C8—C7—C6	118.3 (8)
O—C2—C1	122.2 (7)	C8—C7—H7A	120.8
N—C2—C1	112.3 (6)	C6—C7—H7A	120.8
C8—C3—C4	119.3 (7)	C7—C8—C3	121.0 (7)
C8—C3—N	123.7 (7)	C7—C8—H8A	119.5
C4—C3—N	116.8 (7)	C3—C8—H8A	119.5
C3—N—C2—O	1.4 (13)	N—C3—C4—C5	−177.0 (7)
C3—N—C2—C1	−178.8 (6)	C3—C4—C5—C6	2.7 (13)
Br2—C1—C2—O	50.2 (9)	C4—C5—C6—C7	−3.8 (14)
Br1—C1—C2—O	−70.7 (9)	C4—C5—C6—F	177.9 (7)
Br2—C1—C2—N	−129.7 (6)	C5—C6—C7—C8	3.4 (13)
Br1—C1—C2—N	109.4 (6)	F—C6—C7—C8	−178.3 (7)
C2—N—C3—C8	30.9 (11)	C6—C7—C8—C3	−2.0 (12)

C2—N—C3—C4	−153.6 (7)	C4—C3—C8—C7	1.0 (11)
C8—C3—C4—C5	−1.4 (12)	N—C3—C8—C7	176.4 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0*A*···O^{i}	0.86	2.06	2.868 (7)	156
C1—H1*A*···O^{i}	0.98	2.37	3.178 (9)	140
C8—H8*A*···O	0.93	2.42	2.916 (9)	113

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