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N-(5-Chloro-2-nitrophenyl)-2,2-dimethylpropanamide

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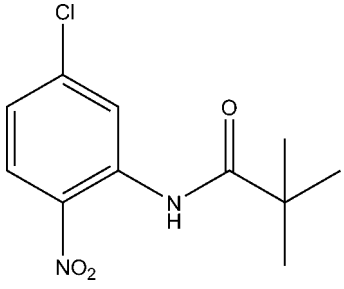
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.064; wR factor = 0.179; data-to-parameter ratio = 13.2.

In the crystal structure of the title compound, $C_{11}H_{13}ClN_2O_3$, molecules are linked through C—H···O hydrogen bonds.

Related literature

For background to the biologically active molecule ezetimibe [systematic name: (3*R*,4*S*)-1-(4-fluorophenyl)-3-[(3*S*)-3-(4-fluorophenyl)-3-hydroxypropyl]-4-(4-hydroxyphenyl)azetidin-2-one, see: Rosenblum *et al.* (1998). For the preparation of the title compound, a derivative of an intermediate in the synthesis of ezetimibe, see: Wang *et al.* (2009). For a related structure, see: Zhu *et al.* (2007).



Experimental

Crystal data

$C_{11}H_{12}ClN_2O_3$
M_r = 255.68
Orthorhombic, *Pnma*
a = 10.401 (2) Å
b = 7.0280 (14) Å
c = 17.106 (3) Å
V = 1250.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 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} = 0.915, T_{max} = 0.970
2432 measured reflections
1244 independent reflections
643 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.179
S = 1.00
1244 reflections
94 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···O3^{i}	0.96	2.35	3.294 (8)	167

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZJ2083).

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supporting information

Acta Cryst. (2012). E**68**, o2771 [doi:10.1107/S1600536812027730]

N-(5-Chloro-2-nitrophenyl)-2,2-dimethylpropanamide

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

S1. 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 part of our studies into the synthesis of Ezetimibe, the title compound *N*-(5-chloro-2-nitrophenyl)-2,2-dimethylpropanamide, (I), which is one of the derivates of a intermediate, is synthesized (Wang *et al.*, 2009). In this paper we report the crystal structure of the title compound.

In the crystal structure,*C*—H···O hydrogen bonds interactions (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure. It's just formed by the accumulation of Molecules.

S2. Experimental

5-chloro-2-nitroaniline ($C_6H_5ClN_2O_2$, 20.64 g, 0.12 mol) in CH_2Cl_2(40 ml) was added 4-dimethylaminopyridine ($C_7H_{10}N_2$, 1.2 g, 0.01 mol) and Et_3N (42.3 ml, 0.31 mol) and cooled the reaction to 273 K. A solution of pivaloyl chloride (C_5H_9ClO, 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 andwashed the organic layer sequentially with NaOH (10%), NaCl (satd) and water. Dried the organic layer over $MgSO_4$ and concentrated to obtain solid product as pure yellow solid. (Wang *et al.*, 2009). Crystals of (I) 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 C—H = 0.93 and 0.97 Å, for aryl and methylene H-atoms, respectively. The U_{iso}(H) were allowed at 1.2U_{eq}(C).

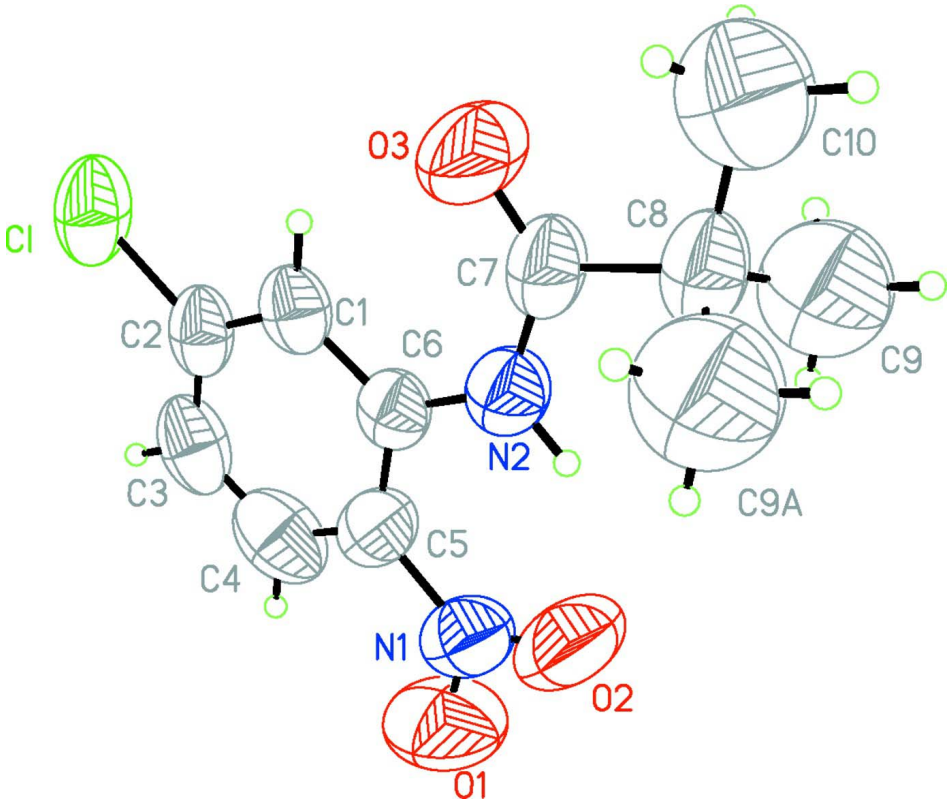

Figure 1

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.

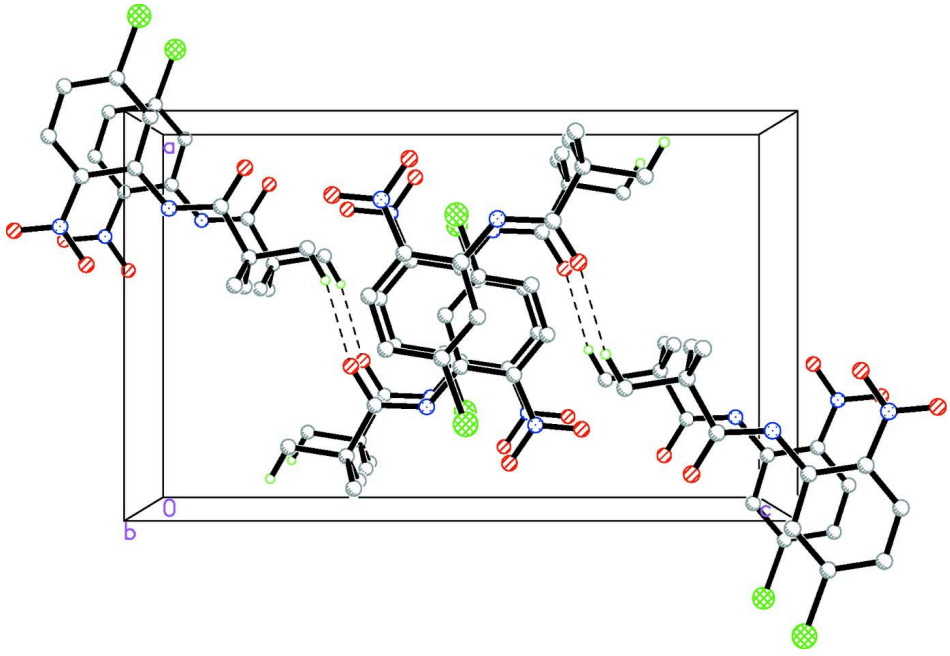

Figure 2

A view of the unit cell packing of the title compound.

N-(5-Chloro-2-nitrophenyl)-2,2-dimethylpropanamide

Crystal data

$C_{11}H_{12}ClN_2O_3$
$M_r = 255.68$
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
$a = 10.401$ (2) Å
$b = 7.0280$ (14) Å
$c = 17.106$ (3) Å
$V = 1250.4$ (4) Å^3
$Z = 4$
$F(000) = 532$
$D_x = 1.358$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–12°
$\mu = 0.30$ mm^{-1}
$T = 293$ K
Block, colorless
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)
$T_{min} = 0.915$, $T_{max} = 0.970$
2432 measured reflections
1244 independent reflections
643 reflections with $I > 2\sigma(I)$
$R_{int} = 0.069$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.3°$
$h = 0 \rightarrow 12$
$k = -8 \rightarrow 0$
$l = -20 \rightarrow 20$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.179$
$S = 1.00$
1244 reflections
94 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
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl	0.24447 (9)	0.2500	0.50682 (10)	0.0967 (6)	

N1	0.7748 (5)	0.2500	0.3855 (3)	0.0880 (14)	
C1	0.4982 (4)	0.2500	0.5237 (3)	0.0623 (11)	
H1A	0.4794	0.2500	0.5769	0.075*	
O1	0.7853 (5)	0.2500	0.3151 (3)	0.143 (2)	
N2	0.7290 (3)	0.2500	0.5525 (2)	0.0706 (11)	
H2A	0.8046	0.2500	0.5321	0.085*	
O2	0.8711 (4)	0.2500	0.4258 (3)	0.1202 (15)	
C2	0.4005 (4)	0.2500	0.4726 (3)	0.0664 (12)	
O3	0.6267 (3)	0.2500	0.6688 (2)	0.1179 (16)	
C3	0.4218 (5)	0.2500	0.3921 (3)	0.0824 (15)	
H3A	0.3541	0.2500	0.3565	0.099*	
C4	0.5469 (7)	0.2500	0.3681 (3)	0.0964 (18)	
H4A	0.5640	0.2500	0.3147	0.116*	
C5	0.6479 (4)	0.2500	0.4186 (3)	0.0732 (13)	
C6	0.6275 (4)	0.2500	0.5004 (3)	0.0585 (11)	
C7	0.7243 (4)	0.2500	0.6321 (3)	0.0720 (14)	
C8	0.8531 (4)	0.2500	0.6754 (3)	0.0802 (15)	
C9	0.9244 (4)	0.4286 (7)	0.6607 (3)	0.156	
H9A	0.9438	0.4385	0.6060	0.233*	
H9B	1.0029	0.4280	0.6902	0.233*	
H9C	0.8726	0.5351	0.6763	0.233*	
C10	0.8297 (7)	0.2500	0.7644 (4)	0.148 (3)	
H10A	0.9098	0.2500	0.7922	0.222*	
H10B	0.7814	0.3615	0.7780	0.222*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0576 (8)	0.0846 (10)	0.1478 (14)	0.000	−0.0208 (7)	0.000
N1	0.091 (3)	0.067 (3)	0.106 (4)	0.000	0.027 (3)	0.000
C1	0.066 (3)	0.043 (2)	0.078 (3)	0.000	−0.013 (3)	0.000
O1	0.168 (4)	0.172 (5)	0.089 (3)	0.000	0.042 (3)	0.000
N2	0.062 (2)	0.075 (3)	0.074 (3)	0.000	−0.001 (2)	0.000
O2	0.087 (3)	0.146 (4)	0.128 (4)	0.000	0.040 (3)	0.000
C2	0.053 (2)	0.045 (2)	0.101 (3)	0.000	−0.015 (2)	0.000
O3	0.060 (2)	0.215 (5)	0.078 (2)	0.000	−0.0037 (18)	0.000
C3	0.088 (4)	0.058 (3)	0.101 (4)	0.000	−0.038 (3)	0.000
C4	0.138 (5)	0.072 (4)	0.079 (4)	0.000	−0.017 (4)	0.000
C5	0.068 (3)	0.059 (3)	0.093 (4)	0.000	0.015 (3)	0.000
C6	0.061 (2)	0.041 (2)	0.074 (3)	0.000	0.000 (2)	0.000
C7	0.048 (2)	0.076 (4)	0.092 (4)	0.000	−0.011 (3)	0.000
C8	0.058 (3)	0.071 (3)	0.111 (4)	0.000	−0.012 (3)	0.000
C9	0.156	0.156	0.156	0.000	0.000	0.000
C10	0.132 (6)	0.175 (8)	0.136 (6)	0.000	−0.015 (5)	0.000

Geometric parameters (Å, º)

Cl—C2	1.725 (4)	C3—H3A	0.9300
N1—O1	1.211 (6)	C4—C5	1.360 (7)
N1—O2	1.215 (6)	C4—H4A	0.9300
N1—C5	1.436 (6)	C5—C6	1.415 (6)
C1—C2	1.340 (6)	C7—C8	1.530 (6)
C1—C6	1.403 (6)	C8—C9	1.479 (5)
C1—H1A	0.9300	C8—C9^{i}	1.479 (5)
N2—C7	1.363 (6)	C8—C10	1.540 (8)
N2—C6	1.381 (5)	C9—H9A	0.9600
N2—H2A	0.8600	C9—H9B	0.9600
C2—C3	1.396 (7)	C9—H9C	0.9600
O3—C7	1.194 (5)	C10—H10A	0.9600
C3—C4	1.365 (7)	C10—H10B	0.9600
O1—N1—O2	119.3 (5)	N2—C6—C1	123.3 (4)
O1—N1—C5	118.4 (6)	N2—C6—C5	121.6 (4)
O2—N1—C5	122.3 (5)	C1—C6—C5	115.1 (4)
C2—C1—C6	122.7 (4)	O3—C7—N2	123.8 (4)
C2—C1—H1A	118.6	O3—C7—C8	119.3 (5)
C6—C1—H1A	118.6	N2—C7—C8	116.9 (4)
C7—N2—C6	128.1 (4)	C9—C8—C9^{i}	116.1 (5)
C7—N2—H2A	115.9	C9—C8—C7	110.8 (3)
C6—N2—H2A	115.9	C9^{i}—C8—C7	110.8 (3)
C1—C2—C3	121.6 (4)	C9—C8—C10	104.3 (4)
C1—C2—Cl	119.5 (4)	C9^{i}—C8—C10	104.3 (4)
C3—C2—Cl	118.9 (4)	C7—C8—C10	109.9 (5)
C4—C3—C2	116.6 (5)	C8—C9—H9A	109.5
C4—C3—H3A	121.7	C8—C9—H9B	109.5
C2—C3—H3A	121.7	H9A—C9—H9B	109.5
C5—C4—C3	123.0 (5)	C8—C9—H9C	109.5
C5—C4—H4A	118.5	H9A—C9—H9C	109.5
C3—C4—H4A	118.5	H9B—C9—H9C	109.5
C4—C5—C6	120.9 (4)	C8—C10—H10A	110.7
C4—C5—N1	117.3 (5)	C8—C10—H10B	108.8
C6—C5—N1	121.8 (5)	H10A—C10—H10B	109.5
C6—C1—C2—C3	0.000 (1)	C2—C1—C6—C5	0.000 (1)
C6—C1—C2—Cl	180.0	C4—C5—C6—N2	180.0
C1—C2—C3—C4	0.000 (1)	N1—C5—C6—N2	0.000 (1)
Cl—C2—C3—C4	180.0	C4—C5—C6—C1	0.0
C2—C3—C4—C5	0.000 (1)	N1—C5—C6—C1	180.0
C3—C4—C5—C6	0.000 (1)	C6—N2—C7—O3	0.000 (2)
C3—C4—C5—N1	180.000 (1)	C6—N2—C7—C8	180.000 (1)
O1—N1—C5—C4	0.000 (1)	O3—C7—C8—C9	−114.8 (4)
O2—N1—C5—C4	180.000 (1)	N2—C7—C8—C9	65.2 (4)
O1—N1—C5—C6	180.0	O3—C7—C8—C9^{i}	114.8 (4)

O2—N1—C5—C6	0.000 (1)	N2—C7—C8—C9^{i}	−65.2 (4)
C7—N2—C6—C1	0.000 (1)	O3—C7—C8—C10	0.000 (2)
C7—N2—C6—C5	180.000 (1)	N2—C7—C8—C10	180.000 (2)
C2—C1—C6—N2	180.0		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O3^{ii}	0.96	2.35	3.294 (8)	167

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