Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-[4-Chloro-3-(trifluoromethyl)phenyl]-2,2-dimethylpropanamide

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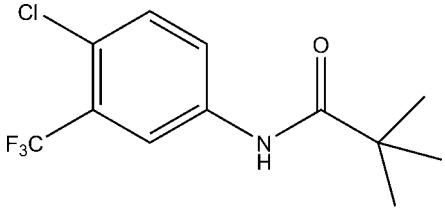
Received 21 June 2012; accepted 11 July 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.061; wR factor = 0.192; data-to-parameter ratio = 14.5.

In the title compound, $C_{12}H_{13}ClF_3NO$, the C—C—N—C torsion angle between the benzene ring and the pivaloyl group is −33.9 (5)°. In the crystal, molecules are linked *via* N—H···O hydrogen bonds to form chains running parallel to the *c* axis. Weak van der Waals interactions are also observed.

Related literature

For background information on related compounds, see: Rosenblum *et al.* (1998); Wang *et al.* (2009). For a related crystal structure, see: Zhu *et al.* (2007).



Experimental

Crystal data

$C_{12}H_{13}ClF_3NO$
M_r = 279.68
Monoclinic, $P2_1/c$
a = 5.8850 (12) Å
b = 21.955 (4) Å
c = 10.307 (2) Å
β = 104.50 (3)°
V = 1289.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 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} = 0.910, T_{max} = 0.969
2599 measured reflections
2364 independent reflections
1477 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.192
S = 1.00
2364 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O	0.86	2.24	3.041 (4)	155

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD-4 EXPRESS*; 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 work was financially supported by the College of Life Science and Pharmaceutical Engineering, Nanjing University of Technology, '973' project (2011CB710803), and the Key Basic Research Program of China, '973' project (2012CB721104).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2427).

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Wang, Y., Zhang, H., Huang, W., Kong, J., Zhou, J. & Zhang, B. (2009). *Eur. J. Med. Chem.* **44**, 1638–1643.
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supporting information

Acta Cryst. (2012). E**68**, o2534 [https://doi.org/10.1107/S1600536812031650]

N-[4-Chloro-3-(trifluoromethyl)phenyl]-2,2-dimethylpropanamide

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S1. Comment

Ezetimibe is a biologically active molecule, and research has shown it to have the useful property of inhibiting the absorption of cholesterol in the intestine (Rosenblum *et al.*, 1998). As part of our studies into the synthesis of Ezetimibe, the title compound, 4-chloro-3-(trifluoromethyl)-N-pivaloylaniline (I), which is a derivate formed as an intermediate, was synthesized (Wang *et al.*, 2009). In the crystal structure, N—H···O hydrogen bonding interactions (Table 1) link the molecules (Fig. 2) into chains running parallel to the *c* axis.

S2. Experimental

4-chloro-3-(trifluoromethyl)aniline ($C_7H_5ClF_3N$, 23.40 g, 0.12 mol) in CH_2Cl_2 (40 ml) was added to 4-dimethylamino-pyridine ($C_7H_{10}N_2$, 1.2 g, 0.01 mol), and Et_3N (42.3 ml, 0.31 mol) and the reaction was cooled 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 then heated to reflux. After 12 h, H_2O and H_2SO_4 (2 N, 75 ml) were added, the layers were separated, and the organic layer was washed sequentially with NaOH (10%), NaCl (satd) and water. The organic layer was dried over $MgSO_4$ and concentrated to obtain the product as a pure yellow solid (Wang *et al.*, 2009). Crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanolic 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, and 0.86 Å for N—H. The U_{iso}(H) were included at 1.5U_{eq}(C) for the methyl groups and 1.2U_{eq} for all other hydrogen atoms.

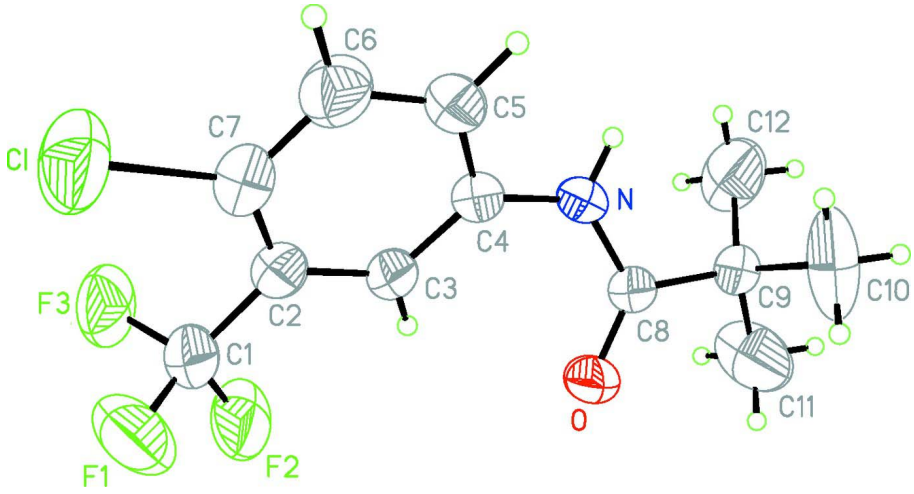

Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radius.

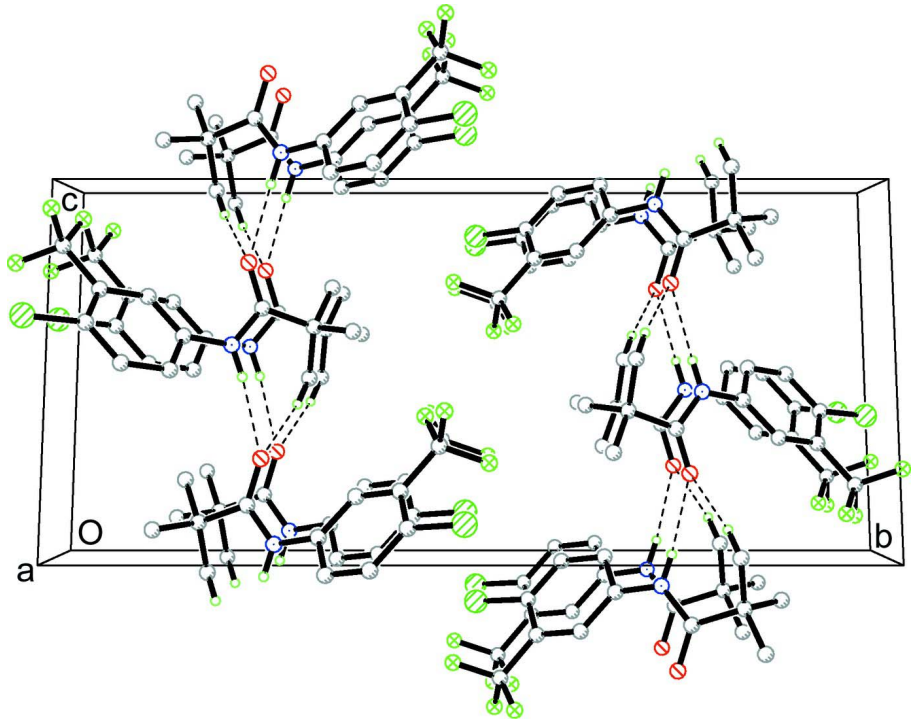

Figure 2

A packing plot of (I), viewed down the *a*-axis of the unit cell.

N-[4-Chloro-3-(trifluoromethyl)phenyl]-2,2-dimethylpropanamide

Crystal data

$C_{12}H_{13}ClF_3NO$
$M_r = 279.68$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

$a = 5.8850\ (12)$ Å
$b = 21.955\ (4)$ Å
$c = 10.307\ (2)$ Å
$\beta = 104.50\ (3)°$

V = 1289.3 (5) Å^3
Z = 4
F(000) = 576
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.32 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} = 0.910, T_{max} = 0.969
2599 measured reflections
2364 independent reflections
1477 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.4°, θ_{min} = 1.9°
h = 0→7
k = 0→26
l = −12→12
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.192
S = 1.00
2364 reflections
163 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.6P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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}
Cl	0.8303 (2)	0.49337 (6)	0.12942 (14)	0.0873 (5)
O	0.1944 (5)	0.25926 (12)	0.2729 (2)	0.0647 (8)
N	0.2134 (5)	0.27644 (13)	0.0605 (3)	0.0494 (7)
H0A	0.1632	0.2645	−0.0211	0.059*
F1	0.7046 (6)	0.47123 (13)	0.3967 (3)	0.1021 (10)
C1	0.4957 (8)	0.46500 (17)	0.3095 (4)	0.0639 (11)
F2	0.3471 (6)	0.44832 (12)	0.3808 (3)	0.1073 (11)
C2	0.5006 (6)	0.42126 (16)	0.1992 (3)	0.0483 (9)
F3	0.4298 (5)	0.52091 (10)	0.2663 (3)	0.0797 (8)
C3	0.3567 (6)	0.37039 (15)	0.1826 (3)	0.0474 (9)

H3A	0.2576	0.3643	0.2389	0.057*
C4	0.3598 (6)	0.32856 (15)	0.0826 (3)	0.0444 (8)
C5	0.5033 (7)	0.33915 (17)	−0.0024 (3)	0.0540 (9)
H5A	0.5024	0.3120	−0.0718	0.065*
C6	0.6482 (7)	0.38958 (19)	0.0147 (4)	0.0623 (11)
H6A	0.7468	0.3957	−0.0420	0.075*
C7	0.6471 (6)	0.43065 (17)	0.1149 (4)	0.0542 (9)
C8	0.1439 (6)	0.24321 (15)	0.1556 (3)	0.0442 (8)
C9	0.0085 (6)	0.18476 (15)	0.1083 (3)	0.0463 (8)
C10	0.1853 (9)	0.1369 (2)	0.0953 (7)	0.107 (2)
H10A	0.2984	0.1321	0.1798	0.161*
H10B	0.2638	0.1493	0.0284	0.161*
H10C	0.1063	0.0989	0.0694	0.161*
C11	−0.1150 (11)	0.1652 (3)	0.2130 (5)	0.110 (2)
H11A	−0.0022	0.1605	0.2976	0.165*
H11B	−0.1931	0.1270	0.1872	0.165*
H11C	−0.2283	0.1955	0.2210	0.165*
C12	−0.1714 (9)	0.1921 (2)	−0.0249 (5)	0.0984 (18)
H12A	−0.0938	0.2042	−0.0923	0.148*
H12B	−0.2838	0.2226	−0.0166	0.148*
H12C	−0.2504	0.1540	−0.0500	0.148*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0823 (8)	0.0705 (8)	0.1155 (10)	−0.0271 (6)	0.0370 (7)	−0.0055 (7)
O	0.107 (2)	0.0532 (16)	0.0354 (13)	−0.0215 (15)	0.0202 (13)	−0.0028 (11)
N	0.074 (2)	0.0427 (16)	0.0316 (13)	−0.0087 (14)	0.0141 (13)	−0.0051 (12)
F1	0.136 (3)	0.078 (2)	0.0703 (16)	0.0004 (17)	−0.0147 (17)	−0.0234 (14)
C1	0.095 (3)	0.043 (2)	0.055 (2)	−0.011 (2)	0.021 (2)	−0.0062 (18)
F2	0.196 (3)	0.0666 (17)	0.0886 (18)	−0.0380 (19)	0.090 (2)	−0.0322 (14)
C2	0.062 (2)	0.0390 (19)	0.0420 (18)	0.0023 (16)	0.0098 (16)	0.0041 (15)
F3	0.105 (2)	0.0383 (13)	0.0994 (18)	0.0013 (12)	0.0319 (15)	−0.0087 (12)
C3	0.068 (2)	0.0380 (19)	0.0395 (17)	−0.0036 (16)	0.0199 (16)	0.0009 (14)
C4	0.060 (2)	0.0382 (18)	0.0355 (17)	0.0003 (15)	0.0126 (15)	0.0012 (14)
C5	0.074 (2)	0.046 (2)	0.0458 (19)	0.0035 (18)	0.0225 (18)	−0.0040 (16)
C6	0.070 (3)	0.060 (2)	0.067 (2)	−0.004 (2)	0.035 (2)	0.002 (2)
C7	0.058 (2)	0.043 (2)	0.061 (2)	−0.0044 (17)	0.0159 (18)	0.0035 (18)
C8	0.060 (2)	0.0380 (18)	0.0351 (18)	0.0023 (15)	0.0136 (15)	0.0013 (14)
C9	0.057 (2)	0.0368 (18)	0.0441 (18)	−0.0022 (15)	0.0118 (15)	0.0005 (15)
C10	0.082 (3)	0.048 (3)	0.186 (6)	0.002 (2)	0.021 (4)	−0.032 (3)
C11	0.144 (5)	0.113 (4)	0.088 (3)	−0.075 (4)	0.056 (3)	−0.027 (3)
C12	0.106 (4)	0.073 (3)	0.090 (3)	−0.028 (3)	−0.026 (3)	0.009 (3)

Geometric parameters (Å, º)

Cl—C7	1.732 (4)	C6—C7	1.372 (5)
O—C8	1.222 (4)	C6—H6A	0.9300

N—C8	1.364 (4)	C8—C9	1.525 (5)
N—C4	1.416 (4)	C9—C11	1.506 (6)
N—H0A	0.8600	C9—C10	1.508 (6)
F1—C1	1.336 (5)	C9—C12	1.518 (5)
C1—F2	1.326 (5)	C10—H10A	0.9600
C1—F3	1.330 (4)	C10—H10B	0.9600
C1—C2	1.494 (5)	C10—H10C	0.9600
C2—C7	1.385 (5)	C11—H11A	0.9600
C2—C3	1.386 (5)	C11—H11B	0.9600
C3—C4	1.384 (4)	C11—H11C	0.9600
C3—H3A	0.9300	C12—H12A	0.9600
C4—C5	1.379 (5)	C12—H12B	0.9600
C5—C6	1.382 (5)	C12—H12C	0.9600
C5—H5A	0.9300		

C8—N—C4	126.6 (3)	O—C8—N	120.9 (3)
C8—N—H0A	116.7	O—C8—C9	122.5 (3)
C4—N—H0A	116.7	N—C8—C9	116.6 (3)
F2—C1—F3	105.3 (4)	C11—C9—C10	109.4 (4)
F2—C1—F1	106.2 (3)	C11—C9—C12	109.0 (4)
F3—C1—F1	105.8 (3)	C10—C9—C12	109.5 (4)
F2—C1—C2	112.6 (3)	C11—C9—C8	108.6 (3)
F3—C1—C2	113.4 (3)	C10—C9—C8	107.3 (3)
F1—C1—C2	112.8 (4)	C12—C9—C8	113.0 (3)
C7—C2—C3	119.9 (3)	C9—C10—H10A	109.5
C7—C2—C1	121.1 (3)	C9—C10—H10B	109.5
C3—C2—C1	119.0 (3)	H10A—C10—H10B	109.5
C4—C3—C2	120.4 (3)	C9—C10—H10C	109.5
C4—C3—H3A	119.8	H10A—C10—H10C	109.5
C2—C3—H3A	119.8	H10B—C10—H10C	109.5
C5—C4—C3	119.0 (3)	C9—C11—H11A	109.5
C5—C4—N	118.6 (3)	C9—C11—H11B	109.5
C3—C4—N	122.3 (3)	H11A—C11—H11B	109.5
C4—C5—C6	120.7 (3)	C9—C11—H11C	109.5
C4—C5—H5A	119.6	H11A—C11—H11C	109.5
C6—C5—H5A	119.6	H11B—C11—H11C	109.5
C7—C6—C5	120.2 (3)	C9—C12—H12A	109.5
C7—C6—H6A	119.9	C9—C12—H12B	109.5
C5—C6—H6A	119.9	H12A—C12—H12B	109.5
C6—C7—C2	119.7 (3)	C9—C12—H12C	109.5
C6—C7—Cl	117.8 (3)	H12A—C12—H12C	109.5
C2—C7—Cl	122.4 (3)	H12B—C12—H12C	109.5

F2—C1—C2—C7	−179.3 (4)	C5—C6—C7—C2	0.2 (6)
F3—C1—C2—C7	61.3 (5)	C5—C6—C7—Cl	−179.2 (3)
F1—C1—C2—C7	−59.1 (5)	C3—C2—C7—C6	0.3 (5)
F2—C1—C2—C3	0.2 (5)	C1—C2—C7—C6	179.8 (4)
F3—C1—C2—C3	−119.2 (4)	C3—C2—C7—Cl	179.7 (3)

F1—C1—C2—C3	120.5 (4)	C1—C2—C7—Cl	−0.8 (5)
C7—C2—C3—C4	0.5 (5)	C4—N—C8—O	4.8 (5)
C1—C2—C3—C4	−179.0 (3)	C4—N—C8—C9	−173.3 (3)
C2—C3—C4—C5	−1.8 (5)	O—C8—C9—C11	19.1 (5)
C2—C3—C4—N	−179.0 (3)	N—C8—C9—C11	−162.8 (4)
C8—N—C4—C5	148.6 (3)	O—C8—C9—C10	−99.1 (5)
C8—N—C4—C3	−34.1 (5)	N—C8—C9—C10	79.0 (4)
C3—C4—C5—C6	2.3 (5)	O—C8—C9—C12	140.2 (4)
N—C4—C5—C6	179.6 (3)	N—C8—C9—C12	−41.7 (5)
C4—C5—C6—C7	−1.5 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0*A*···O	0.86	2.24	3.041 (4)	155