

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

***N*-(3-Chloro-4-fluorophenyl)acetamide**Hoong-Kun Fun,^{a,*} Wan-Sin Loh,^a§ Divya N. Shetty,^b
B. Narayana^b and B. K. Sarojini^c^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bDepartment of Studies in Chemistry, Mangalore University, Mangalagangothri 574 199, India, and ^cDepartment of Chemistry, P.A. College of Engineering, Nadupadavu, Mangalore 574 153, India
Correspondence e-mail: hkfun@usm.my

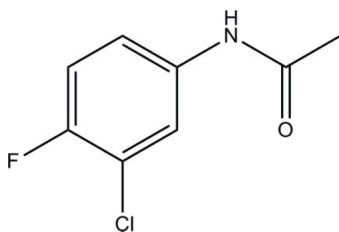
Received 26 March 2012; accepted 1 April 2012

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}—\text{C}) = 0.002$ Å; R factor = 0.047; wR factor = 0.143; data-to-parameter ratio = 36.1.

In the title compound, $\text{C}_8\text{H}_7\text{ClFNO}$, the dihedral angle between the benzene ring and the acetamide side chain is $5.47(6)^\circ$. An $S(6)$ ring motif is formed *via* an intramolecular $\text{C}—\text{H} \cdots \text{O}$ hydrogen bond. In the crystal, $\text{N}—\text{H} \cdots \text{O}$ hydrogen bonds link the molecules into $C(4)$ chains along $[001]$.

Related literature

For background to acetamides, see: Khan *et al.* (2010); Tahir & Shad (2011). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For a related structure, see: Rosli *et al.* (2007). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

 $\text{C}_8\text{H}_7\text{ClFNO}$
 $M_r = 187.60$
Monoclinic, $P2_1/c$
 $a = 7.6776(4)$ Å
 $b = 12.7671(7)$ Å
 $c = 9.8130(4)$ Å
 $\beta = 124.432(3)^\circ$
 $V = 793.35(7)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation $\mu = 0.44$ mm⁻¹
 $T = 100$ K $0.33 \times 0.29 \times 0.15$ mm

Data collection

Bruker SMART APEXII DUO
CCD diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2009)
 $T_{\min} = 0.869$, $T_{\max} = 0.937$ 14562 measured reflections
3971 independent reflections
3173 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.143$
 $S = 1.09$
3971 reflections110 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 1.32$ e Å⁻³
 $\Delta\rho_{\min} = -0.50$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D—H \cdots A$	$D—H$	$H \cdots A$	$D \cdots A$	$D—H \cdots A$
$\text{N1}—\text{H1} \cdots \text{O1}^1$	0.90	2.00	2.8996 (12)	174
$\text{C1}—\text{H1A} \cdots \text{O1}$	0.95	2.20	2.8222 (14)	122

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

HKF and WSL thank Universiti Sains Malaysia (USM) for the Research University Grant (1001/PFIZIK/811160). WSL also thanks the Malaysian Government and USM for the award of the post of Research Officer under the Research University Grant (1001/PFIZIK/811160). BN thanks the UGC SAP for financial assistance for the purchase of chemicals. DNS thanks Mangalore University for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6705).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2009). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Khan, F. N., Roopan, S. M., Malathi, N., Hathwar, V. R. & Akkurt, M. (2010). *Acta Cryst.* **E66**, o2043–o2044.
Rosli, M. M., Karthikeyan, M. S., Fun, H.-K., Razak, I. A. & Patil, P. S. (2007). *Acta Cryst.* **E63**, o67–o68.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
Tahir, M. N. & Shad, H. A. (2011). *Acta Cryst.* **E67**, o443.

* Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: C-7581-2009.

supporting information

Acta Cryst. (2012). E68, o1348 [https://doi.org/10.1107/S160053681201416X]

***N*-(3-Chloro-4-fluorophenyl)acetamide**

Hoong-Kun Fun, Wan-Sin Loh, Divya N. Shetty, B. Narayana and B. K. Sarojini

S1. Comment

To complement earlier studies of acetamides (Khan *et al.*, 2010; Tahir & Shad, 2011), we report herein the crystal structure of the title compound.

In the title compound (Fig. 1), an *S*(6) ring motif (Bernstein *et al.*, 1995) is formed *via* intramolecular C1—H1A···O1 hydrogen bond (Table 1). Bond lengths and angles are within the normal ranges and are comparable with the related structure (Rosli *et al.*, 2007).

In the crystal (Fig. 2), N1—H1···O1 hydrogen bonds (Table 1) link the molecules to form chains along the *c* axis.

S2. Experimental

3-Chloro-4-fluoro aniline (0.145 g, 1 mmol) was dissolved in acetic acid (20 mL) and refluxed for 4 h. The solution was then cooled and poured into 100 ml of ice-cold water with stirring. The precipitate obtained was filtered, washed with water and dried. Orange blocks were grown from DMF solution by the slow evaporation method. *M. P.*: 384 K.

S3. Refinement

N-bound H atoms were located from the difference Fourier map and were refined with a riding model with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{N})$ [N—H = 0.9003 Å]. The remaining H atoms were positioned geometrically and refined with a riding model with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$ [C—H = 0.95 or 0.98 Å]. A rotating group model was applied to the methyl groups. In the final refinement, five outliers were omitted, -3 8 2, -1 0 1, -3 8 1, 1 0 0 and -1 0 4. In the final difference Fourier map, the highest peak and the deepest hole are 0.83 and 0.71 Å from atom Cl1.

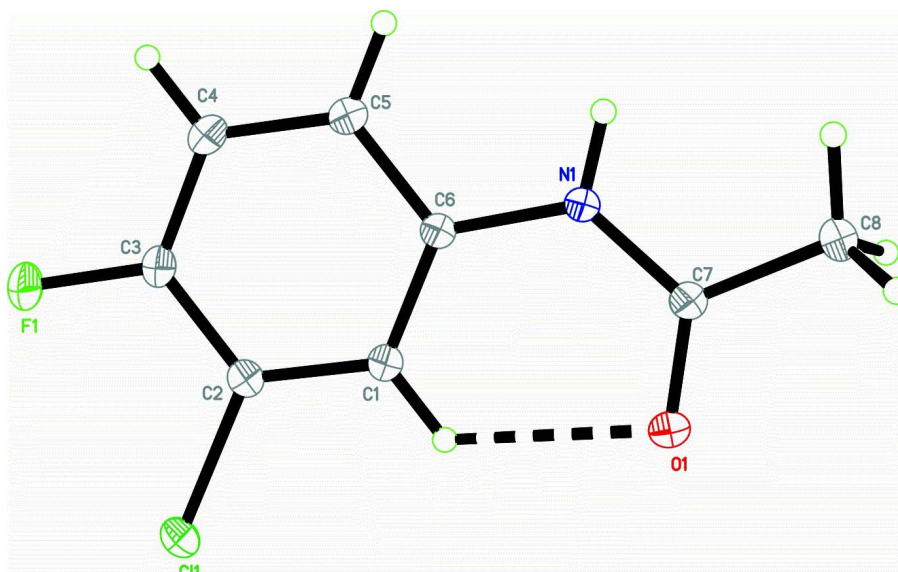


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids. Dashed line indicates the intramolecular hydrogen bond.

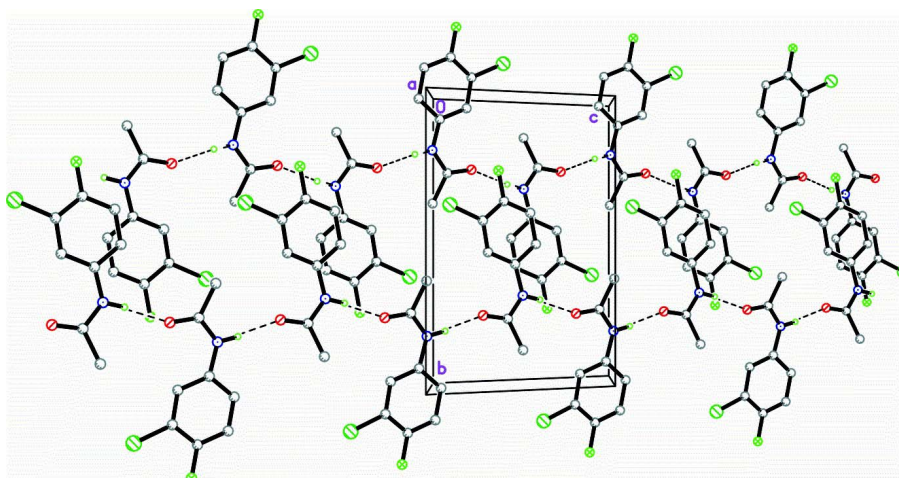


Figure 2

The crystal packing of the title compound, viewed along the *a* axis, showing the chains along the *c* axis. H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

N-(3-Chloro-4-fluorophenyl)acetamide

Crystal data

C_8H_7ClFNO

$M_r = 187.60$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 7.6776\ (4)\ \text{\AA}$

$b = 12.7671\ (7)\ \text{\AA}$

$c = 9.8130\ (4)\ \text{\AA}$

$\beta = 124.432\ (3)^\circ$

$V = 793.35\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 384$

$D_x = 1.571\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5541 reflections

$\theta = 3.0\text{--}36.8^\circ$

$\mu = 0.44\ \text{mm}^{-1}$

$T = 100$ K
Block, orange

$0.33 \times 0.29 \times 0.15$ mm

Data collection

Bruker SMART APEXII DUO CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.869$, $T_{\max} = 0.937$

14562 measured reflections
3971 independent reflections
3173 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\max} = 36.9^\circ$, $\theta_{\min} = 3.2^\circ$
 $h = -12 \rightarrow 12$
 $k = -21 \rightarrow 17$
 $l = -14 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.143$
 $S = 1.09$
3971 reflections
110 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
 $w = 1/[\sigma^2(F_o^2) + (0.0844P)^2 + 0.1402P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.32 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.50 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.32215 (5)	−0.12199 (2)	0.38846 (3)	0.02251 (9)
F1	0.31780 (12)	−0.24025 (5)	0.13350 (9)	0.02207 (16)
O1	0.18905 (15)	0.24972 (7)	0.21236 (10)	0.02070 (17)
N1	0.20506 (15)	0.18521 (7)	0.00234 (11)	0.01588 (16)
H1	0.1979	0.2007	−0.0902	0.019*
C1	0.27231 (17)	0.03769 (8)	0.18824 (12)	0.01625 (18)
H1A	0.2766	0.0831	0.2668	0.019*
C2	0.29704 (17)	−0.06971 (8)	0.21576 (13)	0.01634 (18)
C3	0.29615 (17)	−0.13592 (8)	0.10379 (13)	0.01655 (18)
C4	0.27098 (18)	−0.09670 (9)	−0.03746 (13)	0.01834 (19)
H4A	0.2733	−0.1423	−0.1129	0.022*
C5	0.24229 (17)	0.01024 (9)	−0.06795 (13)	0.01731 (19)

H5A	0.2233	0.0377	−0.1655	0.021*
C6	0.24111 (16)	0.07801 (8)	0.04398 (12)	0.01421 (17)
C7	0.17661 (17)	0.26277 (8)	0.08257 (13)	0.01566 (18)
C8	0.1266 (2)	0.36891 (9)	0.00122 (14)	0.0196 (2)
H8A	0.2467	0.4160	0.0687	0.029*
H8B	0.0013	0.3977	−0.0091	0.029*
H8C	0.0992	0.3619	−0.1088	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.03690 (18)	0.01515 (13)	0.02113 (14)	0.00050 (9)	0.01980 (13)	0.00337 (8)
F1	0.0318 (4)	0.0111 (3)	0.0247 (3)	0.0014 (2)	0.0167 (3)	0.0004 (2)
O1	0.0340 (4)	0.0164 (4)	0.0183 (3)	0.0011 (3)	0.0188 (3)	−0.0001 (3)
N1	0.0246 (4)	0.0120 (3)	0.0155 (3)	−0.0003 (3)	0.0140 (3)	0.0001 (3)
C1	0.0230 (5)	0.0126 (4)	0.0160 (4)	−0.0006 (3)	0.0128 (4)	−0.0006 (3)
C2	0.0214 (4)	0.0136 (4)	0.0162 (4)	−0.0009 (3)	0.0120 (3)	0.0003 (3)
C3	0.0204 (4)	0.0116 (4)	0.0183 (4)	0.0000 (3)	0.0114 (4)	−0.0005 (3)
C4	0.0243 (5)	0.0152 (4)	0.0180 (4)	0.0002 (3)	0.0134 (4)	−0.0029 (3)
C5	0.0238 (5)	0.0153 (4)	0.0165 (4)	−0.0002 (3)	0.0136 (4)	−0.0011 (3)
C6	0.0183 (4)	0.0122 (4)	0.0148 (4)	−0.0007 (3)	0.0109 (3)	−0.0003 (3)
C7	0.0201 (4)	0.0134 (4)	0.0159 (4)	−0.0007 (3)	0.0116 (3)	−0.0003 (3)
C8	0.0281 (5)	0.0136 (4)	0.0211 (5)	0.0011 (3)	0.0163 (4)	0.0019 (3)

Geometric parameters (Å, °)

Cl1—C2	1.7278 (11)	C3—C4	1.3807 (15)
F1—C3	1.3535 (12)	C4—C5	1.3884 (15)
O1—C7	1.2335 (13)	C4—H4A	0.9500
N1—C7	1.3573 (14)	C5—C6	1.4023 (14)
N1—C6	1.4102 (14)	C5—H5A	0.9500
N1—H1	0.9003	C7—C8	1.5081 (15)
C1—C2	1.3896 (15)	C8—H8A	0.9800
C1—C6	1.3938 (14)	C8—H8B	0.9800
C1—H1A	0.9500	C8—H8C	0.9800
C2—C3	1.3832 (15)		
C7—N1—C6	127.56 (9)	C4—C5—C6	120.56 (10)
C7—N1—H1	119.1	C4—C5—H5A	119.7
C6—N1—H1	113.3	C6—C5—H5A	119.7
C2—C1—C6	119.21 (9)	C1—C6—C5	119.61 (10)
C2—C1—H1A	120.4	C1—C6—N1	123.06 (9)
C6—C1—H1A	120.4	C5—C6—N1	117.32 (9)
C3—C2—C1	120.66 (10)	O1—C7—N1	123.81 (10)
C3—C2—Cl1	119.39 (8)	O1—C7—C8	121.05 (10)
C1—C2—Cl1	119.93 (8)	N1—C7—C8	115.14 (9)
F1—C3—C4	120.24 (9)	C7—C8—H8A	109.5
F1—C3—C2	119.04 (10)	C7—C8—H8B	109.5

C4—C3—C2	120.71 (10)	H8A—C8—H8B	109.5
C3—C4—C5	119.22 (10)	C7—C8—H8C	109.5
C3—C4—H4A	120.4	H8A—C8—H8C	109.5
C5—C4—H4A	120.4	H8B—C8—H8C	109.5
C6—C1—C2—C3	1.61 (16)	C2—C1—C6—C5	−2.14 (16)
C6—C1—C2—Cl1	−176.64 (8)	C2—C1—C6—N1	177.04 (10)
C1—C2—C3—F1	−179.12 (10)	C4—C5—C6—C1	1.00 (16)
Cl1—C2—C3—F1	−0.85 (14)	C4—C5—C6—N1	−178.22 (10)
C1—C2—C3—C4	0.09 (16)	C7—N1—C6—C1	−6.20 (17)
Cl1—C2—C3—C4	178.35 (9)	C7—N1—C6—C5	172.99 (10)
F1—C3—C4—C5	177.95 (10)	C6—N1—C7—O1	3.71 (18)
C2—C3—C4—C5	−1.24 (17)	C6—N1—C7—C8	−176.05 (10)
C3—C4—C5—C6	0.69 (17)		

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 $\cdots$ O1 ⁱ	0.90	2.00	2.8996 (12)	174
C1—H1A $\cdots$ O1	0.95	2.20	2.8222 (14)	122

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