



Crystal structure of 2-chloro-*N*-(3-fluorophenyl)acetamide

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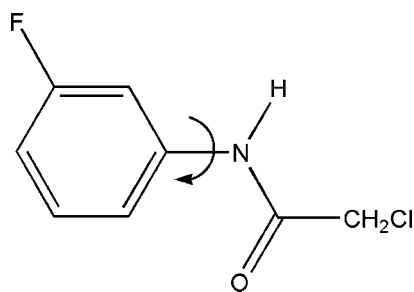
In the title compound, C₈H₇ClFNO, the F atom is disordered over the *meta* positions of the benzene ring in a 0.574 (4):0.426 (4) ratio and the Cl atom is *syn* to the O atom [O—C—C—Cl = 5.6 (3)°]. A short intramolecular C—H...O contact occurs. In the crystal, molecules are linked into amide C(4) chains propagating in [101] by N—H...O hydrogen bonds.

Keywords: crystal structure; disordered F atom; *N*-arylamides; hydrogen bonding.

CCDC reference: 1049536

1. Related literature

For compounds in which the *meta* fluorine substituent of a benzene ring exhibits positional disorder, see: Nayak *et al.* (2012); Sanjeevarayappa *et al.* (2015).



2. Experimental

2.1. Crystal data

C₈H₇ClFNO

M_r = 187.60

Monoclinic, *P*2₁/*n*
a = 5.0441 (2) Å
b = 18.2374 (7) Å
c = 8.8653 (3) Å
β = 99.843 (1)°
V = 803.53 (5) Å³

Z = 4
Cu *Kα* radiation
μ = 3.95 mm⁻¹
T = 293 K
0.31 × 0.24 × 0.19 mm

2.2. Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
T_{min} = 0.368, *T_{max}* = 0.472
6045 measured reflections
1304 independent reflections

1297 reflections with *I* > 2σ(*I*)
R_{int} = 0.041
1 standard reflections every 1 reflections
intensity decay: 1%

2.3. Refinement

R[*F*² > 2σ(*F*²)] = 0.039
wR(*F*²) = 0.105
S = 1.15
1304 reflections
123 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
Δρ_{max} = 0.26 e Å⁻³
Δρ_{min} = -0.25 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C2—H2...O1	0.93	2.33	2.885 (3)	118
N1—H1...O1 ⁱ	0.89 (2)	1.99 (3)	2.843 (2)	160 (2)

Symmetry code: (i) *x* - ½, -*y* + ½, *z* - ½.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7400).

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supporting information

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S1. Synthesis and crystallization

The title compound (scheme 1) was synthesized by the reaction of 2-chloroacetyl chloride with 3-fluoroaniline at room temperature. The reaction mixture was poured into crushed ice and the resulting solid was washed thoroughly with water, dilute hydrochloric acid and filtered.

A small portion of the resulting compound was taken in a 10.0 ml beaker and dissolved in a 1:1 ratio of a mixture of EtOH/H₂O to obtain colourless prisms by a slow evaporation method at ~24°C.

S2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms of the NH groups were located in a difference map and later restrained to N—H = 0.86 (4) Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the *U*_{eq} of the parent atom).

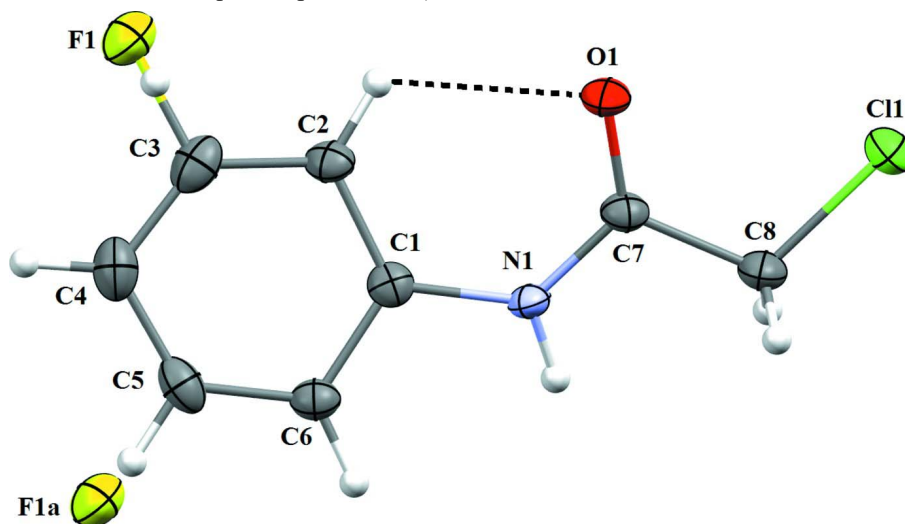


Figure 1

A view of the molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

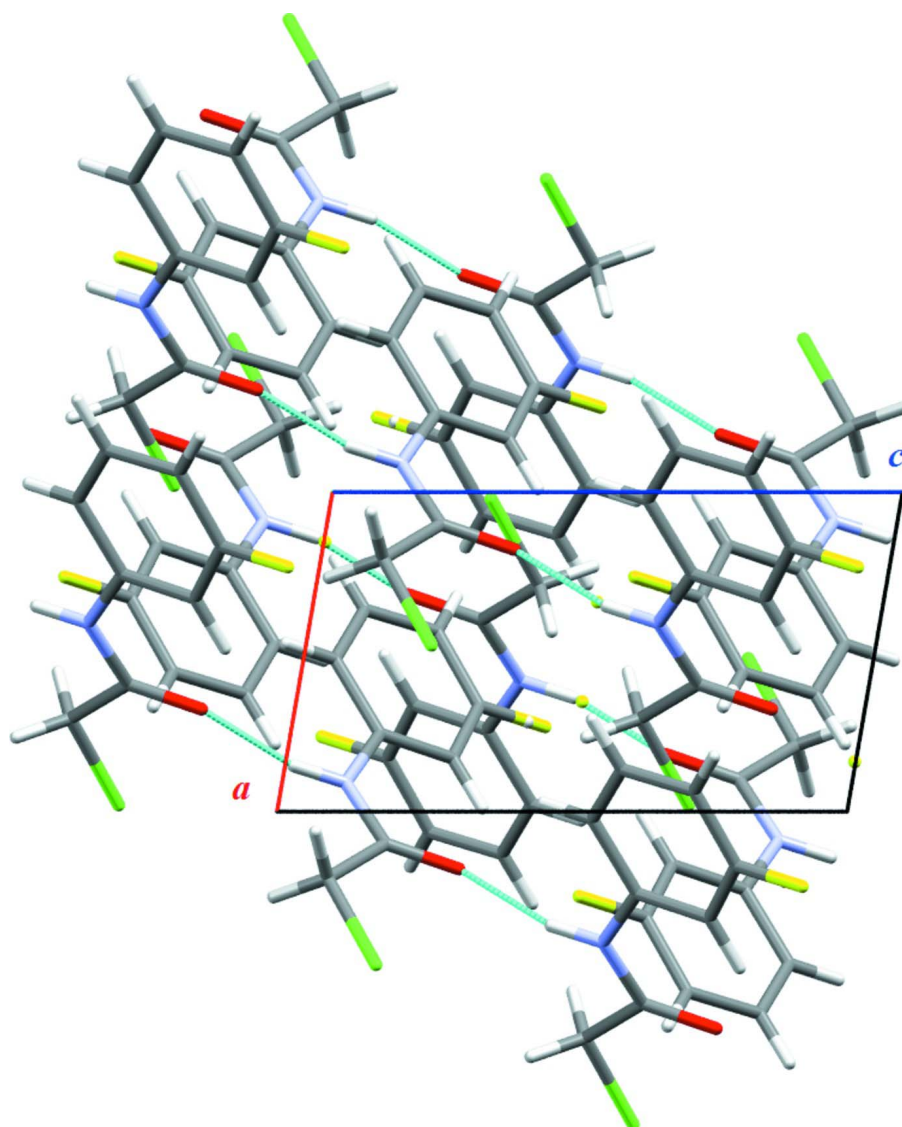


Figure 2

Crystal packing of (I). N—H...O hydrogen bonds are shown as dotted lines.

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

Crystal data

C_8H_7ClFNO

$M_r = 187.60$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 5.0441\ (2)\ \text{\AA}$

$b = 18.2374\ (7)\ \text{\AA}$

$c = 8.8653\ (3)\ \text{\AA}$

$\beta = 99.843\ (1)^\circ$

$V = 803.53\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 384$

Prism

$D_x = 1.551\ \text{Mg m}^{-3}$

Melting point: 385 K

Cu $K\alpha$ radiation, $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 1297 reflections

$\theta = 5.6\text{--}64.3^\circ$

$\mu = 3.95\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, colourless

$0.31 \times 0.24 \times 0.19\ \text{mm}$

Data collection

Bruker APEXII diffractometer	1304 independent reflections
Radiation source: fine-focus sealed tube	1297 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.041$
phi and φ scans	$\theta_{\text{max}} = 64.3^\circ$, $\theta_{\text{min}} = 5.6^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$h = -5 \rightarrow 5$
$T_{\text{min}} = 0.368$, $T_{\text{max}} = 0.472$	$k = -21 \rightarrow 21$
6045 measured reflections	$l = -9 \rightarrow 10$
	1 standard reflections every 1 reflections
	intensity decay: 1%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 0.4965P]$
$S = 1.15$	where $P = (F_o^2 + 2F_c^2)/3$
1304 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
123 parameters	$\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.25 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. Melting point was determined by using open capillary. FT—IR Spectrum was recorded on Jasco FT—IR Spectrometer. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra were recorded on Jeol-400 MHz NMR instrument using DMSO- d_6 as solvent. Chemical shift values were expressed in δ (p.p.m.) relative to tetramethylsilane (TMS) as an internal reference standard. Mass spectrum of the compound was recorded on Shimadzu LC-2010EV with ESI probe. The analysis of various spectra are as follows.

IR wavenumbers (cm^{-1}): C=O 1674.9, C—N 1348–1060, N—H 3510–3120, C—N—C 515–409, C—Cl 850–550, C—Cl 650–515. $^1\text{H-NMR}$ (399.6 MHz, DMSO- d_6) δ : 10.49 (s, 1H, NH), 7.57–7.55 (dd, 1H, Ar—H), 7.34–7.27 (m, 2H, Ar—H), 6.88–6.83 (m, 1H, Ar—H), 2.47 (s, 2H, —CH $_2$ —). $^{13}\text{C-NMR}$ (100 MHz, DMSO- d_6) δ : 165.41, 163.76, 140.67, 130.89, 115.54, 110.80, 106.77, 43.92. MS: Predicted Mass: 187.07; Obtained Mass 188.07 ($M+1$).

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
H1	−0.643 (5)	0.2133 (14)	0.515 (3)	0.032 (7)*	
Cl1	−0.02090 (10)	0.34713 (3)	0.71994 (6)	0.0279 (2)	
O1	−0.3206 (3)	0.22817 (8)	0.83631 (16)	0.0277 (4)	
N1	−0.5907 (3)	0.19920 (9)	0.61148 (19)	0.0191 (4)	
C1	−0.7435 (4)	0.14033 (11)	0.6567 (2)	0.0193 (4)	
C7	−0.3994 (4)	0.23849 (10)	0.7000 (2)	0.0200 (4)	
C6	−0.9766 (4)	0.12164 (11)	0.5553 (2)	0.0222 (5)	

H6	-1.0266	0.1475	0.4645	0.027*	
C2	-0.6672 (4)	0.10139 (11)	0.7913 (2)	0.0220 (5)	
H2	-0.5110	0.1133	0.8589	0.026*	
C4	-1.0643 (4)	0.02475 (12)	0.7255 (3)	0.0286 (5)	
H4	-1.1717	-0.0135	0.7496	0.034*	
C8	-0.2919 (4)	0.29966 (12)	0.6089 (2)	0.0271 (5)	
H8A	-0.4359	0.3340	0.5733	0.032*	
H8B	-0.2326	0.2787	0.5198	0.032*	
C5	-1.1321 (4)	0.06407 (12)	0.5923 (3)	0.0271 (5)	
H5	-1.2875	0.0516	0.5247	0.033*	0.574 (4)
F1A	-1.3441 (5)	0.04244 (15)	0.5018 (3)	0.0275 (9)	0.426 (4)
C3	-0.8308 (5)	0.04424 (12)	0.8222 (3)	0.0273 (5)	
H3	-0.7808	0.0178	0.9122	0.033*	0.426 (4)
F1	-0.7655 (5)	0.00473 (14)	0.9442 (3)	0.0394 (8)	0.574 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0277 (3)	0.0285 (4)	0.0258 (4)	-0.00754 (18)	-0.0003 (2)	-0.00248 (19)
O1	0.0374 (9)	0.0285 (8)	0.0142 (8)	-0.0067 (6)	-0.0040 (6)	0.0009 (6)
N1	0.0232 (9)	0.0215 (8)	0.0118 (9)	-0.0014 (6)	0.0003 (6)	0.0004 (6)
C1	0.0211 (10)	0.0193 (9)	0.0185 (10)	0.0015 (8)	0.0065 (8)	-0.0045 (8)
C7	0.0231 (10)	0.0214 (10)	0.0148 (10)	0.0020 (8)	0.0017 (8)	-0.0019 (8)
C6	0.0231 (10)	0.0247 (11)	0.0187 (10)	0.0012 (8)	0.0036 (8)	-0.0030 (8)
C2	0.0234 (10)	0.0259 (10)	0.0172 (10)	0.0009 (8)	0.0046 (8)	-0.0022 (8)
C4	0.0306 (12)	0.0244 (11)	0.0348 (13)	-0.0028 (9)	0.0166 (10)	-0.0036 (9)
C8	0.0321 (12)	0.0276 (11)	0.0193 (11)	-0.0079 (9)	-0.0020 (9)	0.0015 (9)
C5	0.0229 (11)	0.0278 (11)	0.0319 (12)	-0.0040 (8)	0.0080 (9)	-0.0111 (9)
F1A	0.0209 (15)	0.0318 (17)	0.0282 (17)	-0.0062 (11)	-0.0001 (11)	-0.0039 (12)
C3	0.0348 (12)	0.0252 (11)	0.0252 (11)	0.0023 (9)	0.0143 (9)	0.0017 (9)
F1	0.0487 (16)	0.0436 (15)	0.0266 (13)	-0.0043 (11)	0.0082 (10)	0.0146 (11)

Geometric parameters (Å, °)

C11—C8	1.768 (2)	C2—H2	0.9300
O1—C7	1.221 (2)	C4—C5	1.374 (3)
N1—C7	1.342 (3)	C4—C3	1.380 (3)
N1—C1	1.419 (3)	C4—H4	0.9300
N1—H1	0.89 (2)	C8—H8A	0.9700
C1—C2	1.386 (3)	C8—H8B	0.9700
C1—C6	1.394 (3)	C5—F1A	1.284 (4)
C7—C8	1.530 (3)	C5—H5	0.9300
C6—C5	1.383 (3)	C3—F1	1.295 (3)
C6—H6	0.9300	C3—H3	0.9300
C2—C3	1.385 (3)		
C7—N1—C1	127.59 (17)	C3—C4—H4	121.3
C7—N1—H1	118.2 (17)	C7—C8—C11	111.91 (14)

C1—N1—H1	113.9 (17)	C7—C8—H8A	109.2
C2—C1—C6	120.61 (19)	C11—C8—H8A	109.2
C2—C1—N1	123.14 (18)	C7—C8—H8B	109.2
C6—C1—N1	116.23 (18)	C11—C8—H8B	109.2
O1—C7—N1	125.18 (19)	H8A—C8—H8B	107.9
O1—C7—C8	123.40 (18)	F1A—C5—C4	115.8 (2)
N1—C7—C8	111.42 (16)	F1A—C5—C6	122.1 (2)
C5—C6—C1	118.9 (2)	C4—C5—C6	122.1 (2)
C5—C6—H6	120.6	C4—C5—H5	118.9
C1—C6—H6	120.6	C6—C5—H5	118.9
C3—C2—C1	117.9 (2)	F1—C3—C4	116.4 (2)
C3—C2—H2	121.0	F1—C3—C2	120.6 (2)
C1—C2—H2	121.0	C4—C3—C2	123.1 (2)
C5—C4—C3	117.4 (2)	C4—C3—H3	118.5
C5—C4—H4	121.3	C2—C3—H3	118.5
C7—N1—C1—C2	-18.4 (3)	N1—C7—C8—C11	-175.19 (14)
C7—N1—C1—C6	163.16 (19)	C3—C4—C5—F1A	177.0 (2)
C1—N1—C7—O1	1.9 (3)	C3—C4—C5—C6	-0.8 (3)
C1—N1—C7—C8	-177.30 (18)	C1—C6—C5—F1A	-177.6 (2)
C2—C1—C6—C5	0.7 (3)	C1—C6—C5—C4	0.0 (3)
N1—C1—C6—C5	179.19 (17)	C5—C4—C3—F1	-177.5 (2)
C6—C1—C2—C3	-0.7 (3)	C5—C4—C3—C2	0.8 (3)
N1—C1—C2—C3	-179.04 (18)	C1—C2—C3—F1	178.1 (2)
O1—C7—C8—C11	5.6 (3)	C1—C2—C3—C4	-0.1 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C2—H2...O1	0.93	2.33	2.885 (3)	118
N1—H1...O1 ⁱ	0.89 (2)	1.99 (3)	2.843 (2)	160 (2)

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