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3-Chloro-5-(trifluoromethyl)pyridin-2-amine

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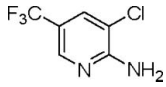
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.049; wR factor = 0.137; data-to-parameter ratio = 9.4.

In the title compound, $C_6H_4ClF_3N_2$, an intermediate in the synthesis of the fungicide fluazinam, the F atoms of the trifluoromethyl group are disordered over two sites in a 0.683 (14):0.317 (14) ratio. In the crystal structure, centrosymmetric dimers arise from pairs of N—H···N hydrogen bonds.

Related literature

For related literature, see: Guo *et al.* (1991).



Experimental

Crystal data

$C_6H_4ClF_3N_2$
M_r = 196.56
Monoclinic, $P2_1/n$
a = 5.801 (1) Å
b = 17.978 (5) Å
c = 7.578 (2) Å
β = 100.19 (4)°
V = 777.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.893, T_{max} = 0.909
3820 measured reflections
1368 independent reflections
904 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.137
S = 1.01
1368 reflections
145 parameters
39 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl1	0.89 (2)	2.60 (3)	2.965 (9)	105.3 (19)
N2—H2B···N1^{i}	0.89 (3)	2.16 (3)	3.049 (9)	171 (3)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2620).

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Guo, Z.-J., Miyoshi, H., Komyoji, T., Haga, T. & Fujita, T. (1991). *Biochim. Biophys. Acta*, **1056**, 89–92.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supporting information

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S1. Comment

The title compound, (I), is an intermediate in the preparation of fluazinam or 3-chloro-*N*-[3-chloro-2,6-dinitro- 4-(trifluoromethyl)phenyl]-5-(trifluoromethyl)-2-pyridinamine which is a kind of pyridine fungicide (Guo *et al.*, 1991).

The F atoms of the trifluoromethyl group are disordered over two sites in a 0.683 (14):0.317 (14) ratio (Fig. 1). An acute intramolecular N—H···Cl interaction occurs and the packing is consolidated by an N—H···N hydrogen bond (Table 1) resulting in inversion dimers.

S2. Experimental

A mixture of 2,3-dichloro-5-(trifluoromethyl)pyridine (216 g, 1 mol) and NH~3 (68 g, 4 mol) in ethanol was heated to 50 atm. After 10 h, the reaction was complete, the resulting solid was filtered off and washed with a little cool ethanol. 50 mg of (I) was dissolved in 20 ml e thanol and the solution was kept at room temperature for 10 d; natural evaporation gave colourless blocks of (I).

S3. Refinement

The N-bound H atoms were located in a difference map and freely refined. The C-bound H atoms were positioned geometrically, with C—H = 0.93Å and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

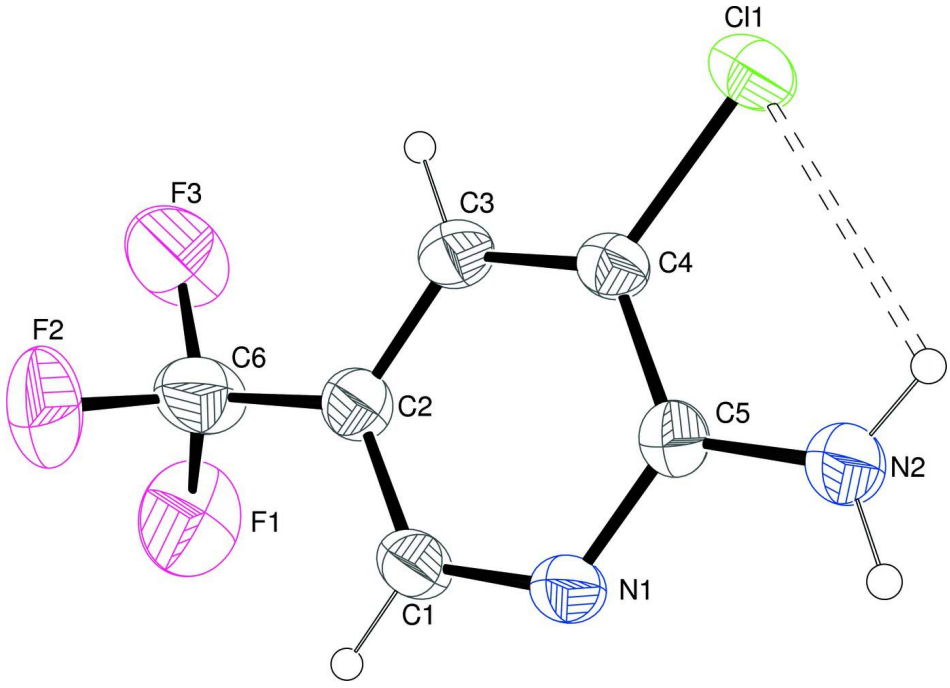

Figure 1

The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms). Only one orientation of the –CF_3 group is shown. The N—H···Cl interaction is shown as a double-dashed line.

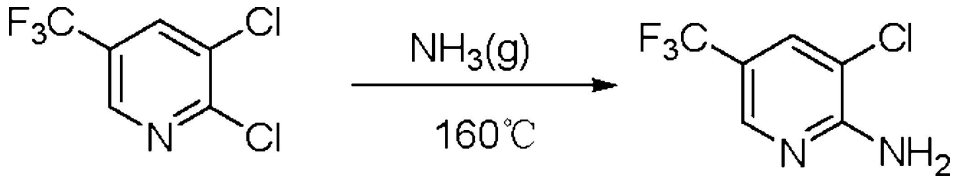

Figure 2

The formation of the title compound.

3-Chloro-5-(trifluoromethyl)pyridin-2-amine

Crystal data

$C_6H_4ClF_3N_2$
M_r = 196.56
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 5.801 (1) Å
b = 17.978 (5) Å
c = 7.578 (2) Å
β = 100.19 (4)°
V = 777.8 (3) Å^3
Z = 4

$F(000)$ = 392
D_x = 1.678 Mg m^{-3}
Melting point = 145–146 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1210 reflections
θ = 2.9–25.9°
μ = 0.49 mm^{-1}
T = 294 K
Block, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.893, T_{max} = 0.909
3820 measured reflections
1368 independent reflections
904 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 25.0°, θ_{min} = 2.3°
$h = -5 \rightarrow 6$
$k = -21 \rightarrow 20$
$l = -9 \rightarrow 5$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.137
S = 1.02
1368 reflections
145 parameters
39 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.3792P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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}	Occ. (<1)
Cl1	0.19339 (18)	0.59163 (5)	0.35440 (14)	0.0682 (4)	
F1	0.075 (2)	0.2514 (7)	0.3252 (15)	0.117 (4)	0.683 (14)
F2	0.2861 (13)	0.2807 (3)	0.1285 (11)	0.108 (2)	0.683 (14)
F3	0.4036 (17)	0.3071 (3)	0.3974 (16)	0.131 (4)	0.683 (14)
F1′	0.078 (4)	0.2487 (13)	0.256 (3)	0.097 (6)	0.317 (14)
F2′	0.392 (4)	0.2973 (9)	0.246 (4)	0.127 (6)	0.317 (14)
F3′	0.267 (3)	0.3002 (6)	0.4870 (18)	0.107 (5)	0.317 (14)
N1	−0.2580 (5)	0.44234 (16)	0.1206 (4)	0.0537 (8)	
N2	−0.2851 (6)	0.56838 (18)	0.1481 (5)	0.0658 (9)	
C1	−0.1364 (6)	0.3800 (2)	0.1569 (5)	0.0548 (9)	
H1	−0.2072	0.3355	0.1145	0.066*	
C2	0.0859 (6)	0.3770 (2)	0.2526 (5)	0.0540 (9)	
C3	0.1917 (6)	0.4434 (2)	0.3143 (5)	0.0540 (9)	
H3	0.3441	0.4439	0.3781	0.065*	
C4	0.0705 (6)	0.50724 (19)	0.2804 (4)	0.0460 (8)	

C5	−0.1582 (6)	0.50666 (18)	0.1823 (4)	0.0468 (9)
C6	0.2091 (10)	0.3053 (3)	0.2854 (8)	0.0837 (15)
H2A	−0.230 (5)	0.6135 (10)	0.181 (5)	0.070 (13)*
H2B	−0.426 (4)	0.5678 (18)	0.078 (4)	0.076 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0704 (7)	0.0546 (6)	0.0765 (7)	−0.0139 (5)	0.0049 (5)	−0.0129 (5)
F1	0.143 (5)	0.074 (5)	0.141 (7)	0.016 (4)	0.037 (6)	0.046 (5)
F2	0.132 (4)	0.077 (3)	0.116 (5)	0.042 (3)	0.027 (4)	−0.011 (3)
F3	0.120 (5)	0.092 (3)	0.150 (6)	0.039 (3)	−0.065 (5)	−0.017 (4)
F1′	0.110 (9)	0.053 (7)	0.115 (10)	0.006 (6)	−0.014 (8)	−0.012 (8)
F2′	0.114 (9)	0.123 (8)	0.153 (11)	0.054 (7)	0.052 (9)	0.024 (8)
F3′	0.135 (9)	0.077 (6)	0.097 (7)	0.039 (6)	−0.009 (7)	0.020 (5)
N1	0.0483 (17)	0.0481 (17)	0.0600 (19)	−0.0008 (14)	−0.0036 (14)	−0.0038 (14)
N2	0.060 (2)	0.0489 (19)	0.082 (2)	0.0067 (17)	−0.0063 (18)	−0.0031 (17)
C1	0.058 (2)	0.046 (2)	0.059 (2)	−0.0055 (18)	0.0029 (18)	−0.0059 (16)
C2	0.055 (2)	0.049 (2)	0.055 (2)	0.0041 (18)	0.0020 (18)	0.0006 (16)
C3	0.046 (2)	0.059 (2)	0.054 (2)	0.0014 (18)	−0.0015 (16)	−0.0009 (17)
C4	0.048 (2)	0.048 (2)	0.0419 (19)	−0.0069 (16)	0.0080 (16)	−0.0039 (15)
C5	0.052 (2)	0.0460 (19)	0.0430 (19)	0.0037 (17)	0.0091 (16)	0.0007 (15)
C6	0.076 (4)	0.060 (3)	0.105 (4)	0.000 (3)	−0.012 (3)	0.002 (3)

Geometric parameters (Å, º)

Cl1—C4	1.727 (5)	N2—H2A	0.891 (11)
F1—C6	1.311 (13)	N2—H2B	0.895 (11)
F2—C6	1.414 (9)	C1—C2	1.364 (6)
F3—C6	1.287 (7)	C1—H1	0.9300
F1′—C6	1.27 (2)	C2—C3	1.385 (6)
F2′—C6	1.162 (14)	C2—C6	1.474 (6)
F3′—C6	1.508 (15)	C3—C4	1.347 (5)
N1—C1	1.327 (5)	C3—H3	0.9300
N1—C5	1.340 (5)	C4—C5	1.401 (6)
N2—C5	1.332 (5)		
C1—N1—C5	118.4 (3)	F2′—C6—F3	55.6 (11)
C5—N2—H2A	123 (2)	F1′—C6—F3	124.4 (11)
C5—N2—H2B	121 (2)	F2′—C6—F1	125.0 (11)
H2A—N2—H2B	115.1 (19)	F1′—C6—F1	23.6 (11)
N1—C1—C2	124.1 (3)	F3—C6—F1	110.7 (8)
N1—C1—H1	117.9	F2′—C6—F2	46.1 (14)
C2—C1—H1	117.9	F1′—C6—F2	82.5 (11)
C1—C2—C3	117.8 (3)	F3—C6—F2	101.0 (7)
C1—C2—C6	120.6 (4)	F1—C6—F2	104.5 (7)
C3—C2—C6	121.6 (4)	F2′—C6—C2	120.2 (8)
C4—C3—C2	119.0 (4)	F1′—C6—C2	114.6 (12)

C4—C3—H3	120.5	F3—C6—C2	115.6 (5)
C2—C3—H3	120.5	F1—C6—C2	113.4 (8)
C3—C4—C5	120.5 (3)	F2—C6—C2	110.4 (5)
C3—C4—Cl1	121.0 (3)	F2′—C6—F3′	101.6 (12)
C5—C4—Cl1	118.4 (3)	F1′—C6—F3′	98.5 (10)
N2—C5—N1	117.5 (3)	F3—C6—F3′	47.3 (6)
N2—C5—C4	122.4 (3)	F1—C6—F3′	75.9 (8)
N1—C5—C4	120.1 (3)	F2—C6—F3′	141.9 (7)
F2′—C6—F1′	113.8 (16)	C2—C6—F3′	103.8 (6)
C5—N1—C1—C2	−0.5 (5)	C1—C2—C6—F2′	125.8 (19)
N1—C1—C2—C3	−0.6 (6)	C3—C2—C6—F2′	−53 (2)
N1—C1—C2—C6	−179.5 (4)	C1—C2—C6—F1′	−15.4 (13)
C1—C2—C3—C4	1.2 (5)	C3—C2—C6—F1′	165.7 (12)
C6—C2—C3—C4	−179.9 (4)	C1—C2—C6—F3	−170.6 (9)
C2—C3—C4—C5	−0.8 (5)	C3—C2—C6—F3	10.5 (11)
C2—C3—C4—Cl1	179.9 (3)	C1—C2—C6—F1	−41.3 (9)
C1—N1—C5—N2	−178.4 (3)	C3—C2—C6—F1	139.8 (7)
C1—N1—C5—C4	0.9 (5)	C1—C2—C6—F2	75.5 (6)
C3—C4—C5—N2	179.0 (3)	C3—C2—C6—F2	−103.3 (6)
Cl1—C4—C5—N2	−1.6 (5)	C1—C2—C6—F3′	−121.6 (9)
C3—C4—C5—N1	−0.3 (5)	C3—C2—C6—F3′	59.5 (10)
Cl1—C4—C5—N1	179.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl1	0.89 (2)	2.60 (3)	2.965 (9)	105 (2)
N2—H2*B*···N1^{i}	0.89 (3)	2.16 (3)	3.049 (9)	171 (3)

Symmetry code: (i) −*x*−1, −*y*+1, −*z*.