

N*-{3-Cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-1*H*-pyrazol-5-yl}acetamide*Zhiping Yang,[‡] Ping Zhong* and Shengrong Guo**

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The title compound, C₁₃H₇Cl₂F₃N₄O, has normal bond lengths and angles. The crystal packing is stabilized by intermolecular N—H···N hydrogen bonds.

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Comment

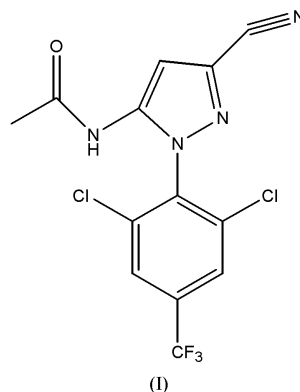
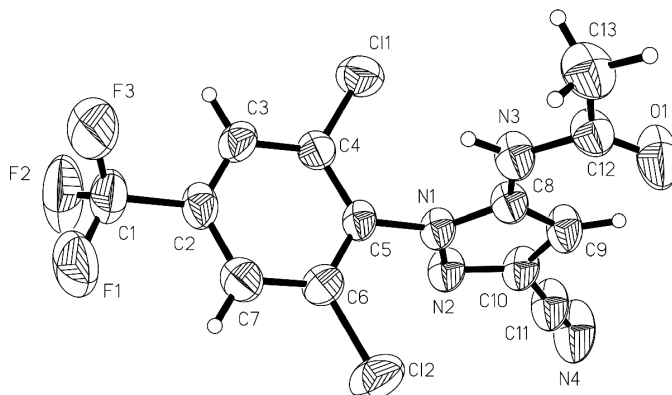
Reaction of 2,6-dichloro-4-trifluoromethylamine with a suspension of nitrosyl sulfuric acid, followed by reaction with a solution of ethyl 2,3-dicyanopropionate in acetic acid, gave 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-pyrazole, which then reacted with acetic anhydride to give the title compound, (I) (Fig. 1).

Key indicators

Single-crystal X-ray study

T = 298 KMean σ (C—C) = 0.004 Å*R* factor = 0.064*wR* factor = 0.170

Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound is used in the synthesis of insecticides, such as 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethylthio)pyrazole, 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethylsulfenyl)pyrazole and 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethylsulfonyl)pyrazole (Hatton *et al.*, 1993).**Figure 1**
View of (I), with the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

All bond lengths and angles in (I) are normal (Table 1). The crystal packing (Fig. 2) is stabilized by the weak intermolecular N—H···N hydrogen bonds (Table 2).

Experimental

Compound (I) was synthesized and purified according to the method of Hatton *et al.* (1993). Single crystals suitable for X-ray data collection were obtained by slow evaporation of an ethyl acetate–toluene (1:1) solution (m.p. 477–478 K). Spectroscopic analysis, IR (KBr, ν cm⁻¹): 3446, 2383, 1698, 1649, 1520; ¹H NMR (CDCl₃, δ , p.p.m.): 7.82 (s, 2H), 7.08 (s, 1H), 7.00 (s, 1H), 2.13 (s, 3H).

Crystal data

C ₁₃ H ₇ Cl ₂ F ₃ N ₄ O	$D_x = 1.559$ Mg m ⁻³
$M_r = 363.13$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 2156 reflections
$a = 18.8282$ (6) Å	$\theta = 2.6$ – 25.2°
$b = 8.6541$ (3) Å	$\mu = 0.46$ mm ⁻¹
$c = 19.0766$ (6) Å	$T = 298$ (2) K
$\beta = 95.583$ (1) $^\circ$	Block, colorless
$V = 3093.62$ (18) Å ³	$0.44 \times 0.16 \times 0.13$ mm
$Z = 8$	

Data collection

Bruker SMART APEX area-detector diffractometer	2768 independent reflections
φ and ω scans	2373 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$R_{int} = 0.025$
$T_{min} = 0.824$, $T_{max} = 0.943$	$\theta_{max} = 25.3^\circ$
7655 measured reflections	$h = -22 \rightarrow 20$
	$k = -10 \rightarrow 8$
	$l = -18 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.079P)^2 + 4.3533P]$
$R[F^2 > 2\sigma(F^2)] = 0.064$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.170$	$(\Delta/\sigma)_{max} = 0.003$
$S = 1.14$	$\Delta\rho_{max} = 0.65$ e Å ⁻³
2768 reflections	$\Delta\rho_{min} = -0.52$ e Å ⁻³
209 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected geometric parameters (Å, $^\circ$).

C1—C4	1.720 (3)	N4—C11	1.130 (5)
N1—C8	1.360 (4)	F1—C1	1.330 (5)
N1—N2	1.361 (3)	C1—C2	1.497 (5)
N1—C5	1.421 (3)	C8—C9	1.364 (4)
N2—C10	1.321 (4)	C9—C10	1.383 (5)
N3—C8	1.378 (4)	C10—C11	1.441 (5)
C8—N1—N2	112.6 (2)	C5—C4—C11	119.2 (2)
N2—N1—C5	119.1 (2)	C4—C5—N1	121.4 (3)
C10—N2—N1	102.6 (2)	N1—C8—N3	120.5 (3)
F3—C1—C2	112.4 (3)	N2—C10—C11	119.9 (3)
C7—C2—C1	119.8 (3)		

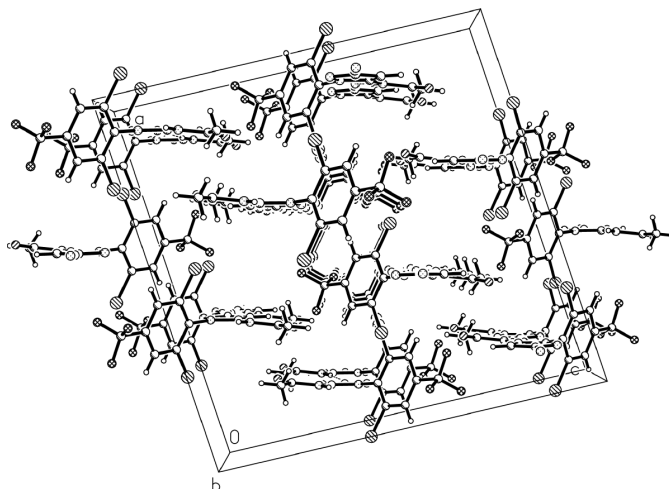


Figure 2

The packing of (I), viewed down the b axis.

Table 2

Hydrogen-bonding geometry (Å, $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3···N4 ⁱ	0.86	2.22	3.060 (4)	166

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

All H atoms were initially located in a difference Fourier map and were placed in geometrically idealized position and constrained to ride on their parent atom, with C—H distances in the range 0.95–1.00 Å and $U_{iso}(H) = 1.2c_q(C)$.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP in SHELXTL (Bruker, 2002); software used to prepare material for publication: SHELXL97.

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