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5-Amino-4-chloro-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-1*H*-pyrazole-3-carbonitrile

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In the title compound, $C_{11}H_4Cl_3F_3N_4$, the dihedral angle between the pyrazole and the N-substituted benzene ring planes is 84.5 (2)°. The crystal structure is stabilized by N—H···N hydrogen bonds.

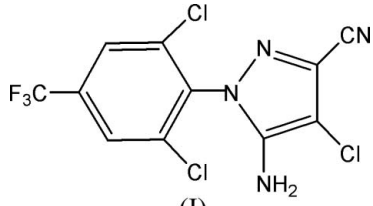
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.069
wR factor = 0.174
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), is similar to the very effective insecticides used to treat animals such as cows and sheep (Philippe, 1997, 2000) and its structure is reported here (Fig. 1 and Table 1). The molecule contains two essentially planar rings, with maximum deviations 0.012 (5) Å for atom C3 from the benzene and 0.004 (2) Å for atom C8 from the pyrazole ring planes. The dihedral angle between these planes is 84.5 (2)°. In the crystal structure, N—H···N interactions form ribbons along the *a* axis, and these ribbons are linked to form a three-dimensional network *via* N4—H4*B*···N3^{ii} hydrogen bonds (Table 1 and Fig. 2).



(I)

Experimental

5-Amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]pyrazole was prepared by the method of Hatton *et al.* (1993). Under a nitrogen atmosphere with the exclusion of moisture, cooling at 273–278 K, a sample of 1.3 mmol sulfonyl dichloride was added dropwise with

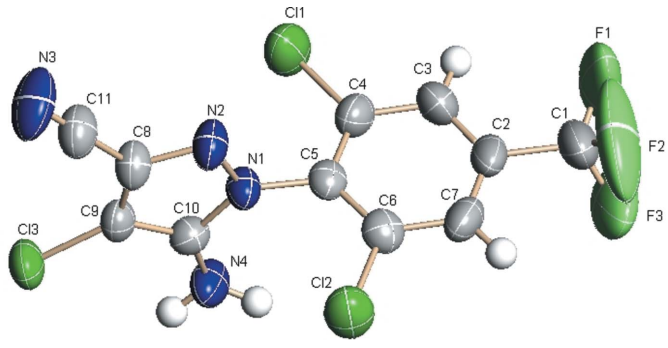


Figure 1
The molecular structure of (I), showing the atom numbering scheme and displacement ellipsoids at the 50% probability level.

continuous stirring to a mixture of 1 mmol 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]pyrazole in 5 ml ethyl acetate, afer stirring for 4 h at 303–323 K, to give the title compound in 93% yield (Okui, 2005). Colorless single crystals suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol–acetone (2:1) solution of (I) (m.p. 452–454 K).

Crystal data

$C_{11}H_4Cl_3F_3N_4$
M_r = 355.53
Orthorhombic, $Pna2_1$
a = 11.4138 (9) Å
b = 9.5508 (8) Å
c = 13.5082 (11) Å
V = 1472.5 (2) Å^3
Z = 4
D_x = 1.604 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 298 (2) K
Block, colorless
0.24 × 0.10 × 0.09 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.859, T_{max} = 0.944
7390 measured reflections
2609 independent reflections
2169 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.174
S = 1.07
2609 reflections
196 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0912P)^2 + 0.7048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 1226 Friedel pairs
Flack parameter: 0.08 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N2^{i}	0.84 (2)	2.35 (4)	3.125 (7)	153 (7)
N4—H4B···N3^{ii}	0.84 (2)	2.26 (2)	3.098 (9)	172 (7)

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

All H atoms were initially located in a difference Fourier map, but the aromatic H atoms were then placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å, and U_{iso}(H) = 1.2$_{eq}$(C). The H atoms on N4 were refined with fixed isotropic displacement parameters (0.08 Å^2) and with their bond distances restrained to 0.84 (2) Å. The high displacement parameters for atoms F1, F2 and F3 indicate disorder in the trifluoromethyl group. However, attempts to refine an appropriate disorder model were unsuccessful. The inability to suitably account for the electron-density distribution in the vicinity of the CF_3 group limits the overall precision of the structure.

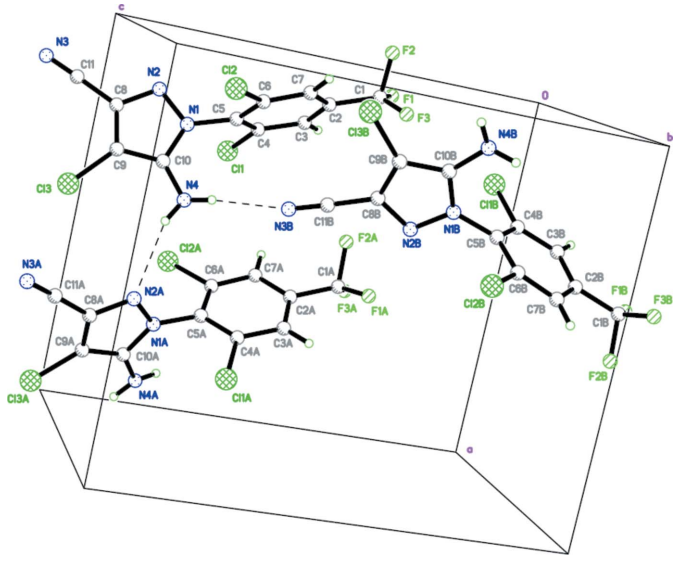


Figure 2
Packing diagram for (I). Hydrogen bonds are shown as dashed lines.

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: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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