Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-{3-Cyano-1-[2,6-dichloro-4-(trifluoromethyl)-phenyl]-1*H*-pyrazol-5-yl}benzamide

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.066
wR factor = 0.168
Data-to-parameter ratio = 13.2

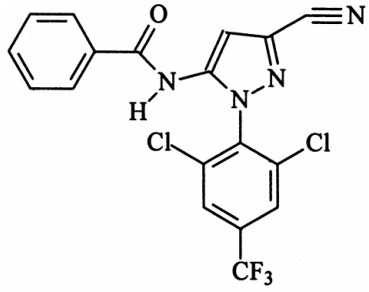
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 November 2004
Accepted 16 November 2004
Online 27 November 2004

The title compound, $C_{18}H_9Cl_2F_3N_4O$, is a tricyclic amide with an overall U-shape. N—H···N hydrogen bonds generate linear chains which extend along the *a* axis.

Comment

The title compound, (I), has been used to synthesize 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-(trifluoromethyl)thiopyrazole, 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-(trifluoromethyl)sulfenylpyrazole and 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-(trifluoromethyl)sulfonylpyrazole, which are all good insecticides (Hatton *et al.*, 1993).



(I)

The structure is shown in Fig. 1, with the atom-numbering scheme. The molecule contains three planar groups, forming an overall U-shape, *viz*. a 2,6-dichloro-4-(trifluoromethyl)-phenyl, a pyrazole and a benzene ring. The dihedral angles between the pyrazole and the C2–C7 and C13–C18 benzene rings are 77.47 (13) and 17.81 (24)°, respectively. In the crystal structure, N—H···N hydrogen bonds (Table 2) result in the formation of linear chains parallel to the *a* axis (Fig. 2).

Experimental

Following the method of Hatton *et al.* (1993), 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 was then reacted with benzoyl chloride to give the title compound, (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate/cyclohexane (1/1) solution (m.p. 483–485 K). IR (KBr, ν cm^{-1}): 3302, 3169, 3065 2246, 1695, 1547; ^{1}H NMR ($CDCl_3$): δ 10.13 (*s*,1H), 8.11 (*s*, 2H), 7.73 (*d*, 2H), 7.58 (*t*, 1H), 7.45 (*m*, 2H), 7.36 (*s*, 1H).

Crystal data

$C_{18}H_9Cl_2F_3N_4O$
M_r = 425.19
Triclinic, $P\overline{1}$
a = 8.4613 (11) Å
b = 9.8923 (13) Å
c = 11.4305 (15) Å
α = 91.463 (2)°
β = 96.002 (2)°
γ = 101.119 (2)°
V = 932.6 (2) Å^3
Z = 2
D_x = 1.514 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1331 reflections
θ = 2.5–24.6°
μ = 0.39 mm^{-1}
T = 298 (2) K
Block, colorless
0.28 × 0.18 × 0.13 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.898, T_{max} = 0.951
4988 measured reflections
3347 independent reflections
2427 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.5°
$h = -10 \rightarrow 9$
$k = -7 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.168
S = 1.03
3347 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0761P)^2 + 0.7071P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.721 (4)	N3—C11	1.135 (5)
F1—C1	1.298 (6)	N4—C12	1.374 (5)
O1—C12	1.213 (4)	N4—C8	1.384 (4)
N1—N2	1.356 (4)	C8—C9	1.361 (5)
N1—C8	1.361 (4)	C9—C10	1.396 (5)
N1—C5	1.431 (4)	C10—C11	1.451 (5)
N2—C10	1.327 (4)	C12—C13	1.479 (5)
N2—N1—C8	112.7 (3)	C8—C9—C10	103.9 (3)
N2—N1—C5	117.7 (3)	N2—C10—C9	113.9 (3)
C8—N1—C5	129.5 (3)	N2—C10—C11	118.0 (3)
C10—N2—N1	102.6 (3)	C9—C10—C11	128.1 (3)
C12—N4—C8	123.7 (3)	N3—C11—C10	177.8 (5)
F1—C1—F3	110.4 (5)	O1—C12—N4	121.6 (4)
F3—C1—C2	113.4 (4)	O1—C12—C13	122.7 (4)
N1—C8—C9	107.0 (3)	N4—C12—C13	115.7 (3)
N1—C8—N4	119.7 (3)	C18—C13—C12	118.9 (4)
C9—C8—N4	133.3 (3)	C14—C13—C12	122.8 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N3^{i}	0.86	2.33	3.149 (4)	159

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

All H atoms were initially located in a difference Fourier map but were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and with U_{iso}(H) = 1.2$_{eq}$(C). Although the F atoms display large ellipsoids, no disorder model could be defined.

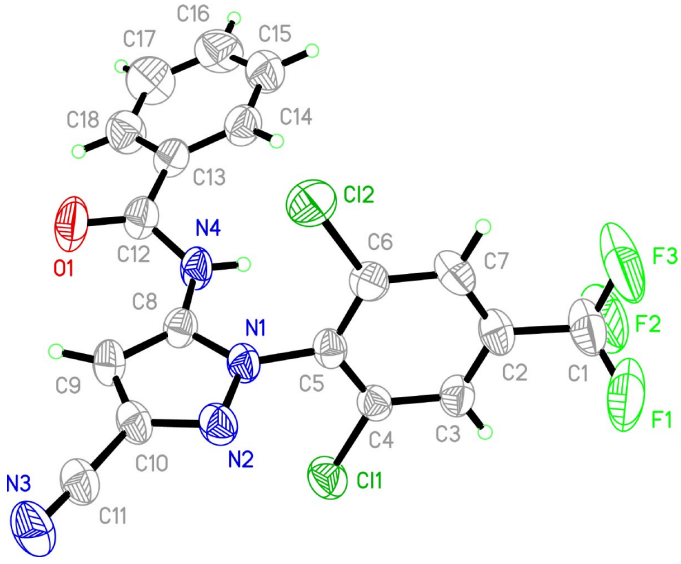


Figure 1
The structure of (I), showing the atomic numbering scheme, with displacement ellipsoids drawn at the 50% probability level.

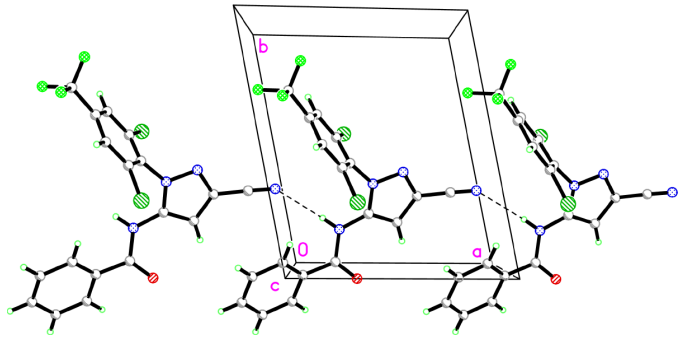


Figure 2
Packing diagram, viewed down the c axis, showing the linear chain generated by N—H···N hydrogen bonds (dashed lines).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL*97.

This work was supported by the National Natural Science Foundation of China (No. 20272043) and the Natural Science Foundation of Zhejiang Province (No. M203001).

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