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

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.054
wR factor = 0.125
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.

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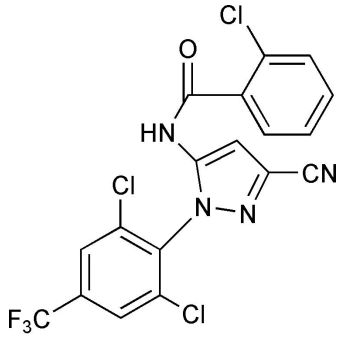
The molecule of the title compound, $C_{18}H_8Cl_3F_3N_4O$, is a tricyclic amide with an overall U-shape. Intermolecular N—H···N hydrogen bonds, with an N(amide)···N(cyano) separation of 3.051 (4) Å, link the molecules into linear chains along the *c* axis.

Comment

The title compound, (I), is an important material for the synthesis of 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethyl)thiopyrazole, 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-(trifluoromethyl)sulfonyl pyrazole, which are well known insecticides (Hatton *et al.*, 1993).



(I)

The structure of (I) is shown in Fig. 1. The molecule contains three planar groups forming an overall U-shape, *viz*. 2,6-dichloro-4-(trifluoromethyl)phenyl, pyrazole and 2-chlorobenzoyl rings. The dihedral angles between the pyrazole and the C2–C7 and C13–C18 benzene rings are 83.4 (2) and 136.5 (1)°, respectively. Intermolecular N—H···N hydrogen bonds (Table 1) link the molecules into linear chains along the *c* axis (Fig. 2).

Experimental

Following the method of Hatton *et al.* (1993), the reaction of 2,6-dichloro-4-(trifluoromethyl)aniline with a suspension of nitrosylsulfuric 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 refluxed with 2-chlorobenzoyl chloride and pyridine in chloroform for about 8 h to give the title compound, (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an acetone solution (m.p. 498–499 K). IR (KBr, ν cm^{-1}): 3271, 2248, 1697, 1553, 1369,

1313, 1173, 1138, 1046; ^{1}H NMR ($CDCl_3$): δ 8.22 (*s*,1H), 7.92 (*s*, 1H), 7.83 (*s*, 2H), 7.45 (*m*, 4H); ^{13}C NMR ($CDCl_3$): δ 164.6 (1C), 152.0 (1C), 138.0 (1C), 136.6 (1C), 136.5 (1C), 134.7 (1C), 132.2 (1C), 132.1 (1C), 131.6 (1C), 128.7 (1C), 126.1(1C), 125.1 (2C), 125.0 (2C), 112.9 (1C), 103.8 (1C), 101.2 (1C).

Crystal data

$C_{18}H_8Cl_3F_3N_4O$
M_r = 459.63
Monoclinic, $P2_1/c$
a = 10.3133 (8) Å
b = 22.7193 (18) Å
c = 8.5237 (7) Å
β = 104.580 (1)°
V = 1932.9 (3) Å^3
Z = 4
D_x = 1.579 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3120 reflections
θ = 2.2–24.1°
μ = 0.52 mm^{-1}
T = 298 (2) K
Block, colourless
0.42 × 0.24 × 0.14 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.811, T_{max} = 0.931
10 250 measured reflections
3509 independent reflections
3069 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.3°
$h = -10 \rightarrow 12$
$k = -27 \rightarrow 20$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.125
S = 1.11
3509 reflections
265 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 1.4918P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N4^{i}	0.85 (3)	2.21 (3)	3.051 (4)	168 (4)

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

Atom H3*A* attached to N3 was located in a difference Fourier map and refined with a restrained N—H distance of 0.86 (1) Å and U_{iso}(H) = 1.2U_{eq}(N). All other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å, with U_{iso} = 1.2U_{eq}(C), and Csp^3—H = 0.96 Å, with U_{iso} = 1.5U_{eq}(C). The crystal packing demonstrates short intermolecular contacts Cl1···O1 (2.92 Å) and Cl2···Cl3 (3.25 Å) caused by the presence of the strong electron-acceptor CF_3 group, which may decrease electron density on atoms Cl1 and Cl2, attached to the same benzene ring.

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

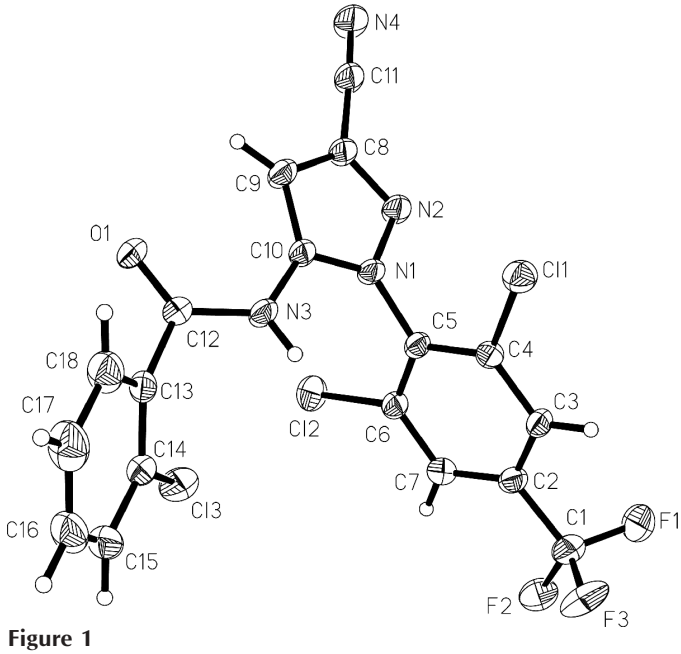


Figure 1
View of (I), showing the atom numbering and displacement ellipsoids at the 50% probability level.

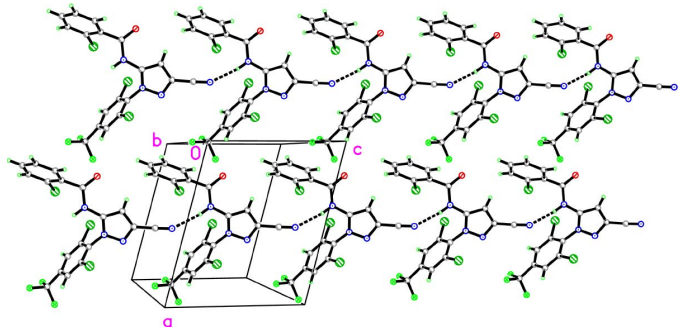


Figure 2
The crystal packing of (I), showing the hydrogen-bonded (dashed lines) linear chains along the *c* axis.

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