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

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.055
wR factor = 0.153
Data-to-parameter ratio = 9.1

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

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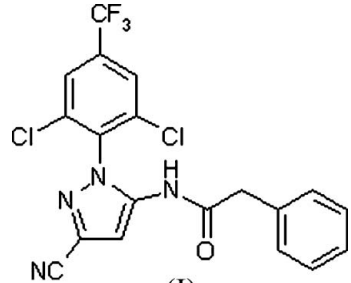
The molecule of the title compound, $C_{19}H_{11}Cl_2F_3N_4O$, is a tricyclic amide with an overall U shape. The dihedral angles between the pyrazole and outermost benzene and phenyl rings are 89.4 (1) and 114.5 (1)°, respectively. In the crystal structure, intermolecular N—H···N hydrogen bonds, with an N(amide)···N(cyano) separation of 3.220 (7) Å, link the molecules into linear chains along the [110] direction.

Comment

The title compound, (I), is an intermediate for the synthesis of 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylthiopyrazole, 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulphenylpyrazole and 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonylpyrazole, which are all good insecticides (Hatton *et al.*, 1993).



(I)

The tricyclic molecule of (I) (Fig. 1) adopts an overall U shape. The bond lengths and angles in (I) (Table 1) are in agreement with those observed in similar compounds (Zhong *et al.*, 2005; Zhang *et al.*, 2005). The dihedral angles between the pyrazole and benzene (C2–C7) and phenyl (C14–C19) rings are 89.4 (1) and 114.5 (1)°, respectively. In the crystal structure, intermolecular N—H···N hydrogen bonds (Table 2) link the molecules into linear chains along the [110] direction (Fig. 2).

Experimental

Following the method of Hatton *et al.* (1993), reaction of 2,6-dichloro-4-trifluoromethylamine (0.01 mol) with a suspension of nitrosylsulfuric acid, followed by reaction with a solution of ethyl 2,3-dicyanopropionate (0.01 mol) in acetic acid, gave 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole (about 0.005 mol), which was then refluxed with phenylacetyl chloride (0.005 mol) and pyridine in chloroform (10 ml) overnight to give the title compound, (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a solution in acetone (m.p. 463–465 K). IR (KBr,

cm^{-1}): ν 3312, 3075, 2924, 2244,1706, 1544, 1311, 1228, 1135; ^{1}H NMR ($CDCl_3$, p.p.m.): δ 9.70 (*s*, 1H), 8.08 (*s*, 2H), 7.25 (*q*, 6H), 3.58 (*s*, 2H); ^{13}C NMR ($CDCl_3$, p.p.m.): δ 168.1 (1C), 140.1(1C), 136.0 (1C), 135.9 (1C), 130.1 (1C), 129.4 (1C), 128.9 (1C),128.0 (1C), 127.9 (1C), 127.8 (1C), 127.5 (1C), 127.1 (1C), 125.2 (1C), 124.1 (1C), 120.4 (1C), 113.1 (1C), 102.8 (2C), 102.0 (1C), 100.2 (1C).

Crystal data

$C_{19}H_{11}Cl_2F_3N_4O$
M_r = 439.22
Monoclinic, *Cc*
a = 15.302 (8) Å
b = 8.604 (5) Å
c = 14.872 (8) Å
β = 90.25 (1)°
V = 1957.9 (19) Å^3
Z = 4
D_x = 1.490 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2534 reflections
θ = 2.7–25.0°
μ = 0.38 mm^{-1}
T = 298 (2) K
Block, colourless
0.42 × 0.28 × 0.25 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.858, T_{max} = 0.912
4794 measured reflections
2389 independent reflections
2254 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.1°
h = −17 → 18
k = −9 → 10
l = −17 → 12

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.153
S = 1.08
2389 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0814P)^2 + 2.8261P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 631 Friedel pairs
Flack parameter: 0.13 (13)

Table 1
Selected geometric parameters (Å, °).

O1—C12	1.201 (6)	C1—C2	1.486 (8)
N1—N2	1.377 (6)	C9—C10	1.356 (7)
N1—C10	1.379 (6)	C12—C13	1.524 (7)
N1—C5	1.421 (7)	C13—C14	1.512 (8)
N4—C12	1.396 (7)		
N2—N1—C10	112.0 (4)	C10—C9—C8	104.7 (4)
N2—N1—C5	120.7 (4)	C9—C10—N4	133.6 (5)
C10—N1—C5	127.1 (4)	O1—C12—N4	122.0 (5)
C8—N2—N1	102.6 (4)	O1—C12—C13	123.3 (5)
C7—C2—C3	120.8 (5)	N4—C12—C13	114.7 (4)
C4—C5—N1	120.9 (5)	C14—C13—C12	112.4 (4)
C6—C5—N1	119.6 (5)	C19—C14—C13	121.5 (5)
N2—C8—C9	114.0 (5)	C19—C18—C17	118.0 (6)
C10—N1—N2—C8	0.9 (6)	C10—N1—C5—C4	−93.0 (7)
C5—N1—N2—C8	175.7 (5)	N1—N2—C8—C9	−0.6 (6)
C7—C2—C3—C4	1.8 (8)	C8—C9—C10—N4	−178.2 (6)
N2—N1—C5—C4	93.0 (6)	C13—C14—C19—C18	179.4 (6)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···N3^{i}	0.86	2.38	3.220 (7)	167

Symmetry code: (i) $x+\frac{1}{2}, y+\frac{1}{2}, z$.

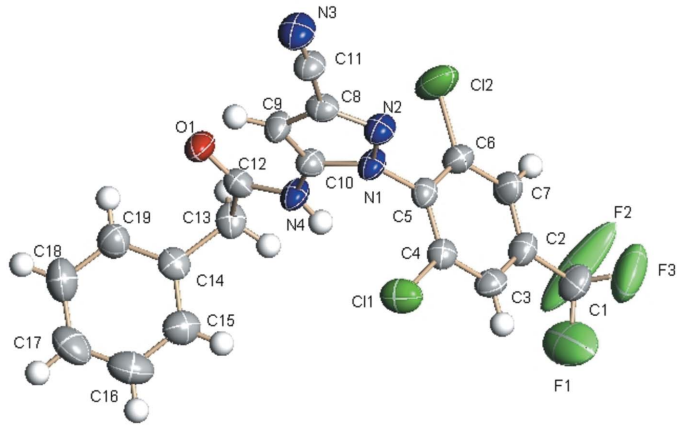


Figure 1
View of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

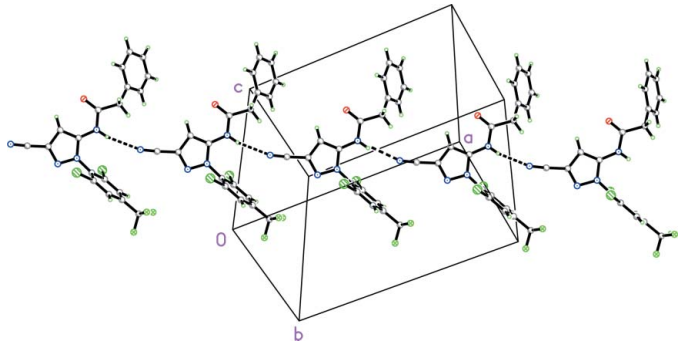


Figure 2
The crystal packing of (I), showing the hydrogen-bonded (dashed lines) linear chains.

All H atoms were positioned geometrically and allowed to ride on their parent atoms; Csp^2—H = 0.93 Å with U_{iso}(H) = 1.2U_{eq}(C), Csp^3—H = 0.97 Å with U_{iso}(H) = 1.5U_{eq}(C), and N—H = 0.86 Å with U_{iso}(H) = 1.5U_{eq}(N). The large values for the atomic displacement parameters for the F atoms and the strong anisotropy of their displacement ellipsoids indicate either large thermal motion or unresolved rotational disorder of the trifluoromethyl group, which is the most probable reason for the rather limited overall precision of the structure.

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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