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1-[2,6-Dichloro-4-(trifluoromethyl)phenyl]-5-(2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl)-1*H*-pyrazole-3-carbonitrile

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Key indicators

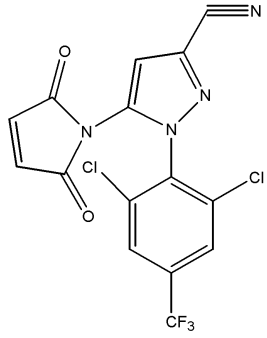
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.055
wR factor = 0.165
Data-to-parameter ratio = 13.7

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

The title compound, $C_{15}H_5Cl_2F_3N_4O_2$, is a tricyclic imide with an overall U-shape, each of the three rings being planar. These include a phenyl ring with two chloro and one trifluoromethyl substituents, a central pyrazole ring with a cyano substituent, and a dioxopyrrolidine ring.

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. When the latter was reacted with maleic anhydride, the title compound, (I), was obtained.



(I)

Compound (I) has been used to synthesize 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-(trifluoromethylsulfonyl)pyrazole, all of which are good insecticides (Hatton *et al.*, 1993).

The molecular structure of compound (I) is illustrated in Fig. 1, and selected bond lengths and angles are given in Table 1. The molecule is composed of three planar moieties, *viz*. a benzene ring, a central pyrazole ring and a dihydropyrrole ring. The angle between the benzene and pyrazole planes is 75.49 (14)°, and that between the dihydropyrrole and pyrazole planes is 49.62 (16)°.

In the crystal structure, the molecules stack along the *b* axis, as shown in Fig. 2, and are connected by weak C—H···O and C—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 analysis were

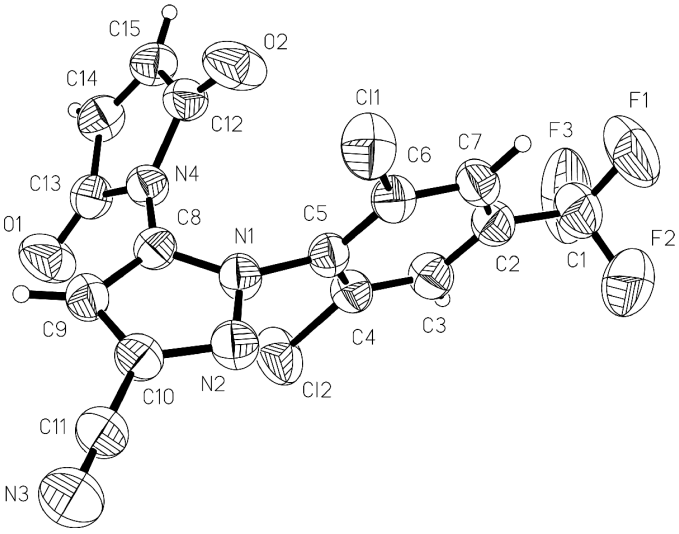


Figure 1
The molecular structure of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

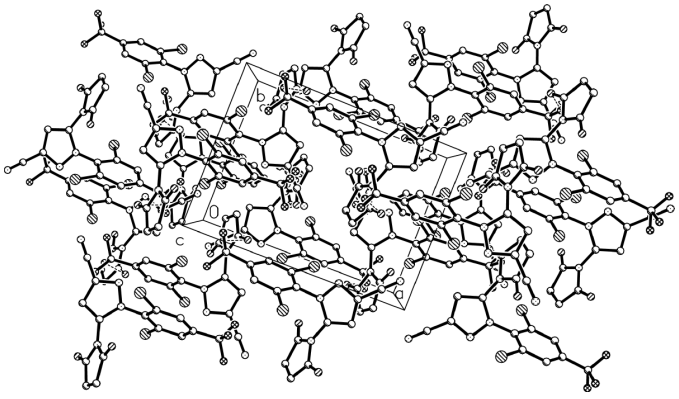


Figure 2
The crystal packing of (I), viewed down the *c* axis.

obtained on slow evaporation of an ethyl acetate/cyclohexane (1:1) solution (m.p. 462–464 K). Spectroscopic analysis, IR (KBr, ν cm^{-1}): 3089, 2253, 1742, 1562, 1497; ^{1}H NMR ($CDCl_3$, p.p.m.): 7.71 (*s*, 2H), 6.88 (*s*, 1H), 6.85 (*s*, 2H).

Crystal data

$C_{15}H_5Cl_2F_3N_4O_2$	D_x = 1.622 Mg m^{-3}
M_r = 401.13	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 3254 reflections
a = 12.8261 (10) Å	θ = 2.4–26.0°
b = 8.9942 (7) Å	μ = 0.45 mm^{-1}
c = 14.6896 (12) Å	T = 298 (2) K
β = 104.217 (1)°	Block, colorless
V = 1642.7 (2) Å^3	0.43 × 0.38 × 0.20 mm
Z = 4	

Data collection

Bruker SMART APEX area-detector diffractometer	3225 independent reflections
φ and ω scans	2660 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	R_{int} = 0.018
T_{min} = 0.832, T_{max} = 0.916	θ_{max} = 26.0°
9003 measured reflections	$h = -15 \rightarrow 14$
	$k = -11 \rightarrow 9$
	$l = -18 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0945P)^2 + 0.8386P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.165	$\Delta\rho_{max}$ = 0.72 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
3225 reflections	
235 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.352 (3)	N2—C10	1.327 (3)
N1—C8	1.359 (3)	C8—C9	1.361 (4)
N1—C5	1.423 (3)	C9—C10	1.395 (4)
N3—C11	1.136 (4)	C10—C11	1.443 (4)
N4—C8	1.401 (3)	C12—C15	1.477 (4)
N4—C13	1.407 (3)	C14—C15	1.319 (4)
N4—C12	1.410 (3)		
N2—N1—C8	111.9 (2)	C8—C9—C10	103.8 (2)
N2—N1—C5	117.93 (19)	N2—C10—C9	113.3 (2)
C8—N1—C5	129.9 (2)	N2—C10—C11	117.8 (2)
C8—N4—C13	124.2 (2)	C9—C10—C11	128.9 (3)
C8—N4—C12	125.5 (2)	N3—C11—C10	178.7 (3)
C13—N4—C12	109.8 (2)	N4—C12—C15	105.7 (2)
C10—N2—N1	103.5 (2)	O1—C13—C14	129.0 (3)
N1—C8—C9	107.6 (2)	N4—C13—C14	105.5 (2)
N1—C8—N4	121.8 (2)	C15—C14—C13	109.5 (3)
C9—C8—N4	130.7 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^{i}	0.93	2.50	3.380 (4)	157
C14—H14···N2^{ii}	0.93	2.49	3.410 (3)	170

Symmetry codes: (i) $x-\frac{1}{2}, \frac{3}{2}-y, z-\frac{1}{2}$; (ii) $x, 1+y, z$.

All H atoms were located in difference Fourier maps but were placed in geometrically idealized position and constrained to ride on their parent atom, with C—H distances of 0.93 Å and U_{iso}(H) = 1.2$_{eq}$(C).

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: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL*97.

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