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1-[2,6-Dichloro-4-(trifluoromethyl)phenyl]-5-[(4-dimethylaminobenzylidene)amino]-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 = 0.066
wR factor = 0.186
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.

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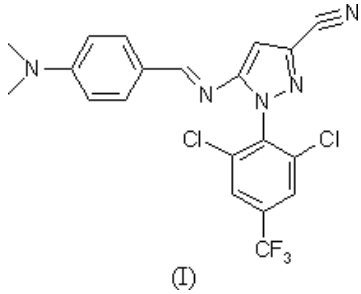
The title compound, $C_{20}H_{14}Cl_2F_3N_5$, is a tricyclic amide with an overall U-shape, each of the the three rings being planar. There are π–π interactions between the pyrazole ring and the benzene ring with the dimethylamine substituent.

Comment

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



(I)

The structure of (I) 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 4-(dimethylamino)-phenyl ring. The dihedral angles between the pyrazole and the C3–C8 and C14–C19 benzene rings are 5.0 (3) and 75.78 (12)°, respectively. There are π–π interactions between the pyrazole ring and the C3–C8 benzene ring.

Experimental

Following the method of Hatton *et al.* (1993), reaction of 2,6-dichloro-4-trifluoromethylamine 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 reacted with 4-(dimethylamino)benzaldehyde and hydrochloric acid in anhydrous ethanol to give the title compound, (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate/petroleum ether (1:2) solution (m.p. 456–458 K). IR (KBr, ν cm^{-1}): 3130, 3075, 2358, 2234, 1587, 1529; ^{1}H NMR ($CDCl_3$): δ 8.77 (*s*,1H), 8.09 (*s*, 2H), 7.62 (*d*, 2H), 7.07 (*s*, 1H), 7.63 (*d*, 2H), 3.07 (*s*, 6H); ^{13}C NMR ($CDCl_3$): δ 165.5 (1C), 155.2 (1C), 154.9 (1C), 138.5 (1C), 136.7 (1C), 133.9 (1C), 132.4 (2C), 128.1 (1C), 126.9 (2C), 126.8 (2C), 123.5 (1C), 114.6 (1C), 112.3 (2C), 97.2 (1C), 40.0 (2C).

Crystal data

$C_{20}H_{14}Cl_2F_3N_5$
$M_r = 452.26$
Triclinic, $P\overline{1}$
$a = 6.5692\ (14)$ Å
$b = 12.365\ (3)$ Å
$c = 13.878\ (3)$ Å
$\alpha = 67.312\ (4)^\circ$
$\beta = 80.447\ (4)^\circ$
$\gamma = 86.740\ (4)^\circ$
$V = 1025.6\ (4)$ Å^3
$Z = 2$
$D_x = 1.464$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2322 reflections
$\theta = 2.8$–24.7°
$\mu = 0.36$ mm^{-1}
$T = 298\ (2)$ K
Block, colorless
0.42 × 0.29 × 0.23 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.863$, $T_{max} = 0.922$
5431 measured reflections
3638 independent reflections
2936 reflections with $I > 2\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 25.3^\circ$
$h = -7 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.186$
$S = 1.05$
3638 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0899P)^2 + 0.8389P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C15	1.713 (4)	N3—C14	1.433 (4)
F1—C20	1.285 (7)	N4—C12	1.332 (4)
N1—C3	1.363 (4)	N5—C13	1.140 (4)
N1—C1	1.447 (4)	C10—C11	1.368 (4)
N2—C9	1.281 (4)	C11—C12	1.394 (4)
N2—C10	1.386 (4)	C12—C13	1.436 (4)
N3—N4	1.357 (3)	C17—C20	1.500 (5)
N3—C10	1.371 (4)		
C9—N2—C10	115.7 (2)	N3—C10—N2	119.4 (2)
N4—N3—C10	113.3 (2)	C10—C11—C12	104.9 (3)
N4—N3—C14	118.7 (2)	N4—C12—C11	113.5 (3)
C10—N3—C14	127.8 (2)	N4—C12—C13	121.1 (3)
C12—N4—N3	102.6 (2)	C11—C12—C13	125.4 (3)
N2—C9—C6	124.8 (3)	N5—C13—C12	174.9 (4)
C11—C10—N3	105.7 (3)	F2—C20—F1	108.5 (7)
C11—C10—N2	134.9 (3)		

All H atoms were initially observed 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 0.93–0.96 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. The high displacement parameters of atoms F1, F2 and F3 indicated the presence of moderate torsional disorder of the trifluoromethyl group, but an attempt to model the group using a disorder model was unsuccessful.

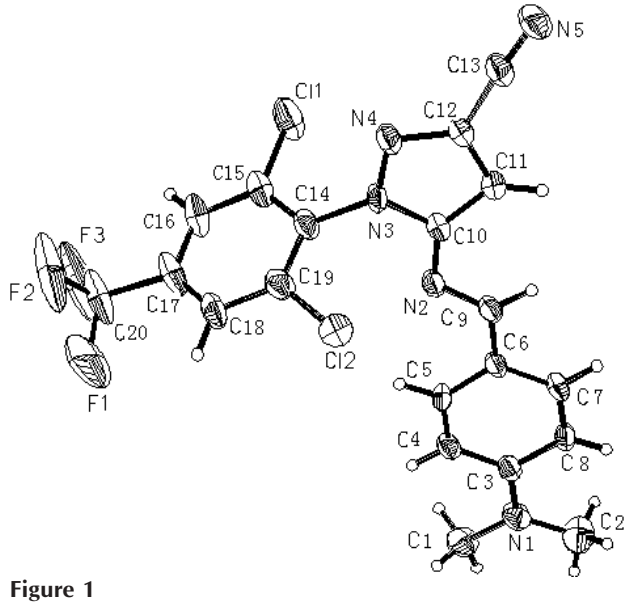


Figure 1
The structure of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

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: *SHELXTL*.

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