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1-[2,6-Dichloro-4-(trifluoromethyl)phenyl]-5-[(4-methylbenzylidene)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
Disorder in main residue
R factor = 0.072
wR factor = 0.215
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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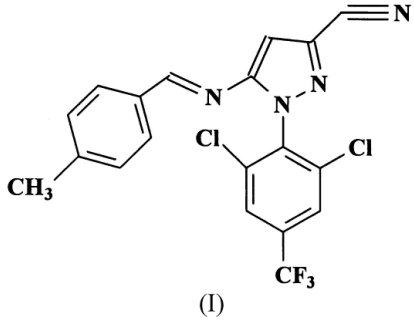
The title compound, $C_{19}H_{11}Cl_2F_3N_4$, is a tricyclic amide with an overall U-shape, each of the three rings being planar. These include a benzene ring with two chloro and one trifluoromethyl substituent, a central pyrazole ring with a cyano substituent, and a benzene ring with one methyl substituent.

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-(trifluoromethyl)thiopyrazole, 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethyl)sulfenylpyrazole and 5-amino-3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(trifluoromethyl)sulfonylpyrazole, all of which are 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-methylphenyl group. The dihedral angles between the pyrazole and the C1–C6 and C12–C17 benzene rings are 3.6 (3) and 78.7 (1)°, respectively.

Experimental

Following the method of Hatton *et al.* (1993), reaction of 2,6-dichloro-4-trifluoromethyl)phenylamine 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 4-methylbenzaldehyde and hydrochloric acid in anhydrous ethanol to give the title compound. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate/petroleum ether (1:2) solution (m.p. 460–462 K). IR (KBr, ν cm^{-1}): 2242, 1603, 1568, 1523, 1507, 1356, 1307, 881, 826; ^{1}H NMR ($CDCl_3$): δ 9.02 (*s*,1H), 8.12 (*s*, 2H), 7.71 (*d*, *J* = 8.0 Hz, 2H), 7.30 (*d*, *J* = 8.0 Hz, 2H), 7.25 (*s*, 1H), 2.39 (*s*, 3H); ^{13}C NMR ($CDCl_3$): δ 166.6 (1C), 154.0 (1C),

145.0 (1C), 138.1 (1C), 134.1 (1C), 133.4 (1C), 130.5 (2C), 130.4 (2C), 128.8 (1C), 127.2 (2C), 127.1 (2C), 119.7 (1C), 114.3 (1C), 98.5 (1C), 21.7 (1C).

Crystal data

$C_{19}H_{11}Cl_2F_3N_4$
M_r = 423.22
Triclinic, $P\overline{1}$
a = 8.3045 (7) Å
b = 10.5134 (10) Å
c = 11.8019 (11) Å
α = 76.106 (1)°
β = 87.116 (2)°
γ = 72.072 (2)°
V = 951.38 (15) Å^3
Z = 2
D_x = 1.477 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2515 reflections
θ = 1.8–25.2°
μ = 0.38 mm^{-1}
T = 298 (2) K
Block, colourless
0.18 × 0.15 × 0.13 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.932, T_{max} = 0.952
5082 measured reflections
3428 independent reflections
2829 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 25.2°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.215
S = 1.06
3359 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1186P)^2 + 0.8905P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C8	1.266 (4)	N4—C12	1.140 (5)
N1—C9	1.390 (4)	C5—C8	1.455 (5)
N2—C11	1.335 (5)	C9—C10	1.364 (5)
N2—N3	1.344 (4)	C10—C11	1.391 (5)
N3—C9	1.367 (4)	C11—C12	1.437 (5)
N3—C13	1.421 (4)		
C8—N1—C9	118.5 (3)	C9—C10—C11	104.8 (3)
C11—N2—N3	102.9 (3)	N2—C11—C10	113.1 (3)
N2—N3—C9	113.2 (3)	N2—C11—C12	120.6 (3)
N2—N3—C13	121.3 (3)	C10—C11—C12	126.4 (3)
C9—N3—C13	125.4 (3)	N4—C12—C11	176.3 (5)
N1—C8—C5	122.7 (3)	C14—C13—N3	121.4 (3)
C10—C9—N3	106.0 (3)	C18—C13—N3	119.9 (3)
C10—C9—N1	136.3 (3)	F2—C19—F1	106.1 (6)
N3—C9—N1	117.7 (3)	F2—C19—C16	115.8 (4)

All H atoms were initially located in a difference Fourier map, and were then 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) = 1.22U_{eq}(C). The low U_{eq} value of C19 compared with its neighbours may be attributed to the three disordered F atoms.

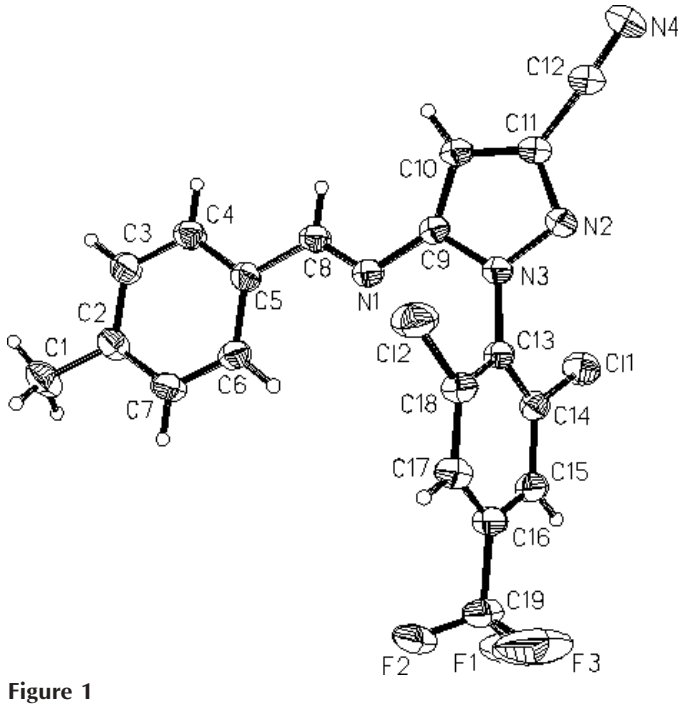


Figure 1
The structure (I), showing the atom-numbering scheme and with displacement ellipsoids at the 50% probability level. Only one disorder component is shown.

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: *SHELXTL* (Bruker, 2002).

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