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5-{[(4-Chlorophenyl)methylene]amino}-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) = 0.004 Å
R factor = 0.050
wR factor = 0.135
Data-to-parameter ratio = 16.9

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

The title compound, $C_{18}H_8Cl_3F_3N_4$, is a tricyclic imide with an overall U-shape, each of the three rings being planar. The packing is stabilized by π–π interactions.

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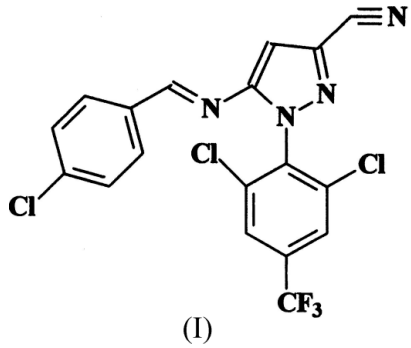
Comment

The molecular structure of the title compound, (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-chlorophenyl ring. Bond lengths and angles (Table 1) are in agreement with those observed in similar compounds (Zhong, Yang & Shi, 2005; Zhong, Yang, Shi *et al.*, 2005; Chen *et al.*, 2005). The dihedral angles between the pyrazole and the C1–C6 and C12–C17 benzene rings are 19.02 (16) and 86.98 (8)°, respectively. There are π–π interactions between the pyrazole ring and the C1–C6 benzene ring. In the crystal structure, the molecules stack along the *a* axis, as shown in Fig. 2.



(I)

Experimental

Following the method of Hatton *et al.* (1993), 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 reacted with 4-chlorobenzaldehyde 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. 452–454 K). IR (KBr, ν cm^{-1}): 3081, 2238, 2234, 1610, 1507, 1310, 880, 825; ^{1}H NMR ($CDCl_3$, δ): 9.08 (*s*, 1H), 8.11 (*s*, 2H), 7.84 (*d*, 2H), 7.50 (*d*, 2H), 7.29 (*s*, 1H); ^{13}C NMR ($CDCl_3$, δ): 166.0 (1C), 153.9 (1C), 140.0 (1C), 137.0 (1C), 135.1 (1C), 134.7 (1C), 132.2 (2C), 130.6 (2C), 128.8 (1C), 127.5 (2C), 127.4 (2C), 123.8 (1C), 114.6 (1C), 99.5 (1C).

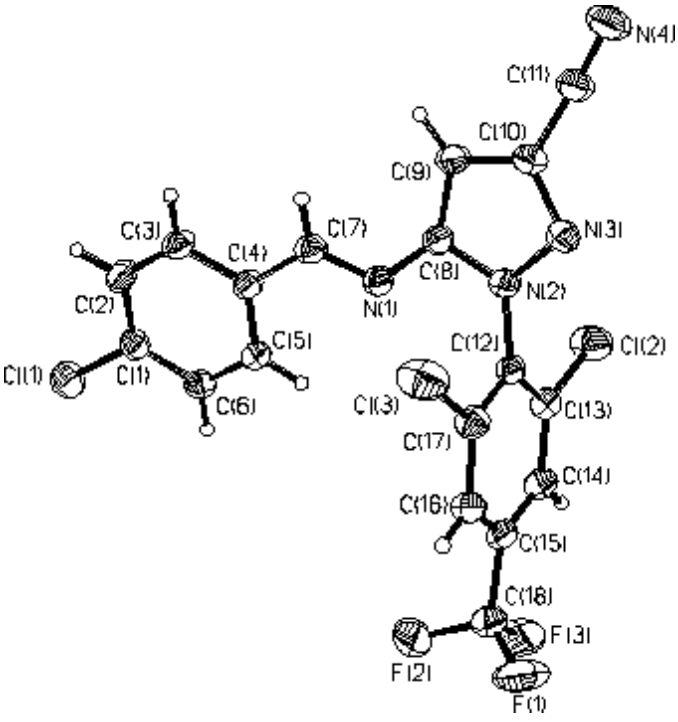


Figure 1
The structure of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

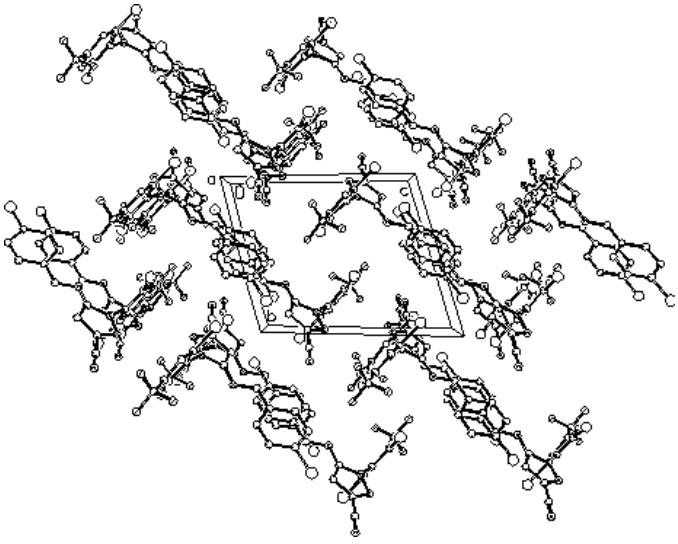


Figure 2
Packing diagram for (I), viewed down the *c* axis. H atoms have been omitted for clarity.

Crystal data

$C_{18}H_8Cl_3F_3N_4$
M_r = 443.64
Triclinic, $P\overline{1}$
a = 9.7553 (9) Å
b = 9.7554 (9) Å
c = 11.7319 (11) Å
α = 68.582 (1)°
β = 68.580 (1)°
γ = 69.340 (1)°
V = 936.06 (15) Å^3
Z = 2
D_x = 1.574 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3541 reflections
θ = 1.9–28.2°
μ = 0.53 mm^{-1}
T = 298 (2) K
Block, colourless
0.22 × 0.18 × 0.15 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.892, T_{max} = 0.924
5873 measured reflections
4627 independent reflections
3320 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 28.2°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.135
S = 1.03
4267 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 0.354P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.740 (2)	N3—C10	1.332 (3)
F1—C18	1.311 (3)	N4—C11	1.133 (3)
N1—C7	1.268 (3)	C4—C7	1.453 (3)
N1—C8	1.387 (3)	C8—C9	1.368 (3)
N2—N3	1.345 (2)	C9—C10	1.392 (3)
N2—C8	1.366 (3)	C10—C11	1.437 (3)
N2—C12	1.424 (2)	C15—C18	1.507 (3)
C7—N1—C8	118.03 (18)	C8—C9—C10	104.57 (19)
N3—N2—C8	113.56 (16)	N3—C10—C9	113.45 (19)
N3—N2—C12	122.26 (17)	N3—C10—C11	120.3 (2)
C8—N2—C12	123.83 (17)	C9—C10—C11	126.2 (2)
C10—N3—N2	102.62 (17)	N4—C11—C10	176.5 (3)
N1—C7—C4	123.2 (2)	C17—C12—N2	119.97 (18)
N2—C8—C9	105.78 (18)	F1—C18—F2	107.3 (3)
N2—C8—N1	118.46 (18)	F1—C18—C15	112.6 (2)
C9—C8—N1	135.8 (2)		

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances set at 0.93 Å and U_{iso}(H) values set at 1.2U_{eq}(C).

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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