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

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.090
wR factor = 0.211
Data-to-parameter ratio = 12.9

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

The title compound, $C_{19}H_{11}Cl_2F_3N_4O$, is a tricyclic imide with an overall U-shape, each of the the three rings being planar. In the crystal structure, the molecular packing is stabilized by π–π interactions occurring between adjacent methoxyphenyl rings.

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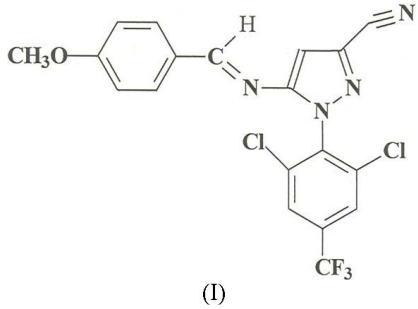
Comment

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.



(I)

Bond lengths and angles (Table 1) are in agreement with those observed in similar compounds (Zhong, Yang, Shi *et al.*, 2005; Zhong, Yang & Shi, 2005; Chen *et al.*, 2005). The dihedral angles between the pyrazole and the C13–C18 and C2–C7 benzene rings are 24.9 (2) and 73.5 (2)°, respectively. In the crystal structure, an overlapped arrangement of the molecules is observed along the *b* axis (Fig. 2). The C13–C18 benzene rings of centrosymmetrically-related molecules at (x, y, z) and $(2 - x, 1 - y, 1 - z)$ are separated by about 3.48 Å, indicating the presence of π-stacking interactions.

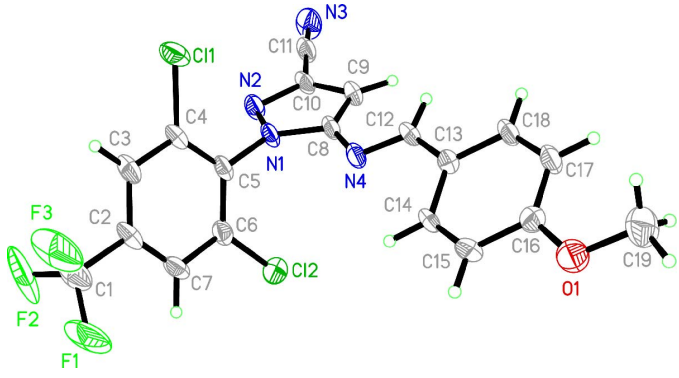


Figure 1
The structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

Experimental

Following the method of Hatton *et al.* (1993), the 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-trifluoromethylphenyl)pyrazole. The compound was then reacted with 4-methoxybenzaldehyde to give the title compound, (I). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution (m.p. 408–410 K). IR (KBr, ν cm^{-1}): 3079, 2928, 2847, 2360, 2237, 1604, 1567, 1518, 1425, 1361, 1259, 1168, 887, 824; ^{1}H NMR ($CDCl_3$): δ 8.61 (*s*, 1H), 7.76 (*s*, 2H), 7.70 (*d, J* = 8.7 Hz, 2H), 6.93 (*d, J* = 8.7 Hz, 2H), 6.73 (*s*, 1H), 3.86 (*s*, 3H); ^{13}C NMR ($CDCl_3$): δ 164.6 (1C), 162.4 (1C), 152.8 (1C), 135.9 (1C), 133.5 (1C), 130.8 (2C), 127.4 (2C), 126.6 (2C), 124.4 (1C), 122.0 (1C), 115.5 (1C), 113.4(1C), 113.1 (2C), 95.6 (1C), 56.2 (1C).

Crystal data

$C_{19}H_{11}Cl_2F_3N_4O$
M_r = 439.22
Triclinic, $P\overline{1}$
a = 6.7145 (12) Å
b = 10.931 (2) Å
c = 13.616 (3) Å
α = 77.464 (3)°
β = 83.116 (3)°
γ = 81.470 (3)°
V = 960.8 (3) Å^3
Z = 2
D_x = 1.518 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1222 reflections
θ = 3.1–24.9°
μ = 0.38 mm^{-1}
T = 298 (2) K
Block, colourless
0.34 × 0.28 × 0.17 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.880, T_{max} = 0.938
4901 measured reflections
3401 independent reflections
2393 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.3°
$h = -6 \rightarrow 8$
$k = -13 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.090
$wR(F^2)$ = 0.211
S = 1.15
3401 reflections
263 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.081P)^2 + 0.8129P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.340 (5)	N4—C8	1.370 (6)
N1—C8	1.371 (5)	N4—C12	1.278 (5)
N2—C10	1.333 (6)		
N2—N1—C8	114.1 (4)	C8—N4—C12	116.6 (4)
N1—N2—C10	103.0 (4)		

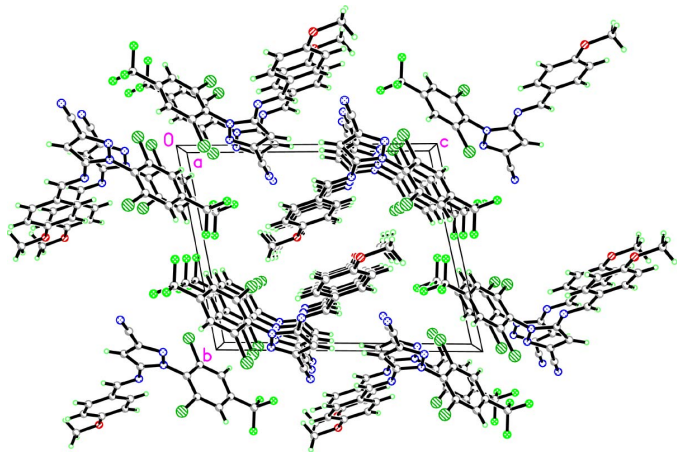


Figure 2
The crystal packing of (I), viewed along the *a* axis.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å, and U_{iso}(H) = 1.2U_{eq}(C) for aryl and CH H atoms, and 1.5U_{eq}(C) for CH_3 H atoms. The CF_3 group may be subject to unresolved disorder, which could account for the weak diffracting ability of the crystal, leading to a rather high R value.

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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