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N-{3-Cyano-1-[2,6-dichloro-4-(trifluoromethyl)-phenyl]-1*H*-pyrazol-5-yl}-4-nitrobenzamide ethanol solvate

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Key indicators

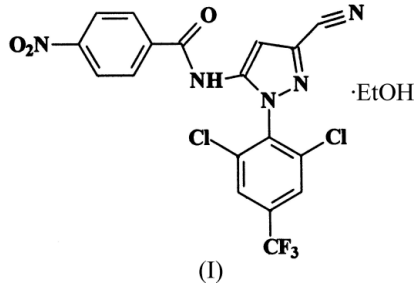
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.055
wR factor = 0.167
Data-to-parameter ratio = 12.4

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

The title compound, $C_{18}H_8Cl_2F_3N_5O_3\cdot C_2H_6O$, is an ethanol solvate of a tricyclic imide with an overall U-shape, each of the 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 nitro substituent. In the crystal packing, weak intermolecular hydrogen-bond contacts result in the formation of zigzag chains running parallel to the *b* axis

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-(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 and atom-numbering scheme of (I) is shown in Fig. 1. The molecule contains three planar groups, forming an overall U-shape. The dihedral angles formed by the C1–C6 and C12–C17 benzene rings with the pyrazole ring are 40.0 (1) and 89.6 (1)°, respectively. Bond distances and angles fall in the normal ranges (Table 1). In the crystal packing, the molecules are connected by weak intermolecular C—H···O, N—H···O and O—H···Cl hydrogen-bond interactions, resulting in the formation of zigzag chains running parallel to the *b* axis (Fig. 2 and Table 2).

Experimental

Following the method of Hatton *et al.* (1993), the reaction of 2,6-dichloro-4-(trifluoromethyl)aniline 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-nitrobenzoyl chloride to give the title compound. Single

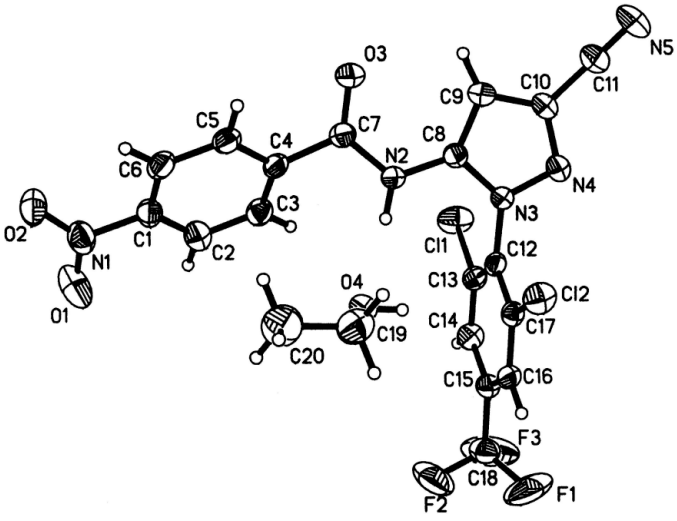


Figure 1
Perspective view of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

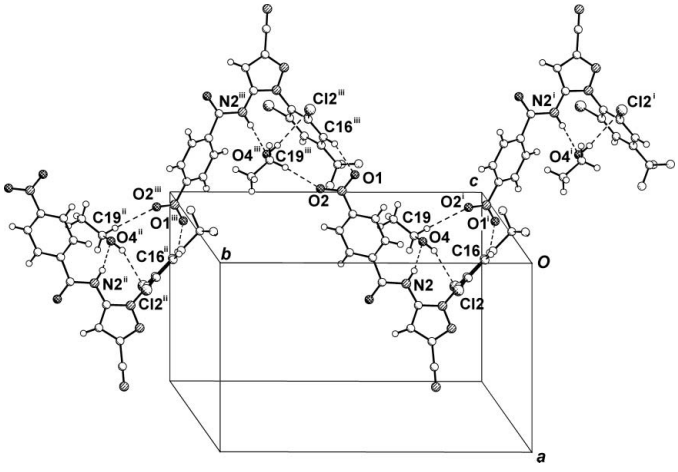


Figure 2
Part of the crystal structure of (I), showing the formation of zigzag chains parallel to the *b* axis, with hydrogen bonds denoted by dashed lines. [Symmetry codes: (i) $-\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, 1+y, z$; (iii) $-\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.]

crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol–ethanol (1:1) solution (m.p. 483–485 K). IR (KBr, ν cm^{-1}): 3478, 2358, 2251, 1699, 1554, 1351, 1310, 865, 811; ^{1}H NMR ($CDCl_3$): δ 10.52 (*s*,1H), 8.29 (*d, J* = 8.8 Hz, 2H), 8.12 (*s*, 2H), 8.00 (*d, J* = 8.8 Hz, 2H), 7.39 (*s*, 1H); ^{13}C NMR ($CDCl_3$): δ 164.5 (1C), 151.0 (1C), 140.8 (1C), 139.6 (1C), 136.9 (1C), 134.7 (1C), 131.3 (1C), 129.2 (2C), 128.3 (2C), 126.4 (2C), 123.4 (2C), 121.5 (1C), 114.1 (1C), 102.8 (1C).

Crystal data

$C_{18}H_8Cl_2F_3N_5O_3\cdot C_2H_6O$
M_r = 516.26
Monoclinic, $P2_1/n$
a = 10.630 (19) Å
b = 18.00 (3) Å
c = 12.54 (2) Å
β = 108.75 (3)°
V = 2272 (7) Å^3
Z = 4
D_x = 1.509 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5801 reflections
θ = 2.1–25.2°
μ = 0.35 mm^{-1}
T = 298 (2) K
Block, colourless
0.25 × 0.22 × 0.18 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.916, T_{max} = 0.937
10256 measured reflections
4099 independent reflections
2643 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.2°
$h = -11 \rightarrow 12$
$k = -21 \rightarrow 21$
$l = -15 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.167
S = 1.07
4099 reflections
307 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0929P)^2 + 0.0142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.215 (4)	N3—N4	1.365 (3)
O2—N1	1.213 (4)	N3—C8	1.369 (4)
N2—C7	1.382 (4)	N4—C10	1.340 (4)
N2—C8	1.387 (4)		
O1—N1—O2	123.3 (3)	N4—N3—C8	112.73 (19)
C7—N2—C8	122.4 (2)	N3—N4—C10	102.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O4	0.86	2.02	2.812 (5)	153
O4—H4A···Cl2	0.82	2.86	3.556 (5)	144
C16—H16A···O1^{i}	0.93	2.58	3.461 (6)	159
C19—H19B···O2^{i}	0.97	2.63	3.458 (7)	144

Symmetry code: (i) $-\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å, N—H = 0.86 Å and O—H = 0.82 Å. U_{iso}(H) values were set at $1.2U_{eq}$(C,N) for aryl, CH, CH_2 and NH H atoms, and at $1.5U_{eq}$(C,O) for CH_3 and OH H atoms. The high displacement parameters of atoms F1, F2, F3, O1, and O2 indicate the presence of moderate torsional disorder of the trifluoromethyl and nitro groups, but an attempt to model the groups using a static disorder model was unsuccessful.

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: *SHELXL*97.

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