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

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In the title compound, $C_{22}H_{16}Cl_3F_3N_4O\cdot C_2H_6O$, the asymmetric unit consists of one molecule of $C_{22}H_{16}Cl_3F_3N_4O$ and one ethanol solvent molecule which are connected through an N—H···O hydrogen bond. The H atom of the hydroxyl group of the solvent molecule is hydrogen bonded to the N atom of the cyano group. These hydrogen-bond interactions result in the formation of zigzag chains parallel to the *a* axis.

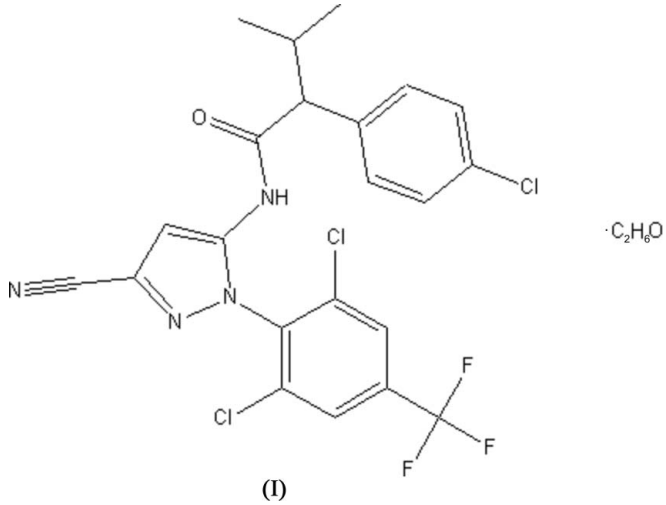
Key indicators

Single-crystal X-ray study
T = 243 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.188
Data-to-parameter ratio = 16.7

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

Comment

Various 1-(substituted phenyl or pyridyl)-5-[substituted alkyl(thio)amino]pyrazole compounds are known to exhibit a number of different types of pesticidal activity, including use as herbicides, plant-growth regulators, insecticides and nematicides (Chapel Hill & Apex, 1996). As part of our continuing interest in the structure–activity relationships of pyrazole derivatives, we have isolated the product, (I), of the condensation reaction of 2-(4-chlorophenyl)-3-methylbutanoyl chloride and 1-(2,6-dichloro-4-trifluoromethylphenyl)-3-cyano-5-aminopyrazole as colorless crystals suitable for X-ray analysis.



(I)

The asymmetric unit of the title compound consists of one molecule of $C_{22}H_{16}Cl_3F_3N_4O$ and one ethanol solvent molecule which are connected through an N—H···O hydrogen bond (Fig. 1, Table 1). The occurrence of hydrogen-bonding interactions between the H atom of the hydroxyl group and the N atom of the cyano group result in the formation of a zigzag chain parallel to the *a* axis (Fig. 2 and Table 1).

The molecule is chiral but, as the space group is centrosymmetric, the two enantiomers are present in the cell and the compound is a racemate.

Experimental

1-(2,6-Dichloro-4-trifluoromethylphenyl)-3-cyano-5-aminopyrazole (2 g, 4.7 mmol), prepared according to the procedure of Hawkins (1988), 2-(4-chlorophenyl)-3-methylbutanoyl chloride (1.15 equivalents), 4-dimethylaminopyridine (0.58 g, 1 equivalent), triethylamine (0.86 ml, 1.3 equivalents) and chloroform (150 ml) were heated at reflux for 48 h. The organic solution was washed with 1% HCl/H_2O once, dried over $MgSO_4$, filtered and concentrated. The residue was purified by silica gel column chromatography, eluted with hexane/ethyl acetate (2:1). The desired product was obtained as a white solid (yield 82%, 2.3 g), which was then recrystallized from ethanol to give colorless blocks (m.p. 437–437 K).

Crystal data

$C_{22}H_{16}Cl_3F_3N_4O\cdot C_2H_6O$
M_r = 561.82
Triclinic, $P\overline{1}$
a = 10.139 (4) Å
b = 10.652 (5) Å
c = 12.827 (7) Å
α = 73.255 (18)°
β = 83.517 (19)°
γ = 89.185 (15)°
V = 1317.9 (11) Å^3
Z = 2
D_x = 1.416 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10553 reflections
θ = 3.1–27.6°
μ = 0.40 mm^{-1}
T = 243 (1) K
Chunk, colorless
0.33 × 0.28 × 0.27 mm

Data collection

Rigaku RAXIS-RAPID diffractometer
ω scans
Absorption correction: multi-scan *ABSCOR* (Higashi, 1995)
T_{min} = 0.874, T_{max} = 0.898
12934 measured reflections
5889 independent reflections
4011 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.026
θ_{max} = 27.5°
$h = -12 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.188
S = 1.01
5889 reflections
353 parameters
H-atom parameters constrained
$w = 1/[0.0038F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: Larson (1970) equation 22
Extinction coefficient: 0.5 (3) × 10^2

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H201···N2^{i}	0.85	2.05	2.895 (3)	172
N1—H101···O2	0.87	1.98	2.839 (2)	167

Symmetry code: (i) $x+1, y, z$.

H atoms were included in calculated positions and treated as riding on their parent atoms with C—H= 0.96 Å (methyl), C—H = 0.97 Å (methylene), O—H = 0.85 Å and N—H = 0.87 Å and U_{iso}(H) = 1.2U_{eq}(CH_2, OH, NH) or 1.5U_{eq}(CH_3). The CF_3 group is disordered with the F atoms distributed over two different sites with occupancy factors in the ratio 0.6:0.4 (the ratio was initially set at 0.5:0.5 but the ratio of 0.6:0.4 was confirmed by the refinement). This disorder was refined using the restraints available within *CRYSTALS* (Betteridge *et al.*, 2003).

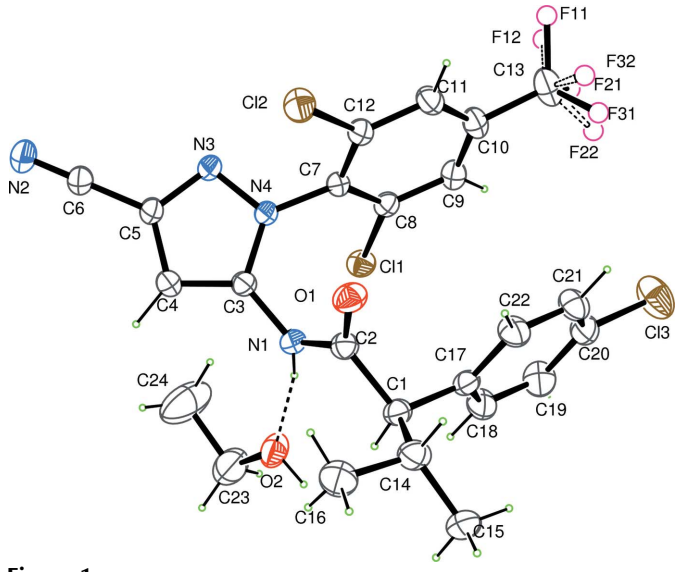


Figure 1
The structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii. The N—H···O hydrogen bond is shown as a dashed line. Both disorder components are shown for the CF_3 group.

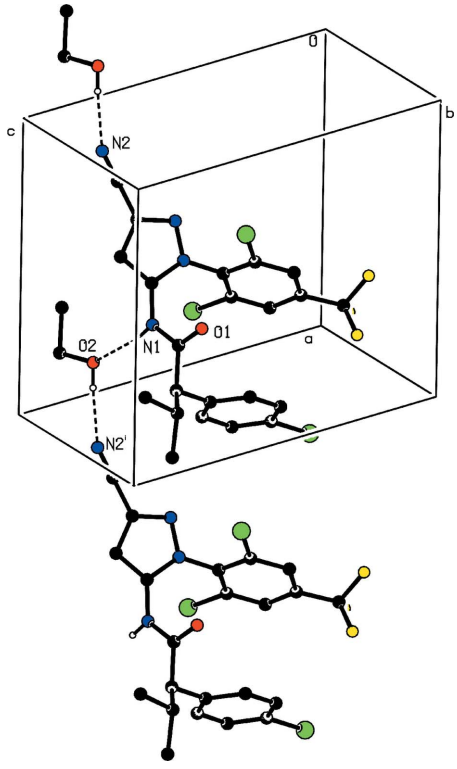


Figure 2
View showing the N—H···O hydrogen bonding and the formation of the zigzag chain. For clarity, only H atoms involved in hydrogen bonding (dashed lines) are shown, and the minor component of the disorder has been omitted. [Symmetry code: (i) $1+x, y, z$].

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1993); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia,1997); *PLATON* (Spek, 2003);

software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC & Rigaku, 2004).

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