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N,*N*′-Bis{3-cyano-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-1*H*-pyrazol-5-yl}-1,4-benzenediamide dimethylformamide disolvate

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Key indicators

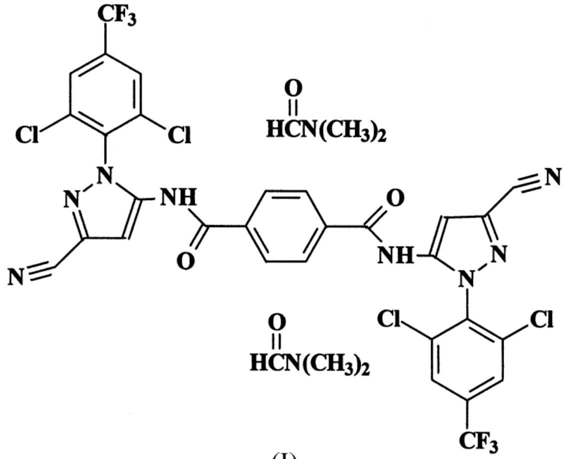
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.098
wR factor = 0.248
Data-to-parameter ratio = 13.8

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

The title compound, $C_{30}H_{12}Cl_4F_6N_8O_2\cdot 2C_3H_7NO$ or $C_6H_4[CONH\text{-}C_3N_2H(CN)\text{-}C_6H_2Cl_2CF_3]_2\cdot 2HCONMe_2$, is a fivecyclic amide with an overall *Z* shape, each of the five rings being planar.

Comment

The title compound, (I), is an important starting material for the synthesis of a number of insecticides (Hatton *et al.*, 1993).



(I)

The molecule occupies a special position on an inversion centre and each of the two peripheral groups is made up of three approximately planar amide (CONH), 3-cyanopyrazole and 2,6-dichloro-4-(trifluoromethyl)phenyl fragments. The amide HNCO plane forms dihedral angles of 39.1 (5) and 11.5 (7)° with the central benzene and pyrazole planes, respectively. The pyrazole forms a dihedral angle of 81.9 (2)° with the dichloro(trifluoromethyl)phenyl plane. The only 'active' amide H atom in each half of the molecule forms a hydrogen bond with the carbonyl O atom of a dimethylformamide solvent molecule (Table 1).

Experimental

According to the method of Hatton *et al.* (1993), the reaction of 2,6-dichloro-4-trifluoromethylamine with a suspension of nitrosylsulfuric acid, followed by reaction with a solution of ethyl 2,3-dicyanopropionate in acetic acid, was used to obtain 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole. To a solution of that compound (10 mmol) in pyridine (8 ml), terephthaloyl dichloride (5 mmol) was added at room temperature. The mixture was stirred continuously for 30 min (reaction monitored by thin-layer chromatography). The reaction mixture was then poured into water (30 ml) to give the title compound, (I), in 86% yield. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a dimethylformamide solution (m.p. 564–565 K). IR (KBr, ν, cm^{-1}): 3145, 3082, 2347, 2251, 1697, 1538, 1492, 875, 817; ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 9.53 (*s*, 2H), 8.08 (*s*, 4H), 7.87 (*s*, 2H), 7.71 (*s*, 4H).

Crystal data

$C_{30}H_{12}Cl_4F_6N_8O_2\cdot 2C_3H_7NO$
$M_r = 918.47$
Monoclinic, $P2_1/c$
$a = 7.7657\ (19)$ Å
$b = 27.655\ (6)$ Å
$c = 9.902\ (2)$ Å
$\beta = 102.526\ (4)^\circ$
$V = 2075.8\ (8)$ Å^3
$Z = 2$
$D_x = 1.469$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1787 reflections
$\theta = 2.2$–24.2°
$\mu = 0.36$ mm^{-1}
$T = 298\ (2)$ K
Block, colourless
$0.35 \times 0.16 \times 0.13$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.883$, $T_{\max} = 0.954$
10 903 measured reflections
3735 independent reflections
3007 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.046$
$\theta_{\max} = 25.2^\circ$
$h = -9 \rightarrow 4$
$k = -31 \rightarrow 33$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.098$
$wR(F^2) = 0.248$
$S = 1.18$
3735 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0905P)^2 + 7.5446P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.022$
$\Delta\rho_{\max} = 0.90$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N4—H4$\cdots$O2	0.86	2.07	2.878 (7)	157

All H atoms were initially located in a difference Fourier map and then placed in geometrically idealized positions and included in the refinement in the riding-model approximation, with N—H distances of 0.86 Å and C—H distances in the range 0.93–0.96 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$ of the carrier atom (1.5$U_{\rm eq}$ in the case of methyl H atoms). High displacement parameters for atoms F1, F2 and F3 indicated either large thermal motion or rotational disorder of the trifluoromethyl group. However, attempts to represent the CF$_3$ group using a disordered model were unsuccessful. The inability to account for the details of electron-density distribution in the vicinity of the CF$_3$ group is the most probable reason for the rather limited overall precision of the structure.

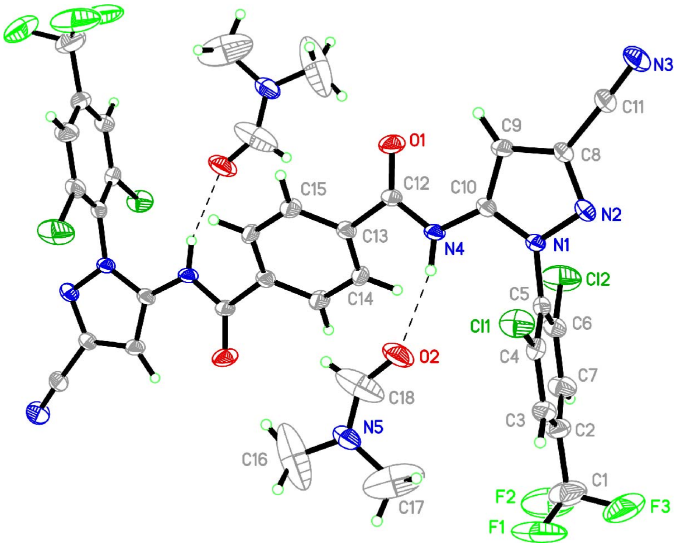


Figure 1
The structure of (I), showing the atom-numbering scheme and with displacement ellipsoids at the 50% probability level. H atoms are shown as small spheres of arbitrary radii and the dashed lines indicate hydrogen bonds. Unlabelled atoms are related to labelled atoms by $1 - x$, $1 - y$, $1 - z$.

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: *SHELXL97*.

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