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4-{(2,4-Dichlorophenyl)[5-hydroxy-1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-4yl]methyl}-1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5-ol monohydrate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.057
wR factor = 0.137
Data-to-parameter ratio = 14.7

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

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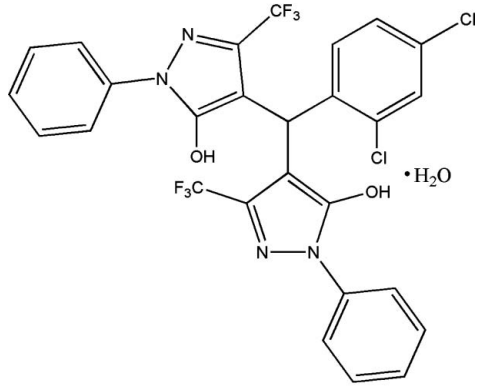
The title compound, $C_{27}H_{16}Cl_2F_6N_4O_2\cdot H_2O$, was obtained by the reaction of 2,4-dichlorobenzaldehyde and 2-phenyl-5-trifluoromethyl-2*H*-pyrazol-3-ol in aqueous media without any catalyst. In the crystal structure, both intra- and intermolecular hydrogen bonds are found.

Comment

Pyrazole derivatives are very attractive for their various bioactivities. For example, pyrazolate is a widely used herbicide (Endo *et al.*, 2004). Some of them can inhibit the release of inflammatory cytokines and tumor necrosis factor (TNF) (John *et al.*, 2003; Clark & Lyon, 2005). Water-insoluble azo dyes are derived from the pyrazole ring (Bernardin & Pechmeze, 1980). Compounds that contain fluorine, such as flumioxazin, are widely used as herbicides (Hermann *et al.*, 2003; Ulrich, 2004). To further study the relationship between structure and bioactivity, we synthesized a series of pyrazole derivatives containing the trifluoromethyl group. We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The dihedral angles between planes C12–C17 and C7–C9/N1/N2, planes C12–C17 and C18–C20/N3/N4, and planes C7–C9/N1/N2 and C18–C20/N3/N4 are 79.4 (1), 76.4 (1) and 57.9 (2)°, respectively. In the molecular structure, there are two intramolecular hydrogen bonds: O1—H1*A*···O3 and O2—H2*A*···O1. The crystal packing is stabilized by two intermolecular hydrogen bonds: O3—H3*A*···N2 and O3—H3*B*···N1 (Fig. 2 and Table 1).

Experimental

The title compound was synthesized, according to the procedure reported by Shi *et al.* (2005), by the reaction of 2,4-dichloro-

benzaldehyde and 2-phenyl-5-trifluoromethyl-2*H*-pyrazol-3-ol in a 1:2 molar ratio in aqueous media without any catalyst at 363 K. After cooling, the precipitate was filtered off and recrystallized from ethanol, giving single crystals suitable for X-ray diffraction.

Crystal data

$C_{27}H_{16}Cl_2F_6N_4O_2\cdot H_2O$
$M_r = 631.35$
Orthorhombic, *Pbca*
$a = 19.214$ (3) Å
$b = 14.821$ (2) Å
$c = 19.639$ (3) Å
$V = 5592.9$ (15) Å^3
$Z = 8$
$D_x = 1.500$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3648 reflections
$\theta = 2.3$–$21.9°$
$\mu = 0.31$ mm^{-1}
$T = 294$ (2) K
Block, colorless
0.30 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick 1996)
$T_{min} = 0.913$, $T_{max} = 0.976$
30100 measured reflections
5733 independent reflections
2698 reflections with $I > 2\sigma(I)$
$R_{int} = 0.108$
$\theta_{max} = 26.4°$
$h = -24 \rightarrow 22$
$k = -15 \rightarrow 18$
$l = -22 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.137$
$S = 1.00$
5733 reflections
391 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 5.8586P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O3	0.82	1.61	2.424 (4)	169
O2—H2A···O1	0.80 (4)	1.89 (4)	2.681 (4)	172 (5)
O3—H3A···N2^{i}	0.86 (5)	1.94 (5)	2.802 (4)	177 (5)
O3—H3B···N4^{ii}	0.82 (5)	1.98 (5)	2.792 (4)	169 (5)

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, z$; (ii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$.

H atoms bonded to O atoms were located in a Fourier difference map and were refined freely, except for that on O1, where the instruction AFIX 3 was used to fix the atomic parameters. Other H atoms were placed in calculated positions, with C—H = 0.93 or 0.98 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$(parent atom). In the molecular structure, the F atoms of one CF_3 group (F1, F2 and F3) were disordered over two positions, with refined site-occupancy factors of 0.889 (7) and 0.111 (7), for which the C—F bond lengths were restrained to 1.32 (1) Å. The C atoms of the C21–C26 aromatic ring and their attached H atoms were disordered over two positions also, with refined site-occupancy factors of 0.890 (3) and 0.110 (3).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

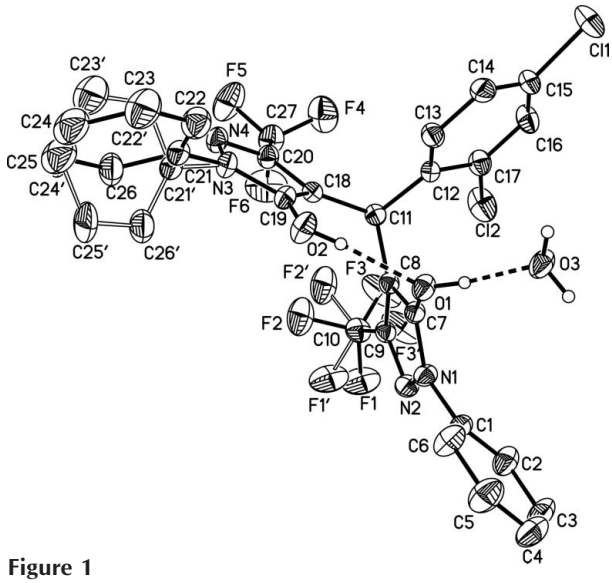


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted. Both disorder components are shown.

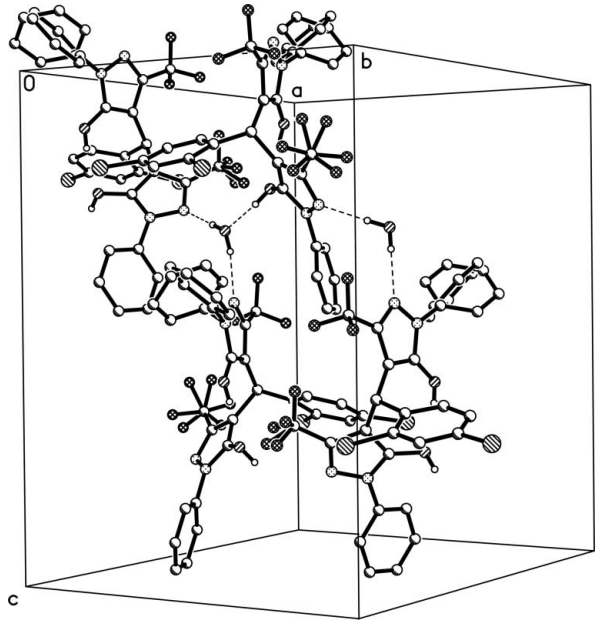


Figure 2
The packing diagram of (I). Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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