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4-Hydroxymethyl-3,5-dimethyl-1*H*-pyrazole chloroform solvate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.076
wR factor = 0.210
Data-to-parameter ratio = 17.2

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

In the title compound, $C_6H_{10}N_2O{\cdot}CHCl_3$, the geometric parameters are in the usual ranges. The structure is stabilized by N—H···O, O—H···N and C—H···O hydrogen bonds.

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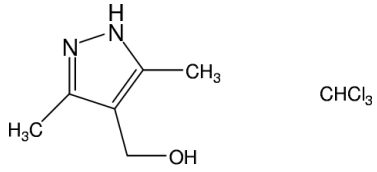
Comment

A perspective view of the title compound, (I), is shown in Fig. 1. The structure is composed of discrete 3,5-dimethyl-4-hydroxymethylpyrazole and chloroform solvent molecules. Bond lengths and angles can be regarded as normal (Cambridge Crystallographic Database, *CONQUEST* Version 1.6 plus three updates, *MOGUL* Version 1.0; Allen, 2002). The pyrazole heterocycle is essentially planar. The hydroxyl group is rotated by −78.1 (6)° out of this plane. The structure is stabilized by N—H···O, O—H···N and C—H···O hydrogen bonds (Fig. 2 and Table 2).



(I)

Experimental

3,5-Dimethyl-4-hydroxymethylpyrazole was synthesized in the following way (Stumpf, 1999): 4-bromo-3,5-dimethylpyrazole was treated with five equivalents of *tert*-butyllithium in THF. Four equivalents of DMF were added and the resulting 3,5-dimethyl-4-formylpyrazole reduced with sodium borohydride in ethanol to the corresponding alcohol.

Crystal data

$C_6H_{10}N_2O{\cdot}CHCl_3$
M_r = 245.53
Monoclinic, $P2_1/c$
a = 12.423 (2) Å
b = 7.843 (1) Å
c = 12.871 (2) Å
β = 109.44 (2)°
V = 1182.6 (3) Å^3
Z = 4
D_x = 1.379 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1700 reflections
θ = 1–25°
μ = 0.74 mm^{-1}
T = 173 (2) K
Plate, colourless
0.40 × 0.30 × 0.05 mm

Data collection

Siemens SMART CCD three-circle diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.756, T_{max} = 0.964
8134 measured reflections
2084 independent reflections
1067 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 25.0°
$h = -14 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 15$

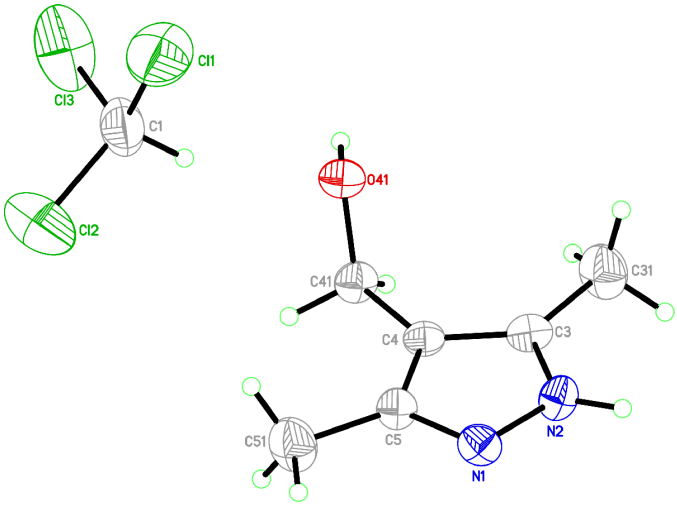


Figure 1
Perspective view of the title compound, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0872P)^2 + 1.7867P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.076$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.210$	$\Delta\rho_{max} = 0.43$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.59$ e Å^{-3}
2084 reflections	
121 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.335 (6)	C3—C4	1.390 (7)
N1—N2	1.344 (6)	C4—C5	1.405 (7)
N2—C3	1.349 (6)		
C5—N1—N2	105.3 (4)	C3—C4—C5	105.5 (4)
N1—N2—C3	113.0 (4)	N1—C5—C4	110.6 (4)
N2—C3—C4	105.7 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O41^{i}	0.88	1.97	2.830 (5)	166
C1—H1···O41	1.00	2.14	3.094 (7)	159
O41—H41···N1^{ii}	0.84	1.88	2.707 (5)	167

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, \frac{3}{2}-y, z-\frac{1}{2}$.

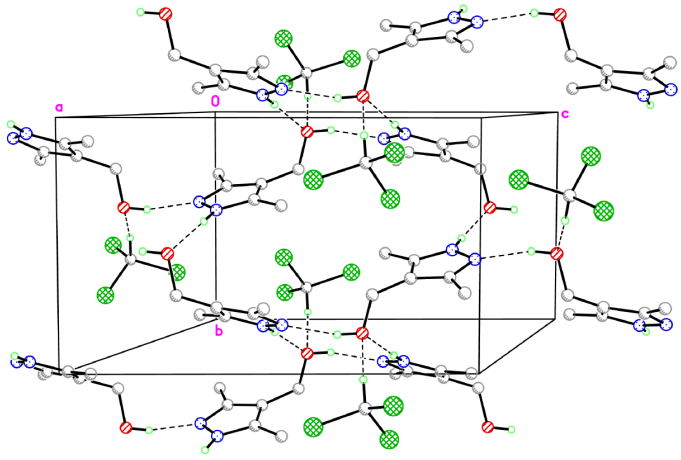


Figure 2
Packing diagram of the title compound, viewed on the *bc* plane. Colour codes: C: shaded black circles; H: small white circles; Cl: cross-hatched green circles; N: dotted blue circles; O: shaded red circles.

H atoms were refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C, N, O) or U(H) = 1.5 U_{eq}(C_{methyl})] using a riding model, with O—H = 0.84 Å, N—H = 0.88 Å and C—H ranging from 0.98 to 1.00 Å. One of the methyl groups was found to be disordered over two positions. The site occupation factor for the two different orientations refined to 0.45 (6)/0.55 (6). The hydroxyl group and the non-disordered methyl group were allowed to rotate but not to tip.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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