

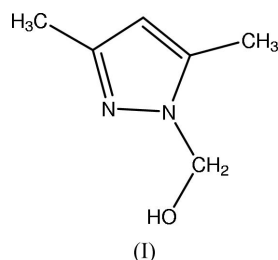
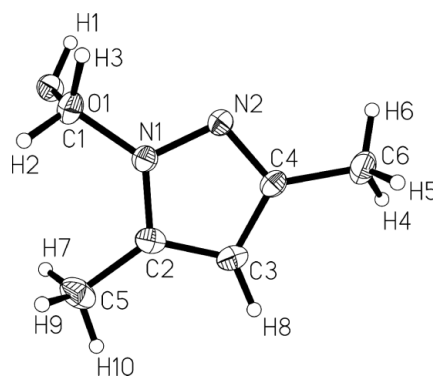
1-(Hydroxymethyl)-3,5-dimethylpyrazole

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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.001$ Å
 R factor = 0.040
 wR factor = 0.100
Data-to-parameter ratio = 17.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The structure of the title compound, $\text{C}_6\text{H}_{10}\text{N}_2\text{O}$, has been determined from single crystals obtained by recrystallization from acetone. Intermolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonding gives rise to $R_2^2(10)$ dimers.

Comment

1-(Hydroxymethyl)-3,5-dimethylpyrazole (HL), (I), was first synthesized by a reaction of 3,5-dimethylpyrazole with paraformaldehyde (Driessen, 1982). HL can act as a chelating ligand, as shown by the examples of four isomorphous cubane-type cluster coordination compounds of the formula $[\text{MXL}(\text{EtOH})_4]$ ($M = \text{Co}^{\text{II}}, \text{Ni}^{\text{II}}, X = \text{Cl}, \text{Br}$; Paap *et al.*, 1985), where the deprotonated species acts as a bidentate ligand, coordinating the metals through its pyridine N atom and the methoxy O atom. With a metal with different coordination preferences, such as Pd, HL acts as a monodentate ligand through the pyrazole N atom, forming a square-planar complex (Boixassa *et al.*, 2002).The molecular structure of (I) is shown in Fig. 1. The substituted pyrazole ring is essentially planar, the largest displacement being 0.01 Å for C4. The $\text{O1}-\text{C1}-\text{N1}-\text{N2}$ torsion angle is $90.19(9)^\circ$. The structure is stabilized by intermolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonding between adjacent molecules [$\text{H}\cdots\text{N} = 1.89(2)$ Å, $\text{O}\cdots\text{N} = 2.760(1)$ Å and $\text{O}-\text{H}\cdots\text{N} = 171.0(2)^\circ$]. These interactions give rise to dimers**Figure 1**
The molecular structure of (I) and the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

with an $R_2^2(10)$ hydrogen-bonding motif (Etter *et al.*, 1990) (Fig. 2).

Experimental

Needle-shaped clear single crystals of (I) were obtained by recrystallization of the commercial product (Aldrich) from acetone.

Crystal data

$C_6H_{10}N_2O$	$D_x = 1.238 \text{ Mg m}^{-3}$
$M_r = 126.16$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 6570 reflections
$a = 7.2877(2) \text{ \AA}$	$\theta = 3.1\text{--}34.3^\circ$
$b = 11.9265(3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 8.1586(2) \text{ \AA}$	$T = 120 \text{ K}$
$\beta = 107.396(1)^\circ$	Needle, white
$V = 676.68(3) \text{ \AA}^3$	$0.80 \times 0.20 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2742 independent reflections
ω scans	2113 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.019$
$T_{\text{min}} = 0.92$, $T_{\text{max}} = 0.98$	$\theta_{\text{max}} = 34.5^\circ$
13356 measured reflections	$h = -11 \rightarrow 11$
	$k = -18 \rightarrow 19$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$1.31T_2(x)$ where T are the Chebychev polynomial terms and $x = F_c/F_{\text{max}}$ (Watkin, 1994; Prince, 1982)
$R[F^2 > 2\sigma(F^2)] = 0.040$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$wR(F^2) = 0.100$	$\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$
$S = 1.00$	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
2113 reflections	
122 parameters	
All H-atom parameters refined	
$w = [1 - (F_o - F_c)/6\sigma(F_o)^2] / [3.44T_0(x) + 4.59T_1(x) +$	

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—C1	1.4007 (10)	C2—C5	1.4918 (13)
N1—C1	1.4530 (11)	C3—C2	1.3814 (13)
N1—C2	1.3568 (11)	C3—C4	1.4073 (12)
N2—N1	1.3627 (10)	C6—C4	1.4934 (12)
N2—C4	1.3362 (10)		
N1—N2—C4	105.40 (7)	C3—C2—N1	106.36 (7)
N2—N1—C1	119.71 (7)	C3—C2—C5	131.14 (8)
N2—N1—C2	111.90 (7)	N1—C2—C5	122.49 (8)
C1—N1—C2	128.31 (7)	C6—C4—C3	128.72 (8)
N1—C1—O1	112.93 (7)	C6—C4—N2	120.72 (8)
C2—C3—C4	105.78 (7)	C3—C4—N2	110.55 (7)

H atoms were found in a difference Fourier map and refined using an isotropic approximation. Refined C—H bond lengths are in the range 0.95 (2)–1.04 (2) \AA and the O—H bond length is 0.88 (2) \AA .

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: CRYSTALS (Betteridge *et al.*, 2003); molecular graphics: ATOMS (Dowty, 2000); software used to prepare material for publication: CRYSTALS.

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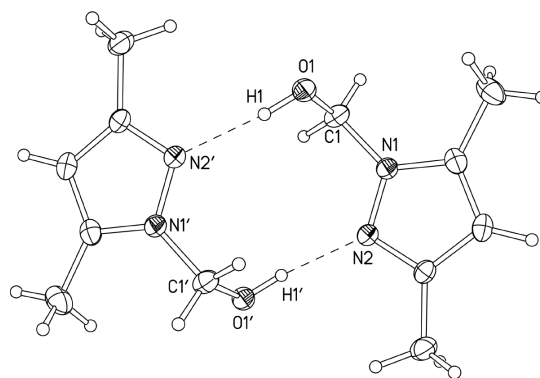


Figure 2
The hydrogen bonding (dashed lines) in (I).

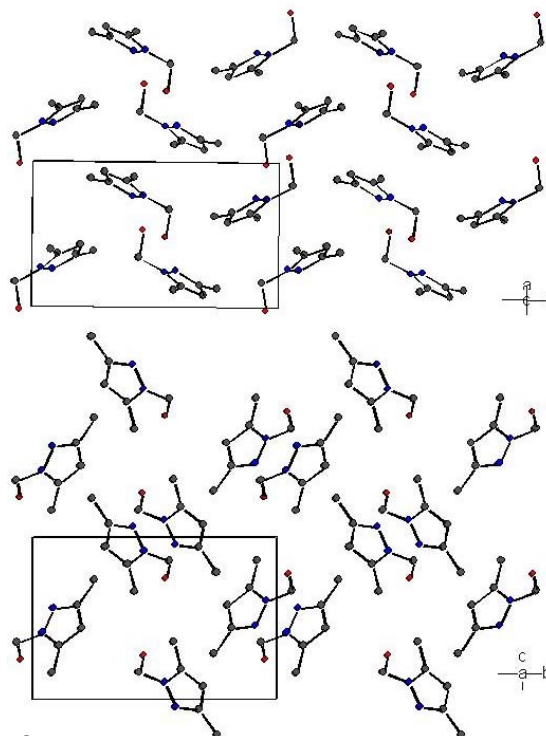


Figure 3
Two views of the packing scheme in (I).

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Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 7.2877$ (2) Å

$b = 11.9265$ (3) Å

$c = 8.1586$ (2) Å

$\beta = 107.396$ (1)°

$V = 676.68$ (3) Å³

$Z = 4$

$F(000) = 272$

$D_x = 1.238$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6570 reflections

$\theta = 3.1$ – 34.3 °

$\mu = 0.09$ mm⁻¹

$T = 120$ K

Prism, white

$0.80 \times 0.20 \times 0.20$ mm

Data collection

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Graphite monochromator

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13356 measured reflections

2742 independent reflections

2113 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\max} = 34.5$ °, $\theta_{\min} = 3.1$ °

$h = -11 \rightarrow 11$

$k = -18 \rightarrow 19$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.100$

$S = 1.00$

2113 reflections

122 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: difference Fourier map

All H-atom parameters refined

Chebyshev polynomial, (Watkin, 1994; Prince,
1982): $w = 1.0/[A_0T_0(x) + A_1T_1(x) \dots +$

$A_{n-1}]T_{n-1}(x)]$,

where A_i are the Chebyshev coefficients listed
below and $x = F_c/F_{\max}$; method = Robust

Weighting (Prince, 1982); $W = w[1 - (\delta F/6\sigma F)^2]^2$;

A_i are 3.44 4.59 1.31

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.40$ e Å⁻³

$\Delta\rho_{\min} = -0.31$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	-0.02535 (9)	-0.04616 (6)	0.23811 (8)	0.0220
N1	0.26298 (10)	0.04284 (6)	0.40283 (9)	0.0190
N2	0.25525 (10)	0.08755 (6)	0.55433 (9)	0.0189

C1	0.16421 (12)	-0.06190 (7)	0.34201 (12)	0.0226
C2	0.36052 (11)	0.10985 (8)	0.32329 (10)	0.0211
C3	0.42099 (11)	0.20151 (7)	0.42920 (12)	0.0218
C4	0.35202 (11)	0.18413 (7)	0.57094 (11)	0.0194
C5	0.38717 (15)	0.08069 (11)	0.15405 (13)	0.0317
C6	0.37831 (13)	0.25601 (8)	0.72617 (13)	0.0280
H1	-0.101 (2)	-0.0516 (14)	0.304 (2)	0.040 (4)*
H2	0.237 (2)	-0.0995 (12)	0.2760 (19)	0.028 (3)*
H3	0.166 (2)	-0.1056 (13)	0.446 (2)	0.033 (4)*
H4	0.362 (3)	0.3355 (17)	0.692 (2)	0.053 (5)*
H5	0.508 (3)	0.2465 (15)	0.808 (3)	0.055 (5)*
H6	0.277 (3)	0.2393 (16)	0.783 (2)	0.051 (5)*
H7	0.265 (3)	0.0639 (15)	0.071 (2)	0.047 (5)*
H8	0.496 (2)	0.2622 (12)	0.408 (2)	0.031 (3)*
H9	0.480 (3)	0.0133 (16)	0.167 (2)	0.048 (4)*
H10	0.455 (3)	0.1415 (17)	0.119 (3)	0.059 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0180 (2)	0.0279 (3)	0.0187 (3)	-0.0024 (2)	0.0034 (2)	-0.0020 (2)
N1	0.0176 (3)	0.0203 (3)	0.0181 (3)	-0.0014 (2)	0.0041 (2)	-0.0009 (2)
N2	0.0180 (3)	0.0193 (3)	0.0190 (3)	-0.0019 (2)	0.0049 (2)	-0.0013 (2)
C1	0.0191 (3)	0.0201 (3)	0.0260 (4)	0.0000 (2)	0.0028 (3)	-0.0044 (3)
C2	0.0164 (3)	0.0278 (4)	0.0183 (3)	0.0008 (3)	0.0038 (2)	0.0043 (3)
C3	0.0174 (3)	0.0225 (3)	0.0242 (3)	-0.0022 (2)	0.0042 (3)	0.0053 (3)
C4	0.0158 (3)	0.0188 (3)	0.0222 (3)	-0.0013 (2)	0.0033 (2)	-0.0003 (3)
C5	0.0273 (4)	0.0490 (6)	0.0191 (4)	0.0018 (4)	0.0075 (3)	0.0018 (4)
C6	0.0247 (4)	0.0270 (4)	0.0313 (4)	-0.0050 (3)	0.0068 (3)	-0.0095 (3)

Geometric parameters (Å, °)

O1—C1	1.4007 (10)	C6—H6	1.000 (18)
N1—C1	1.4530 (11)	C6—H4	0.98 (2)
N1—C2	1.3568 (11)	O1—H1	0.878 (17)
N2—N1	1.3627 (10)	C1—H2	0.972 (14)
N2—C4	1.3362 (10)	C1—H3	0.995 (16)
C2—C5	1.4918 (13)	C3—H8	0.952 (15)
C3—C2	1.3814 (13)	C5—H7	0.963 (18)
C3—C4	1.4073 (12)	C5—H10	0.97 (2)
C6—C4	1.4934 (12)	C5—H9	1.038 (19)
C6—H5	0.988 (19)		
C4—C6—H5	111.2 (11)	H2—C1—H3	111.2 (12)
C4—C6—H6	110.7 (11)	C2—C3—C4	105.78 (7)
H5—C6—H6	110.5 (15)	C2—C3—H8	125.3 (9)
C4—C6—H4	109.8 (11)	C4—C3—H8	128.9 (9)
H5—C6—H4	108.1 (14)	C3—C2—N1	106.36 (7)

H6—C6—H4	106.2 (15)	C3—C2—C5	131.14 (8)
C1—O1—H1	107.9 (11)	N1—C2—C5	122.49 (8)
N1—N2—C4	105.40 (7)	C6—C4—C3	128.72 (8)
N2—N1—C1	119.71 (7)	C6—C4—N2	120.72 (8)
N2—N1—C2	111.90 (7)	C3—C4—N2	110.55 (7)
C1—N1—C2	128.31 (7)	C2—C5—H7	110.6 (11)
N1—C1—O1	112.93 (7)	C2—C5—H10	108.5 (12)
N1—C1—H2	106.8 (9)	H7—C5—H10	113.3 (16)
O1—C1—H2	109.3 (9)	C2—C5—H9	110.3 (11)
N1—C1—H3	106.2 (9)	H7—C5—H9	110.4 (15)
O1—C1—H3	110.4 (8)	H10—C5—H9	103.5 (14)
