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## Key indicators

Single-crystal X-ray study  
 $T = 150$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.049  
 $wR$  factor = 0.147  
Data-to-parameter ratio = 18.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## (3,5-Dimethylpyrazol-1-yl)acetic acid

At 150 K, the title compound,  $\text{C}_7\text{H}_{10}\text{N}_2\text{O}_2$ , comprises one-dimensional hydrogen-bonded homochiral helical chains of molecules.

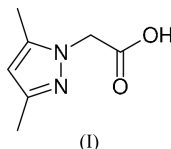
Received 27 April 2004

Accepted 4 May 2004

Online 8 May 2004

## Comment

The molecular structure of the title compound, (I), is shown in Fig. 1 and selected structural parameters are listed in Table 1. The least-squares planes of the pyrazole ring and the carboxylic acid group are almost mutually perpendicular, with a dihedral angle of  $87.57(7)^\circ$ , and atom N2 is close to being coplanar with the carboxylic acid group, lying only  $0.0067(15)$  Å out of the least-squares plane of the latter. The molecules form homochiral helical hydrogen-bonded chains parallel to the  $b$  axis (Fig. 2 and Table 2).



## Experimental

The title compound, (I), was prepared according to the method of Micetich (1970).

## Crystal data

$\text{C}_7\text{H}_{10}\text{N}_2\text{O}_2$   
 $M_r = 154.17$   
Orthorhombic,  $P2_12_12_1$   
 $a = 4.8528(4)$  Å  
 $b = 7.0073(6)$  Å  
 $c = 23.256(3)$  Å  
 $V = 790.82(13)$  Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.295$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
Cell parameters from 6939  
reflections  
 $\theta = 3.0\text{--}34.8^\circ$   
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 150(2)$  K  
Lath, colourless  
 $0.60 \times 0.25 \times 0.10$  mm

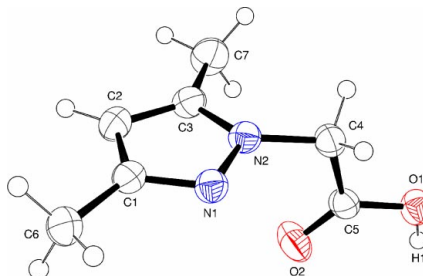


Figure 1

View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

**Data collection**

Stoe IPDS-II area-detector  
diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: none  
11979 measured reflections  
2013 independent reflections

1357 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.057$   
 $\theta_{\text{max}} = 34.8^\circ$   
 $h = -7 \rightarrow 10$   
 $k = -11 \rightarrow 10$   
 $l = -37 \rightarrow 37$

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.147$   
 $S = 1.05$   
2013 reflections  
107 parameters  
H atoms treated by a mixture of  
independent and constrained  
refinement

$w = 1/[\sigma^2(F_o^2) + (0.0896P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.26 \text{ e } \text{\AA}^{-3}$   
Extinction correction: *SHELXL97*  
Extinction coefficient: 0.088 (15)

**Table 1**Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

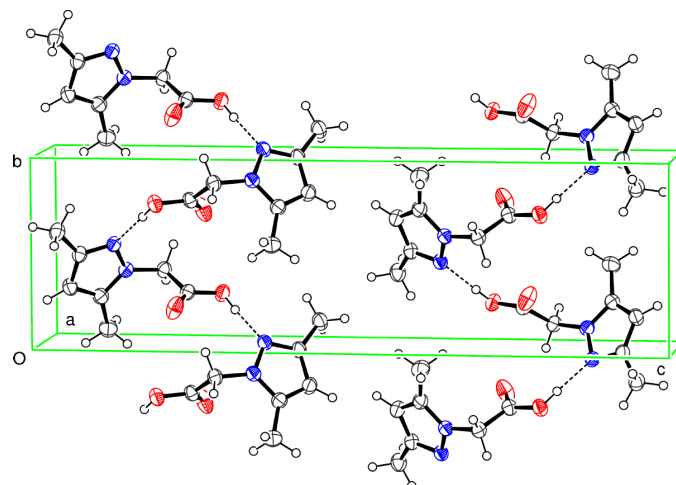
O1—C5	1.318 (2)	C1—C2	1.407 (3)
O2—C5	1.201 (2)	N1—C1	1.331 (2)
N1—N2	1.359 (2)	N2—C4	1.451 (2)
N2—C3	1.348 (2)	C4—C5	1.514 (3)
C2—C3	1.381 (3)		
N1—N2—C4	119.87 (14)	O2—C5—O1	124.93 (18)
N2—C4—C5	110.48 (15)		
N1—N2—C4—C5	87.48 (19)	N2—C4—C5—O2	0.7 (3)

**Table 2**Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 $\cdots$ N1 <sup>i</sup>	0.95 (3)	1.79 (3)	2.723 (2)	169 (3)

Symmetry code: (i)  $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$ .

All H atoms were initially located in a difference Fourier map. The positional and isotropic displacement parameters for the hydroxyl H atom were freely refined. The methyl H atoms were constrained to an ideal geometry, with a C—H distance of 0.98  $\text{\AA}$ , but each group was allowed to rotate freely about its X—C bond. All other C—H atoms

**Figure 2**

The packing and unit cell of (I), viewed approximately down the  $a$  axis. Hydrogen bonds are denoted by dashed lines.

were placed in geometrically idealized positions, with C—H distances of 0.95–0.99  $\text{\AA}$ .  $U_{\text{iso}}(\text{H})$  values were set at  $1.2U_{\text{eq}}(\text{C})$  for all C—H atoms.

Data collection: *X-AREA* (Stoe, 2001); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2001).

**References**

- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.  
Micetich, R. G. (1970). *Can. J. Chem.* **48**, 2006–2015.  
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.  
Spek, A. L. (2001). *PLATON*. University of Utrecht, The Netherlands.  
Stoe. (2001). *X-AREA* and *X-RED32*. Stoe & Cie GmbH, Darmstadt, Germany.

## supporting information

*Acta Cryst.* (2004). E60, o966–o967 [https://doi.org/10.1107/S1600536804010761]

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*Crystal data*

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Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

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$c = 23.256$  (3) Å

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$Z = 4$

$F(000) = 328$

$D_x = 1.295$  Mg m<sup>-3</sup>

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

Cell parameters from 6939 reflections

$\theta = 3.0$ – $34.8^\circ$

$\mu = 0.10$  mm<sup>-1</sup>

$T = 150$  K

Lath, colourless

$0.60 \times 0.25 \times 0.10$  mm

*Data collection*

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diffractometer

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Graphite monochromator

$\varphi$  and  $\omega$  scans

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2013 independent reflections

1357 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.057$

$\theta_{max} = 34.8^\circ$ ,  $\theta_{min} = 3.0^\circ$

$h = -7 \rightarrow 6$

$k = -11 \rightarrow 10$

$l = -37 \rightarrow 37$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.147$

$S = 1.05$

2013 reflections

107 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0896P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.26$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.26$  e Å<sup>-3</sup>

Extinction correction: SHELXL97,

$Fc^* = kFc[1 + 0.001 \times Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.088 (15)

Special details

**Experimental.** The crystal was mounted under the perfluoro-polyether PFO-XR75 (Lancaster Synthesis). A total of 183 frames (3 minute exposure) were collected ( $\phi/\omega$ : 60/0–150, 140/0–33  $\Delta\omega = 1^\circ$ .)

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Weighted least-squares planes through the starred atoms (Nardelli, Musatti, Domiano & Andreetti Ric.Sci.(1965),15(II—A),807).

Plane 1 Atom d s d/s (d/s)\*\*2 N1 \* 0.0052 0.0016 3.329 11.084 N2 \* -0.0068 0.0015 - 4.436 19.679 C1 \* -0.0023 0.0019 - 1.194 1.425 C2 \* -0.0046 0.0021 - 2.203 4.852 C3 \* 0.0093 0.0020 4.733 22.397 C4 0.1719 0.0019 91.699 8408.692 C6 0.0192 0.0022 8.748 76.533 C7 0.0805 0.0025 31.975 1022.373 ===== Sum((d/s)\*\*2) for starred atoms 59.437 Chi-squared at 95% for 2 degrees of freedom: 5.99

Plane 2 Atom d s d/s (d/s)\*\*2 C4 \* -0.0018 0.0019 - 0.939 0.882 C5 \* 0.0065 0.0019 3.369 11.352 O1 \* -0.0012 0.0015 - 0.843 0.710 O2 \* -0.0029 0.0020 - 1.409 1.984 N2 - 0.0067 0.0015 - 4.325 18.707 ===== Sum((d/s)\*\*2) for starred atoms 14.928 Chi-squared at 95% for 1 degrees of freedom: 3.84

Dihedral angles formed by LSQ-planes Plane - plane angle (s.u.) angle (s.u.) 1 2 87.57 (0.07) 92.43 (0.07)

Possible hydrogen bonds Donor-H Donor...Acceptor H...Acceptor Donor-H...Acceptor

O1—H1 O1...N1 (1) H1...N1 (1) O1—H1...N1 (1) 0.945(.029) 2.723(.002) 1.788(.029) 169.21 (2.64)

C4—H4A C4...O2 (3) H4A...O2 (3) C4—H4A...O2 (3) 0.990(.002) 3.090(.003) 2.369(.002) 128.98 (0.12)

C4—H4B C4...O2 (4) H4B...O2 (4) C4—H4B...O2 (4) 0.990(.002) 3.203(.003) 2.295(.002) 152.05 (0.12)

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) etc. and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.7445 (3)	0.7871 (2)	0.20669 (6)	0.0365 (3)
H1	0.857 (6)	0.711 (4)	0.1827 (11)	0.047 (7)*
O2	1.0832 (3)	0.7353 (3)	0.26981 (7)	0.0506 (5)
N1	0.9883 (3)	1.0557 (2)	0.36906 (6)	0.0305 (3)
N2	0.8155 (3)	0.9087 (2)	0.35669 (6)	0.0302 (3)
C1	1.1052 (4)	1.0099 (3)	0.41895 (7)	0.0324 (4)
C2	1.0090 (5)	0.8317 (3)	0.43831 (8)	0.0359 (4)
H2	1.0595	0.7673	0.4727	0.043*
C3	0.8258 (4)	0.7701 (3)	0.39683 (7)	0.0340 (4)
C4	0.6802 (4)	0.9005 (3)	0.30112 (7)	0.0322 (3)
H4A	0.6406	1.0316	0.2876	0.039*
H4B	0.5028	0.8318	0.3049	0.039*
C5	0.8611 (4)	0.7992 (3)	0.25772 (7)	0.0315 (4)
C6	1.3125 (5)	1.1405 (3)	0.44582 (8)	0.0396 (4)
H6A	1.3129	1.2628	0.4254	0.048*
H6B	1.4959	1.0825	0.4434	0.048*
H6C	1.2644	1.1617	0.4862	0.048*
C7	0.6677 (5)	0.5879 (3)	0.39156 (10)	0.0459 (5)
H7A	0.7373	0.5148	0.3587	0.055*
H7B	0.4721	0.6166	0.3857	0.055*
H7C	0.6901	0.5127	0.4268	0.055*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0385 (7)	0.0411 (7)	0.0300 (5)	0.0043 (6)	-0.0031 (5)	-0.0059 (5)
O2	0.0321 (7)	0.0757 (12)	0.0439 (8)	0.0119 (8)	-0.0075 (6)	-0.0208 (8)
N1	0.0321 (7)	0.0315 (7)	0.0277 (6)	-0.0024 (6)	0.0000 (6)	-0.0027 (5)
N2	0.0305 (7)	0.0306 (6)	0.0295 (6)	-0.0011 (6)	0.0010 (6)	-0.0022 (5)
C1	0.0319 (8)	0.0369 (9)	0.0284 (7)	-0.0021 (7)	0.0020 (7)	-0.0011 (6)
C2	0.0388 (9)	0.0390 (9)	0.0299 (7)	-0.0019 (8)	0.0000 (7)	0.0040 (7)
C3	0.0353 (9)	0.0345 (8)	0.0322 (7)	-0.0027 (8)	0.0035 (7)	0.0012 (6)
C4	0.0297 (8)	0.0361 (8)	0.0309 (7)	0.0010 (7)	-0.0026 (7)	-0.0019 (6)
C5	0.0287 (8)	0.0342 (8)	0.0316 (7)	-0.0040 (7)	-0.0017 (6)	-0.0030 (6)
C6	0.0402 (10)	0.0454 (10)	0.0334 (8)	-0.0074 (9)	-0.0029 (8)	-0.0033 (8)
C7	0.0528 (13)	0.0390 (10)	0.0457 (10)	-0.0127 (10)	-0.0002 (10)	0.0028 (8)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C5	1.318 (2)	C2—H2	0.9500
O1—H1	0.95 (3)	C3—C7	1.495 (3)
O2—C5	1.201 (2)	C4—H4A	0.9900
N1—N2	1.359 (2)	C4—H4B	0.9900
N2—C3	1.348 (2)	C6—H6A	0.9800
C2—C3	1.381 (3)	C6—H6B	0.9800
C1—C2	1.407 (3)	C6—H6C	0.9800
N1—C1	1.331 (2)	C7—H7A	0.9800
N2—C4	1.451 (2)	C7—H7B	0.9800
C4—C5	1.514 (3)	C7—H7C	0.9800
C1—C6	1.497 (3)		
C5—O1—H1	108.5 (17)	C5—C4—H4B	109.6
C1—N1—N2	105.34 (15)	H4A—C4—H4B	108.1
C3—N2—N1	112.12 (15)	O2—C5—O1	124.93 (18)
C3—N2—C4	127.23 (16)	O2—C5—C4	122.63 (17)
N1—N2—C4	119.87 (14)	O1—C5—C4	112.42 (16)
N1—C1—C2	110.60 (17)	C1—C6—H6A	109.5
N1—C1—C6	120.19 (17)	C1—C6—H6B	109.5
C2—C1—C6	129.19 (18)	H6A—C6—H6B	109.5
C3—C2—C1	105.50 (16)	C1—C6—H6C	109.5
C3—C2—H2	127.3	H6A—C6—H6C	109.5
C1—C2—H2	127.3	H6B—C6—H6C	109.5
N2—C3—C2	106.42 (17)	C3—C7—H7A	109.5
N2—C3—C7	122.65 (17)	C3—C7—H7B	109.5
C2—C3—C7	130.89 (18)	H7A—C7—H7B	109.5
N2—C4—C5	110.48 (15)	C3—C7—H7C	109.5
N2—C4—H4A	109.6	H7A—C7—H7C	109.5
C5—C4—H4A	109.6	H7B—C7—H7C	109.5
N2—C4—H4B	109.6		

C1—N1—N2—C3	-1.4 (2)	N1—N2—C3—C7	-176.37 (18)
C1—N1—N2—C4	-172.01 (16)	C4—N2—C3—C7	-6.6 (3)
N2—N1—C1—C2	0.6 (2)	C1—C2—C3—N2	-1.1 (2)
N2—N1—C1—C6	179.48 (17)	C1—C2—C3—C7	176.6 (2)
N1—C1—C2—C3	0.3 (2)	C3—N2—C4—C5	-81.6 (2)
C6—C1—C2—C3	-178.4 (2)	N1—N2—C4—C5	87.48 (19)
N1—N2—C3—C2	1.6 (2)	N2—C4—C5—O2	0.7 (3)
C4—N2—C3—C2	171.38 (18)	N2—C4—C5—O1	179.42 (15)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1 $\cdots$ N1 <sup>i</sup>	0.95 (3)	1.79 (3)	2.723 (2)	169 (3)

Symmetry code: (i)  $-x+2, y-1/2, -z+1/2$ .