

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-(3,5-Dimethyl-1H-pyrazol-1-yl)-propanamide

Jian-Feng Zhang,* Feng Huang and Shu-Jiao Chen

State Key Laboratory Base of Novel Functional Materials and Preparation Science, Faculty of Materials Science and Chemical Engineering, Ningbo University, Ningbo, Zhejiang 315211, People's Republic of China

Correspondence e-mail: zjf@nbu.edu.cn

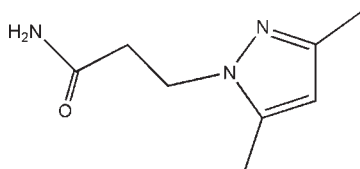
Received 12 June 2009; accepted 21 August 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.033; wR factor = 0.093; data-to-parameter ratio = 9.5.

In the crystal of the title compound, $\text{C}_8\text{H}_{13}\text{N}_3\text{O}$, molecules are linked by intermolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into a three-dimensional network. Additional stabilization is provided by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the potential applications of hemilabile ligands containing substituted pyrazole groups, see: Pal *et al.* (2005); Shaw *et al.* (2004). For the design of various pyrazole ligands with special structural properties to fulfill the specific stereochemical requirement of a particular metal-binding site, see: Mukherjee (2000); Paul *et al.* (2004);



Experimental

Crystal data

$\text{C}_8\text{H}_{13}\text{N}_3\text{O}$
 $M_r = 167.21$
 Orthorhombic, $Fdd2$
 $a = 14.452$ (5) Å
 $b = 33.390$ (7) Å
 $c = 7.4354$ (15) Å

$V = 3588.0$ (16) Å³
 $Z = 16$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ K
 $0.47 \times 0.37 \times 0.36$ mm

Data collection

Rigaku R-Axis RAPID
 diffractometer
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.963$, $T_{\max} = 0.970$

4623 measured reflections
 1067 independent reflections
 890 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.093$
 $S = 1.14$
 1067 reflections
 112 parameters

1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.14$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{i}}$	0.86	2.10	2.936 (3)	164
$\text{N1}-\text{H1B}\cdots\text{N3}^{\text{ii}}$	0.86	2.30	3.084 (3)	152
$\text{C3}-\text{H3B}\cdots\text{O1}^{\text{iii}}$	0.97	2.52	3.413 (3)	154

Symmetry codes: (i) $x - \frac{1}{4}, -y + \frac{1}{4}, z - \frac{1}{4}$; (ii) $-x, -y, z$; (iii) $x + \frac{1}{4}, -y + \frac{1}{4}, z + \frac{1}{4}$

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This project was sponsored by the K. C. Wong Magna Fund of Ningbo University and supported by the Project of Zhejiang Province Science and Technology Program (grant No. 2008 C21043), the Program of Ningbo Natural Science Foundation (grant No. 2007 A610053) and the Project of Zhejiang Province New Talent Program (grant No. 2008R40G2070020). We thank Ms Y. Zhou for the help with the structure analysis and Mr W. Xu for the diffraction data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2847).

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supplementary materials

Acta Cryst. (2009). E65, o2442 [doi:10.1107/S160053680903342X]

3-(3,5-Dimethyl-1*H*-pyrazol-1-yl)propanamide

J.-F. Zhang, F. Huang and S.-J. Chen

Comment

In recent years, there has been considerable interest in the use of hemilabile ligands containing substituted pyrazole groups because of their potential applications in catalysis and their ability for complex construction (Shaw *et al.*, 2004; Pal *et al.*, 2005). Nowadays, much attention has been focused on the design of various pyrazole ligands with special structural properties to fulfill the specific stereochemical requirement of a particular metal-binding site (Mukherjee, 2000; Paul *et al.*, 2004). Herein, we report the crystal structure of the title compound. The molecular structure of the title compound is shown in Fig. 1. In the crystal structure, molecules are linked by intermolecular N—H···N and N—H···O hydrogen bonds into a three dimensional network (see Fig. 2 and Table 1). Additional stabilization is provided by weak intermolecular C—H···O hydrogen bonds.

Experimental

A mixture of 3,5-dimethylpyrazole (3.845 g, 40 mmol), sodium hydroxide (0.2 g, 5 mmol) and *N,N*-dimethylformamide(DMF)(100 ml) was stirred and heated to 373 K. A solution of acrylamide (2.843 g, 40 mmol) in DMF (20 ml) was added dropwise. After 6 h, heating was then terminated. The cooled reaction mixture was filtered and DMF was removed by vacuum distillation to give 3.66 g analytically pure *N*-pyrazolylpropanimide (yield: 54.7%). Recrystallization from ethanol solution yielded colorless single-crystals suitable for X-ray diffraction analysis. Calculated for C₈H₁₃N₃O: C 57.42, H 7.78, N 25.12%; found: C 57.26, H 7.59, N 25.18%.

Refinement

H atoms were positioned geometrically and treated in the subsequent refinement as riding atoms, with C—H = 0.93 (aromatic), 0.97 Å (methylene), 0.96 (methyl) and N—H = 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$ or $1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$. In the absence of significant anomalous dispersion effects Friedel pairs were merged.

Figures

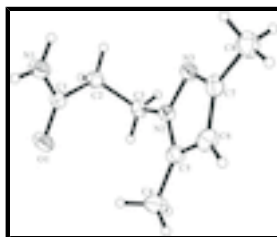


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

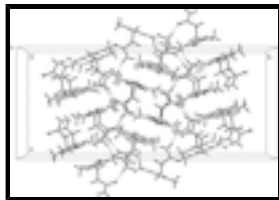


Fig. 2. Part of the crystal structure of the title compound. Dashed lines indicate hydrogen bonds.

3-(3,5-Dimethyl-1H-pyrazol-1-yl)propanamide

Crystal data

$C_8H_{13}N_3O$	$F_{000} = 1440$
$M_r = 167.21$	$D_x = 1.238 \text{ Mg m}^{-3}$
Orthorhombic, <i>Fdd2</i>	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: F 2 -2d	Cell parameters from 4623 reflections
$a = 14.452 (5) \text{ \AA}$	$\theta = 3.1\text{--}27.5^\circ$
$b = 33.390 (7) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 7.4354 (15) \text{ \AA}$	$T = 298 \text{ K}$
$V = 3588.0 (16) \text{ \AA}^3$	Block, colorless
$Z = 16$	$0.47 \times 0.37 \times 0.36 \text{ mm}$

Data collection

Rigaku R-Axis RAPID diffractometer	1067 independent reflections
Radiation source: fine-focus sealed tube	890 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.044$
Detector resolution: 0 pixels mm^{-1}	$\theta_{\text{max}} = 27.5^\circ$
$T = 298 \text{ K}$	$\theta_{\text{min}} = 3.1^\circ$
ω scans	$h = -17 \rightarrow 18$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -43 \rightarrow 43$
$T_{\text{min}} = 0.963$, $T_{\text{max}} = 0.970$	$l = -9 \rightarrow 8$
4623 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H-atom parameters constrained
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 1.8694P]$
$S = 1.14$	where $P = (F_o^2 + 2F_c^2)/3$
1067 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
112 parameters	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$

1 restraint

Extinction correction: SHELXTL'(Sheldrick, 2008),

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

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.0108 (8)

Special details

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.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.02008 (12)	0.12376 (4)	0.3335 (3)	0.0530 (5)
N1	-0.08464 (14)	0.07777 (6)	0.2532 (4)	0.0530 (6)
H1A	-0.1185	0.0952	0.1991	0.064*
H1B	-0.1013	0.0530	0.2556	0.064*
N2	0.15555 (13)	0.04420 (5)	0.1786 (2)	0.0364 (5)
N3	0.12698 (13)	0.00771 (5)	0.1195 (3)	0.0413 (5)
C1	-0.00707 (15)	0.08880 (6)	0.3326 (3)	0.0380 (5)
C2	0.04624 (16)	0.05620 (6)	0.4265 (3)	0.0435 (6)
H2A	0.0163	0.0307	0.4032	0.052*
H2B	0.0441	0.0609	0.5551	0.052*
C3	0.14666 (15)	0.05349 (7)	0.3680 (3)	0.0391 (5)
H3B	0.1771	0.0788	0.3924	0.047*
H3A	0.1775	0.0329	0.4379	0.047*
C4	0.2226 (2)	0.10885 (7)	0.0702 (5)	0.0622 (8)
H4B	0.2746	0.1082	0.1504	0.093*
H4C	0.1747	0.1253	0.1210	0.093*
H4A	0.2414	0.1198	-0.0434	0.093*
C5	0.18687 (15)	0.06742 (6)	0.0435 (3)	0.0422 (5)
C6	0.17798 (19)	0.04516 (8)	-0.1109 (4)	0.0505 (6)
H6A	0.1938	0.0529	-0.2270	0.061*
C7	0.14038 (16)	0.00856 (7)	-0.0582 (3)	0.0438 (6)
C8	0.1147 (2)	-0.02660 (9)	-0.1705 (5)	0.0628 (8)
H8A	0.1465	-0.0499	-0.1274	0.094*
H8B	0.1319	-0.0217	-0.2932	0.094*
H8C	0.0491	-0.0309	-0.1634	0.094*

Atomic displacement parameters (\AA^2)

U^{11} U^{22} U^{33} U^{12} U^{13} U^{23}

supplementary materials

O1	0.0548 (10)	0.0323 (7)	0.0719 (13)	-0.0018 (6)	-0.0196 (10)	-0.0002 (8)
N1	0.0488 (10)	0.0381 (9)	0.0720 (16)	-0.0038 (8)	-0.0163 (12)	0.0039 (11)
N2	0.0386 (10)	0.0338 (10)	0.0369 (10)	0.0008 (7)	-0.0009 (9)	-0.0020 (8)
N3	0.0410 (10)	0.0342 (9)	0.0486 (12)	0.0007 (7)	-0.0011 (10)	-0.0057 (8)
C1	0.0391 (11)	0.0335 (9)	0.0415 (12)	0.0025 (8)	0.0025 (10)	-0.0037 (9)
C2	0.0473 (13)	0.0385 (11)	0.0447 (13)	0.0024 (9)	0.0045 (11)	0.0049 (10)
C3	0.0419 (12)	0.0367 (10)	0.0387 (12)	0.0061 (9)	-0.0027 (11)	-0.0005 (9)
C4	0.0787 (19)	0.0442 (13)	0.0636 (18)	-0.0134 (12)	0.0057 (17)	0.0057 (13)
C5	0.0425 (11)	0.0419 (10)	0.0422 (13)	-0.0005 (9)	0.0004 (12)	0.0018 (10)
C6	0.0521 (14)	0.0604 (16)	0.0390 (12)	-0.0001 (11)	0.0008 (12)	0.0009 (11)
C7	0.0365 (11)	0.0509 (14)	0.0441 (13)	0.0051 (9)	-0.0047 (11)	-0.0113 (11)
C8	0.0568 (15)	0.0671 (16)	0.0645 (19)	0.0018 (12)	-0.0065 (15)	-0.0243 (15)

Geometric parameters (Å, °)

O1—C1	1.232 (2)	C3—H3A	0.9700
N1—C1	1.320 (3)	C4—C5	1.490 (3)
N1—H1A	0.8598	C4—H4B	0.9600
N1—H1B	0.8603	C4—H4C	0.9600
N2—C5	1.347 (3)	C4—H4A	0.9600
N2—N3	1.359 (3)	C5—C6	1.373 (4)
N2—C3	1.448 (3)	C6—C7	1.394 (4)
N3—C7	1.335 (3)	C6—H6A	0.9300
C1—C2	1.505 (3)	C7—C8	1.488 (4)
C2—C3	1.518 (3)	C8—H8A	0.9600
C2—H2A	0.9700	C8—H8B	0.9600
C2—H2B	0.9700	C8—H8C	0.9600
C3—H3B	0.9700		
C1—N1—H1A	120.2	C5—C4—H4B	109.5
C1—N1—H1B	119.8	C5—C4—H4C	109.5
H1A—N1—H1B	120.0	H4B—C4—H4C	109.5
C5—N2—N3	112.13 (18)	C5—C4—H4A	109.5
C5—N2—C3	129.18 (18)	H4B—C4—H4A	109.5
N3—N2—C3	118.66 (18)	H4C—C4—H4A	109.5
C7—N3—N2	104.88 (19)	N2—C5—C6	106.28 (19)
O1—C1—N1	122.5 (2)	N2—C5—C4	123.4 (2)
O1—C1—C2	121.3 (2)	C6—C5—C4	130.3 (2)
N1—C1—C2	116.14 (18)	C5—C6—C7	106.0 (2)
C1—C2—C3	113.56 (19)	C5—C6—H6A	127.0
C1—C2—H2A	108.9	C7—C6—H6A	127.0
C3—C2—H2A	108.9	N3—C7—C6	110.7 (2)
C1—C2—H2B	108.9	N3—C7—C8	120.2 (2)
C3—C2—H2B	108.9	C6—C7—C8	129.1 (3)
H2A—C2—H2B	107.7	C7—C8—H8A	109.5
N2—C3—C2	112.11 (19)	C7—C8—H8B	109.5
N2—C3—H3B	109.2	H8A—C8—H8B	109.5
C2—C3—H3B	109.2	C7—C8—H8C	109.5
N2—C3—H3A	109.2	H8A—C8—H8C	109.5
C2—C3—H3A	109.2	H8B—C8—H8C	109.5

H3B—C3—H3A

107.9

Hydrogen-bond geometry (\AA , $^\circ$)

<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>
N1—H1A \cdots O1 ⁱ	0.86	2.10	2.936 (3)	164
N1—H1B \cdots N3 ⁱⁱ	0.86	2.30	3.084 (3)	152
C3—H3B \cdots O1 ⁱⁱⁱ	0.97	2.52	3.413 (3)	154

Symmetry codes: (i) $x-1/4, -y+1/4, z-1/4$; (ii) $-x, -y, z$; (iii) $x+1/4, -y+1/4, z+1/4$.

Fig. 1

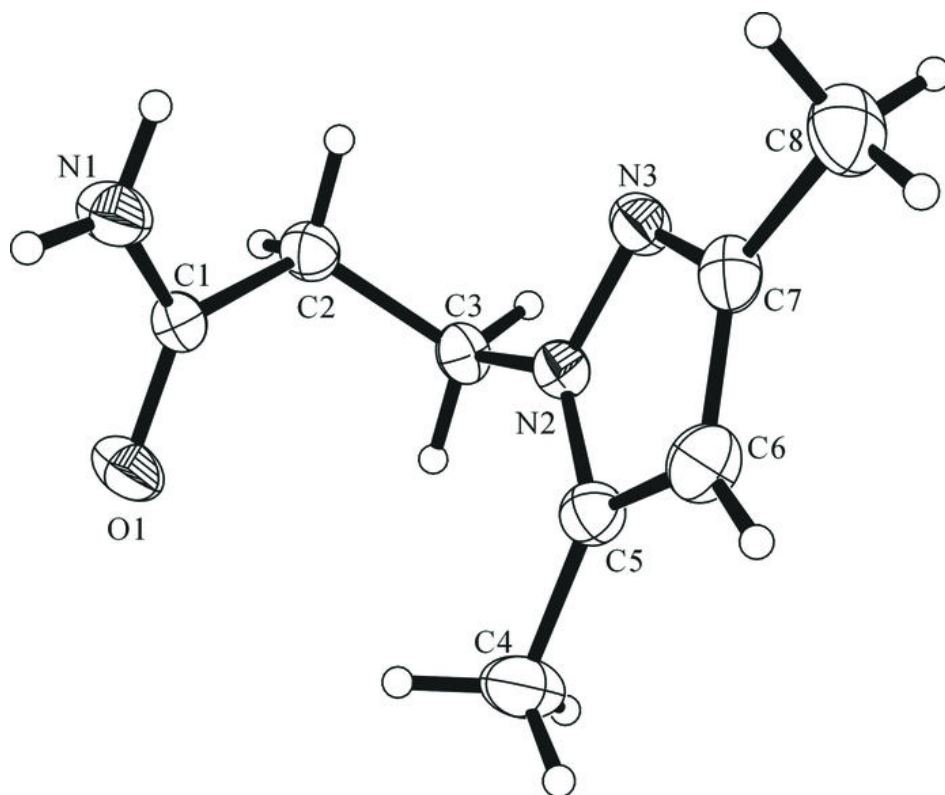


Fig. 2

