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

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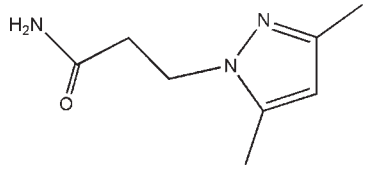
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.033; wR factor = 0.093; data-to-parameter ratio = 9.5.

In the crystal of the title compound, $C_8H_{13}N_3O$, molecules are linked by intermolecular N—H···N and N—H···O hydrogen bonds into a three-dimensional network. Additional stabilization is provided by weak intermolecular C—H···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

$C_8H_{13}N_3O$
M_r = 167.21
Orthorhombic, *Fdd*2
a = 14.452 (5) Å
b = 33.390 (7) Å
c = 7.4354 (15) Å
V = 3588.0 (16) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.47 × 0.37 × 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_{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···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.10	2.936 (3)	164
N1—H1B···N3^{ii}	0.86	2.30	3.084 (3)	152
C3—H3B···O1^{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/MSC, 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*.

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

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supporting information

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

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S1. 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 molecluar 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.

S2. 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 $C_8H_{13}N_3O$: C 57.42, H 7.78, N 25.12%; found: C 57.26, H 7.59, N 25.18%.

S3. 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_{iso}(H) = 1.2 U_{eq}(C,*N*) or 1.5 U_{eq}(C_{methyl}). In the absense of significant anomalous dispersion effects Friedel pairs were merged.

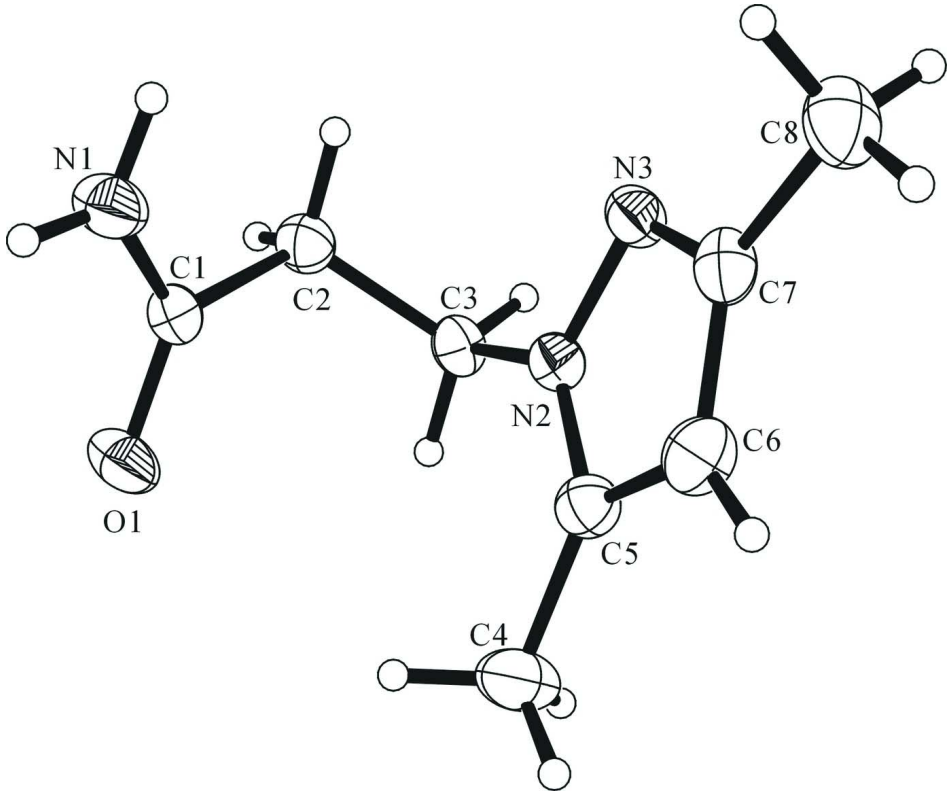

Figure 1

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

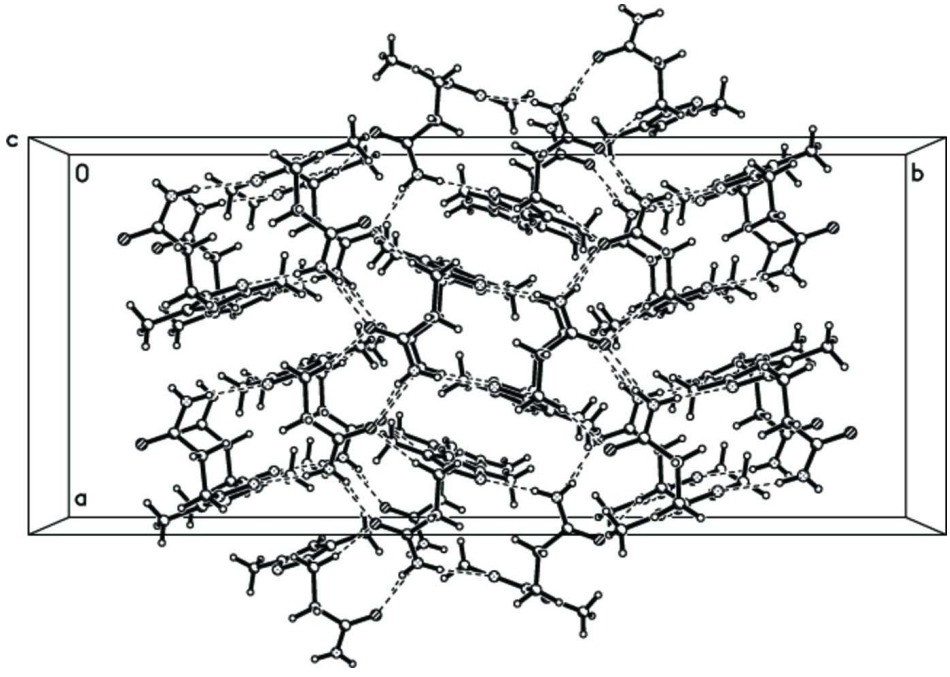

Figure 2

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

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

Crystal data

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

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.963$, $T_{max} = 0.970$
4623 measured reflections
1067 independent reflections
890 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -17\rightarrow18$
$k = -43\rightarrow43$
$l = -9\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.093$
$S = 1.14$
1067 reflections
112 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 1.8694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXTL'*(Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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, º)

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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.86	2.10	2.936 (3)	164
N1—H1*B*···N3^{ii}	0.86	2.30	3.084 (3)	152
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Symmetry codes: (i) $x-1/4, -y+1/4, z-1/4$; (ii) $-x, -y, z$; (iii) $x+1/4, -y+1/4, z+1/4$.