

2-Cyano-2-methylpropanamide

 Jia-Ying Xu^{a*} and Wei-Hua Cheng^b
^aCollege of Chemical and Biological Engineering, Yancheng Institute of Technology, Yinbing Road No. 9 Yancheng, Yancheng 224051, People's Republic of China, and

^bDepartment of Chemical Engineering, Yancheng College of Textile Technology, People's Republic of China

Correspondence e-mail: xujiaying-1984@163.com

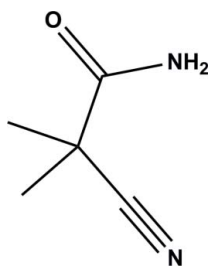
Received 21 March 2012; accepted 27 March 2012

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.050; wR factor = 0.143; data-to-parameter ratio = 12.8.

In the crystal structure of the title compound, $\text{C}_5\text{H}_8\text{N}_2\text{O}$, molecules are linked *via* pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming inversion dimers. These dimers are linked *via* pairs of $\text{N}-\text{H}\cdots\text{H}$ hydrogen bonds into zigzag chains propagating along [101].

Related literature

For the synthesis of the title compound, see: Zhang *et al.* (2011). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_5\text{H}_8\text{N}_2\text{O}$
 $M_r = 112.13$

 Triclinic, $P\bar{1}$
 $a = 5.8916$ (12) Å

 $b = 6.4349$ (14) Å

 $c = 9.1263$ (19) Å

 $\alpha = 95.659$ (4)°

 $\beta = 102.379$ (4)°

 $\gamma = 109.859$ (4)°

 $V = 312.27$ (11) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 293$ K

 $0.20 \times 0.18 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

 Absorption correction: ψ scan

 (North *et al.*, 1968)

 $T_{\min} = 0.983$, $T_{\max} = 0.987$

1699 measured reflections

1077 independent reflections

 1000 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

3 standard reflections every 200 reflections

intensity decay: 1%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.143$
 $S = 1.05$

1077 reflections

84 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{i}}$	0.92 (2)	2.07 (2)	2.9714 (18)	168.2 (18)
$\text{N1}-\text{H1B}\cdots\text{N2}^{\text{ii}}$	0.874 (18)	2.328 (18)	3.166 (2)	160.8 (19)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXTL*.

The authors thank the Center of Testing and Analysis, Nanjing University, for the data collection. They also thank the Foundation of Yancheng Institute of Technology (XKR2010055) and the National Natural Science Foundation of China (No. 31000142) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2395).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Enraf–Nonius (1985). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Zhang, W. L., Wang, P. & Gan, L. X. (2011). Patent Pub. No. WO/2011/091677; Int. Appl. No. PCT/CN2010/078549.

supplementary materials

Acta Cryst. (2012). E68, o1250 [doi:10.1107/S1600536812013360]

2-Cyano-2-methylpropanamide

Jia-Ying Xu and Wei-Hua Cheng

Comment

The title compound has attracted considerable attention in drug research because of its outstanding biological activity. In recent years it has been used as an intermediate in the synthesis of the high blood pressure rennin inhibitor, Aliskiren (Zhang *et al.*, 2011).

The molecular structure of the title compound is shown in Fig. 1. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges.

In the crystal, molecules are connected *via* pairs of N—H···O hydrogen bonds to form inversion dimers (Table 1 and Fig. 2). These dimers are connected *via* pairs of N—H···N hydrogen bonds resulting in the formation of zigzag chains (Table 1 and Fig. 2), propagating along direction [101].

Experimental

The title compound was prepared by the literature procedure (Zhang *et al.*, 2011). To a solution of methyl 2-cyano-2-methylpropanoate (5 g, 39.3 mmol) in methanol (20 ml), ammonia was added slowly at room temperature. After being stirred for 18 h at the room temperature, a yellow solid was obtained. It was dissolved in ethanol and colourless block-like crystals of the title compound, suitable for X-ray diffraction analysis, were obtained by slow evaporation of the solvent over 7 days.

Refinement

The NH₂ H atoms were located in a difference electron density map and refined freely. The methyl H atoms were positioned geometrically and constrained to ride on their parent atoms: C—H = 0.96 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

Computing details

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software* (Enraf–Nonius, 1985); data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXTL* (Sheldrick, 2008).

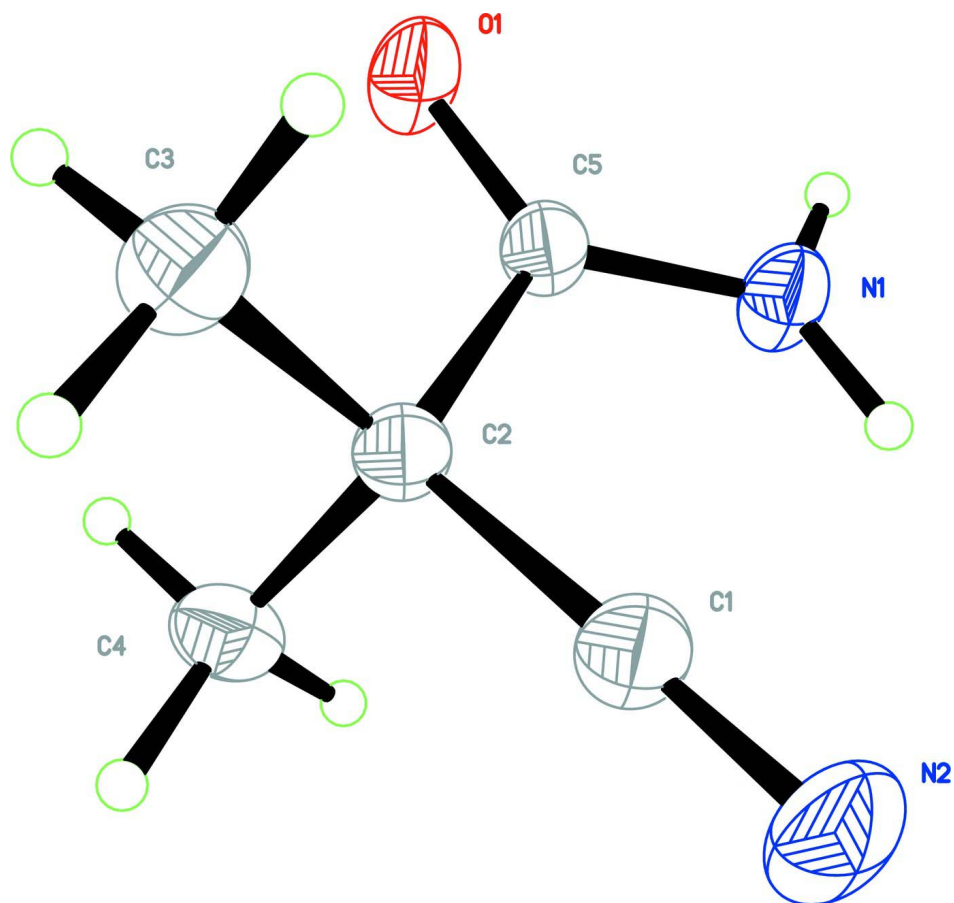


Figure 1

The molecular structure of the title molecule, with atom-numbering. Displacement ellipsoids are drawn at the 35% probability level.

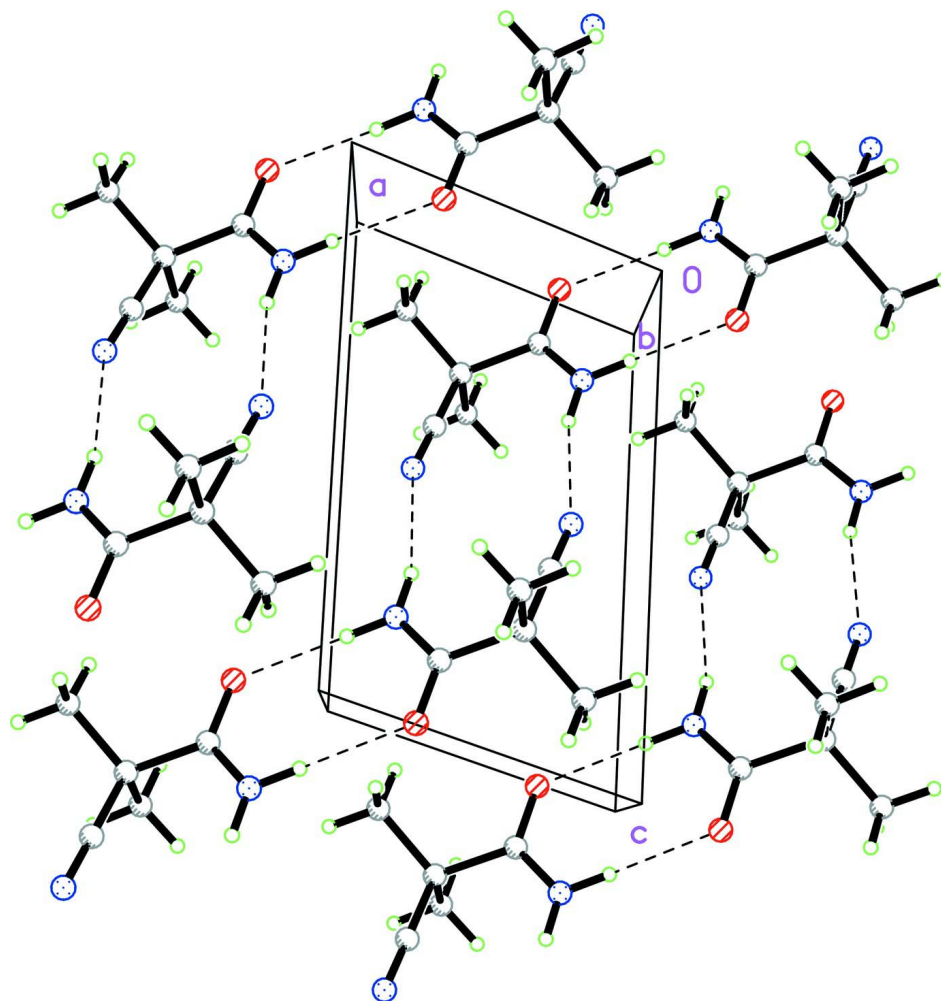


Figure 2

A view along the *b* axis of the crystal packing of the title compound. The N—H···O and N—H···N hydrogen bonds are shown as dashed lines (see Table 1 for details).

2-Cyano-2-methylpropanamide

Crystal data

$C_5H_8N_2O$

$M_r = 112.13$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 5.8916$ (12) Å

$b = 6.4349$ (14) Å

$c = 9.1263$ (19) Å

$\alpha = 95.659$ (4)°

$\beta = 102.379$ (4)°

$\gamma = 109.859$ (4)°

$V = 312.27$ (11) Å³

$Z = 2$

$F(000) = 120$

$D_x = 1.193$ Mg m⁻³

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

Cell parameters from 1603 reflections

$\theta = 2.3$ – 30.1 °

$\mu = 0.09$ mm⁻¹

$T = 293$ K

Block, colourless

$0.20 \times 0.18 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1077 independent reflections 1000 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.021$
Graphite monochromator	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.3^\circ$
$\omega/2\theta$ scans	$h = -6 \rightarrow 6$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$k = -6 \rightarrow 7$
$T_{\text{min}} = 0.983$, $T_{\text{max}} = 0.987$	$l = -10 \rightarrow 7$
1699 measured reflections	3 standard reflections every 200 reflections intensity decay: 1%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.1061P)^2 + 0.0265P]$
$wR(F^2) = 0.143$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1077 reflections	$\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{\AA}^{-3}$
84 parameters	$\Delta\rho_{\text{min}} = -0.26 \text{ e } \text{\AA}^{-3}$
0 restraints	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 2.05 (18)
Secondary atom site location: difference Fourier map	

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.29396 (18)	0.69885 (17)	0.01288 (11)	0.0555 (4)
N1	0.2126 (2)	0.51350 (19)	0.20281 (15)	0.0495 (5)
N2	0.7598 (2)	0.6725 (2)	0.48798 (16)	0.0644 (5)
C1	0.6955 (2)	0.7399 (2)	0.38232 (15)	0.0448 (5)
C2	0.6156 (2)	0.83173 (18)	0.24687 (13)	0.0364 (4)
C3	0.8132 (2)	0.8719 (2)	0.15614 (16)	0.0479 (5)
H3A	0.9726	0.9721	0.2208	0.072*
H3B	0.7658	0.9376	0.0703	0.072*
H3C	0.8246	0.7312	0.1206	0.072*
C4	0.5862 (3)	1.0547 (2)	0.29886 (16)	0.0494 (5)
H4A	0.4580	1.0266	0.3521	0.074*
H4B	0.5407	1.1176	0.2112	0.074*
H4C	0.7418	1.1586	0.3656	0.074*
C5	0.3573 (2)	0.66995 (19)	0.14359 (14)	0.0381 (4)

H1A	0.059 (4)	0.429 (3)	0.138 (2)	0.069 (5)*
H1B	0.254 (4)	0.491 (3)	0.296 (2)	0.064 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0468 (7)	0.0567 (7)	0.0374 (6)	-0.0025 (5)	-0.0094 (4)	0.0185 (5)
N1	0.0391 (7)	0.0511 (8)	0.0394 (7)	0.0004 (5)	-0.0051 (5)	0.0174 (5)
N2	0.0506 (8)	0.0740 (9)	0.0501 (8)	0.0094 (6)	-0.0086 (6)	0.0274 (7)
C1	0.0344 (7)	0.0472 (7)	0.0392 (8)	0.0058 (5)	-0.0033 (5)	0.0101 (6)
C2	0.0332 (7)	0.0376 (7)	0.0308 (7)	0.0087 (5)	-0.0002 (5)	0.0073 (5)
C3	0.0390 (7)	0.0570 (8)	0.0438 (8)	0.0150 (6)	0.0075 (6)	0.0102 (6)
C4	0.0487 (8)	0.0470 (8)	0.0447 (8)	0.0170 (6)	0.0013 (6)	0.0001 (6)
C5	0.0360 (7)	0.0366 (7)	0.0328 (7)	0.0086 (5)	-0.0018 (5)	0.0087 (5)

Geometric parameters (\AA , $^\circ$)

O1—C5	1.2234 (16)	C2—C5	1.5504 (15)
N1—C5	1.3243 (17)	C3—H3A	0.9600
N1—H1A	0.92 (2)	C3—H3B	0.9600
N1—H1B	0.88 (2)	C3—H3C	0.9600
N2—C1	1.1395 (18)	C4—H4A	0.9600
C1—C2	1.4774 (17)	C4—H4B	0.9600
C2—C3	1.5356 (18)	C4—H4C	0.9600
C2—C4	1.5438 (18)		
C5—N1—H1A	114.6 (12)	C2—C3—H3C	109.5
C5—N1—H1B	124.9 (12)	H3A—C3—H3C	109.5
H1A—N1—H1B	120.5 (18)	H3B—C3—H3C	109.5
N2—C1—C2	178.86 (14)	C2—C4—H4A	109.5
C1—C2—C3	109.07 (10)	C2—C4—H4B	109.5
C1—C2—C4	109.37 (10)	H4A—C4—H4B	109.5
C3—C2—C4	110.22 (10)	C2—C4—H4C	109.5
C1—C2—C5	111.37 (9)	H4A—C4—H4C	109.5
C3—C2—C5	109.91 (10)	H4B—C4—H4C	109.5
C4—C2—C5	106.88 (10)	O1—C5—N1	123.35 (11)
C2—C3—H3A	109.5	O1—C5—C2	118.09 (10)
C2—C3—H3B	109.5	N1—C5—C2	118.50 (10)
H3A—C3—H3B	109.5		
N2—C1—C2—C3	78 (8)	C4—C2—C5—O1	76.58 (15)
N2—C1—C2—C4	-42 (8)	C1—C2—C5—N1	18.55 (16)
N2—C1—C2—C5	-160 (8)	C3—C2—C5—N1	139.54 (12)
C1—C2—C5—O1	-164.02 (12)	C4—C2—C5—N1	-100.85 (14)
C3—C2—C5—O1	-43.03 (15)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O1 ⁱ	0.92 (2)	2.07 (2)	2.9714 (18)	168.2 (18)

N1—H1B···N2 ⁱⁱ	0.874 (18)	2.328 (18)	3.166 (2)	160.8 (19)
---------------------------	------------	------------	-----------	------------

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