Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*S*)-(+)-2-Formamido-4-methylpentanoic acid

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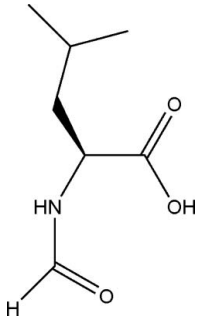
Received 25 May 2007; accepted 3 July 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.086; data-to-parameter ratio = 10.0.

The title compound, $C_7H_{13}NO_3$, was synthesized from (*S*)-(+)-leucine. The stereochemistry is assumed to be unchanged by the reaction and only a single enantiomer is present in the structure. The asymmetric unit consists of a single molecule. The crystal structure displays N—H···O and O—H···O intermolecular hydrogen bonding.

Related literature

For related literature, see: Boyle *et al.* (2004).



Experimental

Crystal data

$C_7H_{13}NO_3$
M_r = 159.18
Orthorhombic, $P2_12_12_1$
a = 9.6041 (7) Å
b = 10.0758 (7) Å
c = 9.2936 (6) Å
V = 899.33 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.36 × 0.25 × 0.24 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
10153 measured reflections
1044 independent reflections
970 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.04
1044 reflections
104 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.00	2.8527 (18)	171
O1—H1A···O3^{ii}	0.82	1.76	2.5729 (18)	175

Symmetry codes: (i) $-x+\frac{3}{2}, -y+2, z+\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Mercury* (Bruno *et al.*, 2002); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank Dr Manuel Fernandes of the Jan Boeyens Structural Chemistry Laboratory at the University of the Witwatersrand for his assistance in the acquisition of the crystallographic data. This work was supported by grants from the National Research Foundation of South Africa (GUN 2046819) and the University of KwaZulu-Natal.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2086).

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 doi:10.1107/S1600536807032503

supplementary materials

Acta Cryst. (2007). E**63**, o3445 [**doi:10.1107/S1600536807032503**]

(*S*)-(+)-2-Formamido-4-methylpentanoic acid

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Comment

The title compound (**I**) was synthesized as an intermediate in the synthesis of a chiral secondary alcohol. This chiral alcohol was in turn used as a chiral source in ligands used for the asymmetric addition of diethylzinc to benzaldehyde (Boyle *et al.*, 2004).

The title compound was synthesized from commercial (S)-(+)-leucine and the stereochemistry is retained during the reaction. The structure therefore contains only a single enantiomer. The unit cell contains a single molecule. C6 and N1 are equatorial to the plane through C1 and C5 along the backbone of the molecule. Fig. 1 illustrates the geometry of the molecule and the labelling scheme employed. Each molecule exhibits intermolecular hydrogen bonding (N1—H1···O2^{i} and O1—H1A···O3^{ii}), resulting in a layered structure with O—H···O hydrogen bonding interactions between molecules within the layer and N—H···O interactions between layers. The molecular packing and hydrogen bonding interactions are shown in Fig. 2.

Experimental

Acetic anhydride (30 mol equivalents) was added dropwise to a stirred solution of (*S*)-(+)-Leucine (1 mol equivalent) in formic acid (approximately 30 ml per 1.0 g of amino acid) at 0°C. After addition of the acetic anhydride, the external ice bath was removed and the solution stirred at room temperature for 24 h. The solution was treated with water (60 ml) and stirred for 1 hr. The solvent was removed under reduced pressure to yield a white residue. This residue was recrystallized from water to yield the pure product (**I**) (Boyle *et al.*, 2004). Yield 72%. Colorless crystals suitable for X-ray diffraction were obtained by evaporation of water at room temperature in a fume hood over a period of 2 days.

Refinement

Hydrogen atoms were first located in the difference map then positioned geometrically and allowed to ride on their respective parent atoms, with C—H = 0.93 or 0.97 Å, N—H = 0.86 Å and O—H = 0.82 Å. U_{iso}(H) values were set equal to 1.5U_{eq} for methyl C and O and 1.2U_{eq} for the remainder.

Figures

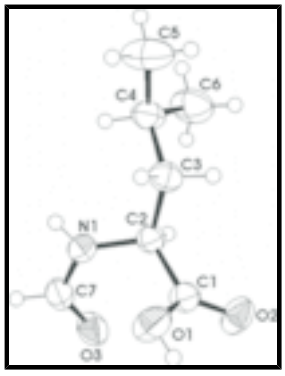

Fig. 1. The asymmetric unit of (**I**) showing atomic numbering scheme and ellipsoids at the 50% probability level.

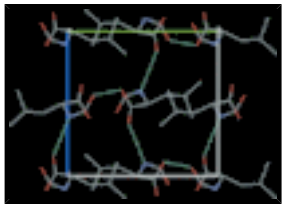

Fig. 2. The molecular packing, viewed down the *a* axis, showing layered structure and hydrogen bonding (dashed lines). H atoms have been omitted.

(*S*)-(+)-2-Formamido-4-methylpentanoic acid

Crystal data

$C_7H_{13}NO_3$	F_{000} = 344
M_r = 159.18	D_x = 1.176 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 5368 reflections
a = 9.6041 (7) Å	θ = 2.9–27.9º
b = 10.0758 (7) Å	μ = 0.09 mm^{-1}
c = 9.2936 (6) Å	*T* = 293 (2) K
V = 899.33 (11) Å^3	Block, colourless
Z = 4	0.36 × 0.25 × 0.24 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	970 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.052
Monochromator: graphite	θ_{max} = 26.0º
T = 293(2) K	θ_{min} = 2.9º
φ and ω scans	*h* = −11→11
Absorption correction: none	*k* = −12→12
10153 measured reflections	*l* = −11→11
1044 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.0822P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$(\Delta/\sigma)_{max} = 0.005$
$wR(F^2) = 0.086$	$\Delta\rho_{max} = 0.12$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
1044 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
104 parameters	Extinction coefficient: 0.029 (5)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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 $F^2 > 2$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}
C1	0.86041 (18)	0.92357 (15)	−0.02094 (17)	0.0387 (4)
C2	0.75039 (17)	1.03083 (14)	−0.00238 (17)	0.0359 (4)
H2	0.7124	1.0530	−0.0972	0.043*
C3	0.81824 (17)	1.15467 (17)	0.0609 (2)	0.0425 (4)
H3A	0.8415	1.1372	0.1607	0.051*
H3B	0.9046	1.1713	0.0099	0.051*
C4	0.7290 (2)	1.27972 (16)	0.0541 (2)	0.0470 (4)
H4	0.6357	1.2573	0.0891	0.056*
C5	0.7892 (3)	1.3853 (2)	0.1529 (3)	0.0833 (8)
H5A	0.7370	1.4660	0.1425	0.125*
H5B	0.8847	1.4012	0.1277	0.125*
H5C	0.7840	1.3554	0.2508	0.125*
C6	0.7153 (3)	1.3315 (2)	−0.0977 (2)	0.0714 (7)
H6A	0.6658	1.2680	−0.1552	0.107*
H6B	0.8062	1.3457	−0.1376	0.107*

H6C	0.6650	1.4139	−0.0966	0.107*
C7	0.55756 (19)	0.88674 (18)	0.0536 (2)	0.0473 (4)
H7	0.4891	0.8601	0.1182	0.057*
N1	0.63713 (14)	0.98712 (13)	0.08936 (14)	0.0400 (3)
H1	0.6219	1.0280	0.1690	0.048*
O1	0.88610 (16)	0.85843 (14)	0.09708 (13)	0.0571 (4)
H1A	0.9464	0.8025	0.0817	0.086*
O2	0.92108 (15)	0.90574 (14)	−0.13295 (13)	0.0551 (4)
O3	0.56841 (16)	0.82565 (13)	−0.06075 (14)	0.0594 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0453 (9)	0.0377 (8)	0.0332 (8)	0.0031 (7)	−0.0015 (7)	0.0019 (6)
C2	0.0408 (8)	0.0339 (8)	0.0331 (8)	0.0015 (7)	0.0003 (7)	0.0010 (6)
C3	0.0418 (8)	0.0386 (8)	0.0470 (9)	−0.0026 (7)	−0.0060 (8)	−0.0008 (8)
C4	0.0540 (10)	0.0344 (8)	0.0525 (10)	−0.0010 (8)	0.0002 (9)	0.0002 (7)
C5	0.118 (2)	0.0465 (11)	0.0858 (16)	0.0005 (13)	−0.0184 (17)	−0.0188 (12)
C6	0.0996 (17)	0.0487 (11)	0.0657 (14)	0.0070 (12)	−0.0080 (13)	0.0137 (10)
C7	0.0509 (10)	0.0458 (9)	0.0450 (9)	−0.0090 (8)	0.0084 (8)	0.0010 (8)
N1	0.0457 (7)	0.0373 (7)	0.0368 (7)	−0.0012 (6)	0.0055 (6)	−0.0052 (6)
O1	0.0795 (9)	0.0556 (8)	0.0361 (6)	0.0296 (7)	0.0052 (6)	0.0085 (6)
O2	0.0637 (8)	0.0651 (8)	0.0364 (6)	0.0222 (7)	0.0098 (6)	0.0078 (6)
O3	0.0778 (9)	0.0524 (7)	0.0481 (7)	−0.0269 (7)	0.0121 (7)	−0.0115 (6)

Geometric parameters (Å, °)

C1—O2	1.206 (2)	C5—H5A	0.9600
C1—O1	1.3018 (19)	C5—H5B	0.9600
C1—C2	1.521 (2)	C5—H5C	0.9600
C2—N1	1.451 (2)	C6—H6A	0.9600
C2—C3	1.526 (2)	C6—H6B	0.9600
C2—H2	0.9800	C6—H6C	0.9600
C3—C4	1.525 (2)	C7—O3	1.233 (2)
C3—H3A	0.9700	C7—N1	1.310 (2)
C3—H3B	0.9700	C7—H7	0.9300
C4—C6	1.510 (3)	N1—H1	0.8600
C4—C5	1.519 (3)	O1—H1A	0.8200
C4—H4	0.9800		
O2—C1—O1	124.09 (15)	C3—C4—H4	108.0
O2—C1—C2	122.62 (14)	C4—C5—H5A	109.5
O1—C1—C2	113.22 (14)	C4—C5—H5B	109.5
N1—C2—C1	111.83 (13)	H5A—C5—H5B	109.5
N1—C2—C3	110.01 (13)	C4—C5—H5C	109.5
C1—C2—C3	109.15 (14)	H5A—C5—H5C	109.5
N1—C2—H2	108.6	H5B—C5—H5C	109.5
C1—C2—H2	108.6	C4—C6—H6A	109.5
C3—C2—H2	108.6	C4—C6—H6B	109.5

C4—C3—C2	114.83 (13)	H6A—C6—H6B	109.5
C4—C3—H3A	108.6	C4—C6—H6C	109.5
C2—C3—H3A	108.6	H6A—C6—H6C	109.5
C4—C3—H3B	108.6	H6B—C6—H6C	109.5
C2—C3—H3B	108.6	O3—C7—N1	123.68 (17)
H3A—C3—H3B	107.5	O3—C7—H7	118.2
C6—C4—C5	110.82 (18)	N1—C7—H7	118.2
C6—C4—C3	111.91 (16)	C7—N1—C2	121.51 (14)
C5—C4—C3	109.87 (17)	C7—N1—H1	119.2
C6—C4—H4	108.0	C2—N1—H1	119.2
C5—C4—H4	108.0	C1—O1—H1A	109.5
O2—C1—C2—N1	142.92 (17)	C2—C3—C4—C6	−71.2 (2)
O1—C1—C2—N1	−40.1 (2)	C2—C3—C4—C5	165.23 (18)
O2—C1—C2—C3	−95.1 (2)	O3—C7—N1—C2	−2.9 (3)
O1—C1—C2—C3	81.89 (18)	C1—C2—N1—C7	−62.4 (2)
N1—C2—C3—C4	−70.11 (19)	C3—C2—N1—C7	176.11 (15)
C1—C2—C3—C4	166.83 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.00	2.8527 (18)	171
O1—H1A···O3^{ii}	0.82	1.76	2.5729 (18)	175

Symmetry codes: (i) −*x*+3/2, −*y*+2, *z*+1/2; (ii) *x*+1/2, −*y*+3/2, −*z*.

Fig. 1

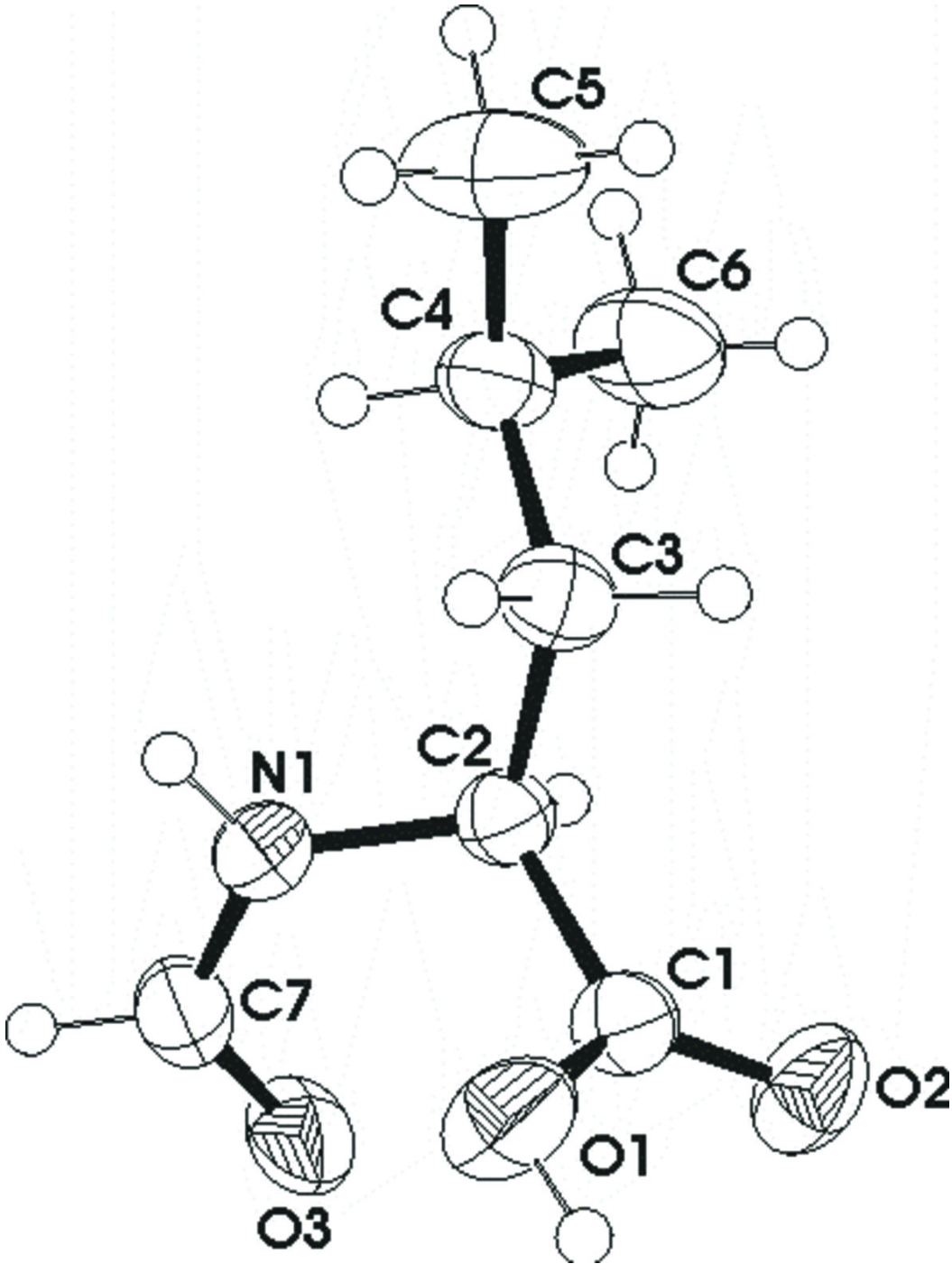

Fig. 2