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Methyl (*E*)-3-acetamido-4-methyl-2-pentenoate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.064
wR factor = 0.198
Data-to-parameter ratio = 19.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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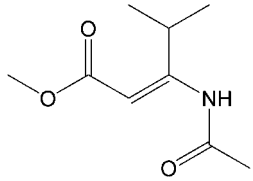
The molecule of the title compound, $C_9H_{15}NO_3$, is an *E* isomer and the crystal structure involves an N—H···O hydrogen bond, forming one-dimensional chains along the *a* axis.

Comment

The title compound, (I), is one of the prochiral olefins widely used in the asymmetric hydrogenation reaction (Hackler & Wickiser, 1985; Lubell *et al.*, 1991) for producing β-amino acids and their derivatives. The enantiomeric excess of the hydrogenation product is significantly influenced by the structure of the substrate. The structure determination of (I) was conducted in order to obtain more stereochemical information about β-amino acids and their derivatives.



(I)

In the structure of (I) (Fig. 1), the bond lengths and angles are in the expected ranges (Table 1). As shown in the packing diagram (Fig. 2), the crystal structure contains intermolecular hydrogen bonds, and infinite molecular chains stretch along the *a* axis.

Experimental

The title compound was synthesized according to Zhu *et al.* (1999). A crystal suitable for X-ray analysis was grown slowly in a mixture of

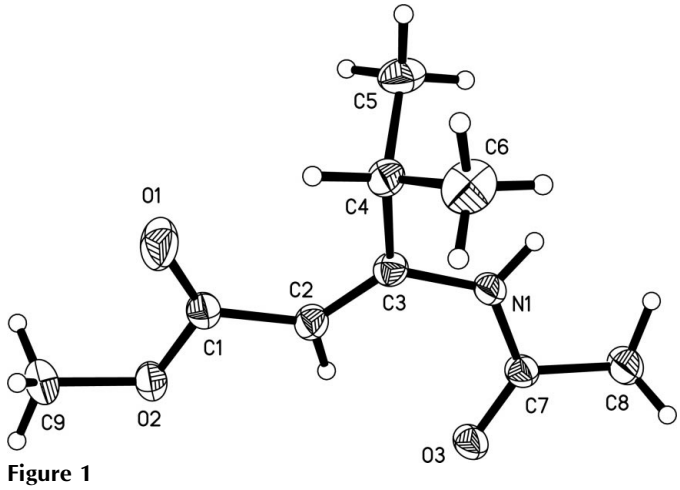


Figure 1
The molecular structure of (I), showing ellipsoids at the 50% probability level (Siemens, 1995).

ethyl acetate and hexane at room temperature. ^{1}H NMR (400 MHz, acetone-d_6, Bruker): δ 1.13 (*d*, J = 7.1 Hz, 6H), 2.09 (*s*, 3H), 3.59 (*s*, 3H), 4.34 (*sep*, J = 7.1 Hz, 1H), 6.99 (*s*, 1H), 8.18 (*br*, 1H).

Crystal data

$C_9H_{15}NO_3$
M_r = 185.22
Orthorhombic, *Pbca*
a = 9.806 (2) Å
b = 12.435 (3) Å
c = 17.067 (4) Å
V = 2081.1 (9) Å^3
Z = 8
D_x = 1.182 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2463 reflections
θ = 1–27.5°
μ = 0.09 mm^{-1}
T = 294 (2) K
Needle, colorless
0.28 × 0.10 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.976, T_{max} = 0.991
13157 measured reflections
2384 independent reflections
888 reflections with $I > 2\sigma(I)$
R_{int} = 0.116
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 16$
$l = -18 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.198
S = 1.02
2384 reflections
123 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.024 (5)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.209 (4)	N1—C7	1.368 (4)
O2—C1	1.333 (4)	N1—C3	1.410 (4)
O2—C9	1.437 (4)	C1—C2	1.449 (4)
O3—C7	1.216 (3)	C4—C6	1.517 (5)
C1—O2—C9	118.0 (3)	O1—C1—O2	121.0 (3)
C7—N1—C3	128.2 (3)	O1—C1—C2	128.4 (3)
C9—O2—C1—O1	−1.6 (5)	C7—N1—C3—C4	163.4 (3)
C9—O2—C1—C2	179.7 (3)	N1—C3—C4—C5	73.2 (3)
C1—C2—C3—N1	−179.7 (3)	C3—N1—C7—C8	178.7 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.28	3.124 (3)	166

Symmetry code: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$.

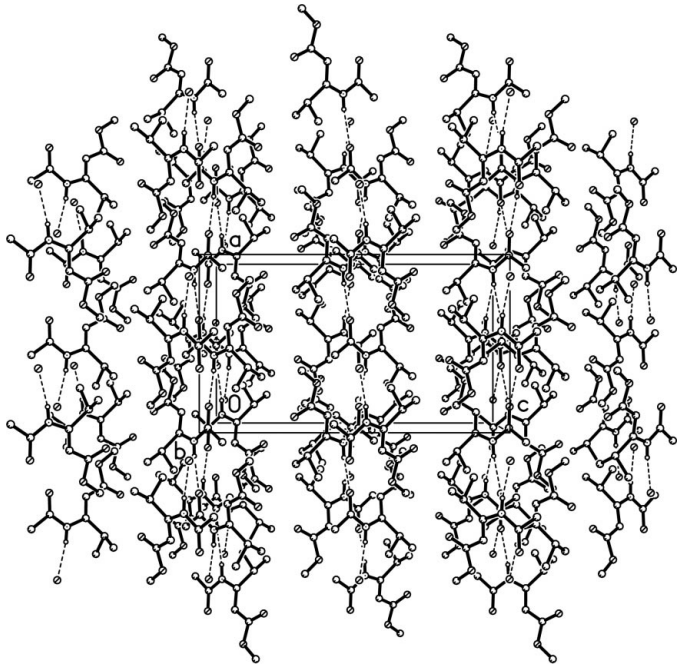


Figure 2
Packing diagram for (I). Hydrogen bonds are indicated by dashed lines.

H atoms were positioned geometrically and refined in the riding-model approximation, with U_{iso} values equal to U_{eq} of the atom to which they are bound.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995) and *SHELXTL* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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