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Key indicators

Single-crystal X-ray study

$T = 294\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

R factor = 0.059

wR factor = 0.155

Data-to-parameter ratio = 19.3

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

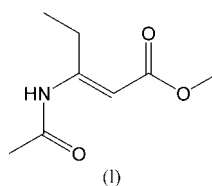
Methyl (*E*)-3-acetamido-2-pentenoate

The title compound, $\text{C}_8\text{H}_{13}\text{NO}_3$, is an *E* isomer and there are two molecules in the asymmetric unit. The molecules are assembled into chains, along the *a* axis, via intermolecular interactions.

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Comment

The title compound, (I), is one of the products obtained from reaction of methyl 3-amine-2-pentenoate with acetic anhydride under reflux for 24 h. This prochiral olefin is a model substrate studied in the asymmetric hydrogenation reaction (Hackler & Wickiser, 1985; Lubell *et al.*, 1991).



The structure determination of (I) was conducted in order to obtain more stereochemical information about the behaviour of these kinds of substrates in hydrogenation reactions. The crystal structure of (I) contains two independent molecules in the asymmetric unit (Fig. 1). A pairwise comparison between these two molecules shows no significant differences in their bond lengths or angles, although the conformations of the two molecules are different. The $\text{C}1-\text{C}2$ bond distance of $1.338(3)\text{ \AA}$ is indicative of double-bond character. The angles $\text{C}1-\text{C}2-\text{C}3$ [$125.1(2)^\circ$] and $\text{C}2-\text{C}1-\text{C}5$ [$124.8(2)^\circ$] are larger, and $\text{N}1-\text{C}1-\text{C}5$ [$112.0(2)^\circ$] smaller than 120° . This results in a close mutual repulsion between the ethyl group on $\text{C}1$ and carbonyl group on $\text{C}3$.

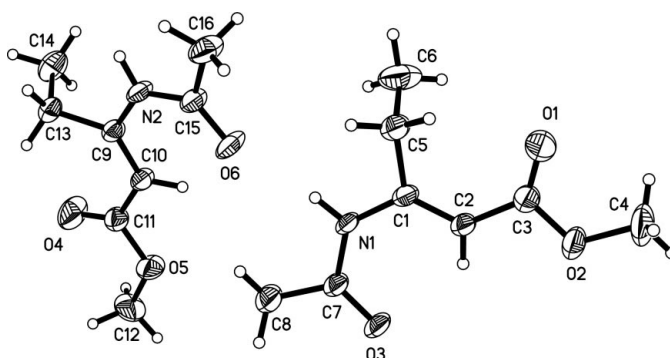


Figure 1

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

The molecules are interconnected by N—H···O hydrogen bonding in the crystal (Table 2). As illustrated in Fig. 2, the hydrogen bonding links the molecules along the *a* axis.

Experimental

The title compound was synthesized according to the literature (Zhu *et al.*, 1999). A crystal suitable for X-ray analysis was slowly grown in a mixed solvent of ethyl acetate and hexane at room temperature. ¹H NMR (400 MHz, acetone-*d*₆, Bruker): δ 1.09–1.12 (*t*, *J* = 7.5 Hz, 3H), 2.06 (*s*, 3H), 2.71–2.77 (*q*, *J* = 7.5 Hz, 2H), 3.59 (*s*, 3H), 6.87 (*s*, 1H), 8.75 (*br*, 1H).

Crystal data

C ₈ H ₁₃ NO ₃	<i>D</i> _x = 1.220 Mg m ⁻³
<i>M</i> _r = 171.19	Mo <i>K</i> α radiation
Monoclinic, <i>P</i> 2 ₁ / <i>n</i>	Cell parameters from 2920 reflections
<i>a</i> = 9.718 (7) Å	<i>θ</i> = 1–27.5°
<i>b</i> = 12.673 (9) Å	<i>μ</i> = 0.09 mm ⁻¹
<i>c</i> = 15.653 (7) Å	<i>T</i> = 294 (2) K
<i>β</i> = 104.825 (15)°	Needle, colorless
<i>V</i> = 1864 (2) Å ³	0.38 × 0.12 × 0.10 mm
<i>Z</i> = 8	

Data collection

Siemens SMART CCD area-detector diffractometer	4308 independent reflections
<i>φ</i> and <i>ω</i> scans	1544 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	<i>R</i> _{int} = 0.057
<i>T</i> _{min} = 0.965, <i>T</i> _{max} = 0.991	<i>θ</i> _{max} = 27.6°
12544 measured reflections	<i>h</i> = -12 → 12
	<i>k</i> = -15 → 16
	<i>l</i> = -16 → 20

Refinement

Refinement on <i>F</i> ²	H-atom parameters constrained
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.059	<i>w</i> = 1/[σ ² (<i>F</i> _o ²) + (0.05 <i>P</i>) ²]
<i>wR</i> (<i>F</i> ²) = 0.156	where <i>P</i> = (<i>F</i> _o ² + 2 <i>F</i> _c ²)/3
<i>S</i> = 1.06	(Δ/σ) _{max} < 0.001
4308 reflections	Δρ _{max} = 0.17 e Å ⁻³
223 parameters	Δρ _{min} = -0.19 e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

O1—C3	1.190 (3)	O4—C11	1.198 (3)
O3—C7	1.219 (3)	O6—C15	1.222 (3)
N1—C7	1.355 (3)	N2—C15	1.356 (3)
N1—C1	1.391 (3)	N2—C9	1.388 (3)
C1—C2	1.338 (3)	C9—C10	1.334 (3)
C2—C3	1.445 (4)	C10—C11	1.443 (3)
C2—C1—C5	124.8 (2)	O1—C3—C2	129.4 (3)
N1—C1—C5	112.0 (2)	O2—C3—C2	109.7 (2)
C1—C2—C3	125.1 (2)	O3—C7—N1	123.0 (2)
O1—C3—O2	120.9 (3)	O3—C7—C8	121.7 (2)
N1—C1—C2—C3	-176.8 (3)	C4—O2—C3—C2	-179.4 (2)
C5—C1—C2—C3	2.6 (4)	C1—N1—C7—O3	-4.0 (5)
C4—O2—C3—O1	0.3 (4)	C1—N1—C7—C8	175.5 (3)

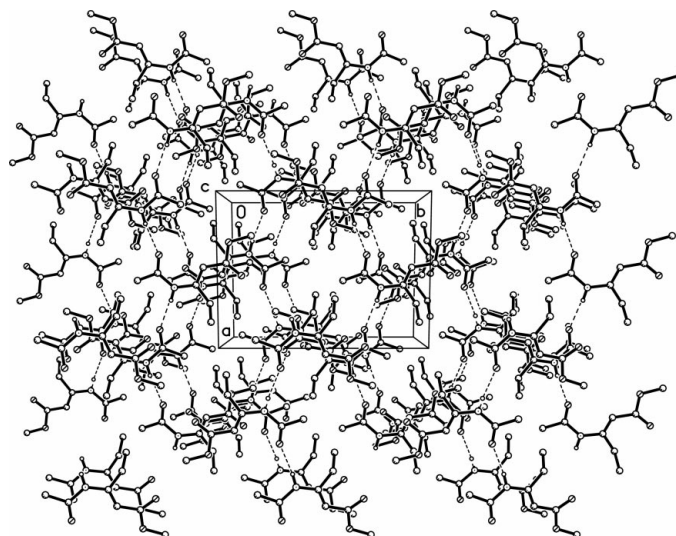


Figure 2
Packing diagram for (I). Hydrogen bonds are indicated by dashed lines.

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···O6	0.86	2.11	2.967 (3)	173
N2—H2A···O3 ⁱ	0.86	2.08	2.936 (3)	174

Symmetry code: (i) *x* - 1, *y*, *z*.

H atoms were included 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: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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