Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl (*E*)-2-acetyl-3-amino-2-pentenoate

Xuanhua Chen* and Tao Chen

Department of Chemistry, Central China Normal University, Wuhan, People's Republic of China

Correspondence e-mail: chxh5211@hotmail.com

Received 17 February 2003
Accepted 10 March 2003
Online 31 March 2003

Key indicators

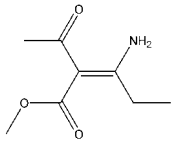
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.087
Data-to-parameter ratio = 18.0

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

The structure of the title compound, $C_8H_{13}NO_3$, refined in space group $Pna2_1$, which was chosen over $Pnam$ as the space group because of the lack of mirror or inversion symmetry. An N—H···O intermolecular hydrogen bond is observed.

Comment

The title compound, (I), is one of the by-products obtained during the synthesis of methyl 3-acetamido-2-pentenoate from the reaction of methyl 3-amino-2-pentenoate refluxed with acetic anhydride in THF for 24 h (Hackler & Wickiser, 1985; Lubell *et al.*, 1991; Yasutake *et al.*, 2001). The structure determination of (I) was conducted in order to obtain more stereochemical information.



(I)

In the crystal structure of (I) (Fig. 1), the N1—C1—C2—C5, C1—C2—C5—O3 and C1—C2—C5—C6 torsion angles are −2.0 (4), −1.1 (4) and 179.4 (3)°, respectively. This shows that atoms N1/C1/C2/C5/O3/C6 are almost coplanar and the C1═C2 and C5═O3 double bonds form a conjugated system (Table 1). The molecules in the crystal structure are interconnected by N—H···O hydrogen bonding (Table 2). As shown in the packing diagram (Fig. 2), the hydrogen bonds links the molecules along the c axis.

Experimental

The title compound, (I), was synthesized according a literature procedure (Zhu *et al.*, 1999). A crystal suitable for X-ray analysis was grown slowly from a mixed solution in ethyl acetate and hexane (1:5) at room temperature.

Crystal data

$C_8H_{13}NO_3$
M_r = 171.19
Orthorhombic, $Pna2_1$
a = 23.998 (7) Å
b = 4.2785 (13) Å
c = 8.944 (3) Å
V = 918.3 (5) Å^3
Z = 4
D_x = 1.238 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1585 reflections
θ = 1–27.5°
μ = 0.10 mm^{-1}
T = 294 (2) K
Block, colorless
0.50 × 0.40 × 0.38 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.954, T_{max} = 0.965
5793 measured reflections
2037 independent reflections
903 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 27.6°
$h = -27 \rightarrow 31$
$k = -5 \rightarrow 5$
$l = -11 \rightarrow 10$

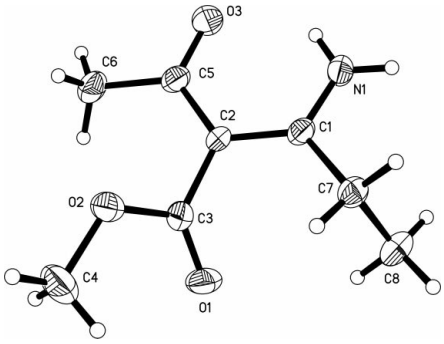


Figure 1
The molecular structure of (I), showing ellipsoids at the 50% probability level (Siemens, 1995).

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.048$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
$wR(F^2) = 0.088$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
$S = 1.03$	Extinction correction: *SHELXL*97
2037 reflections	Extinction coefficient: 0.0098 (18)
113 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = −0.5 (16)
$w = 1/[\sigma^2(F_o^2) + (0.015P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.196 (4)	C1—C2	1.393 (4)
O2—C3	1.339 (3)	C2—C5	1.440 (4)
O3—C5	1.247 (3)	C5—C6	1.499 (4)
N1—C1	1.311 (3)	C7—C8	1.521 (4)
N1—C1—C2	122.5 (3)	C1—C2—C5	121.4 (3)
N1—C1—C7	114.4 (3)	C1—C2—C3	117.1 (2)
N1—C1—C2—C5	−2.0 (4)	C7—C1—C2—C3	−11.7 (4)
C7—C1—C2—C5	172.4 (3)	C1—C2—C5—O3	−1.1 (4)
N1—C1—C2—C3	173.9 (3)	C1—C2—C5—C6	179.4 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3	0.86	1.95	2.597 (3)	131
N1—H1B···O3	0.86	2.04	2.890 (3)	173

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

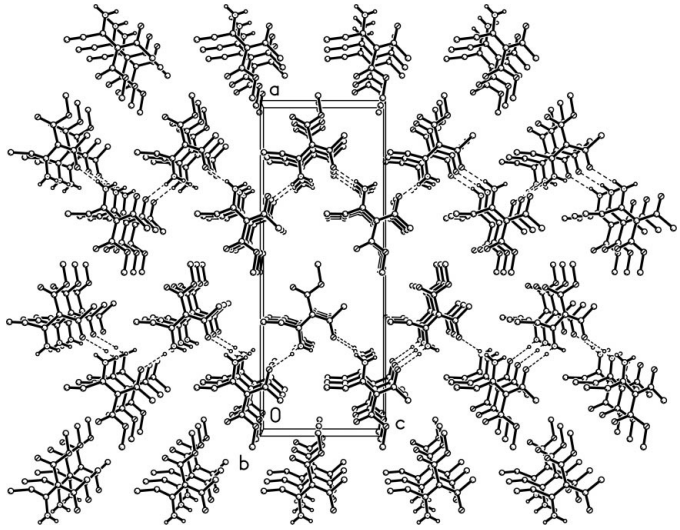


Figure 2
Packing diagram for (I). The hydrogen bonds are indicated by dashed lines.

H atoms were included in the riding-model approximation, with U_{iso} values equal to U_{eq} of the atom to which they are bonded. The Flack (1983) parameter is indeterminate in the absence of significant anomalous scattering.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* 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*.

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Hackler, R. E. & Wickiser, D. I. (1985). Br. UK Patent GB 2 141 712.
Lubell, W. D., Kitamura, M. & Noyori, R. (1991). *Tetrahedron Asymmetry*, **2**, 543–554.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97, University of Göttingen, Germany.
Siemens (1995). *SMART* (Version 5.0), *SAINT* (Version 5.0) and *SHELXTL-NT* (Version 5.10). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Yasutake, M., Gridnew, I. D., Higashi, N. & Imamoto, T. (2001). *Org. Lett.* **3**, 1701–1704.
Zhu, G. X., Chen, Z. G. & Zhang, X. M. (1999). *J. Org. Chem.* **64**, 6907–6910.