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Ethyl (*E*)-2-acetyl-3-amino-3-phenyl-2-propylenoate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.002 Å
R factor = 0.047
wR factor = 0.118
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.

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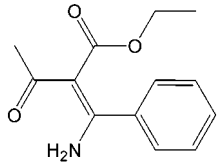
The title compound, $C_{13}H_{15}NO_3$, is an *E* isomer and the phenyl ring does not conjugate with C=C. Both intra- and intermolecular N—H···O hydrogen bonds are found, and the infinite molecular chains stretch along the *b* axis.

Comment

The title compound, (I), is a by-product obtained in the synthesis of ethyl 3-acetamido-3-phenyl-propylenoate, a prochiral olefinic substrate for producing β-amino acids and derivatives by asymmetric hydrogenation (Hackler & Wickiser, 1985; Lubell *et al.*, 1991). The structure determination of (I) was conducted in order to obtain more stereochemical information about β-amino acids and their derivatives. In the structure of (I) (Fig. 1), the C1—C2—C3—C8 and C1—C2—C3—C4 torsion angles are 59.0 (2) and −124.07 (18)°, respectively. This shows that the phenyl ring does not completely conjugate with the C1=C2 double bond in the solid state; the C12—C1—C2—N1, C2—C1—C12—O3 and C2—C1—C12—C13 torsion angles are 5.3 (3), −2.1 (3) and 179.52 (17)°, respectively. This illustrates that the atoms O3—C12—C1—C2—N1—H are almost coplanar and the C12=O3 double bond and C=C form a conjugated system (Table 1). As shown in Table 2 and the packing diagram (Fig. 2), the crystal structure of (I) is stabilized by both intra- and intermolecular hydrogen bonds, and infinite molecular chains stretch along the *b* axis.



(I)

Experimental

The title compound was synthesized according to 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. ^{1}H NMR (400 MHz, acetone-d_6, Bruker): δ 0.63–0.67 (*t*, *J* = 7.1 Hz, 3H), 2.24 (*s*, 3H), 3.64–3.69 (*q*, *J* = 7.1 Hz, 2H), 7.35–7.49 (*m*, 5H), 11.02 (*br*, 1H).

Crystal data

$C_{13}H_{15}NO_3$
M_r = 233.26
Orthorhombic, *Pbca*
a = 17.158 (3) Å
b = 7.6070 (12) Å
c = 18.823 (3) Å
V = 2456.8 (7) Å^3
Z = 8
D_x = 1.261 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4270 reflections
θ = 1–27.5°
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, colorless
0.50 × 0.46 × 0.30 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.974
15490 measured reflections
2820 independent reflections
1423 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 27.5°
$h = -22 \rightarrow 22$
$k = -9 \rightarrow 9$
$l = -24 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.118
S = 1.02
2820 reflections
157 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0249 (15)

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.2098 (19)	C2—C3	1.490 (2)
O3—C12	1.244 (2)	C3—C4	1.380 (2)
C1—C2	1.401 (2)	C4—C5	1.381 (2)
C1—C12	1.451 (2)	C5—C6	1.365 (3)
C1—C9	1.466 (2)	C12—C13	1.504 (3)
C2—C1—C12	120.74 (15)	C3—C4—C5	120.11 (18)
C2—C1—C9	119.41 (14)	C6—C5—C4	120.61 (19)
C12—C1—C9	119.76 (14)	O1—C9—O2	121.87 (16)
N1—C2—C1	122.32 (14)	O1—C9—C1	126.39 (17)
C4—C3—C8	119.08 (15)	O3—C12—C1	121.76 (16)
C4—C3—C2	120.17 (15)		
C12—C1—C2—N1	5.3 (3)	C1—C2—C3—C8	59.0 (2)
C9—C1—C2—N1	−171.24 (16)	C5—C6—C7—C8	−0.3 (3)
C9—C1—C2—C3	11.4 (3)	C4—C3—C8—C7	−0.1 (3)
N1—C2—C3—C4	58.4 (2)	C2—C1—C12—O3	−2.1 (3)
C1—C2—C3—C4	−124.07 (18)	C2—C1—C12—C13	179.52 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3	0.86	1.94	2.592 (2)	131
N1—H1B···O1^{i}	0.86	2.23	3.048 (2)	158

Symmetry code: (i) $x, 1 + y, z$.

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

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

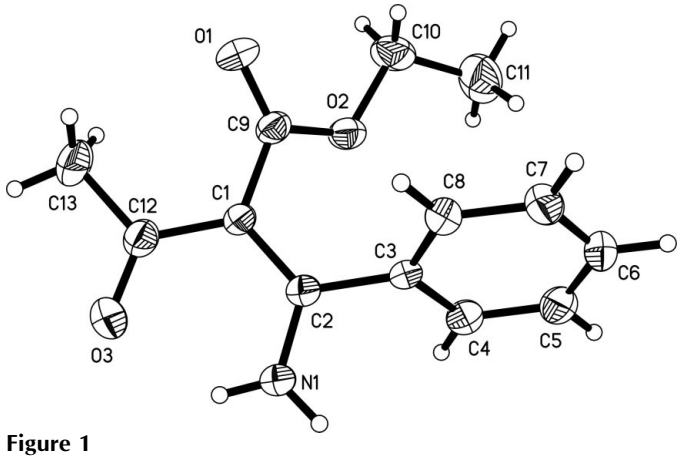


Figure 1
The molecular structure of (I), showing ellipsoids at the 50% probability level (Siemens, 1995).

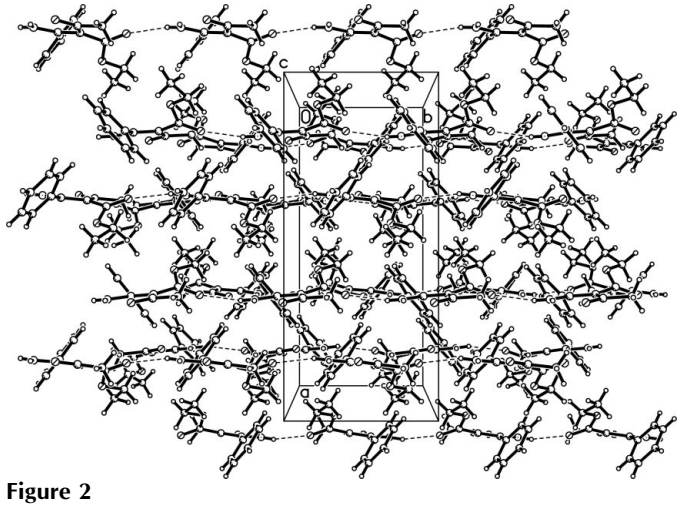


Figure 2
Packing diagram for (I). Hydrogen bonds are indicated by dashed lines.

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References

Hackler, R. E. & Wickiser, D. I. (1985). Br. UK Patent GB 2141712.
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). *SAINT* (Version 5.0), *SMART* (Version 5.0) and *SHELXTL-NT* (Version 5.10). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Zhu, G. X., Chen, Z. G. & Zhang, X. M. (1999). *J. Org. Chem.* **64**, 6907–6910.