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Ethyl (2*E*)-3-oxo-2-(pyrrolidin-2-ylidene)butanoate

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Key indicators

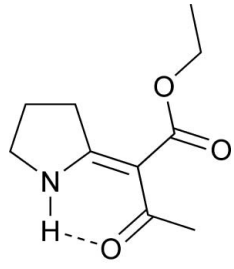
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.064
wR factor = 0.177
Data-to-parameter ratio = 13.8

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

The pyrrolidine ring in the title compound, $C_{10}H_{15}NO_3$, adopts an envelope conformation. Both the secondary amine and acetyl groups participate in bifurcated intra- and intermolecular hydrogen bonding; the former sets up a six-membered hydrogen-bonded ring, while the latter links the molecules into centrosymmetric dimers.

Comment

Cyclic enaminones in which a ring N atom is conjugated through an exocyclic double bond to two different carbonyl groups have found application in the synthesis of alkaloids (Wick *et al.*, 1971), antitubercular indolizinones (Dannhardt *et al.*, 1987) and potential glutamate antagonists (Hitchcock *et al.*, 2003), among others. We required the title compound, (I), a simple example of this type of enaminone, as a precursor in our ongoing programme aimed at the total synthesis of indolizidine alkaloids (Michael *et al.*, 1999). The compound has previously been prepared by condensing pyrrolidin-2-one-derived lactim ethers, thioethers or amide acetals with ethyl acetoacetate (Tsujikawa *et al.*, 1977; Singh *et al.*, 1984; Brunerie *et al.*, 1986).



(I)

The molecular structure of compound (I) clearly establishes its (*E*)-geometry (Fig. 1), previously inferred only from NMR spectroscopic data (Brunerie *et al.*, 1986). The pyrrolidine ring adopts an envelope conformation, with atom C9 (which forms the envelope flap) positioned 0.104 (12) Å above the plane formed by atoms C8, C7, N1 and C10. The Cremer & Pople puckering parameters (Cremer & Pople, 1975) are q_2 = 0.165 (7) Å and φ_2 = 292.0 (17)°.

The crystal structure of (I) is characterized by bifurcated hydrogen bonds between the amine and acetyl groups (Table 1 and Fig. 2). One interaction is an intramolecular N—H···O hydrogen bond between donor atom N1 and acceptor atom O3, described by the graph-set motif *S*(6) (Etter *et al.*, 1990; Bernstein *et al.*, 1995). Atom N1 in the molecule at (*x*, *y*, *z*) also acts as a hydrogen-bond donor *via* atom H1 to atom O3 in

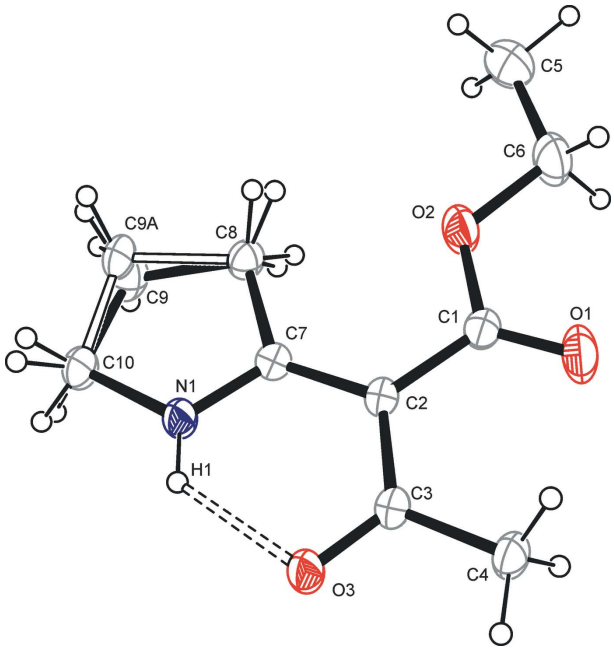


Figure 1
The molecular structure of compound (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Atom C9 is disordered; the minor component is shown with open bonds. The intramolecular hydrogen bond between atoms O3 and H1 is shown with a double dashed line.

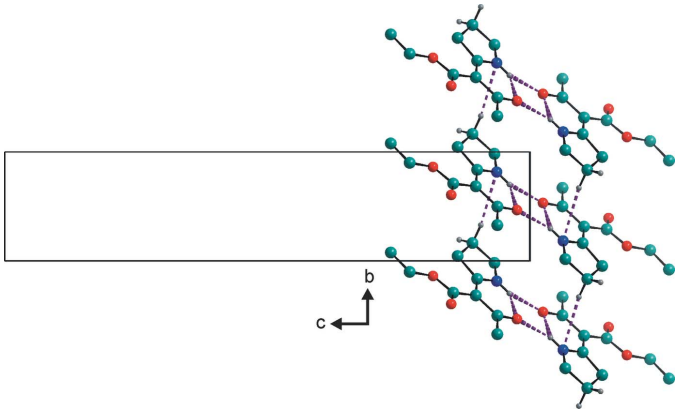


Figure 2
A view down the *a* axis of the crystal packing of compound (I). N—H···O and C—H···N hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

the molecule at $(-x + 1, -y + 1, -z)$. This intermolecular hydrogen bond links the molecules into dimers described by a graph-set motif $R_2^2(12)$. The structure also contains weak C—H···N interactions that link molecules related by translation along the *b* axis into chains.

Experimental

Ethyl (2*E*)-3-oxo-2-pyrrolidin-2-ylidenebutanoate, (I), was prepared from ethyl acetoacetate and 5-methoxy-3,4-dihydro-2*H*-pyrrole as described for the corresponding methyl ester (Wick *et al.*, 1971), giving spectroscopic data identical with those reported elsewhere (Brunerie *et al.* 1986). Crystals of (I) suitable for X-ray crystallography were obtained as fine colourless needles by recrystallization from hexane–ethyl acetate (*ca* 1:1 *v*/*v*).

Crystal data

$C_{10}H_{15}NO_3$
M_r = 197.23
Monoclinic, $P2_1/n$
a = 7.5637 (3) Å
b = 5.2527 (3) Å
c = 25.4713 (13) Å
β = 97.554 (3)°
V = 1003.19 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 (2) K
0.45 × 0.25 × 0.04 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Absorption correction: none
5713 measured reflections
1972 independent reflections
1293 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.177
S = 1.05
1972 reflections
143 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.86 (3)	1.94 (3)	2.570 (3)	129 (2)
N1—H1···O3^{i}	0.86 (3)	2.23 (3)	2.932 (3)	138 (2)
C9—H9*B*···N1^{ii}	0.99	2.71	3.6853 (2)	170

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, y+1, z$.

Disorder in the pyrrolidine ring at atom C9 was resolved by finding alternative positions from the difference Fourier map, and subsequently refined anisotropically over two positions with site occupancy factors of 0.58 (3) for C9 and 0.42 (3) for C9*A*. The ester substituent was also found to be partially disordered. However, attempts to resolve this disorder were unsuccessful owing to the low occupancy of this component. With the exception of H1, all H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H bond lengths of 0.99 Å (CH_2) or 0.98 Å (CH_3), and with U_{iso}(H) = 1.2 (CH_2) or 1.5 (CH_3) times U_{eq}(parent). Atom H1 was found in the difference Fourier map and refined freely.

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: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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