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5-Acetoxy-1-acetyl-3-pyrrolin-2-one

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.090
Data-to-parameter ratio = 9.5

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

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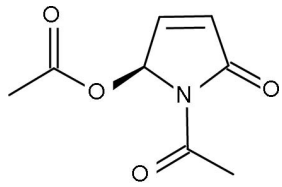
The chiral title compound, $C_8H_9NO_4$, crystallizes in the non-centrosymmetric space group $P2_12_12_1$, but in the absence of an atom with significant anomalous dispersion, the absolute configuration could not be determined. The H atoms in both methyl groups are eclipsed with respect to the C=O bond. The structure features weak C—H···O interactions that link the molecules into a three-dimensional network.

Comment

Optically active 3-pyrrolin-2-ones are multifunctional building blocks that can take part in several stereoselective transformations, such as conjugate additions (Jiménez *et al.*, 1988; Koot *et al.*, 1993), cycloadditions (Koot *et al.*, 1992*a*; Cooper *et al.*, 1995) and acyliminium ion chemistry (Koot *et al.*, 1992*b*). These heterocycles can be used for the preparation of a variety of biologically active compounds, such as gelsemine and peduncularine (Newcombe *et al.*, 1994; Klaver *et al.*, 1989). Examples of pyrrolinone-containing natural products with interesting pharmacological activities are the antitumour alkaloid jatropham (Dittami *et al.*, 1995) and the platelet aggregation inhibitor PI-091 (Shiraki *et al.*, 1996; Iwasawa & Maeyama, 1997).

Racemic 5-acetoxy-1-acetyl-3-pyrrolin-2-one, (I), was synthesized in two steps starting from methoxyfuranone (van der Deen *et al.*, 1996). Both enantiomers of this synthon can be enantiomerically purified in high yield by a chemo–enzymatic route (van der Deen *et al.*, 1996) and the absolute configuration can be determined by circular dichroism measurements or from the crystal structure of the corresponding tetracarbonyl iron complex (Cuiper *et al.*, 1999). This acyloxypyrrolinone can, for example, be converted into the *N*-acyliminium precursor 5-isopropoxypyrrolinone (Goubitz *et al.*, 1996) with retention of configuration by means of a palladium-catalyzed allylic substitution (Cuiper *et al.*, 1998).



(I)

In order to establish unequivocally the structure of the title compound and to obtain structural parameters for a molecular modelling study, an X-ray crystal structure analysis was undertaken (Fig. 1). Although the structure crystallizes in the

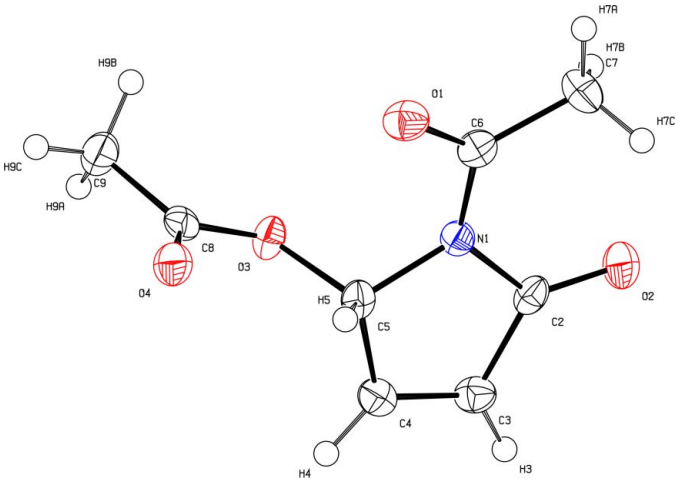


Figure 1
Displacement ellipsoid plot of the title compound drawn at the 50% probability level, including the atom-numbering scheme.

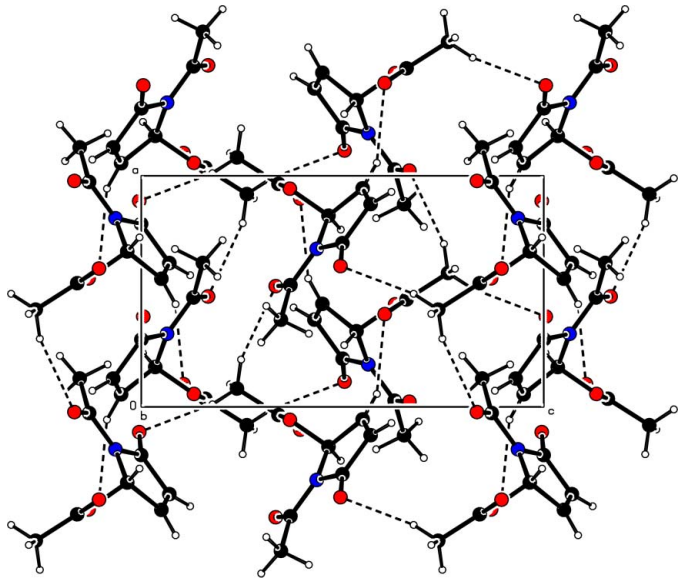


Figure 2
Weakly C—H···O hydrogen-bonded network. Hydrogen bonds are shown as dashed lines.

non-centrosymmetric space group $P2_12_12_1$, the absence of an atom with significant anomalous dispersion made it impossible to determine the absolute configuration.

The H atoms in both methyl groups are eclipsed with respect to the C=O double bond, as expected by analogy with acetaldehyde (Kilb *et al.*, 1957; Iijima & Kimura, 1969), both H—C—C—O torsion angles (H7*A*—C7—C6—O1 and H9*C*—C9—C8—O4) being −1°. The O1—C6—N1—C2—O2 system is close to planar, with O—C—N—C torsion angles of −170.1 (3) and −12.3 (5)°. A search of the Cambridge Structural Database [Version 5.26, with *Conquest* (Version 1.7) (Allen, 2002) shows this to be a common feature for such systems, with torsion angles of predominantly 180/0/−180°. The two C=O groups are, additionally, *cis* to one other, in contrast with the typical metallocyclic complex that would result upon its coordination.

Although there are no classical hydrogen bonds, there are a number of weak C—H···O interactions (Table 1), which lead to the formation of a three-dimensional network (Fig. 2).

Experimental

5-Acetoxy-1-acetyl-3-pyrrolin-2-one was synthesized by the published method (van der Deen *et al.*, 1996) and recrystallized from hot ethanol, to yield colourless crystals suitable for single-crystal X-ray diffraction.

Crystal data

$C_8H_9NO_4$
M_r = 183.16
Orthorhombic, $P2_12_12_1$
a = 6.946 (2) Å
b = 10.204 (2) Å
c = 12.088 (4) Å
V = 856.8 (4) Å^3
Z = 4
D_x = 1.420 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.9–12.0°
μ = 0.12 mm^{-1}
T = 150 (2) K
Block, colourless
0.30 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4T diffractometer
$\omega/2\theta$ scans
4158 measured reflections
1140 independent reflections
855 reflections with $I > 2\sigma(I)$
R_{int} = 0.097
θ_{max} = 27.4°
$h = 0 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.090
S = 1.06
1140 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O4^{i}	0.95	2.46	3.336 (4)	154
C5—H5···O4	1.00	2.27	2.688 (4)	104
C9—H9*A*···O1^{ii}	0.98	2.56	3.508 (4)	162
C9—H9*B*···O2^{iii}	0.98	2.43	3.384 (4)	164

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $x+1, y, z$; (iii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$.

All H atoms were located in a difference map, placed in geometrically idealized positions (C—H = 0.95–1.00 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms. The methyl groups were refined as rigid rotators. In view of the lack of an atom with significant anomalous dispersion, Friedel opposites were merged in the refinement. The absolute configuration was assigned on the basis of the known selectivity of the enzymatic reaction of the purification.

Data collection: locally modified *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *SET4* (de Boer & Duisenberg, 1984); data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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