

1,3-Diacetyl-4-imidazolin-2-one

Rowena Crockett,[‡] Alexander R.
Forrester and R. Alan Howie*Department of Chemistry, University of
Aberdeen, Meston Walk, Aberdeen AB24 3UE,
Scotland[‡] Present address: Swiss Federal Laboratories for
Material Testing and Research (EMPA), Über-
landstrasse 129, 8600 Dübendorf, Switzerland.

Correspondence e-mail: r.a.howie@abdn.ac.uk

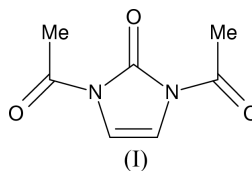
Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.056
 wR factor = 0.113
Data-to-parameter ratio = 8.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The acetyl substituents of the title compound, $\text{C}_7\text{H}_8\text{N}_2\text{O}_3$, are in the *syn* configuration. The bond lengths and angles are as expected for a molecule of this kind.

Comment

The molecule of the title compound, (I), is shown in Fig. 1, and bond lengths and angles involving the non-H atoms are given in Table 1 and are generally as expected for a molecule of this kind. The torsion angles, however, clearly demonstrate the *syn* disposition of the acetyl substituents and are indicative of some departure from planarity in the configuration of the molecule. This departure is further demonstrated by the dihedral angles between the planes of the five-membered ring and those of the acetyl groups [$3.1(4)$ and $5.9(3)^\circ$] and by displacements of the acetyl O and methyl C atoms from the ring plane by as much as $-0.110(8)$ and $0.187(8)\text{ \AA}$ for atoms O1 and C7, respectively. The distribution of the molecules in the unit cell (Fig. 2) can be interpreted in terms of layers (Fig. 3) parallel to (010) and centred on $y = \frac{1}{4}$ and $\frac{3}{4}$. The layer at $y = \frac{3}{4}$ is related to that shown in Fig. 3 by the operation of an *n*-glide plane parallel to (100), which changes the tilt of the molecules from one layer to the next. The whole arrangement brings about the $\text{C}-\text{H}\cdots\text{O}$ contacts given in Table 2, along with a $\text{C}-\text{H}\cdots\pi$ contact involving atoms C7 and H7B and the centroid (*Cg*) of the five-membered ring [this last with symmetry code $(x - \frac{1}{2}, \frac{1}{2} - y, z)$], for which the $\text{C}-\text{H}$, $\text{H}\cdots\text{Cg}$, H_{perp} (the perpendicular distance of H7B from the plane of the ring) and $\text{C7}\cdots\text{Cg}$ distances are 0.96, 2.81, 2.79 and 3.640(3) Å , respectively; the angle at the H atom between $\text{H}\cdots\text{Cg}$ and H_{perp} is 6° , and the $\text{C}-\text{H}\cdots\text{Cg}$ angle is 145° . The contacts involving O1 (Fig. 2 and Table 2) are between the layers and the other two, including the $\text{C}-\text{H}\cdots\pi$ contact noted above, within them.



Experimental

Compound (I) was prepared by heating a suspension of the parent 4-imidazolin-2-one prepared by the method of Haines *et al.* (1982) (0.84 g, 0.01 mol) in acetic anhydride (30 ml) until the solid had dissolved. The excess of acetic anhydride was evaporated to yield (I) (1.53 g, 91%), which was recrystallized from Et_2O as colourless needles [m.p. 379 K, literature m.p. 379 K (Gilbert, 1932)]. ν_{max} (KBr, cm^{-1}): 3130, 1732, 1714, 1385, 1255, 1240, 1132, 1038, 727, 715, 635

Received 9 March 2004
Accepted 11 March 2004
Online 24 March 2004

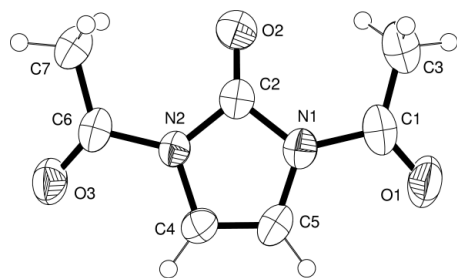


Figure 1

The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

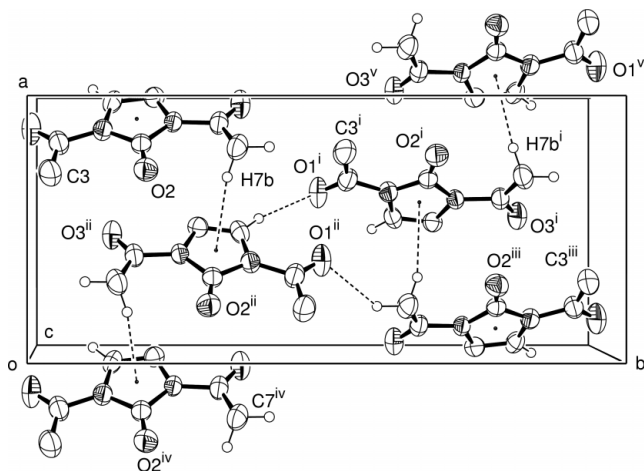


Figure 2

The cell contents of (I). Displacement ellipsoids are drawn at the 50% probability level, H atoms other than those involved in intermolecular contacts (dashed lines) have been omitted and selected atoms are labelled. [Symmetry codes: (i) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} + z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z$; (iii) $1 - x, 1 - y, \frac{1}{2} + z$; (iv) $x - 1, y, z$; (v) $2 - x, 1 - y, \frac{1}{2} + z$.]

and 627; $^1\text{H NMR}$ [$\text{CDCl}_3/(\text{CF}_3\text{CO})_2\text{O}$]: δ 2.59 (6H, s, CH_3), 7.06 (2H, s, CH); m/z 168 (M^+ , 4%): 126 (13), 84 (100), 43 (50).

Crystal data

$\text{C}_7\text{H}_8\text{N}_2\text{O}_3$

$M_r = 168.15$

Orthorhombic, $Pna2_1$

$a = 8.156$ (4) Å

$b = 18.251$ (5) Å

$c = 5.172$ (7) Å

$V = 769.9$ (11) Å³

$Z = 4$

$D_x = 1.451$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 14 reflections

$\theta = 7.6\text{--}10.3^\circ$

$\mu = 0.12$ mm⁻¹

$T = 298$ (2) K

Block, colourless

$0.50 \times 0.40 \times 0.30$ mm

Data collection

Nicolet P3 four-circle diffractometer

θ - 2θ scans

Absorption correction: none

990 measured reflections

990 independent reflections

621 reflections with $I > 2\sigma(I)$

$\theta_{\text{max}} = 27.6^\circ$

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 23$

$l = 0 \rightarrow 6$

2 standard reflections

every 50 reflections

intensity decay: none

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.113$

$S = 1.01$

990 reflections

111 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.18$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

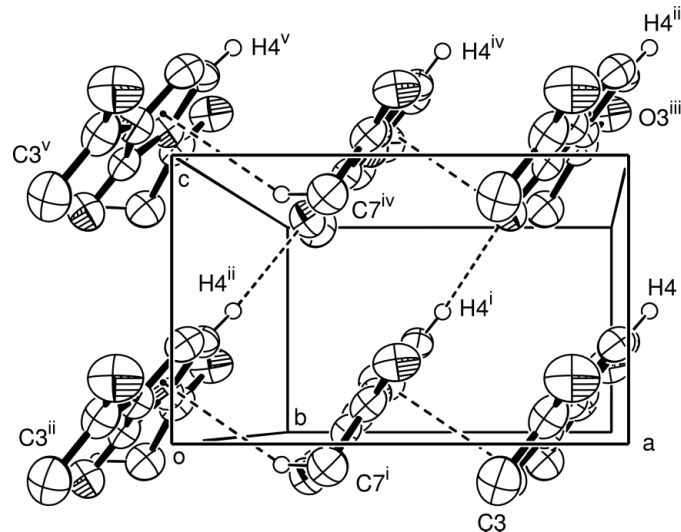


Figure 3

A layer of molecules of (I) parallel to (010) and centred on $y = \frac{1}{4}$. Displacement ellipsoids are drawn at the 50% probability level, H atoms other than those involved in intermolecular contacts (dashed lines) have been omitted and selected atoms are labelled. [Symmetry codes: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z$; (ii) $x - 1, y, z$; (iii) $x, y, 1 + z$; (iv) $x - \frac{1}{2}, \frac{1}{2} - y, 1 + z$; (v) $x - 1, y, 1 + z$.]

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.199 (5)	N2—C2	1.397 (5)
O2—C2	1.205 (5)	N2—C4	1.407 (5)
O3—C6	1.203 (5)	N2—C6	1.426 (4)
N1—C5	1.401 (6)	C1—C3	1.487 (7)
N1—C2	1.405 (5)	C4—C5	1.319 (5)
N1—C1	1.430 (5)	C6—C7	1.485 (7)
C5—N1—C2	110.0 (3)	O2—C2—N2	128.4 (4)
C5—N1—C1	121.4 (4)	O2—C2—N1	128.5 (4)
C2—N1—C1	128.5 (4)	N2—C2—N1	103.1 (4)
C2—N2—C4	110.6 (3)	C5—C4—N2	107.6 (4)
C2—N2—C6	127.7 (4)	C4—C5—N1	108.7 (4)
C4—N2—C6	121.6 (3)	O3—C6—N2	118.0 (4)
O1—C1—N1	118.0 (4)	O3—C6—C7	124.1 (4)
O1—C1—C3	124.4 (4)	N2—C6—C7	117.9 (4)
N1—C1—C3	117.6 (4)	C2—N1—C1—O1	-175.5 (4)
C2—N1—C1—O1	-175.5 (4)	C2—N1—C1—O1	0.9 (6)
C2—N1—C1—C3	5.2 (6)	C2—N1—C1—C3	5.2 (6)
C5—N1—C1—C3	-178.5 (4)	C4—N2—C6—O3	175.8 (4)
		C4—N2—C6—O3	-5.3 (6)
		C2—N2—C6—C7	-5.4 (6)
		C4—N2—C6—C7	173.5 (4)

Table 2

Parameters (Å, °) for C—H...O contacts between molecules of (I).

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
C4—H4...O2 ⁱ	0.93	2.59	3.522 (6)	174.7
C5—H5...O1 ⁱⁱ	0.93	2.54	3.463 (5)	169.8
C7—H7A...O1 ⁱⁱⁱ	0.96	2.56	3.312 (5)	135.0

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

In the final stages of refinement, H atoms were introduced in calculated positions, with C—H = 0.93 Å (alkene H atoms) and 0.96 Å (methyl H atoms), and treated using a riding model, with

$U_{\text{iso}}(\text{H})$ set at $1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{C})$ for alkene and methyl H atoms, respectively. The rotational orientation of the rigid-body methyl groups was also refined. In the absence of any atom of atomic number higher than that of O, the Flack (1983) parameter is, for this refinement, meaningless and the absolute polarity is indeterminate.

Data collection: *Nicolet P3 Software* (Nicolet, 1980); cell refinement: *Nicolet P3 Software*; data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Financial support for this work by the SERC is gratefully acknowledged.

References

- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
Gilbert, G. E. (1932). *J. Am. Chem. Soc.* **54**, 3413–3419.
Haines, D. R., Leonard, N. J. & Wiemer, D. F. (1982). *J. Org. Chem.* **47**, 474–482.
Howie, R. A. (1980). *RDNIC*. University of Aberdeen, Scotland.
Nicolet (1980). *Nicolet P3 Software*. Nicolet XRD Corporation, 10061 Bubb Road, Cupertino, CA 95014, USA.
Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.