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

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Key indicators

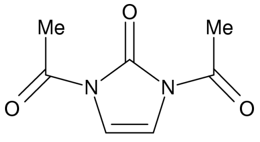
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.056
wR factor = 0.113
Data-to-parameter ratio = 8.9

For 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, $C_7H_8N_2O_3$, 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)°] 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) Å 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 C—H⋯O contacts given in Table 2, along with a C—H⋯π contact involving atoms C7 and H7*B* 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 C—H, H⋯*Cg*, H_{perp} (the perpendicular distance of H7*B* from the plane of the ring) and C7⋯*Cg* distances are 0.96, 2.81, 2.79 and 3.640 (3) Å, respectively; the angle at the H atom between H⋯*Cg* and H_{perp} is 6°, and the C—H⋯*Cg* angle is 145°. The contacts involving O1 (Fig. 2 and Table 2) are between the layers and the other two, including the C—H⋯π contact noted above, within them.



(I)

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

DOI: 10.1107/S1600536804005756

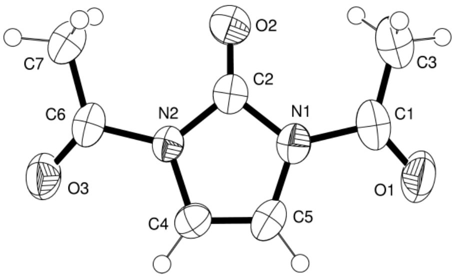


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

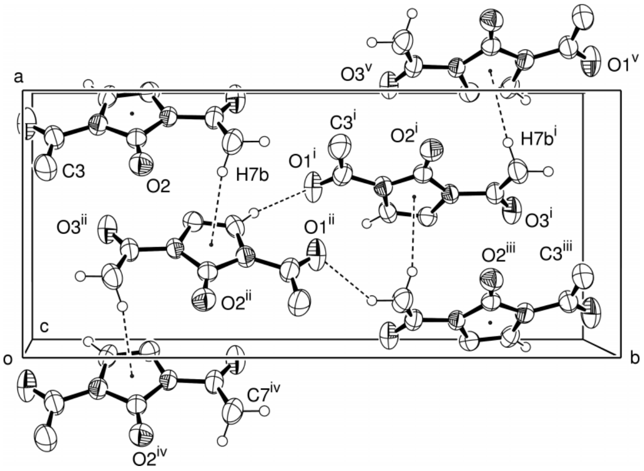


Figure 2
The cell contents of (I). Displacement ellipsoids are drawn at the 50% probability level, H atoms other than those involved in intermolecular contacts (dashed lines) have been omitted and selected atoms are labelled. [Symmetry codes: (i) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} + z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z$; (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}H NMR [$CDCl_3/(CF_3CO)_2O$]: 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

$C_7H_8N_2O_3$
$M_r = 168.15$
Orthorhombic, $Pna2_1$
$a = 8.156$ (4) Å
$b = 18.251$ (5) Å
$c = 5.172$ (7) Å
$V = 769.9$ (11) Å^3
$Z = 4$
$D_x = 1.451$ Mg m^{-3}

Mo *K*α radiation
Cell parameters from 14 reflections
$\theta = 7.6$–$10.3°$
$\mu = 0.12$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.50 × 0.40 × 0.30 mm

Data collection

Nicolet *P*3 four-circle diffractometer
θ–2θ scans
Absorption correction: none
990 measured reflections
990 independent reflections
621 reflections with $I > 2\sigma(I)$

$\theta_{max} = 27.6°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 6$
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

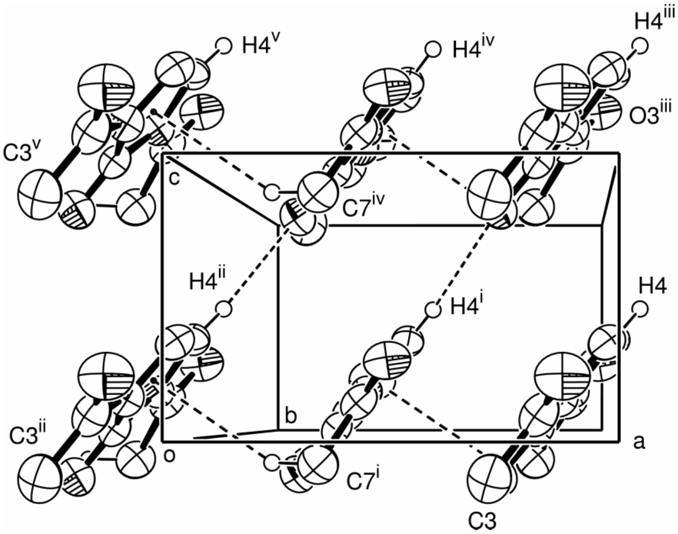


Figure 3
A layer of molecules of (I) parallel to (010) and centred on $y = \frac{1}{4}$. Displacement ellipsoids are drawn at the 50% probability level, H atoms other than those involved in intermolecular contacts (dashed lines) have been omitted and selected atoms are labelled. [Symmetry codes: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z$; (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—N2—C6—O3	175.8 (4)
C5—N1—C1—O1	0.9 (6)	C4—N2—C6—O3	−5.3 (6)
C2—N1—C1—C3	5.2 (6)	C2—N2—C6—C7	−5.4 (6)
C5—N1—C1—C3	−178.5 (4)	C4—N2—C6—C7	173.5 (4)

Table 2
Parameters (Å, °) for C—H···O contacts between molecules of (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*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—H7*A*···O1^{iii}	0.96	2.56	3.312 (5)	135.0

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, 1 + z$; (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_{iso}(H) set at 1.2U_{eq}(C) and 1.5U_{eq}(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 P*3 *Software* (Nicolet, 1980); cell refinement: *Nicolet P*3 *Software*; data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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supporting information

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

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Crystal data

$C_7H_8N_2O_3$
$M_r = 168.15$
Orthorhombic, $Pna2_1$
$a = 8.156$ (4) Å
$b = 18.251$ (5) Å
$c = 5.172$ (7) Å
$V = 769.9$ (11) Å^3
$Z = 4$
$F(000) = 352$
$D_x = 1.451$ Mg m^{-3}
Melting point: 379 (lit. 379) K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 14 reflections
$\theta = 7.6–10.3°$
$\mu = 0.12$ mm^{-1}
$T = 298$ K
Block, colourless
0.50 × 0.40 × 0.30 mm

Data collection

Nicolet P3 four circle diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
θ–2θ scans
990 measured reflections
990 independent reflections
621 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.000$
$\theta_{\text{max}} = 27.6°$, $\theta_{\text{min}} = 2.2°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 6$
2 standard reflections every 50 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.113$
$S = 1.01$
990 reflections
111 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
6.4918 (0.0220) x - 1.6134 (0.1449) y - 3.0973 (0.0135) z = 4.9755 (0.0222)
* 0.0000 (0.0000) C1 * 0.0000 (0.0000) O1 * 0.0000 (0.0000) C3
Rms deviation of fitted atoms = 0.0000
6.3195 (0.0133) x - 0.9068 (0.0342) y - 3.2595 (0.0091) z = 4.8997 (0.0144)
Angle to previous plane (with approximate e.s.d.) = 3.10 (0.41)
* 0.0047 (0.0023) N1 * -0.0050 (0.0022) C2 * 0.0036 (0.0022) N2 * -0.0007 (0.0024) C4 * -0.0026 (0.0025) C5 - 0.1097 (0.0075) O1 - 0.0448 (0.0067) C1 0.0026 (0.0080) C3 - 0.0297 (0.0058) O2 - 0.0402 (0.0066) O3 0.0348 (0.0066) C6 0.1867 (0.0083) C7
Rms deviation of fitted atoms = 0.0036
6.7793 (0.0139) x - 1.4639 (0.1425) y - 2.8453 (0.0121) z = 5.2256 (0.0505)
Angle to previous plane (with approximate e.s.d.) = 5.88 (0.31)
* 0.0000 (0.0000) C6 * 0.0000 (0.0000) O3 * 0.0000 (0.0000) C7
Rms deviation of fitted atoms = 0.0000

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.
H in calculated positions and refined with a riding model.
In the absence of any atom of atomic number higher than that of O the the Flack x parameter is meaningless and the absolute structure therefore indeterminate.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.8759 (4)	0.00337 (15)	0.2277 (7)	0.0721 (12)
O2	0.7257 (3)	0.19844 (14)	−0.1424 (7)	0.0548 (8)
O3	0.9703 (4)	0.35970 (14)	0.2903 (6)	0.0582 (9)
N1	0.8722 (4)	0.12506 (16)	0.1516 (8)	0.0443 (9)
N2	0.9021 (4)	0.24430 (15)	0.1767 (7)	0.0378 (8)
C1	0.8238 (5)	0.0516 (2)	0.0934 (10)	0.0545 (14)
C2	0.8199 (5)	0.1905 (2)	0.0349 (9)	0.0406 (10)
C3	0.7135 (5)	0.0402 (2)	−0.1319 (12)	0.0672 (14)
H3A	0.7773	0.0389	−0.2877	0.101*
H3B	0.6359	0.0797	−0.1412	0.101*
H3C	0.6560	−0.0054	−0.1121	0.101*
C4	0.9979 (4)	0.2123 (2)	0.3726 (9)	0.0416 (9)
H4	1.0627	0.2371	0.4918	0.050*
C5	0.9790 (5)	0.1406 (2)	0.3565 (10)	0.0458 (11)
H5	1.0284	0.1062	0.4636	0.055*
C6	0.8982 (5)	0.3217 (2)	0.1381 (9)	0.0448 (11)
C7	0.8084 (5)	0.3501 (2)	−0.0906 (10)	0.0556 (13)
H7A	0.8094	0.4027	−0.0879	0.083*
H7B	0.6971	0.3329	−0.0861	0.083*
H7C	0.8606	0.3330	−0.2457	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.104 (3)	0.0356 (14)	0.077 (3)	−0.0005 (18)	0.000 (2)	0.0069 (18)
O2	0.0523 (16)	0.0546 (17)	0.057 (2)	−0.0017 (14)	−0.006 (2)	0.0000 (19)
O3	0.083 (2)	0.0376 (15)	0.054 (2)	−0.0015 (15)	0.0041 (19)	−0.0037 (16)
N1	0.0455 (19)	0.0373 (16)	0.050 (2)	0.0015 (15)	0.004 (2)	−0.001 (2)
N2	0.0411 (17)	0.0313 (15)	0.0410 (19)	0.0021 (14)	0.0011 (19)	−0.0001 (18)
C1	0.055 (3)	0.040 (2)	0.068 (4)	−0.007 (2)	0.016 (3)	−0.009 (3)
C2	0.039 (2)	0.039 (2)	0.044 (2)	0.0006 (19)	0.009 (2)	0.000 (2)
C3	0.067 (3)	0.050 (2)	0.084 (4)	−0.007 (2)	0.005 (4)	−0.015 (3)
C4	0.042 (2)	0.0430 (19)	0.039 (2)	0.0012 (18)	0.000 (2)	0.006 (2)
C5	0.053 (2)	0.037 (2)	0.047 (3)	0.0097 (18)	0.008 (3)	0.008 (2)
C6	0.051 (3)	0.034 (2)	0.049 (3)	0.003 (2)	0.014 (3)	0.003 (2)
C7	0.064 (3)	0.042 (2)	0.060 (3)	0.007 (2)	0.001 (3)	0.011 (2)

Geometric parameters (Å, º)

O1—C1	1.199 (5)	C3—H3A	0.9600
O2—C2	1.205 (5)	C3—H3B	0.9600
O3—C6	1.203 (5)	C3—H3C	0.9600
N1—C5	1.401 (6)	C4—C5	1.319 (5)
N1—C2	1.405 (5)	C4—H4	0.9300
N1—C1	1.430 (5)	C5—H5	0.9300
N2—C2	1.397 (5)	C6—C7	1.485 (7)
N2—C4	1.407 (5)	C7—H7A	0.9600
N2—C6	1.426 (4)	C7—H7B	0.9600
C1—C3	1.487 (7)	C7—H7C	0.9600
C5—N1—C2	110.0 (3)	H3B—C3—H3C	109.5
C5—N1—C1	121.4 (4)	C5—C4—N2	107.6 (4)
C2—N1—C1	128.5 (4)	C5—C4—H4	126.2
C2—N2—C4	110.6 (3)	N2—C4—H4	126.2
C2—N2—C6	127.7 (4)	C4—C5—N1	108.7 (4)
C4—N2—C6	121.6 (3)	C4—C5—H5	125.6
O1—C1—N1	118.0 (4)	N1—C5—H5	125.6
O1—C1—C3	124.4 (4)	O3—C6—N2	118.0 (4)
N1—C1—C3	117.6 (4)	O3—C6—C7	124.1 (4)
O2—C2—N2	128.4 (4)	N2—C6—C7	117.9 (4)
O2—C2—N1	128.5 (4)	C6—C7—H7A	109.5
N2—C2—N1	103.1 (4)	C6—C7—H7B	109.5
C1—C3—H3A	109.5	H7A—C7—H7B	109.5
C1—C3—H3B	109.5	C6—C7—H7C	109.5
H3A—C3—H3B	109.5	H7A—C7—H7C	109.5
C1—C3—H3C	109.5	H7B—C7—H7C	109.5
H3A—C3—H3C	109.5		
C2—N1—C1—O1	−175.5 (4)	C1—N1—C2—N2	177.6 (4)

C5—N1—C1—O1	0.9 (6)	C2—N2—C4—C5	0.4 (4)
C2—N1—C1—C3	5.2 (6)	C6—N2—C4—C5	−178.7 (3)
C5—N1—C1—C3	−178.5 (4)	N2—C4—C5—N1	0.2 (5)
C4—N2—C2—O2	178.5 (4)	C2—N1—C5—C4	−0.7 (5)
C6—N2—C2—O2	−2.5 (7)	C1—N1—C5—C4	−177.7 (4)
C4—N2—C2—N1	−0.8 (4)	C2—N2—C6—O3	175.8 (4)
C6—N2—C2—N1	178.2 (4)	C4—N2—C6—O3	−5.3 (6)
C5—N1—C2—O2	−178.4 (4)	C2—N2—C6—C7	−5.4 (6)
C1—N1—C2—O2	−1.7 (7)	C4—N2—C6—C7	173.5 (4)
C5—N1—C2—N2	0.9 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.59	3.522 (6)	175
C5—H5···O1^{ii}	0.93	2.54	3.463 (5)	170
C7—H7*A*···O1^{iii}	0.96	2.56	3.312 (5)	135

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