

## 1,4-Diacetyl-1,2,4-triazolin-5-one

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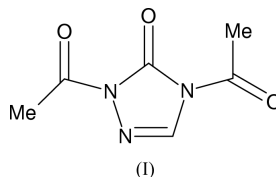
## Key indicators

Single-crystal X-ray study  
T = 298 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
Disorder in main residue  
R factor = 0.040  
wR factor = 0.109  
Data-to-parameter ratio = 12.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of the title compound,  $\text{C}_6\text{H}_7\text{N}_3\text{O}_3$ , lies on the mirror plane of the space group  $P2_1/m$ . The acetyl substituents adopt an *anti* configuration. The bond lengths and angles are as expected for a molecule of this kind.

## Comment

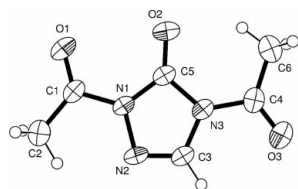
The molecule of the title compound, (I), is shown in Fig. 1 and bond lengths and angles involving the non-H atoms are given in Table 1. The molecule sits on a crystallographic mirror plane and for this reason the torsion angles given in the Table, which clearly demonstrate the *anti* disposition of the acetyl substituents, and those given in the CIF, lack s.u.s, because they are all precisely either 180 or 0°. The remaining bond lengths and angles are unremarkable. The arrangement of the molecules within the unit cell, creating layers parallel to (001), is shown in Fig. 2. Within the layers, molecules related to one another by the operation of a crystallographic twofold screw axis, exemplified by the pair of molecules in the middle of the figure, occur in stacks extending in the direction of **b**, precisely parallel to one another and at regular intervals of  $b/2$ . Within the stacks, the triazole rings are in close proximity, with a perpendicular distance between their planes and a distance between their centroids of 3.099 (2) and 3.995 (1) Å, respectively. These values correspond to a lateral displacement, or slippage, of the rings from one ring to the next in the stack, of 2.521 (2) Å. Intermolecular contacts (dashed lines in Fig. 2) of the form  $\text{C3}-\text{H3}\cdots\text{O2}^{\text{iv}}$  [symmetry code: (iv)  $1-x, y, z$ ] with C—H, H $\cdots$ O2 and C $\cdots$ O2 distances of 0.93, 2.42 and 3.350 (3) Å, respectively, and an angle of 176° at the H atom, connect molecules in adjacent stacks, creating chains of molecules parallel to **a**.



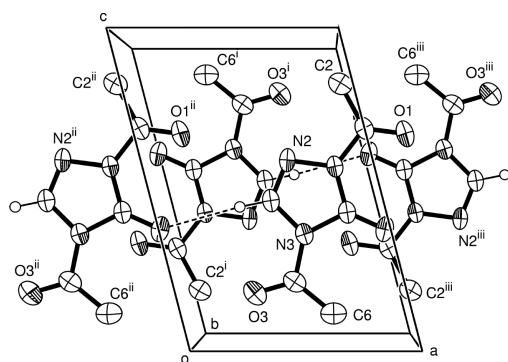
## Experimental

Compound (I) was prepared by heating a suspension of the parent 1,2,4-triazolin-5-one, prepared by the method of Haines *et al.* (1982) (0.85 g, 0.01 mole), in acetic anhydride (30 ml) until the solid had dissolved. The excess of acetic anhydride was evaporated to yield (I) (1.59 g, 94%), which was recrystallized from  $\text{CH}_2\text{Cl}_2$  as colourless plates (m.p. 412–413 K). Found: C 42.47, H 4.16, N 24.84%;  $\text{C}_6\text{H}_7\text{N}_3\text{O}_3$  requires: C 42.61, H 4.17, N 24.84%. IR ( $\nu_{\text{max}}$ , KBr,  $\text{cm}^{-1}$ ): 3094, 1835, 1782, 1738, 1713, 1586, 1429, 1372, 1342, 1256, 1231, 920, 743, 729, 631 and 592;  $^1\text{H}$  NMR [ $\delta$ ,  $\text{CDCl}_3/(\text{CF}_3\text{CO})_2\text{O}$ , p.p.m.] 2.54 (s,  $\text{CH}_3$ ), 2.63 (s,  $\text{CH}_3$ ), 8.16 (s, CH);  $m/z$ : 169 ( $M^+$ , 4%), 127 (37), 85 (46), 43 (100).

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**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 unit-cell contents of (I). Displacement ellipsoids are drawn at the 50% probability level, H atoms other than those involved in C—H...O contacts (dashed lines) have been omitted for clarity, and selected atoms are labelled. Symmetry codes: (i)  $1 - x, \frac{1}{2} + y, 1 - z$ ; (ii)  $x - 1, y, z$ ; (iii)  $2 - x, \frac{1}{2} + y, 1 - z$ .

**Crystal data**

$C_6H_7N_3O_3$   
 $M_r = 169.15$   
 Monoclinic,  $P2_1/m$   
 $a = 6.572$  (3) Å  
 $b = 6.199$  (3) Å  
 $c = 9.463$  (3) Å  
 $\beta = 104.54$  (3)°  
 $V = 373.2$  (3) Å<sup>3</sup>  
 $Z = 2$

$D_x = 1.505$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 14 reflections  
 $\theta = 11.0$ – $12.0$ °  
 $\mu = 0.12$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 Block, colourless  
 $0.35 \times 0.30 \times 0.25$  mm

**Data collection**

Nicolet P3 four-circle diffractometer  
 $\theta$ – $2\theta$  scans  
 Absorption correction: none  
 1010 measured reflections  
 944 independent reflections  
 759 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.015$

$\theta_{max} = 27.6$ °  
 $h = 0 \rightarrow 8$   
 $k = 0 \rightarrow 8$   
 $l = -12 \rightarrow 11$   
 2 standard reflections every 50 reflections  
 intensity decay: none

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.109$   
 $S = 1.04$   
 944 reflections  
 76 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.0599P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.18$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.16$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL  
 Extinction coefficient: 0.087 (13)

**Table 1**

Selected geometric parameters (Å, °).

O1—C1	1.198 (2)	N2—C3	1.276 (2)
O2—C5	1.206 (2)	N3—C3	1.386 (2)
O3—C4	1.198 (3)	N3—C5	1.406 (2)
N1—C5	1.384 (3)	N3—C4	1.419 (3)
N1—N2	1.405 (2)	C1—C2	1.485 (3)
N1—C1	1.415 (2)	C4—C6	1.472 (3)
C5—N1—N2	112.62 (14)	N1—C1—C2	114.11 (16)
C5—N1—C1	127.14 (15)	N2—C3—N3	113.14 (15)
N2—N1—C1	120.23 (16)	O3—C4—N3	117.6 (2)
C3—N2—N1	104.40 (15)	O3—C4—C6	124.4 (2)
C3—N3—C5	107.66 (15)	N3—C4—C6	117.92 (18)
C3—N3—C4	123.37 (15)	O2—C5—N1	129.41 (17)
C5—N3—C4	128.97 (17)	O2—C5—N3	128.42 (18)
O1—C1—N1	120.29 (19)	N1—C5—N3	102.17 (15)
O1—C1—C2	125.60 (19)		
C5—N1—C1—O1	0.0	C3—N3—C4—O3	0.0
N2—N1—C1—O1	180.0	C5—N3—C4—O3	180.0
C5—N1—C1—C2	180.0	C3—N3—C4—C6	180.0
N2—N1—C1—C2	0.0	C5—N3—C4—C6	0.0

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C3-H3 \cdots O2^{ii}$	0.93	2.42	3.350 (3)	176

Symmetry code: (ii)  $x - 1, y, z$ .

In the final stages of refinement the H atoms were introduced in calculated positions, with C—H 0.93 Å (alkene H atoms) and 0.96 Å (methyl H atoms), and refined with a riding model, with  $U_{iso}(H)$  set to 1.2 and  $1.5U_{eq}(C)$  for alkene and methyl H atoms, respectively. For both methyl groups, the H atoms all have occupancies of 0.5 because, following refinement of the rotational orientation of the rigid-body methyl groups, they are found, in each case, to be disordered over two sets of mirror plane symmetry-related sites.

Data collection: P3 software (Nicolet, 1980); cell refinement: P3 software; data reduction: RDNIC (Howie, 1980); program(s) used to solve structure: SHELXS97 (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).

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