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Key indicators

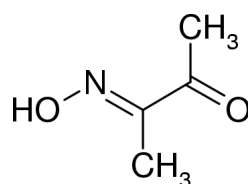
Single-crystal X-ray study
 $T = 120\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.067
 wR factor = 0.181
Data-to-parameter ratio = 19.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Hydrogen-bonded molecular chains in (2*E*)-butane-2,3-dione oxime with $Z' = 3$ The title compound (also known as diacetyl monoxime), $\text{C}_4\text{H}_7\text{NO}_2$, crystallizes with three molecules in the asymmetric unit and forms continuous hydrogen-bonded molecular chains.

Received 23 April 2002

Accepted 29 April 2002

Online 11 May 2002

Comment

The simple title molecule, (I), has been frequently used for the determination of urea concentrations in milk, plasma and urine (*e.g.* Marsh *et al.*, 1957; Butler *et al.*, 1981). It also affects a number of biological mechanisms, including muscle contraction, ionic current flow and synaptic transmission (*e.g.* Sellin & McArdle, 1994).

(I)

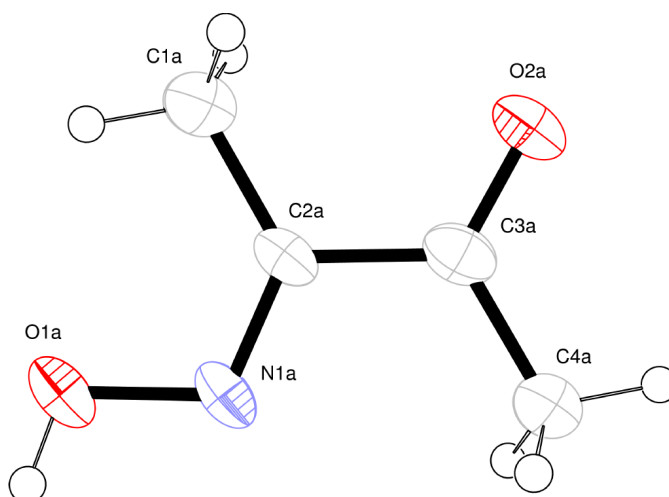
As with 2,3-butanedione (Eriks *et al.*, 1983) and 2,5-diacetyl-3,4-diazahexa-2,4-diene (Korber *et al.*, 1987), the $\text{CH}_3-\text{Csp}^2-\text{Csp}^2-\text{CH}_3$ conformation in the molecule is *trans*. Unfortunately, the conformational isomer with the *cis* arrangement is also known as diacetyl monoxime (*e.g.* Moszner *et al.*, 1997).

Figure 1

The atomic arrangement in molecule A of (I). Displacement ellipsoids are shown at the 50% probability level.


Figure 2

Molecules in a hydrogen-bonded chain. From left to right: molecule C (coordinates transformed by $1/2+x$, $1/2-y$, $1/2-z$), molecule A, molecule B, molecule C (coordinates transformed by $1/2-x$, $1/2-y$, $1/2+z$).

The three molecules (A, B and C) of the asymmetric unit have similar geometries and there are no significant differences between equivalent bond lengths and bond angles. The largest discrepancy among all the equivalent torsions angles (essentially *cis* or *trans*) is $7.0(7)^\circ$ (Table 1).

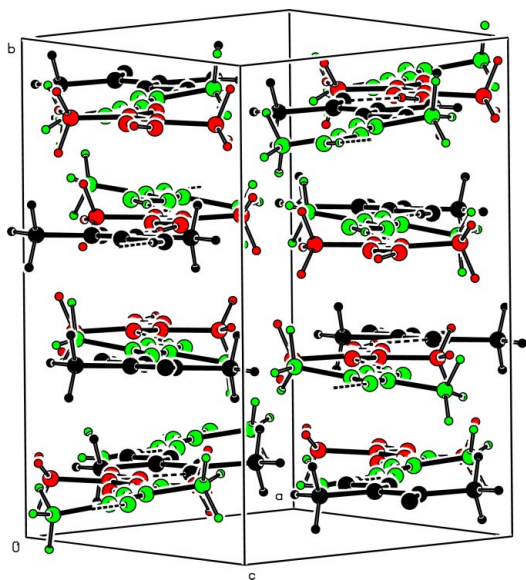
Each molecule is hydrogen bonded to two other molecules, to form a continuous chain, as shown in Fig. 2. In the unit cell, these chains run parallel in the *ac* direction and are stacked along the *b* axis (Fig. 3). Differences in the strengths of the hydrogen bonds are evident as variations in intermolecular separations and linearity (Table 2) are present.

Experimental

Diacetyl monoxime (97%) was purchased from Aldrich (CAS 57-71-6) and was recrystallized from ethanol.

Crystal data

$C_4H_7NO_2$	$D_x = 1.286 \text{ Mg m}^{-3}$
$M_r = 101.11$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 20830 reflections
$a = 11.5354(7) \text{ \AA}$	$\theta = 1.0\text{--}30.5^\circ$
$b = 12.7225(12) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 11.8884(10) \text{ \AA}$	$T = 120(2) \text{ K}$
$\beta = 116.106(3)^\circ$	Block, colourless
$V = 1566.7(2) \text{ \AA}^3$	$0.10 \times 0.10 \times 0.05 \text{ mm}$
$Z = 12$	


Figure 3

Packing diagram, showing chains stacked along the *b* axis and running parallel in the *ac* direction.

Data collection

Enraf-Nonius KappaCCD area-detector diffractometer	3745 independent reflections
φ and ω scans	1343 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SORTAV; Blessing, 1995, 1997)	$R_{\text{int}} = 0.187$
$T_{\text{min}} = 0.990$, $T_{\text{max}} = 0.995$	$\theta_{\text{max}} = 30.2^\circ$
19544 measured reflections	$h = -14 \rightarrow 15$
	$k = -16 \rightarrow 15$
	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.067$	$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2]$
$wR(F^2) = 0.181$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.94$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3745 reflections	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
190 parameters	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1A—N1A	1.392 (3)	C1A—C2A	1.494 (5)
O2A—C3A	1.230 (3)	C2A—C3A	1.481 (5)
N1A—C2A	1.284 (4)	C3A—C4A	1.496 (5)
C2A—N1A—O1A	112.6 (3)	O2A—C3A—C2A	119.5 (3)
N1A—C2A—C3A	114.4 (3)	O2A—C3A—C4A	121.1 (3)
N1A—C2A—C1A	126.9 (3)	C2A—C3A—C4A	119.3 (3)
C3A—C2A—C1A	118.7 (3)		
C1A—C2A—C3A—C4A	−180.0 (3)	C1C—C2C—C3C—C4C	178.4 (3)
C1B—C2B—C3B—C4B	−175.4 (4)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1B—H1B \cdots O2A	0.84	1.90	2.727 (4)	166
O1A—H1A \cdots O2C ⁱ	0.84	1.87	2.699 (4)	168
O1C—H1C \cdots O2B ⁱ	0.84	1.84	2.677 (5)	178

Symmetry code: (i) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$.

Refinement was affected by the limited quality and quantity of the data. The diffraction intensities were very weak from the small crystal with three independent molecules in the asymmetric unit, despite the use of a rotating-anode X-ray source, and the crystal mosaic spread was found to be unusually high ($>1.3^\circ$). The H atoms were initially placed in calculated positions and thereafter made to ride on their attached atoms, each with isotropic displacement parameter $1.2U_{\text{eq}}$ of the parent atom.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97-2* (Sheldrick, 1998); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2001).

We thank the EPSRC for use of the National Crystallographic Service, at Southampton University (X-ray data collection), and for the use of the Chemical Database Service at Daresbury (Fletcher *et al.*, 1996).

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