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Key indicators

Single-crystal X-ray study
T = 130 K
Mean $\sigma(\text{C}-\text{C})$ = 0.003 Å
R factor = 0.028
wR factor = 0.078
Data-to-parameter ratio = 9.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Acetylacetone dioxime

The title compound, $\text{C}_5\text{H}_{10}\text{N}_2\text{O}_2$, has twofold crystallographic symmetry. A set of four molecules packs around a $\bar{4}$ center to form a 12-membered ring that is constructed from four O—H···N hydrogen bonds.Received 22 September 2003
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Comment

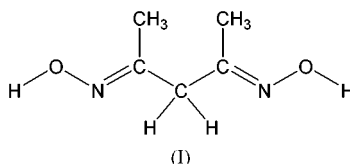
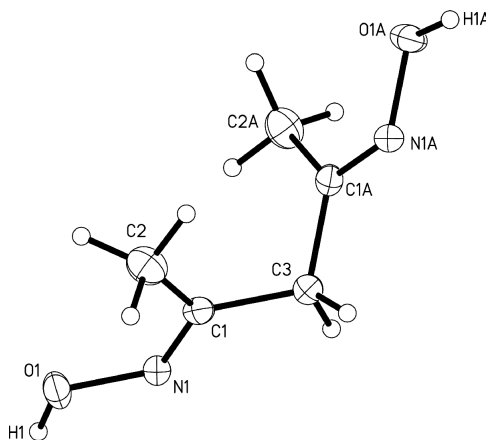
The central C atom (C3) of acetylacetone dioxime, (I), is situated on a crystallographic twofold axis which gives rise to a $\text{HONC}(\text{CH}_3)\text{CH}_2\text{C}(\text{CH}_3)\text{NOH}$ twisted chain (Fig. 1). This differs from the conformation of glyoxime, which has crystallographic $\bar{1}$ symmetry and is planar with the exception of the oxime H atoms (Jeffrey *et al.*, 1982). Bond distances, angles and torsion angles in (I) are presented in Table 1.By virtue of the crystallographic $\bar{4}$ axis, one end of each of four molecules is involved in a tetrameric arrangement of O—H···N hydrogen bonding (Fig. 2). The metrical data (Table 2) suggest that this is a moderately strong hydrogen bond. The various possible conformations about the atoms in the dioxime chain give rise to a number of conformational isomers (Chertanova *et al.*, 1994; Jeffrey *et al.*, 1982). The favorable energetics of the tetrameric arrangement is likely a contrib-

Figure 1

A view of (I) with the atomic numbering scheme [symmetry code: (A) $y - \frac{1}{2}, \frac{1}{2} + x, \frac{1}{2} - z$]. Displacement ellipsoids are drawn at the 35% probability level.

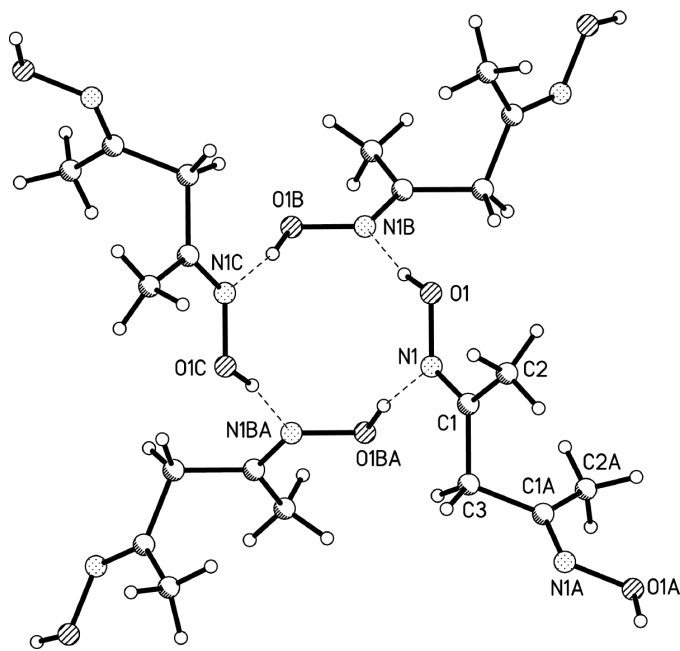


Figure 2
A view of the tetramer that is formed from packing around the $\bar{4}$ symmetry element. [Symmetry codes: (B) $1 - y, x, -z$; (C) $1 - x, 1 - y, z$; (BA) $y, 1 - x, -z$.]

uting factor to the particular conformation that crystallized here (Maurin, 1998).

Experimental

The compound was synthesized as described in the US Patent (Sahbari & Russell, 2001) from acetylacetone and hydroxylamine and recrystallized from perfluorocyclohexane to obtain a crystal suitable for data collection.

Crystal data

$C_5H_{10}N_2O_2$
 $M_r = 130.15$
Tetragonal, $P\bar{4}n2$
 $a = 10.852$ (2) Å
 $c = 5.9751$ (15) Å
 $V = 703.7$ (3) Å³
 $Z = 4$
 $D_x = 1.229$ Mg m⁻³

Data collection

Syntex $P2_1$ diffractometer
 $2\theta - \omega$ scans
Absorption correction: none
777 measured reflections
598 independent reflections
577 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.022$

Cu $K\alpha$ radiation
Cell parameters from 48 reflections
 $\theta = 8.1 - 27.0^\circ$
 $\mu = 0.80$ mm⁻¹
 $T = 130$ (2) K
Block, colorless
 $0.20 \times 0.20 \times 0.20$ mm

$\theta_{max} = 66.8^\circ$
 $h = 0 \rightarrow 12$
 $k = 0 \rightarrow 12$
 $l = 0 \rightarrow 7$
2 standard reflections every 198 reflections
intensity decay: < 0.1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.078$
 $S = 1.12$
598 reflections
63 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.1006P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.13$ e Å⁻³
 $\Delta\rho_{min} = -0.11$ e Å⁻³
Extinction correction: *SHELXL97*
Extinction coefficient: 0.025 (3)

Table 1

Selected geometric parameters (Å, °).

O1—N1	1.4007 (16)	C1—C2	1.486 (3)
O1—H1	0.99 (3)	C1—C3	1.5090 (18)
N1—C1	1.281 (2)	C3—H3	0.98 (2)
N1—O1—H1	99.1 (14)	N1—C1—C3	115.81 (13)
C1—N1—O1	112.30 (13)	C2—C1—C3	120.21 (13)
N1—C1—C2	123.95 (13)	C1—C3—C1 ⁱ	113.00 (18)
H1—O1—N1—C1	175.6 (14)	N1—C1—C3—C1 ⁱ	119.77 (15)
O1—N1—C1—C2	-1.1 (2)	C2—C1—C3—C1 ⁱ	-62.04 (13)
O1—N1—C1—C3	177.05 (13)		

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

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 \cdots N1 ⁱⁱ	0.99 (3)	1.78 (3)	2.767 (2)	170 (2)

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

Molecule (I) crystallized in the non-centrosymmetric space group $P\bar{4}n2$, but the absolute configuration was not determined since only light atoms were present and only a unique set of data was collected. H atoms were freely refined.

Data collection: *P3-PC* (Siemens, 1991); cell refinement: *P3-PC*; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL97*.

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