

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

anti-Ethyl acetohydroximateBarbara Hachuła,^{a*} Anna Polasz,^a Maria Nowak^b and Joachim Kusz^b^aInstitute of Chemistry, University of Silesia, 14 Bankowa Street, 40-007 Katowice, Poland, and ^bInstitute of Physics, University of Silesia, 4 Uniwersytecka Street, 40-007 Katowice, Poland

Correspondence e-mail: bhachula@o2.pl

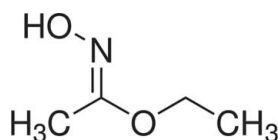
Received 2 August 2013; accepted 8 August 2013

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.034; wR factor = 0.098; data-to-parameter ratio = 14.1.

In the crystal structure of the title compound, $\text{C}_4\text{H}_9\text{NO}_2$, the $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into supra-molecular chains extending along the b -axis direction. The conformation of the NOH group in the nearly planar (r.m.s. deviation = 0.0546 Å) ethyl acetohydroximate molecule is *trans* to $\text{N}=\text{C}$.

Related literature

For related structures, see: Kjaer *et al.* (1977); Larsen (1971). For studies of the IR spectra of hydrogen bonding in oxime derivatives, see: Flakus *et al.* (2012). For typical bond distances, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$\text{C}_4\text{H}_9\text{NO}_2$
 $M_r = 103.12$
 Monoclinic, $C2/c$
 $a = 19.9481$ (9) Å
 $b = 4.4138$ (1) Å
 $c = 13.3277$ (5) Å
 $\beta = 109.027$ (4)°

$V = 1109.35$ (7) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 100$ K
 $0.52 \times 0.18 \times 0.14$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire3 detector
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2006)
 $T_{\min} = 0.505$, $T_{\max} = 1.000$
 6699 measured reflections
 970 independent reflections
 868 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.098$
 $S = 1.08$
 970 reflections
 69 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}^{\text{i}}$	0.871 (19)	1.954 (19)	2.8196 (14)	172.4 (16)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FF2116).

References

- Allen, F. A., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Bernstein, J., Davies, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
 Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
 Flakus, H., Hachuła, B. & Garbacz, A. (2012). *J. Phys. Chem.* **A116**, 11553–11567.
 Kjaer, A., Larsen, I. K. & Sivertsen, P. (1977). *Acta Chem. Scand. Ser. B*, **31**, 415–423.
 Larsen, I. K. (1971). *Acta Chem. Scand.* **25**, 2409–2420.
 Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
 Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Wrocław, Poland.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2013). E69, o1418 [doi:10.1107/S1600536813022368]

anti-Ethyl acetohydroximate

Barbara Hachula, Anna Polasz, Maria Nowak and Joachim Kusz

S1. Comment

Anti-ethyl acetohydroximate [systematic name: ethyl *N*-hydroxyacetimidate], (I), was investigated in a continuation of our studies of the IR spectra of hydrogen bonding in oxime derivatives (Flakus *et al.*, 2012). In order to study interactions occurring *via* hydrogen bonds and molecular packing in this compound, we have now determined the structure of (I) using diffraction data collected at 100 K. Until now, the structures of *syn*-methyl acetohydroximate and *syn*- and *anti*-ethyl benzohydroximate were determined (Kjaer *et al.*, 1977; Larsen *et al.*, 1971). The crystal structure of *syn*-methyl acetohydroximate is composed of layers of molecules, which form cyclic, hydrogen-bonded trimers, whereas the crystals of *syn*- and *anti*-ethyl benzohydroximate are composed of dimers formed by pairs of O—H \cdots N hydrogen-bonded molecules.

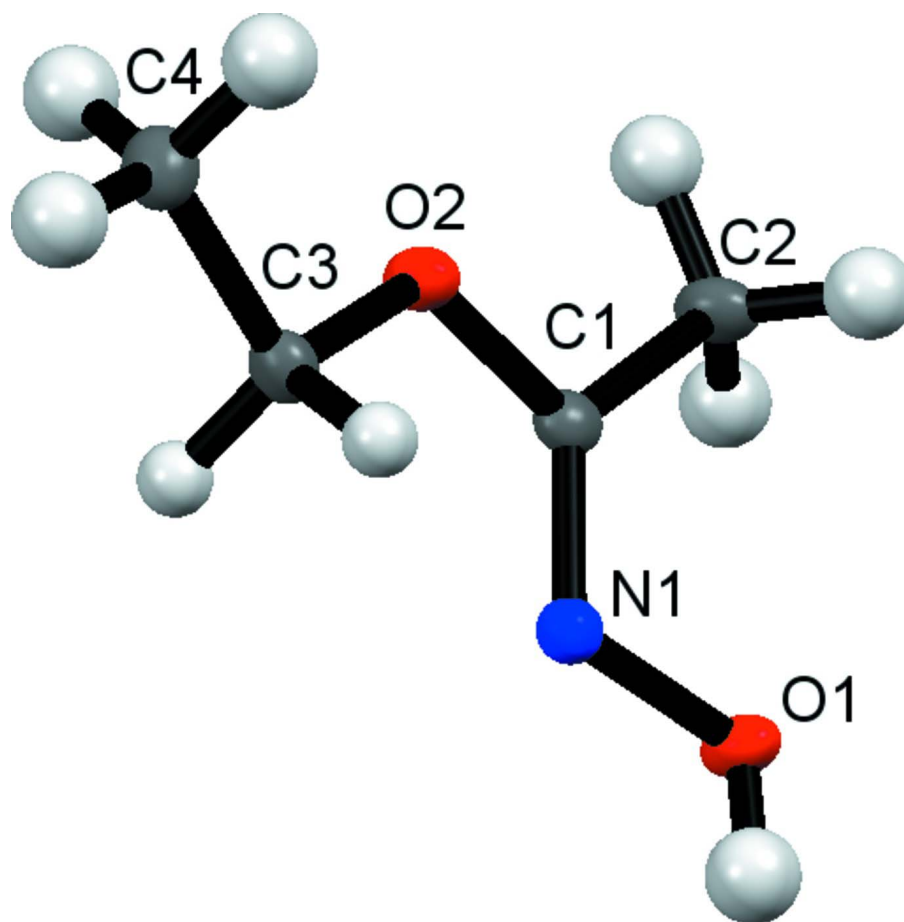
The molecule of (I) is nearly planar (r.m.s. deviations 0.0546 Å for all non-H atoms). The lengths of the bonds C=N (1.2771 (17) Å) and N—O (1.4286 (13) Å) in (I) are comparable to the mean values found in other oximes (C=N 1.281 Å; N—O 1.394 Å) (Allen *et al.*, 1987). The conformation of the NOH group in the planar ethyl acetohydroximate molecule is *trans* to N=C. In the crystal, O—H \cdots N are observed forming infinite chains along the *b* axis (Fig. 2) with a graph-set motif of *C*(3) (Etter *et al.*, 1990; Bernstein *et al.*, 1995).

S2. Experimental

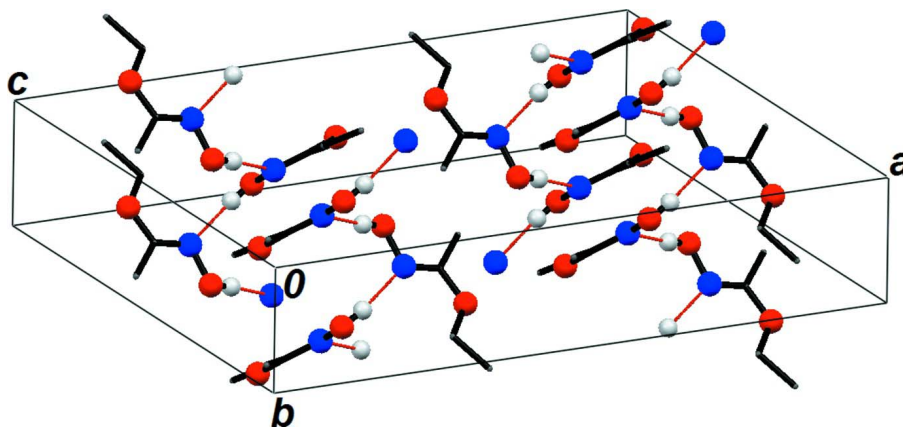
Ethyl acetohydroximate was purchased from Aldrich-Sigma. Crystals of title compound, suitable for X-ray diffraction, were selected directly from purchased sample.

S3. Refinement

The H atoms were introduced in geometrically idealized positions with C—H distances of 0.99 Å and $U_{\text{iso}}(\text{H})$ values set at $1.2U_{\text{eq}}(\text{C})$ for methylene group or 0.98 Å and with $U_{\text{iso}}(\text{H})$ values set at $1.5U_{\text{eq}}(\text{C})$ for methyl groups. The H atom which takes part in hydrogen bonding was located in a difference Fourier map and was refined with $U_{\text{iso}}(\text{H})$ value set at $1.5U_{\text{eq}}(\text{O})$.

**Figure 1**

The asymmetric unit of (I), with the atom-numbering scheme, showing 50% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radius.

**Figure 2**

Part of the crystal structure of (I), showing the C(3) chains. The red lines indicate the hydrogen-bonding interactions. For the sake of clarity, all H atoms bonded to C atoms were omitted.

Ethyl *N*-hydroxyethanecarboximidate

Crystal data

C₄H₉NO₂ $M_r = 103.12$ Monoclinic, *C2/c*Hall symbol: $-C\ 2yc$ $a = 19.9481\ (9)\ \text{\AA}$ $b = 4.4138\ (1)\ \text{\AA}$ $c = 13.3277\ (5)\ \text{\AA}$ $\beta = 109.027\ (4)^\circ$ $V = 1109.35\ (7)\ \text{\AA}^3$ $Z = 8$ $F(000) = 448$ $D_x = 1.235\ \text{Mg m}^{-3}$

Melting point = 296–298 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 6376 reflections

 $\theta = 3.1\text{--}34.4^\circ$ $\mu = 0.10\ \text{mm}^{-1}$ $T = 100\ \text{K}$

Needle, colourless

 $0.52 \times 0.18 \times 0.14\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur

diffractometer with a Sapphire3 detector

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 16.0328 pixels mm^{-1} ω -scan

Absorption correction: multi-scan

(CrysAlis RED; Oxford Diffraction, 2006)

 $T_{\min} = 0.505$, $T_{\max} = 1.000$

6699 measured reflections

970 independent reflections

868 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.025$ $\theta_{\max} = 25.1^\circ$, $\theta_{\min} = 3.2^\circ$ $h = -22 \rightarrow 23$ $k = -2 \rightarrow 5$ $l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.034$ $wR(F^2) = 0.098$ $S = 1.08$

970 reflections

69 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.5949P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.22\ \text{e \AA}^{-3}$ $\Delta\rho_{\min} = -0.22\ \text{e \AA}^{-3}$

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.

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.31644 (5)	0.6668 (2)	0.82875 (7)	0.0201 (3)
O2	0.39491 (4)	0.1706 (2)	0.69431 (7)	0.0185 (3)
N1	0.31725 (5)	0.4501 (2)	0.74954 (8)	0.0165 (3)

C1	0.38102 (7)	0.3704 (3)	0.76216 (10)	0.0158 (3)
C2	0.44623 (7)	0.4799 (3)	0.84598 (10)	0.0215 (3)
H2A	0.4568	0.6871	0.8294	0.032*
H2B	0.4862	0.3472	0.8486	0.032*
H2C	0.4385	0.4774	0.9149	0.032*
C3	0.33586 (7)	0.0728 (3)	0.60358 (10)	0.0187 (3)
H3A	0.3103	0.2503	0.5636	0.022*
H3B	0.3022	-0.0497	0.6271	0.022*
C4	0.36699 (8)	-0.1145 (3)	0.53500 (11)	0.0235 (3)
H4A	0.3987	0.0118	0.5100	0.035*
H4B	0.3287	-0.1929	0.4739	0.035*
H4C	0.3938	-0.2843	0.5765	0.035*
H1	0.2735 (10)	0.740 (4)	0.8065 (13)	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0168 (5)	0.0243 (5)	0.0190 (5)	0.0038 (4)	0.0054 (4)	-0.0041 (4)
O2	0.0153 (5)	0.0230 (5)	0.0163 (5)	0.0012 (3)	0.0041 (4)	-0.0017 (4)
N1	0.0170 (6)	0.0172 (6)	0.0154 (6)	-0.0001 (4)	0.0052 (4)	0.0006 (4)
C1	0.0167 (7)	0.0174 (6)	0.0140 (6)	0.0000 (5)	0.0062 (5)	0.0034 (5)
C2	0.0152 (7)	0.0290 (7)	0.0193 (7)	0.0011 (5)	0.0042 (5)	-0.0014 (6)
C3	0.0175 (7)	0.0201 (7)	0.0164 (7)	-0.0019 (5)	0.0026 (5)	0.0002 (5)
C4	0.0282 (7)	0.0241 (7)	0.0188 (7)	-0.0025 (6)	0.0086 (6)	-0.0006 (5)

Geometric parameters (Å, °)

O1—N1	1.4286 (13)	C2—H2C	0.9800
O1—H1	0.871 (19)	C3—C4	1.5082 (17)
O2—C1	1.3547 (15)	C3—H3A	0.9900
O2—C3	1.4517 (15)	C3—H3B	0.9900
N1—C1	1.2771 (17)	C4—H4A	0.9800
C1—C2	1.4922 (17)	C4—H4B	0.9800
C2—H2A	0.9800	C4—H4C	0.9800
C2—H2B	0.9800		
N1—O1—H1	104.1 (11)	O2—C3—C4	106.57 (10)
C1—O2—C3	117.55 (9)	O2—C3—H3A	110.4
C1—N1—O1	109.76 (10)	C4—C3—H3A	110.4
N1—C1—O2	120.21 (12)	O2—C3—H3B	110.4
N1—C1—C2	126.66 (12)	C4—C3—H3B	110.4
O2—C1—C2	113.11 (10)	H3A—C3—H3B	108.6
C1—C2—H2A	109.5	C3—C4—H4A	109.5
C1—C2—H2B	109.5	C3—C4—H4B	109.5
H2A—C2—H2B	109.5	H4A—C4—H4B	109.5
C1—C2—H2C	109.5	C3—C4—H4C	109.5
H2A—C2—H2C	109.5	H4A—C4—H4C	109.5
H2B—C2—H2C	109.5	H4B—C4—H4C	109.5

O1—N1—C1—O2	-178.49 (9)	C3—O2—C1—C2	-174.04 (10)
O1—N1—C1—C2	0.24 (17)	C1—O2—C3—C4	173.10 (10)
C3—O2—C1—N1	4.85 (16)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...N1 ⁱ	0.871 (19)	1.954 (19)	2.8196 (14)	172.4 (16)

Symmetry code: (i) $-x+1/2, y+1/2, -z+3/2$.