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(*E*)-3,5-Dimethoxybenzaldehyde oxime

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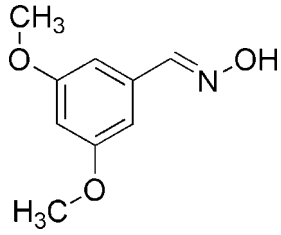
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.081; data-to-parameter ratio = 10.0.

In the title compound, $C_9H_{11}NO_3$, the oxime grouping is twisted by 12.68 (6)° with respect to the dimethoxylbenzene ring. In the crystal, molecules are linked into an infinite [100] chain *via* O—H···N hydrogen bonds, instead of the more common oxime packing motif of dimers with an $R_2^2(6)$ graph-set motif.

Related literature

For backgroud to oximes as therapeutic agents, see: Marrs *et al.* (2006); Jokanovic *et al.* (2009). For related structures, see: Bao (2008); Abbas *et al.* (2010). For graph-set theory, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_9H_{11}NO_3$
M_r = 181.19
Orthorhombic, $P2_12_12_1$
a = 4.4027 (9) Å
b = 13.800 (3) Å
c = 14.300 (3) Å
V = 868.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 113 K
0.20 × 0.18 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.979, T_{max} = 0.990
7173 measured reflections
1239 independent reflections
1115 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.081
S = 1.08
1239 reflections
124 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···N1^{i}	0.916 (19)	1.90 (2)	2.7970 (17)	166.5 (19)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5656).

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supporting information

Acta Cryst. (2010). E**66**, o2719 [https://doi.org/10.1107/S1600536810038766]

(*E*)-3,5-Dimethoxybenzaldehyde oxime

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S1. Comment

Oximes are an therapeutic agent in organophosphorus poisoning (Marrs *et al.*, 2006; Jokanovic *et al.*, 2009). As part of our interest in the study of oxime derivatives, we herein report the crystal structure of the title compound (I).

In the crystal structure of the title compound, Fig. 1, the oxime moiety has an E configuration [C5—C9—N1—O3= 178.22 (11)°] and is twisted with respect to the dimethoxylbenzene ring by 12.68 (6)°. Molecules are linked to form an infinite chain down the *a* axis *via* O—H···N hydrogen bonds (Fig. 2 and Table 1), which differates from the reported $R_2^2(6)$ graph-set motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995; Bao, 2008; Abbas *et al.*, 2010).

S2. Experimental

To a solution of 3,4-dimethoxylbenzaldehyde (0.95 g, 5 mmol) in 25 ml e thanol, hydroxylamine hydrochloride (0.42 g, 6 mmol) and aqueous sodium hydroxide (0.24 g, 6 mmol) were added and the mixture was heated under reflux until completion of the reaction. The reaction mixture was concentrated and water added. The precipitate was collected by filtration, washed with water and dried under vaccu. Colourless blocks of (I) were grown out *via* recrystallization from ethanol.

S3. Refinement

All H atoms were placed in calculated position and treated as riding on their parent atoms with C—H = 0.93 and 0.97Å or O—H =0.82 Å with U_{iso}(H) = 1.2 U_{eq}(C) for aromatic H atoms, or 1.5U_{eq} (O and C) for hydroxyl H and methyl H atom].

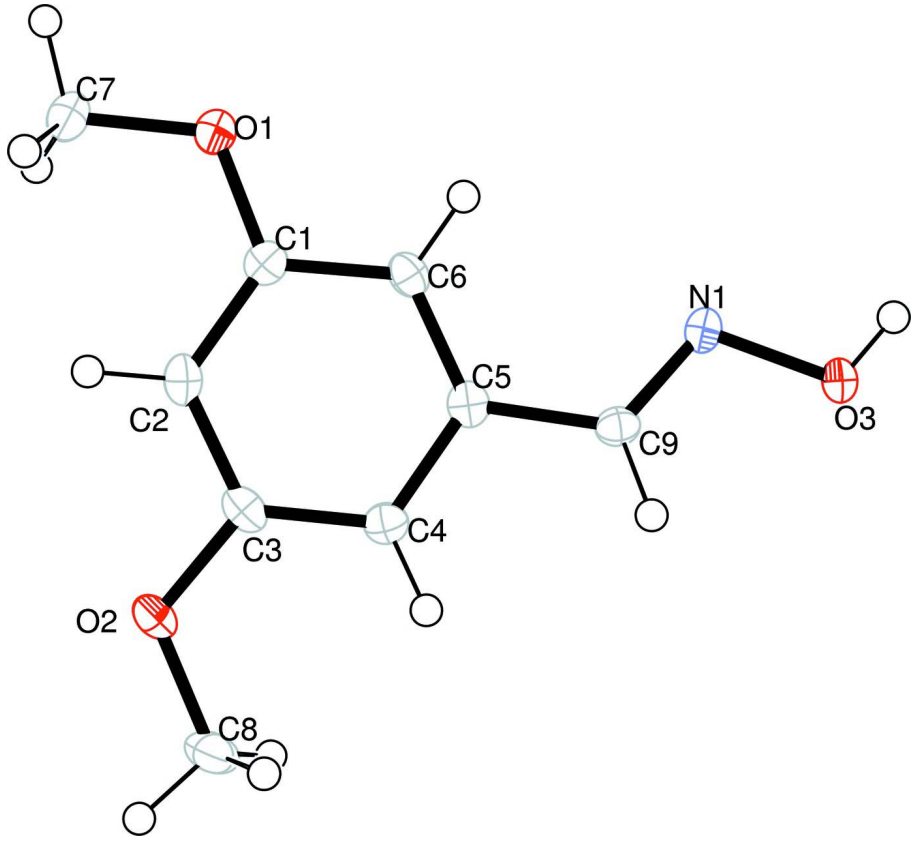

Figure 1

The molecule of (I) showing displacement ellipsoids drawn at the 50% probability level.

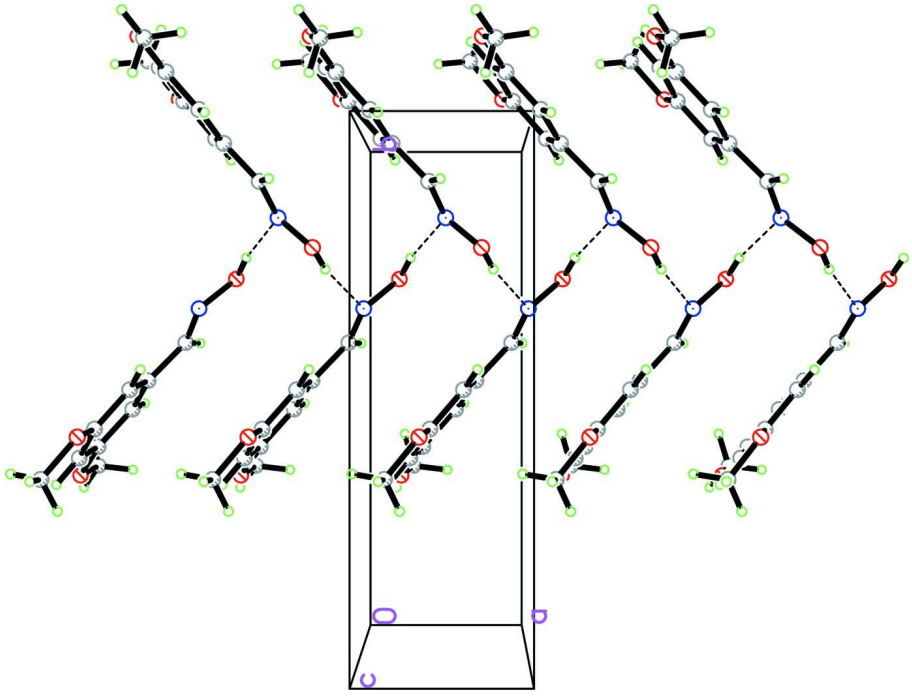

Figure 2

The infinite chain formed *via* O—H···N down the *a* axis.

(*E*)-3,5-Dimethoxybenzaldehyde oxime

Crystal data

$C_9H_{11}NO_3$
$M_r = 181.19$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 4.4027\ (9)$ Å
$b = 13.800\ (3)$ Å
$c = 14.300\ (3)$ Å
$V = 868.9\ (3)$ Å^3
$Z = 4$
$F(000) = 384$
$D_x = 1.385$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3117 reflections
$\theta = 2.1$–$27.9°$
$\mu = 0.11$ mm^{-1}
$T = 113$ K
Block, colorless
0.20 × 0.18 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.979$, $T_{max} = 0.990$
7173 measured reflections
1239 independent reflections
1115 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.1°$
$h = -5 \rightarrow 5$
$k = -18 \rightarrow 12$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.081$
$S = 1.08$
1239 reflections
124 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
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.0067P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.135 (12)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.1440 (3)	0.57833 (8)	1.13939 (7)	0.0215 (3)
O2	1.2719 (3)	0.66671 (7)	0.81997 (7)	0.0212 (3)
O3	0.2763 (3)	0.27744 (7)	0.90865 (7)	0.0195 (3)
H3	0.211 (5)	0.2351 (13)	0.9537 (13)	0.029*
N1	0.4898 (3)	0.33604 (8)	0.95540 (9)	0.0159 (3)
C1	1.0843 (4)	0.56661 (10)	1.04608 (10)	0.0170 (3)
C2	1.2075 (4)	0.62501 (10)	0.97646 (10)	0.0179 (3)
H2	1.3404	0.6767	0.9923	0.021*
C3	1.1337 (4)	0.60688 (10)	0.88299 (10)	0.0169 (3)
C4	0.9316 (4)	0.53398 (10)	0.85910 (10)	0.0168 (3)
H4	0.8775	0.5233	0.7956	0.020*
C5	0.8078 (4)	0.47596 (10)	0.93064 (10)	0.0156 (3)
C6	0.8833 (4)	0.49179 (10)	1.02358 (10)	0.0167 (3)
H6	0.7994	0.4522	1.0714	0.020*
C7	1.3439 (4)	0.65620 (10)	1.16453 (11)	0.0221 (4)
H7A	1.5396	0.6476	1.1329	0.033*
H7B	1.3752	0.6561	1.2324	0.033*
H7C	1.2536	0.7180	1.1455	0.033*
C8	1.2008 (4)	0.65223 (11)	0.72320 (10)	0.0255 (4)
H8A	1.2574	0.5862	0.7048	0.038*
H8B	1.3140	0.6990	0.6852	0.038*
H8C	0.9824	0.6616	0.7135	0.038*
C9	0.5886 (3)	0.40214 (10)	0.90072 (10)	0.0161 (3)

H9	0.5170	0.4035	0.8381	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0254 (6)	0.0209 (5)	0.0180 (5)	−0.0057 (5)	−0.0022 (5)	−0.0001 (4)
O2	0.0242 (6)	0.0191 (5)	0.0204 (5)	−0.0043 (4)	0.0028 (5)	0.0047 (4)
O3	0.0208 (6)	0.0195 (5)	0.0182 (5)	−0.0073 (5)	−0.0012 (5)	−0.0006 (4)
N1	0.0136 (6)	0.0151 (6)	0.0188 (6)	−0.0007 (5)	−0.0006 (6)	−0.0019 (4)
C1	0.0169 (7)	0.0151 (7)	0.0189 (7)	0.0014 (6)	−0.0015 (6)	−0.0009 (5)
C2	0.0161 (7)	0.0137 (6)	0.0238 (7)	−0.0007 (6)	−0.0006 (6)	−0.0007 (5)
C3	0.0156 (7)	0.0135 (7)	0.0218 (7)	0.0024 (6)	0.0032 (6)	0.0038 (5)
C4	0.0175 (7)	0.0163 (7)	0.0166 (7)	0.0015 (6)	0.0001 (6)	0.0009 (5)
C5	0.0132 (7)	0.0134 (6)	0.0203 (7)	0.0018 (6)	−0.0003 (6)	0.0002 (5)
C6	0.0162 (7)	0.0149 (7)	0.0188 (7)	−0.0005 (6)	0.0008 (6)	0.0020 (5)
C7	0.0230 (8)	0.0214 (8)	0.0218 (8)	−0.0030 (6)	−0.0016 (7)	−0.0046 (6)
C8	0.0334 (10)	0.0252 (8)	0.0179 (8)	−0.0010 (7)	0.0049 (7)	0.0048 (6)
C9	0.0157 (7)	0.0171 (7)	0.0156 (7)	0.0010 (6)	−0.0014 (6)	0.0000 (5)

Geometric parameters (Å, º)

O1—C1	1.3697 (18)	C4—C5	1.4089 (19)
O1—C7	1.4349 (19)	C4—H4	0.9500
O2—C3	1.3653 (17)	C5—C6	1.387 (2)
O2—C8	1.4328 (17)	C5—C9	1.467 (2)
O3—N1	1.4087 (15)	C6—H6	0.9500
O3—H3	0.916 (19)	C7—H7A	0.9800
N1—C9	1.2777 (18)	C7—H7B	0.9800
C1—C2	1.391 (2)	C7—H7C	0.9800
C1—C6	1.397 (2)	C8—H8A	0.9800
C2—C3	1.398 (2)	C8—H8B	0.9800
C2—H2	0.9500	C8—H8C	0.9800
C3—C4	1.386 (2)	C9—H9	0.9500
C1—O1—C7	116.76 (12)	C5—C6—C1	119.26 (14)
C3—O2—C8	117.12 (12)	C5—C6—H6	120.4
N1—O3—H3	103.9 (12)	C1—C6—H6	120.4
C9—N1—O3	110.28 (12)	O1—C7—H7A	109.5
O1—C1—C2	123.64 (14)	O1—C7—H7B	109.5
O1—C1—C6	115.67 (13)	H7A—C7—H7B	109.5
C2—C1—C6	120.68 (14)	O1—C7—H7C	109.5
C1—C2—C3	119.34 (14)	H7A—C7—H7C	109.5
C1—C2—H2	120.3	H7B—C7—H7C	109.5
C3—C2—H2	120.3	O2—C8—H8A	109.5
O2—C3—C4	124.22 (13)	O2—C8—H8B	109.5
O2—C3—C2	114.79 (13)	H8A—C8—H8B	109.5
C4—C3—C2	120.98 (13)	O2—C8—H8C	109.5
C3—C4—C5	118.82 (14)	H8A—C8—H8C	109.5

C3—C4—H4	120.6	H8B—C8—H8C	109.5
C5—C4—H4	120.6	N1—C9—C5	122.77 (13)
C6—C5—C4	120.89 (14)	N1—C9—H9	118.6
C6—C5—C9	123.12 (13)	C5—C9—H9	118.6
C4—C5—C9	115.94 (13)		

C7—O1—C1—C2	0.4 (2)	C3—C4—C5—C6	0.7 (2)
C7—O1—C1—C6	−178.53 (13)	C3—C4—C5—C9	178.22 (13)
O1—C1—C2—C3	179.67 (14)	C4—C5—C6—C1	0.2 (2)
C6—C1—C2—C3	−1.4 (2)	C9—C5—C6—C1	−177.15 (13)
C8—O2—C3—C4	0.0 (2)	O1—C1—C6—C5	179.15 (14)
C8—O2—C3—C2	−179.10 (14)	C2—C1—C6—C5	0.2 (2)
C1—C2—C3—O2	−178.50 (13)	O3—N1—C9—C5	178.28 (12)
C1—C2—C3—C4	2.3 (2)	C6—C5—C9—N1	−12.4 (2)
O2—C3—C4—C5	178.97 (14)	C4—C5—C9—N1	170.15 (14)
C2—C3—C4—C5	−2.0 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N1^{i}	0.916 (19)	1.90 (2)	2.7970 (17)	166.5 (19)

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