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3,5-Dimethoxybenzamide oxime

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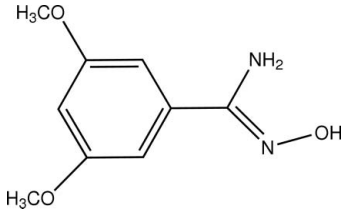
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.057; wR factor = 0.180; data-to-parameter ratio = 14.8.

In the crystal structure of the title compound, $C_9H_{12}N_2O_3$, intermolecular N—H···O and O—H···N hydrogen bonds link the molecules. There is also an intramolecular N—H···O hydrogen bond. The oxime group has an *E* configuration.

Related literature

For general background, see: Marsman *et al.* (1999); Karle *et al.* (1996); Etter *et al.* (1990); Bertolasi *et al.* (1982); Sevagapandian *et al.* (2000); Allen *et al.* (1987). For related structures, see: Hökelek, Batı *et al.* (2001); Hökelek, Zülfikaroğlu *et al.* (2001); Hökelek, Büyükgüngör *et al.* (2004*a*,*b*); Hökelek, Taş *et al.* (2004); Büyükgüngör *et al.* (2003). For related literature, see: Chertanova *et al.* (1994).



Experimental

Crystal data

$C_9H_{12}N_2O_3$
M_r = 196.21
Triclinic, $P\overline{1}$
a = 6.4390 (13) Å
b = 8.0840 (16) Å
c = 10.188 (2) Å
α = 71.29 (3)°
β = 81.60 (3)°
γ = 74.21 (3)°
V = 482.33 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.970, T_{max} = 0.980
2059 measured reflections
1880 independent reflections
1378 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.180
S = 1.11
1880 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*A*···O3	0.86	2.28	2.578 (4)	101
N2—H2*A*···O3^{i}	0.86	2.27	3.083 (4)	158
N2—H2*B*···O2^{ii}	0.86	2.57	3.319 (4)	147
O3—H3*A*···N1^{iii}	0.82	2.04	2.724 (4)	141

Symmetry codes: (i) $-x+3, -y, -z+2$; (ii) $x+1, y, z$; (iii) $-x+2, -y, -z+2$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2275).

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supplementary materials

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3,5-Dimethoxybenzamide oxime

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Comment

The oxime (–C=N—OH) moiety is a functional group that is amphiprotic with a slightly basic N atom and a mildly acidic hydroxyl group. Oxime groups possess stronger hydrogen-bonding capabilities than in alcohols, phenols, and carboxylic acids (Marsman *et al.*, 1999), in which intermolecular hydrogen bonding combines moderate strenght and directionality (Karle *et al.*, 1996) in linking the molecules to form supramolecular structures; this has received considerable attention with respect to directional non-covalent intermolecular interactions (Etter *et al.*, 1990).

The hydrogen-bond systems in the crystal structures of oximes have been analysed and a correlation between a pattern of hydrogen bonding and N—O bond lengths has been suggested (Bertolasi *et al.*, 1982). In general, oxime derivatives are very important compounds in the chemical industry and medicine (Sevagapandian *et al.*, 2000). We report here the crystal structure of the title compound, (I).

In the molecule of the title compound, (I), (Fig. 1), the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). In the oxime moiety, the N1—O3 [1.422 (3) Å], N1—C9 [1.282 (4) Å], C9—C4 [1.479 (4) Å] bonds and N1—C9—C4 [116.0 (3)°] and O3—N1—C9 [111.2 (3)°] angles present no unusual features and are similar to those found in other similar compounds (Hökelek, Batı *et al.*, 2001; Hökelek, Zülfikaroğlu *et al.*, 2001; Hökelek, Büyükgüngör *et al.*, 2004*a*,b; Hökelek, Taş *et al.*, 2004; Büyükgüngör *et al.*, 2003).

The oxime moiety has an E configuration [C4—C9—N1—O3 = −177.7 (3)°; Chertanova *et al.*, 1994]. In this configuration, the oxime group is involved as a donor in intermolecular hydrogen bonding (Table 1). The rings A (C2—C7) and B (N1/N2/O3/C9/H2A) are, of course, planar and the dihedral angle between them is A/B = 37.5 (2)°.

In the crystal structure, intramolecular N—H···O and intermolecular N—H···O and O—H···N hydrogen bonds (Table 1) link the molecules, in which they seem to be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, a mixture of 3,5-dimethoxy -benzonitrile (20 mmol) in ethanol (8 ml), hydroxylamine hydrochloride (20 mmol) in ethanol (6 ml) and potassium carbonate (10 mmol) in water (10 ml) was refluxed for 24 h. After cooling and filtrating, compound (I) was obtained. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH), O—H = 0.82 Å (for OH) and C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*N*,*O*), where x = 1.5 for methyl and OH H, and x = 1.2 for all other H atoms.

Figures

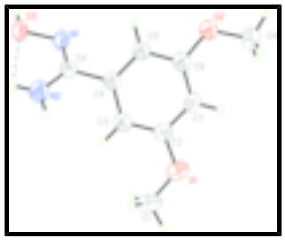

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Intramolecular hydrogen bond is shown as dashed line.

3,5-Dimethoxybenzamide oxime

Crystal data

$C_9H_{12}N_2O_3$	$Z = 2$
$M_r = 196.21$	$F_{000} = 208$
Triclinic, $P\overline{1}$	$D_x = 1.351$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.4390$ (13) Å	Cell parameters from 25 reflections
$b = 8.0840$ (16) Å	$\theta = 10$–14°
$c = 10.188$ (2) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 71.29$ (3)°	$T = 294$ (2) K
$\beta = 81.60$ (3)°	Block, colorless
$\gamma = 74.21$ (3)°	0.30 × 0.20 × 0.20 mm
$V = 482.33$ (17) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.028$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0^\circ$
Monochromator: graphite	$\theta_{min} = 2.1^\circ$
$T = 294(2)$ K	$h = -7 \rightarrow 7$
$\omega/2\theta$ scans	$k = -9 \rightarrow 9$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 12$
$T_{min} = 0.970$, $T_{max} = 0.980$	3 standard reflections
2059 measured reflections	every 120 min
1880 independent reflections	intensity decay: none
1378 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.057$	H-atom parameters constrained

$wR(F^2) = 0.180$	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.9P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} < 0.001$
1880 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
127 parameters	$\Delta\rho_{min} = -0.36$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	1.0788 (4)	0.1642 (4)	0.9438 (3)	0.0451 (7)
O1	1.0049 (5)	0.8092 (4)	0.4557 (3)	0.0641 (8)
C1	1.2181 (7)	0.7582 (6)	0.3939 (4)	0.0601 (11)
H1B	1.2331	0.8433	0.3044	0.090*
H1C	1.2407	0.6406	0.3834	0.090*
H1D	1.3231	0.7565	0.4524	0.090*
O2	0.4585 (4)	0.7003 (3)	0.8199 (3)	0.0522 (7)
C2	0.9512 (5)	0.7037 (4)	0.5843 (3)	0.0435 (8)
N2	1.3735 (4)	0.2953 (4)	0.8772 (3)	0.0529 (8)
H2A	1.4608	0.2016	0.9251	0.063*
H2B	1.4198	0.3887	0.8296	0.063*
O3	1.2317 (4)	0.0190 (3)	1.0254 (3)	0.0537 (7)
H3A	1.1736	−0.0635	1.0674	0.081*
C3	1.0923 (5)	0.5541 (4)	0.6617 (3)	0.0414 (8)
H3B	1.2341	0.5181	0.6278	0.050*
C4	1.0177 (5)	0.4578 (4)	0.7923 (3)	0.0359 (7)
C5	0.8059 (5)	0.5116 (4)	0.8431 (3)	0.0392 (7)
H5A	0.7575	0.4478	0.9303	0.047*
C6	0.6679 (5)	0.6608 (4)	0.7627 (3)	0.0411 (8)
C7	0.7402 (6)	0.7582 (5)	0.6338 (4)	0.0454 (8)
H7A	0.6473	0.8597	0.5809	0.055*
C8	0.3158 (6)	0.8628 (5)	0.7501 (4)	0.0551 (10)
H8A	0.1769	0.8740	0.8004	0.083*
H8B	0.3006	0.8616	0.6581	0.083*
H8C	0.3731	0.9629	0.7447	0.083*

C9	1.1641 (5)	0.2958 (4)	0.8768 (3)	0.0369 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0325 (14)	0.0394 (15)	0.0496 (17)	−0.0041 (12)	−0.0096 (12)	0.0048 (13)
O1	0.0639 (17)	0.0624 (17)	0.0438 (15)	−0.0107 (14)	0.0047 (13)	0.0071 (13)
C1	0.068 (3)	0.068 (3)	0.045 (2)	−0.035 (2)	0.0086 (18)	−0.0075 (19)
O2	0.0359 (13)	0.0527 (15)	0.0506 (15)	−0.0005 (11)	0.0003 (11)	−0.0015 (12)
C2	0.0449 (19)	0.0402 (18)	0.0390 (18)	−0.0099 (15)	0.0006 (14)	−0.0047 (14)
N2	0.0359 (15)	0.0523 (18)	0.060 (2)	−0.0132 (13)	−0.0112 (14)	0.0030 (15)
O3	0.0338 (13)	0.0464 (14)	0.0616 (16)	−0.0019 (10)	−0.0133 (11)	0.0084 (12)
C3	0.0340 (16)	0.0419 (18)	0.0446 (19)	−0.0070 (14)	0.0002 (14)	−0.0109 (15)
C4	0.0302 (15)	0.0356 (16)	0.0384 (17)	−0.0061 (13)	−0.0038 (13)	−0.0071 (13)
C5	0.0341 (16)	0.0375 (17)	0.0411 (18)	−0.0076 (13)	−0.0016 (13)	−0.0059 (14)
C6	0.0337 (16)	0.0416 (18)	0.0445 (19)	−0.0059 (14)	−0.0027 (14)	−0.0105 (15)
C7	0.0436 (19)	0.0382 (18)	0.0428 (19)	−0.0020 (15)	−0.0077 (15)	−0.0005 (15)
C8	0.049 (2)	0.049 (2)	0.055 (2)	0.0023 (17)	−0.0046 (17)	−0.0100 (18)
C9	0.0306 (15)	0.0404 (17)	0.0371 (17)	−0.0077 (13)	−0.0030 (13)	−0.0085 (14)

Geometric parameters (Å, °)

N1—C9	1.282 (4)	N2—H2B	0.8600
N1—O3	1.422 (3)	O3—H3A	0.8200
O1—C2	1.369 (4)	C3—C4	1.400 (4)
O1—C1	1.434 (5)	C3—H3B	0.9300
C1—H1B	0.9600	C4—C5	1.390 (4)
C1—H1C	0.9600	C4—C9	1.479 (4)
C1—H1D	0.9600	C5—C6	1.382 (4)
O2—C6	1.384 (4)	C5—H5A	0.9300
O2—C8	1.418 (4)	C6—C7	1.385 (5)
C2—C7	1.382 (5)	C7—H7A	0.9300
C2—C3	1.382 (5)	C8—H8A	0.9600
N2—C9	1.347 (4)	C8—H8B	0.9600
N2—H2A	0.8600	C8—H8C	0.9600
C9—N1—O3	111.2 (3)	C5—C4—C9	119.5 (3)
C2—O1—C1	118.4 (3)	C3—C4—C9	119.9 (3)
O1—C1—H1B	109.5	C6—C5—C4	119.4 (3)
O1—C1—H1C	109.5	C6—C5—H5A	120.3
H1B—C1—H1C	109.5	C4—C5—H5A	120.3
O1—C1—H1D	109.5	C5—C6—O2	115.2 (3)
H1B—C1—H1D	109.5	C5—C6—C7	120.6 (3)
H1C—C1—H1D	109.5	O2—C6—C7	124.2 (3)
C6—O2—C8	117.9 (3)	C2—C7—C6	119.6 (3)
O1—C2—C7	114.7 (3)	C2—C7—H7A	120.2
O1—C2—C3	124.2 (3)	C6—C7—H7A	120.2
C7—C2—C3	121.1 (3)	O2—C8—H8A	109.5
C9—N2—H2A	120.0	O2—C8—H8B	109.5

C9—N2—H2B	120.0	H8A—C8—H8B	109.5
H2A—N2—H2B	120.0	O2—C8—H8C	109.5
N1—O3—H3A	109.5	H8A—C8—H8C	109.5
C2—C3—C4	118.7 (3)	H8B—C8—H8C	109.5
C2—C3—H3B	120.7	N1—C9—N2	125.0 (3)
C4—C3—H3B	120.7	N1—C9—C4	116.0 (3)
C5—C4—C3	120.7 (3)	N2—C9—C4	119.0 (3)
C1—O1—C2—C7	−177.5 (3)	C8—O2—C6—C7	−7.9 (5)
C1—O1—C2—C3	2.7 (6)	O1—C2—C7—C6	179.7 (3)
O1—C2—C3—C4	179.7 (3)	C3—C2—C7—C6	−0.5 (6)
C7—C2—C3—C4	0.0 (5)	C5—C6—C7—C2	1.1 (5)
C2—C3—C4—C5	0.0 (5)	O2—C6—C7—C2	−177.9 (3)
C2—C3—C4—C9	179.2 (3)	O3—N1—C9—N2	3.0 (5)
C3—C4—C5—C6	0.6 (5)	O3—N1—C9—C4	−177.7 (3)
C9—C4—C5—C6	−178.6 (3)	C5—C4—C9—N1	38.3 (5)
C4—C5—C6—O2	177.9 (3)	C3—C4—C9—N1	−141.0 (3)
C4—C5—C6—C7	−1.2 (5)	C5—C4—C9—N2	−142.4 (3)
C8—O2—C6—C5	173.0 (3)	C3—C4—C9—N2	38.4 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O3	0.86	2.28	2.578 (4)	101
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Fig. 1