(E)-2,4,5-Trimethoxybenzaldehyde oxime

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The title compound, $C_{10}H_{13}NO_4$, which exists as the E isomer, crystallizes with two independent molecules in the asymmetric unit. The bond lengths and angles in both molecules are normal. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds, which link the molecules into cyclic tetramers.

Comment

Substituted benzaldehyde oxime is an important intermediate in organic synthesis (Xu & Jin, 1999), existing in two isomeric forms, viz. Z and E (Sharghi & Sarvari, 2001). We report here the crystal structure of the title compound, (I).

(I)
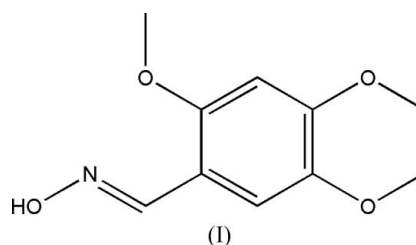
In (I) (Fig. 1), all bond lengths and angles are normal (Allen et al., 1987) and in good agreement with those reported previously (Jerslev, 1983; Jensen, 1970). Atoms O1/O2/O4/C1–C9 lie in the same plane, the largest deviation being 0.040 (2) Å for atom C7, while in the second independent molecule, atoms C11–C20 and O6–O8 are essentially coplanar, the largest deviation from the mean plane being 0.046 (3) Å for atom C19. Intermolecular O—H···O hydrogen bonds (Table 2) link the molecules into nearly planar cyclic tetramers (Fig. 2).
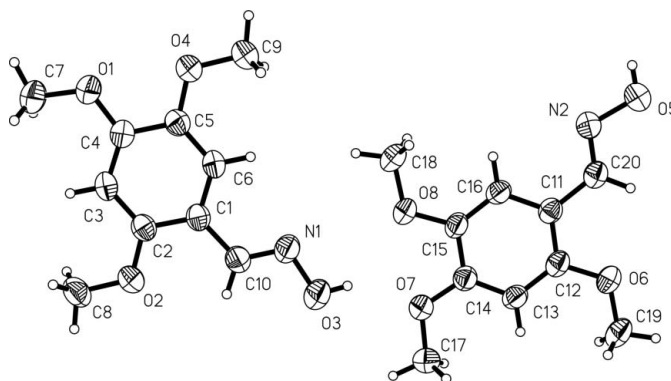
Figure 1
The asymmetric unit of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

Experimental

The title compound was synthesized by the reaction of 2,4,5-trimethoxybenzaldehyde (0.01 mol) with hydroxylamine hydrochloride (0.01 mol) in the presence of sodium carbonate (0.01 mol) in a water–methanol solution (20 ml; 1:5 *v*/*v*) at room temperature (3.5 h). After dilution with water, the solution was extracted with dichloromethane, and the organic phase was evaporated to afford the title product in 92% isolated yield (1.94 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of an ethyl acetate–petroleum ether (1:1 *v*/*v*) solution at room temperature over a period of two weeks.

Crystal data

$C_{10}H_{13}NO_4$	$V = 1081.04$ (2) Å^3
$M_r = 211.21$	$Z = 4$
Triclinic, $P\bar{1}$	$D_x = 1.298$ Mg m^{-3}
$a = 7.44280$ (10) Å	Mo $K\alpha$ radiation
$b = 10.71890$ (10) Å	$\mu = 0.10$ mm^{-1}
$c = 14.0290$ (2) Å	$T = 293$ (2) K
$\alpha = 87.5830$ (10)°	Block, colorless
$\beta = 88.4510$ (10)°	$0.36 \times 0.30 \times 0.22$ mm
$\gamma = 75.2140$ (10)°	

Data collection

Bruker SMART CCD area-detector diffractometer	12599 measured reflections
	3805 independent reflections
φ and ω scans	3079 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.016$
$T_{min} = 0.965$, $T_{max} = 0.978$	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$+ 0.1378P]$
$wR(F^2) = 0.106$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
3805 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
272 parameters	$\Delta\rho_{min} = -0.11$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXTL*
	Extinction coefficient: 0.0058 (13)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5A···O1^i	0.82	2.14	2.8676 (15)	148
O5—H5A···O4^i	0.82	2.29	2.9832 (15)	142

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

H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å and O—H = 0.82 Å, the hydroxy H atom being initially
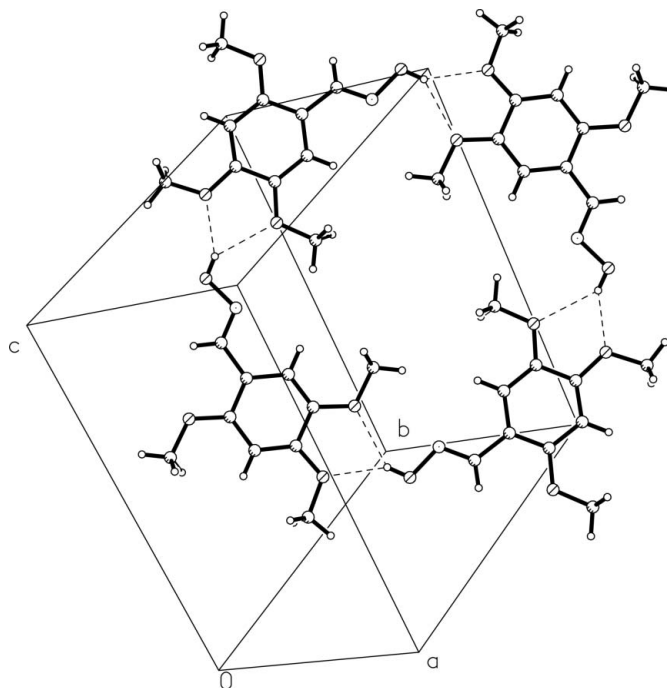

Figure 2
Partial packing plot, showing the hydrogen-bonded (dashed lines) tetramer.

located in a difference Fourier map. They were refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ for the aryl H atoms and $1.5U_{eq}(C,O)$ for the hydroxy and methyl H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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