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Key indicators

Single-crystal X-ray study
 $T = 291$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.044
 wR factor = 0.107
Data-to-parameter ratio = 14.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

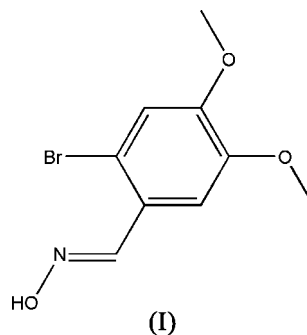
(E)-2-Bromo-4,5-dimethoxybenzaldehyde oxime

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The title compound, $\text{C}_9\text{H}_{10}\text{BrNO}_3$, 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 packing is stabilized by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, which link the molecules into circular tetramers, and by weak $\pi-\pi$ stacking interactions.

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).



Compound (I) crystallizes with two independent molecules in the asymmetric unit (Fig. 1). The bond lengths and angles in both molecules (Table 1) are in agreement with the values reported previously (Jerslev, 1983; Jensen, 1970). The deviation of atom C17 from the mean plane formed by C10–C16/C18/N2/O4–O6/Br1 is 0.106 (3) Å, while in the second independent molecule the atoms C1–C9, N1, O1–O3 and Br2 are essentially coplanar, the largest deviation from the mean plane being 0.040 (2) Å for atom C8. Intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2) link the molecules into nearly planar circular tetramers (Fig. 2). The relatively short distance of 3.829 (4) Å between the centroids of benzene rings C1–C6 and C10–C15 [at $(-x, -\frac{1}{2} + y, \frac{1}{2} - z)$] indicates the presence of weak $\pi-\pi$ interactions, which contribute to the stability of the crystal packing.

Experimental

The title compound was synthesized by the reaction of 2-bromo-4,5-dimethoxybenzaldehyde (0.01 mol) with hydroxylamine hydrochloride (0.01 mol) in the presence of sodium carbonate (0.01 mol) in an aqueous methanol solution (20 ml) at room temperature (3.5 h). After dilution with water, the aqueous solution was extracted with

dichloromethane, and the organic phase was evaporated to afford the title product in 85% yield (2.21 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a mixture of ethyl acetate and petroleum ether (1:1 v/v) at room temperature over a period of two weeks.

Crystal data

$C_9H_{10}BrNO_3$
 $M_r = 260.09$
 Monoclinic, $P2_1/c$
 $a = 8.0870$ (14) Å
 $b = 9.6175$ (17) Å
 $c = 26.795$ (5) Å
 $\beta = 94.964$ (3)°
 $V = 2076.2$ (6) Å³

$Z = 8$
 $D_x = 1.664$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 3.94$ mm⁻¹
 $T = 291$ (2) K
 Block, colourless
 $0.34 \times 0.31 \times 0.25$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.348$, $T_{\max} = 0.439$
 (expected range = 0.296–0.373)

10481 measured reflections
 3653 independent reflections
 2458 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.107$
 $S = 1.04$
 3653 reflections
 253 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.3059P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.66$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.40$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Br1—C10	1.913 (4)	N1—C9	1.233 (5)
Br2—C1	1.903 (4)	N2—C18	1.239 (5)
C3—O1—C7	117.8 (4)	C9—N1—O3	112.2 (4)
C4—O2—C8	117.5 (3)	C18—N2—O6	112.1 (4)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A ⁱ ···O4 ⁱ	0.84	2.36	3.098 (5)	147
O3—H3A ⁱ ···O5 ⁱ	0.84	2.03	2.747 (5)	142
O6—H6A ⁱⁱ ···O1 ⁱⁱ	0.84	2.19	2.942 (4)	150
O6—H6A ⁱⁱ ···O2 ⁱⁱ	0.84	2.33	3.038 (4)	142

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

All H atoms were placed in calculated positions, with C—H = 0.95–0.98 Å and O—H = 0.84 Å, and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the aryl H atoms and $1.5U_{\text{eq}}(\text{O}, \text{C})$ for the hydroxyl and methyl H atoms.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve

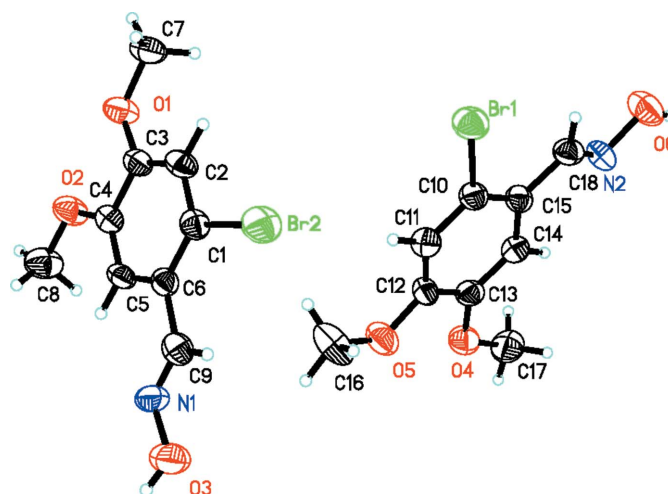


Figure 1

View of the asymmetric unit of (I), with displacement ellipsoids drawn at the 40% probability level.

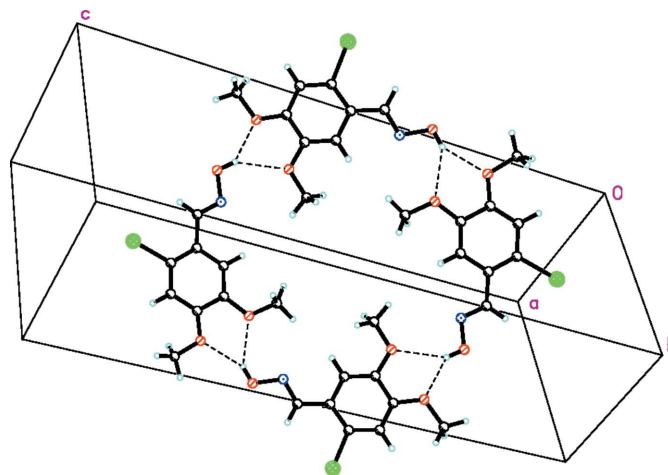


Figure 2

Part of the crystal packing, showing the hydrogen-bonded (dashed lines) tetramer.

structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1999); software used to prepare material for publication: SHELXTL.

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