

## Bis(2-hydroxybenzaldehyde oxime) *O,O'*-butane-1,4-diyl dicarbonyl ether

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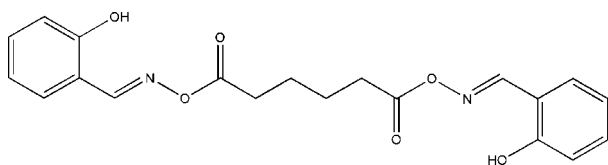
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.047;  $wR$  factor = 0.099; data-to-parameter ratio = 18.5.

The molecule of the title compound,  $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_6$ , lies across a crystallographic inversion centre, the asymmetric unit comprising one half-molecule. An intramolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bond generates a six-membered ring, producing an  $S(6)$  ring motif. Pairs of intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds link neighbouring molecules into a layer with  $R_2^2(38)$  ring motif. The crystal structure is further stabilized by the intermolecular  $\text{C}-\text{H}\cdots\pi$  interactions.

### Related literature

For bond-length data, see Allen *et al.* (1987). For hydrogen bond motifs, see Bernstein *et al.* (1995). For Schiff bases, see: Granovski *et al.*, (1993). For the synthesis, see: Hosseini Sarvari (2003).



### Experimental

#### Crystal data

$\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_6$   
 $M_r = 384.38$   
 Monoclinic,  $C2/c$   
 $a = 13.0293$  (11) Å  
 $b = 5.5464$  (4) Å  
 $c = 25.538$  (2) Å  
 $\beta = 91.348$  (7)°

$V = 1845.0$  (3) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 120$  K  
 $0.45 \times 0.11 \times 0.10$  mm

#### Data collection

STOE IPDSII diffractometer  
 Absorption correction: numerical  
 ( $X\text{-RED32}$ ; Stoe & Cie (2005))  
 $T_{\text{min}} = 0.956$ ,  $T_{\text{max}} = 0.985$

10357 measured reflections  
 2493 independent reflections  
 2098 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.046$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$   
 $wR(F^2) = 0.099$   
 $S = 1.10$   
 2493 reflections  
 135 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.20$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.91 (3)	1.79 (3)	2.5836 (15)	144 (2)
$\text{C4}-\text{H4}\cdots\text{O1}^{\text{i}}$	0.93	2.45	3.1874 (17)	136
$\text{C2}-\text{H2}\cdots\text{Cg1}^{\text{ii}}$	0.93	2.75	3.4659 (14)	134

Symmetry codes: (i)  $x - \frac{1}{2}, y + \frac{1}{2}, z$ ; (ii)  $x + 1, -y - 1, z - \frac{1}{2}$ . Cg1 is the centroid of the C1-C6 benzene ring.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2778).

### References

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**supplementary materials**

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## Bis(2-hydroxybenzaldehyde oxime) *O,O'*-butane-1,4-diyl dicarbonyl ether

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### Comment

Schiff base compounds are some of the most important stereochemical models in transition metal coordination chemistry, with their ease of preparation and structural variations (Granovski *et al.*, 1993). In continuation of our works on the synthesis and structural characterization of Schiff base ligands here we report the structure of the title compound.

The asymmetric unit of the title compound, Fig. 1, lies across a crystallographic inversion centre. Intramolecular O—H $\cdots$ N hydrogen bond generates a six-membered ring, producing an *S(6)* ring motif (Bernstein *et al.*, 1995). Pairs of intermolecular C—H $\cdots$ O hydrogen bonds link neighbouring molecules into a layer with *R*<sup>2</sup><sub>2</sub>(38) ring motif (Fig. 2). The crystal structure is further stabilized by the intermolecular C—H $\cdots$  $\pi$  interactions [Cg1 is the centroid of the C1–C6 benzene ring] (Table 1).

### Experimental

An ethyl acetate solution (40 ml) of salicylaldehyde oxime (2 mmol, 768 mg) was treated with butanedicarboxylic acid chloride (1 mmol, 183 mg) at -5 °C. The mixture was stirred for 30 min and then filtered and the resulting white powder dried under air (Hosseini Sarvari, 2003). Single crystals suitable for X-ray diffraction were obtained from an ethanol solution.

### Refinement

The O-bound and C7 bound hydrogen atoms were located from the difference Fourier map and refined freely. The rest of the hydrogen atoms were positioned geometrically [C—H = 0.93–97 Å] and refined using a riding model approximation with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

### Figures



Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. Intramolecular hydrogen bonds are shown as dashed lines. Symmetry code for A suffix:  $-x + 1/2, -y + 1/2, -z + 1$ .

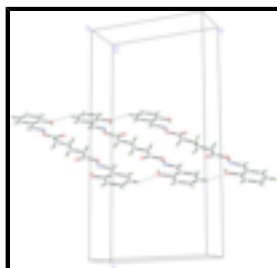


Fig. 2. The crystal packing of the title compound, showing linking of the molecules into a layer through *R*<sup>2</sup><sub>2</sub>(38) motifs. Intermolecular interactions are drawn as dashed lines.

## Bis(2-hydroxybenzaldehyde oxime) *O,O'*-(butane-1,4-diyl)dicarbonyl ether

### Crystal data

$C_{20}H_{20}N_2O_6$	$F_{000} = 808$
$M_r = 384.38$	$D_x = 1.384 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
Hall symbol: $-C 2yc$	$\lambda = 0.71073 \text{ \AA}$
$a = 13.0293 (11) \text{ \AA}$	Cell parameters from 1302 reflections
$b = 5.5464 (4) \text{ \AA}$	$\theta = 3.1\text{--}29.2^\circ$
$c = 25.538 (2) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\beta = 91.348 (7)^\circ$	$T = 120 \text{ K}$
$V = 1845.0 (3) \text{ \AA}^3$	Needle, colourless
$Z = 4$	$0.45 \times 0.11 \times 0.10 \text{ mm}$

### Data collection

STOE IPDS II diffractometer	2493 independent reflections
Radiation source: fine-focus sealed tube	2098 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.046$
Detector resolution: $0.15 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 29.2^\circ$
$T = 120 \text{ K}$	$\theta_{\text{min}} = 3.1^\circ$
rotation method scans	$h = -17 \rightarrow 17$
Absorption correction: numerical (X-RED32; Stoe & Cie (2005))	$k = -7 \rightarrow 7$
$T_{\text{min}} = 0.956, T_{\text{max}} = 0.985$	$l = -35 \rightarrow 34$
10357 measured reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 1.8216P]$
$S = 1.10$	where $P = (F_o^2 + 2F_c^2)/3$
2493 reflections	$(\Delta/\sigma)_{\text{max}} = 0.004$
135 parameters	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
	Extinction correction: none

*Special details*

**Experimental.** The low-temperature data was collected with the Oxford Cryosystem Cobra low-temperature attachment.

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.79600 (9)	-0.3740 (2)	0.32698 (5)	0.0186 (3)
C2	0.73433 (10)	-0.5276 (2)	0.29667 (5)	0.0204 (3)
H2	0.7620	-0.6677	0.2828	0.025*
C3	0.63188 (10)	-0.4721 (2)	0.28720 (5)	0.0206 (3)
H3	0.5909	-0.5768	0.2674	0.025*
C4	0.58953 (9)	-0.2610 (3)	0.30699 (5)	0.0212 (3)
H4	0.5207	-0.2245	0.3004	0.025*
C5	0.65095 (9)	-0.1061 (2)	0.33650 (5)	0.0192 (3)
H5	0.6230	0.0355	0.3495	0.023*
C6	0.75482 (9)	-0.1590 (2)	0.34720 (5)	0.0168 (2)
C7	0.81678 (10)	0.0064 (2)	0.37953 (5)	0.0193 (3)
H7	0.7866 (13)	0.154 (3)	0.3912 (7)	0.027 (4)*
C8	1.05836 (10)	0.0434 (2)	0.43627 (5)	0.0204 (3)
C9	1.11260 (10)	0.2321 (2)	0.46898 (5)	0.0208 (3)
H9A	1.0741	0.2617	0.5004	0.025*
H9B	1.1155	0.3816	0.4494	0.025*
C10	1.22133 (10)	0.1530 (2)	0.48435 (5)	0.0206 (3)
H10B	1.2588	0.1167	0.4529	0.025*
H10A	1.2181	0.0069	0.5051	0.025*
N1	0.90918 (8)	-0.0542 (2)	0.39115 (4)	0.0224 (3)
O1	0.89508 (7)	-0.4424 (2)	0.33553 (4)	0.0289 (3)
H1	0.9268 (18)	-0.333 (5)	0.3572 (10)	0.062 (7)*
O2	0.96033 (7)	0.11970 (18)	0.42331 (4)	0.0223 (2)
O3	1.09270 (8)	-0.14750 (19)	0.42343 (4)	0.0271 (2)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0151 (5)	0.0196 (6)	0.0210 (6)	0.0028 (4)	-0.0008 (4)	0.0010 (5)
C2	0.0222 (6)	0.0177 (6)	0.0213 (6)	0.0026 (5)	0.0002 (5)	-0.0012 (5)
C3	0.0191 (6)	0.0219 (6)	0.0207 (6)	-0.0031 (5)	-0.0022 (4)	-0.0009 (5)

## supplementary materials

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C4	0.0146 (5)	0.0261 (7)	0.0227 (6)	0.0016 (5)	-0.0013 (4)	-0.0001 (5)
C5	0.0172 (6)	0.0199 (6)	0.0204 (6)	0.0031 (5)	0.0011 (4)	-0.0008 (5)
C6	0.0163 (5)	0.0171 (6)	0.0170 (5)	-0.0003 (4)	-0.0008 (4)	0.0010 (5)
C7	0.0202 (6)	0.0185 (6)	0.0191 (6)	-0.0007 (5)	-0.0006 (4)	-0.0001 (5)
C8	0.0194 (6)	0.0224 (7)	0.0192 (6)	-0.0037 (5)	-0.0019 (4)	0.0009 (5)
C9	0.0209 (6)	0.0207 (6)	0.0206 (6)	-0.0033 (5)	-0.0025 (5)	-0.0011 (5)
C10	0.0204 (6)	0.0216 (6)	0.0197 (6)	-0.0032 (5)	-0.0035 (5)	-0.0012 (5)
N1	0.0197 (5)	0.0231 (6)	0.0241 (6)	-0.0047 (4)	-0.0043 (4)	-0.0052 (5)
O1	0.0170 (5)	0.0292 (6)	0.0402 (6)	0.0085 (4)	-0.0070 (4)	-0.0111 (5)
O2	0.0199 (5)	0.0217 (5)	0.0250 (5)	-0.0030 (4)	-0.0048 (4)	-0.0048 (4)
O3	0.0250 (5)	0.0231 (5)	0.0330 (5)	0.0006 (4)	-0.0067 (4)	-0.0060 (4)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C1—O1	1.3583 (15)	C7—H7	0.958 (18)
C1—C2	1.3935 (18)	C8—O3	1.1985 (17)
C1—C6	1.4105 (18)	C8—O2	1.3784 (16)
C2—C3	1.3857 (17)	C8—C9	1.5047 (18)
C2—H2	0.9300	C9—C10	1.5255 (18)
C3—C4	1.3943 (19)	C9—H9A	0.9700
C3—H3	0.9300	C9—H9B	0.9700
C4—C5	1.3850 (18)	C10—C10 <sup>i</sup>	1.526 (2)
C4—H4	0.9300	C10—H10B	0.9700
C5—C6	1.4054 (17)	C10—H10A	0.9700
C5—H5	0.9300	N1—O2	1.4226 (14)
C6—C7	1.4639 (17)	O1—H1	0.91 (3)
C7—N1	1.2782 (17)		
O1—C1—C2	116.85 (12)	C6—C7—H7	119.1 (10)
O1—C1—C6	123.07 (12)	O3—C8—O2	123.73 (12)
C2—C1—C6	120.08 (11)	O3—C8—C9	126.36 (12)
C3—C2—C1	120.10 (12)	O2—C8—C9	109.91 (11)
C3—C2—H2	120.0	C8—C9—C10	111.35 (11)
C1—C2—H2	120.0	C8—C9—H9A	109.4
C2—C3—C4	120.75 (12)	C10—C9—H9A	109.4
C2—C3—H3	119.6	C8—C9—H9B	109.4
C4—C3—H3	119.6	C10—C9—H9B	109.4
C5—C4—C3	119.31 (11)	H9A—C9—H9B	108.0
C5—C4—H4	120.3	C9—C10—C10 <sup>i</sup>	111.85 (14)
C3—C4—H4	120.3	C9—C10—H10B	109.2
C4—C5—C6	121.19 (12)	C10 <sup>i</sup> —C10—H10B	109.2
C4—C5—H5	119.4	C9—C10—H10A	109.2
C6—C5—H5	119.4	C10 <sup>i</sup> —C10—H10A	109.2
C5—C6—C1	118.56 (11)	H10B—C10—H10A	107.9
C5—C6—C7	119.65 (12)	C7—N1—O2	112.42 (11)
C1—C6—C7	121.79 (11)	C1—O1—H1	109.0 (15)
N1—C7—C6	118.03 (12)	C8—O2—N1	110.45 (10)
N1—C7—H7	122.9 (10)		
O1—C1—C2—C3	178.68 (12)	C2—C1—C6—C7	179.53 (12)

C6—C1—C2—C3	-1.30 (19)	C5—C6—C7—N1	175.82 (12)
C1—C2—C3—C4	1.0 (2)	C1—C6—C7—N1	-3.07 (19)
C2—C3—C4—C5	-0.1 (2)	O3—C8—C9—C10	0.66 (19)
C3—C4—C5—C6	-0.6 (2)	O2—C8—C9—C10	179.67 (10)
C4—C5—C6—C1	0.30 (19)	C8—C9—C10—C10 <sup>i</sup>	177.66 (13)
C4—C5—C6—C7	-178.63 (12)	C6—C7—N1—O2	-179.06 (10)
O1—C1—C6—C5	-179.35 (12)	O3—C8—O2—N1	-2.57 (18)
C2—C1—C6—C5	0.63 (18)	C9—C8—O2—N1	178.39 (10)
O1—C1—C6—C7	-0.4 (2)	C7—N1—O2—C8	178.03 (11)

Symmetry codes: (i)  $-x+5/2, -y+1/2, -z+1$ .

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1 $\cdots$ N1	0.91 (3)	1.79 (3)	2.5836 (15)	144 (2)
C4—H4 $\cdots$ O1 <sup>ii</sup>	0.93	2.45	3.1874 (17)	136
C2—H2 $\cdots$ Cg1 <sup>iii</sup>	0.93	2.75	3.4659 (14)	134

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

Fig. 1

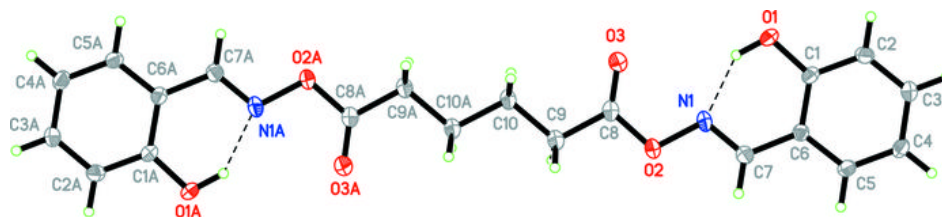




Fig. 2

