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

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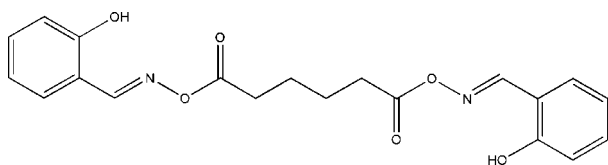
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 Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.047; wR factor = 0.099; data-to-parameter ratio = 18.5.

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) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 120$ 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⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

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).

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supporting information

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

Bijan Etemadi, Reza Kia, Hashem Sharghi and Mona Hosseini Sarvari

S1. 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*²₂(38) ring motif (Fig. 2). The crystal structure is further stabilized by the intermolecular C—H \cdots π interactions [*Cg*1 is the centroid of the C1—C6 benzene ring] (Table 1).

S2. Experimental

An ethyl acetate solution (40 ml) of salicylaldoxime (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.

S3. 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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

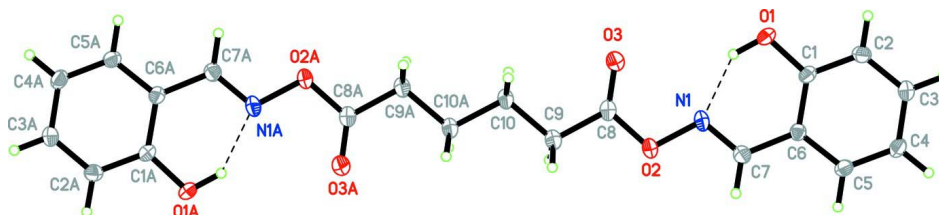
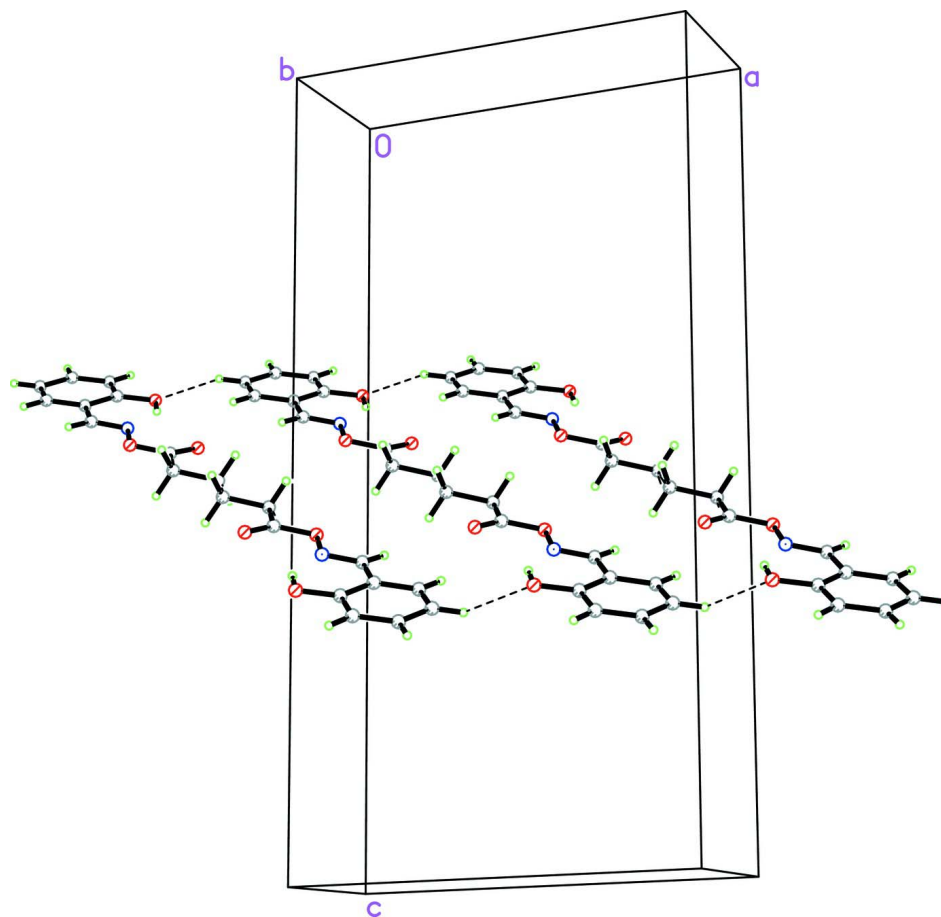


Figure 1

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: $-x + 1/2, -y + 1/2, -z + 1$].

**Figure 2**

The crystal packing of the title compound, showing linking of the molecules into a layer through $R^2_2(38)$ motifs. Intermolecular interactions are drawn as dashed lines.

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

Crystal data

$C_{20}H_{20}N_2O_6$
 $M_r = 384.38$
 Monoclinic, $C2/c$
 Hall symbol: $-C 2yc$
 $a = 13.0293$ (11) Å
 $b = 5.5464$ (4) Å
 $c = 25.538$ (2) Å
 $\beta = 91.348$ (7)°
 $V = 1845.0$ (3) Å³
 $Z = 4$

$F(000) = 808$
 $D_x = 1.384$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 1302 reflections
 $\theta = 3.1$ – 29.2 °
 $\mu = 0.10$ mm⁻¹
 $T = 120$ K
 Needle, colourless
 $0.45 \times 0.11 \times 0.10$ mm

Data collection

STOE IPDS II
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 0.15 pixels mm⁻¹

rotation method scans
 Absorption correction: numerical
 (X -RED32; Stoe & Cie (2005))
 $T_{\min} = 0.956$, $T_{\max} = 0.985$
 41515 measured reflections

2493 independent reflections
 2098 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.091$
 $\theta_{\text{max}} = 29.2^\circ$, $\theta_{\text{min}} = 3.1^\circ$

$h = -17 \rightarrow 17$
 $k = -7 \rightarrow 7$
 $l = -35 \rightarrow 34$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.099$
 $S = 1.10$
 2493 reflections
 135 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
 $w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 1.8216P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.004$
 $\Delta\rho_{\text{max}} = 0.31 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å²)

	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)
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 (Å, °)

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 ⁱ	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 ⁱ	111.85 (14)
C3—C4—H4	120.3	C9—C10—H10B	109.2
C4—C5—C6	121.19 (12)	C10 ⁱ —C10—H10B	109.2
C4—C5—H5	119.4	C9—C10—H10A	109.2
C6—C5—H5	119.4	C10 ⁱ —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 ⁱ	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 code: (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 ⁱⁱ	0.93	2.45	3.1874 (17)	136
C2—H2 \cdots Cg1 ⁱⁱⁱ	0.93	2.75	3.4659 (14)	134

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