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2,2′-Dihydroxybiphenyl-3,3′-dicarbaldehyde dioxime

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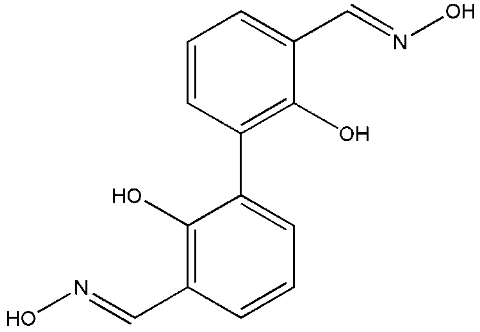
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.146; data-to-parameter ratio = 14.0.

The molecule of the title compound, $C_{14}H_{12}N_2O_4$, lies across a crystallographic inversion centre situated at the mid-point of the C—C intra-annular bond. The molecule is not planar, the dihedral angle between the aromatic rings being 50.1 (1)°. The oxime group is in an E position with respect to the –OH group and forms an intramolecular O—H···N hydrogen bond. In the crystal structure, intermolecular O—H···O hydrogen bonds link molecules into chains propagating along [001]. The crystal structure is further stabilized by intermolecular stacking interactions between the rings [centroid-to-centroid distance = 3.93 (1) Å], resulting in layers parallel to the bc plane.

Related literature

For the use of oximes as chelating ligands in coordination and analytical chemistry and extraction metallurgy, see: Kukushkin *et al.* (1996); Chaudhuri (2003). For the use of oxime ligands to obtain polynuclear compounds in the fields of molecular magnetism and supramolecular chemistry, see: Cervera *et al.* (1997); Costes *et al.* (1998). Oxime-containing ligands have been found to efficiently stabilize high oxidation states of metal ions such as Cu(III) and Ni(III), see: Fritsky *et al.* (2006); Kanderal *et al.* (2005). For C=N and N—O bond lengths in oximes, see: Mokhir *et al.* (2002); Onindo *et al.* (1995); Sliva *et al.* (1997). For the synthesis of 2,2′-dihydroxybiphenyl-3,3′-dicarbaldehyde, see: Wünnemann *et al.* (2008).



Experimental

Crystal data

$C_{14}H_{12}N_2O_4$
M_r = 272.26
Monoclinic, $C2/c$
a = 24.2780 (14) Å
b = 3.9279 (4) Å
c = 16.6466 (12) Å
β = 129.652 (6)°
V = 1222.2 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 120 K
0.19 × 0.09 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.976, T_{max} = 0.993
4331 measured reflections
1388 independent reflections
812 reflections with $I > 2\sigma(I)$
R_{int} = 0.073

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.146
S = 1.02
1388 reflections
99 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.91 (3)	1.79 (3)	2.609 (2)	148 (2)
O2—H2···O1^{i}	1.00 (3)	1.96 (3)	2.871 (2)	151 (3)

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

Data collection: *COLLECT* (Bruker–Nonius, 2004); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2095).

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supplementary materials

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2,2'-Dihydroxybiphenyl-3,3'-dicarbaldehyde dioxime

E. Golovnia, **E. V. Prisyazhnaya**, **T. S. Iskenderov**, **M. Haukka** and **I. O. Fritsky**

Comment

Oximes are a traditional class of chelating ligands widely used in coordination and analytical chemistry and extraction metallurgy (Kukushkin *et al.*, 1996; Chaudhuri, 2003). Due to marked ability to from bridges between metal ions oxime ligands may be used for obtaining polynuclear compounds in the field of molecular magnetism and supramolecular chemistry (Cervera *et al.*, 1997; Costes *et al.*, 1998). Also, the oxime ligands are strong donors and therefore the oxime-containing ligands were found to efficiently stabilize high oxidation states of metal ions like Cu(III) and Ni(III) (Kanderal *et al.*, 2005; Fritsky *et al.*, 2006). The presence of additional donor groups together with the oxime group in the ligand molecule may result in significant increase of chelating efficiency and ability to form polynuclear complexes. The present investigation is dedicated to the study of the molecular structure of the title compound (**I**) which is a new polynuclear ligand containing both oxime and phenolic functions.

Molecules of **I** lie across a crystallographic inversion centre situated in the midpoint of the C—C intra-annular bond (Fig. 1). The molecule is not planar, the dihedral angle between the phenyl rings is 50.1 (1)°. The oxime group is in the *E*-position with respect to the OH group and forms an intramolecular O—H···N hydrogen bond. The C=N and N—O bond lengths are normal for oximes (Onindo *et al.*, 1995; Sliva *et al.*, 1997; Mokhir *et al.*, 2002).

In the crystal structure, intermolecular O—H···O hydrogen bonds between the phenolic groups of the translational molecules link the molecules into chains propagating along [001]. The crystal structure is further stabilized by the intermolecular stacking interactions between the phenyl rings with centroid-to-centroid distances equal to 3.93 Å resulting in layers parallel to the *yz* plane (Fig. 2).

Experimental

2,2'-Dihydroxybiphenyl-3,3'-dicarbaldehyde (2.57 g, 10 mmol) dissolved in 20 ml of methanol was added to a solution obtained by dissolving sodium (0.51 g, 22 mmol) in 10 ml of methanol with addition of hydroxylamine hydrochloride (1.52 g, 22 mmol). The mixture was stirred for 30 min and filtered. In 2–3 h the filtrate produced white crystalline precipitate which was filtered off and dried. Yield 85%. Single crystals suitable for X-ray analysis were obtained as a result of recrystallization from aqueous (40%) ethanol. 2,2'-Dihydroxybiphenyl-3,3'-dicarbaldehyde was synthesized according to the reported method (Wünnemann *et al.*, 2008).

Refinement

The O—H hydrogen atoms were located from the difference Fourier map and refined isotropically. The C—H hydrogen atoms of the phenyl rings were positioned geometrically and were constrained to ride on their parent atoms, with C—H = 0.95 Å, and $U_{\rm iso}$ = 1.2 $U_{\rm eq}$(parent atom).

Figures

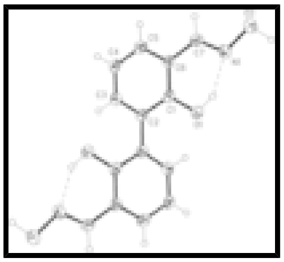

Fig. 1. A view of compound (I), with displacement ellipsoids shown at the 50% probability level. H atoms are drawn as spheres of an arbitrary radius.

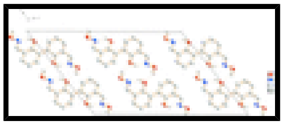

Fig. 2. A packing diagram of the title compound. Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

2,2'-Dihydroxy-1,1'-biphenyl-3,3'-dicarbaldehyde dioxime

Crystal data

$C_{14}H_{12}N_2O_4$

$M_r = 272.26$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 24.2780\ (14)$ Å

$b = 3.9279\ (4)$ Å

$c = 16.6466\ (12)$ Å

$\beta = 129.652\ (6)^\circ$

$V = 1222.2\ (2)$ Å^3

$Z = 4$

$F_{000} = 568$

$D_x = 1.480$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 516 reflections

$\theta = 4.5–27.0^\circ$

$\mu = 0.11$ mm^{-1}

$T = 120$ K

Block, pale-yellow

0.19 × 0.09 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: horizontally mounted graphite crystal

Detector resolution: 9 pixels mm^{-1}

$T = 120$ K

φ scans and ω scans with κ offset

Absorption correction: multi-scan (SADABS; Sheldrick, 2001)

$T_{\min} = 0.976$, $T_{\max} = 0.993$

4331 measured reflections

1388 independent reflections

812 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.073$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 4.4^\circ$

$h = -30 \rightarrow 30$

$k = -5 \rightarrow 4$

$l = -18 \rightarrow 21$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.056$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.146$	$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
1388 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
99 parameters	$\Delta\rho_{min} = -0.29$ 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 $F^2 > \sigma(F^2)$ 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}
O1	0.50535 (8)	0.1656 (4)	0.11701 (11)	0.0286 (5)
O2	0.64023 (9)	−0.1055 (4)	0.07166 (13)	0.0350 (5)
N1	0.60748 (10)	0.0232 (5)	0.11062 (14)	0.0279 (5)
C1	0.55751 (12)	0.2918 (5)	0.21487 (16)	0.0236 (6)
C2	0.53803 (11)	0.4208 (6)	0.27199 (16)	0.0235 (6)
C3	0.59205 (12)	0.5499 (6)	0.37151 (16)	0.0265 (6)
H3	0.5795	0.6439	0.4105	0.032*
C4	0.66275 (12)	0.5455 (6)	0.41490 (17)	0.0269 (6)
H4	0.6983	0.6329	0.4832	0.032*
C5	0.68185 (12)	0.4140 (6)	0.35911 (16)	0.0272 (6)
H5	0.7308	0.4102	0.3893	0.033*
C6	0.62978 (11)	0.2855 (6)	0.25813 (16)	0.0237 (6)
C7	0.65242 (12)	0.1435 (6)	0.20269 (17)	0.0265 (6)
H7	0.7019	0.1402	0.2358	0.032*
H1	0.5270 (14)	0.081 (7)	0.0923 (19)	0.042 (8)*
H2	0.5979 (18)	−0.165 (8)	−0.002 (3)	0.067 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0229 (9)	0.0387 (10)	0.0217 (9)	−0.0034 (7)	0.0132 (8)	−0.0059 (7)

O2	0.0324 (10)	0.0468 (11)	0.0296 (10)	0.0008 (8)	0.0217 (9)	−0.0042 (8)
N1	0.0299 (11)	0.0321 (11)	0.0277 (11)	0.0015 (9)	0.0212 (10)	−0.0001 (8)
C1	0.0244 (13)	0.0233 (12)	0.0192 (12)	−0.0006 (9)	0.0121 (11)	0.0013 (9)
C2	0.0235 (12)	0.0218 (12)	0.0213 (11)	−0.0002 (9)	0.0124 (11)	0.0017 (9)
C3	0.0306 (14)	0.0266 (13)	0.0231 (12)	−0.0015 (10)	0.0176 (11)	0.0000 (10)
C4	0.0253 (13)	0.0301 (13)	0.0178 (11)	−0.0044 (10)	0.0103 (10)	−0.0024 (9)
C5	0.0211 (12)	0.0290 (14)	0.0257 (12)	−0.0018 (10)	0.0123 (11)	0.0011 (10)
C6	0.0237 (13)	0.0246 (12)	0.0204 (12)	−0.0012 (9)	0.0130 (11)	0.0020 (9)
C7	0.0207 (12)	0.0311 (13)	0.0252 (12)	−0.0008 (10)	0.0136 (11)	0.0008 (10)

Geometric parameters (Å, °)

O1—C1	1.368 (3)	C3—C4	1.373 (3)
O1—H1	0.91 (3)	C3—H3	0.9500
O2—N1	1.402 (2)	C4—C5	1.376 (3)
O2—H2	1.00 (3)	C4—H4	0.9500
N1—C7	1.276 (3)	C5—C6	1.402 (3)
C1—C2	1.399 (3)	C5—H5	0.9500
C1—C6	1.409 (3)	C6—C7	1.453 (3)
C2—C3	1.396 (3)	C7—H7	0.9500
C2—C2^{i}	1.490 (4)		
C1—O1—H1	107.9 (16)	C3—C4—C5	119.7 (2)
N1—O2—H2	101.8 (18)	C3—C4—H4	120.1
C7—N1—O2	112.73 (17)	C5—C4—H4	120.1
O1—C1—C2	118.89 (19)	C4—C5—C6	120.7 (2)
O1—C1—C6	120.46 (19)	C4—C5—H5	119.7
C2—C1—C6	120.6 (2)	C6—C5—H5	119.7
C3—C2—C1	118.0 (2)	C5—C6—C1	118.83 (19)
C3—C2—C2^{i}	120.9 (2)	C5—C6—C7	118.8 (2)
C1—C2—C2^{i}	121.1 (2)	C1—C6—C7	122.31 (19)
C4—C3—C2	122.1 (2)	N1—C7—C6	121.6 (2)
C4—C3—H3	118.9	N1—C7—H7	119.2
C2—C3—H3	118.9	C6—C7—H7	119.2
O1—C1—C2—C3	−179.69 (18)	C4—C5—C6—C7	−178.9 (2)
C6—C1—C2—C3	1.6 (3)	O1—C1—C6—C5	−179.3 (2)
O1—C1—C2—C2^{i}	0.3 (3)	C2—C1—C6—C5	−0.6 (3)
C6—C1—C2—C2^{i}	−178.47 (16)	O1—C1—C6—C7	−0.8 (3)
C1—C2—C3—C4	−1.7 (3)	C2—C1—C6—C7	177.9 (2)
C2^{i}—C2—C3—C4	178.39 (17)	O2—N1—C7—C6	−179.16 (18)
C2—C3—C4—C5	0.8 (3)	C5—C6—C7—N1	−179.9 (2)
C3—C4—C5—C6	0.3 (3)	C1—C6—C7—N1	1.5 (3)
C4—C5—C6—C1	−0.3 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.91 (3)	1.79 (3)	2.609 (2)	148 (2)

O2—H2···O1^{ii}	1.00 (3)	1.96 (3)	2.871 (2)	151 (3)

Symmetry codes: (ii) −*x*+1, −*y*, −*z*.

Fig. 1

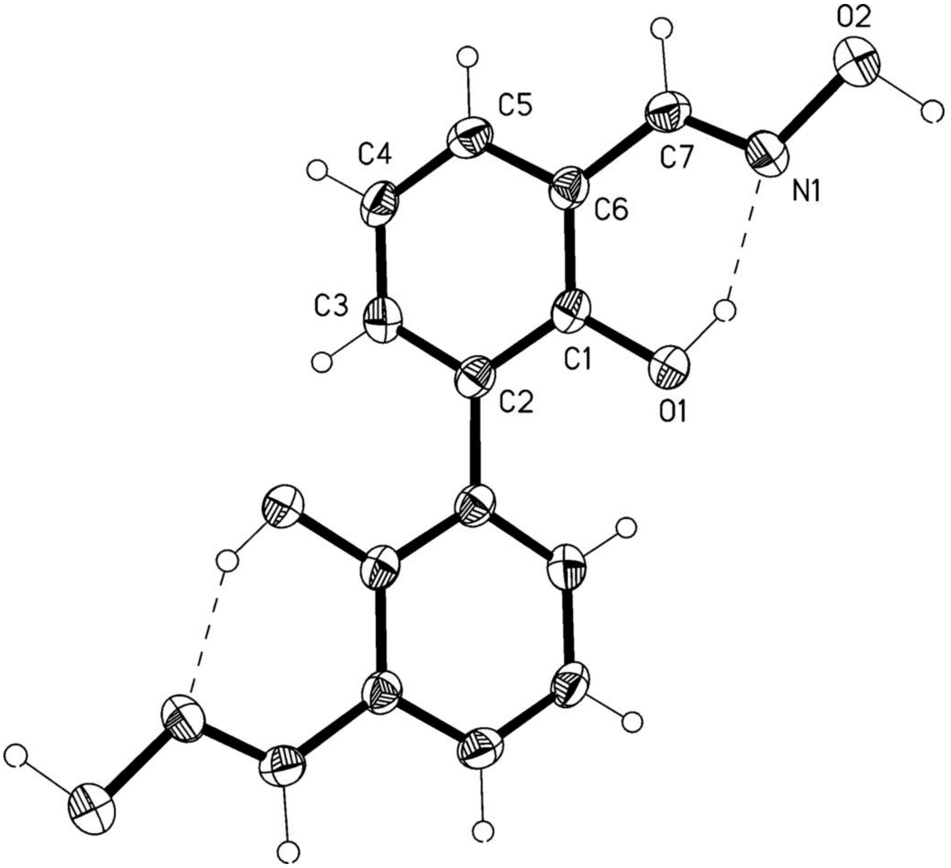

Fig. 2