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2,2′-[1,1′-(Ethylenedioxydinitrilo)diethylidyne]diphenol

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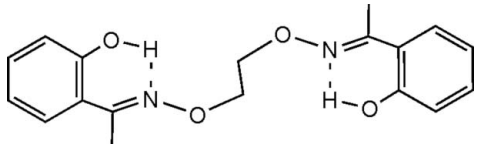
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.175; data-to-parameter ratio = 13.6.

The title compound, $C_{18}H_{20}N_2O_4$, has been characterized structurally by ^{1}H NMR and X-ray crystallography. The molecule is centrosymmetric. The two benzene rings are parallel to each other with a perpendicular interplanar spacing of *ca* 1.4 Å. Intramolecular O—H···N hydrogen bonds are formed between the oxime and hydroxy groups.

Related literature

For related literature, see: Atwood & Harvey (2001); Dong & Feng (2006); Dong, Feng & Yang (2006); Dong, Duan *et al.* (2006); Dong *et al.* (2007); Duan *et al.* (2007); Katsuki (1995).



Experimental

Crystal data

$C_{18}H_{20}N_2O_4$
M_r = 328.36
Monoclinic, $P2_1/n$
a = 6.9018 (12) Å
b = 17.2184 (18) Å
c = 7.3063 (14) Å
β = 103.003 (2)°
V = 846.0 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.63 × 0.58 × 0.45 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.944, T_{max} = 0.960
4172 measured reflections
1485 independent reflections
952 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.175
S = 1.07
1485 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N1	0.82	1.85	2.56 (2)	145

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2302).

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supplementary materials

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2,2'-[1,1'-(Ethylenedioxydinitrilo)diethylidyne]diphenol

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Comment

(*N*,*N*'-bis(salicylidence)ethylenediamine), salen, and its analogues have been extensively investigated for decades (Katsuki, 1995; Atwood & Harvey, 2001). At present, a new class of salen-type bisoxime compounds have been synthesized by using an *O*-alkyloxime unit (—CH=N—O—$(CH)_n$—O—N=CH—) instead of the (—CH=N—$(CH)_n$—N=CH—) group (Dong & Feng, 2006; Dong, Feng & Yang, 2006; Dong, Duan *et al.*, 2006; Duan *et al.*, 2007) as the large electronegativity of oxygen atoms is expected to affect strongly the electronic properties of N_2O_2 coordination sphere, which can lead to different and novel properties and structures of the resulting complexes.

Herein, we report on the crystal structure of 2,2'-[ethylenedioxy- bis(nitriloethylidyne)]diphenol (I), shown in Fig. 1. The structure of (I) consists of discrete $C_{18}H_{20}N_2O_4$ molecules in which all bond lengths are in normal ranges. The molecule is disposed about a cyrstallographic centre of symmetry with the (—CH=N—O—$(CH)_2$—O—N=CH—) bridge adopting an anti-symmetrized conformation. The two benzene rings of (I) are parallel to each other and separated by *ca* 1.4 Å. There is an intra-molecular hydrogen bond, O2—H2···N1 (*d*(O2—H2) = 0.82 Å, d(H2···N1) = 1.85 Å, d(O2···N1) = 2.56 (2) Å, O2—H2···N1 = 145.0°).

Experimental

2,2'-[ethylenedioxybis(nitriloethylidyne)]diphenol (I) was synthesized according to our previous work (Dong *et al.*, 2007). To an ethanol solution (5 ml) of 2'-hydroxyacetophenone (283.0 mg, 2.00 mmol) was added dropwise an ethanol solution (5 ml) of 1,2-bis(aminooxy)ethane (92.1 mg, 1.00 mmol). The mixture solution was stirred at 328 K for 2 h. After cooling to room temperature, the precipitate was filtered off, and washed successively with ethanol and ethanol-hexane (1:4). The product was dried *in vacuo* and purified by recrystallization from ethanol to yield 223.1 mg (Yield, 67.9%) of colorless microcrystals; m.p. 387 − 389 K. Anal. Calcd. for $C_{18}H_{20}N_2O_4$: C, 65.84; H, 6.14; N, 8.53; Found: C, 65.71; H, 6.24; N, 8.42%. ^{1}H NMR (400 MHz, $CDCl_3$): 2.34 (s, 6H), 4.50 (s, 4H), 6.89 (dd, J = 7.8, 1.4 Hz, 2H), 6.95 (dd, J = 8.4, 1.2 Hz, 2H), 7.25 (dd, J = 6.8, 1.6 Hz, 2H), 7.41 (dd, J = 8.0, 1.6 Hz, 2H), 11.10 (s, 2H).

Single crystals were obtained by slow evaporation from a solution of ethanol of 2,2'-[ethylenedioxybis(nitriloethylidyne)]diphenol at room temperature.

Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.97 (CH_2), or 0.93 Å (CH),*O*—H = 0.82 Å, and U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O).

Figures

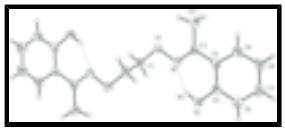

Fig. 1. Molecular structure of (I).

2,2'-[1,1'-(Ethylenedioxydinitrilo)diethylidyne]diphenol

Crystal data

$C_{18}H_{20}N_2O_4$	F_{000} = 348
M_r = 328.36	D_x = 1.289 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1276 reflections
a = 6.9018 (12) Å	θ = 2.4–25.1º
b = 17.2184 (18) Å	μ = 0.09 mm^{-1}
c = 7.3063 (14) Å	T = 298 (2) K
β = 103.003 (2)º	Prismatic, colorless
V = 846.0 (2) Å^3	0.63 × 0.58 × 0.45 mm
Z = 2	

Data collection

Bruker SMART CCD area-detector diffractometer	1485 independent reflections
Radiation source: fine-focus sealed tube	952 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.065
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −8→8
T_{min} = 0.944, T_{max} = 0.960	k = −20→18
4172 measured reflections	l = −5→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.058	H-atom parameters constrained
$wR(F^2)$ = 0.175	$w = 1/[\sigma^2(F_o^2) + (0.0801P)^2 + 0.199P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
1485 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
109 parameters	$\Delta\rho_{min}$ = −0.28 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.7373 (3)	0.04598 (11)	0.7165 (3)	0.0475 (6)
O1	0.7376 (2)	0.00177 (11)	0.8782 (2)	0.0579 (6)
O2	0.8973 (2)	0.09878 (10)	0.4578 (2)	0.0575 (6)
H2	0.8928	0.0734	0.5516	0.086*
C1	0.9323 (4)	−0.03004 (15)	0.9451 (4)	0.0534 (7)
H1A	0.9850	−0.0472	0.8395	0.064*
H1B	0.9250	−0.0747	1.0243	0.064*
C2	0.3971 (4)	0.07355 (16)	0.7411 (4)	0.0592 (8)
H2A	0.4379	0.0478	0.8600	0.089*
H2B	0.2932	0.0442	0.6613	0.089*
H2C	0.3488	0.1245	0.7600	0.089*
C3	0.5701 (3)	0.08004 (13)	0.6508 (3)	0.0408 (6)
C4	0.5586 (3)	0.12616 (12)	0.4789 (3)	0.0406 (6)
C5	0.7196 (3)	0.13387 (13)	0.3919 (3)	0.0437 (6)
C6	0.7024 (4)	0.17870 (15)	0.2319 (4)	0.0577 (7)
H6	0.8099	0.1832	0.1753	0.069*
C7	0.5280 (5)	0.21646 (16)	0.1564 (4)	0.0698 (9)
H7	0.5182	0.2471	0.0499	0.084*
C8	0.3672 (5)	0.20922 (16)	0.2377 (5)	0.0720 (9)
H8	0.2484	0.2344	0.1853	0.086*
C9	0.3829 (4)	0.16474 (15)	0.3962 (4)	0.0580 (8)
H9	0.2734	0.1602	0.4500	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0400 (12)	0.0603 (13)	0.0418 (12)	0.0038 (9)	0.0084 (9)	0.0099 (10)
O1	0.0428 (10)	0.0820 (12)	0.0491 (11)	0.0102 (8)	0.0108 (8)	0.0218 (9)
O2	0.0397 (10)	0.0758 (12)	0.0591 (12)	0.0100 (8)	0.0158 (8)	0.0167 (10)
C1	0.0509 (15)	0.0637 (16)	0.0454 (15)	0.0134 (12)	0.0108 (12)	0.0118 (12)
C2	0.0444 (15)	0.0755 (18)	0.0603 (17)	0.0048 (13)	0.0171 (12)	0.0075 (14)

C3	0.0335 (12)	0.0459 (13)	0.0423 (14)	−0.0013 (10)	0.0070 (10)	−0.0045 (11)
C4	0.0356 (12)	0.0392 (12)	0.0438 (14)	−0.0001 (9)	0.0022 (10)	−0.0038 (10)
C5	0.0383 (13)	0.0441 (13)	0.0467 (15)	−0.0016 (10)	0.0054 (11)	−0.0018 (11)
C6	0.0574 (17)	0.0613 (16)	0.0559 (17)	−0.0037 (13)	0.0158 (13)	0.0097 (13)
C7	0.073 (2)	0.0629 (18)	0.070 (2)	0.0011 (15)	0.0074 (16)	0.0243 (15)
C8	0.0552 (18)	0.0686 (19)	0.086 (2)	0.0131 (14)	0.0026 (16)	0.0265 (17)
C9	0.0421 (14)	0.0581 (16)	0.073 (2)	0.0078 (12)	0.0111 (13)	0.0109 (14)

Geometric parameters (Å, °)

N1—C3	1.287 (3)	C3—C4	1.473 (3)
N1—O1	1.405 (2)	C4—C9	1.396 (3)
O1—C1	1.432 (3)	C4—C5	1.405 (3)
O2—C5	1.355 (3)	C5—C6	1.384 (3)
O2—H2	0.8200	C6—C7	1.370 (4)
C1—C1^{i}	1.500 (5)	C6—H6	0.9300
C1—H1A	0.9700	C7—C8	1.377 (4)
C1—H1B	0.9700	C7—H7	0.9300
C2—C3	1.493 (3)	C8—C9	1.372 (4)
C2—H2A	0.9600	C8—H8	0.9300
C2—H2B	0.9600	C9—H9	0.9300
C2—H2C	0.9600		
C3—N1—O1	113.06 (19)	C9—C4—C5	117.2 (2)
N1—O1—C1	108.54 (17)	C9—C4—C3	120.0 (2)
C5—O2—H2	109.5	C5—C4—C3	122.7 (2)
O1—C1—C1^{i}	110.0 (3)	O2—C5—C6	116.6 (2)
O1—C1—H1A	109.7	O2—C5—C4	122.8 (2)
C1^{i}—C1—H1A	109.7	C6—C5—C4	120.6 (2)
O1—C1—H1B	109.7	C7—C6—C5	120.3 (3)
C1^{i}—C1—H1B	109.7	C7—C6—H6	119.8
H1A—C1—H1B	108.2	C5—C6—H6	119.8
C3—C2—H2A	109.5	C6—C7—C8	120.3 (3)
C3—C2—H2B	109.5	C6—C7—H7	119.9
H2A—C2—H2B	109.5	C8—C7—H7	119.9
C3—C2—H2C	109.5	C9—C8—C7	119.7 (3)
H2A—C2—H2C	109.5	C9—C8—H8	120.1
H2B—C2—H2C	109.5	C7—C8—H8	120.1
N1—C3—C4	115.8 (2)	C8—C9—C4	121.8 (3)
N1—C3—C2	122.7 (2)	C8—C9—H9	119.1
C4—C3—C2	121.5 (2)	C4—C9—H9	119.1
C3—N1—O1—C1	−176.97 (19)	C9—C4—C5—C6	0.6 (3)
N1—O1—C1—C1^{i}	80.3 (3)	C3—C4—C5—C6	−179.1 (2)
O1—N1—C3—C4	−178.84 (17)	O2—C5—C6—C7	−179.7 (2)
O1—N1—C3—C2	0.9 (3)	C4—C5—C6—C7	0.2 (4)
N1—C3—C4—C9	179.5 (2)	C5—C6—C7—C8	−0.9 (4)
C2—C3—C4—C9	−0.3 (3)	C6—C7—C8—C9	0.8 (5)
N1—C3—C4—C5	−0.8 (3)	C7—C8—C9—C4	0.0 (5)
C2—C3—C4—C5	179.4 (2)	C5—C4—C9—C8	−0.7 (4)

C9—C4—C5—O2	−179.5 (2)	C3—C4—C9—C8	179.0 (2)
C3—C4—C5—O2	0.8 (3)		

Symmetry codes: (i) −*x*+2, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.82	1.85	2.56 (2)	145

Fig. 1