Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′-[1,1′-(Ethylenedioxydinitrilo)-diethylidyne]di-1-naphthol

Wen-Kui Dong,* Jian-chao Wu, Jian Yao, Li Li and Shang-sheng Gong

School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, People's Republic of China
Correspondence e-mail: dongwk@mail.lzjtu.cn

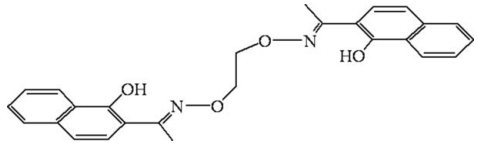
Received 11 June 2009; accepted 12 June 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.122; data-to-parameter ratio = 13.1.

The complete molecule of the title compound, $C_{26}H_{24}N_2O_4$, is generated by a crystallographic centre of inversion. There are two intramolecular O—H···N hydrogen bonds. In the crystal structure, intermolecular C—H···O hydrogen bonds result in zigzag chains.

Related literature

For the applications of Shiff base ligands, see: Calligaris & Randaccio (1987) For the applications bisoxime derivatives of salen-type compounds, see: Sun *et al.* (2004); Wang *et al.* (2007). For related structures, see: Dong *et al.* (2008*a*,*b*,*c*);



Experimental

Crystal data

$C_{26}H_{24}N_2O_4$
M_r = 428.47
Monoclinic, $C2/c$
a = 12.6682 (18) Å
b = 9.3728 (15) Å
c = 18.335 (2) Å
β = 97.478 (2)°
V = 2158.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.39 × 0.37 × 0.13 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.966, T_{max} = 0.989
5220 measured reflections
1894 independent reflections
1027 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.122
S = 1.06
1894 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 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	1.84	2.562 (3)	146
C10—H10···O2^{i}	0.93	2.63	3.446 (3)	146

Symmetry code: (i) $-x+\frac{3}{2}, -y-\frac{1}{2}, -z+1$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge finanical support from the 'Jing Lan' Talent Engineering Funds of Lanzhou Jiaotong University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2524).

References

Calligaris, M. & Randaccio, L. (1987). *Comprehensive Coordination Chemistry*, Vol. 2, edited by G. Wilkinson, pp. 715-738. London: Pergamon.
Dong, W.-K., He, X.-N., Guan, Y.-H., Xu, L. & Ren, Z.-L. (2008*c*). *Acta Cryst.* E**64**, o1600–o1601.
Dong, W.-K., He, X.-N., Li, L., Lv, Z.-W. & Tong, J.-F. (2008*a*). *Acta Cryst.* E**64**, o1405.
Dong, W.-K., Zhao, C.-Y., Zhong, J.-K., Tang, X.-L. & Yu, T.-Z. (2008*b*). *Acta Cryst.* E**64**, o1323.
Sheldrick, G. M. (1996). *SADABS*. University of Goöttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sun, S. S., Stern, C. L., Nguyen, S. T. & Hupp, J. T. (2004). *J. Am. Chem. Soc.* **126**, 6314–6326.
Wang, L., He, X., Sun, Y. & Xu, L. (2007). *Acta Cryst.* E**63**, o4517.

supporting information

Acta Cryst. (2009). E**65**, o1598 [doi:10.1107/S1600536809022508]

2,2′-[1,1′-(Ethylenedioxydinitrilo)diethylidyne]di-1-naphthol

Wen-Kui Dong, Jian-chao Wu, Jian Yao, Li Li and Shang-sheng Gong

S1. Comment

There has been considerable interest in Schiff base ligand containing oxygen and imine nitrogen atoms due to their variety of applications, especially for catalysis and enzymatic reactions, magnetism, and supramolecular architectures (Calligaris & Randaccio, 1987). salen-type compounds and its bisoxime derivatives are a new class of multidentate ligand, which can be used as elemental building blocks for construction of supramolecular structures *via* intermolecular hydrogen bonding or short contact interaction (Sun *et al.*, 2004; Wang *et al.*, 2007). As an extension of our work (Dong *et al.*, 2008*a*; Dong *et al.*, 2008*b*) on the structural characterization of salen-type bisoxime compounds, here report the synthesis and structure of the title compound (Fig. 1).

The single-crystal structure of the title compound is built up by discrete $C_{26}H_{24}N_2O_4$ molecules, in which all bond lengths are in normal ranges. The molecule has a crystallographic twofold rotation axis (symmetry code: -*x*, *y*, 1/2 - *z*) and screw axis (symmetry code: 1/2 - *x*, 1/2 + *y*, 1/2 - *z*), and adopts a distorted E-configuration. This structure is similar to what was observed in our previously reported E-configuration compounds of 2,2′-[1,1′-Ethylenedioxybis(nitriloethylidyne)]diphenol (Wang *et al.*, 2007). The dihedral angle formed by the two naphthalene rings in each molecule of the title compound is about 43.20 °. There are two intramolecular hydrogen bonds, O2—H2···N1 (*d*(O2—H2) = 0.82 Å, d(H2···N1) = 1.84 Å, d(O2···N1) = 2.562 (3) Å, <O2—H2···N1 = 146°). Besides in the crystal structure, four intermolecular hydrogen bonds, C10—H10···O2 (*d*(C10—H10) = 0.93 Å, d(H10···O2) = 2.63 Å, d(C10···O2) = 3.446 (3) Å, <C10—H10···O2 = 146°), link two other molecules into infinite zigzag supramolecular structure (Fig. 2).

S2. Experimental

2,2′-[1,1′-Ethylenedioxybis(nitriloethylidyne)]dinaphthol was synthesized according to an analogous method reported earlier (Dong *et al.*, 2008*c*). To an ethanol solution (5 ml) of 2-acetyl-1-naphthol (392.1 mg, 2.10 mmol) was added dropwise an ethanol solution (3 ml) of 1,2-bis(aminooxy)ethane (96.2 mg, 1.04 mmol). The mixture solution was stirred at 328–433 K for 72 h. After cooling to room temperature, the precipitate was filtered off, and washed successively three times with ethanol. The product was dried *in vacuo* and purified by recrystallization from ethanol to yield 313.7 mg (Yield, 70.1%) of powder; m.p. 471–472 K. Colorless block-like single crystals suitable for X-ray diffraction studies were obtained by slow evaporation from a solution of dichloromethane of 2,2′-[1,1′-ethylenedioxybis(nitriloethylidyne)]dinaphthol at room temperature for about three weeks. Anal. Calc. for $C_{26}H_{24}N_2O_4$: C, 73.28; H, 5.92; N, 6.33; Found: C, 73.25; H, 5.97; N, 6.29.

S3. Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.97 (CH_2), C—H = 0.96 (CH_3), 0.93 Å (CH), 0.82 Å (OH), and U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O).

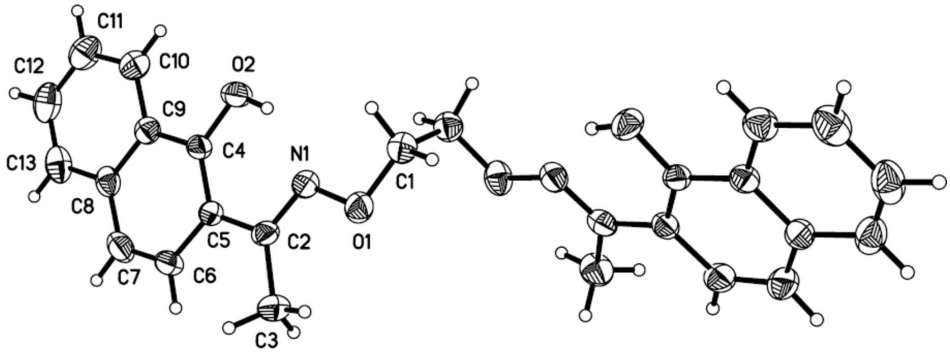


Figure 1

The molecular structure of the title compound with atom numbering scheme [Symmetry codes: -*x*+2, *y*, -*z* + 3/2]. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

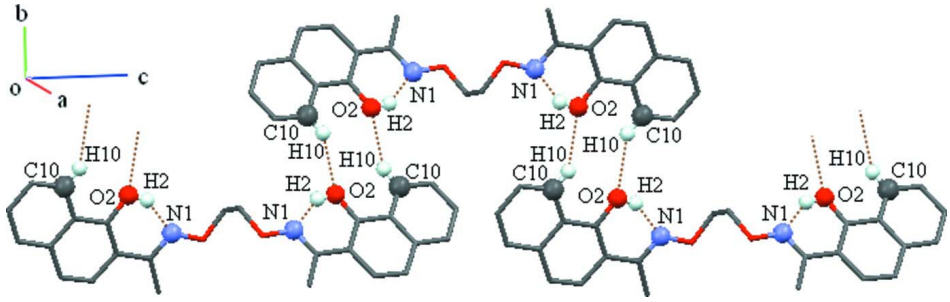


Figure 2

Part of the supramolecular structure of the title compound. Intra- and intermolecular hydrogen bonds are shown as dashed lines.

2,2′-[1,1′-(Ethylenedioxydinitrilo)diethylidyne]di-1-naphthol

Crystal data

$C_{26}H_{24}N_2O_4$
M_r = 428.47
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 12.6682 (18) Å
b = 9.3728 (15) Å
c = 18.335 (2) Å
β = 97.478 (2)°
V = 2158.6 (5) Å^3
Z = 4
F(000) = 904
D_x = 1.318 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1610 reflections
θ = 2.2–26.7°
μ = 0.09 mm^{-1}
T = 298 K
Block-shaped, colorless
0.39 × 0.37 × 0.13 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.966, T_{max} = 0.989
5220 measured reflections
1894 independent reflections
1027 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −15→14
k = −11→9
l = −18→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.122$
$S = 1.06$
1894 reflections
145 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 1.5505P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.97510 (17)	0.0879 (2)	0.61829 (11)	0.0528 (6)
O1	1.04967 (14)	0.11327 (19)	0.68140 (9)	0.0610 (5)
O2	0.84279 (14)	−0.06506 (17)	0.53429 (9)	0.0620 (6)
H2	0.8831	−0.0456	0.5717	0.093*
C1	1.0479 (2)	−0.0049 (3)	0.73030 (14)	0.0567 (7)
H1A	1.1113	−0.0024	0.7661	0.068*
H1B	1.0493	−0.0929	0.7025	0.068*
C2	0.9707 (2)	0.1902 (3)	0.57048 (14)	0.0500 (7)
C3	1.0376 (2)	0.3226 (3)	0.58230 (16)	0.0714 (9)
H3A	1.0938	0.3072	0.6220	0.107*
H3B	0.9941	0.4009	0.5942	0.107*
H3C	1.0678	0.3444	0.5382	0.107*
C4	0.83740 (19)	0.0469 (3)	0.48738 (13)	0.0464 (6)
C5	0.89572 (19)	0.1710 (2)	0.50274 (12)	0.0469 (6)
C6	0.8818 (2)	0.2819 (3)	0.44918 (15)	0.0610 (8)
H6	0.9202	0.3660	0.4582	0.073*
C7	0.8146 (2)	0.2692 (3)	0.38547 (16)	0.0676 (8)
H7	0.8065	0.3454	0.3527	0.081*
C8	0.7568 (2)	0.1424 (3)	0.36816 (14)	0.0552 (7)
C9	0.7676 (2)	0.0289 (3)	0.42035 (13)	0.0490 (7)
C10	0.7106 (2)	−0.0991 (3)	0.40331 (15)	0.0632 (8)
H10	0.7163	−0.1737	0.4370	0.076*
C11	0.6470 (2)	−0.1137 (4)	0.33734 (18)	0.0774 (9)
H11	0.6097	−0.1982	0.3266	0.093*
C12	0.6378 (2)	−0.0025 (4)	0.28598 (17)	0.0794 (10)

H12	0.5957	−0.0146	0.2409	0.095*
C13	0.6896 (2)	0.1227 (4)	0.30122 (15)	0.0707 (9)
H13	0.6807	0.1966	0.2671	0.085*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0581 (14)	0.0524 (13)	0.0491 (13)	−0.0069 (11)	0.0112 (11)	−0.0060 (11)
O1	0.0618 (12)	0.0706 (13)	0.0511 (11)	−0.0170 (10)	0.0085 (9)	−0.0068 (9)
O2	0.0768 (13)	0.0463 (10)	0.0591 (12)	−0.0120 (9)	−0.0055 (10)	0.0078 (9)
C1	0.0569 (18)	0.0582 (17)	0.0548 (17)	0.0022 (13)	0.0063 (13)	−0.0011 (14)
C2	0.0553 (16)	0.0410 (14)	0.0572 (17)	−0.0059 (13)	0.0205 (13)	−0.0099 (13)
C3	0.085 (2)	0.0560 (18)	0.076 (2)	−0.0235 (16)	0.0195 (17)	−0.0080 (15)
C4	0.0559 (17)	0.0381 (14)	0.0474 (15)	0.0028 (12)	0.0152 (13)	0.0019 (12)
C5	0.0582 (16)	0.0398 (14)	0.0458 (15)	−0.0022 (13)	0.0176 (13)	−0.0033 (12)
C6	0.080 (2)	0.0431 (15)	0.0637 (19)	−0.0059 (15)	0.0249 (16)	0.0047 (14)
C7	0.086 (2)	0.0622 (19)	0.0587 (19)	0.0079 (17)	0.0262 (17)	0.0161 (15)
C8	0.0593 (18)	0.0602 (18)	0.0491 (16)	0.0143 (15)	0.0180 (14)	0.0030 (14)
C9	0.0485 (16)	0.0489 (16)	0.0509 (16)	0.0069 (13)	0.0117 (13)	−0.0021 (13)
C10	0.0617 (18)	0.0606 (18)	0.0659 (19)	−0.0008 (15)	0.0034 (16)	−0.0052 (15)
C11	0.064 (2)	0.087 (2)	0.079 (2)	−0.0027 (18)	−0.0008 (18)	−0.0190 (19)
C12	0.061 (2)	0.116 (3)	0.059 (2)	0.016 (2)	0.0015 (16)	−0.012 (2)
C13	0.065 (2)	0.094 (2)	0.0537 (19)	0.0235 (19)	0.0121 (16)	0.0088 (17)

Geometric parameters (Å, º)

N1—C2	1.295 (3)	C5—C6	1.425 (3)
N1—O1	1.415 (2)	C6—C7	1.358 (3)
O1—C1	1.427 (3)	C6—H6	0.9300
O2—C4	1.353 (3)	C7—C8	1.410 (4)
O2—H2	0.8200	C7—H7	0.9300
C1—C1^{i}	1.490 (5)	C8—C13	1.412 (4)
C1—H1A	0.9700	C8—C9	1.426 (3)
C1—H1B	0.9700	C9—C10	1.413 (3)
C2—C5	1.473 (3)	C10—C11	1.370 (3)
C2—C3	1.504 (3)	C10—H10	0.9300
C3—H3A	0.9600	C11—C12	1.400 (4)
C3—H3B	0.9600	C11—H11	0.9300
C3—H3C	0.9600	C12—C13	1.355 (4)
C4—C5	1.387 (3)	C12—H12	0.9300
C4—C9	1.428 (3)	C13—H13	0.9300
C2—N1—O1	113.2 (2)	C7—C6—C5	122.5 (3)
N1—O1—C1	108.64 (18)	C7—C6—H6	118.8
C4—O2—H2	109.5	C5—C6—H6	118.8
O1—C1—C1^{i}	112.64 (18)	C6—C7—C8	121.1 (3)
O1—C1—H1A	109.1	C6—C7—H7	119.5
C1^{i}—C1—H1A	109.1	C8—C7—H7	119.5

O1—C1—H1B	109.1	C7—C8—C13	122.9 (3)
C1^{i}—C1—H1B	109.1	C7—C8—C9	118.4 (2)
H1A—C1—H1B	107.8	C13—C8—C9	118.7 (3)
N1—C2—C5	116.6 (2)	C10—C9—C8	118.9 (2)
N1—C2—C3	122.7 (2)	C10—C9—C4	122.1 (2)
C5—C2—C3	120.8 (2)	C8—C9—C4	118.9 (2)
C2—C3—H3A	109.5	C11—C10—C9	120.3 (3)
C2—C3—H3B	109.5	C11—C10—H10	119.8
H3A—C3—H3B	109.5	C9—C10—H10	119.8
C2—C3—H3C	109.5	C10—C11—C12	120.5 (3)
H3A—C3—H3C	109.5	C10—C11—H11	119.8
H3B—C3—H3C	109.5	C12—C11—H11	119.8
O2—C4—C5	122.7 (2)	C13—C12—C11	120.7 (3)
O2—C4—C9	115.4 (2)	C13—C12—H12	119.7
C5—C4—C9	121.9 (2)	C11—C12—H12	119.7
C4—C5—C6	117.2 (2)	C12—C13—C8	120.9 (3)
C4—C5—C2	122.8 (2)	C12—C13—H13	119.6
C6—C5—C2	120.1 (2)	C8—C13—H13	119.6
C2—N1—O1—C1	179.8 (2)	C6—C7—C8—C9	2.2 (4)
N1—O1—C1—C1^{i}	−75.0 (3)	C7—C8—C9—C10	−179.5 (2)
O1—N1—C2—C5	179.31 (19)	C13—C8—C9—C10	−0.1 (4)
O1—N1—C2—C3	−0.8 (3)	C7—C8—C9—C4	−0.9 (3)
O2—C4—C5—C6	−179.2 (2)	C13—C8—C9—C4	178.5 (2)
C9—C4—C5—C6	1.1 (3)	O2—C4—C9—C10	−1.9 (3)
O2—C4—C5—C2	1.7 (4)	C5—C4—C9—C10	177.9 (2)
C9—C4—C5—C2	−178.1 (2)	O2—C4—C9—C8	179.6 (2)
N1—C2—C5—C4	−4.3 (3)	C5—C4—C9—C8	−0.7 (3)
C3—C2—C5—C4	175.8 (2)	C8—C9—C10—C11	0.6 (4)
N1—C2—C5—C6	176.6 (2)	C4—C9—C10—C11	−177.9 (2)
C3—C2—C5—C6	−3.3 (4)	C9—C10—C11—C12	0.1 (4)
C4—C5—C6—C7	0.2 (4)	C10—C11—C12—C13	−1.5 (5)
C2—C5—C6—C7	179.3 (2)	C11—C12—C13—C8	2.1 (5)
C5—C6—C7—C8	−1.8 (4)	C7—C8—C13—C12	178.1 (3)
C6—C7—C8—C13	−177.2 (3)	C9—C8—C13—C12	−1.3 (4)

Symmetry code: (i) −*x*+2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.82	1.84	2.562 (3)	146
C10—H10···O2^{ii}	0.93	2.63	3.446 (3)	146

Symmetry code: (ii) −*x*+3/2, −*y*−1/2, −*z*+1.