

2,2'-[1,1'-[Butane-1,4-diylbis(oxy-nitrilo)]diethylidene]di-1-naphthol

Wen-Kui Dong,* Jian-Chao Wu, Yin-Xia Sun, Jian Yao and Jun-Feng Tong

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

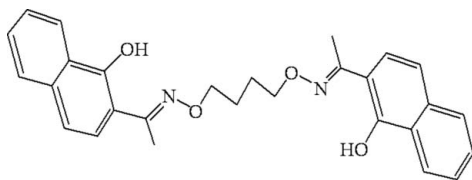
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.054; wR factor = 0.173; data-to-parameter ratio = 13.2.

The title compound, $\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_4$, was synthesized by the reaction of 2-acetyl-1-naphthol with 1,4-bis(aminooxy)butane in ethanol. The molecule, which lies about an inversion centre, adopts a linear structure, in which the oxime groups and naphthalene ring systems assume an *anti* conformation. The intramolecular interplanar distance between parallel naphthalene rings is 1.054 (3) Å. Intramolecular O—H...N hydrogen bonds are formed between the oxime nitrogen and hydroxy groups.

Related literature

For salen-type compounds, see: Atwood & Harvey (2001); Okabe & Oya (2000). For related structures, see: Dong *et al.* (2007, 2008).



Experimental

Crystal data

 $\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_4$
 $M_r = 456.52$

Triclinic, $P\bar{1}$
 $a = 6.9590$ (15) Å
 $b = 8.6598$ (18) Å
 $c = 10.596$ (2) Å
 $\alpha = 105.841$ (2)°
 $\beta = 105.940$ (2)°
 $\gamma = 91.689$ (1)°

$V = 586.9$ (2) Å³
 $Z = 1$
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ K
 $0.50 \times 0.43 \times 0.20$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.958$, $T_{\max} = 0.983$

3020 measured reflections
2026 independent reflections
1350 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.173$
 $S = 1.02$
2026 reflections

154 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2...N1	0.82	1.84	2.557 (2)	145

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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2505).

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supplementary materials

Acta Cryst. (2009). E65, o1248 [doi:10.1107/S160053680901647X]

2,2'-{1,1'-[Butane-1,4-diylbis(oxynitrilo)]diethylidyne}di-1-naphthol

W.-K. Dong, J.-C. Wu, Y.-X. Sun, J. Yao and J.-F. Tong

Comment

Salen-type compounds and their analogues are one of the most prevalent multidentate ligands in the field of modern coordination chemistry (Atwood & Harvey, 2001), which can coordinate to transition or rare earth ions yielding complexes with interesting properties that are useful in materials science and in biological systems (Okabe & Oya, 2000).

Herein, we report on the crystal structure of 2,2'-[(butane-1,4-diylbis(oxynitrilo)]diethylidyne}dinaphthol, shown in Fig. 1. The structure of the title compound consists of discrete $C_{28}H_{28}N_2O_4$ molecule, in which all bond lengths and angles are in normal ranges. The molecule is disposed about a crystallographic centre of symmetry with the $(-CH=N-O-(CH)_4-O-N=CH-)$ bridge adopting a linear-shaped structure. The oxime groups and naphthalene rings assume an anti conformation. This structure is not similar to what was observed in our previously reported series salen-type compounds containing four-methene bridge, which often adopt an E configuration (Dong *et al.*, 2007, 2008).

The two naphthalene rings in each molecule of the title compound are parallel and the distance between them is 1.054 (3) Å. Two intramolecular O—H...N hydrogen bonds are formed between the oxime nitrogen and hydroxy groups.

Experimental

2,2'-[(Butane-1,4-diylbis(oxynitrilo)]diethylidyne}dinaphthol was synthesized according to our previous work (Dong *et al.*, 2007). To an ethanol solution (5 ml) of 2-acetyl-1-naphthol (760.0 mg, 2.02 mmol) was added dropwise an ethanol solution (3 ml) of 1,4-bis(aminoxyl)butane (243.0 mg, 1.01 mmol). The mixture solution was stirred at 328–333 K for 75 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 618.6 mg (yield 67.0%) of powder. Single crystals were obtained by slow evaporation from a solution of ethanol/dichloromethane (1:2) of 2,2'-[(butane-1,4-diylbis(oxynitrilo)]diethylidyne}dinaphthol at room temperature for several weeks. Anal. Calcd. for $C_{28}H_{28}N_2O_4$: C, 73.66; H, 6.18; N, 6.14; Found: C, 73.61; H, 6.23; N, 6.09%.

Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.97 (CH₂), C—H = 0.96 (CH₃), 0.93 Å (CH), 0.82 Å (OH), and $U_{iso}(H) = 1.2 U_{eq}(C)$ and $1.5 U_{eq}(O)$.

Figures

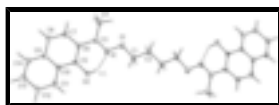


Fig. 1. The molecule structure of the title compound with atom numbering [Symmetry codes: $-x + 1, -y + 1, -z$]. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

2,2'-[1,1'-[Butane-1,4-diylbis(oxynitrilo)]diethyldiylidene]di-1-naphthol

Crystal data

$C_{28}H_{28}N_2O_4$	$Z = 1$
$M_r = 456.52$	$F_{000} = 242$
Triclinic, $P\bar{1}$	$D_x = 1.292 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 6.9590 (15) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.6598 (18) \text{ \AA}$	Cell parameters from 1246 reflections
$c = 10.596 (2) \text{ \AA}$	$\theta = 2.5\text{--}27.6^\circ$
$\alpha = 105.841 (2)^\circ$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 105.940 (2)^\circ$	$T = 298 \text{ K}$
$\gamma = 91.6890 (10)^\circ$	Block, pale-yellow
$V = 586.9 (2) \text{ \AA}^3$	$0.50 \times 0.43 \times 0.20 \text{ mm}$

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2026 independent reflections
Radiation source: fine-focus sealed tube	1350 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 8$
$T_{\text{min}} = 0.958, T_{\text{max}} = 0.983$	$k = -9 \rightarrow 10$
3020 measured reflections	$l = -12 \rightarrow 6$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.054$	H-atom parameters constrained
$wR(F^2) = 0.173$	$w = 1/[\sigma^2(F_o^2) + (0.096P)^2 + 0.0813P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
2026 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
154 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
	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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.4299 (3)	0.3289 (2)	0.31934 (16)	0.0435 (5)
O1	0.3505 (2)	0.37340 (18)	0.19981 (14)	0.0515 (5)
O2	0.7182 (2)	0.30688 (18)	0.52155 (15)	0.0505 (5)
H2	0.6665	0.3326	0.4519	0.076*
C1	0.5112 (3)	0.4251 (3)	0.1562 (2)	0.0455 (6)
H1A	0.5966	0.3396	0.1410	0.055*
H1B	0.5923	0.5189	0.2259	0.055*
C2	0.4211 (3)	0.4667 (3)	0.0252 (2)	0.0449 (6)
H2A	0.3277	0.5459	0.0400	0.054*
H2B	0.3460	0.3705	-0.0449	0.054*
C3	0.2958 (3)	0.2877 (2)	0.3702 (2)	0.0402 (5)
C4	0.0759 (3)	0.2939 (3)	0.3091 (2)	0.0627 (7)
H4A	0.0578	0.3501	0.2408	0.094*
H4B	0.0198	0.3496	0.3798	0.094*
H4C	0.0091	0.1859	0.2673	0.094*
C5	0.5732 (3)	0.2467 (2)	0.5630 (2)	0.0372 (5)
C6	0.3690 (3)	0.2344 (2)	0.49419 (19)	0.0365 (5)
C7	0.2306 (3)	0.1639 (2)	0.5453 (2)	0.0433 (5)
H7	0.0937	0.1554	0.5009	0.052*
C8	0.2912 (3)	0.1088 (2)	0.6565 (2)	0.0438 (6)
H8	0.1961	0.0608	0.6851	0.053*
C9	0.4976 (3)	0.1238 (2)	0.7294 (2)	0.0386 (5)
C10	0.6397 (3)	0.1961 (2)	0.6838 (2)	0.0370 (5)
C11	0.8449 (3)	0.2185 (3)	0.7606 (2)	0.0506 (6)
H11	0.9400	0.2668	0.7321	0.061*
C12	0.9047 (4)	0.1696 (3)	0.8761 (3)	0.0600 (7)
H12	1.0397	0.1872	0.9270	0.072*
C13	0.7640 (4)	0.0933 (3)	0.9182 (2)	0.0564 (7)
H13	0.8068	0.0574	0.9955	0.068*
C14	0.5661 (3)	0.0711 (3)	0.8475 (2)	0.0479 (6)
H14	0.4743	0.0207	0.8770	0.057*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0575 (12)	0.0489 (11)	0.0329 (9)	0.0084 (9)	0.0176 (8)	0.0215 (8)
O1	0.0585 (10)	0.0695 (11)	0.0386 (8)	0.0082 (8)	0.0167 (7)	0.0329 (7)
O2	0.0468 (9)	0.0664 (10)	0.0556 (10)	0.0120 (7)	0.0240 (7)	0.0362 (8)
C1	0.0570 (13)	0.0519 (13)	0.0402 (12)	0.0112 (10)	0.0242 (10)	0.0234 (10)
C2	0.0573 (14)	0.0482 (12)	0.0360 (12)	0.0094 (11)	0.0180 (10)	0.0188 (9)
C3	0.0498 (12)	0.0394 (11)	0.0345 (11)	0.0036 (9)	0.0159 (9)	0.0121 (9)
C4	0.0544 (15)	0.0897 (19)	0.0525 (14)	0.0030 (13)	0.0111 (11)	0.0397 (13)
C5	0.0447 (12)	0.0363 (11)	0.0393 (11)	0.0080 (9)	0.0219 (9)	0.0152 (9)
C6	0.0443 (12)	0.0380 (11)	0.0313 (10)	0.0048 (9)	0.0155 (9)	0.0124 (8)
C7	0.0434 (12)	0.0523 (13)	0.0374 (11)	0.0003 (10)	0.0131 (9)	0.0178 (10)
C8	0.0487 (13)	0.0495 (13)	0.0404 (12)	-0.0015 (10)	0.0203 (10)	0.0182 (10)
C9	0.0505 (13)	0.0354 (11)	0.0375 (11)	0.0105 (9)	0.0202 (9)	0.0149 (9)
C10	0.0438 (12)	0.0367 (11)	0.0379 (11)	0.0138 (9)	0.0177 (9)	0.0157 (9)
C11	0.0466 (13)	0.0589 (14)	0.0597 (14)	0.0164 (11)	0.0218 (11)	0.0315 (11)
C12	0.0498 (14)	0.0742 (17)	0.0654 (16)	0.0220 (12)	0.0132 (12)	0.0376 (13)
C13	0.0643 (16)	0.0638 (15)	0.0534 (14)	0.0232 (12)	0.0170 (12)	0.0356 (12)
C14	0.0621 (15)	0.0493 (13)	0.0455 (13)	0.0138 (11)	0.0239 (11)	0.0262 (10)

Geometric parameters (\AA , $^\circ$)

N1—C3	1.285 (3)	C5—C10	1.427 (3)
N1—O1	1.398 (2)	C6—C7	1.423 (3)
O1—C1	1.425 (2)	C7—C8	1.357 (3)
O2—C5	1.350 (2)	C7—H7	0.9300
O2—H2	0.8200	C8—C9	1.415 (3)
C1—C2	1.503 (3)	C8—H8	0.9300
C1—H1A	0.9700	C9—C10	1.413 (3)
C1—H1B	0.9700	C9—C14	1.414 (3)
C2—C2 ⁱ	1.509 (4)	C10—C11	1.414 (3)
C2—H2A	0.9700	C11—C12	1.365 (3)
C2—H2B	0.9700	C11—H11	0.9300
C3—C6	1.475 (3)	C12—C13	1.400 (3)
C3—C4	1.498 (3)	C12—H12	0.9300
C4—H4A	0.9600	C13—C14	1.354 (3)
C4—H4B	0.9600	C13—H13	0.9300
C4—H4C	0.9600	C14—H14	0.9300
C5—C6	1.394 (3)		
C3—N1—O1	113.72 (17)	C5—C6—C7	117.71 (18)
N1—O1—C1	109.26 (15)	C5—C6—C3	122.01 (18)
C5—O2—H2	109.5	C7—C6—C3	120.26 (18)
O1—C1—C2	107.96 (18)	C8—C7—C6	122.4 (2)
O1—C1—H1A	110.1	C8—C7—H7	118.8
C2—C1—H1A	110.1	C6—C7—H7	118.8
O1—C1—H1B	110.1	C7—C8—C9	120.59 (19)

C2—C1—H1B	110.1	C7—C8—H8	119.7
H1A—C1—H1B	108.4	C9—C8—H8	119.7
C1—C2—C2 ⁱ	112.2 (2)	C10—C9—C14	118.88 (19)
C1—C2—H2A	109.2	C10—C9—C8	118.87 (17)
C2 ⁱ —C2—H2A	109.2	C14—C9—C8	122.24 (19)
C1—C2—H2B	109.2	C9—C10—C11	118.88 (18)
C2 ⁱ —C2—H2B	109.2	C9—C10—C5	119.50 (18)
H2A—C2—H2B	107.9	C11—C10—C5	121.61 (19)
N1—C3—C6	116.56 (18)	C12—C11—C10	120.5 (2)
N1—C3—C4	122.53 (18)	C12—C11—H11	119.8
C6—C3—C4	120.91 (18)	C10—C11—H11	119.8
C3—C4—H4A	109.5	C11—C12—C13	120.4 (2)
C3—C4—H4B	109.5	C11—C12—H12	119.8
H4A—C4—H4B	109.5	C13—C12—H12	119.8
C3—C4—H4C	109.5	C14—C13—C12	120.6 (2)
H4A—C4—H4C	109.5	C14—C13—H13	119.7
H4B—C4—H4C	109.5	C12—C13—H13	119.7
O2—C5—C6	122.92 (17)	C13—C14—C9	120.8 (2)
O2—C5—C10	116.22 (18)	C13—C14—H14	119.6
C6—C5—C10	120.86 (18)	C9—C14—H14	119.6
C3—N1—O1—C1	176.83 (17)	C7—C8—C9—C14	178.56 (19)
N1—O1—C1—C2	178.10 (15)	C14—C9—C10—C11	-2.1 (3)
O1—C1—C2—C2 ⁱ	176.3 (2)	C8—C9—C10—C11	176.85 (18)
O1—N1—C3—C6	178.15 (15)	C14—C9—C10—C5	178.64 (16)
O1—N1—C3—C4	-2.3 (3)	C8—C9—C10—C5	-2.4 (3)
O2—C5—C6—C7	177.98 (17)	O2—C5—C10—C9	-176.58 (16)
C10—C5—C6—C7	-2.5 (3)	C6—C5—C10—C9	3.8 (3)
O2—C5—C6—C3	-0.4 (3)	O2—C5—C10—C11	4.2 (3)
C10—C5—C6—C3	179.16 (17)	C6—C5—C10—C11	-175.34 (18)
N1—C3—C6—C5	7.8 (3)	C9—C10—C11—C12	0.5 (3)
C4—C3—C6—C5	-171.77 (19)	C5—C10—C11—C12	179.74 (19)
N1—C3—C6—C7	-170.53 (17)	C10—C11—C12—C13	1.5 (4)
C4—C3—C6—C7	9.9 (3)	C11—C12—C13—C14	-2.0 (4)
C5—C6—C7—C8	-0.4 (3)	C12—C13—C14—C9	0.3 (3)
C3—C6—C7—C8	178.04 (18)	C10—C9—C14—C13	1.7 (3)
C6—C7—C8—C9	1.8 (3)	C8—C9—C14—C13	-177.2 (2)
C7—C8—C9—C10	-0.4 (3)		

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

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>
O2—H2 \cdots N1	0.82	1.84	2.557 (2)	145

Fig. 1

