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2,2′-[(Butane-1,4-diyldioxy)bis(nitrilomethylidyne)]dinaphthalene

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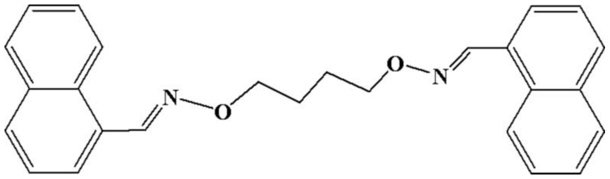
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.120; data-to-parameter ratio = 13.7.

The title complex, $C_{26}H_{24}N_2O_2$, was synthesized by the reaction of 1-naphthaldehyde with 1,4-bis(aminooxy)butane in ethanol. The molecule has crystallographic inversion symmetry. The intermolecular distance between the nearest naphthalene rings is 3.141 (2) Å, indicating a strong intermolecular π–π stacking interaction.

Related literature

Related structures were reported by Dong, Duan *et al.* (2006); Dong & Feng (2006); Dong, Feng & Yang (2006); Duan *et al.* (2007).

For related literature, see: Akine *et al.* (2001, 2006); Akine, Takanori, Dong & Nabeshima (2005); Akine, Takanori, Taniguchi & Nabeshima (2005); Aysegul *et al.* (2005); Cordes & Jencks (1962); Koehler *et al.* (1964); Lacroix (2001); Niederhoffer *et al.* (1984); Srinivasan *et al.* (1986); Sundari *et al.* (1997); Tisato *et al.* (1994); Zhang *et al.* (1990).



Experimental

Crystal data

$C_{26}H_{24}N_2O_2$
M_r = 396.47
Monoclinic, $P2_1/c$
a = 9.0910 (18) Å
b = 5.8499 (13) Å
c = 20.019 (3) Å
β = 94.779 (2)°
V = 1061.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 273 (2) K
0.37 × 0.33 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.971, T_{max} = 0.988
5177 measured reflections
1868 independent reflections
1031 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.121
S = 0.99
1868 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2003); 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: OM2146).

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supplementary materials

Acta Cryst. (2007). E**63**, o4080 [**doi:10.1107/S1600536807040822**]

2,2'-[(Butane-1,4-diyldioxy)bis(nitrilomethylidyne)]dinaphthalene

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Comment

Schiff bases have been widely used as versatile ligands in the formation of transition metal complexes (Aysegul *et al.*, 2005). They have been found to possess potent activities including inorganic biochemistry (Niederhoffer *et al.*, 1984), catalysis (Srinivasan *et al.*, 1986; Zhang *et al.*, 1990), medical imaging (Tisato *et al.*, 1994), optical materials (Lacroix, 2001) and thin films (Sundari *et al.*, 1997). Although most Schiff base derivatives are stable in solution and in the solid state, C=N bonds often suffer an exchange reaction (Koehler *et al.*, 1964) as well as hydrolysis (Cordes & Jencks, 1962). Rate constants of oxime formation are smaller than those of imine formation, and the equilibrium constants are larger by several orders of magnitude (Akine, Takanori, Taniguchi & Nabeshima, 2005). Hence, the title compound should be stable enough to resist the metathesis of the C=N bonds (Akine *et al.*, 2001). In recent years, we have been very much interested in the chemistry of salen-type derivatives, such as 2,2'-[(1,4-butylene)dioxybis(nitrilomethylidyne)]dinaphthol (Dong, Duan *et al.*, 2006), 4,4'-dibromo-2,2'-[ethylenedioxybis(nitrilomethylidyne)]diphenol (Dong & Feng, 2006),4,4'-dibromo-2,2'-[(1,3-propylene) dioxybis(nitrilomethylidyne)]diphenol (Dong, Feng & Yang, 2006), and 2,2'-[(propane-1,3-diyldioxy)bis(nitrilomethylidyne)]diphenol (Duan *et al.*, 2007). In this paper, a bisoxime ligand, 2,2'-[(1,4-butylene)dioxybis(nitrilomethylidyne)]dinaphthane was designed and synthesized, and shown in Fig. 1. The molecule is disposed about a crystallographic centre of symmetry, and the molecule adopts an extended conformation where the two naphthaldoxime moieties are apart from each other. The oxime groups have the anti-conformation, which is similar to what is observed in our previously reported salen-type bisoxime of 2,2'-[(1,4-butylene)dioxybis(nitrilomethylidyne)]dinaphthol (Dong, Duan *et al.*, 2006). Noteworthy is that the distance between the nearest naphthane rings, parallel to that of another molecule, is 3.141 (2) Å, revealing a strong intermolecular π-π stacking interaction as shown in Fig. 2.

Experimental

2,2'-[(1,4-butylene)dioxybis(nitrilomethylidyne)]dinaphthane was synthesized according to an analogous method reported earlier (Akine, Takanori, Dong & Nabeshima, 2005; Akine *et al.*, 2006). To an ethanol solution (5 ml) of 1-naphthaldehyde (213.6 mg, 1.36 mmol) was added an ethanol (3 ml) solution of 1,4-bis(aminooxy)butane (70.8 mg, 0.67 mmol). The solution was stirred at 328 K for 5 h, then concentrated to about 2 ml under reduced pressure. The precipitate was filtered and washed successively with ethanol and hexane. The product was dried under vacuum and purified by recrystallization from ethanol to yield 182.5 mg of the title compound. Yield, 68.7%. mp. 363–364 K. Anal. Calc. for $C_{26}H_{24}N_2O_2$: C, 78.76; H, 6.10; N, 7.07. Found: C, 78.68; H, 6.03; N, 7.21. IR: νC=N, 1624 cm^{-1} and νC-O, 1175 $cm^{-1.}$ ^{1}H NMR (400 MHz, $CDCl_3$) 1.97 (s, 4H), 4.36 (s, 4H), 7.46 (t, J= 7.6 Hz, 4H), 7.51 (t, J= 7.8 Hz, 2H), 7.55 (dd, J= 7.0 Hz, 2H), 7.75 (d, J= 7.8 Hz, 2H), 7.86 (d, J= 8.0 Hz, 2H), 8.54(d, J= 8.8 Hz, 2H), 8.74 (s, 2H). Colorless block-shaped single crystals suitable for X-ray diffraction studies were obtained after several weeks by slow evaporation from an ethanol solution of 2,2'-[(1,4-butylene)dioxybis(nitrilomethylidyne)]dinaphthane.

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), and U_{iso}(H) = 1.2 U_{eq}(C).

Figures

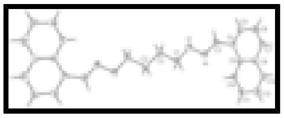

Fig. 1. The molecule structure with the atom numbering scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

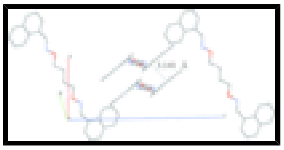

Fig. 2. A view showing the formation of π-π interactions.

2,2'-[(Butane-1,4-diyldioxy)bis(nitrilomethylidyne)]dinaphthalene

Crystal data

$C_{26}H_{24}N_2O_2$	F_{000} = 420
M_r = 396.47	D_x = 1.241 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.0910 (18) Å	Cell parameters from 625 reflections
b = 5.8499 (13) Å	θ = 2.3–20.6°
c = 20.019 (3) Å	μ = 0.08 mm^{-1}
β = 94.779 (2)°	T = 273 (2) K
V = 1061.0 (3) Å^3	Block, colorless
Z = 2	0.37 × 0.33 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1868 independent reflections
Radiation source: fine-focus sealed tube	1031 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
T = 273(2) K	θ_{max} = 25.1°
φ and ω scans	θ_{min} = 2.0°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
T_{min} = 0.971, T_{max} = 0.988	$k = -6 \rightarrow 6$
5177 measured reflections	$l = -23 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.121$

$S = 0.99$

1868 reflections

136 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.10$ e Å^{-3}

$\Delta\rho_{min} = -0.11$ e Å^{-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 > 2sigma(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	0.2816 (2)	0.9484 (3)	0.08370 (9)	0.0617 (5)
O1	0.29043 (17)	1.1226 (2)	0.03499 (7)	0.0710 (5)
C1	0.4221 (3)	1.2525 (4)	0.05003 (11)	0.0651 (7)
H1A	0.5076	1.1527	0.0527	0.078*
H1B	0.4185	1.3296	0.0928	0.078*
C2	0.4328 (2)	1.4251 (3)	−0.00515 (11)	0.0621 (6)
H2A	0.4349	1.3451	−0.0475	0.075*
H2B	0.3453	1.5207	−0.0079	0.075*
C3	0.1568 (3)	0.8486 (4)	0.07499 (10)	0.0578 (6)
H3	0.0902	0.9063	0.0414	0.069*
C4	0.1082 (2)	0.6522 (3)	0.11266 (10)	0.0515 (6)
C5	−0.0180 (3)	0.5460 (4)	0.08575 (11)	0.0639 (6)
H5	−0.0691	0.6097	0.0481	0.077*
C6	−0.0726 (3)	0.3454 (4)	0.11292 (12)	0.0716 (7)
H6	−0.1583	0.2776	0.0935	0.086*
C7	0.0009 (3)	0.2513 (4)	0.16784 (12)	0.0670 (7)
H7	−0.0326	0.1145	0.1847	0.080*
C8	0.1274 (2)	0.3570 (3)	0.19992 (11)	0.0553 (6)

C9	0.1829 (2)	0.5628 (3)	0.17299 (10)	0.0496 (6)
C10	0.3057 (2)	0.6688 (4)	0.20813 (11)	0.0605 (6)
H10	0.3429	0.8035	0.1915	0.073*
C11	0.3708 (3)	0.5778 (4)	0.26582 (12)	0.0726 (7)
H11	0.4514	0.6509	0.2881	0.087*
C12	0.3168 (3)	0.3742 (4)	0.29177 (13)	0.0786 (8)
H12	0.3613	0.3134	0.3314	0.094*
C13	0.1998 (3)	0.2660 (4)	0.25924 (13)	0.0712 (7)
H13	0.1666	0.1292	0.2763	0.085*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0703 (14)	0.0557 (11)	0.0602 (12)	−0.0035 (10)	0.0109 (10)	0.0136 (9)
O1	0.0752 (12)	0.0672 (10)	0.0696 (11)	−0.0148 (9)	0.0007 (8)	0.0229 (8)
C1	0.0700 (18)	0.0629 (15)	0.0626 (15)	−0.0090 (13)	0.0065 (12)	0.0065 (12)
C2	0.0685 (16)	0.0572 (14)	0.0603 (14)	−0.0061 (11)	0.0040 (12)	0.0096 (11)
C3	0.0609 (16)	0.0590 (14)	0.0538 (14)	0.0015 (12)	0.0072 (11)	0.0073 (11)
C4	0.0541 (14)	0.0513 (13)	0.0503 (13)	0.0003 (11)	0.0113 (11)	−0.0043 (11)
C5	0.0660 (16)	0.0705 (16)	0.0558 (14)	−0.0066 (14)	0.0085 (12)	−0.0017 (12)
C6	0.0682 (17)	0.0765 (17)	0.0709 (17)	−0.0187 (14)	0.0107 (13)	−0.0089 (14)
C7	0.0762 (18)	0.0506 (14)	0.0781 (18)	−0.0114 (13)	0.0290 (14)	−0.0064 (13)
C8	0.0615 (16)	0.0474 (13)	0.0595 (14)	0.0048 (12)	0.0204 (12)	0.0014 (11)
C9	0.0507 (13)	0.0489 (13)	0.0508 (13)	0.0059 (11)	0.0132 (11)	−0.0002 (10)
C10	0.0585 (15)	0.0623 (14)	0.0607 (15)	−0.0027 (12)	0.0060 (12)	0.0076 (12)
C11	0.0614 (16)	0.0891 (18)	0.0664 (17)	0.0012 (14)	−0.0001 (13)	0.0146 (14)
C12	0.0694 (19)	0.094 (2)	0.0727 (17)	0.0194 (16)	0.0103 (14)	0.0299 (15)
C13	0.0727 (19)	0.0606 (16)	0.0838 (19)	0.0141 (14)	0.0278 (15)	0.0187 (14)

Geometric parameters (Å, °)

N1—C3	1.274 (3)	C6—C7	1.355 (3)
N1—O1	1.417 (2)	C6—H6	0.9300
O1—C1	1.429 (2)	C7—C8	1.412 (3)
C1—C2	1.506 (3)	C7—H7	0.9300
C1—H1A	0.9700	C8—C13	1.413 (3)
C1—H1B	0.9700	C8—C9	1.428 (3)
C2—C2^{i}	1.504 (4)	C9—C10	1.413 (3)
C2—H2A	0.9700	C10—C11	1.361 (3)
C2—H2B	0.9700	C10—H10	0.9300
C3—C4	1.463 (3)	C11—C12	1.404 (3)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.375 (3)	C12—C13	1.357 (3)
C4—C9	1.434 (3)	C12—H12	0.9300
C5—C6	1.402 (3)	C13—H13	0.9300
C5—H5	0.9300		
C3—N1—O1	109.64 (17)	C7—C6—H6	120.4
N1—O1—C1	109.40 (15)	C5—C6—H6	120.4

O1—C1—C2	107.81 (17)	C6—C7—C8	121.3 (2)
O1—C1—H1A	110.1	C6—C7—H7	119.4
C2—C1—H1A	110.1	C8—C7—H7	119.4
O1—C1—H1B	110.1	C7—C8—C13	121.4 (2)
C2—C1—H1B	110.1	C7—C8—C9	119.82 (19)
H1A—C1—H1B	108.5	C13—C8—C9	118.7 (2)
C2^{i}—C2—C1	113.0 (2)	C10—C9—C8	118.12 (18)
C2^{i}—C2—H2A	109.0	C10—C9—C4	123.95 (19)
C1—C2—H2A	109.0	C8—C9—C4	117.91 (18)
C2^{i}—C2—H2B	109.0	C11—C10—C9	121.4 (2)
C1—C2—H2B	109.0	C11—C10—H10	119.3
H2A—C2—H2B	107.8	C9—C10—H10	119.3
N1—C3—C4	126.4 (2)	C10—C11—C12	120.3 (2)
N1—C3—H3	116.8	C10—C11—H11	119.9
C4—C3—H3	116.8	C12—C11—H11	119.9
C5—C4—C9	119.1 (2)	C13—C12—C11	120.2 (2)
C5—C4—C3	115.5 (2)	C13—C12—H12	119.9
C9—C4—C3	125.35 (19)	C11—C12—H12	119.9
C4—C5—C6	122.5 (2)	C12—C13—C8	121.3 (2)
C4—C5—H5	118.8	C12—C13—H13	119.4
C6—C5—H5	118.8	C8—C13—H13	119.4
C7—C6—C5	119.2 (2)		

C3—N1—O1—C1	172.74 (18)	C7—C8—C9—C4	−1.3 (3)
N1—O1—C1—C2	176.28 (17)	C13—C8—C9—C4	179.82 (19)
O1—C1—C2—C2^{i}	179.6 (2)	C5—C4—C9—C10	−174.4 (2)
O1—N1—C3—C4	176.55 (19)	C3—C4—C9—C10	7.1 (3)
N1—C3—C4—C5	−167.4 (2)	C5—C4—C9—C8	4.1 (3)
N1—C3—C4—C9	11.1 (3)	C3—C4—C9—C8	−174.37 (19)
C9—C4—C5—C6	−3.6 (3)	C8—C9—C10—C11	0.4 (3)
C3—C4—C5—C6	175.0 (2)	C4—C9—C10—C11	179.0 (2)
C4—C5—C6—C7	0.1 (4)	C9—C10—C11—C12	0.2 (3)
C5—C6—C7—C8	2.9 (3)	C10—C11—C12—C13	0.4 (4)
C6—C7—C8—C13	176.6 (2)	C11—C12—C13—C8	−1.6 (4)
C6—C7—C8—C9	−2.2 (3)	C7—C8—C13—C12	−176.6 (2)
C7—C8—C9—C10	177.3 (2)	C9—C8—C13—C12	2.2 (3)
C13—C8—C9—C10	−1.6 (3)		

Symmetry codes: (i) $-x+1, -y+3, -z$.

Fig. 1

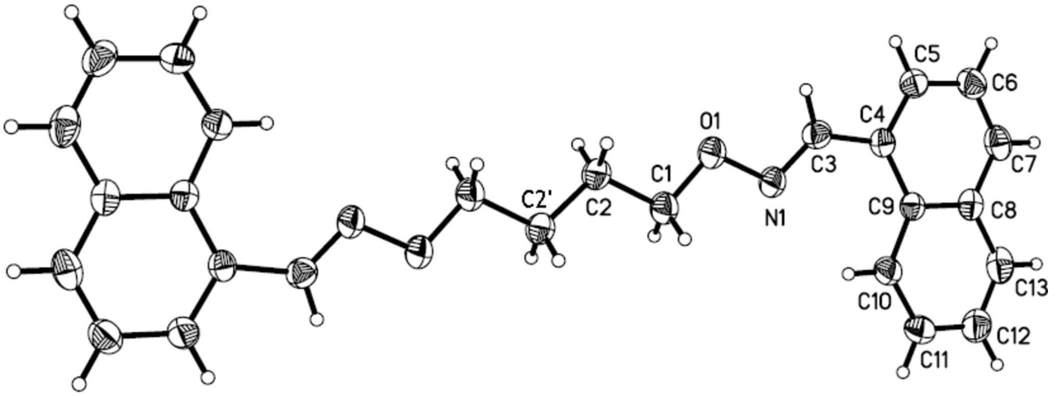

Fig. 2