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5,5′-Bis(diethylamino)-2,2′-[ethylenedioxybis(nitrilomethylidyne)]diphenol

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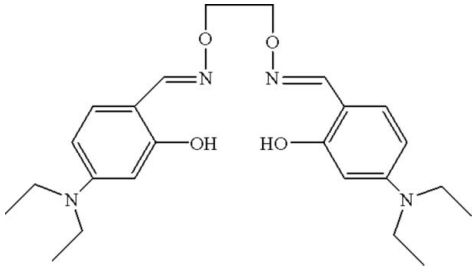
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.123; data-to-parameter ratio = 14.4.

The centrosymmetric title compound, $C_{24}H_{34}N_4O_4$, has been characterized structurally by ^{1}H NMR and X-ray crystallography. The two benzene rings are parallel to each other. The compound forms intramolecular hydrogen bonds, where the oxime group acts as a hydrogen-bond donor and the OH group acts as a hydrogen-bond acceptor.

Related literature

For related literature, see: Atwood & Harvey (2001); Bunzli & Piguet (2002); Campbell & Nguyen (2001); Costes *et al.* (2000); Di Bella & Fragala (2000); Dong *et al.* (2007); Katsuki (1995); Lacroix (2001); Mohand *et al.* (1995); Morris *et al.* (2001); Sun *et al.* (2004).



Experimental

Crystal data

$C_{24}H_{34}N_4O_4$
M_r = 442.55
Monoclinic, $P2_1/c$
a = 7.3705 (10) Å
b = 18.386 (2) Å
c = 8.9368 (16) Å
β = 97.063 (2)°
V = 1201.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.56 × 0.46 × 0.40 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.954, T_{max} = 0.967
6175 measured reflections
2114 independent reflections
1249 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.05
2114 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 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)	1.96 (2)	2.68 (2)	146 (1)

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: HG2270).

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supplementary materials

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5,5'-Bis(diethylamino)-2,2'-[ethylenedioxybis(nitrilomethylidyne)]diphenol

Y.-P. Zhang, X. Chen, J.-Y. Shi, L. Xu and W.-K. Dong

Comment

There has been a growing interest in Schiff-base ligands, mainly because of their wide application in the fields of biochemistry, catalysis (Mohand *et al.*, 1995; Campbell & Nguyen, 2001), and synthesis of new ligands and their complexes (Atwood & Harvey, 2001; Morris *et al.*, 2001). A series of *N*,*N*'-bis(salicylidence)ethylenediamine (salen) and its analogues have been used as catalysts in various organic reactions (Katsuki, 1995), nonlinear optical materials (Di Bella & Fragala, 2000; Lacroix, 2001), and exhibit interesting magnetic properties (Costes *et al.*, 2000; Bunzli & Piguet, 2002). In addition, in biological and artificial systems allosteric regulation is effective in controlling molecular functions, such as molecular recognition and biological activity (Sun *et al.*, 2004). In context with this background, we report here on the crystal structure of 5,5'-di(*N*,*N*'-diethylamino)-2,2'- [ethylenedioxybis(nitrilomethylidyne)]diphenol (I), shown in Fig. 1. The molecule adopts an extended conformation where the two salicylaldoxime moieties are apart from each other. The oxime groups and phenolic groups have the anti-conformation, and there is an intramolecular hydrogen bond, O2—H2···N1(*d*(O2—H2) = 0.82 (1) Å, d(H2···N1) = 1.96 (2) Å, d(O2···N1) = 2.68 (2) Å, <O2—H2···N1 = 146.0 (1) °).

Experimental

5,5'-Di(*N*,*N*'-diethylamino)-2,2'-[ethylenedioxybis (nitrilomethylidyne)]diphenol (I) was synthesized according to our previous work (Dong *et al.*, 2007). A solution of 1, 2-bis(aminooxy)ethane (48.5 mg, 0.53 mmol) in ethanol(4 ml) was added to a solution of 4-(*N*,*N*-Diethylamino)-2-hydroxybenzaldehyde (204.0 mg, 1.06 mmol) in ethanol(4 ml), and the mixture solution was heated to 328 K under stirring for 4 h. The solution was concentrated to 2 ml *in vacuo*. After cooling to room temperature, the precipitate was filtered, and washed successively with ethanol-hexane (1:4) and hexane, respectively. The product was dried under reduced pressure, and purified with recrystallization from ethanol to yield colorless crystals. Yield, 68.6%. mp. 398 − 399 K. Anal. Calcd for $C_{24}H_{34}N_4O_4$: C, 65.14; H, 7.74; N, 12.66. Found: C, 65.10; H, 7.65; N, 12.68. ^{1}H NMR (400 MHz, $CDCl_3$): 1.17 (t, J= 7.4 Hz, 12H), 3.35 (dd, J= 14.0 Hz, 6.8 Hz, 8H), 4.38 (s, 4H), 6.20 (d, J= 2.8 Hz, 2H), 6.22 (t, J= 2.4 Hz, 2H), 6.94(d, J= 8.8 Hz, 2H), 8.11 (s, 2H), 9.80 (s, 2H). Colorless prismatic single crystals suitable for X-ray diffraction studies were obtained after about one month by slow evaporation at room temperature from an acetone solution of 5,5'-di(*N*,*N*'-diethylamino)-2,2'-[ethylenedioxybis (nitrilomethylidyne)]diphenol.

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

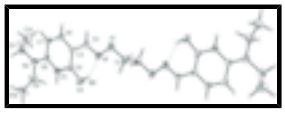

Fig. 1. Molecular structure of (I)

5,5'-Bis(diethylamino)-2,2'-[ethylenedioxybis(nitrilomethylidyne)]diphenol

Crystal data

$C_{24}H_{34}N_4O_4$	F_{000} = 476
M_r = 442.55	D_x = 1.223 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1428 reflections
a = 7.3705 (10) Å	θ = 2.2–22.2º
b = 18.386 (2) Å	μ = 0.08 mm^{-1}
c = 8.9368 (16) Å	T = 298 (2) K
β = 97.063 (2)º	Prism, colorless
V = 1201.9 (3) Å^3	0.56 × 0.46 × 0.40 mm
Z = 2	

Data collection

Bruker SMART CCD area-detector diffractometer	2114 independent reflections
Radiation source: fine-focus sealed tube	1249 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −6→8
T_{min} = 0.954, T_{max} = 0.967	k = −19→21
6175 measured reflections	l = −10→10

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.123	$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 0.4459P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
2114 reflections	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
147 parameters	$\Delta\rho_{min}$ = −0.16 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.7756 (3)	0.48722 (11)	0.1956 (2)	0.0493 (5)
N2	0.3985 (2)	0.32076 (11)	0.7116 (2)	0.0521 (6)
O1	0.7825 (2)	0.52817 (9)	0.06124 (18)	0.0582 (5)
O2	0.8711 (2)	0.41916 (11)	0.4589 (2)	0.0755 (6)
H2	0.8884	0.4424	0.3836	0.113*
C1	0.9679 (3)	0.53421 (13)	0.0339 (3)	0.0526 (7)
H1A	1.0441	0.5442	0.1280	0.063*
H1B	0.9800	0.5746	−0.0340	0.063*
C2	0.6097 (3)	0.47704 (12)	0.2187 (2)	0.0432 (6)
H2A	0.5166	0.4960	0.1500	0.052*
C3	0.5613 (3)	0.43738 (12)	0.3469 (2)	0.0381 (5)
C4	0.6890 (3)	0.40947 (13)	0.4609 (3)	0.0449 (6)
C5	0.6359 (3)	0.37169 (13)	0.5808 (2)	0.0452 (6)
H5	0.7245	0.3536	0.6544	0.054*
C6	0.4517 (3)	0.35996 (12)	0.5943 (2)	0.0403 (5)
C7	0.3223 (3)	0.38893 (12)	0.4812 (2)	0.0439 (6)
H7	0.1981	0.3825	0.4866	0.053*
C8	0.3788 (3)	0.42628 (12)	0.3639 (2)	0.0433 (6)
H8	0.2904	0.4454	0.2913	0.052*
C9	0.5293 (3)	0.28426 (14)	0.8212 (3)	0.0605 (7)
H9A	0.6319	0.2680	0.7716	0.073*
H9B	0.4719	0.2416	0.8585	0.073*
C10	0.5996 (4)	0.33191 (18)	0.9531 (3)	0.0809 (9)
H10A	0.6590	0.3738	0.9174	0.121*
H10B	0.6853	0.3050	1.0214	0.121*
H10C	0.4992	0.3473	1.0045	0.121*
C11	0.2080 (3)	0.31521 (14)	0.7367 (3)	0.0580 (7)
H11A	0.1447	0.3591	0.6995	0.070*
H11B	0.2016	0.3124	0.8443	0.070*
C12	0.1107 (4)	0.25025 (17)	0.6610 (3)	0.0808 (9)
H12A	0.0987	0.2565	0.5536	0.121*

H12B	−0.0084	0.2459	0.6931	0.121*
H12C	0.1800	0.2070	0.6882	0.121*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0503 (13)	0.0544 (13)	0.0452 (11)	−0.0010 (10)	0.0147 (9)	0.0087 (10)
N2	0.0444 (12)	0.0628 (14)	0.0497 (12)	−0.0040 (10)	0.0083 (9)	0.0118 (11)
O1	0.0512 (10)	0.0713 (13)	0.0552 (10)	0.0023 (8)	0.0189 (8)	0.0155 (9)
O2	0.0326 (10)	0.1101 (16)	0.0841 (13)	0.0027 (9)	0.0079 (8)	0.0416 (12)
C1	0.0488 (15)	0.0563 (17)	0.0561 (16)	−0.0067 (12)	0.0205 (12)	0.0061 (12)
C2	0.0396 (13)	0.0487 (15)	0.0419 (13)	0.0039 (11)	0.0069 (10)	−0.0007 (11)
C3	0.0345 (12)	0.0416 (13)	0.0391 (13)	0.0018 (10)	0.0085 (10)	−0.0012 (10)
C4	0.0323 (13)	0.0485 (15)	0.0546 (15)	0.0012 (10)	0.0090 (11)	0.0043 (12)
C5	0.0369 (13)	0.0497 (15)	0.0481 (14)	0.0034 (10)	0.0014 (10)	0.0098 (12)
C6	0.0406 (13)	0.0401 (14)	0.0412 (13)	−0.0016 (10)	0.0098 (10)	−0.0010 (10)
C7	0.0313 (12)	0.0518 (15)	0.0491 (14)	−0.0011 (10)	0.0071 (10)	0.0009 (12)
C8	0.0373 (13)	0.0470 (14)	0.0449 (14)	0.0035 (10)	0.0022 (10)	0.0020 (11)
C9	0.0654 (17)	0.0575 (17)	0.0587 (16)	−0.0053 (13)	0.0079 (13)	0.0175 (14)
C10	0.087 (2)	0.088 (2)	0.0627 (18)	−0.0123 (17)	−0.0096 (16)	0.0095 (17)
C11	0.0563 (16)	0.0679 (19)	0.0524 (15)	−0.0083 (13)	0.0167 (12)	0.0044 (13)
C12	0.0655 (19)	0.077 (2)	0.099 (2)	−0.0174 (16)	0.0063 (16)	0.0011 (19)

Geometric parameters (Å, °)

N1—C2	1.279 (3)	C5—H5	0.9300
N1—O1	1.424 (2)	C6—C7	1.407 (3)
N2—C6	1.369 (3)	C7—C8	1.361 (3)
N2—C9	1.452 (3)	C7—H7	0.9300
N2—C11	1.452 (3)	C8—H8	0.9300
O1—C1	1.422 (2)	C9—C10	1.508 (4)
O2—C4	1.357 (2)	C9—H9A	0.9700
O2—H2	0.8200	C9—H9B	0.9700
C1—C1^{i}	1.498 (4)	C10—H10A	0.9600
C1—H1A	0.9700	C10—H10B	0.9600
C1—H1B	0.9700	C10—H10C	0.9600
C2—C3	1.439 (3)	C11—C12	1.510 (4)
C2—H2A	0.9300	C11—H11A	0.9700
C3—C8	1.387 (3)	C11—H11B	0.9700
C3—C4	1.397 (3)	C12—H12A	0.9600
C4—C5	1.373 (3)	C12—H12B	0.9600
C5—C6	1.394 (3)	C12—H12C	0.9600
C2—N1—O1	110.35 (18)	C6—C7—H7	120.0
C6—N2—C9	122.09 (19)	C7—C8—C3	123.5 (2)
C6—N2—C11	121.92 (19)	C7—C8—H8	118.3
C9—N2—C11	116.0 (2)	C3—C8—H8	118.3
C1—O1—N1	108.75 (17)	N2—C9—C10	113.2 (2)
C4—O2—H2	109.5	N2—C9—H9A	108.9

O1—C1—C1^{i}	111.2 (2)	C10—C9—H9A	108.9
O1—C1—H1A	109.4	N2—C9—H9B	108.9
C1^{i}—C1—H1A	109.4	C10—C9—H9B	108.9
O1—C1—H1B	109.4	H9A—C9—H9B	107.7
C1^{i}—C1—H1B	109.4	C9—C10—H10A	109.5
H1A—C1—H1B	108.0	C9—C10—H10B	109.5
N1—C2—C3	122.5 (2)	H10A—C10—H10B	109.5
N1—C2—H2A	118.7	C9—C10—H10C	109.5
C3—C2—H2A	118.7	H10A—C10—H10C	109.5
C8—C3—C4	116.2 (2)	H10B—C10—H10C	109.5
C8—C3—C2	120.0 (2)	N2—C11—C12	113.7 (2)
C4—C3—C2	123.8 (2)	N2—C11—H11A	108.8
O2—C4—C5	117.0 (2)	C12—C11—H11A	108.8
O2—C4—C3	121.4 (2)	N2—C11—H11B	108.8
C5—C4—C3	121.6 (2)	C12—C11—H11B	108.8
C4—C5—C6	121.3 (2)	H11A—C11—H11B	107.7
C4—C5—H5	119.3	C11—C12—H12A	109.5
C6—C5—H5	119.3	C11—C12—H12B	109.5
N2—C6—C5	121.4 (2)	H12A—C12—H12B	109.5
N2—C6—C7	121.1 (2)	C11—C12—H12C	109.5
C5—C6—C7	117.4 (2)	H12A—C12—H12C	109.5
C8—C7—C6	120.0 (2)	H12B—C12—H12C	109.5
C8—C7—H7	120.0		
C2—N1—O1—C1	−176.15 (19)	C9—N2—C6—C7	173.7 (2)
N1—O1—C1—C1^{i}	78.7 (3)	C11—N2—C6—C7	−8.3 (3)
O1—N1—C2—C3	−179.86 (19)	C4—C5—C6—N2	178.3 (2)
N1—C2—C3—C8	−178.2 (2)	C4—C5—C6—C7	−0.6 (3)
N1—C2—C3—C4	3.2 (3)	N2—C6—C7—C8	−178.5 (2)
C8—C3—C4—O2	−177.8 (2)	C5—C6—C7—C8	0.4 (3)
C2—C3—C4—O2	0.9 (3)	C6—C7—C8—C3	0.8 (3)
C8—C3—C4—C5	1.6 (3)	C4—C3—C8—C7	−1.8 (3)
C2—C3—C4—C5	−179.7 (2)	C2—C3—C8—C7	179.5 (2)
O2—C4—C5—C6	178.9 (2)	C6—N2—C9—C10	88.5 (3)
C3—C4—C5—C6	−0.4 (4)	C11—N2—C9—C10	−89.6 (3)
C9—N2—C6—C5	−5.2 (3)	C6—N2—C11—C12	91.9 (3)
C11—N2—C6—C5	172.8 (2)	C9—N2—C11—C12	−90.0 (3)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.82 (1)	1.96 (2)	2.68 (2)	146 (1)

Fig. 1