organic compounds

1,3-Bis[(4-methylbenzylidene)amino-oxy]propane

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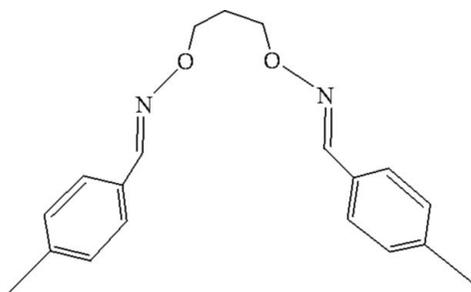Correspondence e-mail: dongwk@126.com

The title bisoxime compound, $C_{19}H_{22}N_2O_2$, synthesized by the reaction of 4-methyl-2-hydroxybenzaldehyde with 1,3-bis-(aminooxy)propane in ethanol, adopts a V-shaped conformation. The dihedral angle between the rings is 84.59 (3)°. The molecule is disposed about a crystallographic twofold rotation axis, with one C atom lying on the axis. In the crystal, molecules are packed by C—H···π(Ph) interactions, forming chains.

Related literature

For bisoximes and their applications, see: Akine *et al.* (2005); Atwood & Harvey (2001); Dong *et al.* (2008, 2009); He *et al.* (2008); Yeap *et al.* (2008).

Experimental

Crystal data

$C_{19}H_{22}N_2O_2$	$V = 1740.7$ (3) Å^3
$M_r = 310.39$	$Z = 4$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 29.843$ (2) Å	$\mu = 0.08$ mm^{-1}
$b = 4.8668$ (7) Å	$T = 298$ K
$c = 12.1202$ (11) Å	$0.43 \times 0.13 \times 0.07$ mm
$\beta = 98.568$ (1)°	

Data collection

Siemens SMART CCD area-detector diffractometer	4227 measured reflections
	1530 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	831 reflections with $I > 2\sigma(I)$
$T_{min} = 0.968$, $T_{max} = 0.995$	$R_{int} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$	106 parameters
$wR(F^2) = 0.175$	H-atom parameters constrained
$S = 1.12$	$\Delta\rho_{max} = 0.13$ e Å^{-3}
1530 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10C···Cg1	0.96	2.73	3.614 (2)	153

Cg1 is the centroid of the C4–C9 ring.

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

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1,3-Bis[(4-methylbenzylidene)aminooxy]propane

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S1. Comment

Much interest has been focused on bisoxime compounds, in which the large electronegativity of O atoms is expected to affect strongly the electronic properties of the nitrogen atoms, and exhibit high stability against imine metathesis reactions (Akine *et al.*, 2005). Some of them or their metal complexes are used in wide field due to their variety of applications, especially for catalysis and biological processes, magnetism, and supramolecular architectures (Atwood *et al.*, 2001; Yeap *et al.*, 2008; Dong *et al.*, 2008). Herein, the synthesis and structure of 4,4′-dimethyl-1,3-[propenedioxy-bis(nitrilomethylidyne)]dibenzene (I) is reported (Fig. 1).

The single-crystal structure of (I) is built up by discrete $C_{19}H_{22}N_2O_2$ molecules, in which all bond lengths are in normal ranges. The title compound adopts a V-shaped configuration with the dihedral angle between the two halves of the molecule is 85.82 (3)°. The molecules are disposed about a crystallographic two-fold rotation axis. This structure is similar to that observed in our previously reported salen-type bisoxime compounds (He *et al.*, 2008). The packing of the molecule is controlled by C—H···π(Ph) interactions linking molecules into infinite supramolecular structure along *b* axis.

S2. Experimental

4,4′-Dimethyl-1,3-[propenedioxybis(nitrilomethylidyne)]dibenzene was synthesized according to an analogous method reported earlier (Dong *et al.*, 2009). To an ethanol solution (4 ml) of 4-methyl-2-hydroxybenzaldehyde (243.2 mg, 2.02 mmol) was added an ethanol solution (4 ml) of 1,3-bis(aminooxy)propane (108.3 mg, 1.02 mmol). The reaction mixture was stirred at 328–333 K for 14 h. After cool to room temperature, no precipitate was formed, which was concentrated to about 1 ml under reduced pressure. The precipitate formed was separated by filtration, and washed several times with n-hexane. The product was dried under vacuum to yield 189.4 mg of (I). Yield, 60.4%. m. p. 329–330 K. Anal. Calcd. for $C_{19}H_{22}N_2O_2$: C, 73.52; H, 7.14; N, 9.03. Found: C, 73.49; H, 7.01; N, 9.39.

Colorless needle-like single crystals suitable for X-ray diffraction studies were obtained after about four weeks by slow evaporation from an acetonitrile solution of (I).

S3. Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.96 Å (CH_3), 0.97 Å (CH_2), 0.93 Å (CH) and $U_{iso}(H) = 1.2\ U_{eq}(C)$.
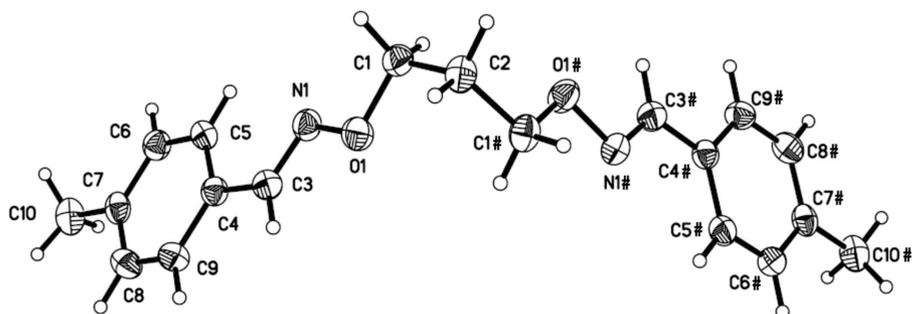
Figure 1

The molecule structure of the title compound with atom numbering [Symmetry codes: -*x*, *y*, 1/2 - *z*]. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.
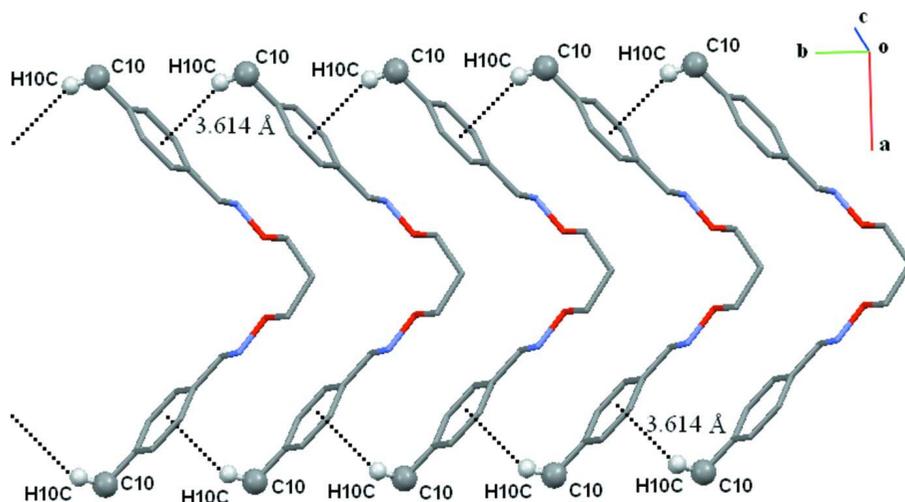

Figure 2

Part of the supramolecular structure of the title compound. C—H···*π*(Ph) interactions are shown as dashed lines.

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Crystal data

$C_{19}H_{22}N_2O_2$	$F(000) = 664$
$M_r = 310.39$	$D_x = 1.184$ Mg m^{-3}
Monoclinic, $C2/c$	Melting point = 329–330 K
Hall symbol: -C 2yc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 29.843$ (2) Å	Cell parameters from 780 reflections
$b = 4.8668$ (7) Å	$\theta = 2.8$–$25.2°$
$c = 12.1202$ (11) Å	$\mu = 0.08$ mm^{-1}
$\beta = 98.568$ (1)°	$T = 298$ K
$V = 1740.7$ (3) Å^3	Needle-like, colorless
$Z = 4$	$0.43 \times 0.13 \times 0.07$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
Radiation source: fine-focus sealed tube	$T_{min} = 0.968$, $T_{max} = 0.995$
Graphite monochromator	4227 measured reflections
phi and ω scans	1530 independent reflections

831 reflections with $I > 2\sigma(I)$		$h = -34 \rightarrow 25$
$R_{int} = 0.062$		$k = -5 \rightarrow 5$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.8°$		$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.058$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.175$	
$S = 1.12$	H-atom parameters constrained
1530 reflections	$w = 1/[\sigma^2(F_o^2) + (0.071P)^2]$
106 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.13$ e Å^{-3}
	$\Delta\rho_{min} = -0.20$ 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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
N1	0.58112 (8)	0.7298 (5)	0.3832 (2)	0.0491 (7)
O1	0.54391 (7)	0.5473 (4)	0.36901 (16)	0.0562 (7)
C1	0.54272 (10)	0.4006 (7)	0.2661 (2)	0.0530 (9)
H1A	0.5691	0.2826	0.2691	0.064*
H1B	0.5426	0.5283	0.2046	0.064*
C2	0.5000	0.2312 (9)	0.2500	0.0532 (12)
H2	0.4999	0.1136	0.3146	0.064*
C3	0.58513 (10)	0.8441 (7)	0.4781 (2)	0.0493 (8)
H3	0.5646	0.7979	0.5258	0.059*
C4	0.62026 (9)	1.0435 (6)	0.5161 (2)	0.0436 (8)
C5	0.65245 (10)	1.1316 (7)	0.4504 (2)	0.0494 (8)
H5	0.6518	1.0609	0.3790	0.059*
C6	0.68507 (10)	1.3223 (7)	0.4909 (2)	0.0518 (9)
H6	0.7060	1.3778	0.4458	0.062*
C7	0.68736 (10)	1.4340 (6)	0.5977 (2)	0.0487 (8)
C8	0.65537 (11)	1.3458 (7)	0.6621 (2)	0.0558 (9)
H8	0.6561	1.4163	0.7336	0.067*
C9	0.62262 (10)	1.1564 (6)	0.6225 (2)	0.0518 (8)
H9	0.6016	1.1026	0.6678	0.062*
C10	0.72298 (11)	1.6435 (7)	0.6406 (3)	0.0653 (10)
H10A	0.7515	1.5904	0.6194	0.098*
H10B	0.7258	1.6534	0.7204	0.098*

H10C	0.7143	1.8201	0.6092	0.098*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0415 (15)	0.0518 (18)	0.0529 (15)	−0.0036 (13)	0.0040 (11)	−0.0018 (13)
O1	0.0463 (13)	0.0644 (16)	0.0579 (13)	−0.0126 (11)	0.0075 (10)	−0.0103 (12)
C1	0.0468 (19)	0.052 (2)	0.0585 (18)	0.0048 (17)	0.0032 (14)	−0.0096 (17)
C2	0.050 (3)	0.044 (3)	0.063 (3)	0.000	−0.001 (2)	0.000
C3	0.0466 (18)	0.054 (2)	0.0473 (17)	0.0002 (16)	0.0078 (13)	−0.0002 (16)
C4	0.0435 (18)	0.0414 (19)	0.0448 (16)	0.0022 (14)	0.0030 (13)	0.0016 (14)
C5	0.0487 (19)	0.058 (2)	0.0404 (15)	0.0010 (17)	0.0023 (14)	−0.0006 (15)
C6	0.0481 (19)	0.055 (2)	0.0524 (18)	0.0002 (17)	0.0069 (14)	0.0061 (16)
C7	0.053 (2)	0.0372 (19)	0.0520 (17)	0.0036 (16)	−0.0058 (15)	0.0032 (16)
C8	0.071 (2)	0.051 (2)	0.0448 (16)	0.0016 (18)	0.0070 (16)	−0.0050 (16)
C9	0.057 (2)	0.055 (2)	0.0456 (17)	−0.0020 (17)	0.0117 (14)	−0.0012 (16)
C10	0.069 (2)	0.050 (2)	0.071 (2)	−0.0028 (19)	−0.0081 (18)	0.0022 (18)

Geometric parameters (Å, º)

N1—C3	1.268 (3)	C5—C6	1.381 (4)
N1—O1	1.412 (3)	C5—H5	0.9300
O1—C1	1.433 (3)	C6—C7	1.397 (4)
C1—C2	1.506 (4)	C6—H6	0.9300
C1—H1A	0.9700	C7—C8	1.388 (4)
C1—H1B	0.9700	C7—C10	1.508 (4)
C2—C1^i	1.506 (4)	C8—C9	1.377 (4)
C2—H2	0.9700	C8—H8	0.9300
C3—C4	1.452 (4)	C9—H9	0.9300
C3—H3	0.9300	C10—H10A	0.9600
C4—C9	1.394 (4)	C10—H10B	0.9600
C4—C5	1.403 (4)	C10—H10C	0.9600
C3—N1—O1	110.7 (2)	C4—C5—H5	119.7
N1—O1—C1	109.6 (2)	C5—C6—C7	121.7 (3)
O1—C1—C2	107.2 (2)	C5—C6—H6	119.2
O1—C1—H1A	110.3	C7—C6—H6	119.2
C2—C1—H1A	110.3	C8—C7—C6	117.3 (3)
O1—C1—H1B	110.3	C8—C7—C10	121.6 (3)
C2—C1—H1B	110.3	C6—C7—C10	121.1 (3)
H1A—C1—H1B	108.5	C9—C8—C7	121.6 (3)
C1—C2—C1^i	113.6 (4)	C9—C8—H8	119.2
C1—C2—H2	108.8	C7—C8—H8	119.2
C1^i—C2—H2	108.8	C8—C9—C4	121.4 (3)
N1—C3—C4	123.2 (3)	C8—C9—H9	119.3
N1—C3—H3	118.4	C4—C9—H9	119.3
C4—C3—H3	118.4	C7—C10—H10A	109.5
C9—C4—C5	117.4 (3)	C7—C10—H10B	109.5

C9—C4—C3	119.4 (3)	H10A—C10—H10B	109.5
C5—C4—C3	123.2 (3)	C7—C10—H10C	109.5
C6—C5—C4	120.7 (3)	H10A—C10—H10C	109.5
C6—C5—H5	119.7	H10B—C10—H10C	109.5
C3—N1—O1—C1	−174.3 (2)	C4—C5—C6—C7	0.1 (5)
N1—O1—C1—C2	−174.5 (2)	C5—C6—C7—C8	−0.1 (4)
O1—C1—C2—C1^i	65.14 (18)	C5—C6—C7—C10	−179.6 (3)
O1—N1—C3—C4	−179.6 (2)	C6—C7—C8—C9	−0.1 (4)
N1—C3—C4—C9	−179.1 (3)	C10—C7—C8—C9	179.4 (3)
N1—C3—C4—C5	1.2 (5)	C7—C8—C9—C4	0.3 (5)
C9—C4—C5—C6	0.2 (4)	C5—C4—C9—C8	−0.4 (4)
C3—C4—C5—C6	179.9 (3)	C3—C4—C9—C8	179.9 (3)

Symmetry code: (i) −x+1, y, −z+1/2.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10C···Cg1	0.96	2.73	3.614 (2)	153