

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,2'-[1,1'-[Pentane-1,5-diylbis(oxy-nitrilo)]diethylidene]diphenol

Wen-Kui Dong,* Zhong-Wu Lv, Xue-Ni He, Yong-Hong Guan 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

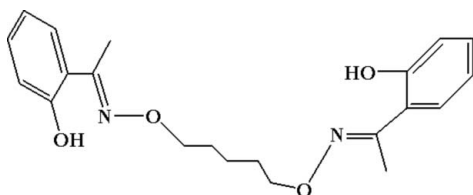
Received 28 July 2008; accepted 28 September 2008

 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.042; wR factor = 0.123; data-to-parameter ratio = 7.9.

In the title compound, $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_4$, there is half a molecule in the asymmetric unit with a crystallographic twofold rotation axis passing through the central C atom of the $-\text{CH}=\text{N}-\text{O}-$ $(\text{CH}_2)_5-\text{O}-\text{N}=\text{CH}-$ bridge. The dihedral angle formed by the two benzene rings is $80.85(2)^\circ$. Strong intramolecular $\text{O}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds help to establish the molecular conformation. There are also weak intermolecular $\pi-\pi$ stacking interactions between neighbouring benzene rings [centroid-centroid separation = $3.502(3)$ Å].

Related literature

For general background, see: Bhadbhade & Srinivas (1993). For related structures, see: Dong *et al.* (2007, 2008); Wang *et al.* (2007); Xu *et al.* (2007).



Experimental

Crystal data

$\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_4$	$V = 976.1(3)$ Å ³
$M_r = 370.44$	$Z = 2$
Monoclinic, $C2$	Mo $K\alpha$ radiation
$a = 12.9691(13)$ Å	$\mu = 0.09$ mm ⁻¹
$b = 4.601(1)$ Å	$T = 298(2)$ K
$c = 16.3639(16)$ Å	$0.48 \times 0.40 \times 0.32$ mm
$\beta = 91.621(1)^\circ$	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	2421 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	972 independent reflections
$T_{\min} = 0.959$, $T_{\max} = 0.973$	646 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$	2 restraints
$wR(F^2) = 0.123$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.15$ e Å ⁻³
972 reflections	$\Delta\rho_{\text{min}} = -0.12$ e Å ⁻³
123 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2}\cdots\text{N1}$	0.82	1.84	2.553 (4)	144
$\text{C4}-\text{H4A}\cdots\text{O1}$	0.96	2.16	2.631 (5)	109

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.

This work was supported by the Foundation of the Education Department of Gansu Province (No. 0604-01) and the 'Qing Lan' Talent Engineering Fund of Lanzhou Jiaotong University (No. QL-03-01A), which are gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2048).

References

- Bhadbhade, M. M. & Srinivas, D. (1993). *Inorg. Chem.* **32**, 6122–6130.
 Dong, W. K., He, X. N., Dong, C. M., Wang, L., Zhong, J. K., Chen, X. & Yu, T. Z. (2007). *Z. Kristallogr. New Cryst. Struct.* **222**, 289–290.
 Dong, W.-K., He, X.-N., Zhong, J.-K., Chen, X. & Yu, T.-Z. (2008). *Acta Cryst.* **E64**, o1098.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Siemens (1996). *SMART and SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Wang, L., He, X., Sun, Y. & Xu, L. (2007). *Acta Cryst.* **E63**, o4517.
 Xu, L., Zhang, Y. P., Sun, Y. X., Shi, J. Y. & Dong, W. K. (2007). *Chin. J. Struct. Chem.* **23**, 1999–2002.

supporting information

Acta Cryst. (2008). E64, o2059 [doi:10.1107/S160053680803136X]

2,2'-[1,1'-(Pentane-1,5-diylbis(oxynitrilo)]diethylidyne}diphenol

Wen-Kui Dong, Zhong-Wu Lv, Xue-Ni He, Yong-Hong Guan and Jun-Feng Tong

S1. Comment

salen-type compounds containing strong donor sites such as oxygen and imine nitrogen atoms and their metal complexes have been the subject of extensive investigation (Bhadbhade & Srinivas, 1993). Structures of salen-type compounds derived from *O*-alkyl oxime moiety ($-\text{CH}=\text{N}-\text{O}-(\text{CH}_2)_n-\text{O}-\text{N}=\text{CH}-$) instead of the imine moiety and closely related to the title compound (Wang *et al.*, 2007; Dong *et al.*, 2008; Dong *et al.*, 2007), are known. In this research, we report the synthesis and crystal structure of the title compound, and shown in Fig. 1.

The single-crystal structure of the title compound is built up by discrete $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_4$ molecules, within all bond lengths are in normal ranges. There is 1/2 molecule per asymmetric unit with a crystallographic twofold rotation axis passing through the central carbon (symmetry code: $-x, y, -z$) of the five carbon atoms in the ($-\text{CH}=\text{N}-\text{O}-(\text{CH}_2)_5-\text{O}-\text{N}=\text{CH}-$) bridge. The dihedral angle formed by the two benzene rings in the molecule of the title compound is $80.85(2)^\circ$. The strong intramolecular $\text{O2}\cdots\text{H2}\cdots\text{N1}$ and $\text{C4}\cdots\text{H4A}\cdots\text{O1}$ hydrogen bonds play an important role in the stability of the crystal structure in the title compound. The five carbon atoms in the $\text{C1}-\text{C2}-\text{C3}-\text{C2A}-\text{C1A}$ bridge are almost in the same plane with slight deviation of 0.015 and 0.029 Å below for C1, C2A, 0.015 and 0.029 Å above the plane for C2 and C1A (symmetry code A: $-x, y, -z + 1$), respectively. In the crystal structure, there is weak intermolecular $\pi-\pi$ stacking interaction between the neighbouring benzene rings, and the inter-molecular plane-to-plane distance is 3.502 (3) Å along *b* axis. This structure is not similar to what was observed in our previously reported series of salen-type compound containing two- (Wang *et al.*, 2007), three- (Dong *et al.*, 2008) and four-methene (Dong *et al.*, 2007) bridge.

S2. Experimental

2,2'-[1,1'-(Pentane-1,5-diylbis(oxynitrilo)]diethylidyne}diphenol was synthesized according to an analogous method reported earlier (Wang *et al.*, 2007; Xu *et al.*, 2007). To an ethanol solution (5 ml) of 2'-hydroxyacetophenone (516.2 mg, 4.00 mmol) was added an ethanol solution (3 ml) of 1,5-bis(aminooxy)pentane (268.4 mg, 2.00 mmol). The reaction mixture was stirred at 328 K for 4 h. The formed precipitate was separated by filtration, and washed successively with ethanol and ethanol-hexane (1:4), respectively. The product was dried under vacuum to yield 410.9 mg of the title compound. Yield, 55.5%. mp. 344–345 K. Anal. Calc. for $\text{C}_{17}\text{H}_{16}\text{Cl}_2\text{N}_2\text{O}_2$: C, 68.09; H, 7.07; N, 7.56. Found: C, 68.19; H, 7.21; N, 7.42.

Colorless block-like single crystals suitable for X-ray diffraction studies were obtained after two weeks by slow evaporation from a diethyl-ether solution of the title compound.

S3. Refinement

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

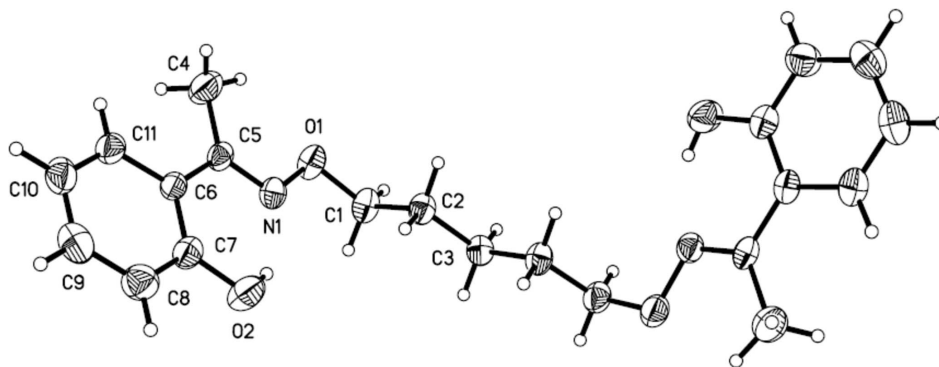


Figure 1

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

2,2'-(1,1'-[Pentane-1,5-diylbis(oxynitrilo)]diethyldiyne)diphenol

Crystal data

$C_{21}H_{26}N_2O_4$

$M_r = 370.44$

Monoclinic, $C2$

Hall symbol: $C\ 2y$

$a = 12.9691(13)\ \text{\AA}$

$b = 4.601(1)\ \text{\AA}$

$c = 16.3639(16)\ \text{\AA}$

$\beta = 91.621(1)^\circ$

$V = 976.1(3)\ \text{\AA}^3$

$Z = 2$

$F(000) = 396$

$D_x = 1.260\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 813 reflections

$\theta = 2.5\text{--}23.2^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Needle-like, colourless

$0.48 \times 0.40 \times 0.32\ \text{mm}$

Data collection

Siemens 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)

$T_{\min} = 0.959$, $T_{\max} = 0.973$

2421 measured reflections

972 independent reflections

646 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.5^\circ$

$h = -15 \rightarrow 15$

$k = -5 \rightarrow 5$

$l = -19 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.123$

$S = 1.04$

972 reflections

123 parameters

2 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

$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.2844P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.15\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.12\ \text{e \AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.1857 (2)	0.7552 (7)	0.30501 (17)	0.0533 (8)	
O1	0.23002 (19)	0.6005 (8)	0.37063 (14)	0.0656 (7)	
O2	0.0337 (2)	0.9262 (9)	0.21462 (17)	0.0796 (9)	
H2	0.0629	0.8223	0.2488	0.119*	
C1	0.1512 (3)	0.4342 (11)	0.4082 (2)	0.0607 (10)	
H1A	0.1834	0.2927	0.4447	0.073*	
H1B	0.1126	0.3286	0.3662	0.073*	
C2	0.0778 (3)	0.6170 (10)	0.4558 (2)	0.0551 (9)	
H2A	0.1169	0.7333	0.4952	0.066*	
H2B	0.0418	0.7489	0.4186	0.066*	
C3	0.0000	0.4391 (13)	0.5000	0.0526 (12)	
H3A	0.0361	0.3147	0.5392	0.063*	0.50
H3B	-0.0361	0.3147	0.4608	0.063*	0.50
C4	0.3610 (3)	0.9300 (15)	0.2926 (2)	0.0836 (14)	
H4A	0.3736	0.8041	0.3386	0.125*	
H4B	0.3787	1.1259	0.3074	0.125*	
H4C	0.4023	0.8688	0.2479	0.125*	
C5	0.2494 (3)	0.9162 (9)	0.2673 (2)	0.0494 (9)	
C6	0.2084 (3)	1.0860 (9)	0.1985 (2)	0.0485 (9)	
C7	0.1043 (3)	1.0870 (11)	0.1759 (2)	0.0573 (9)	
C8	0.0682 (3)	1.2544 (12)	0.1116 (2)	0.0745 (12)	
H8	-0.0017	1.2535	0.0971	0.089*	
C9	0.1348 (4)	1.4212 (13)	0.0693 (2)	0.0773 (13)	
H9	0.1099	1.5343	0.0260	0.093*	
C10	0.2363 (4)	1.4250 (12)	0.0892 (2)	0.0735 (12)	
H10	0.2812	1.5389	0.0595	0.088*	
C11	0.2732 (3)	1.2605 (10)	0.1533 (2)	0.0610 (11)	
H11	0.3433	1.2656	0.1670	0.073*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0547 (18)	0.0514 (18)	0.0545 (16)	0.0036 (18)	0.0125 (14)	-0.0018 (17)
O1	0.0574 (15)	0.0702 (16)	0.0701 (15)	0.0067 (17)	0.0167 (12)	0.0136 (17)
O2	0.0532 (16)	0.0868 (19)	0.099 (2)	-0.006 (2)	0.0049 (14)	0.018 (2)
C1	0.065 (2)	0.053 (2)	0.066 (2)	0.011 (3)	0.0173 (19)	0.009 (2)

C2	0.062 (2)	0.045 (2)	0.059 (2)	-0.001 (2)	0.0143 (17)	-0.002 (2)
C3	0.062 (3)	0.041 (3)	0.054 (3)	0.000	0.009 (2)	0.000
C4	0.055 (2)	0.107 (4)	0.089 (3)	-0.014 (3)	0.004 (2)	0.015 (4)
C5	0.0469 (19)	0.048 (2)	0.0541 (19)	-0.003 (2)	0.0155 (17)	-0.010 (2)
C6	0.053 (2)	0.0447 (19)	0.0478 (18)	-0.011 (2)	0.0105 (16)	-0.010 (2)
C7	0.056 (2)	0.055 (2)	0.062 (2)	-0.004 (3)	0.0132 (18)	-0.005 (3)
C8	0.071 (3)	0.078 (3)	0.074 (3)	-0.001 (3)	-0.004 (2)	0.005 (3)
C9	0.106 (4)	0.067 (3)	0.058 (2)	0.001 (3)	0.000 (2)	0.001 (3)
C10	0.098 (4)	0.066 (3)	0.058 (2)	-0.023 (3)	0.014 (2)	-0.004 (3)
C11	0.063 (2)	0.062 (3)	0.059 (2)	-0.014 (3)	0.013 (2)	-0.012 (3)

Geometric parameters (Å, °)

N1—C5	1.282 (4)	C4—H4A	0.9600
N1—O1	1.398 (4)	C4—H4B	0.9600
O1—C1	1.430 (5)	C4—H4C	0.9600
O2—C7	1.349 (5)	C5—C6	1.459 (5)
O2—H2	0.8200	C6—C7	1.389 (5)
C1—C2	1.504 (5)	C6—C11	1.390 (5)
C1—H1A	0.9700	C7—C8	1.375 (5)
C1—H1B	0.9700	C8—C9	1.360 (6)
C2—C3	1.501 (5)	C8—H8	0.9300
C2—H2A	0.9700	C9—C10	1.348 (6)
C2—H2B	0.9700	C9—H9	0.9300
C3—C2 ⁱ	1.501 (5)	C10—C11	1.370 (6)
C3—H3A	0.9700	C10—H10	0.9300
C3—H3B	0.9700	C11—H11	0.9300
C4—C5	1.495 (5)		
C5—N1—O1	114.0 (3)	C5—C4—H4C	109.5
N1—O1—C1	108.6 (3)	H4A—C4—H4C	109.5
C7—O2—H2	109.5	H4B—C4—H4C	109.5
O1—C1—C2	113.2 (4)	N1—C5—C6	117.0 (3)
O1—C1—H1A	108.9	N1—C5—C4	121.6 (4)
C2—C1—H1A	108.9	C6—C5—C4	121.3 (4)
O1—C1—H1B	108.9	C7—C6—C11	117.0 (4)
C2—C1—H1B	108.9	C7—C6—C5	122.5 (3)
H1A—C1—H1B	107.7	C11—C6—C5	120.4 (3)
C3—C2—C1	112.9 (3)	O2—C7—C8	116.4 (4)
C3—C2—H2A	109.0	O2—C7—C6	122.7 (4)
C1—C2—H2A	109.0	C8—C7—C6	120.8 (4)
C3—C2—H2B	109.0	C9—C8—C7	119.9 (4)
C1—C2—H2B	109.0	C9—C8—H8	120.1
H2A—C2—H2B	107.8	C7—C8—H8	120.1
C2—C3—C2 ⁱ	113.9 (5)	C10—C9—C8	121.0 (5)
C2—C3—H3A	108.8	C10—C9—H9	119.5
C2 ⁱ —C3—H3A	108.8	C8—C9—H9	119.5
C2—C3—H3B	108.8	C9—C10—C11	119.7 (4)

C2 ⁱ —C3—H3B	108.8	C9—C10—H10	120.2
H3A—C3—H3B	107.7	C11—C10—H10	120.2
C5—C4—H4A	109.5	C10—C11—C6	121.6 (4)
C5—C4—H4B	109.5	C10—C11—H11	119.2
H4A—C4—H4B	109.5	C6—C11—H11	119.2
C5—N1—O1—C1	179.0 (3)	C5—C6—C7—O2	-1.6 (6)
N1—O1—C1—C2	-72.2 (4)	C11—C6—C7—C8	0.0 (6)
O1—C1—C2—C3	-175.9 (3)	C5—C6—C7—C8	178.7 (4)
C1—C2—C3—C2 ⁱ	-176.9 (4)	O2—C7—C8—C9	-179.6 (4)
O1—N1—C5—C6	-179.5 (3)	C6—C7—C8—C9	0.1 (6)
O1—N1—C5—C4	0.5 (5)	C7—C8—C9—C10	0.2 (7)
N1—C5—C6—C7	2.9 (5)	C8—C9—C10—C11	-0.5 (8)
C4—C5—C6—C7	-177.1 (4)	C9—C10—C11—C6	0.6 (7)
N1—C5—C6—C11	-178.5 (3)	C7—C6—C11—C10	-0.3 (6)
C4—C5—C6—C11	1.5 (6)	C5—C6—C11—C10	-179.1 (4)
C11—C6—C7—O2	179.7 (4)		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2...N1	0.82	1.84	2.553 (4)	144
C4—H4A...O1	0.96	2.16	2.631 (5)	109