organic compounds

4,4′-Dichloro-2,2′-[(1E,1′E)-propane-1,3-diylbis(nitrilomethylidyne)]diphenol

Lei Shi, Zhu-Ping Xiao, Zhong Zhuang, Zhao-Zhao Zhong and Hai-Liang Zhu*

State Key Laboratory of Pharmaceutical Biotechnology, Nanjing University, Nanjing 210093, People's Republic of China

Correspondence e-mail: hailiang_zhu@163.com

The title compound, $C_{17}H_{16}Cl_2N_2O_2$, forms two intramolecular O—H⋯N hydrogen bonds. The dihedral angle between the two benzene rings is 68.44 (11)°.

Related literature

For related literature, see: Allen *et al.* (1987); Li *et al.* (2006); Shi *et al.* (2007).
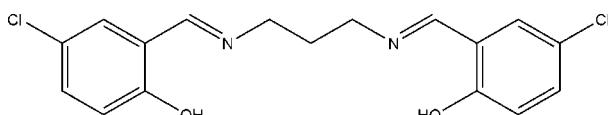
Experimental

Crystal data

$C_{17}H_{16}Cl_2N_2O_2$	$V = 1706.9$ (5) Å³
$M_r = 351.22$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 11.2479$ (18) Å	$\mu = 0.39$ mm^{-1}
$b = 8.9729$ (14) Å	$T = 293$ (2) K
$c = 17.007$ (2) Å	$0.30 \times 0.30 \times 0.20$ mm
$\beta = 96.06$ (3)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	3462 measured reflections
Absorption correction: ψ scan (North *et al.*, 1968)	3292 independent reflections
$T_{min} = 0.892$, $T_{max} = 0.926$	1461 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.047$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$	208 parameters
$wR(F^2) = 0.161$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
3292 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1A⋯N1	0.82	1.86	2.591 (5)	148
O2—H2A⋯N2	0.82	1.81	2.548 (4)	149

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2172).

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supplementary materials

4,4'-Dichloro-2,2'-[(1*E*,1'*E*)-propane-1,3-diylbis(nitrilomethylidyne)]diphenol

L. Shi, Z.-P. Xiao, Z. Zhuang, Z.-Z. Zhong and H.-L. Zhu

Comment

Schiff bases of salicylaldehyde and its derivatives play an important role in organic chemistry (Shi *et al.*, 2007). Recently, we have reported the structural characterization of one Schiff base compound derived from the condensation of 5-chloro-salicylaldehyde and primary amines (Li *et al.*, 2006). As an extension of this work, we report here the crystal structure of the title compound, (I). In (I), all bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). The dihedral angle between the two benzene rings is 68.44 (11)°. Intramolecular O—H⋯N hydrogen bonds are formed between atoms O1 and N1, O2 and N2.

Experimental

Propane-1,3-diamine (37 mg, 0.5 mmol) and 5-chlorosalicylaldehyde (156 mg, 1 mmol) were dissolved in methanol (10 ml) at 323 K. The mixture was stirred for 10 min to give a clear yellow solution. After keeping the solution in air for 7 d with slow evaporation of the solvent, yellow block crystals were formed at the bottom of the vessel, with 85% yield. The crystals were isolated, washed three times with methanol and dried in a vacuum desiccator containing anhydrous $CaCl_2$.

Refinement

All H atoms were positioned geometrically (O—H = 0.82 Å, C—H = 0.93 Å for the aromatic H atoms and C—H = 0.96 Å for the aliphatic H atoms) and were refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C,O)$.

Figures
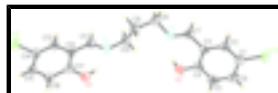
Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

4,4'-Dichloro-2,2'-[(1E,1'*E*)-propane-1,3-diylbis(nitrilomethylidyne)]diphenol

Crystal data

$C_{17}H_{16}Cl_2N_2O_2$	$F_{000} = 728$
$M_r = 351.22$	$D_x = 1.367$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.2479$ (18) Å	Cell parameters from 875 reflections
$b = 8.9729$ (14) Å	$\theta = 2.6–25.5°$
$c = 17.007$ (2) Å	$\mu = 0.39$ mm^{-1}
$\beta = 96.06$ (3)°	$T = 293$ (2) K

$V = 1706.9\ (5)\ \text{Å}^3$

$Z = 4$

Block, yellow

$0.30 \times 0.30 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	3292 independent reflections
Radiation source: fine-focus sealed tube	1461 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.047$
$T = 293(2)$ K	$\theta_{max} = 26.0°$
$\omega/2\theta$ scans	$\theta_{min} = 2.1°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 13$
$T_{min} = 0.892$, $T_{max} = 0.926$	$k = 0 \rightarrow 11$
3462 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.066$	H-atom parameters constrained
$wR(F^2) = 0.161$	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
3292 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.19$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	$U_{iso}*/U_{eq}$
C1	−0.1940 (4)	0.6503 (5)	0.0598 (2)	0.0630 (11)
C2	−0.1202 (3)	0.5367 (5)	0.0883 (2)	0.0568 (11)
H2C	−0.1535	0.4460	0.1008	0.068*

C3	0.0033 (3)	0.5538 (4)	0.0990 (2)	0.0486 (10)
C4	0.0526 (4)	0.6901 (5)	0.0777 (2)	0.0596 (11)
C5	−0.0239 (5)	0.8023 (5)	0.0499 (3)	0.0800 (14)
H5A	0.0083	0.8933	0.0368	0.096*
C6	−0.1452 (4)	0.7853 (6)	0.0409 (3)	0.0772 (14)
H6A	−0.1945	0.8638	0.0223	0.093*
C7	0.0794 (3)	0.4329 (5)	0.1287 (2)	0.0575 (11)
H7A	0.0437	0.3440	0.1417	0.069*
C8	0.2623 (3)	0.3116 (5)	0.1678 (3)	0.0747 (13)
H8A	0.2912	0.2592	0.1237	0.090*
H8B	0.2114	0.2440	0.1937	0.090*
C9	0.3670 (3)	0.3572 (5)	0.2256 (3)	0.0724 (13)
H9A	0.3378	0.4145	0.2680	0.087*
H9B	0.4054	0.2684	0.2487	0.087*
C10	0.4575 (3)	0.4479 (5)	0.1883 (2)	0.0719 (13)
H10A	0.4920	0.3882	0.1490	0.086*
H10B	0.4185	0.5333	0.1619	0.086*
C11	0.6566 (3)	0.4489 (5)	0.2474 (2)	0.0622 (11)
H11A	0.6722	0.3784	0.2097	0.075*
C12	0.7545 (3)	0.4979 (4)	0.3050 (2)	0.0525 (10)
C13	0.8677 (3)	0.4405 (5)	0.3036 (2)	0.0615 (11)
H13A	0.8822	0.3699	0.2657	0.074*
C14	0.9592 (3)	0.4867 (5)	0.3579 (2)	0.0598 (11)
C15	0.9377 (4)	0.5900 (5)	0.4151 (2)	0.0642 (12)
H15A	0.9999	0.6211	0.4519	0.077*
C16	0.8249 (4)	0.6468 (5)	0.4178 (2)	0.0665 (12)
H16A	0.8109	0.7161	0.4565	0.080*
C17	0.7320 (4)	0.6016 (4)	0.3635 (2)	0.0563 (11)
Cl1	−0.34717 (10)	0.62447 (17)	0.04676 (8)	0.0977 (5)
Cl2	1.10171 (10)	0.41310 (17)	0.35786 (8)	0.0959 (5)
N1	0.1924 (3)	0.4413 (4)	0.13828 (18)	0.0587 (9)
N2	0.5519 (3)	0.4989 (4)	0.24715 (19)	0.0655 (10)
O1	0.1711 (3)	0.7112 (4)	0.08454 (17)	0.0866 (10)
H1A	0.2049	0.6353	0.1019	0.104*
O2	0.6225 (2)	0.6574 (3)	0.36736 (17)	0.0801 (9)
H2A	0.5762	0.6218	0.3318	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.070 (3)	0.066 (3)	0.053 (3)	0.006 (3)	0.007 (2)	0.000 (2)
C2	0.061 (3)	0.054 (3)	0.056 (3)	−0.004 (2)	0.009 (2)	0.007 (2)
C3	0.060 (2)	0.046 (3)	0.040 (2)	−0.012 (2)	0.0050 (19)	−0.0043 (19)
C4	0.072 (3)	0.053 (3)	0.053 (3)	−0.015 (3)	0.001 (2)	0.002 (2)
C5	0.114 (4)	0.041 (3)	0.082 (3)	−0.008 (3)	0.000 (3)	0.014 (3)
C6	0.101 (4)	0.060 (3)	0.068 (3)	0.020 (3)	−0.005 (3)	−0.003 (3)
C7	0.059 (3)	0.049 (3)	0.065 (3)	−0.008 (2)	0.006 (2)	0.007 (2)
C8	0.062 (3)	0.068 (3)	0.091 (3)	0.003 (3)	−0.003 (2)	0.002 (3)

C9	0.060 (3)	0.078 (3)	0.077 (3)	0.002 (2)	−0.001 (2)	0.011 (3)
C10	0.053 (2)	0.090 (4)	0.070 (3)	0.003 (3)	−0.005 (2)	−0.002 (3)
C11	0.059 (3)	0.065 (3)	0.063 (3)	0.005 (2)	0.007 (2)	−0.009 (2)
C12	0.055 (2)	0.051 (3)	0.051 (2)	0.003 (2)	0.006 (2)	−0.002 (2)
C13	0.055 (2)	0.071 (3)	0.058 (3)	0.008 (2)	0.005 (2)	−0.006 (2)
C14	0.048 (2)	0.072 (3)	0.059 (3)	0.006 (2)	0.004 (2)	0.009 (2)
C15	0.065 (3)	0.058 (3)	0.066 (3)	−0.006 (2)	−0.010 (2)	0.006 (2)
C16	0.079 (3)	0.046 (3)	0.073 (3)	0.004 (2)	−0.001 (3)	−0.009 (2)
C17	0.064 (3)	0.041 (2)	0.064 (3)	0.008 (2)	0.008 (2)	0.001 (2)
Cl1	0.0711 (8)	0.1190 (12)	0.1029 (10)	0.0248 (8)	0.0086 (7)	0.0233 (9)
Cl2	0.0544 (7)	0.1236 (12)	0.1081 (10)	0.0187 (7)	0.0010 (6)	−0.0046 (9)
N1	0.056 (2)	0.056 (2)	0.063 (2)	−0.0067 (19)	0.0038 (17)	0.0032 (18)
N2	0.0503 (19)	0.079 (3)	0.065 (2)	0.008 (2)	−0.0026 (17)	−0.004 (2)
O1	0.079 (2)	0.075 (2)	0.104 (2)	−0.0294 (18)	−0.0005 (18)	0.0175 (19)
O2	0.0683 (19)	0.068 (2)	0.102 (2)	0.0257 (16)	−0.0010 (17)	−0.0266 (18)

Geometric parameters (Å, °)

C1—C2	1.371 (5)	C9—H9B	0.970
C1—C6	1.381 (6)	C10—N2	1.454 (5)
C1—Cl1	1.729 (4)	C10—H10A	0.970
C2—C3	1.390 (5)	C10—H10B	0.970
C2—H2C	0.930	C11—N2	1.260 (4)
C3—C4	1.406 (5)	C11—C12	1.463 (5)
C3—C7	1.440 (5)	C11—H11A	0.930
C4—O1	1.339 (4)	C12—C13	1.375 (5)
C4—C5	1.375 (6)	C12—C17	1.405 (5)
C5—C6	1.366 (6)	C13—C14	1.372 (5)
C5—H5A	0.930	C13—H13A	0.930
C6—H6A	0.930	C14—C15	1.384 (5)
C7—N1	1.267 (4)	C14—Cl2	1.734 (4)
C7—H7A	0.930	C15—C16	1.373 (5)
C8—N1	1.463 (5)	C15—H15A	0.930
C8—C9	1.509 (5)	C16—C17	1.381 (5)
C8—H8A	0.970	C16—H16A	0.930
C8—H8B	0.970	C17—O2	1.338 (4)
C9—C10	1.495 (5)	O1—H1A	0.820
C9—H9A	0.970	O2—H2A	0.820
C2—C1—C6	119.6 (4)	H9A—C9—H9B	107.8
C2—C1—Cl1	120.2 (4)	N2—C10—C9	111.1 (3)
C6—C1—Cl1	120.2 (4)	N2—C10—H10A	109.4
C1—C2—C3	121.6 (4)	C9—C10—H10A	109.4
C1—C2—H2C	119.2	N2—C10—H10B	109.4
C3—C2—H2C	119.2	C9—C10—H10B	109.4
C2—C3—C4	118.5 (4)	H10A—C10—H10B	108.0
C2—C3—C7	120.8 (4)	N2—C11—C12	122.2 (4)
C4—C3—C7	120.7 (4)	N2—C11—H11A	118.9
O1—C4—C5	120.5 (4)	C12—C11—H11A	118.9
O1—C4—C3	121.1 (4)	C13—C12—C17	119.8 (4)

C5—C4—C3	118.4 (4)	C13—C12—C11	120.6 (4)
C6—C5—C4	122.6 (4)	C17—C12—C11	119.6 (4)
C6—C5—H5A	118.7	C14—C13—C12	120.3 (4)
C4—C5—H5A	118.7	C14—C13—H13A	119.8
C5—C6—C1	119.2 (4)	C12—C13—H13A	119.8
C5—C6—H6A	120.4	C13—C14—C15	120.1 (4)
C1—C6—H6A	120.4	C13—C14—Cl2	120.8 (3)
N1—C7—C3	123.5 (4)	C15—C14—Cl2	119.0 (3)
N1—C7—H7A	118.3	C16—C15—C14	120.2 (4)
C3—C7—H7A	118.3	C16—C15—H15A	119.9
N1—C8—C9	111.2 (4)	C14—C15—H15A	119.9
N1—C8—H8A	109.4	C15—C16—C17	120.4 (4)
C9—C8—H8A	109.4	C15—C16—H16A	119.8
N1—C8—H8B	109.4	C17—C16—H16A	119.8
C9—C8—H8B	109.4	O2—C17—C16	119.6 (4)
H8A—C8—H8B	108.0	O2—C17—C12	121.3 (4)
C10—C9—C8	113.0 (4)	C16—C17—C12	119.2 (4)
C10—C9—H9A	109.0	C7—N1—C8	119.5 (4)
C8—C9—H9A	109.0	C11—N2—C10	120.3 (4)
C10—C9—H9B	109.0	C4—O1—H1A	109.5
C8—C9—H9B	109.0	C17—O2—H2A	109.5
C6—C1—C2—C3	0.1 (6)	N2—C11—C12—C17	1.5 (6)
Cl1—C1—C2—C3	179.6 (3)	C17—C12—C13—C14	−1.6 (6)
C1—C2—C3—C4	−1.8 (6)	C11—C12—C13—C14	−179.9 (4)
C1—C2—C3—C7	−179.8 (4)	C12—C13—C14—C15	0.9 (6)
C2—C3—C4—O1	−178.0 (3)	C12—C13—C14—Cl2	179.0 (3)
C7—C3—C4—O1	0.0 (6)	C13—C14—C15—C16	0.0 (6)
C2—C3—C4—C5	2.3 (6)	Cl2—C14—C15—C16	−178.1 (3)
C7—C3—C4—C5	−179.7 (4)	C14—C15—C16—C17	−0.1 (6)
O1—C4—C5—C6	179.1 (4)	C15—C16—C17—O2	179.6 (4)
C3—C4—C5—C6	−1.2 (7)	C15—C16—C17—C12	−0.6 (6)
C4—C5—C6—C1	−0.4 (7)	C13—C12—C17—O2	−178.7 (4)
C2—C1—C6—C5	1.0 (6)	C11—C12—C17—O2	−0.4 (6)
Cl1—C1—C6—C5	−178.5 (3)	C13—C12—C17—C16	1.4 (6)
C2—C3—C7—N1	178.7 (4)	C11—C12—C17—C16	179.8 (4)
C4—C3—C7—N1	0.7 (6)	C3—C7—N1—C8	−179.1 (3)
N1—C8—C9—C10	−65.5 (5)	C9—C8—N1—C7	−140.4 (4)
C8—C9—C10—N2	175.6 (4)	C12—C11—N2—C10	179.5 (4)
N2—C11—C12—C13	179.8 (4)	C9—C10—N2—C11	111.9 (5)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.82	1.86	2.591 (5)	148
O2—H2A···N2	0.82	1.81	2.548 (4)	149

Fig. 1