

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4,4',6,6'-Tetrachloro-2,2'-[(1*E*,1'*E*)-propane-1,3-diylbis(nitrilomethanylylidene)]diphenol

 Hadi Kargar,^a Reza Kia,^{b*‡} Amir Adabi Ardakani^a and Muhammad Nawaz Tahir^{c*}
^aDepartment of Chemistry, Payame Noor University, PO BOX 19395-3697 Tehran, I. R. of IRAN, ^bDepartment of Chemistry, Science and Research Branch, Islamic Azad University, Tehran, Iran, and ^cDepartment of Physics, University of Sargodha, Punjab, Pakistan

Correspondence e-mail: zsrkk@yahoo.com, dmntahir_uos@yahoo.com

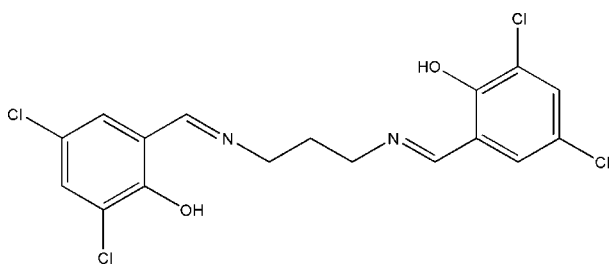
Received 16 June 2012; accepted 28 June 2012

 Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.027; wR factor = 0.068; data-to-parameter ratio = 17.0.

The title compound, $\text{C}_{17}\text{H}_{14}\text{Cl}_4\text{N}_2\text{O}_2$, is generated by crystallographic twofold symmetry. The two benzene rings are inclined to one another by $80.17(10)^\circ$. There are two intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds, which make $S(6)$ ring motifs. In the crystal, molecules are linked by $\text{C}-\text{H}\cdots\text{O}$ and weak $\text{C}-\text{H}\cdots\text{Cl}$ interactions, forming a three-dimensional network.

Related literature

For standard bond lengths, see: Allen *et al.*, (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related Schiff base ligands, see: Kargar *et al.* (2011); Kia *et al.* (2010).



Experimental

Crystal data

 $\text{C}_{17}\text{H}_{14}\text{Cl}_4\text{N}_2\text{O}_2$
 $M_r = 420.10$

 Orthorhombic, $Fdd2$
 $a = 24.9797(14)$ Å

 $b = 31.666(3)$ Å

 $c = 4.4495(2)$ Å

 $V = 3519.6(4)$ Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.69$ mm⁻¹
 $T = 291$ K

 $0.26 \times 0.23 \times 0.18$ mm

Data collection

 Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.842$, $T_{\max} = 0.886$

 7926 measured reflections
 1960 independent reflections
 1634 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.068$
 $S = 1.04$

1960 reflections

115 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.14$ e Å⁻³
 $\Delta\rho_{\min} = -0.16$ e Å⁻³

 Absolute structure: Flack (1983),
 842 Friedel pairs

Flack parameter: 0.08 (7)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.84	2.574 (2)	147
$\text{C5}-\text{H2}\cdots\text{O1}^i$	0.93	2.43	3.336 (2)	166
$\text{C8}-\text{H5B}\cdots\text{Cl1}^{ii}$	0.97	2.89	3.851 (2)	169

 Symmetry codes: (i) $x + \frac{1}{4}, -y + \frac{1}{4}, z + \frac{1}{4}$; (ii) $x + \frac{1}{4}, -y + \frac{1}{4}, z + \frac{3}{4}$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); 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 and PLATON (Spek, 2009).

HK and AAA thank PNU for financial support. MNT thanks GC University of Sargodha, Pakistan for the research facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2464).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Kargar, H., Kia, R., Pahlavani, E. & Tahir, M. N. (2011). *Acta Cryst.* **E67**, o614.
- Kia, R., Kargar, H., Tahir, M. N. & Kianoosh, F. (2010). *Acta Cryst.* **E66**, o2296.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

‡ Present address: Structural Dynamics of (Bio)Chemical Systems, Max Planck Institute for Biophysical Chemistry, Am Fassberg 11 37077 Göttingen, Germany.

supplementary materials

Acta Cryst. (2012). E68, o2323 [doi:10.1107/S1600536812029443]

4,4',6,6'-Tetrachloro-2,2'-[(1*E*,1'*E*)-propane-1,3-diylbis(nitrilomethanylylidene)]diphenol

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Comment

In continuation of our work on the crystal structure analyses of Schiff base ligands (Kargar *et al.*, (2011); Kia *et al.*, (2010), we synthesized the title compound and report herein on its crystal structure.

The title compound, Fig. 1, a potential tetradentate Schiff base ligand, possesses two-fold rotation symmetry, atom C9 is located on the 2-fold axis. The bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges. The two symmetry related benzene rings are inclined to one another by 80.17 (10) °. There are two intramolecular O—H···N hydrogen bonds which make *S(6)* ring motifs (Table 1; Bernstein *et al.*, 1995).

In the crystal, molecules are linked by C—H···O and weak C—H···Cl interactions to form a three-dimensional network (Table 1 and Fig. 2).

Experimental

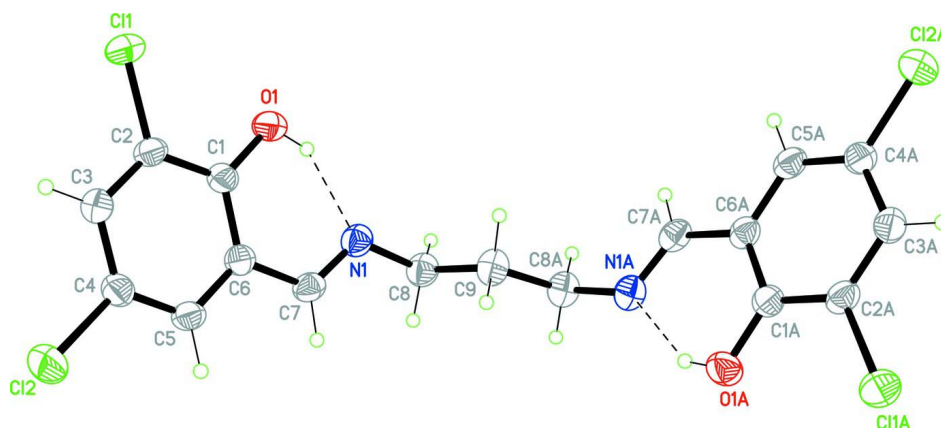
The title compound was synthesized by adding 3,5-dichlorosalicylaldehyde (2 mmol) to a solution of propylenediamine (1 mmol) in ethanol (30 ml). The mixture was refluxed with stirring for 30 min. The resultant solution was filtered. Light-yellow prismatic single crystals of the title compound, suitable for *X*-ray structure determination, were recrystallized from ethanol by slow evaporation of the solvents at room temperature over several days.

Refinement

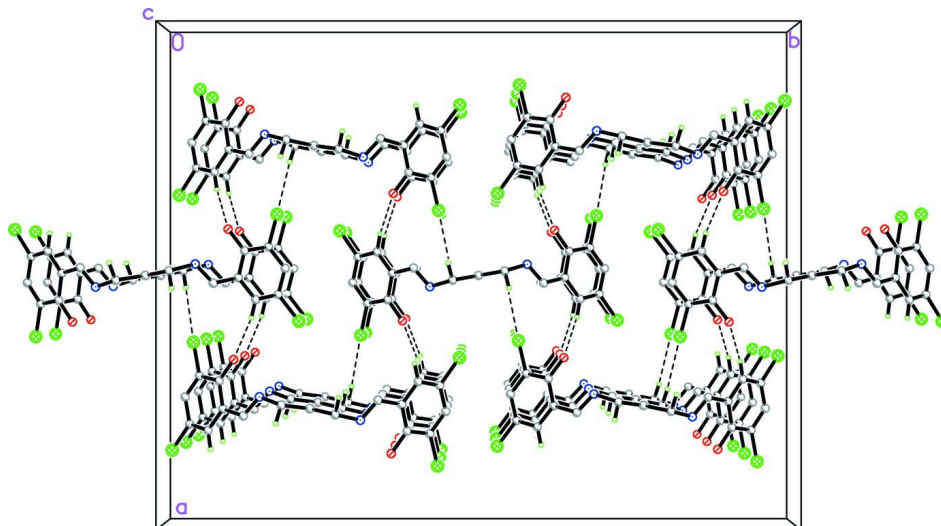
The OH and C-bound H atoms were included in calculated positions and treated as riding atoms: O-H = 0.82 Å, C-H = 0.93 and 0.96 Å, with $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{O}, \text{C})$ where $k = 1.5$ for OH and CH₃ H atoms and = 1.2 for other H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); 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* (Sheldrick, 2008) and *PLATON* (Spek, 2009).


Figure 1

The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering [symmetry code for suffix A = -x, -y, z].


Figure 2

The crystal packing diagram of the title compound viewed down the *c*-axis, showing linking of molecules through C—H...O and weak C—H...Cl interactions (dashed lines).

4,4',6,6'-Tetrachloro-2,2'-[(1*E*,1'*E*)-propane-1,3- diylbis(nitrilomethanylylidene)]diphenol

Crystal data

$C_{17}H_{14}Cl_4N_2O_2$
 $M_r = 420.10$
 Orthorhombic, *Fdd2*
 Hall symbol: *F 2 -2d*
 $a = 24.9797$ (14) Å
 $b = 31.666$ (3) Å
 $c = 4.4495$ (2) Å
 $V = 3519.6$ (4) Å³
 $Z = 8$

$F(000) = 1712$
 $D_x = 1.586$ Mg m⁻³
 Mo *K*α radiation, $\lambda = 0.71073$ Å
 Cell parameters from 1234 reflections
 $\theta = 2.5$ – 27.5°
 $\mu = 0.69$ mm⁻¹
 $T = 291$ K
 Prism, light-yellow
 0.26 × 0.23 × 0.18 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	7926 measured reflections
Radiation source: fine-focus sealed tube	1960 independent reflections
Graphite monochromator	1634 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.028$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$\theta_{\text{max}} = 27.3^\circ$, $\theta_{\text{min}} = 2.1^\circ$
$T_{\text{min}} = 0.842$, $T_{\text{max}} = 0.886$	$h = -32 \rightarrow 32$
	$k = -40 \rightarrow 40$
	$l = -5 \rightarrow 5$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.027$	$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 1.6825P]$
$wR(F^2) = 0.068$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1960 reflections	$\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{\AA}^{-3}$
115 parameters	$\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$
1 restraint	Absolute structure: Flack (1983), 842 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.08 (7)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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)
C1	-0.04810 (7)	0.14212 (6)	0.7828 (4)	0.0378 (4)	
C2	-0.05597 (7)	0.17577 (6)	0.5854 (5)	0.0405 (5)	
C3	-0.01420 (7)	0.19985 (6)	0.4810 (5)	0.0429 (5)	
H9	-0.0203	0.2218	0.3466	0.052*	
C4	0.03720 (8)	0.19078 (6)	0.5799 (5)	0.0419 (5)	
C5	0.04673 (7)	0.15902 (6)	0.7793 (5)	0.0409 (5)	
H2	0.0814	0.1538	0.8453	0.049*	
C6	0.00437 (7)	0.13440 (6)	0.8845 (4)	0.0374 (4)	
C7	0.01466 (8)	0.10036 (6)	1.0958 (4)	0.0401 (5)	
H4	0.0489	0.0972	1.1753	0.048*	
C8	-0.00878 (9)	0.03959 (6)	1.3730 (4)	0.0458 (5)	
H5A	-0.0378	0.0351	1.5143	0.055*	
H5B	0.0234	0.0461	1.4863	0.055*	
C9	0.0000	0.0000	1.1876 (7)	0.0465 (7)	
H6A	-0.0309	-0.0043	1.0589	0.056*	0.50

H6B	0.0309	0.0043	1.0589	0.056*	0.50
Cl1	-0.12035 (2)	0.187207 (19)	0.46609 (15)	0.05937 (17)	
Cl2	0.09006 (2)	0.220654 (18)	0.43888 (16)	0.06188 (18)	
N1	-0.02186 (7)	0.07479 (5)	1.1743 (4)	0.0424 (4)	
O1	-0.08951 (5)	0.11861 (4)	0.8692 (4)	0.0503 (4)	
H1	-0.0790	0.0999	0.9826	0.075*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0349 (9)	0.0355 (9)	0.0429 (12)	-0.0002 (8)	-0.0034 (8)	-0.0052 (9)
C2	0.0322 (9)	0.0401 (10)	0.0490 (12)	0.0056 (8)	-0.0083 (8)	-0.0044 (9)
C3	0.0415 (10)	0.0356 (9)	0.0518 (13)	0.0015 (8)	-0.0029 (10)	0.0001 (10)
C4	0.0349 (10)	0.0359 (10)	0.0550 (13)	-0.0016 (8)	0.0015 (9)	-0.0067 (9)
C5	0.0314 (9)	0.0402 (10)	0.0512 (13)	0.0052 (8)	-0.0060 (8)	-0.0087 (9)
C6	0.0368 (9)	0.0344 (9)	0.0409 (11)	0.0028 (7)	-0.0035 (8)	-0.0085 (9)
C7	0.0374 (10)	0.0420 (11)	0.0410 (11)	0.0067 (8)	-0.0083 (8)	-0.0084 (9)
C8	0.0521 (12)	0.0446 (11)	0.0407 (12)	0.0032 (9)	-0.0053 (9)	-0.0001 (10)
C9	0.0567 (17)	0.0430 (15)	0.0399 (15)	0.0048 (13)	0.000	0.000
Cl1	0.0371 (3)	0.0610 (3)	0.0800 (4)	0.0024 (2)	-0.0162 (3)	0.0145 (3)
Cl2	0.0434 (3)	0.0532 (3)	0.0891 (5)	-0.0076 (2)	0.0080 (3)	0.0055 (3)
N1	0.0464 (9)	0.0411 (9)	0.0397 (9)	0.0039 (7)	-0.0062 (8)	-0.0015 (8)
O1	0.0366 (7)	0.0503 (9)	0.0639 (10)	-0.0058 (6)	-0.0093 (6)	0.0124 (7)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.331 (2)	C6—C7	1.453 (3)
C1—C2	1.395 (3)	C7—N1	1.269 (2)
C1—C6	1.408 (2)	C7—H4	0.9300
C2—C3	1.373 (3)	C8—N1	1.460 (3)
C2—Cl1	1.7318 (19)	C8—C9	1.517 (3)
C3—C4	1.387 (3)	C8—H5A	0.9700
C3—H9	0.9300	C8—H5B	0.9700
C4—C5	1.362 (3)	C9—C8 ⁱ	1.517 (3)
C4—Cl2	1.741 (2)	C9—H6A	0.9700
C5—C6	1.395 (3)	C9—H6B	0.9700
C5—H2	0.9300	O1—H1	0.8200
O1—C1—C2	119.98 (16)	N1—C7—C6	121.61 (17)
O1—C1—C6	122.23 (17)	N1—C7—H4	119.2
C2—C1—C6	117.79 (17)	C6—C7—H4	119.2
C3—C2—C1	122.02 (17)	N1—C8—C9	109.52 (18)
C3—C2—Cl1	119.06 (15)	N1—C8—H5A	109.8
C1—C2—Cl1	118.92 (15)	C9—C8—H5A	109.8
C2—C3—C4	118.75 (19)	N1—C8—H5B	109.8
C2—C3—H9	120.6	C9—C8—H5B	109.8
C4—C3—H9	120.6	H5A—C8—H5B	108.2
C5—C4—C3	121.42 (18)	C8 ⁱ —C9—C8	114.1 (3)
C5—C4—Cl2	120.22 (15)	C8 ⁱ —C9—H6A	108.7
C3—C4—Cl2	118.35 (16)	C8—C9—H6A	108.7

C4—C5—C6	119.90 (17)	C8 ⁱ —C9—H6B	108.7
C4—C5—H2	120.1	C8—C9—H6B	108.7
C6—C5—H2	120.1	H6A—C9—H6B	107.6
C5—C6—C1	120.07 (18)	C7—N1—C8	119.53 (17)
C5—C6—C7	119.82 (17)	C1—O1—H1	109.5
C1—C6—C7	120.10 (18)		
O1—C1—C2—C3	-177.41 (19)	C4—C5—C6—C7	179.60 (17)
C6—C1—C2—C3	2.7 (3)	O1—C1—C6—C5	177.77 (18)
O1—C1—C2—Cl1	2.0 (2)	C2—C1—C6—C5	-2.4 (3)
C6—C1—C2—Cl1	-177.84 (15)	O1—C1—C6—C7	-1.3 (3)
C1—C2—C3—C4	-1.2 (3)	C2—C1—C6—C7	178.59 (16)
Cl1—C2—C3—C4	179.35 (16)	C5—C6—C7—N1	-173.18 (19)
C2—C3—C4—C5	-0.7 (3)	C1—C6—C7—N1	5.9 (3)
C2—C3—C4—Cl2	178.41 (15)	N1—C8—C9—C8 ⁱ	-174.57 (19)
C3—C4—C5—C6	1.0 (3)	C6—C7—N1—C8	176.32 (17)
Cl2—C4—C5—C6	-178.09 (15)	C9—C8—N1—C7	-97.64 (19)
C4—C5—C6—C1	0.5 (3)		

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

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>
O1—H1...N1	0.82	1.84	2.574 (2)	147
C5—H2...O1 ⁱⁱ	0.93	2.43	3.336 (2)	166
C8—H5B...Cl1 ⁱⁱⁱ	0.97	2.89	3.851 (2)	169

Symmetry codes: (ii) $x+1/4, -y+1/4, z+1/4$; (iii) $x+1/4, -y+1/4, z+5/4$.