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

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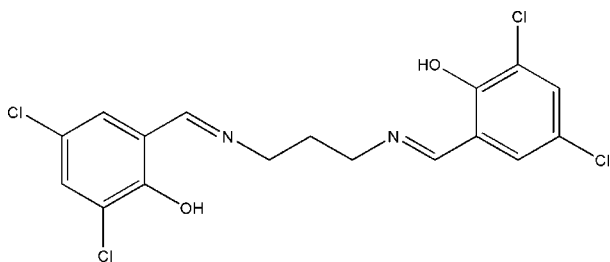
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 Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.027; wR factor = 0.068; data-to-parameter ratio = 17.0.

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).

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

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supporting information

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

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S1. 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).

S2. 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.

S3. 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.

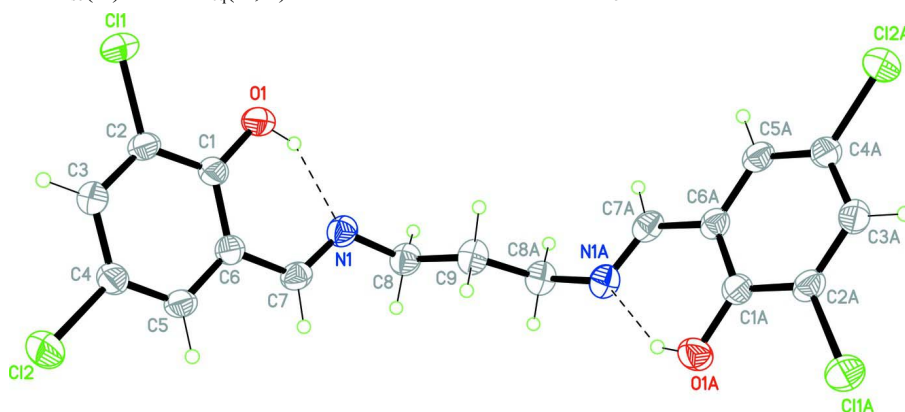


Figure 1

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

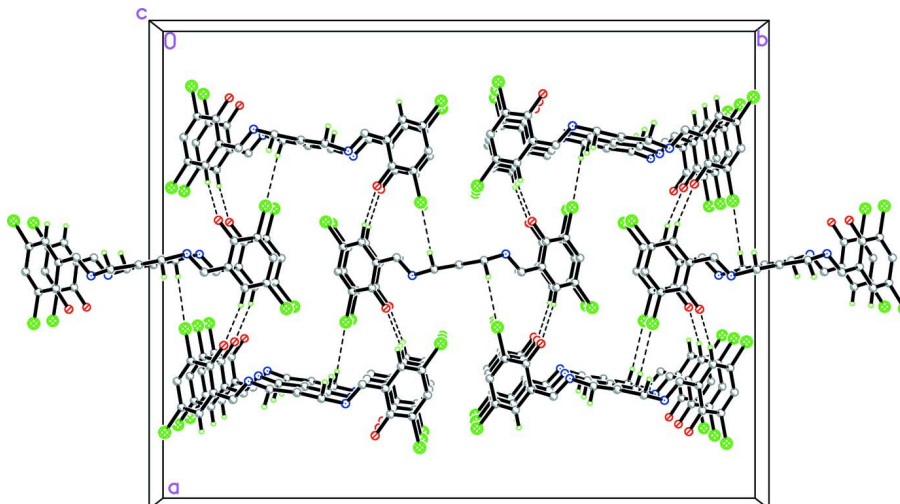


Figure 2

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 -2*d*

$a = 24.9797$ (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

$0.26 \times 0.23 \times 0.18$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.842$, $T_{\max} = 0.886$

7926 measured reflections

1960 independent reflections

1634 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 27.3^\circ$, $\theta_{\min} = 2.1^\circ$

$h = -32 \rightarrow 32$

$k = -40 \rightarrow 40$

$l = -5 \rightarrow 5$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.068$

$S = 1.03$

1960 reflections

115 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 1.6825P]$

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

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.14 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{Å}^{-3}$

Absolute structure: Flack (1983), 842 Friedel
 pairs
 Absolute structure parameter: 0.08 (7)

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 (Å^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | 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 |
| C11 | -0.12035 (2) | 0.187207 (19) | 0.46609 (15) | 0.05937 (17) | |
| C12 | 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 (Å^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 |
| C11 | 0.0371 (3) | 0.0610 (3) | 0.0800 (4) | 0.0024 (2) | -0.0162 (3) | 0.0145 (3) |
| C12 | 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 (Å, °)

| | | | |
|--------------|--------------|--------------------------|--------------|
| 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—C11 | 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—C12 | 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—C11 | 119.06 (15) | N1—C8—H5A | 109.8 |
| C1—C2—C11 | 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—C12 | 120.22 (15) | C8 ⁱ —C9—H6A | 108.7 |
| C3—C4—C12 | 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—C11 | 2.0 (2) | C2—C1—C6—C5 | -2.4 (3) |
| C6—C1—C2—C11 | -177.84 (15) | O1—C1—C6—C7 | -1.3 (3) |
| C1—C2—C3—C4 | -1.2 (3) | C2—C1—C6—C7 | 178.59 (16) |
| C11—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—C12 | 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) |
| C12—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—H...A</i> | <i>D—H</i> | <i>H...A</i> | <i>D...A</i> | <i>D—H...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...C11 ⁱⁱⁱ | 0.97 | 2.89 | 3.851 (2) | 169 |

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