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(*E*,*E*)-4,4′,6,6′-Tetrachloro-2,2′-(1,1′-azinodiethylene)diphenol

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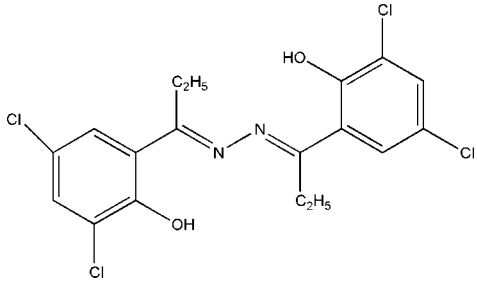
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.026; wR factor = 0.075; data-to-parameter ratio = 13.6.

The title compound, $C_{18}H_{16}Cl_4N_2O_2$, was synthesized by the reaction of 1-(3,5-dichloro-2-hydroxyphenyl)propan-1-one with hydrazine hydrate. The molecule has a centre of symmetry at the mid-point of the N—N bond. The crystal structure is stabilized by intramolecular O—H···N and intermolecular C—H···Cl hydrogen bonds.

Related literature

For further details of the chemistry of the title compound, see: Kundu *et al.* (2005); Kesslen *et al.* (1999); Zheng *et al.* (2005). For literature on similar structures, see: Glaser *et al.* (1995); Hunig *et al.* (2000).



Experimental

Crystal data

$C_{18}H_{16}Cl_4N_2O_2$
M_r = 434.13
Triclinic, $P\overline{1}$
a = 7.8251 (5) Å
b = 8.0294 (4) Å
c = 8.3899 (5) Å
α = 65.894 (2)°
β = 80.961 (4)°
γ = 74.821 (2)°
V = 463.63 (5) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 273 (2) K
0.33 × 0.22 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.813, T_{max} = 0.932
5333 measured reflections
1635 independent reflections
1494 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.075
S = 1.06
1635 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.82	2.538 (1)	146
C6—H6···Cl2^{i}	0.93	2.80	3.6678 (15)	156

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2144).

References

Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Glaser, R., Chen, G. S., Anthamatten, M. & Barnes, C. L. (1995). *J. Chem. Soc. Perkin Trans. 2*, pp. 1449–1458.
Hunig, S., Kemmer, M. & Wenner, H. (2000). *Chem. Eur. J.* **6**, 2618–2632.
Kesslen, E. C., Euler, W. B. & Foxman, B. M. (1999). *Chem. Mater.* **11**, 336–340.
Kundu, N., Chatterjee, P. B., Chaudhury, M. & Tiekink, E. R. T. (2005). *Acta Cryst.* E**61**, m1583–m1585.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Zheng, P.-W., Wang, W. & Duan, X.-M. (2005). *Acta Cryst.* E**61**, o3020–o3021.

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supplementary materials

Acta Cryst. (2007). E**63**, o3382 [**doi:10.1107/S1600536807031844**]

(*E*,*E*)-4,4',6,6'-Tetrachloro-2,2'-(1,1'-azinodiethylene)diphenol

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Comment

Recently, a number of azine compounds containing both a diimine linkage and N—N bonding have been investigated in terms of their crystallography and coordination chemistry (Kundu *et al.*, 2005; Kesslen *et al.*, 1999; Zheng *et al.*, 2005;). As an extension of work on the structural characterization of azine derivatives,the title compound, (I),was synthesized and its crystal structure is reported here.

In the title compound, there is a crystallographic centre of symmetry at the midpoint of the N—N bond (Fig. 1.). The molecule displays an (E, E) conformation with respect to the C7=N1 and its symmetry related *c*7*a*=N1a double bond (Fig. 1.). This configuration agrees with those commonly found in similar compounds (Glaser *et al.*, 1995; Hunig *et al.*, 2000)·The crystal structure is stabilized by intramolecular O—H···N and intermolecular C—H···Cl hydrogen bonds·(Table 1. and Fig. 2).

Experimental

An ethanol solution (50 ml) of hydrazine (0.02 mol) and 1-(3,5-dichloro-2-hydroxyphenyl)propan-1-one (0.04 mol) was refluxed and stirred for 5 h; the mixture was cooled and the resulting solid product, (I), was collected by filtration·Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in 1,2-dichloroethane.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms,with *C*—*H*(methyl) = 0.96 Å, *C*—*H*(methylene) = 0.97 Å,*C*—H(aromatic) = 0.93 Å, O—H = 0.82 Å and with U_{iso}(H) =1.5U_{eq}(C_{methyl},*O*) and 1.2U_{eq}($C_{aromatic}$,$C_{methylene}$).

Figures

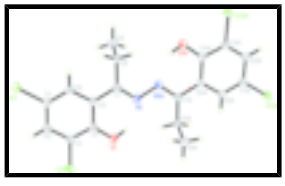


Fig. 1. The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

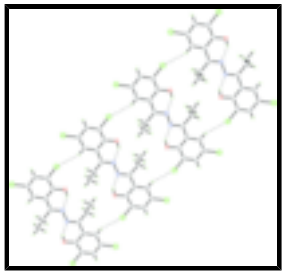


Fig. 2. Packing diagram of (I), showing intramolecular O—H···N and intermolecular C—H···Cl hydrogen bonds.(dashed lines).

(*E*,*E*)-4,4',6,6'-Tetrachloro-2,2'-(1,1'-azinodiethylene)diphenol

Crystal data

$C_{18}H_{16}Cl_4N_2O_2$	$Z = 1$
$M_r = 434.13$	$F_{000} = 222$
Triclinic, $P\overline{1}$	$D_x = 1.555$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.8251$ (5) Å	Cell parameters from 3274 reflections
$b = 8.0294$ (4) Å	$\theta = 0.00$–0.00°
$c = 8.3899$ (5) Å	$\mu = 0.65$ mm^{-1}
$\alpha = 65.894$ (2)°	$T = 273$ (2) K
$\beta = 80.961$ (4)°	Plate, yellow
$\gamma = 74.821$ (2)°	0.33 × 0.22 × 0.11 mm
$V = 463.63$ (5) Å^3	

Data collection

Bruker APEXII CCD area-detector diffractometer	1635 independent reflections
Radiation source: fine-focus sealed tube	1494 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.018$
$T = 273$(2) K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 2.7^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -8 \rightarrow 9$
$T_{min} = 0.813$, $T_{max} = 0.932$	$k = -9 \rightarrow 9$
5333 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.026$	H-atom parameters constrained
$wR(F^2) = 0.075$	$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.1214P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$

1635 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
120 parameters	$\Delta\rho_{min} = -0.21$ 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^ > 2sigma(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}
Cl1	0.18719 (6)	−0.14153 (6)	0.90825 (6)	0.05554 (16)
Cl2	0.43014 (6)	0.46317 (6)	0.80112 (7)	0.05983 (17)
O1	0.73361 (16)	0.29828 (16)	0.62779 (17)	0.0504 (3)
H1	0.8185	0.2477	0.5804	0.076*
N1	0.92133 (17)	0.03912 (18)	0.53574 (17)	0.0390 (3)
C1	0.3518 (2)	−0.0141 (2)	0.82541 (19)	0.0372 (3)
C2	0.3274 (2)	0.1548 (2)	0.84262 (19)	0.0396 (4)
H2	0.2247	0.1997	0.8986	0.048*
C3	0.4578 (2)	0.2547 (2)	0.7753 (2)	0.0392 (4)
C4	0.6141 (2)	0.1919 (2)	0.68970 (19)	0.0370 (3)
C5	0.63793 (19)	0.0177 (2)	0.67474 (18)	0.0336 (3)
C6	0.5032 (2)	−0.0823 (2)	0.74394 (19)	0.0365 (3)
H6	0.5167	−0.1966	0.7345	0.044*
C7	0.80203 (19)	−0.0610 (2)	0.59174 (18)	0.0345 (3)
C8	0.8265 (2)	−0.2498 (2)	0.5844 (2)	0.0400 (4)
H8A	0.9018	−0.2539	0.4819	0.048*
H8B	0.7123	−0.2690	0.5733	0.048*
C9	0.9097 (3)	−0.4059 (2)	0.7471 (3)	0.0615 (5)
H9A	1.0241	−0.3890	0.7568	0.092*
H9B	0.9227	−0.5244	0.7387	0.092*
H9C	0.8348	−0.4029	0.8487	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0414 (2)	0.0580 (3)	0.0741 (3)	−0.02249 (19)	0.0186 (2)	−0.0330 (2)
Cl2	0.0591 (3)	0.0465 (3)	0.0872 (4)	−0.0123 (2)	0.0113 (2)	−0.0441 (2)
O1	0.0486 (7)	0.0448 (6)	0.0683 (8)	−0.0217 (5)	0.0167 (6)	−0.0323 (6)
N1	0.0353 (7)	0.0386 (7)	0.0457 (7)	−0.0114 (5)	0.0096 (5)	−0.0209 (6)

C1	0.0338 (8)	0.0399 (8)	0.0384 (8)	−0.0107 (6)	0.0030 (6)	−0.0159 (6)
C2	0.0358 (8)	0.0425 (8)	0.0414 (8)	−0.0042 (6)	0.0023 (6)	−0.0214 (7)
C3	0.0423 (8)	0.0353 (8)	0.0446 (8)	−0.0062 (6)	−0.0005 (7)	−0.0220 (7)
C4	0.0397 (8)	0.0350 (7)	0.0390 (8)	−0.0109 (6)	0.0010 (6)	−0.0166 (6)
C5	0.0340 (8)	0.0347 (7)	0.0330 (7)	−0.0083 (6)	0.0026 (6)	−0.0150 (6)
C6	0.0387 (8)	0.0337 (7)	0.0406 (8)	−0.0109 (6)	0.0021 (6)	−0.0175 (6)
C7	0.0363 (8)	0.0352 (7)	0.0339 (7)	−0.0102 (6)	0.0017 (6)	−0.0152 (6)
C8	0.0352 (8)	0.0424 (8)	0.0505 (9)	−0.0118 (6)	0.0080 (6)	−0.0276 (7)
C9	0.0623 (12)	0.0390 (9)	0.0791 (13)	−0.0084 (8)	−0.0086 (10)	−0.0184 (9)

Geometric parameters (Å, °)

Cl1—C1	1.7372 (15)	C4—C5	1.416 (2)
Cl2—C3	1.7281 (15)	C5—C6	1.401 (2)
O1—C4	1.3331 (18)	C5—C7	1.479 (2)
O1—H1	0.8200	C6—H6	0.9300
N1—C7	1.2980 (19)	C7—C8	1.503 (2)
N1—N1^{i}	1.393 (2)	C8—C9	1.521 (2)
C1—C6	1.372 (2)	C8—H8A	0.9700
C1—C2	1.383 (2)	C8—H8B	0.9700
C2—C3	1.372 (2)	C9—H9A	0.9600
C2—H2	0.9300	C9—H9B	0.9600
C3—C4	1.402 (2)	C9—H9C	0.9600
C4—O1—H1	109.5	C1—C6—H6	119.5
C7—N1—N1^{i}	115.44 (15)	C5—C6—H6	119.5
C6—C1—C2	121.22 (13)	N1—C7—C5	115.61 (13)
C6—C1—Cl1	119.55 (12)	N1—C7—C8	124.08 (13)
C2—C1—Cl1	119.22 (12)	C5—C7—C8	120.24 (12)
C3—C2—C1	118.61 (14)	C7—C8—C9	111.55 (13)
C3—C2—H2	120.7	C7—C8—H8A	109.3
C1—C2—H2	120.7	C9—C8—H8A	109.3
C2—C3—C4	122.34 (13)	C7—C8—H8B	109.3
C2—C3—Cl2	118.75 (12)	C9—C8—H8B	109.3
C4—C3—Cl2	118.89 (12)	H8A—C8—H8B	108.0
O1—C4—C3	118.32 (13)	C8—C9—H9A	109.5
O1—C4—C5	123.32 (14)	C8—C9—H9B	109.5
C3—C4—C5	118.35 (13)	H9A—C9—H9B	109.5
C6—C5—C4	118.54 (13)	C8—C9—H9C	109.5
C6—C5—C7	119.96 (13)	H9A—C9—H9C	109.5
C4—C5—C7	121.48 (13)	H9B—C9—H9C	109.5
C1—C6—C5	120.93 (14)		
C6—C1—C2—C3	0.4 (2)	C2—C1—C6—C5	−0.3 (2)
Cl1—C1—C2—C3	−179.48 (11)	Cl1—C1—C6—C5	179.58 (11)
C1—C2—C3—C4	0.2 (2)	C4—C5—C6—C1	−0.4 (2)
C1—C2—C3—Cl2	−178.49 (12)	C7—C5—C6—C1	178.35 (13)
C2—C3—C4—O1	179.77 (14)	N1^{i}—N1—C7—C5	177.43 (14)
Cl2—C3—C4—O1	−1.6 (2)	N1^{i}—N1—C7—C8	0.6 (3)
C2—C3—C4—C5	−0.8 (2)	C6—C5—C7—N1	−178.17 (13)

Cl2—C3—C4—C5	177.85 (11)	C4—C5—C7—N1	0.5 (2)
O1—C4—C5—C6	−179.73 (13)	C6—C5—C7—C8	−1.2 (2)
C3—C4—C5—C6	0.9 (2)	C4—C5—C7—C8	177.50 (13)
O1—C4—C5—C7	1.6 (2)	N1—C7—C8—C9	88.61 (19)
C3—C4—C5—C7	−177.81 (13)	C5—C7—C8—C9	−88.11 (18)

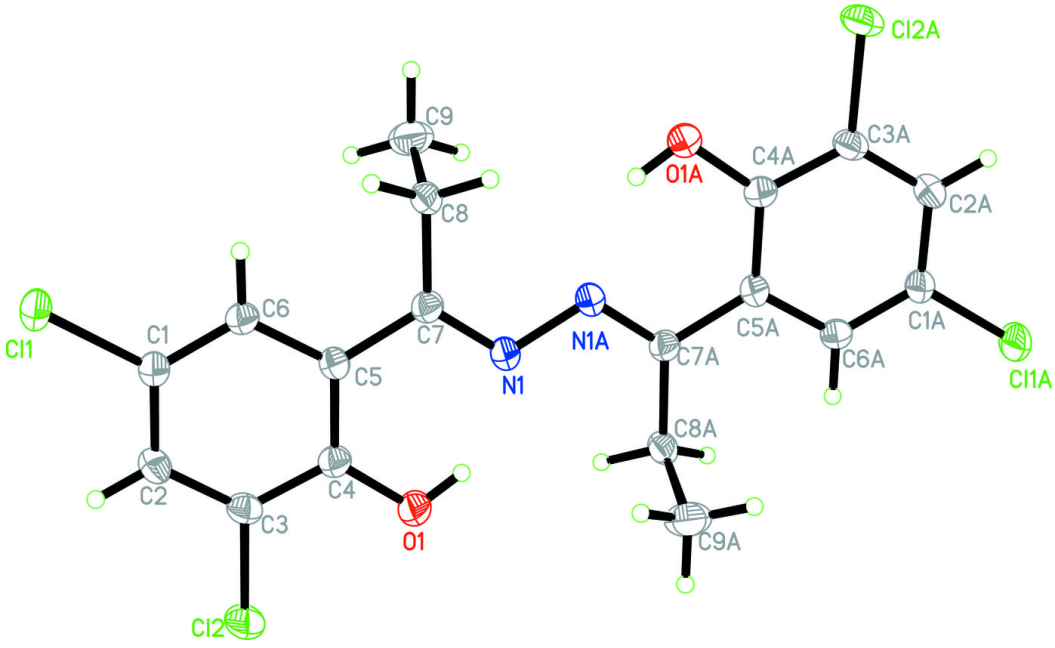
Symmetry codes: (i) −*x*+2, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.82	2.538 (1)	146
C6—H6···Cl2^{ii}	0.93	2.80	3.6678 (15)	156

Symmetry codes: (ii) *x*, *y*−1, *z*.

Fig. 1


Cl2A
C9
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C5
N1
C7A
C6A
Cl1A
C8A
C2
C4
C3
C9A
O1
Cl2

Fig. 2