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(*E*,*E*)-4,4′-Dichloro-2,2′-(1,1′-azinodimethylene)diphenol

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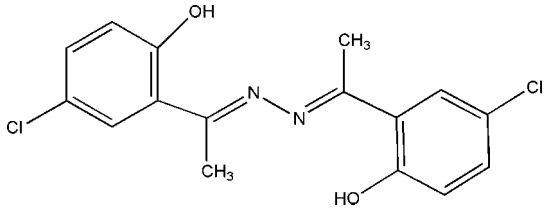
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.081; data-to-parameter ratio = 12.9.

The title compound, $C_{16}H_{14}Cl_2N_2O_2$, was synthesized by the reaction of 1-(5-chloro-2-hydroxyphenyl)ethanone with hydrazine hydrate. The molecule sits on a crystallographic center of symmetry at the mid-point of the central N—N bond and both N atoms are acceptors in an intramolecular hydrogen bond. In the crystal structure, the planar molecules are arranged in a sheet-like motif with one C—H···O bond linking molecules within the sheets and a second C—H···O interaction between neighboring sheets. There is also a close Cl···Cl intermolecular approach of 3.54 (1) Å within the sheets.

Related literature

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



Experimental

Crystal data

$C_{16}H_{14}Cl_2N_2O_2$
M_r = 337.19
Triclinic, $P\overline{1}$
a = 3.9105 (2) Å
b = 6.2985 (4) Å
c = 15.1479 (9) Å
α = 89.520 (2)°
β = 88.792 (1)°
γ = 86.164 (2)°
V = 372.17 (4) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 273 (2) K
0.25 × 0.18 × 0.12 mm

Data collection

Bruker APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.897, T_{max} = 0.949
4295 measured reflections
1315 independent reflections
1010 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.081
S = 1.00
1315 reflections
102 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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	1.83	2.551 (2)	146
C8—H8B···O1^{i}	0.96	2.60	3.368 (2)	137
C8—H8C···O1^{ii}	0.96	2.63	3.455 (3)	145

Symmetry codes: (i) $x-1, y+1, z$; (ii) $x, y+1, z$.

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: FL2158).

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supplementary materials

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(*E*,*E*)-4,4'-Dichloro-2,2'-(1,1'-azinodimethylene)diphenol

J.-G. Chang, G.-F. He and Y.-F. Li

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;). 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 center of symmetry at the midpoint of the N—N bond (Fig. 1.). The molecule displays an (E, E) conformation with respect to the symmetry related C=N double bonds. This configuration agrees with those commonly found in similar compounds (Glaser *et al.*, 1995; Hunig *et al.*, 2000). In the crystal the planar molecules are arranged in a sheet like motif with one C—H···O bond linking molecules within the sheets and a second C—H···O interaction between neighboring sheets. There is also a close Cl—Cl intermolecular approach of 3.54 (1) Å within the sheets. (Table 1 and Fig. 2).

Experimental

An ethanol solution (50 ml) of hydrazine (0.02 mol) and 1-(5-chloro-2-hydroxyphenyl)ethanone(0.04 mol) was refluxed and stirred for 3 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 acetone.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms,with *C*—*H*(methyl) = 0.96 Å, C—H(aromatic) = 0.93 Å,*O*—H = 0.82 Å and with U_{iso}(H) = 1.5U_{eq}(C_{methyl},*O*) anddn3057 1.2U_{eq}($C_{aromatic}$).

Figures

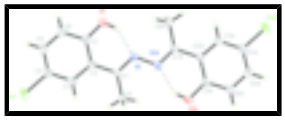

Fig. 1. The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level·Dashed lines show intramolecular hydrogen bonds.

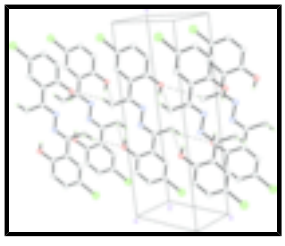

Fig. 2. Packing diagram of (I), showing intermolecular C—H···O hydrogen bonds.(dashed lines).

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

Crystal data

$C_{16}H_{14}Cl_2N_2O_2$	$Z = 1$
$M_r = 337.19$	$F_{000} = 174$
Triclinic, $P\overline{1}$	$D_x = 1.504$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 3.9105$ (2) Å	Cell parameters from 1047 reflections
$b = 6.2985$ (4) Å	$\theta = 2.7$–23.0º
$c = 15.1479$ (9) Å	$\mu = 0.44$ mm^{-1}
$\alpha = 89.520$ (2)º	$T = 273$ (2) K
$\beta = 88.792$ (1)º	Plate, colorless
$\gamma = 86.164$ (2)º	0.25 × 0.18 × 0.12 mm
$V = 372.17$ (4) Å^3	

Data collection

Bruker APEX II CCD area-detector diffractometer	1315 independent reflections
Radiation source: fine-focus sealed tube	1010 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 273$(2) K	$\theta_{max} = 25.0$º
phi and ω scans	$\theta_{min} = 1.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -4 \rightarrow 4$
$T_{min} = 0.897$, $T_{max} = 0.949$	$k = -7 \rightarrow 7$
4295 measured reflections	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.081$	$w = 1/[\sigma^2(F_o^2) + (0.0223P)^2 + 0.229P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.003$
1315 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
102 parameters	$\Delta\rho_{min} = -0.18$ 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}
Cl1	0.07776 (19)	0.29780 (11)	0.07597 (4)	0.0668 (3)
O1	0.6578 (4)	−0.3076 (2)	0.33622 (10)	0.0531 (4)
H1	0.6553	−0.2565	0.3859	0.080*
N1	0.5071 (4)	−0.0325 (3)	0.45587 (10)	0.0392 (4)
C1	0.5138 (5)	−0.1637 (3)	0.27931 (13)	0.0408 (5)
C2	0.3675 (5)	0.0368 (3)	0.30718 (13)	0.0357 (5)
C3	0.2340 (5)	0.1759 (3)	0.24204 (13)	0.0409 (5)
H3	0.1387	0.3095	0.2581	0.049*
C4	0.2415 (6)	0.1185 (4)	0.15515 (14)	0.0458 (6)
C5	0.3773 (6)	−0.0796 (4)	0.12886 (14)	0.0525 (6)
H5	0.3771	−0.1182	0.0697	0.063*
C6	0.5124 (6)	−0.2187 (4)	0.19101 (14)	0.0509 (6)
H6	0.6046	−0.3521	0.1736	0.061*
C7	0.3551 (5)	0.0988 (3)	0.40064 (12)	0.0347 (5)
C8	0.1725 (6)	0.3052 (3)	0.42815 (14)	0.0450 (6)
H8A	0.0690	0.2888	0.4856	0.068*
H8B	−0.0020	0.3455	0.3864	0.068*
H8C	0.3335	0.4136	0.4300	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0850 (5)	0.0718 (5)	0.0411 (3)	0.0148 (4)	−0.0136 (3)	0.0121 (3)
O1	0.0727 (12)	0.0382 (9)	0.0462 (9)	0.0140 (8)	−0.0056 (8)	0.0026 (7)
N1	0.0479 (11)	0.0350 (10)	0.0339 (8)	0.0045 (8)	−0.0053 (8)	0.0026 (7)
C1	0.0436 (13)	0.0364 (12)	0.0418 (12)	0.0005 (10)	−0.0021 (10)	0.0051 (9)
C2	0.0367 (12)	0.0343 (11)	0.0360 (11)	−0.0007 (9)	−0.0035 (9)	0.0029 (9)
C3	0.0423 (13)	0.0381 (12)	0.0415 (12)	0.0042 (10)	−0.0050 (9)	0.0025 (9)
C4	0.0487 (14)	0.0502 (14)	0.0380 (12)	0.0026 (11)	−0.0061 (10)	0.0068 (10)
C5	0.0630 (16)	0.0585 (16)	0.0356 (12)	0.0004 (12)	−0.0030 (11)	−0.0049 (11)
C6	0.0621 (16)	0.0440 (13)	0.0453 (13)	0.0068 (12)	−0.0010 (11)	−0.0057 (10)
C7	0.0344 (12)	0.0312 (11)	0.0383 (11)	−0.0005 (9)	−0.0034 (9)	0.0038 (9)

C8	0.0510 (14)	0.0393 (12)	0.0432 (12)	0.0099 (10)	−0.0053 (10)	0.0017 (10)

Geometric parameters (Å, °)

Cl1—C4	1.743 (2)	C3—H3	0.9300
O1—C1	1.350 (2)	C4—C5	1.382 (3)
O1—H1	0.8200	C5—C6	1.370 (3)
N1—C7	1.297 (2)	C5—H5	0.9300
N1—N1^{i}	1.400 (3)	C6—H6	0.9300
C1—C6	1.385 (3)	C7—C8	1.499 (3)
C1—C2	1.415 (3)	C8—H8A	0.9600
C2—C3	1.400 (3)	C8—H8B	0.9600
C2—C7	1.471 (3)	C8—H8C	0.9600
C3—C4	1.367 (3)		
C1—O1—H1	109.5	C6—C5—H5	120.4
C7—N1—N1^{i}	115.28 (19)	C4—C5—H5	120.4
O1—C1—C6	117.51 (19)	C5—C6—C1	121.1 (2)
O1—C1—C2	122.30 (18)	C5—C6—H6	119.5
C6—C1—C2	120.19 (19)	C1—C6—H6	119.5
C3—C2—C1	117.37 (18)	N1—C7—C2	116.84 (17)
C3—C2—C7	120.79 (18)	N1—C7—C8	123.10 (18)
C1—C2—C7	121.85 (17)	C2—C7—C8	120.06 (17)
C4—C3—C2	121.11 (19)	C7—C8—H8A	109.5
C4—C3—H3	119.4	C7—C8—H8B	109.5
C2—C3—H3	119.4	H8A—C8—H8B	109.5
C3—C4—C5	121.08 (19)	C7—C8—H8C	109.5
C3—C4—Cl1	119.64 (17)	H8A—C8—H8C	109.5
C5—C4—Cl1	119.27 (17)	H8B—C8—H8C	109.5
C6—C5—C4	119.2 (2)		
O1—C1—C2—C3	178.2 (2)	C4—C5—C6—C1	0.2 (4)
C6—C1—C2—C3	−1.9 (3)	O1—C1—C6—C5	−178.6 (2)
O1—C1—C2—C7	−1.7 (3)	C2—C1—C6—C5	1.4 (4)
C6—C1—C2—C7	178.2 (2)	N1^{i}—N1—C7—C2	179.4 (2)
C1—C2—C3—C4	0.8 (3)	N1^{i}—N1—C7—C8	−0.7 (3)
C7—C2—C3—C4	−179.3 (2)	C3—C2—C7—N1	−174.95 (19)
C2—C3—C4—C5	0.8 (4)	C1—C2—C7—N1	4.9 (3)
C2—C3—C4—Cl1	−179.15 (17)	C3—C2—C7—C8	5.1 (3)
C3—C4—C5—C6	−1.3 (4)	C1—C2—C7—C8	−175.0 (2)
Cl1—C4—C5—C6	178.66 (19)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.83	2.551 (2)	146
C8—H8B···O1^{ii}	0.96	2.60	3.368 (2)	137
C8—H8C···O1^{iii}	0.96	2.63	3.455 (3)	145

Symmetry codes: (ii) *x*−1, *y*+1, *z*; (iii) *x*, *y*+1, *z*.

Fig. 1

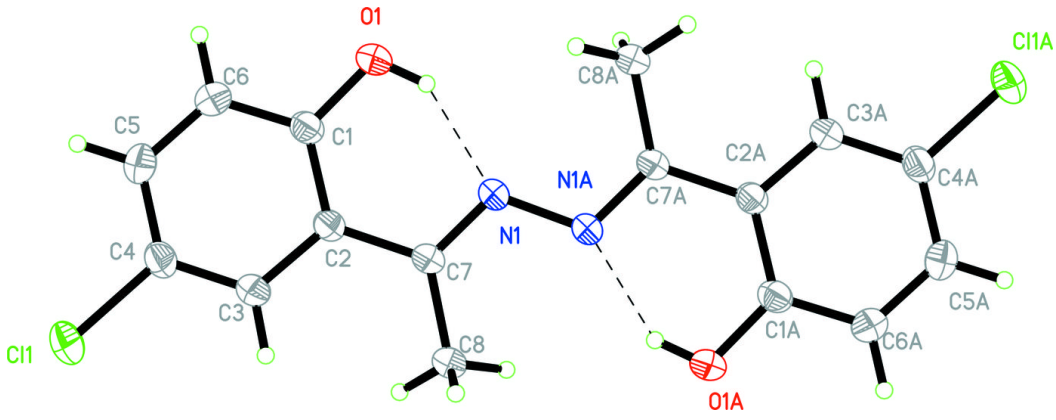
O1
C6
C5
C1
C4
C2
C3
Cl1
C7
C8
N1
N1A
C8A
C7A
C2A
C3A
Cl1A
C4A
C5A
C1A
C6A
O1A

Fig. 2