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

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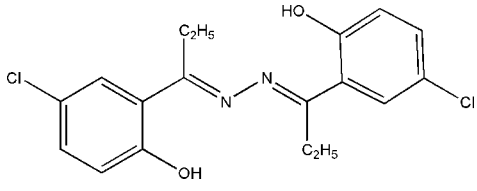
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.089; data-to-parameter ratio = 13.6.

The title compound, $C_{18}H_{18}Cl_2N_2O_2$, was synthesized by the reaction of 1-(5-chloro-2-hydroxyphenyl)propan-1-one with hydrazine hydrate. The molecule possesses a crystallographically imposed centre of symmetry at the midpoint of the N—N bond. Intramolecular O—H···N hydrogen bonds, together with C—H···π interactions, help to stabilize the structure.

Related literature

For related literature, see: Glaser *et al.* (1995); Hunig *et al.* (2000); Kesslen *et al.* (1999); Kundu *et al.* (2005); Zheng *et al.* (2005).



Experimental

Crystal data

$C_{18}H_{18}Cl_2N_2O_2$
M_r = 365.24
Monoclinic, $P2_1/c$
a = 9.3706 (3) Å
b = 13.9089 (4) Å
c = 6.7640 (2) Å
β = 103.021 (1)°
V = 858.92 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 273 (2) K
0.33 × 0.23 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.882, T_{max} = 0.958
9653 measured reflections
1507 independent reflections
1352 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.089
S = 1.08
1507 reflections
111 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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	2.5513 (16)	146
C2—H2···$Cg1^{i}$	0.93	2.68	3.496 (2)	147

Symmetry code: (i) $x, -y-\frac{1}{2}, z-\frac{1}{2}$.

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: *ORTEP-3 for Windows* (Farrugia, 1997) and *XP* in *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: DN2201).

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supplementary materials

Acta Cryst. (2007). E**63**, o3297 [**doi:10.1107/S1600536807030140**]

(*E*,*E*)-4,4'-Dichloro-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 the (E, E) conformation with respect to the C7=N1 and its symmetry related $C7^{i}$=$N1^{i}$ 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 hydrogen bond and weak C—H···\p interactions. (Table 1. and Fig. 2).

Experimental

An ethanol solution (50 ml) of hydrazine (0.02 mol) and 1-(5-chloro-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

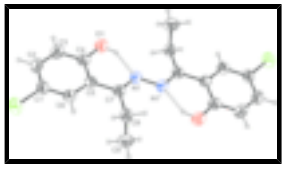

Fig. 1. The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines show intramolecular hydrogen bonds. [Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*].

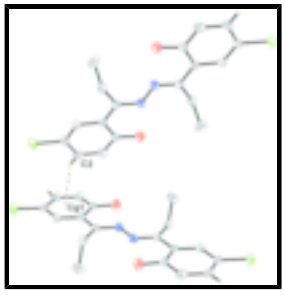

Fig. 2. View showing the C—H···π interactio. H bond is shown as dashed line.

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

Crystal data

$C_{18}H_{18}Cl_2N_2O_2$	F_{000} = 380
M_r = 365.24	D_x = 1.412 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4397 reflections
a = 9.3706 (3) Å	θ = 2.2–28.1º
b = 13.9089 (4) Å	μ = 0.39 mm^{-1}
c = 6.7640 (2) Å	T = 273 (2) K
β = 103.021 (1)º	Plate, yellow
V = 858.92 (4) Å^3	0.33 × 0.23 × 0.11 mm
Z = 2	

Data collection

Bruker APEXII CCD area-detector diffractometer	1507 independent reflections
Radiation source: fine-focus sealed tube	1352 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 273(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −10→11
T_{min} = 0.882, T_{max} = 0.958	k = −16→16
9653 measured reflections	l = −8→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.032	H-atom parameters constrained
$wR(F^2)$ = 0.089	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.2176P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max} < 0.001$

1507 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
111 parameters	$\Delta\rho_{min} = -0.29$ 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	−0.00768 (5)	0.36471 (3)	1.03985 (8)	0.0643 (2)
O1	0.58588 (12)	0.37295 (8)	0.90005 (17)	0.0499 (3)
H1	0.5916	0.4059	0.8016	0.075*
N1	0.48939 (13)	0.47487 (9)	0.58463 (18)	0.0384 (3)
C1	0.16828 (19)	0.36915 (10)	0.9962 (2)	0.0433 (4)
C2	0.28142 (19)	0.32278 (11)	1.1277 (2)	0.0473 (4)
H2	0.2641	0.2898	1.2396	0.057*
C3	0.41983 (19)	0.32587 (11)	1.0916 (2)	0.0470 (4)
H3	0.4961	0.2947	1.1801	0.056*
C4	0.44800 (17)	0.37479 (10)	0.9253 (2)	0.0385 (3)
C5	0.33229 (15)	0.42224 (9)	0.7891 (2)	0.0351 (3)
C6	0.19278 (16)	0.41832 (10)	0.8310 (2)	0.0402 (3)
H6	0.1152	0.4496	0.7453	0.048*
C7	0.35581 (15)	0.47347 (9)	0.6084 (2)	0.0354 (3)
C8	0.23007 (16)	0.52225 (11)	0.4663 (2)	0.0419 (4)
H8A	0.1402	0.4879	0.4673	0.050*
H8B	0.2457	0.5198	0.3294	0.050*
C9	0.2138 (2)	0.62645 (13)	0.5253 (3)	0.0613 (5)
H9A	0.2073	0.6295	0.6648	0.092*
H9B	0.1265	0.6530	0.4409	0.092*
H9C	0.2973	0.6626	0.5076	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0603 (3)	0.0673 (3)	0.0767 (4)	−0.0043 (2)	0.0394 (3)	0.0087 (2)
O1	0.0403 (6)	0.0577 (7)	0.0509 (7)	0.0051 (5)	0.0086 (5)	0.0132 (5)
N1	0.0386 (7)	0.0399 (6)	0.0387 (6)	0.0025 (5)	0.0127 (5)	0.0059 (5)

C1	0.0519 (9)	0.0366 (8)	0.0471 (9)	−0.0037 (6)	0.0229 (7)	−0.0017 (6)
C2	0.0658 (11)	0.0397 (8)	0.0399 (8)	−0.0032 (7)	0.0193 (7)	0.0032 (6)
C3	0.0567 (10)	0.0432 (9)	0.0388 (8)	0.0014 (7)	0.0059 (7)	0.0052 (6)
C4	0.0407 (8)	0.0346 (7)	0.0398 (8)	−0.0004 (6)	0.0083 (6)	−0.0022 (6)
C5	0.0394 (8)	0.0301 (7)	0.0369 (7)	−0.0012 (6)	0.0110 (6)	−0.0012 (5)
C6	0.0414 (8)	0.0371 (7)	0.0440 (8)	0.0018 (6)	0.0133 (6)	0.0019 (6)
C7	0.0368 (8)	0.0331 (7)	0.0367 (7)	−0.0003 (5)	0.0094 (6)	−0.0018 (5)
C8	0.0354 (8)	0.0501 (8)	0.0406 (8)	0.0004 (6)	0.0092 (6)	0.0068 (6)
C9	0.0636 (12)	0.0553 (11)	0.0637 (12)	0.0208 (8)	0.0116 (9)	0.0072 (8)

Geometric parameters (Å, °)

Cl1—C1	1.7404 (17)	C4—C5	1.417 (2)
O1—C4	1.3410 (19)	C5—C6	1.400 (2)
O1—H1	0.8200	C5—C7	1.4738 (19)
N1—C7	1.2976 (18)	C6—H6	0.9300
N1—N1^{i}	1.393 (2)	C7—C8	1.504 (2)
C1—C6	1.373 (2)	C8—C9	1.520 (2)
C1—C2	1.381 (2)	C8—H8A	0.9700
C2—C3	1.374 (2)	C8—H8B	0.9700
C2—H2	0.9300	C9—H9A	0.9600
C3—C4	1.390 (2)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C4—O1—H1	109.5	C1—C6—H6	119.4
C7—N1—N1^{i}	115.44 (14)	C5—C6—H6	119.4
C6—C1—C2	120.81 (15)	N1—C7—C5	116.02 (12)
C6—C1—Cl1	119.62 (13)	N1—C7—C8	123.55 (13)
C2—C1—Cl1	119.57 (12)	C5—C7—C8	120.41 (12)
C3—C2—C1	119.32 (14)	C7—C8—C9	111.89 (13)
C3—C2—H2	120.3	C7—C8—H8A	109.2
C1—C2—H2	120.3	C9—C8—H8A	109.2
C2—C3—C4	121.22 (15)	C7—C8—H8B	109.2
C2—C3—H3	119.4	C9—C8—H8B	109.2
C4—C3—H3	119.4	H8A—C8—H8B	107.9
O1—C4—C3	117.23 (13)	C8—C9—H9A	109.5
O1—C4—C5	122.97 (13)	C8—C9—H9B	109.5
C3—C4—C5	119.78 (14)	H9A—C9—H9B	109.5
C6—C5—C4	117.58 (13)	C8—C9—H9C	109.5
C6—C5—C7	120.52 (13)	H9A—C9—H9C	109.5
C4—C5—C7	121.90 (13)	H9B—C9—H9C	109.5
C1—C6—C5	121.28 (14)		

Symmetry codes: (i) −*x*+1, −*y*+1, −*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	2.5513 (16)	146
C2—H2···Cg1^{ii}	0.93	2.68	3.496 (2)	147

Symmetry codes: (ii) *x*, −*y*−1/2, *z*−1/2.

Fig. 1

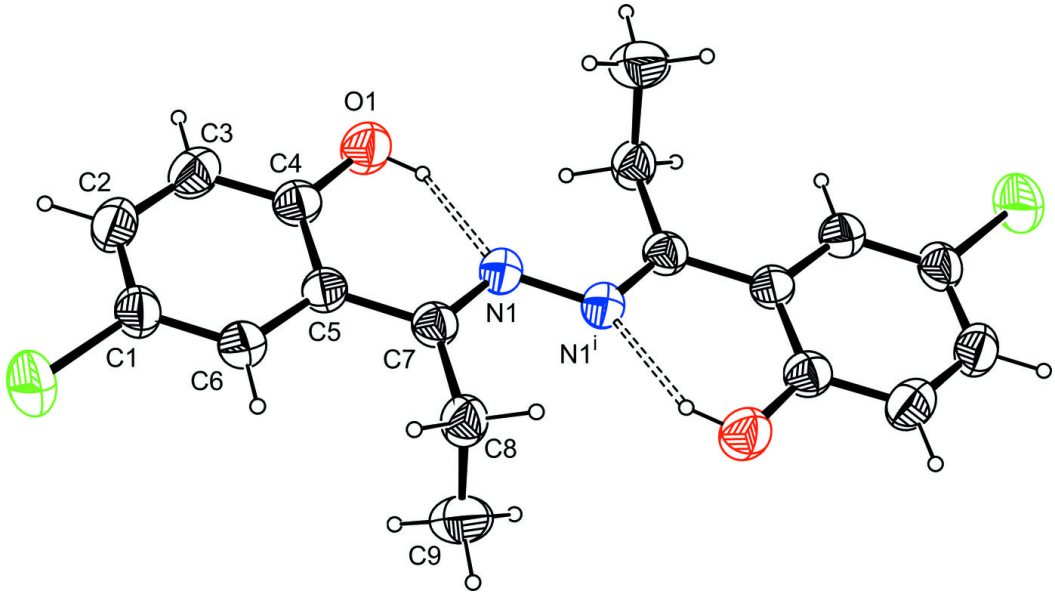

Fig. 2