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(*E*,*E*)-4,4′-Dichloro-2,2′-[azinobis(phenylmethylidyne)]diphenol

Jian-Guo Chang

Department of Materials Science and Chemical Engineering, Taishan University, 271021 Taian,Shandong, People's Republic of China
Correspondence e-mail: tsucjg@163.com

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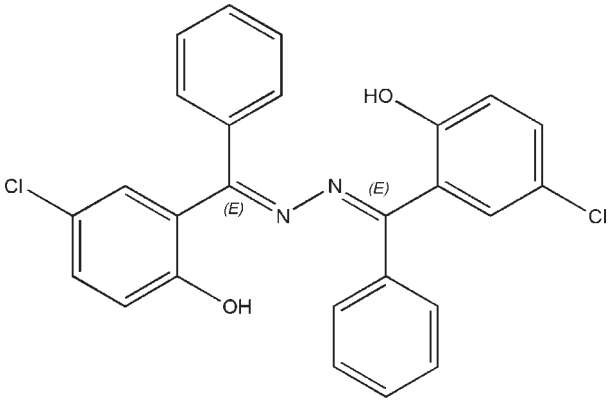
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.163; data-to-parameter ratio = 13.7.

The title compound, $C_{26}H_{18}Cl_2N_2O_2$, was synthesized by the reaction of (5-chloro-2-hydroxyphenyl)(phenyl)methanone with hydrazine hydrate. The molecule possesses a crystallographically imposed centre of symmetry at the mid-point of the N—N bond. The conformation of the molecule is stabilized by an intramolecular O—H···N hydrogen bond.

Related literature

For further details of the chemistry of the title compound, see: Glaser *et al.* (1995); Hunig *et al.* (2000). For similar structures, see: Kundu *et al.* (2005); Chang *et al.* (2007); Kesslen *et al.* (1999).



Experimental

Crystal data

$C_{26}H_{18}Cl_2N_2O_2$
M_r = 461.32
Orthorhombic, *Pbcn*
a = 13.1622 (11) Å
b = 10.6184 (9) Å
c = 16.0671 (13) Å
V = 2245.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 295 K
0.21 × 0.19 × 0.15 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.925, T_{max} = 0.960
11062 measured reflections
1995 independent reflections
1448 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.163
S = 1.00
1995 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 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	1.85	2.572 (3)	145

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2195).

References

Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chang, J.-G., He, G.-F. & Li, Y.-F. (2007). *Acta Cryst.* E**63**, o3982.
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. (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2767 [https://doi.org/10.1107/S1600536809041427]

(*E*,*E*)-4,4′-Dichloro-2,2′-[azinobis(phenylmethylidyne)]diphenol

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S1. 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; Chang *et al.*, 2007;). As an extension of work on the structural characterization of azine derivatives, the title compound 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 benzene rings, C1—C6(A), C8—C13(B) make dihedral angles of 85.26 (10)°. The conformation of the molecule is stabilized by intramolecular O—H···N hydrogen bonds. (Table 1. and Fig. 1).

S2. Experimental

An ethanol solution (50 ml) of hydrazine (0.02 mol) and (5-chloro-2-hydroxyphenyl)(phenyl)methanone (0.04 mol) was refluxed and stirred for 6 h; the mixture was cooled and the resulting solid product, was collected by filtration. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in acetone.

S3. Refinement

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

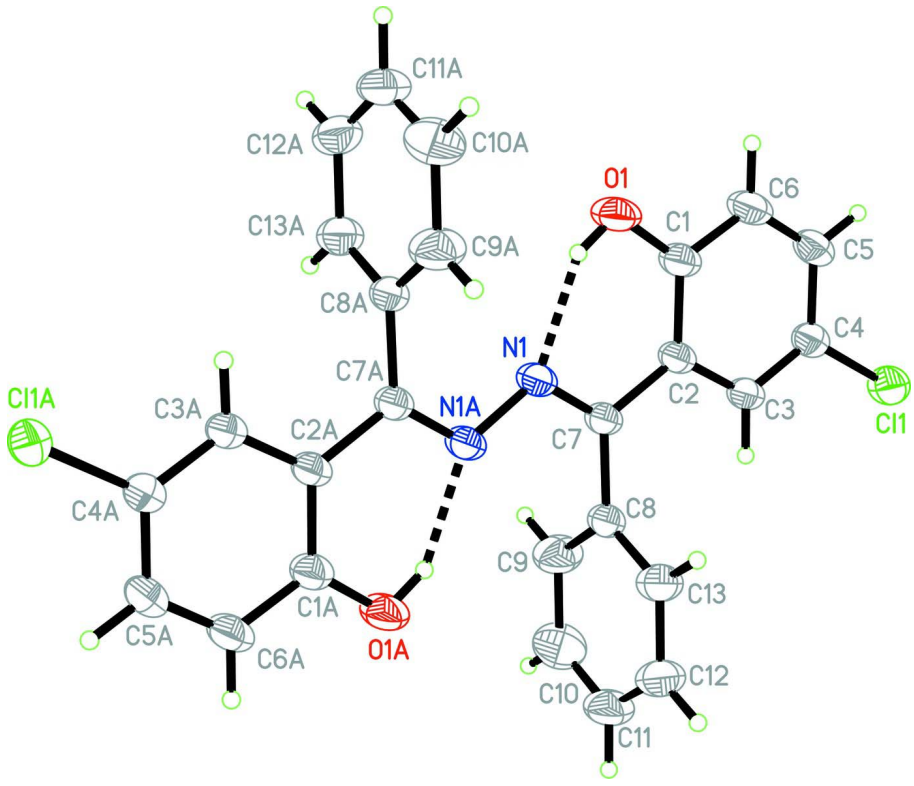

Figure 1

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

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Crystal data

$C_{26}H_{18}Cl_2N_2O_2$
M_r = 461.32
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
a = 13.1622 (11) Å
b = 10.6184 (9) Å
c = 16.0671 (13) Å
V = 2245.6 (3) Å^3
Z = 4
F(000) = 952
D_x = 1.365 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2411 reflections
θ = 2.5–24.9°
μ = 0.32 mm^{-1}
T = 295 K
Plate, yellow
0.21 × 0.19 × 0.15 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.925, T_{max} = 0.960
11062 measured reflections
1995 independent reflections
1448 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 25.1°, θ_{min} = 2.5°
$h = -7 \rightarrow 15$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.163$
$S = 1.00$
1995 reflections
146 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.113P)^2 + 0.120P]$ where $P = (F_o$^2^ + $2F_c$^2^)/3
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.23969 (7)	0.00971 (8)	0.37894 (5)	0.0751 (4)
O1	0.0498 (2)	−0.25287 (17)	0.10094 (15)	0.0776 (7)
H1	0.0336	−0.2049	0.0632	0.116*
N1	0.01697 (18)	−0.03788 (18)	0.03234 (13)	0.0534 (6)
C1	0.0915 (2)	−0.1871 (2)	0.16297 (18)	0.0584 (8)
C2	0.09948 (19)	−0.0543 (2)	0.16200 (16)	0.0487 (7)
C3	0.1464 (2)	0.0039 (2)	0.22956 (15)	0.0502 (7)
H3	0.1534	0.0911	0.2300	0.060*
C4	0.1822 (2)	−0.0646 (2)	0.29475 (16)	0.0555 (7)
C5	0.1737 (2)	−0.1941 (3)	0.29588 (19)	0.0671 (8)
H5	0.1983	−0.2402	0.3408	0.081*
C6	0.1291 (3)	−0.2536 (3)	0.2306 (2)	0.0709 (9)
H6	0.1235	−0.3409	0.2312	0.085*
C7	0.06111 (19)	0.0215 (2)	0.09230 (15)	0.0468 (6)
C8	0.0749 (2)	0.1609 (2)	0.09250 (16)	0.0503 (7)
C9	−0.0003 (3)	0.2398 (3)	0.1189 (2)	0.0801 (10)
H9	−0.0609	0.2068	0.1391	0.096*
C10	0.0135 (3)	0.3694 (3)	0.1158 (3)	0.1007 (13)
H10	−0.0377	0.4230	0.1342	0.121*
C11	0.1022 (3)	0.4180 (3)	0.0857 (2)	0.0905 (11)
H11	0.1108	0.5048	0.0827	0.109*
C12	0.1777 (3)	0.3402 (3)	0.0602 (2)	0.0782 (10)
H12	0.2384	0.3737	0.0405	0.094*
C13	0.1644 (2)	0.2118 (3)	0.06340 (19)	0.0660 (8)
H13	0.2164	0.1587	0.0457	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0985 (7)	0.0654 (6)	0.0615 (6)	−0.0123 (4)	−0.0284 (4)	0.0114 (3)
O1	0.124 (2)	0.0360 (11)	0.0729 (14)	−0.0034 (11)	−0.0286 (14)	0.0010 (9)
N1	0.0740 (15)	0.0362 (11)	0.0500 (12)	0.0003 (10)	−0.0115 (10)	0.0046 (9)
C1	0.0757 (19)	0.0383 (14)	0.0612 (17)	0.0019 (12)	−0.0088 (14)	0.0026 (12)
C2	0.0576 (15)	0.0352 (12)	0.0533 (14)	0.0035 (11)	−0.0037 (11)	0.0052 (11)
C3	0.0596 (15)	0.0379 (12)	0.0531 (15)	−0.0002 (11)	−0.0045 (11)	0.0052 (11)
C4	0.0639 (16)	0.0462 (14)	0.0565 (15)	0.0000 (12)	−0.0074 (13)	0.0065 (12)
C5	0.084 (2)	0.0506 (16)	0.0673 (18)	0.0046 (15)	−0.0175 (15)	0.0158 (14)
C6	0.099 (2)	0.0402 (14)	0.0733 (19)	0.0036 (14)	−0.0188 (17)	0.0107 (13)
C7	0.0564 (15)	0.0362 (13)	0.0477 (13)	0.0009 (10)	−0.0035 (11)	0.0037 (10)
C8	0.0636 (16)	0.0387 (13)	0.0486 (13)	−0.0017 (12)	−0.0098 (12)	0.0031 (11)
C9	0.080 (2)	0.0472 (17)	0.113 (3)	0.0045 (15)	0.0075 (18)	−0.0080 (16)
C10	0.112 (3)	0.057 (2)	0.133 (3)	0.022 (2)	−0.006 (2)	−0.015 (2)
C11	0.128 (3)	0.0418 (18)	0.102 (3)	−0.0125 (19)	−0.032 (2)	0.0059 (17)
C12	0.104 (2)	0.0579 (19)	0.0724 (19)	−0.0258 (18)	−0.0146 (18)	0.0126 (16)
C13	0.0825 (19)	0.0504 (16)	0.0653 (17)	−0.0083 (14)	−0.0043 (15)	0.0053 (13)

Geometric parameters (Å, º)

Cl1—C4	1.739 (3)	C6—H6	0.9300
O1—C1	1.335 (3)	C7—C8	1.491 (4)
O1—H1	0.8200	C8—C9	1.365 (4)
N1—C7	1.290 (3)	C8—C13	1.378 (4)
N1—N1^{i}	1.388 (4)	C9—C10	1.388 (4)
C1—C6	1.387 (4)	C9—H9	0.9300
C1—C2	1.414 (4)	C10—C11	1.365 (5)
C2—C3	1.394 (4)	C10—H10	0.9300
C2—C7	1.469 (3)	C11—C12	1.355 (5)
C3—C4	1.360 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.376 (4)
C4—C5	1.381 (4)	C12—H12	0.9300
C5—C6	1.359 (4)	C13—H13	0.9300
C5—H5	0.9300		
C1—O1—H1	109.5	N1—C7—C8	122.8 (2)
C7—N1—N1^{i}	114.9 (2)	C2—C7—C8	120.0 (2)
O1—C1—C6	117.7 (2)	C9—C8—C13	119.0 (3)
O1—C1—C2	122.9 (2)	C9—C8—C7	121.5 (3)
C6—C1—C2	119.4 (3)	C13—C8—C7	119.5 (2)
C3—C2—C1	117.8 (2)	C8—C9—C10	120.2 (4)
C3—C2—C7	120.2 (2)	C8—C9—H9	119.9
C1—C2—C7	122.0 (2)	C10—C9—H9	119.9
C4—C3—C2	121.1 (2)	C11—C10—C9	120.0 (4)
C4—C3—H3	119.5	C11—C10—H10	120.0
C2—C3—H3	119.5	C9—C10—H10	120.0

C3—C4—C5	121.0 (2)	C12—C11—C10	120.2 (3)
C3—C4—Cl1	120.50 (19)	C12—C11—H11	119.9
C5—C4—Cl1	118.5 (2)	C10—C11—H11	119.9
C6—C5—C4	119.2 (2)	C11—C12—C13	120.0 (3)
C6—C5—H5	120.4	C11—C12—H12	120.0
C4—C5—H5	120.4	C13—C12—H12	120.0
C5—C6—C1	121.5 (2)	C12—C13—C8	120.6 (3)
C5—C6—H6	119.3	C12—C13—H13	119.7
C1—C6—H6	119.3	C8—C13—H13	119.7
N1—C7—C2	117.2 (2)		

O1—C1—C2—C3	−178.7 (3)	C1—C2—C7—N1	1.8 (4)
C6—C1—C2—C3	0.8 (4)	C3—C2—C7—C8	1.7 (4)
O1—C1—C2—C7	0.6 (4)	C1—C2—C7—C8	−177.6 (2)
C6—C1—C2—C7	−179.9 (3)	N1—C7—C8—C9	83.9 (4)
C1—C2—C3—C4	−0.8 (4)	C2—C7—C8—C9	−96.8 (3)
C7—C2—C3—C4	179.9 (2)	N1—C7—C8—C13	−94.4 (3)
C2—C3—C4—C5	0.4 (4)	C2—C7—C8—C13	84.9 (3)
C2—C3—C4—Cl1	−179.5 (2)	C13—C8—C9—C10	0.5 (5)
C3—C4—C5—C6	0.2 (5)	C7—C8—C9—C10	−177.8 (3)
Cl1—C4—C5—C6	−180.0 (3)	C8—C9—C10—C11	0.4 (6)
C4—C5—C6—C1	−0.2 (5)	C9—C10—C11—C12	−1.2 (6)
O1—C1—C6—C5	179.2 (3)	C10—C11—C12—C13	1.0 (5)
C2—C1—C6—C5	−0.3 (5)	C11—C12—C13—C8	−0.1 (5)
N1^{i}—N1—C7—C2	−179.4 (3)	C9—C8—C13—C12	−0.6 (4)
N1^{i}—N1—C7—C8	0.0 (4)	C7—C8—C13—C12	177.7 (3)
C3—C2—C7—N1	−178.9 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.85	2.572 (3)	145