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4-Chloro-2-[(*E*)-(2-chlorophenyl)iminomethyl]phenol

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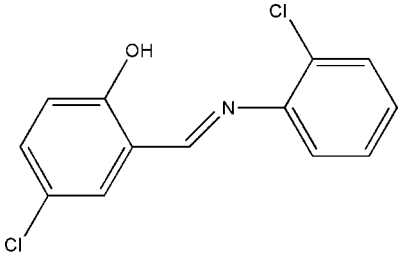
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.099; data-to-parameter ratio = 13.6.

The title compound, $C_{13}H_9Cl_2NO$, was crystallized from a methanol solution of 5-chlorosalicylaldehyde and *o*-chloroaniline. The molecule displays a *trans* configuration with respect to the imine C=N double bond. The N atom is involved in an intramolecular O—H···N hydrogen bond. The two aromatic rings are essentially coplanar, the dihedral angle between them being 7.1 (1)°. A C—H···π interaction is present in the crystal.

Related literature

For the biological properties of Schiff bases containing O and N atoms, see: Antony *et al.* (1999); Lumme & Elo (1984); Yao *et al.* (1999). For its chemical behaviour, see: Ueno *et al.* (2006).



Experimental

Crystal data

$C_{13}H_9Cl_2NO$
M_r = 266.11
Orthorhombic, *Pbca*
a = 7.2693 (13) Å
b = 13.0037 (19) Å
c = 25.2711 (16) Å
V = 2388.8 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 298 (2) K
0.50 × 0.48 × 0.47 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
T_{min} = 0.780, T_{max} = 0.791
11102 measured reflections
2103 independent reflections
1496 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
S = 1.08
2103 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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	1.87	2.603 (3)	147
C11—H11···$Cg1^{i}$	0.93	2.97	3.549 (3)	122

Symmetry code: (i) $x-\frac{1}{2}, y, -z+\frac{1}{2}$. $Cg1$ is the centroid of C8–C13 phenyl ring.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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: BQ2118).

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supporting information

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4-Chloro-2-[(*E*)-(2-chlorophenyl)iminomethyl]phenol

Xinli Zhang

S1. Comment

The Schiff base containing some O and N atoms is a new important biological ligand and it shows some interesting biological properties, such as antibacterial, antiphlogistic, anticancer and high catalytic activities (Antony *et al.*, 1999; Lumme & Elo *et al.*, 1984; Yao *et al.*, 1999), so the chemical behavior of the Schiff base has drawn our attention (Ueno *et al.*, 2006). Our research emphasis is focused on the synthesis of the Schiff base. Then, a new crystal structure of the title compound, (I), is reported here.

The molecular structure of (I) are illustrated in Fig. 1. In the structure of (I), the whole molecule is essentially planar with a 7.1 (2)° dihedral angle between the two phenyl rings. The C1═N1 bond distance [1.277 (3)Å] is shorter than the standart 1.28Å value of C═N double bond, indicating a delocalization of π-electron density across the phenyl ring. In addition to the intramolecular O-H..N hydrogen bond, there is also an intermolecular C-H..π interaction (Table 1.)

S2. Experimental

A solution of 5-chlorosalicylaldehyde (0.1 mmol, 15.7 mg) in methanol (10 ml) was added dropwise to the methanol (10 ml) solution of *o*-chloroaniline (0.1 mmol, 12.8 mg) with stirring. The mixture was stirred at room temperature for one hour and then filtered. After allowing the filtrate to stand in air for 3 d, yellow block-shaped crystals of the title compound were formed in slow evaporation of the solvent. The crystals were collected, washed with methanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 60%).

S3. Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances 0.93Å and U_{iso}(H) = 1.2 U_{eq}(C) and O—H distances 0.82Å and U_{iso}(H) = 1.5 U_{eq}(O).

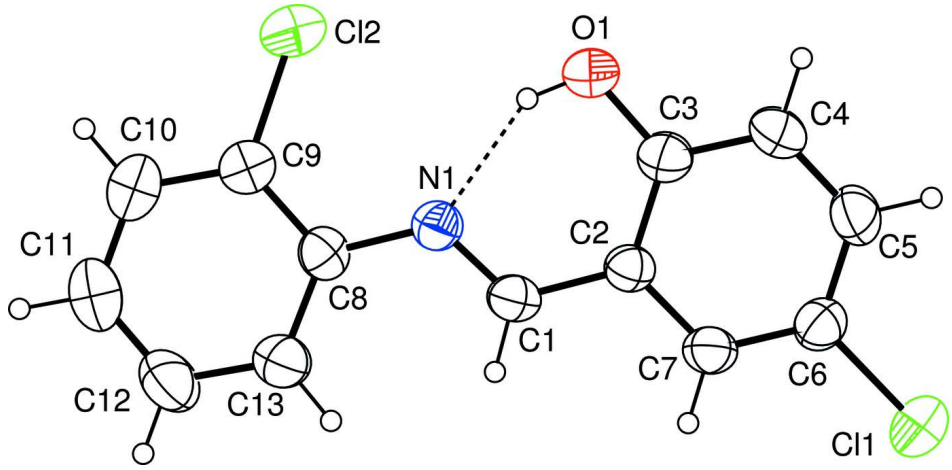

Figure 1

The structure of the title compound with 30% probability ellipsoids. The dashed line represents hydrogen bond.

4-Chloro-2-[(*E*)-(2-chlorophenyl)iminomethyl]phenol

Crystal data

$C_{13}H_9Cl_2NO$
$M_r = 266.11$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 7.2693$ (13) Å
$b = 13.0037$ (19) Å
$c = 25.2711$ (16) Å
$V = 2388.8$ (6) Å^3
$Z = 8$
$F(000) = 1088$
$D_x = 1.480$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3178 reflections
$\theta = 2.9$–$26.3°$
$\mu = 0.52$ mm^{-1}
$T = 298$ K
Block, yellow
0.50 × 0.48 × 0.47 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
$T_{min} = 0.780$, $T_{max} = 0.791$
11102 measured reflections
2103 independent reflections
1496 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.6°$
$h = -8 \rightarrow 8$
$k = -15 \rightarrow 11$
$l = -30 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.099$
$S = 1.08$
2103 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 1.7853P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0069 (6)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.07629 (12)	1.13163 (6)	1.12839 (3)	0.0653 (3)
Cl2	0.41980 (14)	0.79479 (6)	0.83555 (3)	0.0760 (3)
N1	0.3625 (3)	0.96742 (15)	0.90783 (8)	0.0407 (5)
O1	0.2932 (4)	0.81908 (14)	0.97404 (8)	0.0734 (7)
H1	0.3252	0.8457	0.9461	0.110*
C1	0.3120 (3)	1.03135 (19)	0.94334 (9)	0.0396 (6)
H1A	0.3132	1.1013	0.9357	0.048*
C2	0.2530 (3)	0.99774 (18)	0.99498 (9)	0.0366 (6)
C3	0.2445 (4)	0.89294 (19)	1.00872 (10)	0.0482 (7)
C4	0.1860 (4)	0.8649 (2)	1.05868 (11)	0.0591 (8)
H4	0.1806	0.7956	1.0677	0.071*
C5	0.1357 (4)	0.9376 (2)	1.09521 (11)	0.0519 (7)
H5	0.0972	0.9178	1.1288	0.062*
C6	0.1427 (4)	1.04066 (19)	1.08177 (10)	0.0432 (6)
C7	0.1990 (4)	1.07049 (19)	1.03254 (10)	0.0422 (6)
H7	0.2014	1.1400	1.0239	0.051*
C8	0.4229 (3)	0.99867 (19)	0.85743 (9)	0.0393 (6)
C9	0.4587 (4)	0.9228 (2)	0.82003 (11)	0.0462 (7)
C10	0.5255 (4)	0.9469 (2)	0.77026 (11)	0.0588 (8)
H10	0.5487	0.8949	0.7459	0.071*
C11	0.5574 (4)	1.0476 (3)	0.75699 (12)	0.0627 (8)
H11	0.6026	1.0641	0.7236	0.075*
C12	0.5224 (5)	1.1235 (2)	0.79298 (12)	0.0635 (9)
H12	0.5439	1.1918	0.7839	0.076*
C13	0.4556 (4)	1.1001 (2)	0.84244 (11)	0.0544 (7)
H13	0.4319	1.1529	0.8663	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0859 (6)	0.0587 (5)	0.0515 (4)	0.0011 (4)	0.0102 (4)	−0.0135 (4)
Cl2	0.1089 (8)	0.0424 (4)	0.0766 (6)	−0.0022 (4)	0.0226 (5)	−0.0113 (4)
N1	0.0478 (13)	0.0375 (12)	0.0367 (11)	−0.0015 (10)	−0.0029 (10)	−0.0003 (10)
O1	0.128 (2)	0.0355 (10)	0.0562 (12)	0.0156 (12)	0.0259 (13)	0.0034 (9)
C1	0.0434 (15)	0.0330 (13)	0.0424 (14)	−0.0013 (11)	−0.0043 (12)	0.0023 (12)
C2	0.0385 (13)	0.0335 (12)	0.0377 (13)	−0.0001 (10)	−0.0036 (12)	0.0011 (11)

C3	0.0614 (18)	0.0379 (14)	0.0452 (15)	0.0094 (13)	0.0001 (14)	0.0028 (12)
C4	0.088 (2)	0.0384 (15)	0.0512 (16)	0.0129 (15)	0.0078 (16)	0.0119 (14)
C5	0.0633 (19)	0.0532 (17)	0.0391 (14)	0.0078 (14)	0.0032 (14)	0.0097 (13)
C6	0.0467 (16)	0.0428 (15)	0.0402 (14)	0.0026 (12)	−0.0029 (12)	−0.0038 (12)
C7	0.0481 (16)	0.0343 (13)	0.0443 (15)	−0.0037 (11)	−0.0030 (12)	−0.0001 (11)
C8	0.0390 (14)	0.0436 (14)	0.0353 (13)	−0.0006 (12)	−0.0059 (11)	0.0024 (11)
C9	0.0472 (17)	0.0435 (15)	0.0479 (15)	0.0012 (12)	−0.0004 (13)	−0.0024 (12)
C10	0.060 (2)	0.068 (2)	0.0478 (16)	0.0039 (16)	0.0083 (15)	−0.0077 (15)
C11	0.063 (2)	0.080 (2)	0.0456 (16)	−0.0032 (17)	0.0071 (15)	0.0107 (17)
C12	0.082 (2)	0.0572 (18)	0.0518 (17)	−0.0089 (17)	0.0014 (16)	0.0151 (15)
C13	0.073 (2)	0.0429 (15)	0.0475 (16)	−0.0042 (14)	−0.0005 (15)	0.0022 (13)

Geometric parameters (Å, º)

Cl1—C6	1.738 (3)	C5—H5	0.9300
Cl2—C9	1.733 (3)	C6—C7	1.366 (3)
N1—C1	1.277 (3)	C7—H7	0.9300
N1—C8	1.407 (3)	C8—C9	1.391 (4)
O1—C3	1.347 (3)	C8—C13	1.393 (4)
O1—H1	0.8200	C9—C10	1.384 (4)
C1—C2	1.441 (3)	C10—C11	1.371 (4)
C1—H1A	0.9300	C10—H10	0.9300
C2—C7	1.397 (3)	C11—C12	1.367 (4)
C2—C3	1.408 (3)	C11—H11	0.9300
C3—C4	1.381 (4)	C12—C13	1.375 (4)
C4—C5	1.371 (4)	C12—H12	0.9300
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.383 (4)		
C1—N1—C8	122.5 (2)	C6—C7—H7	119.6
C3—O1—H1	109.5	C2—C7—H7	119.6
N1—C1—C2	121.6 (2)	C9—C8—C13	117.1 (2)
N1—C1—H1A	119.2	C9—C8—N1	117.9 (2)
C2—C1—H1A	119.2	C13—C8—N1	124.9 (2)
C7—C2—C3	118.4 (2)	C10—C9—C8	121.5 (3)
C7—C2—C1	119.6 (2)	C10—C9—Cl2	118.7 (2)
C3—C2—C1	122.0 (2)	C8—C9—Cl2	119.8 (2)
O1—C3—C4	119.2 (2)	C11—C10—C9	119.9 (3)
O1—C3—C2	121.2 (2)	C11—C10—H10	120.1
C4—C3—C2	119.6 (2)	C9—C10—H10	120.1
C5—C4—C3	121.0 (3)	C12—C11—C10	119.7 (3)
C5—C4—H4	119.5	C12—C11—H11	120.1
C3—C4—H4	119.5	C10—C11—H11	120.1
C4—C5—C6	119.5 (3)	C11—C12—C13	120.7 (3)
C4—C5—H5	120.2	C11—C12—H12	119.6
C6—C5—H5	120.2	C13—C12—H12	119.6
C7—C6—C5	120.6 (2)	C12—C13—C8	121.2 (3)
C7—C6—Cl1	120.5 (2)	C12—C13—H13	119.4

C5—C6—Cl1	118.9 (2)	C8—C13—H13	119.4
C6—C7—C2	120.7 (2)		
C8—N1—C1—C2	−179.1 (2)	C1—C2—C7—C6	−179.9 (2)
N1—C1—C2—C7	180.0 (2)	C1—N1—C8—C9	−174.2 (2)
N1—C1—C2—C3	−1.2 (4)	C1—N1—C8—C13	8.1 (4)
C7—C2—C3—O1	179.4 (3)	C13—C8—C9—C10	0.6 (4)
C1—C2—C3—O1	0.6 (4)	N1—C8—C9—C10	−177.4 (3)
C7—C2—C3—C4	−0.8 (4)	C13—C8—C9—Cl2	−179.5 (2)
C1—C2—C3—C4	−179.6 (3)	N1—C8—C9—Cl2	2.5 (3)
O1—C3—C4—C5	179.8 (3)	C8—C9—C10—C11	−0.1 (4)
C2—C3—C4—C5	0.1 (5)	Cl2—C9—C10—C11	180.0 (2)
C3—C4—C5—C6	0.3 (5)	C9—C10—C11—C12	−0.2 (5)
C4—C5—C6—C7	0.1 (4)	C10—C11—C12—C13	0.1 (5)
C4—C5—C6—Cl1	179.4 (2)	C11—C12—C13—C8	0.4 (5)
C5—C6—C7—C2	−0.9 (4)	C9—C8—C13—C12	−0.7 (4)
Cl1—C6—C7—C2	179.8 (2)	N1—C8—C13—C12	177.1 (3)
C3—C2—C7—C6	1.2 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.87	2.603 (3)	147
C11—H11···*Cg*1^{i}	0.93	2.97	3.549 (3)	122

Symmetry code: (i) $x-1/2, y, -z+1/2$.