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

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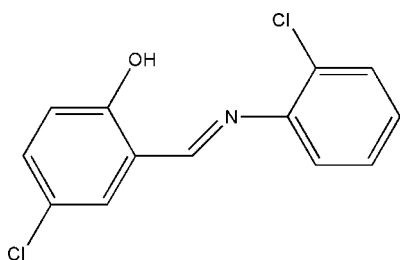
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.037; wR factor = 0.099; data-to-parameter ratio = 13.6.

The title compound, $\text{C}_{13}\text{H}_9\text{Cl}_2\text{NO}$, was crystallized from a methanol solution of 5-chlorosalicylaldehyde and *o*-chloroaniline. The molecule displays a *trans* configuration with respect to the imine $\text{C}=\text{N}$ double bond. The N atom is involved in an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond. The two aromatic rings are essentially coplanar, the dihedral angle between them being $7.1(1)^\circ$. A $\text{C}-\text{H}\cdots\pi$ 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

$\text{C}_{13}\text{H}_9\text{Cl}_2\text{NO}$
 $M_r = 266.11$
 Orthorhombic, *Pbca*
 $a = 7.2693(13)$ Å
 $b = 13.0037(19)$ Å
 $c = 25.2711(16)$ Å
 $V = 2388.8(6)$ Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.52$ mm⁻¹
 $T = 298(2)$ K
 $0.50 \times 0.48 \times 0.47$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

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, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.87	2.603 (3)	147
$\text{C11}-\text{H11}\cdots\text{Cg1}^i$	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: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; 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).

References

- Antony, F. M., O'Mahony, R. S. & Joyce, M. W. (1999). *Croat. Chem. Acta*, **72**, 685–703.
 Lumme, P. & Elo, H. (1984). *Inorg. Chim. Acta*, **92**, 241–251.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Siemens (1996). *SMART*, *SAINTE* and *SADABS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Ueno, T., Yokoi, N., Unno, M., Matsui, T., Tokita, Y., Yamada, M., Ikeda-Saito, M., Nakajima, H. & Watanabe, Y. (2006). *PNAS*, **103**, 9416–9421.
 Yao, K. M., Li, N., Huang, Q. H., Shen, L.-F. & Yuan, H.-Z. (1999). *Sci. China Ser. B Chem.* **42**, 53–61.

supplementary materials

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

X. Zhang

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.)

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 CaCl₂ (yield 60%).

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 $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ and O—H distances 0.82Å and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

Figures

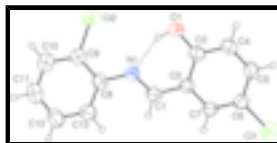


Fig. 1. The structure of the title compound with 30% probability ellipsoids. The dashed line represents hydrogen bond.

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

Crystal data

C₁₃H₉Cl₂NO

$F_{000} = 1088$

supplementary materials

$M_r = 266.11$

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) Å³

$Z = 8$

$D_x = 1.480$ 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

$0.50 \times 0.48 \times 0.47$ mm

Data collection

Siemens SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ K

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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.041$

$\theta_{\max} = 25.0^\circ$

$\theta_{\min} = 1.6^\circ$

$h = -8 \rightarrow 8$

$k = -15 \rightarrow 11$

$l = -30 \rightarrow 29$

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

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),

$$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.07629 (12)	1.13163 (6)	1.12839 (3)	0.0653 (3)
C12	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 (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0859 (6)	0.0587 (5)	0.0515 (4)	0.0011 (4)	0.0102 (4)	-0.0135 (4)
C12	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)

supplementary materials

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, °)

C11—C6	1.738 (3)	C5—H5	0.9300
C12—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—C12	118.7 (2)
C3—C2—C1	122.0 (2)	C8—C9—C12	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—C11	120.5 (2)	C12—C13—H13	119.4
C5—C6—C11	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—C12	-179.5 (2)

C1—C2—C3—C4	-179.6 (3)	N1—C8—C9—C12	2.5 (3)
O1—C3—C4—C5	179.8 (3)	C8—C9—C10—C11	-0.1 (4)
C2—C3—C4—C5	0.1 (5)	C12—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—C11	179.4 (2)	C11—C12—C13—C8	0.4 (5)
C5—C6—C7—C2	-0.9 (4)	C9—C8—C13—C12	-0.7 (4)
C11—C6—C7—C2	179.8 (2)	N1—C8—C13—C12	177.1 (3)
C3—C2—C7—C6	1.2 (4)		

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.82	1.87	2.603 (3)	147
C11—H11 \cdots Cg1 ⁱ	0.93	2.97	3.549 (3)	122

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

Fig. 1

