

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-[(*E*)-(2-Chlorophenyl)iminomethyl]-6-methylphenol

Peihua Zhu,* Jiemei Yu, Hongyan Wang, Chunlai Zhang and Dongming Yang

School of Chemistry and Chemical Engineering, University of Jinan, Jinan 250022, People's Republic of China

Correspondence e-mail: chm_zhuph@ujn.edu.cn

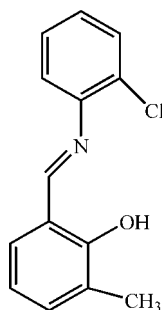
Received 18 August 2010; accepted 23 August 2010

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.051; wR factor = 0.096; data-to-parameter ratio = 15.7.

The title compound, $\text{C}_{14}\text{H}_{12}\text{ClNO}$, a Schiff base derived from 3-methylsalicylaldehyde, crystallizes in the phenol-imine tautomeric form with an *E* conformation for the imine functionality. The molecule is not planar, the dihedral angle between the aromatic rings being 36.38 (5)°. The hydroxy H atom is involved in a strong intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond, generating an *S*(6) ring.

Related literature

For background information and applications of Schiff base complexes, see: Barton & Ollis (1979); Layer (1963); Ingold (1969); Cohen *et al.* (1964); Henrici-Olive & Olive (1984); Garnovskii *et al.* (1993). For related structures, see: Köysal *et al.* (2007); Kılıç *et al.* (2009); Şahin *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{14}\text{H}_{12}\text{ClNO}$ $M_r = 245.70$ Orthorhombic, $P2_12_12_1$ $a = 7.8318$ (14) Å $b = 11.693$ (2) Å $c = 13.250$ (2) Å $V = 1213.4$ (4) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.30$ mm⁻¹ $T = 293$ K $0.21 \times 0.11 \times 0.06$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 2003)

 $T_{\min} = 0.940$, $T_{\max} = 0.982$

6803 measured reflections

2477 independent reflections

1486 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.070$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.051$ $wR(F^2) = 0.096$ $S = 1.00$

2477 reflections

158 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.15$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Absolute structure: Flack (1983),

1034 Friedel pairs

Flack parameter: -0.06 (9)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.87 (1)	1.84 (2)	2.615 (3)	148 (3)

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; 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.

This work was supported by Shandong Province (2007BS02016).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2056).

References

- Barton, D. & Ollis, W. D. (1979). *Comprehensive Organic Chemistry*, Vol. 2. Oxford: Pergamon Press.
- Bruker (2001). *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cohen, M. D., Schmidt, G. M. J. & Flavian, S. (1964). *J. Chem. Soc.* pp. 2041–2051.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Garnovskii, A. D., Nivorozhkin, A. L. & Minkin, V. I. (1993). *Coord. Chem. Rev.* **126**, 1–69.
- Henrici-Olive, G. & Olive, S. (1984). *The Chemistry of the Catalyzed Hydrogenation of Carbon Monoxide*. Berlin: Springer.
- Ingold, C. K. (1969). *Structure and Mechanism in Organic Chemistry*, 2nd ed. Ithaca: Cornell University Press.
- Kılıç, I., Işık, Ş., Açar, E. & Erşahin, F. (2009). *Acta Cryst.* **E65**, o1347.
- Köysal, Y., Işık, Ş. & Açar, A. (2007). *Acta Cryst.* **E63**, o4916.
- Layer, R. W. (1963). *Chem. Rev.* **63**, 489–510.
- Şahin, Z. S., Işık, Ş., Erşahin, F. & Açar, E. (2009). *Acta Cryst.* **E65**, o811.
- Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2010). E66, o2460 [https://doi.org/10.1107/S1600536810033969]

2-[(*E*)-(2-Chlorophenyl)iminomethyl]-6-methylphenol**Peihua Zhu, Jiemei Yu, Hongyan Wang, Chunlai Zhang and Dongming Yang****S1. Comment**

Schiff bases are used as starting materials in the synthesis of important drugs (Layer, 1963; Ingold, 1969). A large number of Schiff bases and their complexes have been studied for their interesting and important properties, *e.g.* catalytic activity (Henrici-Olive & Olive, 1984), photochromic properties (Cohen *et al.*, 1964), biological activity (Barton *et al.*, 1979). On the other hand, Schiff base ligands play a vital role in coordination chemistry due to their metal binding ability (Garnovskii *et al.*, 1993).

The structure of the title compound is shown in Fig. 1. The C7=N1 double bond of 1.283 (3) Å is slightly longer than the literature values found in similar structures (Köysal *et al.*, 2007; Kılıç *et al.*, 2009; Şahin *et al.*, 2009) in the range of 1.262 (8)-1.279 (3) Å. The title molecule is not planar with a dihedral angle between the aromatic rings C1/C6 and C8/C13 of 36.38 (5)°. The imino group is coplanar with the hydroxyphenyl ring with the torsion angle C13—C8—C7—N1 of 1.6 (4)°.

The molecular structure is stabilized by a strong intramolecular O—H⋯N hydrogen bond.

S2. Experimental

A solution of 3-methylsalicylaldehyde (0.0681 g, 0.5 mmol) in ethanol (10 ml) was added to a solution of 2-chlorobenzenamine (0.0638 g, 0.5 mmol) in ethanol (20 ml). The reaction mixture was stirred for 2 h under reflux. Single crystals suitable for a X-ray analysis were obtained from ethanol by slow evaporation (0.0749 g, 61%).

S3. Refinement

The H atom bounded to O1 was located in the difference Fourier map and freely refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. All other H atoms were placed in calculated positions and refined using a riding-model approximation with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms, and with C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

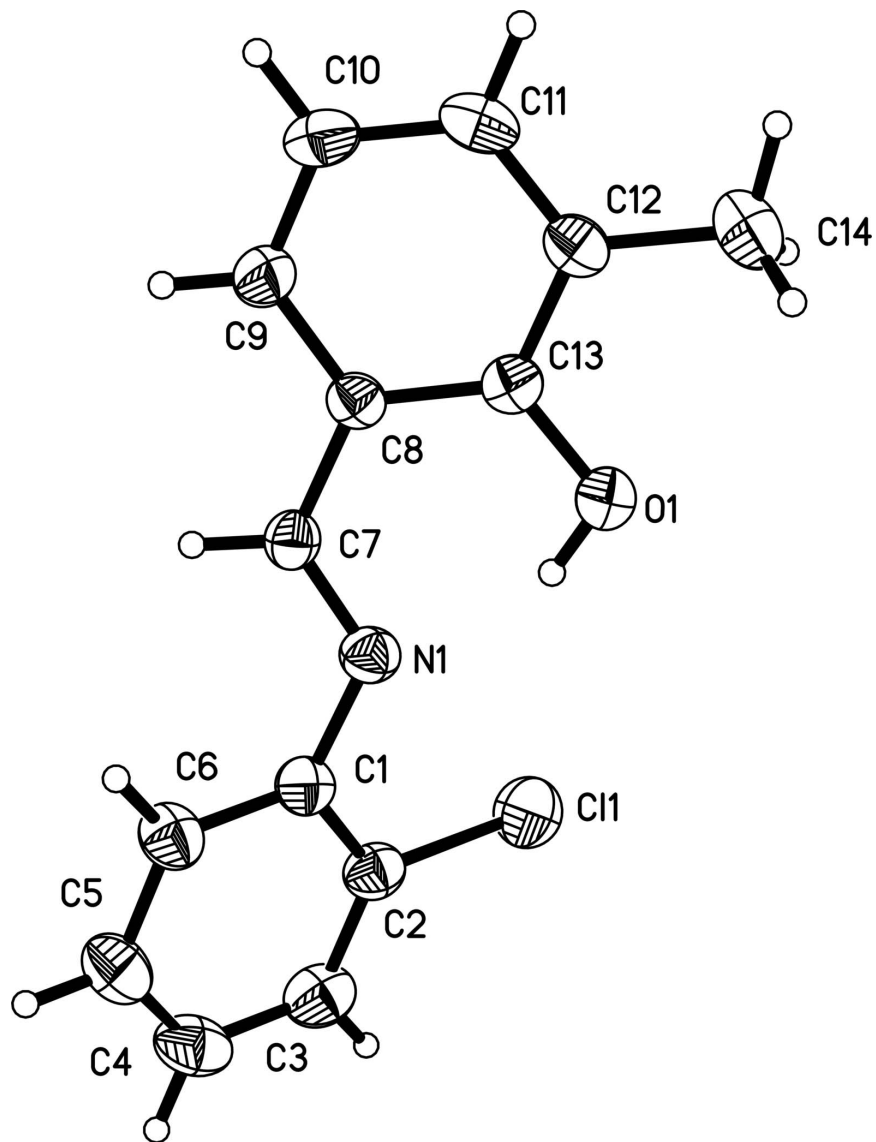


Figure 1

The molecular structure of the title compound, with the atom-numbering scheme and 25% probability displacement ellipsoids for the non-H atoms.

2-[(*E*)-(2-Chlorophenyl)iminomethyl]-6-methylphenol

Crystal data

$C_{14}H_{12}ClNO$

$M_r = 245.70$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.8318 (14) \text{ \AA}$

$b = 11.693 (2) \text{ \AA}$

$c = 13.250 (2) \text{ \AA}$

$V = 1213.4 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 512$

$D_x = 1.345 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1163 reflections

$\theta = 3.1\text{--}28.8^\circ$

$\mu = 0.30 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Needle, yellow

$0.21 \times 0.11 \times 0.06 \text{ mm}$

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)
 $T_{\min} = 0.940$, $T_{\max} = 0.982$

6803 measured reflections
2477 independent reflections
1486 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.070$
 $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 3.1^\circ$
 $h = -9 \rightarrow 9$
 $k = -14 \rightarrow 14$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.096$
 $S = 1.00$
2477 reflections
158 parameters
1 restraint
Primary atom site location: structure-invariant
direct methods
Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 0.0414P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$
Absolute structure: Flack (1983), 1034 Friedel
pairs
Absolute structure parameter: -0.06 (9)

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	-0.30427 (11)	-0.84282 (9)	-0.18201 (8)	0.0873 (3)
C11	-0.2023 (4)	-0.5269 (3)	-0.6436 (3)	0.0609 (9)
H11	-0.1358	-0.4949	-0.6945	0.073*
C2	-0.6147 (5)	-0.8244 (3)	-0.0959 (3)	0.0673 (9)
H2	-0.5727	-0.8740	-0.0469	0.081*
C4	-0.8405 (4)	-0.7124 (3)	-0.1622 (3)	0.0704 (10)
H4	-0.9518	-0.6851	-0.1577	0.085*
C13	-0.2304 (4)	-0.5959 (2)	-0.4763 (2)	0.0460 (7)
N1	-0.4592 (3)	-0.68266 (18)	-0.32559 (19)	0.0510 (6)
C8	-0.4038 (3)	-0.6210 (2)	-0.4942 (2)	0.0446 (7)
C5	-0.7384 (3)	-0.6793 (3)	-0.2418 (3)	0.0600 (8)
H5	-0.7817	-0.6305	-0.2910	0.072*
C1	-0.5125 (4)	-0.7905 (2)	-0.1741 (2)	0.0546 (8)
C3	-0.7793 (5)	-0.7849 (3)	-0.0899 (3)	0.0726 (10)
H3	-0.8492	-0.8075	-0.0368	0.087*

C9	-0.4702 (4)	-0.5986 (2)	-0.5894 (2)	0.0568 (8)
H9	-0.5835	-0.6167	-0.6030	0.068*
C7	-0.5128 (3)	-0.6658 (2)	-0.4159 (2)	0.0476 (7)
H7	-0.6257	-0.6830	-0.4313	0.057*
C12	-0.1285 (3)	-0.5488 (2)	-0.5514 (2)	0.0509 (8)
C6	-0.5706 (3)	-0.7186 (2)	-0.2491 (2)	0.0495 (7)
C10	-0.3721 (4)	-0.5507 (3)	-0.6631 (2)	0.0638 (9)
H10	-0.4190	-0.5342	-0.7260	0.077*
C14	0.0568 (4)	-0.5250 (3)	-0.5305 (3)	0.0781 (11)
H14C	0.1143	-0.5953	-0.5150	0.117*
H14A	0.0662	-0.4737	-0.4742	0.117*
H14B	0.1081	-0.4907	-0.5889	0.117*
O1	-0.1569 (2)	-0.61668 (18)	-0.38591 (17)	0.0618 (6)
H1	-0.233 (3)	-0.648 (3)	-0.3472 (19)	0.074*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0762 (5)	0.1058 (7)	0.0799 (7)	0.0203 (5)	-0.0073 (5)	0.0146 (6)
C11	0.072 (2)	0.0524 (19)	0.058 (2)	0.0033 (17)	0.0238 (19)	0.0008 (17)
C2	0.089 (3)	0.054 (2)	0.059 (2)	-0.0124 (19)	-0.002 (2)	0.0131 (19)
C4	0.064 (2)	0.065 (2)	0.083 (3)	-0.0066 (17)	0.016 (2)	0.000 (2)
C13	0.0515 (19)	0.0393 (16)	0.0471 (19)	0.0069 (13)	0.0008 (15)	-0.0042 (15)
N1	0.0543 (13)	0.0510 (14)	0.0477 (16)	-0.0051 (11)	0.0029 (14)	0.0016 (14)
C8	0.0473 (18)	0.0412 (17)	0.0452 (19)	0.0026 (12)	0.0044 (15)	-0.0075 (15)
C5	0.0581 (18)	0.0538 (19)	0.068 (2)	-0.0021 (15)	0.0058 (17)	0.0120 (18)
C1	0.0629 (18)	0.0511 (16)	0.0499 (19)	-0.0042 (15)	-0.0015 (18)	-0.0013 (18)
C3	0.087 (3)	0.064 (2)	0.067 (2)	-0.0186 (19)	0.022 (2)	0.002 (2)
C9	0.0564 (17)	0.0652 (18)	0.049 (2)	0.0009 (15)	-0.0027 (17)	-0.0051 (19)
C7	0.0468 (15)	0.0460 (16)	0.050 (2)	-0.0023 (14)	-0.0025 (16)	-0.0025 (17)
C12	0.0527 (17)	0.0432 (16)	0.057 (2)	0.0023 (14)	0.0098 (18)	-0.0034 (16)
C6	0.0539 (18)	0.0423 (16)	0.0523 (19)	-0.0094 (13)	0.0014 (16)	-0.0013 (17)
C10	0.079 (2)	0.071 (2)	0.042 (2)	0.0057 (18)	0.0028 (18)	-0.0004 (19)
C14	0.057 (2)	0.085 (2)	0.093 (3)	-0.0041 (17)	0.0114 (19)	0.004 (2)
O1	0.0516 (12)	0.0784 (16)	0.0554 (15)	-0.0011 (11)	-0.0025 (11)	0.0044 (12)

Geometric parameters (Å, °)

C11—C1	1.745 (3)	C8—C9	1.388 (4)
C11—C12	1.376 (4)	C8—C7	1.442 (4)
C11—C10	1.384 (4)	C5—C6	1.396 (4)
C11—H11	0.9300	C5—H5	0.9300
C2—C1	1.369 (4)	C1—C6	1.378 (4)
C2—C3	1.372 (4)	C3—H3	0.9300
C2—H2	0.9300	C9—C10	1.363 (4)
C4—C3	1.366 (4)	C9—H9	0.9300
C4—C5	1.379 (4)	C7—H7	0.9300
C4—H4	0.9300	C12—C14	1.503 (4)

C13—O1	1.351 (3)	C10—H10	0.9300
C13—C12	1.389 (4)	C14—H14C	0.9600
C13—C8	1.410 (4)	C14—H14A	0.9600
N1—C7	1.283 (3)	C14—H14B	0.9600
N1—C6	1.402 (3)	O1—H1	0.87 (3)
C12—C11—C10	122.2 (3)	C2—C3—H3	120.1
C12—C11—H11	118.9	C10—C9—C8	121.2 (3)
C10—C11—H11	118.9	C10—C9—H9	119.4
C1—C2—C3	119.7 (3)	C8—C9—H9	119.4
C1—C2—H2	120.1	N1—C7—C8	122.2 (3)
C3—C2—H2	120.1	N1—C7—H7	118.9
C3—C4—C5	120.5 (3)	C8—C7—H7	118.9
C3—C4—H4	119.8	C11—C12—C13	117.9 (3)
C5—C4—H4	119.8	C11—C12—C14	122.3 (3)
O1—C13—C12	117.5 (3)	C13—C12—C14	119.8 (3)
O1—C13—C8	121.4 (3)	C1—C6—C5	117.5 (3)
C12—C13—C8	121.1 (3)	C1—C6—N1	119.9 (3)
C7—N1—C6	121.1 (2)	C5—C6—N1	122.5 (3)
C9—C8—C13	118.3 (3)	C9—C10—C11	119.3 (3)
C9—C8—C7	120.0 (3)	C9—C10—H10	120.3
C13—C8—C7	121.7 (3)	C11—C10—H10	120.3
C4—C5—C6	120.4 (3)	C12—C14—H14C	109.5
C4—C5—H5	119.8	C12—C14—H14A	109.5
C6—C5—H5	119.8	H14C—C14—H14A	109.5
C2—C1—C6	122.0 (3)	C12—C14—H14B	109.5
C2—C1—C11	119.4 (3)	H14C—C14—H14B	109.5
C6—C1—C11	118.6 (2)	H14A—C14—H14B	109.5
C4—C3—C2	119.9 (3)	C13—O1—H1	108 (2)
C4—C3—H3	120.1		
O1—C13—C8—C9	-179.2 (2)	C10—C11—C12—C14	178.9 (3)
C12—C13—C8—C9	0.7 (4)	O1—C13—C12—C11	-179.7 (2)
O1—C13—C8—C7	2.5 (4)	C8—C13—C12—C11	0.4 (4)
C12—C13—C8—C7	-177.6 (2)	O1—C13—C12—C14	1.0 (4)
C3—C4—C5—C6	-0.6 (4)	C8—C13—C12—C14	-178.9 (3)
C3—C2—C1—C6	-1.1 (5)	C2—C1—C6—C5	1.1 (4)
C3—C2—C1—C11	-179.1 (3)	C11—C1—C6—C5	179.1 (2)
C5—C4—C3—C2	0.6 (5)	C2—C1—C6—N1	177.5 (3)
C1—C2—C3—C4	0.2 (5)	C11—C1—C6—N1	-4.5 (3)
C13—C8—C9—C10	-1.8 (4)	C4—C5—C6—C1	-0.2 (4)
C7—C8—C9—C10	176.5 (3)	C4—C5—C6—N1	-176.6 (3)
C6—N1—C7—C8	175.4 (2)	C7—N1—C6—C1	146.1 (3)
C9—C8—C7—N1	-176.7 (3)	C7—N1—C6—C5	-37.6 (4)
C13—C8—C7—N1	1.6 (4)	C8—C9—C10—C11	1.8 (5)
C10—C11—C12—C13	-0.4 (4)	C12—C11—C10—C9	-0.7 (5)

Hydrogen-bond geometry (Å, °)

<i>D—H⋯A</i>	<i>D—H</i>	<i>H⋯A</i>	<i>D⋯A</i>	<i>D—H⋯A</i>
O1—H1⋯N1	0.87 (1)	1.84 (2)	2.615 (3)	148 (3)
