

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-[[2-(4-Chlorophenyl)hydrazinylidene]-methyl]phenol

Kong Mun Lo and Seik Weng Ng*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

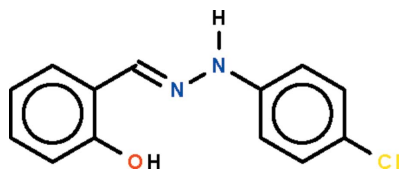
Received 9 May 2011; accepted 12 May 2011

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;
 R factor = 0.031; wR factor = 0.089; data-to-parameter ratio = 16.1.

In the title Schiff base molecule, $\text{C}_{13}\text{H}_{11}\text{ClN}_2\text{O}$, the non-H atoms are approximately coplanar (r.m.s. deviation = 0.115 Å) and the two benzene rings are twisted by 9.36 (3)° with respect to each other. The hydroxy group is hydrogen bonded to the azomethine N atom. In the crystal, an $\text{N}-\text{H}\cdots\pi$ interaction is observed between the imino group and the hydroxybenzene ring of an adjacent molecule.

Related literature

For the synthesis of the compound, see: Auwers (1909).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{11}\text{ClN}_2\text{O}$ $M_r = 246.69$ Orthorhombic, $Pbca$ $a = 10.7590$ (1) Å $b = 7.3189$ (1) Å $c = 28.9222$ (3) Å $V = 2277.45$ (4) Å³ $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 0.32$ mm⁻¹

$T = 100$ K
 $0.25 \times 0.25 \times 0.15$ mm

Data collection

Bruker SMART APEX
diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.925$, $T_{\max} = 0.954$

20107 measured reflections
2614 independent reflections
2326 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.089$
 $S = 1.04$
2614 reflections
162 parameters

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C1–C6 benzene ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1–H1 \cdots N1	0.84 (2)	1.88 (2)	2.6382 (13)	149.8 (19)
N2–H2 \cdots Cg1 ⁱ	0.859 (18)	2.73 (2)	3.3675 (12)	132.3 (17)

Symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the University of Malaya (grant No. RG020/09AFR) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5209).

References

- Auwers, K. (1909). *Justus Liebigs Ann. Chem.* **365**, 314–342.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2011). E67, o1452 [doi:10.1107/S1600536811017958]

2-[[2-(4-Chlorophenyl)hydrazinylidene]methyl]phenol

Kong Mun Lo and Seik Weng Ng

S1. Comment

There is an enormous amount of literature on Schiff bases, which are synthesized by reaction of a primary amine with a carbonyl function. The synthesis of the title hydrazone (Scheme I) was reported a century ago (Auwers, 1909). The $C_{13}H_{11}ClN_2O$ molecule (Scheme I) is twisted along the $-CH=N-NH-$ portion that connects the two aromatic rings. The non-hydrogen atoms approximately lie on plane (r.m.s. deviation 0.115 Å), the rings being twisted by $9.36(3)^\circ$. The hydroxy group is hydrogen-bond donor to the azomethine N atom (Fig. 1). The amino H atom is involved in the $N-H\cdots\pi$ interaction in the crystal structure (Table 1).

S2. Experimental

Salicylaldehyde (1 ml, 10 mmol) and 4-chlorophenylhydrazine hydrochloride (1.8 g, 10 mmol) were dissolved in ethanol (100 ml). No HCl-abstracting reagent was added. The solution was heated for an hour. Slow evaporation of the solvent gave colorless crystals of the Schiff base.

S3. Refinement

Carbon-bound H atoms were placed in calculated positions ($C-H$ 0.95 Å) and were included in the refinement in the riding model approximation, with $U(H)$ set to 1.2 times $U_{eq}(C)$. The amino and hydroxy H atoms were located in a difference Fourier map, and were freely refined.

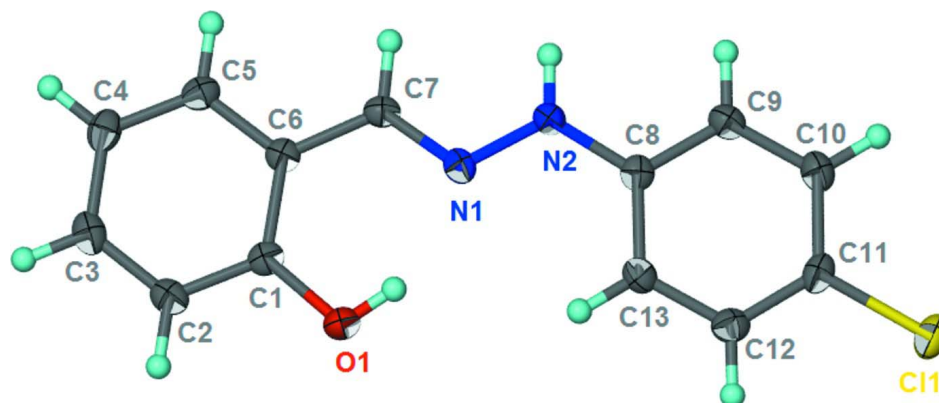


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{13}H_{11}ClN_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

2-[[2-(4-Chlorophenyl)hydrazinylidene]methyl]phenol

Crystal data

C₁₃H₁₁ClN₂O $M_r = 246.69$ Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

 $a = 10.7590$ (1) Å $b = 7.3189$ (1) Å $c = 28.9222$ (3) Å $V = 2277.45$ (4) Å³ $Z = 8$ $F(000) = 1024$ $D_x = 1.439$ Mg m⁻³Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 7363 reflections

 $\theta = 2.4$ – 28.3° $\mu = 0.32$ mm⁻¹ $T = 100$ K

Block, colorless

 $0.25 \times 0.25 \times 0.15$ mm

Data collection

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.925$, $T_{\max} = 0.954$

20107 measured reflections

2614 independent reflections

2326 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.033$ $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 2.4^\circ$ $h = -13 \rightarrow 13$ $k = -9 \rightarrow 9$ $l = -37 \rightarrow 37$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.031$ $wR(F^2) = 0.089$ $S = 1.04$

2614 reflections

162 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 atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.9769P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.37$ e Å⁻³ $\Delta\rho_{\min} = -0.19$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.72028 (3)	0.56809 (4)	0.484943 (10)	0.02413 (12)
O1	0.75260 (9)	0.62244 (14)	0.75810 (3)	0.0202 (2)
N1	0.57723 (9)	0.76810 (14)	0.70608 (3)	0.0160 (2)
N2	0.52407 (10)	0.81449 (15)	0.66514 (4)	0.0192 (2)
C1	0.68256 (11)	0.67666 (16)	0.79476 (4)	0.0158 (2)
C2	0.72583 (11)	0.63711 (18)	0.83900 (4)	0.0183 (3)
H2A	0.8022	0.5739	0.8429	0.022*
C3	0.65750 (12)	0.68996 (16)	0.87755 (4)	0.0197 (3)

H3	0.6883	0.6646	0.9077	0.024*
C4	0.54397 (12)	0.77994 (17)	0.87237 (4)	0.0198 (3)
H4	0.4971	0.8152	0.8987	0.024*
C5	0.50049 (12)	0.81715 (16)	0.82826 (4)	0.0174 (2)
H5	0.4230	0.8778	0.8247	0.021*
C6	0.56790 (11)	0.76755 (16)	0.78874 (4)	0.0150 (2)
C7	0.51856 (11)	0.81180 (16)	0.74331 (4)	0.0157 (2)
H7	0.4414	0.8743	0.7410	0.019*
C8	0.57272 (11)	0.75285 (16)	0.62353 (4)	0.0157 (2)
C9	0.50203 (11)	0.77689 (17)	0.58344 (4)	0.0183 (3)
H9	0.4223	0.8322	0.5854	0.022*
C10	0.54686 (12)	0.72102 (17)	0.54082 (4)	0.0193 (3)
H10	0.4986	0.7382	0.5137	0.023*
C11	0.66308 (12)	0.63963 (17)	0.53830 (4)	0.0180 (2)
C12	0.73438 (12)	0.61342 (17)	0.57764 (4)	0.0182 (3)
H12	0.8137	0.5569	0.5754	0.022*
C13	0.68960 (11)	0.66998 (16)	0.62040 (4)	0.0171 (2)
H13	0.7383	0.6524	0.6474	0.020*
H1	0.7170 (18)	0.662 (3)	0.7343 (7)	0.047 (6)*
H2	0.4532 (17)	0.868 (3)	0.6662 (6)	0.032 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0330 (2)	0.02229 (18)	0.01713 (17)	0.00084 (13)	0.00504 (12)	-0.00208 (11)
O1	0.0173 (4)	0.0242 (5)	0.0191 (5)	0.0049 (4)	0.0013 (4)	-0.0008 (4)
N1	0.0172 (5)	0.0153 (5)	0.0154 (5)	-0.0010 (4)	-0.0023 (4)	0.0006 (4)
N2	0.0184 (5)	0.0240 (6)	0.0152 (5)	0.0063 (4)	-0.0014 (4)	0.0001 (4)
C1	0.0158 (6)	0.0126 (5)	0.0189 (6)	-0.0020 (4)	0.0007 (4)	-0.0014 (4)
C2	0.0186 (6)	0.0145 (6)	0.0218 (6)	-0.0009 (5)	-0.0036 (5)	0.0005 (5)
C3	0.0260 (6)	0.0158 (6)	0.0171 (6)	-0.0041 (5)	-0.0036 (5)	0.0014 (4)
C4	0.0250 (6)	0.0177 (6)	0.0166 (6)	-0.0040 (5)	0.0034 (5)	-0.0009 (5)
C5	0.0172 (6)	0.0143 (5)	0.0208 (6)	-0.0004 (5)	0.0026 (5)	-0.0005 (4)
C6	0.0155 (5)	0.0121 (5)	0.0174 (6)	-0.0022 (4)	-0.0004 (4)	0.0000 (4)
C7	0.0141 (6)	0.0133 (5)	0.0195 (6)	-0.0001 (4)	0.0002 (4)	0.0005 (4)
C8	0.0170 (6)	0.0139 (5)	0.0162 (6)	-0.0025 (4)	0.0007 (4)	0.0012 (4)
C9	0.0166 (6)	0.0185 (6)	0.0199 (6)	0.0001 (5)	-0.0021 (5)	0.0012 (5)
C10	0.0217 (6)	0.0194 (6)	0.0169 (6)	-0.0022 (5)	-0.0033 (5)	0.0010 (5)
C11	0.0241 (6)	0.0152 (5)	0.0147 (5)	-0.0024 (5)	0.0038 (5)	-0.0010 (4)
C12	0.0185 (6)	0.0153 (6)	0.0210 (6)	0.0008 (5)	0.0024 (5)	0.0024 (5)
C13	0.0175 (6)	0.0167 (6)	0.0171 (6)	-0.0006 (5)	-0.0016 (4)	0.0022 (4)

Geometric parameters (Å, °)

C11—C11	1.7418 (12)	C5—C6	1.4014 (16)
O1—C1	1.3600 (15)	C5—H5	0.9500
O1—H1	0.84 (2)	C6—C7	1.4539 (16)
N1—C7	1.2883 (15)	C7—H7	0.9500

N1—N2	1.3580 (14)	C8—C9	1.3978 (16)
N2—C8	1.3878 (15)	C8—C13	1.3991 (17)
N2—H2	0.859 (18)	C9—C10	1.3855 (17)
C1—C2	1.3919 (17)	C9—H9	0.9500
C1—C6	1.4123 (17)	C10—C11	1.3870 (18)
C2—C3	1.3905 (18)	C10—H10	0.9500
C2—H2A	0.9500	C11—C12	1.3857 (18)
C3—C4	1.3958 (19)	C12—C13	1.3901 (17)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.3859 (17)	C13—H13	0.9500
C4—H4	0.9500		
C1—O1—H1	106.7 (14)	C1—C6—C7	122.36 (11)
C7—N1—N2	117.40 (10)	N1—C7—C6	121.41 (11)
N1—N2—C8	121.06 (10)	N1—C7—H7	119.3
N1—N2—H2	117.2 (11)	C6—C7—H7	119.3
C8—N2—H2	121.0 (11)	N2—C8—C9	118.24 (11)
O1—C1—C2	118.08 (11)	N2—C8—C13	122.42 (11)
O1—C1—C6	121.69 (11)	C9—C8—C13	119.33 (11)
C2—C1—C6	120.22 (11)	C10—C9—C8	120.76 (11)
C1—C2—C3	120.17 (11)	C10—C9—H9	119.6
C1—C2—H2A	119.9	C8—C9—H9	119.6
C3—C2—H2A	119.9	C9—C10—C11	119.17 (11)
C2—C3—C4	120.51 (11)	C9—C10—H10	120.4
C2—C3—H3	119.7	C11—C10—H10	120.4
C4—C3—H3	119.7	C10—C11—C12	121.02 (11)
C5—C4—C3	119.15 (11)	C10—C11—C11	119.62 (9)
C5—C4—H4	120.4	C12—C11—C11	119.36 (10)
C3—C4—H4	120.4	C11—C12—C13	119.83 (12)
C4—C5—C6	121.68 (11)	C11—C12—H12	120.1
C4—C5—H5	119.2	C13—C12—H12	120.1
C6—C5—H5	119.2	C12—C13—C8	119.88 (11)
C5—C6—C1	118.27 (11)	C12—C13—H13	120.1
C5—C6—C7	119.37 (11)	C8—C13—H13	120.1
C7—N1—N2—C8	-171.80 (11)	C1—C6—C7—N1	0.68 (18)
O1—C1—C2—C3	179.94 (11)	N1—N2—C8—C9	168.57 (11)
C6—C1—C2—C3	-1.21 (19)	N1—N2—C8—C13	-12.10 (18)
C1—C2—C3—C4	1.21 (19)	N2—C8—C9—C10	178.83 (12)
C2—C3—C4—C5	-0.44 (18)	C13—C8—C9—C10	-0.52 (18)
C3—C4—C5—C6	-0.32 (18)	C8—C9—C10—C11	0.32 (19)
C4—C5—C6—C1	0.31 (18)	C9—C10—C11—C12	0.10 (19)
C4—C5—C6—C7	-179.31 (11)	C9—C10—C11—C11	179.78 (10)
O1—C1—C6—C5	179.26 (11)	C10—C11—C12—C13	-0.30 (19)
C2—C1—C6—C5	0.46 (18)	C11—C11—C12—C13	-179.99 (10)
O1—C1—C6—C7	-1.13 (18)	C11—C12—C13—C8	0.10 (18)
C2—C1—C6—C7	-179.93 (11)	N2—C8—C13—C12	-179.01 (11)
N2—N1—C7—C6	179.85 (10)	C9—C8—C13—C12	0.31 (18)

C5—C6—C7—N1 -179.72 (11)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C1—C6 benzene ring.

<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	0.84 (2)	1.88 (2)	2.6382 (13)	149.8 (19)
N2—H2...Cg1 ⁱ	0.859 (18)	2.73 (2)	3.3675 (12)	132.3 (17)

Symmetry code: (i) $-x+1, y+1/2, -z+3/2$.