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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 = 0.042
 wR factor = 0.132
Data-to-parameter ratio = 10.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*N,N'*-Bis(5-chlorosalicylidene)ethane-
1,2-diamine

The title compound, $\text{C}_{16}\text{H}_{14}\text{Cl}_2\text{N}_2\text{O}_2$, crystallizes in a centrosymmetric space group with one-half molecule in the asymmetric unit. The salicylideneimine moiety is almost planar. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond is observed [$\text{O}-\text{H}\cdots\text{N} = 2.611(3)$ Å]. The packing of the molecules in the crystal structure is stabilized by $\pi-\pi$ stacking interactions between salicylideneimine moieties.

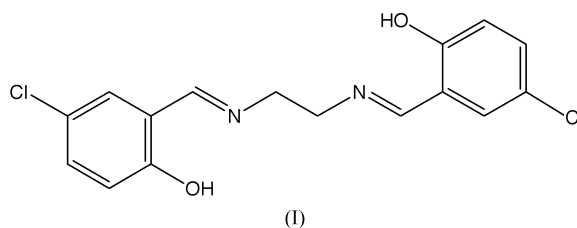
Received 29 June 2004

Accepted 13 July 2004

Online 24 July 2004

Comment

Salicylidene compounds exhibit photochromism in the solid state by intramolecular proton transfer from the hydroxyl O atom to the imino N atom (Zi \acute{o} łek *et al.*, 2003; Zhao *et al.*, 2001). We have been studying the optical and electrical properties of supramolecular compounds constructed by intermolecular hydrogen bonds, $\pi-\pi$ interactions and halogen-hydrogen contacts. Very recently, the title compound, (I), in the crystalline state, has given interesting photo-acoustic and photoluminescence spectra (Kushida *et al.*, 2004). This paper describes the crystal and molecular structure of (I).



The molecule crystallizes in the space group $P2_1/n$, lying on a center of inversion. The bond lengths and angles observed in the salicylideneimine moiety are all in the normal ranges and comparable with those of other related compounds (Elerman *et al.*, 1998; Kabak, 2003). The salicylideneimine moiety is almost planar, with a maximum deviation of 0.0120(2) Å for atom C3. The torsion angles $\text{C}8-\text{N}1-\text{C}7-\text{C}6$ and $\text{C}7-\text{N}1-$

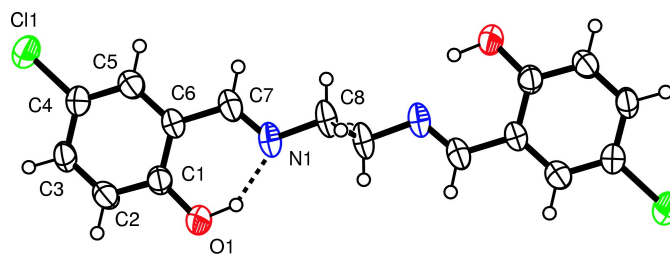


Figure 1

View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

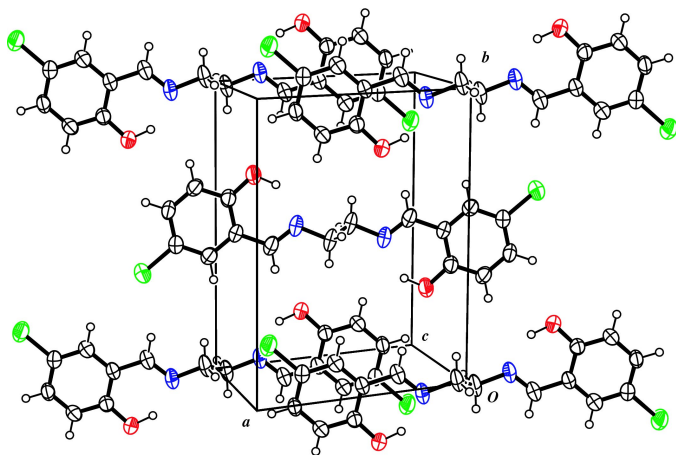


Figure 2
The packing of the molecules of (I).

C8—C8ⁱ are $-179.4(2)$ and $-125.2(3)^\circ$, respectively [symmetry code: (i) $-x, -y, -z$]. The intramolecular hydrogen bond distance of O1—H1 \cdots N1 is $2.611(3)$ Å.

In the crystal structure, the shortest intermolecular C \cdots C distance is $3.477(4)$ Å for C3 \cdots C7ⁱⁱ [symmetry code: (ii) $1-x, -y, 1-z$]. The packing of the molecules in the crystal structure is stabilized by π - π stacking interactions between salicylideneimine moieties.

Experimental

Compound (I) was prepared from 5-chlorosalicylaldehyde and 1,2-diaminoethane. A solution of 1,2-diaminoethane (5 mmol) in methanol (30 ml) was added to a stirred hot solution of 5-chlorosalicylaldehyde (10 mmol), dissolved in methanol (100 ml). The mixture was refluxed for 30 min and then cooled to room temperature, giving yellow crystals suitable for X-ray analysis.

Crystal data

C ₁₆ H ₁₄ Cl ₂ N ₂ O ₂	$D_x = 1.451$ Mg m ⁻³
$M_r = 337.19$	Mo K α radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
$a = 9.379(2)$ Å	$\theta = 15.1$ – 17.1°
$b = 11.655(3)$ Å	$\mu = 0.43$ mm ⁻¹
$c = 7.322(3)$ Å	$T = 298.1$ K
$\beta = 105.31(3)^\circ$	Prism, yellow
$V = 772.0(4)$ Å ³	$0.30 \times 0.20 \times 0.20$ mm
$Z = 2$	

Data collection

Rigaku AFC-7R diffractometer	$h = -6 \rightarrow 12$
ω - 2θ scans	$k = 0 \rightarrow 15$
4540 measured reflections	$l = -9 \rightarrow 9$
1776 independent reflections	3 standard reflections
1072 reflections with $F^2 > 2\sigma(F^2)$	every 150 reflections
$R_{\text{int}} = 0.026$	intensity decay: 0.8%
$\theta_{\text{max}} = 27.5^\circ$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[0.0031F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.98$	$\Delta\rho_{\text{max}} = 0.33$ e Å ⁻³
1069 reflections	$\Delta\rho_{\text{min}} = -0.28$ e Å ⁻³
107 parameters	

Table 1

Selected geometric parameters (Å, °).

C11—C4	1.746 (3)	N1—C7	1.259 (4)
O1—C1	1.351 (3)	N1—C8	1.460 (3)
C7—N1—C8	119.3 (3)	N1—C7—C6	122.4 (3)
C2—C1—O1	119.3 (2)	N1—C8—C8 ⁱ	110.2 (2)
C6—C1—O1	121.1 (2)	C8—N1—C7—C6	$-179.4(2)$
C8—N1—C7—C6	$-179.4(2)$	C1—C6—C7—N1	$-0.7(4)$
O1—C1—C6—C7	0.7 (4)	C7—N1—C8—C8 ⁱ	$-125.2(3)$

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

Table 2

Contact distances (Å).

C1 \cdots C4 ⁱⁱ	3.631 (4)	C4 \cdots C6 ⁱⁱ	3.574 (4)
C1 \cdots C5 ⁱⁱ	3.500 (4)	C4 \cdots C7 ⁱⁱ	3.727 (4)
C2 \cdots C5 ⁱⁱ	3.626 (4)	C5 \cdots C6 ⁱⁱ	3.720 (4)
C3 \cdots C6 ⁱⁱ	3.615 (4)	C6 \cdots C6 ⁱⁱ	3.910 (3)
C3 \cdots C7 ⁱⁱ	3.477 (4)		

Symmetry code: (ii) $1-x, -y, 1-z$.

Table 3

Hydrogen-bonding geometry (Å, °).

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
O1—H1 \cdots N1	0.97	1.80	2.611 (3)	138

The H atoms of the hydroxy groups were found in a difference Fourier map. The other H atoms were placed at idealized positions, with C—H = 0.95 Å. All H atoms were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *WinAFC* (Rigaku/MS, 2003); cell refinement: *WinAFC*; data reduction: *CrystalStructure* (Rigaku/MS, 2003); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MS, 2003).

This work was partially supported by a Grant-in-Aid (No. 16750061) for Scientific Research from the Japan Society for the Promotion of Science.

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