

Refinement

Refinement on F^2 $R(F) = 0.040$ $wR(F^2) = 0.056$ $S = 1.87$

1482 reflections

118 parameters

All H atoms refined

 $w = 1/\sigma^2(F^2)$ $(\Delta/\sigma)_{\max} < 0.01$ $\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$

Extinction correction:

Zachariasen (1963, 1968)

Extinction coefficient:

 $5.4(8) \times 10^{-7}$

Scattering factors from

Stewart *et al.* (1965) (H)

and Creagh & McAuley

(1992) (C, N, O)

Table 1. Selected geometric parameters (\AA , $^\circ$)

O1—C1	1.247 (2)	C1—C2	1.520 (2)
O2—C1	1.251 (2)	C2—C3	1.522 (2)
N1—C3	1.484 (2)	C2—C4	1.528 (3)
H1O—O3—H2O	114 (1)	O2—C1—C2	117.6 (1)
O1—C1—O2	124.3 (1)	N1—C3—C2	112.3 (1)
O1—C1—C2	118.0 (1)		

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1N...O3 ⁱ	0.96 (2)	1.95 (2)	2.809 (2)	149 (2)
N1—H2N...O1 ⁱⁱ	1.05 (2)	1.73 (2)	2.758 (2)	166 (1)
N1—H3N...O2 ⁱⁱⁱ	0.97 (2)	1.83 (2)	2.799 (2)	171 (1)
O3—H1O...O2 ^{iv}	0.86 (2)	1.90 (2)	2.755 (2)	170 (2)
O3—H2O...O1	0.90 (2)	1.84 (2)	2.739 (2)	175 (2)

Symmetry codes: (i) $x, y, z - 1$; (ii) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

Table 3. First- and basic second-level graph-set descriptors involving hydrogen bonds, designated *a–e* in the order given in Table 2

	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>	<i>e</i>
<i>a</i>	<i>D</i>	$D_2^3(5)$	$D_2^3(5)$	$C_3^2(8)$	$C_3^2(8)$
<i>b</i>		$C(6)$	$C_3^2(6)$	$D_2^1(5)$	$D_2^1(3)$
<i>c</i>			$R_2^2(12)$	$D_2^1(3)$	$D_2^1(5)$
<i>d</i>				<i>D</i>	$C_3^2(6)$
<i>e</i>					<i>D</i>

Scan widths were $(1.50 + 0.35 \tan \theta)^\circ$ in ω , with a background/scan time-ratio of 0.5. The data were corrected for Lorentz and polarization effects. The Laue group assignment, systematic absences and centrosymmetric intensity statistics indicated space group *Pbca* (No. 61); since refinement proceeded well it was adopted. Fourier difference methods were used to locate the initial H-atom positions and the H atoms were then refined isotropically. The refined C—H distances ranged from 0.97 (2) to 1.06 (2) \AA with a mean value 1.01 (3) \AA ; refined N—H and O—H distances are given in Table 2. The maximum effect of extinction is 2.3% of F_o for 200. The maximum positive residual peak is located $\sim 0.3 \text{ \AA}$ from O1 and 1.1 \AA from C1; the maximum negative peak is located $\sim 0.7 \text{ \AA}$ from O3.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1106). Services for accessing these data are described at the back of the journal.

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***N*-(3,5-Dichlorophenyl)naphthalidimine**

AYHAN ELMALI,^a YALÇIN ELERMAN,^a INGRID SVOBODA^b
AND HARTMUT FUESS^b

^a*Department of Physics Engineering, Faculty of Sciences, University of Ankara, 06100 Besevler, Ankara, Turkey, and*
^b*Strukturforschung, Fachbereich Materialwissenschaft, Technische Universität Darmstadt, Petersenstraße 23, D-64287 Darmstadt, Germany. E-mail: elmali@science.ankara.edu.tr*

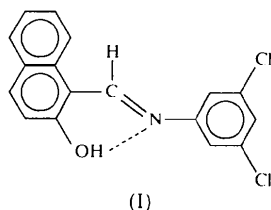
(Received 8 December 1997; accepted 19 January 1998)

Abstract

Molecules of the title compound [1-(3,5-dichlorophenyl)iminomethyl]-2-naphthol, C₁₇H₁₁Cl₂NO] are nearly planar. Each contains a strong intramolecular N...H—O hydrogen bond between the imine and hydroxyl groups [2.570 (3) \AA].

Comment

Although many structures of transition metal complexes with Schiff bases have been determined, a relatively small number of free Schiff bases have been structurally characterized (Calligaris & Randaccio, 1987). In the course of a systematic structural investigation of Schiff bases (Elerman *et al.*, 1991, 1992, 1994, 1995, 1997, 1998; Elmali *et al.*, 1995; Kevran *et al.*, 1996), the structure of the title compound, (I), was determined. Schiff bases are of interest because they have long been known to show photochromism and thermochromism in the solid state; this may involve reversible proton transfer from the hydroxyl O atom to the imine N atom (Cohen *et al.*, 1964; Moustakali *et al.*, 1978; Hadjoudis *et al.*, 1987).



The title molecule is nearly planar; moieties *A* (C1–C6, C11, C12, N1) and *B* (C7–C17, O1) [both planar with a maximum deviation of 0.049 (2) Å] are inclined at an angle of 3.14 (9)°, reflecting mainly the twist about C1–N1 [C6–C1–N1–C7 = 2.5 (4)°].

Two types of intramolecular hydrogen bonds (either N–H···O or N···H–O) can exist in Schiff bases (Garnovskii *et al.*, 1993). The Schiff bases derived from salicylaldehyde always form the N···H–O type of hydrogen bonding regardless of the nature of the N substituent (alkyl or aryl) (Gavranić *et al.*, 1996). In the aldimine compounds derived from 2-hydroxy-1-naphthaldehyde, both types of hydrogen bonds were found (Kaitner & Pavlović, 1996; Elerman *et al.*, 1998). In (I), the N···O distance [2.570 (3) Å] indicates a strong intramolecular hydrogen. The position of the H

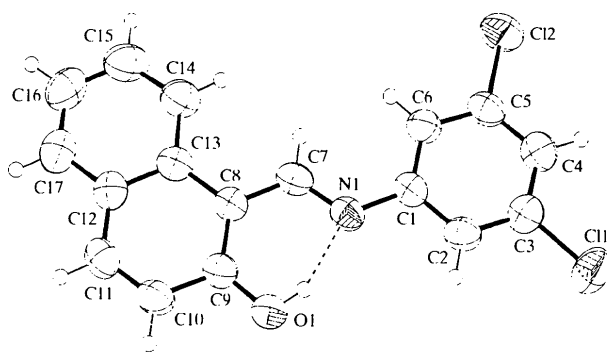


Fig. 1. The molecular structure and atomic labelling scheme of the title compound. Displacement ellipsoids are plotted at the 50% probability level (ORTEP; Johnson, 1976).

atom was deduced from a small peak in the final difference map, 0.83 Å from O1, along with the angle N···H–O1 of 152°, which indicated the presence of an N···H–O hydrogen bond. The bond lengths within the vicinity of the N and O atoms are consistent with this interpretation. The O1···N1 distance is comparable to those observed for 1-[*N*-(4-methyl-2-pyridyl)amino-methylidene]-2(1*H*)-naphthalenone [2.532 (3) Å; Elerman *et al.*, 1998], 4-ethyl-2-(β-hydroxy-α-naphthylmethylidene)amino-5-methyl-3-thiophenecarbonitrile [2.575 (3) Å; Elerman *et al.*, 1997] and *N*-*n*-propyl-2-oxo-1-naphthylidenemethylamine [2.578 (2) Å; Kaitner *et al.*, 1996].

Experimental

The title compound was obtained from the reaction of 2-hydroxynaphthalene-1-carbaldehyde (0.01 mol) with a solution of 2-bromo-4-methylaniline (0.01 mol) in 40 ml of ethanol. The precipitate which separated from the solution was recrystallized for 1–2 d from 1.7 g of Schiff base in 70 ml of acetonitrile solution.

Crystal data

C₁₇H₁₁Cl₂NO
M_r = 316.19
 Orthorhombic
*P*2₁2₁2₁
a = 4.7410 (9) Å
b = 15.293 (5) Å
c = 19.610 (5) Å
V = 1421.8 (6) Å³
Z = 4
D_x = 1.477 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 2.47–12.00°
 μ = 0.453 mm⁻¹
T = 293 (2) K
 Prism
 0.525 × 0.325 × 0.275 mm
 Yellow

Data collection

Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 T_{\min} = 0.818, T_{\max} = 0.900
 2326 measured reflections
 2240 independent reflections

1912 reflections with $I > 2\sigma(I)$
 R_{int} = 0.023
 θ_{\max} = 25.47°
 h = -5 → 3
 k = -18 → 0
 l = -23 → 0
 3 standard reflections
 frequency: 120 min
 intensity decay: 3.7%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.036
 $wR(F^2)$ = 0.103
 S = 1.077
 2240 reflections
 194 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.2183P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.158 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.183 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

C1—N1	1.407 (3)	C7—N1	1.290 (3)
C3—C11	1.738 (3)	C9—O1	1.323 (3)
C5—C12	1.735 (3)		
C2—C1—N1	116.6 (2)	C4—C5—C12	118.3 (2)
C6—C1—N1	124.5 (2)	N1—C7—C8	122.8 (2)
C2—C3—C11	119.9 (2)	O1—C9—C10	116.6 (2)
C4—C3—C11	117.9 (2)	O1—C9—C8	122.5 (3)
C6—C5—C12	119.2 (2)	C7—N1—C1	122.6 (2)

The structure was solved by direct phase determination. It was possible to refine the parameters of the complete structure by full-matrix anisotropic least squares. All phenyl rings were refined without constraints. All H-atom positions (except that of the hydroxyl H atom) were calculated using a riding model and were considered with fixed isotropic *U*'s in all refinements. The H1O atom was located in the difference Fourier maps calculated at the end of the refinement process as a small positive electron density and was not refined.

Data collection: *Diffraction Control Software* (Nonius, 1993). Cell refinement: *Diffraction Control Software*. Data reduction: *REDU4* (Stoe & Cie, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1210). Services for accessing these data are described at the back of the journal.

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Racemic 1-Acetyl-5-phenyl-3-styryl-2-pyrazoline

R. V. KRISHNAKUMAR,^a S. NATARAJAN,^a
V. VIJAYABASKAR^b AND S. PERUMAL^b

^aSchool of Physics, Madurai Kamaraj University, Madurai 625 021, India, and ^bSchool of Chemistry, Madurai Kamaraj University, Madurai 625 021, India. E-mail: sopmku@pronet.xlweb.com

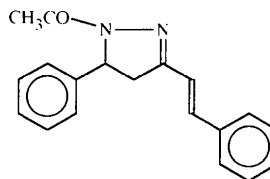
(Received 26 March 1997; accepted 6 January 1998)

Abstract

The asymmetric unit of the racemic title compound, C₁₉H₁₈N₂O, contains two enantiomeric pyrazoline molecules, differing in the configuration at C3. All the rings in the molecules are essentially planar.

Comment

The syntheses of the title compound and its derivatives (Vijayabaskar, 1997) are important as it is known that pyrazolines display various biological properties, such as pesticidal, fungicidal, insecticidal and antiviral activities, and have industrial applications, such as their use as polymer intermediates (Wang *et al.*, 1995; El-Hashash *et al.*, 1995). In order to deduce the configuration (*vis. s-cis* or *s-trans*) of the title pyrazoline, (I), the X-ray structure analysis was undertaken.



(I)