Instrumental Achievements

Crystal Structure of *N,N*-Bis(4-hydroxy-benzylidine)ethylenediamine·methanol

Tuncer HÖKELEK*† and Canan ÜNALEROĞLU**

**Hacettepe University, Department of Physics, 06532 Beytepe-Ankara, Turkey*
***Hacettepe University, Department of Chemistry, 06532 Beytepe-Ankara, Turkey*

(Received July 3, 2000; Accepted February 15, 2001)

Schiff bases have often been used as chelating ligands in the field of coordination chemistry for obtaining thermotropic liquid crystalline polymers. Further, their metal complexes have been used as radiopharmaceuticals for cancer targeting, as dioxygen carriers and as model systems for biological macromolecules.[1] Although a series of Schiff-base complexes have been investigated crystallographically, there are only a very limited number of reports about free Schiff bases in the literature.[2–6]

The title molecule was prepared from a mixture of ethylenediamine (0.123 g, 2.05 mmol) and *p*-hydroxybenzaldehyde (0.526 g, 4.31 mmol) in ethanol (20 ml). The mixture was heated at 333 K for 2 h. The crude product was dissolved in methanol and set aside for crystallization.

The results of an X-ray structure determination are given in Tables 1 – 4.

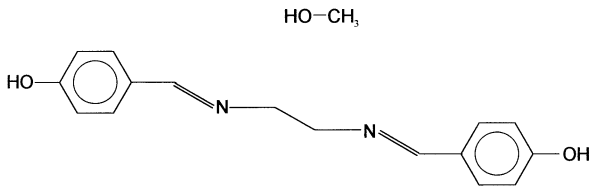

Fig. 1 Chemical diagram.

Table 1 Crystal and experimental data

Formula: $C_{16}H_{16}N_2O_2{\cdot}2CH_3OH$
Formula weight = 332.40
Crystal system: orthorhombic
Space group: *P*bca $Z = 4$
$a = 12.221(1)$Å
$b = 8.086(1)$Å
$c = 18.707(1)$Å
$V = 1848.6(4)$Å^3
$D_x = 1.19$ g/cm^3
μ(Cu K$_\alpha$) = 0.66 mm^{-1}
$T = 293$ K
Colorless
Crystal size: 0.15 × 0.20 × 0.25 mm
λ(Cu K$_\alpha$) = 1.54184 Å
$R = 0.062$ $wR = 0.069$
No. of reflections measured = 1352
No. of reflections used = 515, [$F > 3.0\,\sigma(F)$]
No. of parameters = 109
Goodness-of-fit = 1.01
$(\Delta/\sigma)_{max} = 0.00$
$(\Delta\rho)_{max} = 0.28$ eÅ^{-3}
$(\Delta\rho)_{min} = -0.33$ eÅ^{-3}
$2\theta_{max} = 108.6^\circ$
Measurements: Enraf-Nonius CAD-4 diffractometer
Program system: CAD-4 EXPRESS Software
Structure determination: MolEN
Treatment of hydrogen atoms: geometric calculation
Refinement: full matrix least-squares

† To whom correspondence should be addressed.

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	*x*	*y*	*z*	B_{eq}/Å^2
O1	0.2549(4)	0.2477(7)	0.1109(2)	4.6(1)
O2	0.3286(5)	0.6606(6)	-0.0151(2)	5.3(1)
N1	0.0373(4)	0.1202(7)	0.4188(3)	3.3(1)
C1	0.1747(6)	0.2124(8)	0.2954(3)	3.3(2)
C2	0.2328(5)	0.253(1)	0.2361(3)	3.4(2)
C3	0.1934(6)	0.2067(8)	0.1684(3)	3.2(2)
C4	0.0956(6)	0.1235(9)	0.1642(3)	3.8(2)
C5	0.0380(5)	0.0841(9)	0.2247(3)	3.9(2)
C6	0.0764(5)	0.1282(8)	0.2916(3)	2.9(1)
C7	0.0125(6)	0.0810(9)	0.3553(3)	3.6(2)
C8	-0.0349(5)	0.0597(9)	0.4757(3)	4.0(2)
C9	0.3424(8)	0.505(1)	-0.0399(4)	6.4(2)

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

Table 3 Bond distances (Å) and angles (°)

O1-C3	1.354(7)	C3-C4	1.37(1)
N1-C7	1.267(8)	C4-C5	1.371(9)
N1-C8	1.467(8)	C5-C6	1.384(9)
C1-C2	1.358(9)	C6-C7	1.475(9)
C1-C6	1.382(9)	C9-O2	1.35(1)
C2-C3	1.404(8)		
C7-N1-C8	116.9(5)	C3-C4-C5	120.8(6)
C2-C1-C6	122.1(6)	C4-C5-C6	120.9(6)
C1-C2-C3	119.5(6)	C1-C6-C5	117.9(6)
O1-C3-C2	117.4(6)	C1-C6-C7	123.1(6)
O1-C3-C4	123.8(5)	C5-C6-C7	119.0(6)
C2-C3-C4	118.7(6)	N1-C7-C6	124.4(6)

Table 4 Torsion angles (°)

C8-N1-C7-C6	-178.9(6)	C5-C6-C7-N1	-177.1(7)
C7-N1-C8-C8'	116.7(6)	N1-C8-C8'-N1'	-180.0(4)

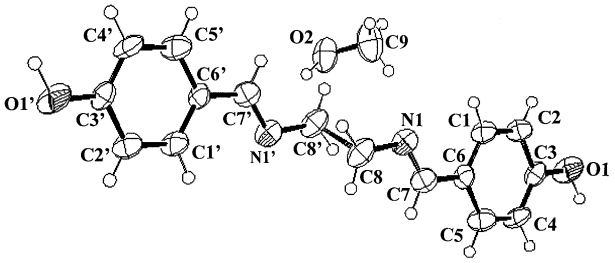

Fig. 2 Molecular structure of the title compound along with the atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

The title molecule (Fig. 2) is crystallographically centrosymmetric and in the anti conformation according to the C=N imine bonds. The asymmetric unit contains one half molecule and a methanol molecule as a solvent. The phenyl ring and the conjugated imine group are slightly distorted from planarity. The C=N imine bonds and the C–N–C bond angles are smaller than the 1.313(8)Å and 122.5(6)˚ values in 2-hydroxy-*N*-*n*-propyl-1-naphthaldimine,[3] which contains hydrogen bonding.

References

1. T. Hökelek, Z. Kılıç, M. Işıklan, and M. Toy, *J. Mol. Struct*., **2000**, *523*, 61.
2. M. Yıldız, Z. Kılıç, and T. Hökelek, *J. Mol. Struct*., **1998**, *441*, 1.
3. T. Hökelek, N. Gündüz, Z. Hayvalı, and Z. Kılıç, *Acta Crystallogr*., **1995**, *C51*, 880.
4. T. Hökelek, N. Gündüz, Z. Hayvalı, and Z. Kılıç, *J. Chem. Crystallogr*., **1995**, *25*, 831.
5. M. Gavranic, B. Kaitner, and E. Mestrovic, *J. Chem. Crystallogr*., **1996**, *26*, 23.
6. H. H. Freedman, *J. Am. Chem. Soc*., **1961**, *83*, 2900.