Instrumental Achievements

Crystal Structure of 1,5-Di[*N*-2-oxyphenyl-salicylidene]-3-oxapentane

Tuncer HÖKELEK,*† Nurcan AKDURAN,* Mustafa YILDIZ, and Zeynel KILIÇ****

**Hacettepe University, Department of Physics, 06532 Beytepe–Ankara, Turkey*
***Ankara University, Department of Chemistry, 06100 Tandoğan–Ankara,Turkey*

(Received October 1, 1999; Accepted February 4, 2000)

2-Hydroxy Schiff base ligands are of interest mainly due to the existence of (O–H···N and O···H–N) type hydrogen bonds and tautomerism between phenol-imine and *keto*-amine forms.[1–3] In these types of ligands, short hydrogen bonds are observed between the 2-hydroxy group and the imine nitrogen atom. In some instances, the hydrogen from the phenol group is completely transferred to the imine nitrogen.[4,5] The hydrogen bond type depends neither on the stereochemistry of the molecule nor on the sort of the substituent to the imine atom, but on the kind of aldehyde used.[5] The title ligand was prepared from a mixture of diethylene glycol bis(2-aminophenyl ether) (1.00 g, 0.0035 mol) and THF (100 mL) solution of salicylaldehyde (0.85 g, 0.007 mol). After the evaporation of THF, the residue was crystallized from $CHCl_3$:hexane (3:1).

The results of X-ray structure determination are given in Tables 1 – 3. The title molecule (Fig. 2) contains short

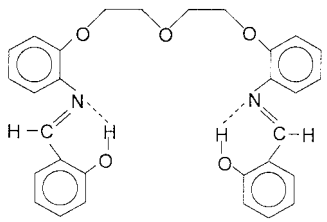

Fig. 1 Chemical diagram.

Table 1 Crystal and experimental data

Formula: $C_{30}H_{28}N_2O_5$
Formula weight = 496.57
Crystal system: monoclinic
Space group: $P2_1$ $Z = 2$
a = 15.336(1)Å
b = 5.735(1)Å
c = 15.679(2)Å
β = 110.91(2)˚
V = 1288.0(3)Å^3
D_x = 1.28 g/cm^3
μ(Cu K$_\alpha$) = 0.67 mm^{-1}
T = 293 K
Orange
Crystal size: 0.20 × 0.25 × 0.30 mm
λ(Cu K$_\alpha$) = 1.54184 Å
R = 0.034 wR = 0.035
No. of reflections measured = 2764
No. of reflections used = 1823
[$F > 3.0\sigma(F)$]
No. of parameters = 342
Goodness-of-fit = 0.98
$(\Delta/\sigma)_{max}$ = 0.02
$(\Delta\rho)_{max}$ = 0.10
$(\Delta\rho)_{min}$ = –0.07
$2\theta_{max}$= 148.7˚
Measurements: Enraf-Nonius CAD-4 diffractometer
Program system: CAD-4 EXPRESS Software
Structure determination: MolEN
Refinement: Full matrix least-squares

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	x	y	z	B_{eq}/Å^2
O1	0.1178(2)	0.3468(5)	0.7000(2)	0.0762(6)
O2	0.3685(1)	0.4921(5)	0.7950(1)	0.0677(6)
O3	0.4001(1)	0.2553(5)	0.9846(1)	0.0715(6)
O4	0.2163(1)	0.1083(5)	0.9830(1)	0.0699(6)
O5	0.3180(2)	0.0627(6)	1.2160(2)	0.0899(7)
N1	0.2172(2)	0.6849(6)	0.6738(2)	0.0605(7)
N2	0.1969(2)	-0.1915(6)	1.1003(2)	0.0539(6)
C1	0.0422(2)	0.4794(7)	0.6561(2)	0.0568(8)
C2	-0.0461(2)	0.3949(7)	0.6465(2)	0.0696(9)
C3	-0.1241(2)	0.5254(8)	0.6015(2)	0.0735(9)
C4	-0.1168(2)	0.7405(8)	0.5659(2)	0.0700(9)
C5	-0.0301(2)	0.8264(7)	0.5755(2)	0.0628(8)
C6	0.0504(2)	0.6988(6)	0.6216(2)	0.0524(7)
C7	0.1410(2)	0.7981(7)	0.6341(2)	0.0592(8)
C8	0.3054(2)	0.7831(7)	0.6843(2)	0.0605(8)
C9	0.3177(2)	0.9727(8)	0.6354(2)	0.0799(9)
C10	0.4054(2)	1.0534(9)	0.6457(2)	0.0875(9)
C11	0.4822(2)	0.9426(9)	0.7046(2)	0.0837(9)
C12	0.4726(2)	0.7555(9)	0.7550(2)	0.0748(9)
C13	0.3843(2)	0.6730(7)	0.7460(2)	0.0612(8)
C14	0.4493(2)	0.3774(8)	0.8579(2)	0.0723(9)
C15	0.4179(2)	0.1862(8)	0.9055(2)	0.0729(9)
C16	0.3207(2)	0.3996(7)	0.9695(2)	0.0697(9)
C17	0.2297(2)	0.2786(7)	0.9211(2)	0.0652(8)
C18	0.1440(2)	-0.0444(7)	0.9485(2)	0.0574(8)
C19	0.0813(2)	-0.0392(7)	0.8586(2)	0.0629(8)
C20	0.0106(2)	-0.2029(8)	0.8309(2)	0.0652(8)
C21	0.0005(2)	-0.3657(8)	0.8894(2)	0.0681(9)
C22	0.0617(2)	-0.3696(7)	0.9799(2)	0.0616(8)
C23	0.1341(2)	-0.2098(7)	1.0097(2)	0.0530(7)
C24	0.2060(2)	-0.3445(7)	1.1622(2)	0.0584(8)
C25	0.2679(2)	-0.3055(7)	1.2550(2)	0.0559(8)
C26	0.2758(2)	-0.4635(9)	1.3229(2)	0.0799(9)
C27	0.3343(2)	-0.429(1)	1.4120(2)	0.0875(9)
C28	0.3874(2)	-0.2314(9)	1.4326(2)	0.0849(9)
C29	0.3817(3)	-0.0687(9)	1.3680(2)	0.0849(9)
C30	0.3221(2)	-0.1014(7)	1.2778(2)	0.0663(9)

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

† To whom correspondence should be addressed.

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

O2-C13	1.364(5)	O5-C30	1.335(5)
O2-C14	1.438(4)	N1-C7	1.288(4)
O3-C15	1.417(5)	N1-C8	1.421(4)
O3-C16	1.420(4)	N2-C23	1.408(3)
O4-C17	1.442(5)	N2-C24	1.277(4)
O4-C18	1.364(4)	C1-O1	1.349(4)
C13-O2-C14	116.8(3)	C7-N1-C8	121.3(3)
C15-O3-C16	116.0(2)	C23-N2-C24	124.3(3)
C17-O4-C18	117.3(2)		

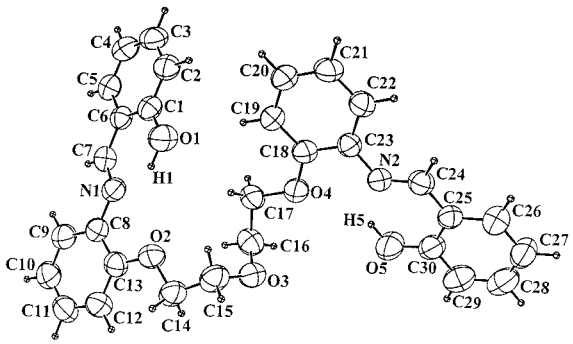

Fig. 2 Molecular structure of the title compound with atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

intramolecular O–H···N hydrogen bonds [O1–H1 0.864(4), H1···N1 1.865(3), N1···O1 2.587(4) and O5–H5 1.056(3), H5···N2 1.603(4), N2···O5 2.542(4)Å], which means that the compound is in phenol-imine form as in 1,8-di[*N*-2-oxyphenyl-salicylidene]-3,6-dioxaoctane[1] [O–H 1.154(3), H···N 1.488(3), O···N 2.578(3)Å]. The C=N imine bonds and C–N–C bond angles can be compared with 1.270(3)Å and 123.5(2)° values in 1,8-di[*N*-2-oxyphenyl-salicylidene]-3,6-dioxaoctane.[1] The H atoms were calculated geometrically, 0.95 Å from the corresponding atoms and refined using a riding model, while the H1 and H5 positions were found from difference synthesis and were refined isotropically.

References

1. M. Yıldız, Z. Kılıç, and T. Hökelek, *J. Mol. Struct.*, **1998**, *441*, 1.
2. J. Costamagna, J. Vargas, R. Latorre, A. Alvarado, and G. Mena, Coord. *Chem. Rev*., **1992**, *119*, 67.
3. S. R. Salman, S. H. Shawkat, and G. M. Al-Obaidi, *Can. J. Spectrosc*., **1990**, *35*, 25.
4. M. Gavranic, B. Kaitner, and E. Mestrovic, *J. Chem. Cryst*., **1996**, *26*, 23.
5. B. Kaitner and G. Pavlovic, *Acta Crystallogr*., **1996**, *C52*, 2573.