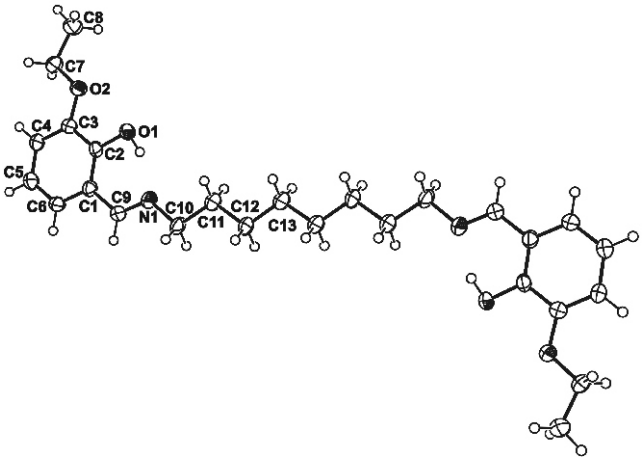
Crystal structure of *N*,*N*′-bis(3-ethoxy-2-hydroxybenzylidene)-octane-1,8-diamine, $C_{26}H_{36}N_2O_4$

Kwang Ha*

Chonnam National University, School of Applied Chemical Engineering, Research Institute of Catalysis, Gwangju 500-757, Republic of Korea

Received March 3, 2011, accepted and available on-line May 20, 2011; CCDC no. 1267/3422



Abstract

$C_{26}H_{36}N_2O_4$, triclinic, $P\bar{1}$ (no. 2), a = 6.905(1) Å, b = 6.910(1) Å, c = 12.981(2) Å, α = 90.123(4)°, β = 101.625(4)°, γ = 101.530(4)°, V = 593.8 Å^3, Z = 1, $R_{gt}(F)$ = 0.059, $wR_{ref}(F^2)$ = 0.176, T = 200 K.

Source of material

1,8-Diaminooctane (1.0098 g, 7.000 mmol) and 3-ethoxy-salicylaldehyde (2.3266 g, 14.001 mmol) in EtOH (20 ml) were stirred for 5 h at room temperature. After addition of pentane (30 ml) to the reaction mixture, the formed precipitate was separated by filtration, washed with ether, and dried at 50 °C, to give a yellow powder (2.7674 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3CN solution.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.95 Å (CH), 0.99 Å (CH_2) or 0.98 Å (CH_3) and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}). The hydroxy H atom was located from Fourier difference maps and refined isotropically: d(O—H) = 0.89(3) Å. The highest peak (0.38 e·Å^{-3}) and the deepest hole (–0.23 e·Å^{-3}) in the difference Fourier map are located 1.29 Å and 0.91 Å from the atoms O1 and C1, respectively.

Discussion

The title compound is a tetradentate Schiff-base, which can act as a dibasic ligand, i.e. the N and O donor atoms can coordinate one or two metal ions. The compound crystallizes in the same space group as the analogous compounds with propylene chain ($C_{21}H_{26}N_2O_4$) [1] and butylene chain ($C_{22}H_{28}N_2O_4$) [2], whereas the analogous Schiff-base with ethylene group ($C_{20}H_{24}N_2O_4$) crystallizes in the monoclinic space group $C2/c$ [3]. A center of inversion is located at the centroid of the title molecule, and therefore the asymmetric unit contains one half of the molecule, and the two benzene rings are exactly parallel. In the crystal structure, the ethoxy group is located approximately parallel to the carrier benzene ring. The N—C bond lengths and the C–N–C bond angle indicate that the imino N1 atom is sp^2-hybridized with d(N1=C9) = 1.270(3) Å, d(N1—C10) = 1.468(3) Å and ∠C9–N1–C10 = 121.1(2)°. The Schiff-base displays strong intramolecular O–H···N hydrogen bonding between the hydroxy O atom and the imino N atom with d(O1···N1) = 2.575(3) Å thus forming a nearly planar six-membered ring. There are also weak intermolecular C–H···O hydrogen bonds with d(C···O) = 3.235(3) Å. The torsion angles for the four atoms within the diiminooctylene chain indicate that the chain is almost perfectly in the anti conformation with ∠N1–C10–C11–C12 = 179.6(2)°, ∠C10–C11–C12–C13 = –179.8(2)° and ∠C11–C12–C13–C13^{i} = 178.5(3)° (symmetry code i: 1–x,2–y,–z).

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.14 × 0.24 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.83 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3645, 2303
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1514
$N(param)_{refined}$:	150
Programs:	SHELXS-97, SHELXL-97 [4], ORTEP-3 [5], PLATON [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.891(5)	0.315(5)	0.239(3)	0.07(1)
H(4)	2*i*	1.2818	–0.1797	0.4069	0.041
H(5)	2*i*	1.5514	0.0040	0.3413	0.045
H(6)	2*i*	1.5075	0.2784	0.2437	0.041
H(7A)	2*i*	1.0172	–0.2662	0.5007	0.043
H(7B)	2*i*	0.9641	–0.3849	0.3886	0.043
H(8A)	2*i*	0.6702	–0.2843	0.4945	0.068
H(8B)	2*i*	0.7100	–0.5031	0.4836	0.068
H(8C)	2*i*	0.6153	–0.3977	0.3815	0.068
H(9)	2*i*	1.2966	0.5151	0.1666	0.039

* e-mail: hakwang@chonnam.ac.kr

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(10A)	2*i*	1.0304	0.6627	0.0446	0.043
H(10B)	2*i*	1.0894	0.7933	0.1529	0.043
H(11A)	2*i*	0.7477	0.7134	0.1709	0.045
H(11B)	2*i*	0.6893	0.5848	0.0625	0.045

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(12A)	2*i*	0.8302	1.0046	0.0785	0.042
H(12B)	2*i*	0.7711	0.8757	–0.0300	0.042
H(13A)	2*i*	0.4909	0.9135	0.0994	0.045
H(13B)	2*i*	0.4331	0.7898	–0.0105	0.045

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*i*	0.8686(2)	0.2066(3)	0.2756(1)	0.0283(9)	0.042(1)	0.051(1)	0.0161(7)	0.0116(7)	0.0183(8)
O(2)	2*i*	0.9083(2)	–0.1104(2)	0.3800(1)	0.0320(9)	0.0354(9)	0.049(1)	0.0115(7)	0.0129(7)	0.0171(7)
N(1)	2*i*	1.0187(3)	0.4985(3)	0.1734(2)	0.036(1)	0.034(1)	0.039(1)	0.0141(9)	0.0084(9)	0.0111(9)
C(1)	2*i*	1.2130(3)	0.2659(3)	0.2522(2)	0.031(1)	0.035(1)	0.030(1)	0.013(1)	0.0068(9)	0.0050(9)
C(2)	2*i*	1.0519(3)	0.1573(3)	0.2907(2)	0.025(1)	0.034(1)	0.031(1)	0.0115(9)	0.0038(9)	0.0039(9)
C(3)	2*i*	1.0779(3)	–0.0123(3)	0.3479(2)	0.028(1)	0.032(1)	0.032(1)	0.0058(9)	0.008(1)	0.0037(9)
C(4)	2*i*	1.2636(3)	–0.0671(3)	0.3671(2)	0.032(1)	0.034(1)	0.038(1)	0.012(1)	0.005(1)	0.009(1)
C(5)	2*i*	1.4243(4)	0.0423(3)	0.3282(2)	0.031(1)	0.040(1)	0.044(2)	0.013(1)	0.006(1)	0.009(1)
C(6)	2*i*	1.3983(4)	0.2056(3)	0.2709(2)	0.030(1)	0.036(1)	0.038(1)	0.009(1)	0.012(1)	0.009(1)
C(7)	2*i*	0.9198(4)	–0.2918(3)	0.4325(2)	0.040(1)	0.027(1)	0.043(1)	0.009(1)	0.010(1)	0.010(1)
C(8)	2*i*	0.7105(4)	–0.3766(4)	0.4495(2)	0.041(2)	0.036(1)	0.060(2)	0.006(1)	0.014(1)	0.015(1)
C(9)	2*i*	1.1857(3)	0.4415(3)	0.1922(2)	0.030(1)	0.036(1)	0.034(1)	0.008(1)	0.008(1)	0.008(1)
C(10)	2*i*	0.9961(4)	0.6763(3)	0.1143(2)	0.042(1)	0.030(1)	0.037(1)	0.015(1)	0.007(1)	0.012(1)
C(11)	2*i*	0.7806(4)	0.7036(3)	0.1005(2)	0.039(1)	0.033(1)	0.044(2)	0.013(1)	0.007(1)	0.012(1)
C(12)	2*i*	0.7395(3)	0.8853(3)	0.0407(2)	0.038(1)	0.030(1)	0.037(1)	0.014(1)	0.005(1)	0.009(1)
C(13)	2*i*	0.5229(4)	0.9082(3)	0.0286(2)	0.040(1)	0.032(1)	0.044(1)	0.013(1)	0.009(1)	0.012(1)

Acknowledgment. This work was supported by Priority Research Centers Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (grant no. 2010-0029626)

References

1. Ha, K.: Crystal structure of *N*,*N*′-bis(3-ethoxy-2-hydroxybenzylidene)-propane-1,3-diamine, $C_{21}H_{26}N_2O_4$. Z. Kristallogr. NCS **225** (2010) 737-738.
2. Fun, H.-K.; Kia, R.; Kargar, H.; Jamshidvand, A.: *N*,*N*′-Bis(2-hydroxy-3-ethoxybenzylidene)butane-1,4-diamine. Acta Crystallogr. **E65** (2009) 706.
3. Bermejo, M. R.; Fernández, M. I.; Gómez-Fórneas, E.; González-Noya, A.; Maneiro, M.; Pedrido, R.; Rodríguez, M. J.: Self-assmebly of dimeric Mn^{III}-Schiff-base complexes tuned by perchlorate anions. Eur. J. Inorg. Chem. (2007) 3789-3797.
4. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
5. Farrugia, L. J.: ORTEP-3 for Windows - a version of ORTEP-III with a Graphical User Interface (GUI) J. Appl. Crystallogr. **30** (1997) 565.
6. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.