Synthesis and charaterization of two novel salen type symmetrical Schiff base ligands

IRAN SHEIKHSHOAIE

Chemistry Department, Shahid-Bahonar University of Kerman, Kerman (Iran)

(Received: December 27, 2007; Accepted: March 14, 2008)

ABSTRACT

Two novel Schiff base ligands bis-(2-hydroxy-1-napthaldimine)-N-2,2-dimethyl-1, 3-diaminopropane[L₁] and bis-(2-hydroxy-1-napthaldimine)-N-diethylene triamine[L₂] have been prepared by condensation of 2,2-dimethyl-1,3-diaminopropane and diethylentriamine with 1-napthaldehyde. The products have been characterized by C, H, N analysis and some spectroscopic methods.

Key words: Schiff base, Napthaldehyde, Diethlentriamine, 2,2-Dimethy-I,3-diaminopropane.

INTRODUCTION

Schiff bases are considered as a very important class of organic compounds. Azomethine compounds have wide applications in many biological aspects, viz., proteins visual pigments, enzymic aldolization and decarboxylation reactions¹⁻⁴. Also Schiff bases and the relevant transition metal complexes are still found to be of great interest in inorganic chemistry. Some Schiff base and their metal complexes exhibit biological activity as antibiotics, antiviral and antitumor agents⁵. In this paper, we report the synthesis and characterization of two tetra and penta dentate Schiff base compounds L, and L_a (Fig. 1).

EXPERIMENTAL

Materials and methods

All chemicals were commercial reagent grade and used as received from Aldrich, Fluka or Merck companies and all reagents were used without further purification. Fourier transformed infrared (FT-IR) spectra were recorded at room temperature in a KBr disk using a spectrophotometer (Unicam-400). The electronic spectra were recorded on a Beckman DU-7000 UV-vis spectrophotometer in (DMF). Elemental analysis (C, H, and N) data were obtained with an Exeter Analytical CE-440 elemental analyzer. Melting points were taken using



Fig. 1: The structure of L, and L, Schiff base compounds

an electro thermal IA 9100 apparatus in open capillary tubes.

¹H NMR and ¹³C NMR spectra were obtained on Bruker (250MHz.) NMR spectrometer in dimethlsufoxid DMSO-d6 solvent. Proton chemical shifts are reported in parts per million (ppm) relative to an internal Me_4Si standard.

Synthesis of L₁ and L₂ Schiff base compounds

Bis-(2-hydroxy-1-napthaldimine)-N-2,2dimethyl-1,3-diaminopropane[L₁] and bis-(2hydroxy-1-napthaldimine)-N-diethylen triamine [L₂] Schiff base compounds were prepared according to a general procedure reported by Schiff [6]. The L₁ and L₂Schiff bases have been synthesized by adding the ethanolic solution of napthaldehyde (2 mmol) with ethanolic solution of 2,2-dimethyl-1,3-propan diamine (1 mmol) or diethylen tri amine (1 mmol). The reaction mixtures separately were then refluxed for 5-6 h, and the condensation products were filtered, thoroughly washed with some drops of ethanol, recrystallized at room temperature. The purity of the synthesized compounds was monitored by TLC using silica gel.

Comp. $L_{1,}$ Yellow crystals. Yield: 80%, mp. 125°C, Molecular weight: 410g/mol. Anal. Calcd. for L_1 : C, 79.02; H, 6.34; N, 6.82. Found: C, 78.98; H, 6.20; N, 6.73.

 ^{1}H NMR (CDCl_3): ^{1}H NMR(CDCl_3): 5 2.2 (s, 6H, CH_3), 3.5 (q, 4H, CH_2), 6.9 (m, 2H, HC=N), 12.4 (s, 2H, OH), 7.8-8.2 (m, 12H, napthyle).

Comp. L_2 Yellow crystals. Yield: 57%, m.p. 117°C, molecular weight: 411g/mol. Anal. Calcd. for L_1 : C, 75.91; H, 6.08; N, 10.21. Found: C, 75.72; H, 6.10; N, 10.11.

 $^{1}\mathrm{H}$ NMR (CDCl_3): δ 2.25 (m, 1H, NH), 2.76 (m, 8H, CH_2), 8 (s, 2H, HC=N), 7.11-7.22 (m, 12H, naphtyle).

In the UV-Vis spectra of L₁ and L₂ there is an intense band in high-energy region (about 300 and 259 nm) of their spectra of these ligands which are related to $\pi \rightarrow \pi^*$ transitions of naphtyl rings⁷.

RESULTS AND DISCUSSION

Salen type Schiff base compounds L_1 and L_2 can be rapidly prepared by Schiff method. These compounds are air stable. Infrared spectral data of these ligands display a sharp band at 1641-1644 cm⁻¹ that are attributed to v (C=N) stretching frequency. Some quantum chemical calculations study shows above two compounds are as tetra dentate ligand with N (in C=N groups) and O (in hydroxyl groups) active coordination sites.

REFERENCES

- B. Witkop, L.K. Ramachandran, *Metabolism*, 13: 1016 (1964).
- R.A. Morton, G.A.*J. Pitt, Bio*chem. J., **59**: 128 (1955).
- I. Fridovitch, F.H. Westheimer, J. Am. Chem. Soc. 84: 3208 (1962).
- 4. E. Grazi, R.T. Rowley, T. Cheng, O. Tchola,

B.L. Horecker, *Biochem. Biophys.* Res. Commun. **9**: 38 (1962).

- 5. D.R. Williams, *Chem. Rev.*, **72**: 203 (1972).
- 6. H. Schiff, Ann. Chim. (Paris), **131**: 118 (1964).
- A. Bottcher, T. Takeuchi, K.I. Hardcastle, T.J. Meade, H.B. Gray, *Inorg. Chem.*, 36: 2498 (1997).