C:\Aftab1.PM5\31

Material Science Research India Volume 03, No. (1) 85-88 (2005)

PHYSICO-CHEMICAL STUDY OF FUROSEMIDE SALICYLDEHYDE SCHIFF BASE COMPLEX WITH Hg(II). AND Ag(I) METAL IONS

Mamta Bhattacharya, S.A. Iqbal* and Suman Malik

Department of Chemistry, S.V. College, Bairagarh, Bhopal- 462 03 (India)
*Department of Chemistry, Saifia College of Science and Education, Bhopal- 462 001 (India)

(Received, March 31, 2005)

ABSTRACT

In the present paper, we describe the synthesis and characterization of metal complexes of Hg (II) and Ag (I) using Schiff base ligand salicyldehyde-furosemide. Furosemide (FSD) is a diuretic drug and it is used to increase the rate of urination through kidneys. Analytical data and stoichiometry suggest ligand metal ratio as 2:1 for Hg complex and 1 : 1 for Ag complex. The structure assigned to the complexes, synthesized via Schiff base formation is supported by infra-red spectral studies.

INTRODUCTION

Schiff base and their metal have been the subject of intensive research due to their novel structural features and their industrial and biological importance and hence have well been studied in past[1-7]. Survey of Literature reveals some studies on pharmacological activities of metal complexes of Schiff base. We report herein the synthesis and characterization of some bipositive 3d metal ion chelats of Furosemide-Schiff base. In continuation of our previous work on metal complexes of established drugs[8-11] the synthesis and structural studies of Furosemide-Hg and Furosemide-Ag complexes are described below.

EXPERIMENTAL

Pure sample of furosemide (FSD) molecular formula $C_{12}H_{11}N_2O_5S$, molecular wt.330.75, was obtained from Geno Pharmaceuticals Private Limited, Goa. Metal salts were of *Qualigen* chemicals. Solvents used were methyl alcohol and acetone which were of analytical grade.

Equimolar solutions of pure drug (0.01 M) and salicyldehyde (0.01 M) were taken in methanol-water mixture (50:50 ratio). Both the solutions were mixed and refluxed for three hours. The reaction mixture was kept overnight. Light yellow crystals of furosemide- Schiff base were formed in the reaction mixture, which was washed with 50:50 mixture of methanol: water, filtered, dried and weighed. Melting point was recorded.

Ligand-Metal ratio and stoichiometry :

To confirm the ligand metal ratio, conductometric titrations using monovariation method were carried out on Systronics conductometer and dip type electrode. Titrations were carried out at 21°C and 20°C respectively for $HgCl_2$ and $AgNO_3$.

0.01M solution of furosemide-Schiff base was prepared in 60 : 40 mixtures of acetone and water, diluted to 200 ml with same solvent. This Schiff base solution was titrated against 0.02M metal solutions using monovariation method. After making volume corrections the results were plotted in form of a graph which shows the ligand metal ratio for Hg as 2:1 and

for Ag 1 : 1. Formation of 2:1 complex and 1:1 complex was further confirmed by Job's method of continuous variation modified by Turner and Anderson.[12-13] The stability constants and free energy changes were calculated.

Synthesis of complexes :

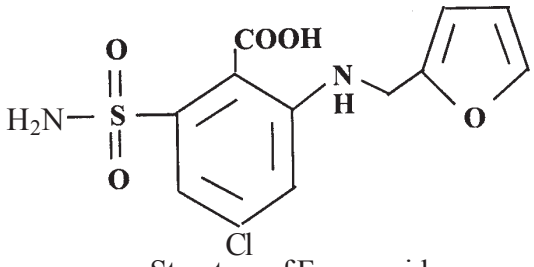
For the synthesis of complexes of furosemide-Hg and furosemide-Ag, 0.006 M ligand solution was prepared in 60:40 mixture of acetone water and refluxed for four hours with 0.003M solution of $HgCl_2$ and 0.006M solution of $AgNO_3$.The refluxed solutions were kept for two days. Solid crystalline compounds appeared in the solutions. Complexes of furosemide-Hg and furosemide-Ag were washed with 60:40 mixture of acetone: water, filtered, dried and weighed. Melting points were recorded.

Table 1. Synthesis and Physico-chemical Characteristics of Complexes

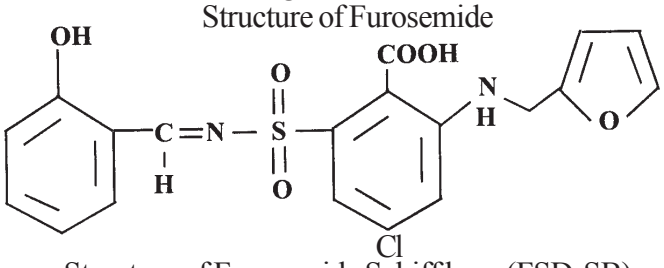
Ligand & Complexes	Ligand/metal ratio	Colour	% Yield	Stabilty Constant logK L/mole	Free energy change Δ F (K.Cal/mol)
FSD-SB	-	Pale yellow crystals	65	-	-
$(FSD)_2Hg$	2:1	Grey crystals	51	11.20	-15.8371
(FSD)Ag	1:1	Pale White crystals	78	11.42	-16.1746

Table 2. Analytical data of Complexes

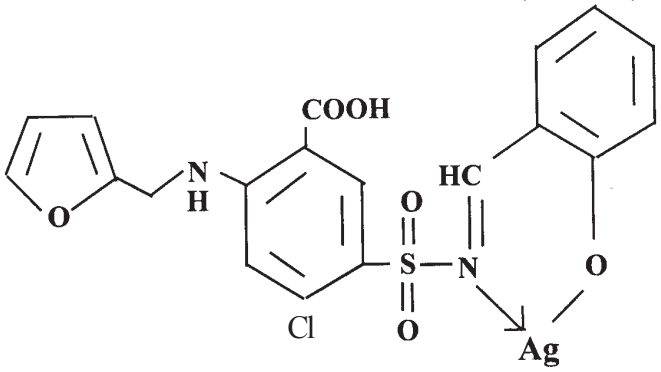
Complex (Mol. Wt.)	Compo-sition	Elemental analysis %		Found (Calculated)			m.p.^{0} C
	C	H	Cl	N	S	Metal	
$(C_{19}H_{14}ClN2O_6S)_2Hg$ (1068)	46.75 (36.00)	2.87 (2.91)	6.64 (2.91)	5.24 (16.67)	6.01 (17.50)	18.78 (8.91)	360
$(C_{11}H_9N_4O_4S_2)Ag$ (975.3)	36.86 (36.89)	2.57 (2.51)	7.27 (2.91)	5.74 (16.17)	6.58 (17.50)	11.06 (8.61)	275



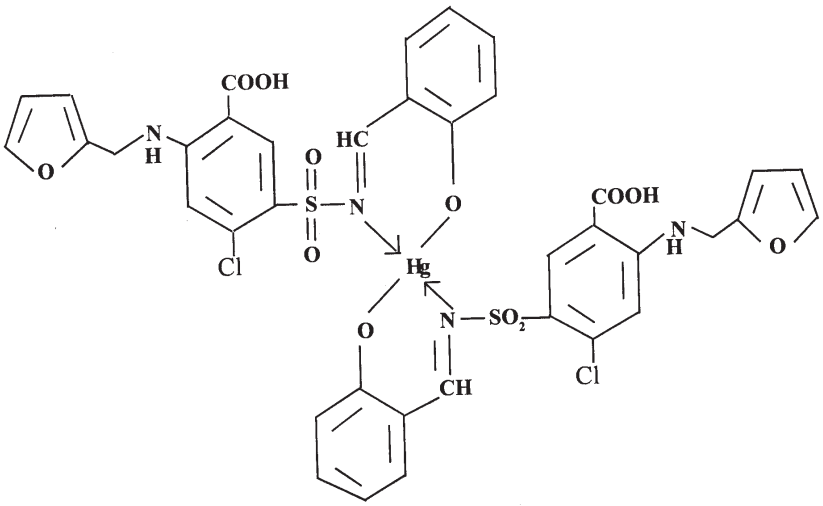
Structure of Furosemide



Structure of Furosemide Schiff base (FSD-SB)



Structure of Furosemide Schiff base silver complex



Structure of Furosemide Schiff base mercury complex

RESULTS AND DISCUSSION

Conductometric studies monovariation method and Job's method of continuous variation modified by Turner and Anderson indicate the formation of 2:1 (L: M) complex of Schiff base of furosemide with Hg and formation of 1:1 (L: M) complex of Schiff base of furosemide with Ag lons.

Analytical data of these complexes are in agreement with the composition $(C_{19}H_{14}C1N2O_6S)_2$ Hg and $(C_{19}H_{14}C1N2O_6S)$ Ag.

Proposed structure was further supported by IR spectral data[14-18]. Bands observed in the IR spectra of FSD-SA-Hg and FSD-SA-Ag at 1172 cm^{-1} and 1163 cm^{-1} are characteristic of SO_2-N group. Absorption band at 1411 cm^{-1} and 1420cm^{-1} shows the presence of chelate ring. Frequency at 664 cm^{-1} and 680cm^{-1} is characteristics of M-O linkage. Bands at 582cm^{-1} and 579 cm^{-1} are because of M-N linkage. Frequencies at 789 cm^{-1} and 880 cm^{-1} indicate the S-N linkage.

The disappearance of frequencies of phenolic -OH in complex supports its involvement in complexation.

All these evidences support the proposed structure of the prepared complexes.

REFERENCES

l. Pardeshi, R. K., Bondage S. P. and N.G. Palaskar, *Asian J. Chem.,* **13**(4), 1513-1517 (2001).

2. Gotmare, R. Sulekha and Mayadev, Acta Cienc. Indica, **I7C,** 245 (1991).

3. Kumar, A.,Singh G., Handa R.N., *Indian J. of Chem.,* **38A,** pp 613-617. June (1999).

4. Mohan, J. Kataria S., *Indian J. of heterocyclic Chem,* **6,** 317 (1997).

5. Qureshi R. and Iqbal S.A., *Asian J. Chem.,* **2(3)**, 321(1990).

6. Tan S.F. and Ang K.P, *Trans. Met. Chem.*, 9, 390 (1984).,13, 64(1988).

7. N.R. Rao and M.C. Ganorkar, *Indian J. Chem.,* **24A,** 877 (1985), 26A, 887 (1987), 27A, 52 (1988).

8. Iqbal S.A. and Qureshi R., *Asian J. Exp. Sci.,* **I,** 68 (1985).

9. Iqbal S.A. and Siddiqui S., Qureshi R.and Desnavi A., *Orient. J. Chem.,* **1,** 32 (1985).

10. Qureshi R. and Iqbal S. A., *Orient. J. Chem.,* **13(3)** (1997).

11. Qureshi R. and Iqbal S.A., *Asian J. Chem.,* **2(3),** 321 (1990).

12. Job P., *Ann Chim.,* **10,** 113,(1928).

13. Turner S.E. and Anderson, R.C., *J. Amer. Chem., Soc.,* **71,** 912, (1949).

14. Vogel I. *Quantitative Inorganic Analysis,* Longman, Green and Co. (London) 455 (1954).

15. Bellamy, L.J., *Chemical Application Of spectroscopy* (Int. Sci. Pub.) New York, (1956).

16. Nakamotto K., *Infrared spectra of Inorg and co-ord. Compounds,* (John Wiley), New York (1963).

17. Weissberger A., *Chem Application of spectroscopy* (Int. Sci. Pub.) New York, (1956).

18. Rao C. N. R., *Chem Application of IR spectroscopy,* Academic Press New York, (1963).