

Studies on dapsone salicyldiamine complex with transition metal ions

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ABSTRACT

Complexes of Fe(II), Co (II) Ag (I) and Hg (II) have been synthesized with the drug Dapsone that is 4-4'diaminodiphenyl sulfone. Conductometric studies and elemental analysis suggest the ML_2 stoichiometry for Fe(II), Co (II) Hg (II) complexes and ML_1 for Ag (I) complex. Molar conductance values indicate the non-electrolytic nature of the complexes. The ligand behaves as bidentate ligand and forms co-ordinate bond through azomethine nitrogen(-C=N-). The structure assigned to the complexes, synthesized via Schiff base formation is supported by infrared spectral studies.

Key words: Schiff base, antibacterial, Dapsone, Infra red and partical size.

INTRODUCTION

Schiff base metal chalets are widely applicable because of their industrial and biological importance and hence have well been studied in past.¹⁻⁴ Survey of literature reveals some studies on pharmacological activities of metal complexes of Schiff base.

We report herein the synthesis and characterization of some transition metal ion chelates of Dapsone -Schiff base. In continuation of our previous work on metal complexes of established drugs⁵⁻¹⁰ The synthesis and structural studies of Dapsone-

Fe(II), Co (II) Hg (II) and Ag (I) complex is described below.

EXPERIMENTAL

Pure sample of Dapsone (DPN) molecular formula $C_{12}H_{12}N_2O_2S$, molecular wt.248.30, was obtained from Bengal Chemicals private limited. Kolkata Metal salts were of Qualigen chemicals.

Solvents used were absolute alcohol is of analytical grade.

Equimolar solutions of pure drug (0.01M) and salicyldehyde (0.01M) were taken in absolute alcohol Both the solutions were mixed and refluxed for $\frac{1}{2}$ an hour . While heating, light yellow crystals of Dapsone- Schiff base were formed in the reaction mixture, which was washed with absolute alcohol, 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.

0.01M solution of Dapsone-Schiff base was prepared in 60% acetone and diluted to 200ml 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 Fe(II), Co (II) Hg (II) and Zn as 2:1. and 1:1 for Ag (I). L:M ratio for all complex was further confirmed by Job's method of continuous Variation, modified by Turner and Anderson. The stability constants and free energy changes were calculated.

Synthesis of complexes

For the synthesis of complexes of Dapsone 0.006 M ligand solution was prepared in 60% acetone and refluxed for four hours with 0.003M solution of metal salts. The refluxed solutions were kept for two days. Solid crystalline compounds appeared in the solutions. Complexes were washed with 60% acetone, filtered, dried and weighed.

Melting points were recorded

Results and discussion-Conductometric studies monovariation method and Job's method of continuous variation modified by Turner and

Anderson¹⁰⁻¹¹ indicate the formation of 2:1 (L: M) complexes of Schiff base of Dapsone with Fe(II), Co (II) Hg (II) complexes and 1:1 for Ag (I) complex. Analytical data of these complexes are in agreement with the composition $(C_{19}H_{15}N_2O_3S)_2 M$.

Proposed structure was further confirmed by IR spectra data¹²⁻¹⁶. Bands observed at 1173 cm^{-1} and 1143 cm^{-1} characteristic of SO_2-N group. Absorption band at 1413 cm^{-1} shows chelate ring. Frequency at 892 cm^{-1} is characteristics of M-O linkage. Bands at 709 cm^{-1} and 715 cm^{-1} are characteristic of M-N linkage. Frequencies at 1362 cm^{-1} and 1212 cm^{-1} indicate the S-N linkage.

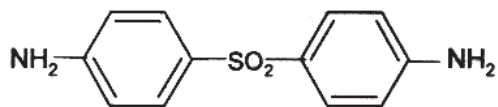
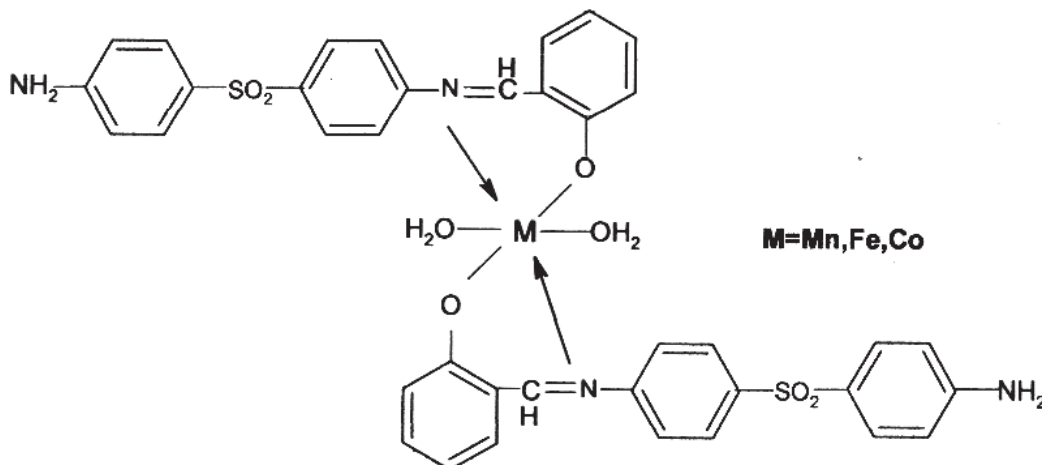
Absorption band at 1004 cm^{-1} and 974 cm^{-1} shows the presence of Sulphur in heterocyclic ring. The disappearance of frequencies of phenolic -OH in complex supports its involvement in complexation.

Table 1: Synthesis and Physiochemical Characteristics of Complexes

Ligand/Complex	Ligand/Metal Ratio	Colour	% Yield	Stability Constant logK L/mole	Free energy change F (K.Cal/mol)
DPN-SB	-	Lt. yellow crystals	65	-	-
$(DPN-SA)_2Fe$	2:1	Brown crystals	32	9.32	-11.456
$(DPN-SA)_2Co$	2:1	Pink crystals	60	11.3263	-15.650
$(DPN-SA)Ag$	1:1	Yellow crystals	49	6.0755	-8.3391
$(DPN-SA)_2Hg$	2:1	Yellow crystals	75	12.9928	-17.8837

Table 2: Analytical data of Complexes

Complex	Elemental analysis Found (Calculated) %					m.p. °C
	C	H	N	S	Metal	
$(C_{19}H_{16}N_2O_3S)$	64.0 (64.77)	5.01 (4.54)	8.00 (7.95)	9.30 (9.09)	-	220
$(C_{11}H_9N_4O_4S_2)_2Fe$	36.98 (36.00)	2.5 (2.91)	15.69 (16.67)	17.93 (17.50)	8.86 (8.91)	200
$(C_{19}H_{15}N_2O_3S)_2Co$	36.98 (36.00)	2.5 (2.91)	15.69 (16.67)	17.93 (17.50)	8.86 (8.91)	300
$(C_{19}H_{15}N_2O_3S)_2Ag$	36.98 (36.00)	2.5 (2.91)	15.69 (16.67)	17.93 (17.50)	8.86 (8.91)	250
$(C_{19}H_{15}N_2O_3S)_2Hg$	36.98 (36.00)	2.5 (2.91)	15.69 (16.67)	17.93 (17.50)	8.86 (8.91)	230

**Dapsone****Dapsone - Salicyldimine Metal complex**

Anti bacterial activity- The ligand Dapsone(DPN) and its Fe(II), Co (II) Ag (I) and Hg (II) metal complexes were screened for antibacterial activity.

The ligand Dapsone(DPN) and its Fe(II), Co (II) Ag (I) and Hg (II) metal complexes were tested against Gram +ve and Gram -ve bacteria . Results reveal that metal complexes are more active than the free ligand.

REFERENCES

- De A.K., Deb D., Nath Bhowmik K.R. and Dutta R.N. *J.Indian Chem Soc*, 6, 76-82, Jan 2009.
- Vishwanathmurthy P., Kavitha R. and Muthukumar M., *J.Indian Chem Soc*, **85**: 735-738 (2008).
- Qureshi R. and Iqbal S.A., *Asian J. Chem.*, **2**(3): 321 (1990)
- S.F.Tan and K.P.Ang, *Trans. Met. Chem.*, **9**: 390 (1984); **13**: 64 (1988).
- N.R.Rao and M.C. Ganorkar, *Indian J.Chem.*, **24A**: 877 (1985); **26A**: 887 (1987); **27A**: 52 (1988).
- Iqbal S.A. and Qureshi R., *Asian J.Exp. Sci.*, **1**: 68 (1985).
- Iqbal S.A. and Siddiqui S., Qureshi R. and Desnavi A., *Oriental J. Chem.*, **1**: 32 (1985).
- Qureshi R. and Iqbal S.A., *Oriental J. Chem* **13**(3) (1997)
- Nadkarni S. *Indian material medica* 2: 51 (1976).
- Job P., *Ann Chim.*, 10: 113 (1928).
- Turner S.E., and Anderson, R.C., *J. Amer. Chem., Soc.*, 71,912, (1949).

12. Vogel I. Quantitative Inorganic Analysis, Longman, Green and Co. (London) 455(1954).
13. Bellamy, L.J., *Chemical Appli. of spectroscopy* (Int.Sci.Pub.) New York,(1956).
14. Nakamoto K., *Infrared spectra of Inorg. and co-ord. Compounds*, (John Wiley), New York,(1963).
15. Weissberger A., *Chem. Appli. of spectroscopy* (Int. Sci. Pub.) New York, (1956).
16. Rao C.N.R., *Chem. Appli. of IR spectroscopy*, Academic Press New York, (1963).