

Synthesis and characterisation of sulphasomidine complexes with copper, silver, zinc, mercury, iron and cobalt

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ABSTRACT

Complexes of sulphasomidine with Cu(II), Ag (I), Zn (II), Hg (II), Fe (II) and Co (II) have been synthesized and characterized using different physico-chemical techniques. The ligand and metal chelates were screened against *Gram positive* and *Gram negative* bacteria. Metal chelates were found to be more potent than the ligand and metals.

Key words : Complexes, sulphasomidine, transition metals.

INTRODUCTION

Sulpha drugs is a group of compounds used for eliminating a wide range of infections in human and other animal systems. Many chemotherapeutically important sulpha drugs like sulphadimidine, sulphamerazine and sulphaacetamide, possess SO₂ NH moiety which is an important toxophoric function¹. Sulphonamides are drugs extensively used for the treatment of certain infections caused by *Gram positive* and *Gram negative* microorganisms². Metal complexes of sulphadugs have been found to be more bacteriostatic than the drug themselves³⁻⁴. In the search for sulphadugs, sulphasomidine (SSD) is proved to be another useful drug. Hence it was proposed to study few transition metals of sulphasomidine. It is chemically N' 2-6 dimethyl 4 pyrimidinyl sulphanilamide. It is an isomer of sulphadimidine or sulphamethazine (SDD). The metals selected for this study are copper, silver, zinc, mercury, iron and cobalt.

EXPERIMENTAL

Pure sample of sulphasomidine with the molecular formula C₁₂H₁₄N₄O₂S was procured from M/s Pure Drugs (India), Bombay.

Metal salts used were all of AR grade. Solvents used were distilled water, DMSO and DMF. Elemental analysis were carried out by using Carlo Erba 1106 Thomas and Coleman Analyzer. Conductivity measurements were carried out with "Digital Conductivity Meter" a dip type conductivity cell. IR spectra of the ligand and its complexes were recorded in KBr pellets on Perkin Elmer Model 283 'B' spectrophotometer. The IR spectra were recorded between 4000-400 cm⁻¹ range. Metals were estimated by using standard methods⁵⁻⁷.

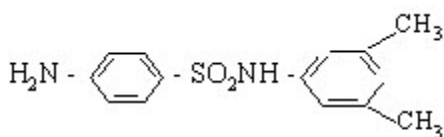
Synthesis of Complexes

2 moles of ligand (sulphasomidine) and 1 mole of metal salt (except Ag, where one mole of ligand and one mole of salt were taken), were dissolved separately in distilled water. Metal solution was gradually added to sulphasomidine with constant stirring. On adding metal solution to the sulphasomidine, the metal complex was precipitated. The Precipitate was filtered, washed many times with distilled water, dried and weighed (yield % are given in the table 1).

RESULTS AND DISCUSSION

The physico-chemical and analytical data of the complexes are summarized in Table 1. The

analytical data corresponds with the general formulae $M(L)$, $M'(L_2)$ and $M''(L_2) \cdot H_2O$ where $M=Ag$, $M' = Cu, Zn, Hg$ and $M'' = Co, Fe$. The complexes are soluble in DMSO. 2:1 (L:M) stoichiometry for Cu, Zn, Hg, Fe and Cu complexes and 1:1 (L:M) stoichiometry for Ag complexes was confirmed by Job's method of continuous variation modified by Turner and Anderson. The molar conductance values of the complexes indicate their non electrolytic nature⁸⁻⁹.



Structure of ligand (Sulphasomidine)

IR Spectra

Absorption spectral data of sulphasomidine and its metal complexes. As far as possible, assignments have been given to various absorption bands. Few important bands are discussed here.

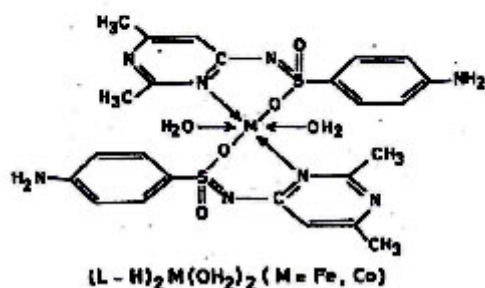
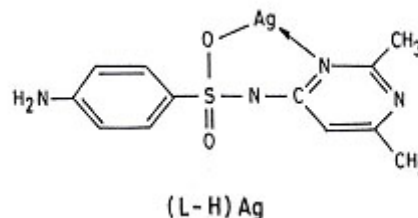
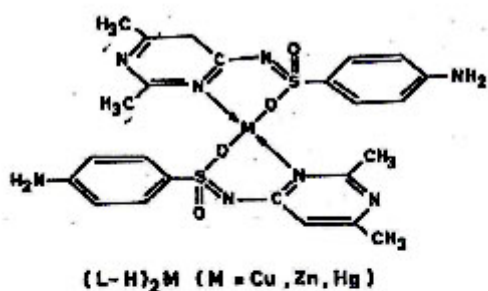
In all the metal complexes, a chelate ring frequency at $660 \pm 5 \text{ cm}^{-1}$ was observed which is in agreement with the results obtained by Gupta and Kaushal¹¹. The metal oxygen frequencies with various metals was observed are Cu-O 670 cm^{-1} , Ag-O 670 cm^{-1} , Zn-O 680 cm^{-1} , Hg-O, 680 cm^{-1} , Co-O 670 cm^{-1} . Similarly M-N frequencies were observed as Cu-N $455 \pm 5 \text{ cm}^{-1}$. Ag-N, $310 \pm 350 \text{ cm}^{-1}$, Zn-N, $440 \pm 5 \text{ cm}^{-1}$, Hg-N, $545 \pm 5 \text{ cm}^{-1}$, Fe-N, $565 \pm 5 \text{ cm}^{-1}$, Co-N $550 \pm 5 \text{ cm}^{-1}$. The SO₂-N frequency is observed at $1280 \pm 5 \text{ cm}^{-1}$. Similarly S-N frequency is observed at $1065 \pm 10 \text{ cm}^{-1}$. The Ar-S frequency is observed in the range $710 \pm 10 \text{ cm}^{-1}$.

Further more the NH-deformations of SO₂ NH are observed at $3080 \pm 40 \text{ cm}^{-1}$ and frequency C=N was observed in the range of $2360 \pm 20 \text{ cm}^{-1}$.

The frequency due to Ar-NH₂ is observed at $1285 \pm 5 \text{ cm}^{-1}$. Broad bands of strong to very strong frequencies were observed in sulphasomidine for Fe-complex $3350 \pm 10 \text{ cm}^{-1}$ and co-complex $3355 \pm 5 \text{ cm}^{-1}$. It has also been observed that M-O and M-N frequencies are absent in the ligand.

Table 1: Elemental analyses, colour, yield, molar conductance and composition of the complexes

S. No.	Complex	Colour	Yield %	Molar Conductance (W ⁻¹ cm ² mol ⁻¹)	Analysis % : Found (Calculated)				m.p. °C	L:M ratio	
					Metal	Carbon	Hydrogen	Nitrogen			Sulphur
1	(C ₁₂ H ₁₄ N ₄ O ₂ S) ₂ Cu	Green	55	13.6	15.63 (15.19)	44.08 (43.91)	4.60 (4.30)	17.01 (17.07)	9.71 (9.77)	198	2:1
2	(C ₁₂ H ₁₄ N ₄ O ₂ S)Ag	White	66	12.1	28.99 (28.38)	36.12 (37.92)	3.93 (3.71)	14.22 (14.74)	8.51 (8.44)	290	1:1
3	(C ₁₂ H ₁₄ N ₄ O ₂ S) ₂ Zn	White	52	11.8	11.18 (10.51)	45.65 (46.35)	4.70 (4.52)	17.69 (18.01)	10.03 (10.31)	272	2:1
4	(C ₁₂ H ₁₄ N ₄ O ₂ S) ₂ Hg	White	71	9.5	26.51 (26.48)	37.50 (38.07)	4.10 (3.73)	14.08 (14.79)	8.09 (8.47)	245	2:1
5	(C ₁₂ H ₁₄ N ₄ O ₂ S) ₂ Fe.2H ₂ O	Brown	48	12.9	25.23 (24.77)	38.09 (39.02)	3.91 (3.82)	14.75 (15.17)	8.71 (8.68)	250	2:1
6	(C ₁₂ H ₁₄ N ₄ O ₂ S) ₂ Co.2H ₂ O	Blue	48	11.4	14.87 (14.64)	42.20 (44.36)	4.65 (4.34)	16.95 (17.24)	9.96 (9.86)	340	2:1



On the basis of the various absorption bands observed for sulphasomidine and its complexes following structures have been assigned for its complexes.

Antibacterial Screening

The ligand and the complexes were analysed for their antimicrobial activity¹⁰⁻¹² *in vitro* against *E. coli* and *B. subtilis* by using Disc plate method¹³. The results obtained are given in Table 2. From Table 2, it is seen that the ligand sulphasomidine is resistant to *E. coli* and markedly sensitive to *Bacillus subtilis*. But complexes like Mercury, Zinc, Silver, Cobalt, Iron and Copper all shows markedly sensitive with *E. coli* and all shows resistance to *Bacillus subtilis*.

Table 2: Antibacterial activity of sulphasomidine (SSD) and their metal complexes, zone of inhibition in mm

S. No.	Compounds	Microorganisms	
		<i>Escherichia coli</i>	<i>Bacillus subtilis</i>
1.	SSD	---	+++
2.	(SSD) ₂ - Hg	+++	-
3.	(SSD) ₂ - Zn	+++	-
4.	SSD - Ag	+++	-
5.	(SSD) ₂ - Co	+++	-
6.	(SSD) ₂ - Fe	+++	-
7.	(SSD) ₂ - Cu	+++	-

(-, +, ++, +++, +++) Resistant
Zone of Inhibition

Weakly Sensitive - 0-0 mm
Moderately Sensitive - 5-10 mm
Markedly Sensitive - 10-15 mm
Highly Sensitive - more than 15 mm

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