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Synthesis and Thermal Study of Some Transition Metal ion Complexes with N¹-(5,6-dimethoxypyrimidin-4-yl) sulphanilamide

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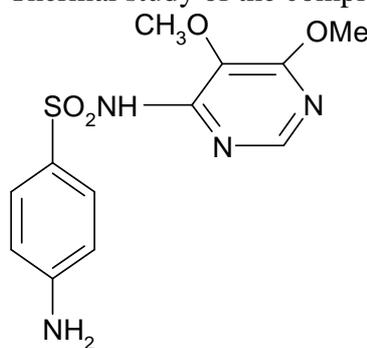
ABSTRACT

This paper reports the synthesis of metal complexes of Fe(II), Co(II) and Cu(II) derived from N¹-(5,6 dimethoxypyrimidin-4-yl) sulphanilamide. All metal complexes were characterized by elemental analysis, analytical and thermal study.

Key words: Synthesis of metal complexes of Fe(II), Co(II) and Cu(II), Thermal study.

INTRODUCTION

N¹-(5,6 dimethoxypyrimidin-4-yl) sulphanilamide is a antibacterial antimalarial drug. The molecular formula is C₁₂H₁₄N₄O₄S. This compound contains primary amine, secondary amine and tertiary nitrogen in the ring. The compound contains -SO₂ and -OCH₃ groups. As this compound contains nitrogen, oxygen and sulphur donor atoms, therefore we have tried to use this compound as a complexing agent to prepare metal complexes of Fe(II), Co(II) and Cu(II)¹⁻⁴. By different trial and error preparative methods the complexes of Fe(II), Co(II), Cu(II) were prepared. The prepared complexes were dried and the physical and chemical parameters were recorded. The elemental analysis and Thermal study of the complexes were studied.



N¹ (5,6-dimethoxypyrimidin-4-yl) sulphanilamide

Preparation of Dichloro bis N¹-(5,6-Dimethoxypyrimidin-4-yl) sulphanilamide Fe (II) complex :0.1M solution of N¹-(5,6-dimethoxypyrimidin-4-yl)-sulphanilamide were prepared in warm ethyl alcohol. 0.1 M Iron chloride (FeCl₂) solution were also prepared in warmed ethyl alcohol. Metal ion solution and ligand solution were mixed in round bottom flask. The pH of reaction mixture were maintained about 6.8 to 7.1 by putting alcoholic ammonia solution drop by drop. The round bottom flask was fixed with vertical water condenser. The reaction mixture was refluxed for three hours by keeping the round bottom flask on steam bath, chrome yellow colour precipitate was obtained. It is filtered through whatmann filter paper. The complex was purified by washing with warm alcohol. The complex is dried and yield were recorded and stored in the glass sample bottle.

Preparation of Dichloro N¹-(5,6-dimethoxypyrimidin-4-yl) sulphanilamide diaquo Co (II) complex :0.1 M N¹-(5,6-dimethoxypyrimidin-4-yl) sulphanilamide solution were prepared in excess warmed ethyl alcohol. Also cobalt chloride BDH make 0.1 M solution was prepared in warmed ethyl alcohol. The metal ion solution and N¹-(5,6-dimethoxypyrimidin-4-yl)sulphanilamide solution were mixed. Reaction mixture was refluxed in the round bottom flask for three hours. Before that the p^H of reaction mixture was maintained at 6.5 by adding alcoholic ammonia. Mehendi green colour precipitate appears. The precipitate of complex were filtered and purified by washing it with ethyl alcohol also by acetone. After the repeated washing of the complex it is dried and pure complex stored in a sample bottle.

Preparation of Monochloro N¹-(5,6-dimethoxypyrimidin-4-yl) sulphanilamide monoquo Cu (II) chloride complex : The BDH make copper chloride 0.1 M solution were prepared in ethyl alcohol. Also N¹-(5,6-dimethoxypyrimidin-4-yl) sulphanilamide 0.1 M solution were prepared in warm ethyl alcohol. These two reacting solutions were mixed in round bottom flask. The p^H of reaction mixture was also maintained by putting alcoholic ammonia drop by drop. Digestion of the reaction mixture was carried using steam bath and vertical water condenser. After four hours, light turkish blue colour precipitate appears. It is filtered through whatmann filter paper. Washing were done by ethyl alcohol. After purification of complex it is dried by keeping under sun. The dried complex was stored in labeled glass stopper bottle.

Characterization of the prepared metal complexes :

After preparation of above three metal complexes, physical characterization like nature, colour, decomposition point were measured and elemental analysis of metal complexes was done by the reported method⁵ was obtained from IICT Hyderabad. These observations are recorded in the table No.1. The solubility of the metal complexes also tested by dissolving.

Table No. 1: Analytical data and other physical properties of metal complexes

metal complex	Mol. wt	Colour	D.P °C	% of yield	M.L. ratio	Molar Cond. (S cm ² mol ⁻¹)	μ _{eff}	Elemental analysis Found (Calcd)			
								C	H	N	S
[Fe(DMPS) ₂ Cl ₂] H ₂ O	765.64	Chrome yellow	285	73	1:2	12.5	5.0 5	37.12 (37.61)	3.76 (3.91)	13.98 (14.62)	8.21 (8.35)
[Co (DMPS)Cl ₂ 2H ₂ O]	476.32	Mehandi green	239	81	1:1	9.50	3.8 5	29.98 (30.23)	3.48 (3.77)	11.63 (11.75)	6.65 (6.71)
[Cu (DMPS)ClH ₂ O]Cl	462.82	Light Turkish blue	247	79	1:1	8.26	2.0 8	31.42 (31.11)	3.26 (3.45)	12.34 (12.09)	7.04 (6.91)

Metal ligand ratio : To find out metal ligand ratio usual methods were adopted. The known weight of metal complexes were heated in the nickel crucible at high temperature. The complexes decomposes, the metal oxide of respective metal complex remains in the crucible. Further M:L ratio of the metal complex were determined.

The molar conductance of the prepared complexes was determined by conductometric method. The complex is dissolved in DMF, 0.01 M solution were prepared. After decomposing the prepared complex in the nitric acid, the complex solution were transferred to 25 ml standard flask, distilled water is added up to the mark. Estimation of metal ion is done by usual method chloride ions are estimated by vollard's method⁶. In Fe(II) complex chloride ion is found to be 10.04% matches with theoretical value which is 9.27% In Co(II) and Cu(II) complex % of chloride ion is 15.83 and 15.12 matches with the theoretical value 14.90 and 15.14 percent respectively.

Estimation of Fe (II) ion [Fe(DMPS)₂Cl₂] H₂O were carried out by volumetric titration. The Cobalt ion from [Co(DMPS)Cl₂2H₂O] complex were estimated by EDTA titration. Also Copper ions from [Cu(DMPS)ClH₂O]Cl complex were estimated by Iodometric method.

Table No. 2: Percent metal ion in prepared metal complex

Sr. No.	Name of Complex	Amount		Method used
		Observed %	Calculated %	
1.	[Fe(DMPS) ₂ Cl ₂] H ₂ O	6.99	7.31	Oxi-Red. Titration
2.	[Co(DMPS)Cl ₂ 2H ₂ O]	12.71	12.38	Co-EDTA Titration
3.	[Cu (DMPS) Cl H ₂ O] Cl	14.01	13.72	Iodometric method

Thermal Study :To study the effect of temperature during the decomposition of metal complex TGA-DTA study were used. The effect of temperature range, theoretical loss and experimental loss at different temperature is given in the table No. 3

Thermo analytical result of metal complexes of N¹(5,6-dimethoxypyrimidine-4-yl) sulphanilamide.

Table 3

Complex	Total loss		Temp range ^o C	Loss (%)
	Theort.(%)	Expt. (%)		
[Fe(DMPS) ₂ Cl ₂] H ₂ O	79.11	78.65	R.T.-95 95-475 475-550 ^o C 550 – 810 810-1000	2.3 45.17 14.18 17.00 20.02 (res.) 20.89 (Calcd.)
[Co(DMPS)Cl ₂ 2H ₂ O]	84.26	84.64	R.T.-200 200-335 335-814 814-1000	7.82 45.85 30.97 15.04 (res.) 15.74 (Calcd.)
[Cu(DMPS)Cl H ₂ O]Cl	83.80	82.86	R.T.-149 149-795 795-1000	3.99 78.87 16.96 (res.) 16.20 (Calcd.)

Thermal study of [Fe(DMPS)₂Cl₂] H₂O: TGA of Dichloro bis N¹-(5,6-dimethoxypyrimidin-4-yl) sulphanimide Fe (II) complex indicates decomposition in three steps. There is a loss in mass 2.3% at 95⁰C indicates the presence of lattice water⁷. As the temperature increases to 475⁰C, there is a loss of 45.17 indicating loss of some organic part of the metal complex. Further at 475⁰C– 550⁰C, 14.18% loss is observed indicating loss of remaining part of organic molecule. Further of 550-810⁰C, 17% loss observed indicating removal of chloride moiety. At 810⁰C, 20.02% of the complex remain in the form of oxide as Fe₂O₃.

Thermal study of [Co (DMPS)Cl₂ 2H₂O] complex : Dichloro N¹-(5,6-dimethoxypyrimidin-4-yl) sulphanimide diaquo Co(II) complex shows sharp decrease in weight. Weight loss at 200⁰C is found to be 7.82 percent which is close to calculated value 7.56 indicating loss of coordinated water. In the temperature range 200- 335⁰C, 45.85 percent loss occurs indicating decomposition of organic part. Further at 335-814⁰C loss of 30.97 percent were occurs indicating loss of chloride moiety. At 814⁰C weight of residual metal oxide⁸ was found to be 15.04 percent which is close to theoretical value 15.74.

Thermal Study of [Cu (DMPS) Cl H₂O] Cl : The TGA study of monochloro N¹-(5,6-dimethoxypyrimidin-4-yl) aquo Cu(II) chloride complex shows the loss of coordinated water by endothermic peak. The percentage loss of coordinated water molecule at 149⁰C was found to be 3.99 percent which is very close to theoretical value 3.89. Further decomposition continues up to 795⁰C indicating the loss of organic part of the complex and the percent weight loss of organic part of complex was found to be 78.87. At 795⁰C horizontal nature of the curve indicates the presence of thermally stable residual metal oxide⁹. The percentage of residual metal oxide was found to be 16.96 which it very close to theoretical value 16.20.

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