



## PHYSICOCHEMICAL STUDY OF COMPLEXES WITH SOME TRANSITION METAL ION'S WITH NEWLY DERIVED CONDENSED SULFA DRUG

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### ABSTRACT:

Newly condense sulfa drugs have been prepared by the condensation of Benzene sulphonamide with benzoyl isothiocyanate (BbsTC) . The sulfa drug was characterized by elemental analysis and spectral method. The metal complexes of Fe<sup>II</sup> , and Co<sup>II</sup> and Cu<sup>II</sup> and have been prepared by the reaction with BbsTC ligand. The analytical, spectral and magnetic study data support the Octahedral, Tetrahedral and Square planar complexes respectively. Thermalanalysis results are in good agreement with the composition of the complexes.

### INTRODUCTION:

Ligands derived from the reaction of aromatic aldehydes and aromatic amines or amides represent an important series of widely studied organic ligands. The relevant transition metal complexes are still found to be of great interest in inorganics chemistry, although this subject has been studies extensively [1,2,3]. A combination of distinctly different metal ion binding sites within one ligand can lead to material with interesting new properties [4,5], e.g. specific sensor, molecular wires, and magnetic and optical devices. In continuation of unpublished work; we report here the synthesis and physicochemical characterization of iron(II), cobalt(II), copper(II) with 1- Benzoly -3 (benzene sulfonamido) thiocarbamide (BbsTC).

#### 1. Physical Measurements:

The magnetic moments have been obtained by Gouy's method using mercurytetrathiocyanato cobaltate-(II) as a calibrant ( $\chi_g = 16.44 \times 10^{-6}$

c.g.s. units at 25°C). Diamagnetic correction were made using Pascal's constants. I.R. spectra were recorded on FTIR spectrophotometer at Regional Sophisticated Instrumentation Centre (RSIC) IIT Mumbai. Electronic spectra of the complexes were recorded in the 190-1200 nm range on Shimadzu Spectrophotometer with automatic recorder at Department of Chemistry, Nagpur University, Nagpur. The elemental analysis of the carbon, hydrogen and nitrogen were performed at RSIC Chandigarh. Sulphur estimation was done by standard messenger's method. The metal contents of the complexes were analyzed by EDTA titration [7] Thermogravimetric analysis (TGA) were obtained by -2 Perkin Elmer computer controlled Thermogravimetric analyzer model TGS-2 at RSIC Nagpur.

### MATERIAL & METHODS

Preparation of the organic ligand

The organic ligands, 1-Benzoyl-3 (benzene sulfonamido ) thiocarbamide (BbsTC). The preparation of BbsTC was carried out by dissolving benzene sulphonamide in minimum quantity of acetone in conical flask and the solution was warmed in the water bath to this solution benzoyl isothiocyanate was added drop by drop in the warm condition. The conical flask was heated for a few minutes in order to complete the reaction. Then the whole content was quickly poured in a petridish and allowed it to stand for few hours, in order to solidify. The excess of acetone present in the reaction evaporated automatically and we got a solid condensed product. The product was then recrystallised with ethanol. Yield 90% m.p : 155°C for BbsTC and yield 90% .

Preparation of the complexes :

The BbsTC-M<sup>II</sup> , [M=Fe<sup>II</sup>, Co<sup>II</sup>, and Cu<sup>II</sup>] complexes were synthesized by a following method,

Metal and Ligand was taken in the molar ratio 1:1 for reaction metal acetate [Fe(II) acetylacetonate in case of Fe(II)] and ligand was dissolved in minimum quantity of acetone separately. Then the solution was warmed in the waterbath. Both the solution was mixed in warm condition with constant stirring. Then the reaction mixture was digested for few minutes and quickly poured into a petridish and allowed it to stand for few hours in order to solidify. The excess of acetone present in the reaction gets evaporated automatically and we got a solid complex. The excess of metal present in this reaction was removed by washing with hot water.

#### RESULT & DISCUSSION:

Elemental analysis data, formula weights, percentage yields, melting points and magnetic moments are given in Table- 1. The results of the elemental analysis of the organic ligand BbsTC and its complexes are in good agreement with those required by the proposed formula.

I.R. Spectra:

The I. R. spectra of the complexes, in comparison with those of the free ligands, display certain changes which give an idea about the type of bonds and their structure. The ligand BbsTC, exhibit a broad bands in the region 3100-3650 may be assigned to  $\nu(\text{OH})$  stretching vibrating [8]. It is further observed that bands in the region 3400-3500 $\text{cm}^{-1}$  are disappeared in the complexes. In the free Ligand indicates bands around the region 1320 $\text{cm}^{-1}$  which may be assigned to be  $\nu(\text{C-O})$  stretching vibration[8]. In case of complexes the band shifts to higher frequency region 1330-1350  $\text{cm}^{-1}$  indicating the bonding of oxygen atom with metal. It is further supported by the appearance of new short and strong band in the region 530-600 $\text{cm}^{-1}$  in all complexes [9]. The  $\nu(\text{C=S})$  frequency of BbsTC occurs at 1100 $\text{cm}^{-1}$ . This band shift to lower energy by 10-30 $\text{cm}^{-1}$  in complexes indicating coordination through the sulphur [10]. The frequencies in the 550-560  $\text{cm}^{-1}$  ranges are attributed to metal coordination with sulphur [11,12]. The persual of the I.R. of BbsTC ligand indicates band around 1160-1140 $\text{cm}^{-1}$  which may be due to  $-\text{C-S-NH}$  thiocarbamide stretching vibrations [13]. In case of coordination all bands observed in the same range indicating no coordination through Nitrogen atom. In Ligand BbsTC broad band in the range 3390 $\text{cm}^{-1}$  and 3260 $\text{cm}^{-1}$  indicates presences of  $-\text{NH-}$  stretching frequencies [14] in complexes bands were appeared at same region indicating non-participation in the coordination of  $-\text{NH-}$  group. A well defined short band appears at 1600 $\text{cm}^{-1}$  in Ligand may be assigned to p- disubstituted benzene [13]. These bands appeared almost at the same frequency in the complexes indicating non-participation in coordination. The persual of the I.R. of BbsTC Ligand indicates two sharp bands in the regions 1150  $\text{cm}^{-1}$  and 1325 $\text{cm}^{-1}$  which may be  $\text{SO}_2\text{-NH}$  stretching vibration [15]. In coordination complexes all the bands

appeared almost at the same frequency region, indicating non-participation of  $-SO_2 - NH$  group in the coordination.

### Magnetic :

The magnetic moment of the complexes are given in Table-1 the values for the Iron(II) complex is 4.91 which suggest high spin octahedral geometry [16]. The magnetic moment of cobalt(II) complex is 3.98 which suggest high spin tetrahedral geometry [17]. In the investigating of cu(II) complex was found to be diamagnetic. This may be due to coupling of neighbouring spins leading to antiferromagnetic metal- metal interaction [16] square planer geometry of cu(II) complexes . Suggested on the basis of magnetic and spectral studies [ 18, 19, 20].

### Electronic Spectra :

The electronic spectra of the complexes were recorded in the 400-1200nm range. The electronic spectra of Iron(II) complex exhibit absorption band at 13.15 kK which are assigned to the transition  $5T_{2g} \rightarrow 5E_g$  for BbsTC complex suggesting octahedral geometry [21, 22]. The cobalt(II) complexes exhibit one broad band at 13.51kK for BbsTC complex and may be assigned to transition  $4A_2 \rightarrow 4T_1$  which can be assigned to tetrahedral geometry [23]. The electronic spectra of the copper(II) complexes exhibits band at 13.51 kK which corresponds to  $2B_{1g} \rightarrow 2A_{1g}$  transitions this transition can be assigned to square planner geometry [24].

Thermogravimetric Analysis :-

It has been observed that in Fe(II) complex show a loss in weight between 120-290°C indicating that presence of crystal water and coordinated water molecule in the Fe(II) complex. It is observed from the T.G. curves of the all complexes that the decomposition of organic ligands species from 300°- 700°C in one or two step's In all cases the remaining residue are metal oxides. These results are in good

agreement with the composition of the complexes.

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**Table :- 1 Elemental and Magnetic data for metal complexes of BbsTC Ligand**

Compounds Empirical formula	Formula weight	Yield %	% Found (Calculated)					M. eff. B.M.	M. P °C.
			C	H	N	S	Metal		
BbsTC C <sub>14</sub> H <sub>13</sub> N <sub>3</sub> O <sub>2</sub> S <sub>2</sub>	335	90	50.01 (50.14)	3.75 (3.90)	12.20 (12.52)	18.98 (19.12)	-	-	155°
Fe(II) (BbsTc) <sub>2</sub> 2H <sub>2</sub> O	761.84	75	45.27 (45.16)	8.69 (3.78)	11.33 (11.28)	17.29 (17.22)	7.56 (7.49)	4.91	-
Co(II)- (BbsTC) <sub>2</sub>	728.93	75	46.44 (46.08)	3.42 (3.59)	11.69 (11.51)	17.45 (17.57)	7.98 (8.07)	3.98	-
Cu(II)- (BbsTC) <sub>2</sub>	733.54	80	45.91 (45.79)	3.39 (3.56)	11.68 (11.44)	17.27 (17.46)	8.77 (8.65)	Diamagnetic	-

**Table :- 2 Thermoanalytical Data and Decomposition Temperature for BbsTc complexe**

Complexes	Temp. Range (c°)	Stage of Decompositio n	Species degraded	% Weight Loss	
				Found	Cal.
Fe(II)-(BbsTc) <sub>2</sub> 2H <sub>2</sub> O	175- 250	First	Two Water molecule and phenyl group	23.00	23.35
	250-380	Second	C <sub>6</sub> H <sub>5</sub> SO <sub>2</sub> NH <sub>2</sub>	42.80	42.43
Co(II)- (BbsTC) <sub>2</sub>	175-250	First	Phenyl or SO <sub>2</sub> - NH <sub>2</sub>	22.78	21.37
	250-450	Second	C <sub>6</sub> H <sub>5</sub> SO <sub>2</sub> NH <sub>2</sub>	41.11	43.30
Cu(II)- (BbsTC) <sub>2</sub>	175-425	First	Phenyl Group & SO <sub>2</sub> NH <sub>2</sub>	42.78	43.35
	425-575	Second	Phenyl group , - NH	25.40	25.32