



**PHYSICOCHEMICAL AND BIOLOGICAL STUDY OF SOME
TRANSITION METAL COMPLEXES WITH NEWLY DERIVED
CONDENSED SULFA DRUG**

N. C. Kongre¹ , P. N. Patil²

¹Department of Chemistry, Jawaharlal Neharu Arts, Commerce and
Science College, Wadi, Nagpur-440023 India . Email-

²R.T.M.Nagpur Universitys Bar.Wankhede College of Education Nagpur-
Corresponding author E-mail : n.kongre@rediffmail.com

Abstract :

Newly condense sulfa drug have been prepared by the condensation of Sulphaguanidine with benzoyl isothiocyanate (BbsgTC).The sulfa drug was characterized by elemental analysis and spectral method. The metal complexes of Ni^{II} ,Zn^{II}, Co^{II}and Fe^{II} have been prepared by the reaction with BbsgTC ligand. The analytical, spectral and magnetic study data support the Tetrahedral and octahedral complexes respectively. The biological activities of ligand and their complexes have been studied by screening the compounds, against micro organisms such as E.Coli and S. aureus

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 [4-6] e.g. specific sensor, molecular wires, and magnetic and optical devices. Recently it has been proved that several coumarins and sulfonamides derived Schiff-bases and their metal complexes are less cytotoxic and the decrease in cythotoxicity is due to coordination with the metal ion [7,8]. In some complexes, it has been



observed that minor changes in the structure of the ligands containing hard/soft donor atoms, e.g. nitrogen, sulfur and oxygen markedly affect the activity of the compound [9,10]. In continuation of unpublished work we report here the synthesis and physicochemical characterization of nickel(II), zinc(II), cobalt(II) and iron(II) complexes with 1- Benzoly-3(benzene sulphoguanido)- thiocarbamide (BbsgTC).

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 [11]

Experimental :-

Preparation of the organic ligand

The organic ligand 1- benzoly -3 (benzene sulphoguanido) – thiocarbamide (BbsgTC).The preparation of BbsgTC was carried out by dissolving benzene sulphaguanidine in minimum quantity of acetone in conical flask and the solution was warned in the water bath to this solution benzoly 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: 190°C for BbsgTC.

Preparation of the complexes :

The BbsgTC- M^{II} , [$M=Ni^{II}$, Zn^{II} , Co^{II} ,& Fe^{II}] complexes were synthesized by a following method,

Metal and Ligand was taken in the molar ratio 1:1 for reaction metal acetate and ligand was dissolved in minimum quantity of acetone separately. Then the solution was warmed in the water bath. 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 Petridis 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.

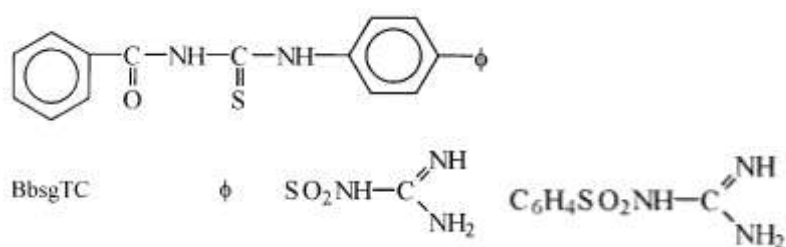
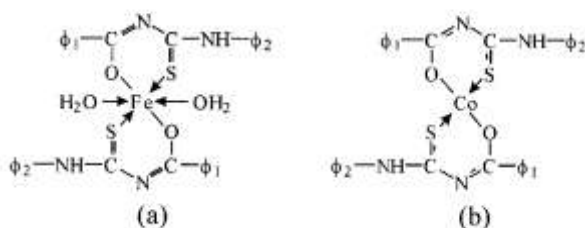


Fig. 1 : Structure of the ligand



Where

ϕ_1 C_6H_5

ϕ_2

Fig. 2: Suggested structure of the complexes.



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 BbsgTC 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 BbsgTC exhibit a broad bands in the region 3100-3650 may be assigned to $\nu(\text{OH})$ stretching vibrating [12]. It is further observed that bands in the region 3400-3500 cm^{-1} are disappeared in the complexes. In the free Ligand indicates band around the region 1320 cm^{-1} which may be assigned to be $\nu(\text{C-O})$ stretching vibration [12]. In case of complexes the band shifts to higher frequency region 1330-1350 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 cm^{-1} in call complexes [13]. The $\nu(\text{C=S})$ frequency of BbsgTC occurs at 1100 cm^{-1} . This band shift to lower energy by 10-30 cm^{-1} in complexes indicating coordination through the sulphur[14]. The frequencies in the 550-560 cm^{-1} ranges are attributed to metal coordination with sulphur [15,16]. The persual of the I.R. of BbsgTC ligand indicates band around 1160-1140 cm^{-1} which may be due to $-\text{C-S-NH}$ thiocarbamide stretching vibrations [17]. In case of coordination all bands observed in the same range indicating no coordination through Nitrogen atom. In Ligand BbsgTC broad band in the range 3260 cm^{-1} indicates presences of $-\text{NH}-$ strecting frequencies [18] in complexes



bands were appeared at same region indicating non-participation in the coordination of -NH- group. A well defined short band appears at 1600cm^{-1} in Ligand may be assigned to p- disubstituted benzene [17]. These bands appeared almost at the same frequency in the complexes indicating non-participation in coordination. The persual of the I.R. of BbsgTC Ligand indicates two sharp bands in the regions 1150 cm^{-1} and 1340 cm^{-1} which may be $\text{SO}_2\text{-NH}$ stretching vibration [19]. In coordination complexes all the bands appeared almost at the same frequency region, indicating non-participation of $\text{-SO}_2\text{-NH}$ group in the coordination. In ligand BbsgTC band at 1530cm^{-1} indicates presences of imine (C=N) group stretching vibrations [17] this band appeared at the same frequency after coordination with metal ion indicate non-participation of the imine group in the coordination.

Magnetic :

The magnetic moment of the complexes are given in Table-1 the values for the Nickel(II) complexes was 3.76 B.M. which suggest the tetrahedral geometry [20] The magnetic moment of Zn(II) complexes are found to be diamagnetic in nature [21]. The magnetic moment of Co(II) complexes was 4.11 B.M suggesting high spin tetrahedral geometry [21]. The magnetic moment of Fe (II) complexes are 5.09 B.M. The lowering of the value may be due to sulphur present in the ligand [20]

Electronic Spectra :

The electronic spectra of the complexes were recorded in the 400-1200nm range. The electronic spectra of Nickel (II) complexes exhibit absorption band at 13.51 kk which are assigned to the transition ${}^3\text{T}_1 \rightarrow {}^3\text{T}_1(\text{P})$ for BbsgTC complexes suggesting tetrahedral geometry [22]. The Zinck(II) complexes electronic spectra indicate no d-d transition as expected for d^{10} system[23]. The electronic spectra of the Cobalt (II)



complexes exhibits bands at 12.98 kK which corresponds to ${}^4A_2 \rightarrow {}^4T_1$ transitions. This transition can be assigned to tetrahedral geometry [24]. The Iron (II) complexes electronic spectra exhibits bands at 13.15 kK which corresponds to ${}^5T_{2g} \rightarrow {}^5E_g$ transitions. This transition can be assigned to high spin octahedral geometry [22].

Biological Activity:-

All of these complexes, ligand and parent compound are screened for their antibacterial activity against *E. coli* and *S. aureus* using Disc Diffusion Method [25]. The reading and interpretation of result were done according to Cruickshank et al [26]. The diameter of the zones of inhibition of growth, including 6mm diameter of the cup, was measured by viewing the plate against a ruler. Considering *E. coli* in series B strain, the ligand B2 showed enhanced activity than the parent compound (B1) and the metal complexes. All the metal complexes showed lower activity. In the *S. aureus* strain all the compounds showed lesser activity as compared to the parent compound. Zones of inhibition of growth of compounds are listed in Table. No.2 The activity showed by the metal complexes are in the order as follows

Series B: *E. coli*- Fe > Ni > Co > Zn *S. aureus*- Fe > Ni > Co > Zn

Acknowledgements :

We wish to express our gratitude to the Head, Department of Chemistry, Rashtrasant Tukdoji Maharaj Nagpur University Nagpur for providing the necessary laboratory facilities also thankful to the Director RSIC IIT, Mumbai. RSIC Chandigarh for IR and elemental analysis respectively.



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

Compounds Empirical formula	Formula weight	Yield %	% Found (Calculated)					M. eff. B.M.	M. P °C.
			C	H	N	S	Metal		
Bbsg TC C ₁₅ H ₁₅ N ₅ O ₃ S ₂	377	90	47.65 (47.73)	3.89 (4.00)	18.26 (18.55)	17.09 (16.98)	-	-	190°
Ni(II) (BbsgTC) ₂	845.84	75	44.19 (44.28)	3.43 (3.69)	17.04 (17.21)	15.69 (15.76)	7.46 (7.21)	5.09	-
Zn(II)- (BbsgTC) ₂	812.58	70	44.06 (43.92)	3.83 (3.68)	16.90 (17.07)	15.57 (15.75)	7.71 (7.97)	Diamagnetic	-
Co(II)- (BbsgTC) ₂	817.54	70	44.36 (44.27)	3.62 (3.71)	16.98 (17.21)	15.57 (15.75)	7.41 (7.24)	4.11	-
Fe(II)- (BbsgTC) ₂ 2H ₂ O	843.70	70	43.58 (43.47)	3.84 (3.89)	17.01 (16.90)	15.39 (15.47)	6.81 (6.73)	5.09	

Table :- 2 Zones of inhibition of growth of compounds

Compounds	E.coli (mm)	S. aureus (mm)
B1. Sulphaguanidine	26	20
B2. BbsgTC	28	18
B3. Ni (BbsgTC) ₂	22	11
B4. Zn (BbsgTC) ₂	20	09
B5. Co (BbsgTC) ₂	21	10
B6. Fe (BbsgTC) ₂ 2H ₂ O	23	13

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