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Coordination Chemistry of Cobalt Biguanide Complex: Ligand Interaction and Metal Complex Stability

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Abstract: ***Biguanides are versatile organic compounds known for their broad applications in medicinal chemistry, coordination chemistry, and catalysis. Biguanide, $C_2N_5H_7$ is a nitrogen donor bidentate ligand. Structurally, they contain two linked guanidine moieties, which enable strong chelation with metal ions, forming stable complexes. The ability of biguanides to interact with transition metals has led to the development of metal-biguanide complexes with remarkable biological and catalytic properties. These complexes exhibit diverse coordination modes influenced by steric and electronic factors, resulting in distinct physicochemical behaviours. Spectroscopic and crystallographic studies reveal their structural variations, while computational methods provide insights into their electronic configurations and reactivity. Biguanide-metal complexes have been investigated for their role in antimicrobial, anticancer, and catalytic applications, making them essential in modern chemistry. This abstract outline the synthesis, characterisation, and potential applications of biguanide-based metal complexes, emphasizing their importance in material science and medicinal research.***
Keywords: Biguanide, Cobalt, mixed ligand, diamagnetic, IR, UV.

I. INTRODUCTION

Biguanide exist as open chain systems, though some cyclic biguanides have been utilized in medicinal chemistry, where they exhibit their active state. Due to their electron -rich nature, they are highly basic and are primarily employed in their monoprotonated form in medicinal applications. The first synthesis of biguanide ($C_2N_5H_7$) was reported in 1879 by Rathke, utilizing cyanimide and guanidine. Later, in 1929, Slotta and Tschesche identified the hypoglycemics activity of the biguanide derivative metformin in animal studies.[1]. Biguanide and its derivatives have long been utilized in medical science due to their diverse pharmacological applications [2]. One notable example is metformin, widely prescribed oral hypoglycemics agent for managing type 2 diabetes. Similarly, progu-anil serves as an antimalarial drug. Additionally, phenyl aminopropyl biguanide find use as both a disinfectant and an antimalarial agent [3][4][5]. Structurally, biguanide is formed through the condensation of two guanidine molecule. Biguanide is a bidentate ligand and it coordinates to the central metal here Co (III) through its imino and amino N-atoms. Biguanide is formed through the condensation of two guanidine molecules, resulting in removal of ammonia molecule.

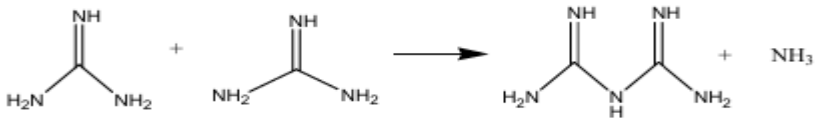

Fig 1.Condesation reaction of guanidine molecules to form biguanide with the elimination of ammonia

II. EXPERIMENTAL

A. Materials and Methods

Dicyandiamide, Aluminium Chloride, Benzylamine, Copper Sulphate and all the other chemicals required were available commercially and used as such.

Physical Measurements: The results of C, H, S and N were obtained from SAIF, COCHIN using the CHNS Analyser with instrument name as ELEMTAR VARIO EL III. The result of UV was recorded on 5000 UV-VIS-NIR spectrometer and IR spectra were recorded on FT-IR spectrometer, SAIF, COCHIN. The ligand and the complexes were prepared by the reported method [6].

B. Synthesis of ligand

1) *Preparation of Biguanide Sulphate* [7]: Biguanide Sulphate was prepared according to the method described by Smolka and Fridrich with slight modification. In this method, a mixture of ammonium chloride (NH_4Cl) and Dicyandiamide $C_2H_4N_4$, in ratio of 2:1 was dried in oven and mixed thoroughly in mortar and pestle and the mixture so obtained was then transferred in Pyrex beaker and heated by means of Bunsen burner. During this process, the mixture was constantly stirred with the help of glass rod and the temperature was maintained at 155^oC for ten minutes as soon as the mixture changed into molten mass. The molten mass was then poured into large volume of water and then treated with Cuprammonium sulphate $[Cu(NH_3)_4]SO_4$. As a result, rose red coloured precipitate of copper biguanide sulphate was then obtained at once. The precipitate should be kept over Buchner funnel and was washed thoroughly with cold water. The precipitate should be kept over Buchner funnel till water gets drained. The moist Copper biguanide sulphate was then decomposed with cold solution of 10%H_2SO_4. A blue solution was obtained which on keeping in cold deposited large crystals of Biguanide Sulphate.
2) *Synthesis of Dibenzyl amine bisbiguanidinium Cobalt (III) hydroxide:* The Complex was synthesized by adding calculated amount of Biguanide sulphate dissolved in slight excess of Sodium hydroxide solution of Cobalt (II) Sulphate with continuous stirring. As a result, the yellow silky ppt was obtained which was filtered quickly to avoid oxidation on Buchner funnel and was washed with ice cold water. The yellow-coloured complex so obtained was then transferred to an aeration flask. To this suspension small amount of Benzylamine, $C_6H_5CH_2NH_2$ was added and then a brisk current of air was passed through it to oxidize Co (II) complex to Co (III) complex. Due to aeration, the silky yellow bisbiguanidinium Cobalt (II) Sulphate gradually dissolve to a dark red solution with the separation of a slight black oxide of Cobalt due to the decomposition of the complex. The mixture was then filtered through a quantitative filter paper and the filtrate was left in cold for crystallisation. These were filtered and recrystallised with hot water.

III.RESULTS AND DISCUSSION

A. Physico-Chemical Analysis

The yield of synthesized Biguanide is 3gm when 8gm of Dicyandiamide with 16gm of Ammonium Chloride was fused. The colour of the ligand and complex is white and orange coloured. The prepared complex was stable in air. The melting point of the ligand 231-232^oC. The value of molar mass is 235.24.

Table I
Physico-Chemical Properties of Synthesized ligand and Complex

SL.No.	PROPERTIES	LIGAND VALUE	COMPLEX VALUE
1.	NAME	Biguanide Sulphate	Dibenzylamine bisbiguanidinium hydroxide
2.	Colour	White crystalline	Brownish Orange
3.	Physical State	Crystalline Solid	Crystalline Solid
4.	Yield	3gm	4.4gm
5.	Stability	Stable in Air	Stable in Air
6.	Recrystallisation Solvent	Distilled Water	Ethanol

B. Elemental Analysis

The Elemental Analysis provides insights about the Carbon, Nitrogen, Hydrogen, Oxygen.It is done by ELEMTAR VARIO EL III instrument. The Metal content is determined by Gravimetric Method.

Table II
Elemental Content Of ligand and Complex

SL. No		CARBON	NITROGEN	HYDROGEN	OXYGEN	METAL
1.	LIGAND	10.21%	29.90%	4.3%	31.71%	-
2.	COMPLEX	61.07%	13.29%	10.26%	1.74%	6.41%

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C. FT-IR Spectral Analysis

The FT-IR spectra of the biguanide ligand [8][9] was recorded using Nicolet iS50 FTIR instrument in the frequency range of 4000-450 $cm^{-1.}$ The IR spectral frequency bands of the free biguanide ligand is mentioned in the Table III.

Table III
FT-IR Spectral Band Positions of Synthesized Biguanide Ligand

WAVENUMBER (in cm^{-1})	ASSIGNMENT
$3389cm^{-1}$	NH_2 amine
$3019cm^{-1}$,$2399cm^{-1}$	N-H stretching NH_3^+
$929cm^{-1}$	N-H deform
$769cm^{-1}$	N-H wagging
$1069cm^{-1}$	C-N sym stretching
$1626cm^{-1}$	C=N sym stretching

The IR spectrum of Biguanide ligand shows stretches between $3389cm^{-1}$ and 1626 cm^{-1} and these belongs to NH_2 group, NH_3^+
The N-H deformation band is $929cm^{-1}$ and the N-H out of plane vibration occurs at $769cm^{-1}$ The C-N stretching vibrations is shown at $1069cm^{-}$
The IR spectral frequency bands of the Biguanide Cobalt Complex is mentioned in the Table IV.

Table IV
FT-IR Spectral Band Positions of Synthesized Biguanide Cobalt Complex

WAVENUMBER(in cm^{-1})	ASSIGNMENT
$3416.68cm^{-1}$	O-H bond stretching
$1634.99cm^{-1}$,$1525cm^{-1}$	C=C aromatic
$1305cm^{-1}$	C-N stretching amine
$517cm^{-1}$	Metal ligand

The IR spectrum of Biguanide ligand shows stretches between $3416.68cm^{-1}$ and $517cm^{-1}$. The metal complex shows broad band in the range $3000cm^{-1}$ and 1305 cm^{-1} assignable to the vibrations due to -OH and -CN primary amine. The metal-nitrogen bond is observed at $517cm^{-1}$ indicating the metal ligand bond formation or Metal Complex formation.

D. Electronic spectral Analysis

The Co (III) complexes shows two band, one around 480-490 nm is attributed to ($^1A_{1g} \rightarrow {}^1T_{1g}$) and other at around 260-265 nm due to ($^1A_{1g} \rightarrow {}^1T_{2g}$) and charge transfer transition. The diamagnetic behaviour and absorption at these bands suggest octahedral structure for the complexes of Co (III) ion with Biguanide and Benzylamine ligands.

IV. CONCLUSION

On the basis of stoichiometry, magnetic measurements, spectral studies (IR & electronic absorption) and physico-chemical properties, Octahedral structure have been proposed for these mixed ligand metal biguanide complexes. Following below is the proposed structure of Metal Complex

V. ACKNOWLEDGMENT

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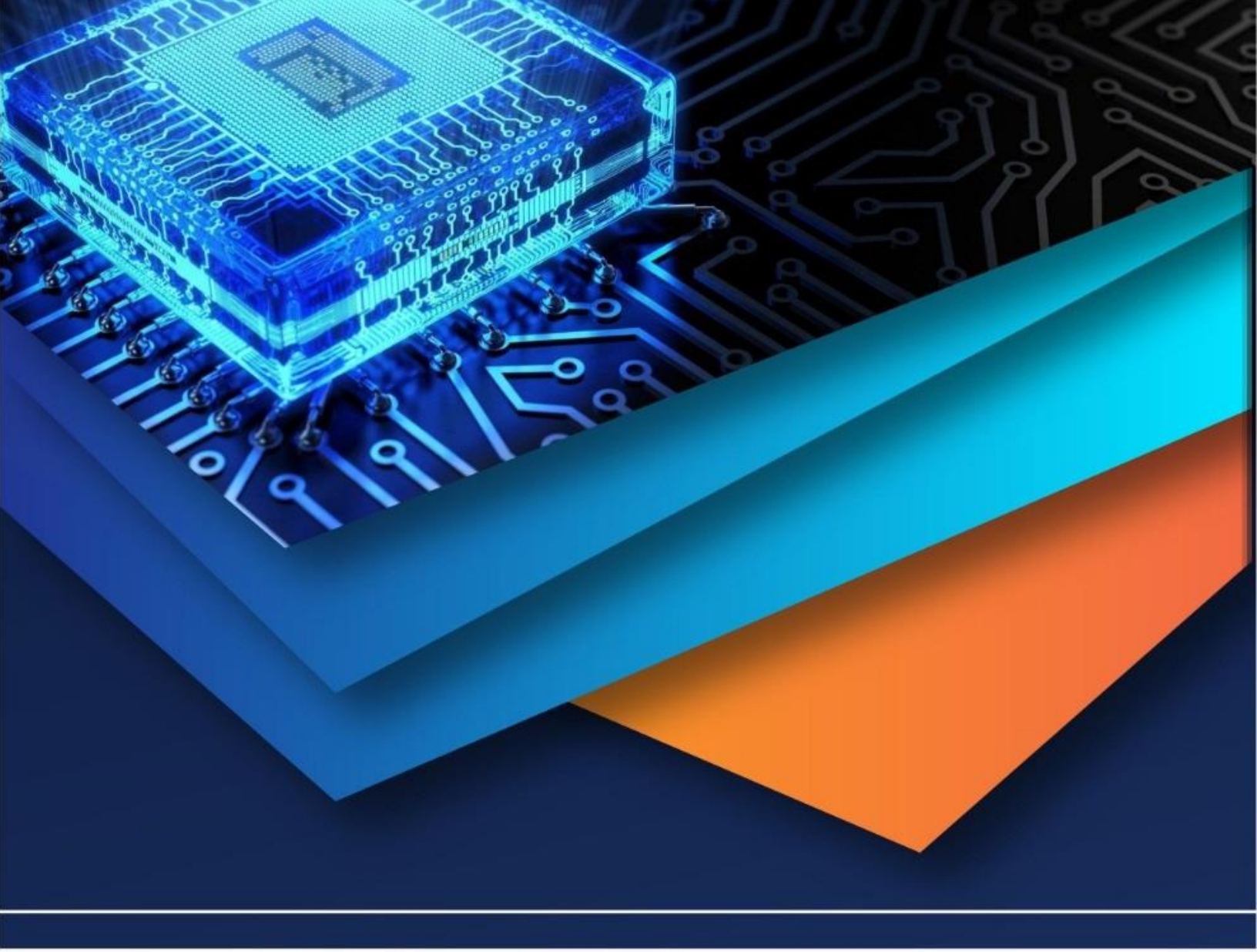

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