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SYNTESIS AND SPECTRAL STUDIES OF MIXED LIGAND COMPLEXES OF CHLOROPROPAMIDE /GLICLAZIDE WITH IRON AND COBALT

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ABSTRACT

Co-ordination compounds of chloropropamide (CPM) N-Propyl-N-(P-chlorobenzene sulphonyl) urea (trade name, *diabinese*) and gliclazide (GCZ) 1 - (3 - azabicyclo [3,3,0] oct - 3yl) - 3 - (P - totyl sulphonyl) urea. (trade name, *diamicron*) are oral hypoglycemic were synthesize with iron(II) and cobalt(II), isolated in pure powderd form, yields are substantial. Stoichiometry suggest 1:1:1 ratio between drugs and metal ion. Analytical data of the complexes agrees with the compositions are $C_{10}H_{12}ClN_2O_3S$-Fe-$C_{15}H_{20}N_3O_3S$ $2H_2O$ and $C_{10}H_{12}ClN_2O_3S$-Co-$C_{15}H_{20}N_3O_3S.2H_2O$ The general structure assigned to these complexes is supported by infra-red and electronic spectral studies.

Key Words:- Complexation chloropropamide, gliclazide, iron and cobalt.

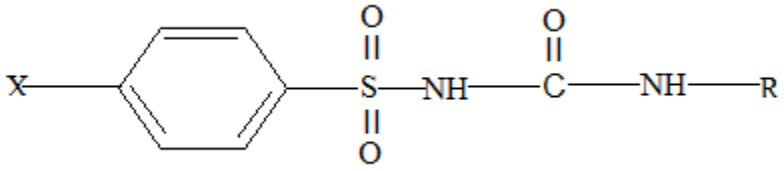
INTRODUCTION

Sulphonyl urea represented by general structure 'I' have long been in use as hypoglycemic agents in the treatment of *Diabetes mellitus*[1-5]. In continuation of our previous work[6-7], on the synthesis and characterization of metal complexes of various drugs with transition metals, it has been desired necessary to synthesise and study the new type of complexes of mixed drugs in order to see as to whether it increases the potencies of the complexes or not.

Therefore in the present communication we are describing the synthesis of mixed CPM/GCZ complexes with iron and cobalt. A survey of literature indicates that Yoshinaga and Yamamotto have synthesized and studied Zn (II), Cd (II) and Hg (II) complexes with suphlonyl ureas[8-10] and have recommended their use in mercury poisoning.

MATERIALS AND METHODS

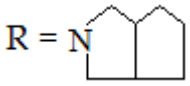
Pure sample of CPM, Mf $C_{10}H_{13}ClN_2O_3S$ m.p. 127° (lit., 127°-129°C) and GCZ, Mf $C_{15}H_{13}N_3O_3S$ m.p. 180°C (lit., 180°-182°C), CPM and TBM are soluble in absolute alcohol. Salts like ferrous sulphate and cobalt chloride were of AnalaR grade.



(I) (Open Chain general structure)

1 X = Cl Chloropropamide (L_1)
R = C_3H_7

2 X = CH3 Gliclazide (L_2)



SYNTHESIS :

The ligand CMP (L_1) trade name, (*Diabinese*) 0.691g and GCZ L_2 (*Dimicron*) 0.808g while metal salts Viz., iron 0.695g and cobalt 0.594g are in 1:1:1 molar ratio L_1L_2M were dissolved separately in minimum quantity of absolute alcohol. Ligand solution was added gradually with constant stirring to metal salt solution, a thick dark brown precipitate (CPM-Fe-GCZ.$2H_2O$) and light pink precipitate of (CPM-Co-GCZ.$2H_2O$) complexes were obtained on adjusting the pH upto 6.0 by adding very dilute NaOH solution and refluxing for four hours. Dark brown crystals of Fe complex, and light pink crystals of cobalt complex were obtained. The complexes were washed with absolute alcohol, dried and weighed. Yields were 50.20% and 55.63% respectively. The decomposition temperatures are given in table 1.

Iron was estimated in complex by Cupfurron method as oxide, cobalt was estimated as, tetrapyridine dithionate[12], nitrogen by Kjeldahls method using modified digestion mixture[13], sulphur was estimated by Messenger's method.

Table 1. Physico-Chemical characterisation of chcorpropamide/gliclazide complex

S N	Composition of complexes	Colour	DC.°C (%yield)	Metal % of Analysis/(Calcd) Metal	C	H	N	S	H_2O
1	$C_{10}H_{12}ClN_2O_3S$-Fe-$C_{15}H_{20}N_3O_3S.2H_2O$	Dark brown	220 (50.20)	8.43 (8.50)	43.20 (43.33)	4.94 (5.02)	9.80 (10.11)	8.80 (9.76)	5.10 (5.20)
2	$C_{10}H_{12}ClN_2O_3S$-Co-$C_{15}H_{20}N_3O_3S$ $2H_2O$	Light pink	210 (55.63)	8.80 (9.09)	43.19 (43.52)	4.24 4.64	10.01 (10.15)	9.50 (9.72)	5.19 (5.22)

DC = Decomposition temperature

Table 2. IR absorption data of the complex

Ligand and Complexes	SO_2-NH (cm-1)	C=O (cm^{-1})	C-O	M-O (cm^{-1})	NH-R (cm^{-1})	C=N (cm^{-1})	C_6H_5-S (cm^{-1})	Water of ordinaton (cm^{-1})
CPM,	1170 ± 10 (J.D/38)	1669 KN/184	-	-	-	-	750	-
GCZ	1165 ± 10	16640	-	-	-	-	-	-
Complex of iron	3080 ± 40 (A.W)		1650 ± 10	669	3070	2510	710	3399
Complex of cobalt	3070 ± 30		1649 ± 10	669	3080	2520	730	3390

Table 3. Electronic spectra and magnetic moment values, and possible assignments of the Fe (II) and Co (II) complexes of mixed ligands

Complexes	Magnetic	Electronic moment	Possible assignment bands (cm^{-1})	Proposed geometry
$Fe(L_1L_2)(OH_2)$	5.41	27860 21790	Charge transfer $^5Eg \leftarrow {}^5T_2g$	Octahedral
$Co(L_1L_2)(OH_2)$	4.10	21450 14750 12900	$^4T_1g \leftarrow {}^4T_1g$ (F) $^4A_2g \leftarrow {}^4T_1g$ (F) $^4T_2g \leftarrow {}^4T_1g$ (F)	Octahedral

RESULTS AND DISCUSSION

The infrared absorption frequencies of the various groups like SO_2NH, C = O, aromatic ring and alkyl group etc. found in ligand are modified on complexation in intensity as well as in the frequency. The new frequencies observed in the complexes (Fe and Co) like C-O1650 ± 10cm^{-1}/1649 ± 10cm^{-1},M-O669cm^{-1}/669cm^{-1}, C = N2510cm^{-1}/2520cm^{-1}, NH-R3070cm^{-1}/3080cm^{-1} and C_6H_5-S 710cm^{-1}/730cm^{-1}. In addition to this we have proposed structure 2 for Fe (II) and Co (II) complexes which have two molecules of water of coordination satisfying the six coordination number of Fe^{++} and Co^{++} present. In case of structure 2, is further supported by presence of very strong broad bands observe at 3399cm^{-1}/3390cm-1 for water of coordination. IR results support the structure of the complex which was already suggested on the basis of stoichiometry and analytical data (table 2)[14-17].

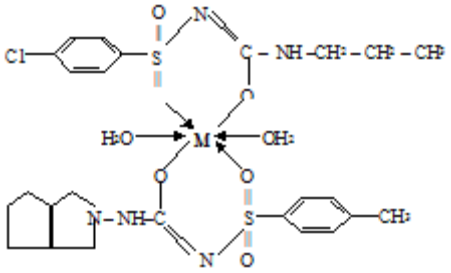


Fig. 2 (M = Fe and Co)

The Electronic spectra of Fe (II) complex shows two bands at 27860 cm^{-1} and 22790 cm^{-1}, the first band is assigned to charge transfer while second band would be due to $^5Eg \leftarrow {}^5T_2g$ transition suggesting an octahedral geometry around Fe (II) ion[18]. The magnetic moment μeff is 5.41 B.M. agrees with octahedral symmetry of Fe (II) complex[18].

The Electronic spectra of Co (II) complex shows three bands observed at 21450 cm^{-1}, 14750 cm^{-1} and 12900 cm^{-1} assignable to ^4T_1g $(P)_4 \leftarrow {}^4T_1g$ (F), ^4A_2g (F) $\leftarrow {}^4T_1g$ (F) and ^4T_2g (F) $\leftarrow {}^4T_1g$ (F) respectively, favours the octahedral environment of Co (II) complex. The μeff valves of Co-complex has been found to be 4.10 B.M. these values of electronic absorption bands and magnetic moment indicate an octahedral geometry of Co (II) ions[19-20]. Therefore a common structure 2, can be assigned to Fe (II) and Co (II) complexes.

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