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X-RAY DIFFRACTION STUDIES OF Co(II), Cu(II), MO(II), COMPLEXES WITH GLICLAZIDE 1-(3-azobicyclo[3,3,0] Oct-3yl)-3-(p-tolysulphonyl) UREAS AS AN ANTIDIABETIC SCREENING

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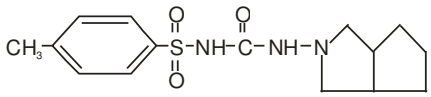
Abstract:
GLICLAZIDE 1-(3-azobicyclo[3,3,0] Oct-3yl)-3-(p-tolysulphonyl) *was used to synthesize* **Co(II), Cu(II), MO(II),** *complexes. Metal complxes were characterized by elemental analysis, IR, TGA. The crystal structure of complexes were further determined by X-ray diffraction method. The XRD diffraction method. The XRD data was used to index the compound for tetragonal and octahedral system.*
Keywords: *Glibenclamide, Crystal structure,* **Co(II), Cu(II), MO(II),** *complex.*

Introduction:

Polyfunctionally rings compounds and synthesis of their metal complex which have various biological activities and include hetero atom, have been formed in organic synthesis and coordination chemistry.[1.3] Many trasition and inner trasition metal complexes have been synthesized for analytical and commercial applicatisis many of medicinal use.[4-5] literature survey reveals that the transition metal complexes generally crystallized with tetrahedral, octahedral geometry.[6-3]

Structure of Gliclazide



Experimental:

All the chemicals used for the preparation of complexes are of Hi-media AR grade mark. Metal complexes are synthesized by adding metal salt solution in appropriate solvent to the solution of the ligand. The mixture was refluxed for 3-4 hours. Then the precipitate of metal complxes was obtained. It is filtered, washed and dried in vacuum desiccators.

All selected metals forms 1:2 complexes with glibenclamide were confirmed by to be method as modified by Turner and Anderson.[9]

Result and Discussion:

The result of ESR spectra and X-ray diffraction of , **Co(II), Cu(II), MO(II),** complexes with Gliclazide were obtained and summarized in following tables. Al reflections has been indexed for h, k, l values using reported lite[14-16] rutur[12-13] and full proof suit XRD software v.2.0 by using fullpray suite XRD software the d-values of metal complexes were obtained. From ESR spectra of complxes the value of 9:g_4g_3 and gav can be determined. This value are tabulated in table No. (2)

In case of Cu(II) complex gav value is 2.09 which is less than 2.271. the values of indicates the presence of sufficient covalence between the metal ion and donar atom.[17] In case of Zn(II) complex gav value is found to be 2.21. this value is less than 2.25. it is assignable to the presence of covalent character in metal ion and donar atom. In case of Mo(II) complex g_{av} values found to be 2.30 and 2.09 respectively. This values Sc less than 2.387 and 2.247 indicates presence of covalent characters in coordinate bond.

Table -3.32 Physico-Chemical Characteristics Of Gliclazide Metal Complexes

Sl. No.	Formulae of Complexes	Colour	%Yield	m.p.°C	Molar conductance $\Omega^{-1}cm^{-1}mole^{-1}$
3.	$(C_{15}H_{21}N_3O_3S)_2Mo{\cdot}2H_2O$	Purple	60.00	169	30.1
5.	$(C_{15}H_{21}N_3O_3S)_2Co{\cdot}2H_2O$	Light Pink	55.80	210	35.0
6.	$(C_{15}H_{21}N_3O_3S)_2Cu$	Light Green	68.00	180	34.1

Table -3.33 Analytical Data Of Gliclazide Complexes

Sl. No.	Molecular formula of complexes	Molecular weight	% Analysis found (calculated)						
			C	H	N	O	S	Metal	H_2O
3.	$(C_{15}H_{21}N_3O_3S)_2Mo{\cdot}2H_2O$	776.764	46.82 (46.34)	5.28 (5.14)	10.38 (10.81)	12.20 (12.35)	8.13 (8.23)	12.12 (12.35)	5.23 (4.63)
5.	$(C_{15}H_{21}N_3O_3S)_2Co{\cdot}2H_2O$	739.754	42.26 (48.66)	5.64 (5.40)	8.60 (11.35)	13.04 (12.97)	8.50 (8.65)	7.12 (7.96)	5.24 (4.86)
6.	$(C_{15}H_{21}N_3O_3S)_2Cu$	708.364	36.07 (50.82)	6.03 (5.64)	8.46 (11.85)	13.84 (13.55)	8.98 (9.03)	7.99 (8.96)	-

X-ray diffraction study-

1 X-ray diffraction study of Gliclazide and their Co,Cu and Mo complexes

Gliclazide (GLC)

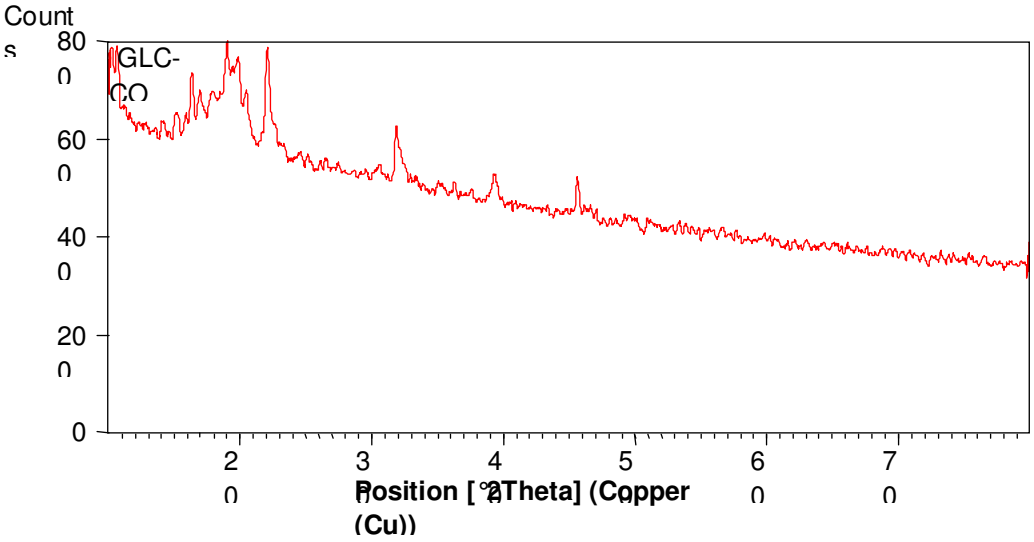


Figure.9.1: X-ray difractogram of GLC-Co Complex

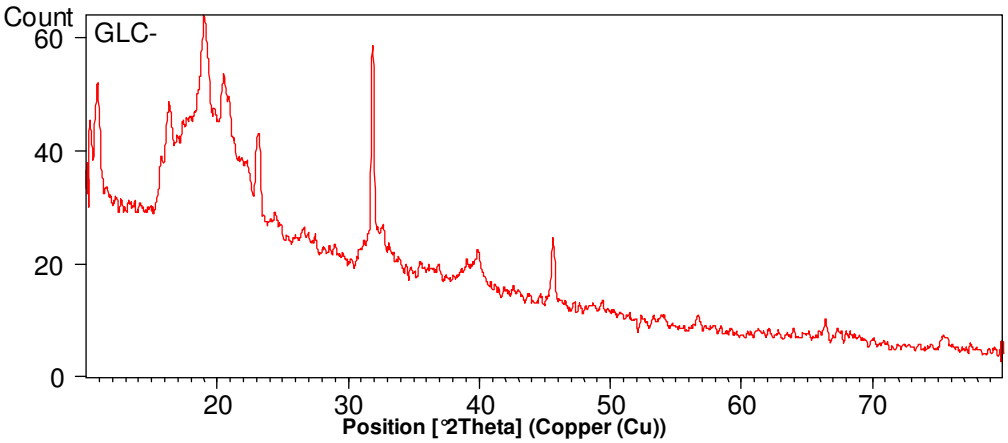


Figure.9.2: X-ray difractogram of GLC-Cu Complex

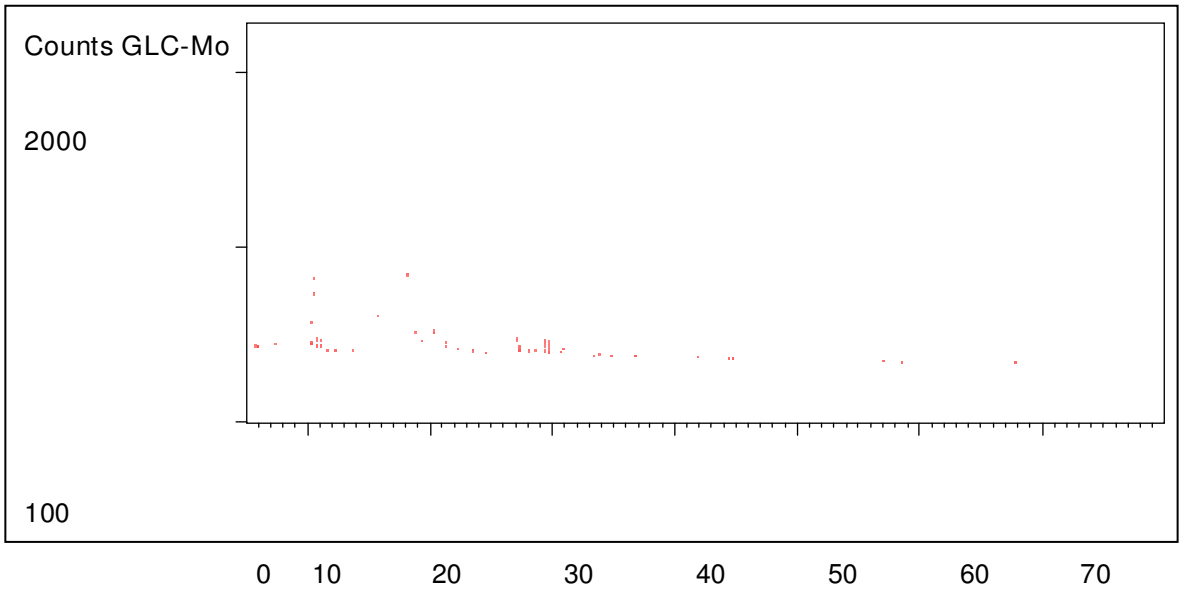


Figure.9.3: X-ray difractogram of GLC-Mo Complex

Table 9.1: Cell data and crystal parameter of GLC-Co complex

a(Å) = 21.6990 Volume(abcsinβ)Å= 13880.931
b(Å) = 23.1881 Dcal = 4.02100 g/cm³
c(Å) = 27.5891 Dobs = 4.03241 g/cm³
Standard deviation = 0.024% Crystal system = Monoclinic
α=90°, β=89.4°, λ=90° Porosity(%) = 2.837
Density = 0.05329g/cm³ Particle size = 15.794microns
Space group = Pmmm

2θ	I/I0	D(Obs)	D(Cal)	h	k	l
10.5540	69.89	8.38237	8.46731	1	0	3
16.3437	60.23	5.42369	5.42470	4	0	0
19.0109	99.40	4.66834	4.64949	2	3	4
19.8736	82.38	4.46760	4.46387	3	4	1
20.4293	53.81	4.34731	4.34770	1	4	4
22.0439	100.00	4.03241	4.02100	5	2	1
31.8513	52.99	2.80964	2.80664	7	1	4
39.3397	29.09	2.29037	2.28905	1	4	11
45.6199	37.40	1.98697	1.98754	9	3	7

Table 9.2: Cell data and crystal parameter of GLC-Cu Complex

a(Å) = 21.4126 Volume(a³)Å = 13855.00
b(Å) = 23.4321 Dcal = 2.80001 g/cm³
c(Å) = 27.6138 Dobs = 2.80932 g/cm³
Standard deviation = 0.031% Crystal system = Orthorhombic
α=90°, β=90°, λ=90° Porosity(%) = 0.3321
Density = 0.0511g/cm³ Particle size = 16.014microns
Space group = Pm

2θ	I/I0	D(Obs)	D(Cal)	h	k	l
10.9099	55.22	8.10977	8.24452	7	-1	2
16.3138	36.73	5.43355	5.48380	3	1	3
23.1943	32.13	3.83495	3.83060	-5	1	3
31.8550	100.00	2.80932	2.80001	-6	1	6
45.6099	31.43	1.98738	1.98747	0	11	5
56.7244	3.54	1.62288	1.62164	3	6	15
66.4314	7.33	1.40735	1.40608	5	5	4

Table 9.3: Cell data and crystal parameter of GLC-Mo Complex

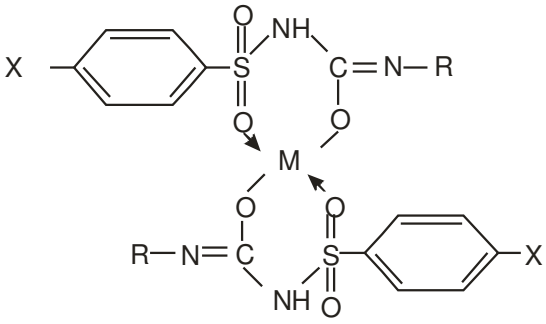
a(Å) =21.7628 Volume (abcsinβ)Å= 14071.30322
b(Å) = 23.4281 Dcal = 13.80660g/cm³
c(Å) = 27.6010 Dobs = 13.85955 g/cm³
Standard deviation = 0.0034% Crystal system = Monoclinic
α =90°, β=89.2°, γ =90° Porosity(%) = 0.3835
Density = 0.055209g/cm³ Particle size = 15.556microns
Space group = Pm Point group = m

2θ	I/I0	D(Obs)	D(Cal)	h	k	l
6.3774	100.00	13.85955	13.80660	1	1	1
10.8970	26.15	8.11932	8.26023	1	-2	2
11.4807	3.82	7.70780	7.65932	2	-2	1
14.4063	5.14	6.14843	6.16741	3	2	0
15.2123	5.68	5.82442	5.82736	2	0	4
16.9963	15.53	5.21656	5.21643	1	1	5
19.2834	30.43	4.60299	4.60017	0	0	6
19.9557	28.96	4.44940	4.43686	0	5	2
22.2511	26.35	3.99534	3.98622	4	4	0
23.4571	3.85	3.79257	3.80192	5	3	0
24.7115	3.22	3.60283	3.59621	6	0	1
28.6699	4.14	3.11376	3.11288	1	7	3
32.1833	1.47	2.78142	2.78123	3	7	4
33.5511	3.97	2.67108	2.67159	4	0	9
35.2684	2.14	2.54486	2.54429	3	4	9

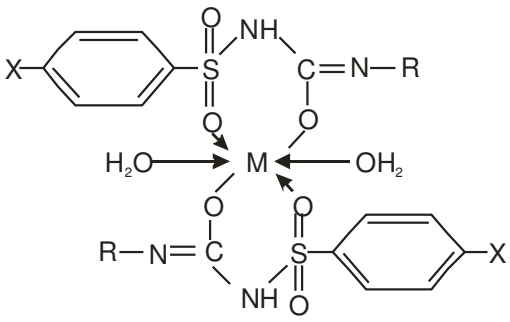
Conclusion:

On the basis of magnetic moment, crystal lactice parameters and spectral data complexes of Cu(II),) exhibit Planner structure were as Mo(II) and Co(II) complexes exhibit octahedral structure.

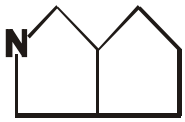
The proposed structure of **Co(II), Cu(II), MO(II),**complexes Sc as shown in the following figure.



M = Cu (Cu = Square Planner)



(M = Mo, Co (Octahedral)

X = CH_3 , R = 

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