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X-ray Diffraction Studies of Co(II), Sm(III) and Nd(III) Complexes with Gliclazide (*N*-(hexahydrocyclopenta[*c*] pyrrol-2(1*H*)-carbamoyl)-4-methylbenzenesulfonamide, An Oral Antidiabetic Drug

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ABSTRACT

Gliclazide(N-hexahydrocyclopentapyrrol-2-carbamoyl)-4-methylbenzenesulphonamide was used to synthesize Co(II),Sm(III) , Nd(III) complexes. Metal complxes were characterized by elemental analysis, IR, NMR,TGA. The crystal structure of complexes were further determined by X-ray diffraction method. The XRD data was used to calculate various parameters like crystal system, volume,density,porosity,particle size etc.which shows that the complexes of Co(II),Sm(III) and Nd(III) are octahedral structure.

Key words: Gliclazide, Crystal structure,.Co(II),Sm(III) and Nd(III) 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[1-6]. Many trasition and inner trasition metal complexes have been synthesized for analytical and commercial applications many of medicinal use[7-9,24-28]. Literature survey reveals that the transition and inner transition metal complexes generally crystallized with tetrahedral, octahedral geometry[10-12].

EXPERIMENTAL

All the chemicals used for the preparation of complexes are of Hi-media AR grade E-merk. 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 gliclazide were confirmed by Jobs method as modified by Turner and Anderson[13-14].

RESULTS AND DISCUSSION

The X-ray diffraction of Co(II),Sm(III) and Nd (III) complexes with Gliclazide were obtained and summarized in following tables. All reflections has been indexed for *h, k, l* values using reported literature[15-23] and full proof suit XRD software v.2.0 by using foolproof suite XRD software the d-values of metal complexes were obtained.

Table 1: Physico-chemical and analytical data of gliclazide complexes

S. No.	Composition of complex	Metal Ligand Ratio	Colour	% Yield	M.P. (°C)	% of Metal observed/ Required
1	$(C_{15}H_{20}N_3O_3S)_2Nd2H_2O$	1:2	Off White	45	192	17.48 (17.33)
2	$(C_{15}H_{20}N_3O_3S)_2Co2H_2O$	1:2	Pink	56	220	7.76 (7.76)
3	$(C_{15}H_{20}N_3O_3S)_2Sm\ 2H_2O$	1:2	Pale Yellow	53	216	18.08 (17.96)

S. No.	% of Carbon observed/ (Required)	% of H observed/ (Required)	% N observed/ Required	% of S observed/ Required	Stability constant log k lit/mole	Free Energy Change (-ΔF)
1	43.63 (42.80)	5.33 (4.86)	10.18 (9.90)	7.75 (6.77)	9.7219	-11.7438
2	48.66 (49.70)	5.84 (4.86)	11.35 (7.75)	8.65 (5.80)	10.6973	-13.7099
3	43.31 (42.70)	5.29 (4.86)	10.10 (9.70)	7.69 (7.21)	9.6677	-11.6692

Table 1: Cell data and crystal parameter of GLZ-Co complex

a(Å) = 21.6990
b(Å) = 23.1881
c(Å) = 27.5891
Standard deviation = 0.024%
α=90°, β=89.4°, γ =90°
Density = 0.05329g/cm^3

Volume(abcsinβ)Å= 13880.931
Dcal=4.02100 g/cm^3
Dobs= 4.03241 g/cm^3
Crystal system = Monoclinic
Porosity(%) = 2.837
Particle size = 15.794microns
Space group = Pm

2θ	I/I_0	$D_{(Obs)}$	$D_{(Cal)}$	*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 2: Cell data and crystal parameters for [$(GLZ)_2Sm2H_2O$] complex

a(Å) = 21.7621	Volume (abcsinβ)Å= 14065.269
b(Å) = 23.4271	Dcal = 13.86574 g/cm^3
c(Å) = 27.5913	Dobs = 14.17908 g/cm^3
Standard deviation = 0.0034%	Crystal system = Monoclinic(Octahedral)
α =90°, β=89.2°, γ =90°	Porosity(%) = 3.0055 %
	Density = 0.059094g/cm^3
Space group = Pm	Particle size =23.5720 microns

2 θ	I/I_0	D(Obs)	D(Cal)	h	k	l
6.2336	100	14.17908	13.86574	1	1	1
16.2587	34.89	5.45184	5.43999	4	0	0
28.1979	21.43	3.16480	3.16244	-6	1	4
29.3552	34.62	3.04261	3.04027	2	3	8
41.0622	19.06	2.19818	2.19539	-4	6	9
50.4845	14.68	1.80782	1.80644	8	3	11
58.0457	8.22	1.58780	1.58759	10	1	12

Table 3: Cell data and crystal parameter of GLZ-Nd Complex

a(Å) = 21.762	Volume (abcsin β)Å= 14065.307
b(Å) = 23.4271	Dcal = 13.86574 g/cm^3
c(Å) = 27.274	Dobs =14.26441 g/cm^3
Standard deviation = 0.0026%	Crystal system = monoclinic
α =90°, β=89.2°, γ =90°	Porosity(%) = 3.0055
Density = 0.0586592g/cm^3	Particle size = 15.484microns
Space group = Pm	

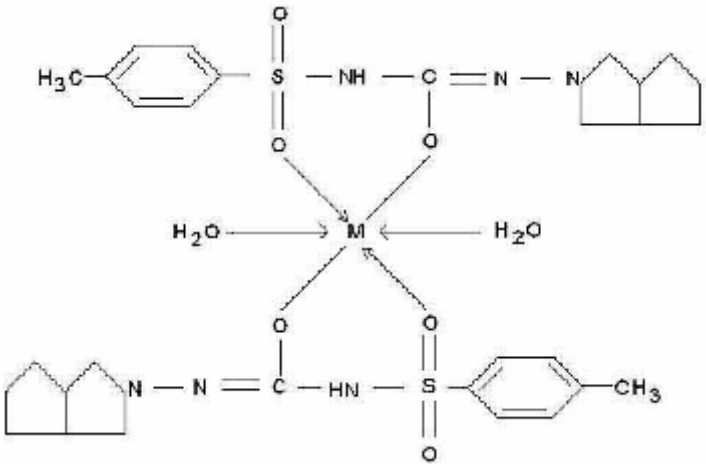
2θ	I/I_0	$D_{(Obs)}$	$D_{(Cal)}$	*h*	*k*	*l*
6.1963	100	14.26441	13.86574	1	1	1
16.0639	16.52	5.55751	5.51772	0	0	5
18.2478	2.92	4.86181	4.85591	-3	1	4
27.9094	10.13	3.19686	3.19271	-3	5	5
28.9225	15.47	2.76823	3.08474	-6	3	3
32.3407	2.16	2.42315	2.76573	7	3	3
37.1028	0.92	2.22463	2.42107	4	2	10
40.5523	8.43	2.09735	2.22284	3	10	1
43.1325	1.82	1.82716	2.09549	6	7	7
49.9132	6.76	1.76742	1.82558	-8	1	11
51.7224	0.86	1.61184	1.76595	12	3	0
57.1485	2.36	1.54030	1.61049	-6	13	1
60.0677	0.80	1.42229	1.53910	3	9	14

X-ray diffraction study of Gliclazide complexes

The X-ray diffraction pattern of Co(II), Sm(III) and Nd(III) complexes has been determined 2è range from 6.1963 to 79.97884°,Diffractograms (Fig-1,2,3,) and data has been summarized in the following table..

Table 4:

Molecular Formula	complexes	Mol^r Weight (gm/mole)	Crystal/ System
$(C_{15}H_{20}N_3O_3\ S)_2Co2H_2O$	Co(II)	739.75	Monoclinic
$(C_{15}H_{20}N_3O_3\ S)_2Nd2H_2O$	Nd(III)	825.06	Monoclinic
$(C_{15}H_{20}N_3O_3\ S)_2Sm2H_2O$	Sm(III)	831.18	Monoclinic



Where, M = Co(II),Sm(III) and Nd(III)

Scheme 1: Proposed structure of $(GLZ)_2Co2H_2O$, $(GLZ)_2Sm2H_2O$ and $(GLZ)_2Nd2H_2O$ complexes

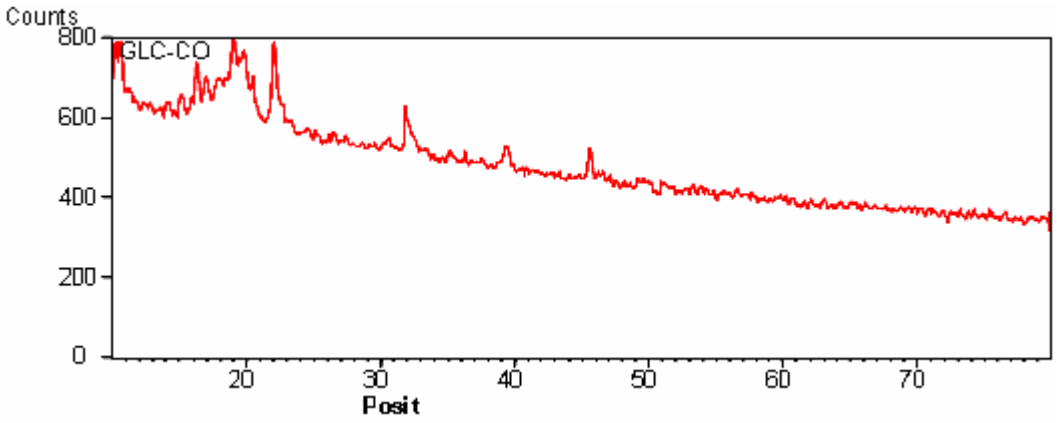


Fig. 1: X-ray difractogram of GLZ-Co Complex

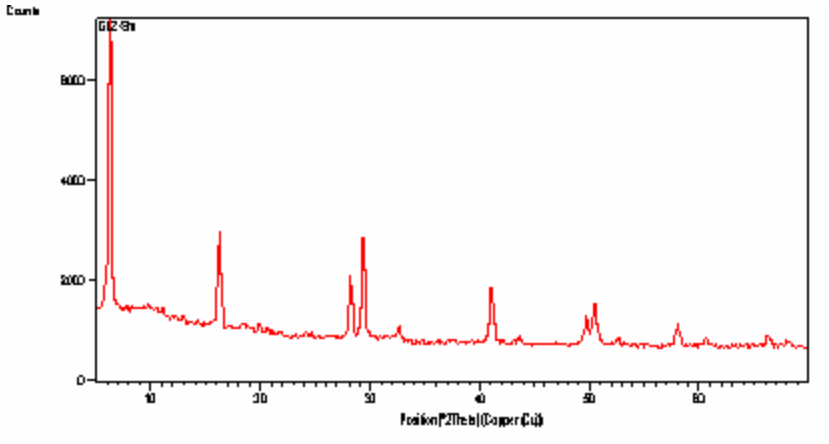


Fig. 2: X-ray difractogram of GLZ-Sm Complex

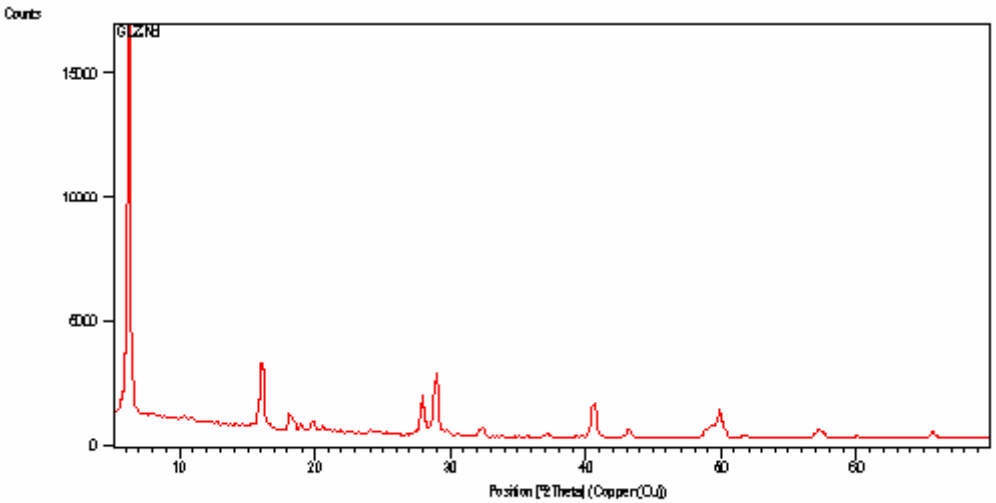


Fig. 3: X-ray difractogram of GLZ-Nd Complex

Gliclazide (GLZ-Co)

From the cell data and crystal lattice one can conclude that Co(II),Sm(III) and Nd(III) complex is having *monoclinic crystal*

CONCLUSION

X-ray diffraction studies also confirms the complexes and formation of new bonds. The number of peaks in Gliclazide are 22 while that of $(GLC)_2$ Nd $(GLC)_2$Co and $(GLC)_2$Sm. are 24,9 and 8 respectively . Thus indicating that complexes formed are a well kit one moreover the X-ray pattern of neither Gliclazide all the reflections present are new ones and the patterns are fairly strong. On comparing the pattern obtained with available literature. It is evident that its pattern is not in good agreement with available information and thus confirms the formation of totally new complexes The X-ray pattern have been indexed by using computer software(FPSUIT 2.0V) and applying interactive trial and error method keeping in mind the characterstics of the various symmetry system,till a good fit was obtained between the observed and the calculated $Sin^2è$ value.The unit cell parameters were calculated from the indexed data, from cell data and crystal lattice parameters of system. $(GLC)_2Co.2H_2O$ and $(GLC)_2Sm.2H_2O$ and $(GLZ)_2Nd2H_2O$ complexes attributed to Monoclinic crystal system .

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