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THERMAL AND XRD STUDY OF METAL COMPLEX OF *N*-(HEXAHYDROCYCLOPENTA[*C*]PYRROL-2(1*H*)-LCARBAMOYL) - 4-METHYLBENZENESULFONAMIDE

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Abstract

The present work describes Thermal and XRD studies of cobalt complex of *N*-(hexahydrocyclopenta[*c*]pyrrol-2(1*H*)-ylcarbamoyl)-4-methylbenzenesulfonamide or Gliglazide, an oral antidiabetic drug. Complex of cobalt was synthesized and characterized by TGA and X-ray diffraction Metal ligand ratio was determined by Monovariation method indicate the formation of non-ionic complex and L_2M (2:1) type and further confirmed by Job's method of continuous variation. Analytical data agrees with the molecular formula of complex viz.$(C_{15}H_{20}N_3O_3S)_2Co2H_2O$. Structure (Scheme-2) have been discussed and suggested upon Elemental analysis, Electronic spectra. Powder X-ray diffraction data was performed on metal complex. XRD pattern indicate crystalline nature of complex. XRD data have been used to calculate particle size, porosity, volume of unit cell and density of synthesized complex. The freeman-Carroll and Sharp-Wentworth methods was used to calculate activation energy and thermal stability. Thermodynamic parameters such as free energy change (ΔG), entropy change (-ΔS), order of reaction (n) is also determined on the basis of TGA/DTA curves by using data of the Freeman-Carroll method.

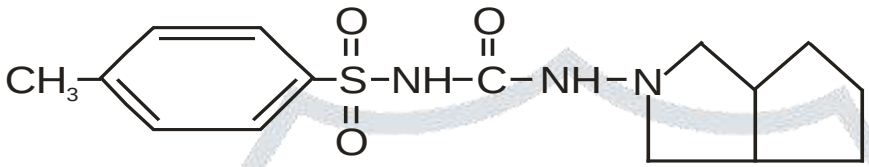
Keywords: G*liclazide, complex, TGA and X-Ray diffraction*

1. INTRODUCTION

Gliclazide is an important anti-diabetes medicine Metal ions are required for many critical functions in humans. Scarcity of some metal ions can lead to disease. Well known example are *pernicious anemia* resulting from iron deficiency; growth retardation arising from insufficient dietary of zinc, and heart disease in infants owing to copper deficiency. Diabetes is a deceptive disease and if not detected in early stage may cause even death. It is considered hereditary but actual genetic disorder is still a mystery. Several million people are suffering from this disease all over the world. Zinc- insulin was discovered as early as in 1921 and later it proved to be a very efficacious medicine in the treatment of diabetes mellitus 1. To avoid the daily pricks of hypodermic syringe, oral hypoglycemic agents were discovered which has revolutionized the treatment of diabetes. It is worthwhile

to mention here that the majority of the essential metallic elements of biological importance are transition metals, whose ability to form coordination complexes and chalets are the characteristic aspects of their chemistry.

In recent years much attention is given to the use of sulphonylureas because of their high complexing nature with essential metals (3-4). Sulphonylureas are effective for non- insulin dependent *diabetes mellitus*. These compounds are completely absorbed on oral administration. They are metabolized by liver and are excreted predominantly through urine. Complexation of sulphonylureas with lighter transition metals has been studied in detail by Yoshinaga and Yamamotto 5-6 and Iqbal *at.el 7-8* ., It is interesting to have an insight into the synthesis of cobalt complex with gliclazide and to diagnose various structural aspects of the isolated complex . The general structure of gliclazide is given in Scheme-1.



Scheme-1 Structure of gliclazide

2. EXPERIMENT

2. 1.LIGAND-METAL RATIO

a)Pure Gliclazide 1.617g (0.005M) was dissolved in 100ml of ethanol, and metal salt in same solvent.10ml ligand solution was diluted in 100ml of ethanol in a beaker. This was titrated conductometrically against cobaltus chloride solution taken in burette using fractions of 1ml. Conductance was recorded after each addition with proper stirring at temperature 30±1 ^{0}C. Results were plotted in the form of a graph between corrected conductance and volume of metal salt. From the equivalent point in the graph, ratio between metal and ligand was noted to be 1:2.

b)Formation of 2:1 (L_2M) ratio was further confirmed by Job's method (9) of continuous variation as modified by Turner and Anderson 10 (Table-1 and Fig.1) using absorbance as index property, from these values the stability constant (log k) and free energy change (-ΔF), were also calculated by using formula $$k = \frac{x}{(a_1 - x)(b_1 - 2x)^2} = \frac{x}{(a_2 - x)(b_2 - 2x)^2}$$

Where, k = Stability constant, x = Conc. of complex and $\Delta G = -RT \ln k$ respectively

Table 1 Glicalzide with cobaltus chloride(Modified Job's method)

S. No.	Metal:Ligand ratio	Absorbance		Corrected Absorbance	
		0.002M	0.005M	0.002M	0.005M
1	0:12	0.012	0.019	0.00	0.00
2	1:11	0.043	0.059	0.031	0.040
3	2:10	0.087	0.109	0.071	0.090
4	3:9	0.129	0.179	0.117	0.160
5	4:8	0.192	0.240	0.180	0.221
6	5:7	0.16	0.172	0.148	0.153
7	6:6	0.132	0.155	0.120	0.136
8	7:5	0.121	0.141	0.109	0.122
9	8:4	0.099	0.13	0.087	0.111
10	9:3	0.077	0.101	0.063	0.082
11	10:2	0.038	0.098	0.043	0.078
12	11:1	0.043	0.056	0.027	0.035
13	12:0	0.019	0.023	0.00	0.00

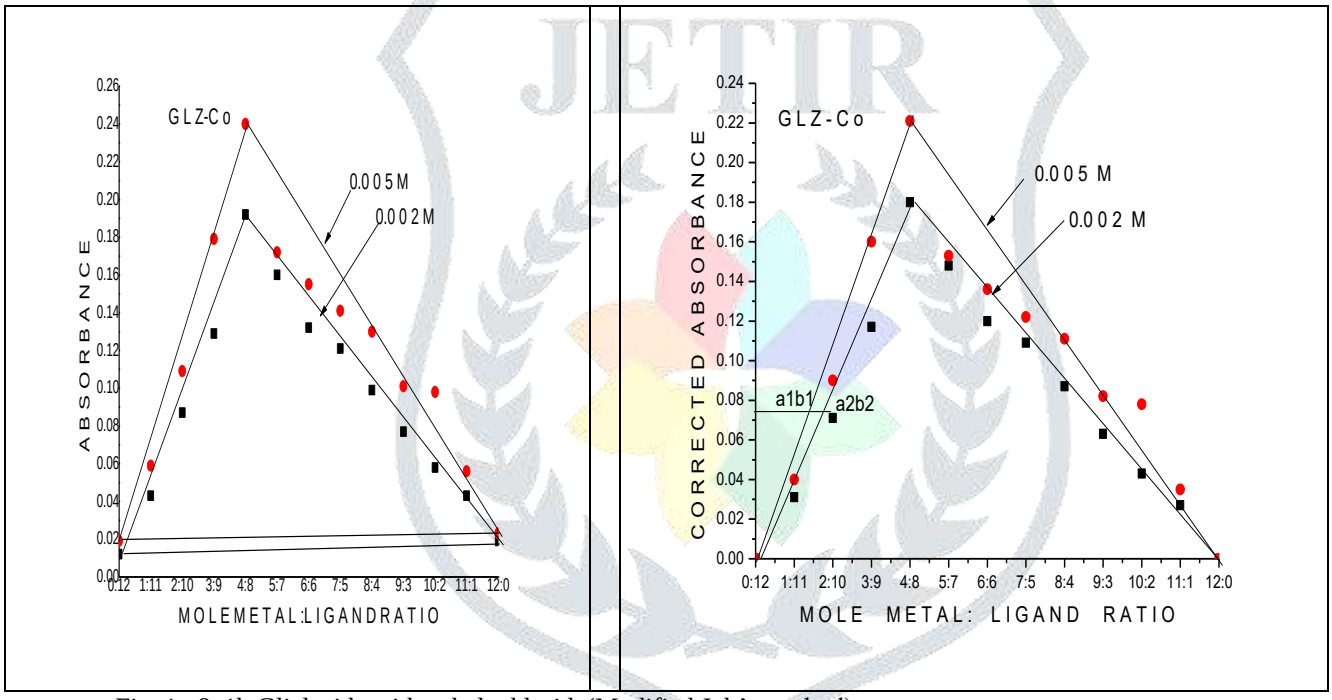


Fig. 1a & 1b Gliclazide with cobalt chloride(Modified Job's method)

2.2 SYNTHESIS OF COMPLEX

a) The chemicals used in synthesis were all of AnalaR grade. Pure 1.617 g. "gliclazide" (L) and 0.594 g. of cobaltus chloride were dissolved separately in 90% ethanol. The ligand solution was added gradually to the cobaltus chloride solution with constant stirring, pH of the solution was adjusted (6.0) by adding dilute NaOH solution. A pink coloured precipitate was obtained. The whole solution was reflux for three and half hour, cooled and filtered. A pink coloured crystalline complex was formed. The complex form was washed with alcohol and dried. Finally the weight was taken (Yield, 56.57%).

2.3 Instrumentation

The elemental analysis of the isolated complex was carried out using Coleman Analyzer at the Departmental Microanalytical Laboratory C.D.R.I, Lucknow.. metal analysis was carried out in Qualichem Laboratory, Nagpur, by atomic absorption spectroscopy (AAS).. X-ray diffractometer model Rigaku D-max/B, with 12 kW Rotating Anode X-ray generator was used for scanning the ligand and respective complex. Anode X-ray generator was used for scanning the ligand and respective complex at SAIF, Punjab University, Chandigarh, India. The radiation used was Cu, Ka (1W = 1.5060 °A). The sample was scanned

in the range from 10.0084 to 79.9804 (2ɵ). Powder data were indexes using computer software (FPSUIT V 2.0). TGA spectra were recorded in IIT Bombay, India.

From stoichoimetry and analytical data, the composition of the complex comes out to be $(C_{15}H_{20}N_3O_3S)_2Co.2H_2O$ for which favours 2:1 (L_2M) ratio. The tentative structure (9) assigned to complex on the basis of analytical data, X-ray and TGA.

3. RESULTS

3.1 Physico-chemical data of gliclazide-cobalt complex: Yield: 56.57%; m.p:180 ºC; colour: pink; –ΔF: 16.65; **Log**K: 11.041; Anal.Calcd for $(C_{15}H_{20}N_3O_3S)_2$ Co2H_2O:C,42.26; H,5.64; N,8.60; S,8.50; M,7.98 Found:C,43.66; H,5.40;N, 8.35; S,8.65; M,7.70.

3.2 Electronic spectral Studies

UV-VIS(EtOH) (λ_{max} / nm) The electronic spectrum of Co(II) complex shows three bands at 300-700 nm due to $^4T_{1g} \longrightarrow {}^4T_{2G}$, $^4T_{1g} \longrightarrow {}^4A_{2G}$,and $^4T_{1g} \longrightarrow {}^4T_{1g}$.

3.3 Thermal Study of gliclazide-cobalt complex

Thermal study gives quick information about the complex that it contains coordination water molecule. In the present investigation, the weight loss for complex was calculated within corresponding temperature ranges. The complex is thermally decomposed in three decomposition steps within the temperature range of 50-600°C. The TGA/DTA curves for the complexes are shown in Figure. 2.

The thermoanalytical data are presented in table-2 and 3. In studying the decomposition, kinetics methods mentioned in the literature was used in each case, the least square plots were drawn. The first few points that did not fall on straight line were discarded. These types of deviations of points are reported in literature by several research workers 11-13**.** This is explained as due to the failure of obeying as first order kinetics always by the solids in their decomposition in the early stages.

Theoretical Consideration

To provide further evidence regarding the degradation system of analyzed compound we derived the TG curves by applying an analytical method proposed by **Freeman-Carroll** 14 and **Sharp-Wentworth** 16

Freeman-Carroll Method

The straight-line equation derived by Freeman and Carroll, which is in the form of

$$\frac{\Delta log\frac{dw}{dt}}{\Delta log\, Wr} = n - \frac{E_a}{2.303R}.\frac{\Delta\frac{1}{T}}{\Delta log\, Wr}$$

Where, $\frac{dw}{dt}$ = rate of change of weight with time, Wr = Wc-W

W_c = Wt. loss at completion of reaction, W = Total wt. loss up to time‘t’

E_a = Energy of activation, n = Order of reaction

The plot between the term $\frac{\Delta log\frac{dw}{dT}}{\Delta \log Wr}$ Vs $\frac{\Delta\frac{1}{T}}{\Delta \log Wr}$ gives a straight line (Figure. 3) from which slope can be calculated. The energy of activation (Ea) and intercept on Y-axis as order of reaction (n) and entropy are calculated.

Sharp-Wentworth Method

Using the equation derived by Sharp and Wentworth

$$\frac{\Delta log\frac{dc}{dT}}{(1-c)} = log\frac{A}{B} - \frac{Eq}{2.303\,R}.\frac{1}{T}$$

Where, $\frac{dc}{dT}$ = Rate of change of fraction of weight with change in temperature.

β = Linear heating rate $\frac{dT}{dt}$, By plotting the graph between

$$\frac{\Delta log\frac{dc}{dT}}{(1-c)} \text{ Vs } = \frac{1}{T},$$

We obtained the straight line (Fig.4) which gives energy of activation (Ea) from its slope.

The thermodynamic activation parameters of decomposition process of dehydrate complex namely activation energy (Ea), Entropy (ΔS) and Gibb's free energy change of decomposition (ΔG°) are evaluated graphically by employing Freeman-Carroll and Sharp-Wentworth relation. The data are summarized in Table -4. The activation energies of decomposition are found to be in the range 33 to109 KJ Mol^{-1}. The high value of activation energies reflects the thermal stability of complex. The entropy of activation is found negative values in complex which indicate that decomposition reactions process with lower rate than the normal ones beside these parameters, in TGA curve the initial loss is due to water of crystallization in complex.

Table-2 TGA data of gliclazide cobalt complex by Sharp-wentworth method

Temp. (°C)	Temp (°K)	$\frac{1000}{T}$	% Mass Loss	Change in Wt. 'c' grams	1-c	$\frac{dc}{dt}$	$log\frac{dc}{dt}$	$log(1-c)$	$\frac{log\left(\frac{dc}{dt}\right)}{(1-c)}$	Weight (%)
30	303	3.30033	1.381	0.00019	0.99981	0.00036	-3.44273	-0.00008	-3.44338	98.619
50	323	3.09598	2.928	0.00040	0.99960	0.00057	-3.24651	-0.00017	-3.24780	97.072
70	343	2.91545	4.752	0.00065	0.99935	0.00075	-3.12209	-0.00028	-3.12411	95.248
90	363	2.75482	6.499	0.00088	0.99912	0.00089	-3.05055	-0.00038	-3.05325	93.501
110	383	2.61097	7.842	0.00107	0.99893	0.00100	-2.99942	-0.00046	-3.00262	92.158
130	403	2.48139	8.928	0.00121	0.99879	0.00113	-2.94832	-0.00053	-2.95191	91.072
150	423	2.36407	10.064	0.00137	0.99863	0.00128	-2.89309	-0.00060	-2.89705	89.936
170	443	2.25734	11.414	0.00155	0.99845	0.00146	-2.83590	-0.00067	-2.84032	88.586
190	463	2.15983	13.007	0.00177	0.99823	0.00172	-2.76460	-0.00077	-2.76951	86.993
210	483	2.07039	15.239	0.00207	0.99793	0.00213	-2.67264	-0.00090	-2.67819	84.761
230	503	1.98807	18.666	0.00254	0.99746	0.00256	-2.59115	-0.00110	-2.59774	81.334
250	523	1.91205	22.575	0.00307	0.99693	0.00295	-2.52985	-0.00134	-2.53764	77.425
270	543	1.84162	26.213	0.00357	0.99643	0.00328	-2.48401	-0.00155	-2.49290	73.787
290	563	1.77620	29.356	0.00399	0.99601	0.00355	-2.45015	-0.00174	-2.45997	70.644
310	583	1.71527	31.94	0.00435	0.99565	0.00374	-2.42724	-0.00189	-2.43783	68.060
330	603	1.65837	33.869	0.00461	0.99539	0.00390	-2.40885	-0.00201	-2.42001	66.131
350	623	1.60514	35.443	0.00482	0.99518	0.00405	-2.39225	-0.00210	-2.40384	64.557
370	643	1.55521	36.875	0.00502	0.99498	0.00420	-2.37703	-0.00218	-2.38901	63.125
390	663	1.50830	38.224	0.00520	0.99480	0.00435	-2.36200	-0.00226	-2.37435	61.776
410	683	1.46413	39.58	0.00539	0.99461	0.00448	-2.34843	-0.00235	-2.36114	60.420
430	703	1.42248	40.865	0.00556	0.99444	0.00464	-2.33339	-0.00242	-2.34643	59.135
450	723	1.38313	42.283	0.00575	0.99425	0.00480	-2.31835	-0.00251	-2.33177	57.717
470	743	1.34590	43.768	0.00596	0.99404	0.00488	-2.31180	-0.00259	-2.32565	56.232
490	763	1.31062	44.602	0.00607	0.99393	0.00493	-2.30734	-0.00264	-2.32143	55.398
510	783	1.27714	45.139	0.00614	0.99386	0.00495	-2.30544	-0.00268	-2.31969	54.861

Table 3.Thermogravimetric data of gliclazide-cobalt complex by Freeman and Carroll method

Temp. (°C)	% Mass Loss	Change in Wt. (gm.)	Time in Sec.	$\frac{dw}{dt}$	$log\frac{dw}{dt}$	wr = wc-w	log wr	T(K)	$\frac{1}{T}(K^{-1})$	$\frac{log\frac{dw}{dt}}{log w_r}$	$\frac{\frac{1}{T}}{log w_r}$	$\alpha = \frac{wt}{wc}$	$\frac{1-(1-\alpha)^{1-n}}{1-n}$	$T^3 \times 10^{-7}$	$\frac{g(\alpha)}{T^3 \times 10^7}$	$\frac{1}{T} \times 10^{-3}$	$log\left(\frac{g(\alpha)}{T^3}\right)$
30	1.381	0.00019	90	0.000389	-3.410	0.00599	-2.2226	303	0.00330	1.534	-0.0015	0.0304	0.0309	2.7818	0.0003	3.3003	-108.8234
50	2.928	0.00040	150	0.000627	-3.203	0.00578	-2.2381	323	0.00310	1.431	-0.0014	0.0645	0.0666	3.3698	0.0013	3.0960	-70.2398
70	4.752	0.00065	210	0.000852	-3.070	0.00553	-2.2572	343	0.00292		-0.0013	0.1047	0.1104	4.0354	0.0029	2.9155	-48.0044
90	6.499	0.00088	270	0.001023	-2.990	0.00529	-2.2763	363	0.00275	1.314	-0.0012	0.1431	0.1542	4.7832	0.0046	2.7548	-34.6226
110	7.842	0.00107	330	0.001161	-2.935	0.00511	-2.2915	383	0.00261	1.281	-0.0011	0.1727	0.1892	5.6182	0.0058	2.6110	-26.4437
130	8.928	0.00121	390	0.001309	-2.883	0.00496	-2.3043	403	0.00248	1.251	-0.0011	0.1966	0.2185	6.5451	0.0066	2.4814	-20.8855
150	10.064	0.00137	450	0.001485	-2.828	0.00481	-2.3180	423	0.00236	1.220	-0.0010	0.2216	0.2500	7.5687	0.0073	2.3641	-16.6010
170	11.414	0.00155	510	0.001692	-2.772	0.00462	-2.3349	443	0.00226	1.187	-0.0010	0.2514	0.2887	8.6938	0.0083	2.2573	-13.1039
190	13.007	0.00177	570	0.001985	-2.702	0.00441	-2.3558	463	0.00216	1.147	-0.0009	0.2865	0.3364	9.9253	0.0097	2.1598	-10.2373
210	15.239	0.00207	630	0.002436	-2.613	0.00410	-2.3868	483	0.00207	1.095	-0.0009	0.3356	0.4072	11.2679	0.0121	2.0704	-7.6705
230	18.666	0.00254	690	0.002945	-2.531	0.00364	-2.4391	503	0.00199	1.038	-0.0008	0.4111	0.5267	12.7264	0.0170	1.9881	-5.2213
250	22.575	0.00307	750	0.003413	-2.467	0.00311	-2.5078	523	0.00191	0.984	-0.0008	0.4972	0.6828	14.3056	0.0237	1.9120	-3.2795
270	26.213	0.00357	810	0.003816	-2.418	0.00261	-2.5831	543	0.00184	0.936	-0.0007	0.5773	0.8538	16.0103	0.0308	1.8416	-1.9191
290	29.356	0.00399	870	0.004146	-2.382	0.00218	-2.6608	563	0.00178	0.895	-0.0007	0.6465	1.0292	17.8454	0.0373	1.7762	-0.9913
310	31.94	0.00435	930	0.004391	-2.357	0.00183	-2.7371	583	0.00172	0.861	-0.0006	0.7034	1.2009	19.8155	0.0426	1.7153	-0.3698
330	33.869	0.00461	990	0.004592	-2.338	0.00157	-2.8042	603	0.00166	0.834	-0.0006	0.7459	1.3515	21.9256	0.0460	1.6584	0.0161
350	35.443	0.00482	1050	0.004776	-2.321	0.00136	-2.8679	623	0.00161	0.809	-0.0006	0.7806	1.4941	24.1804	0.0482	1.6051	0.2762
370	36.875	0.00502	1110	0.004950	-2.305	0.00116	-2.9353	643	0.00156	0.785	-0.0005	0.8121	1.6444	26.5848	0.0502	1.5552	0.4726
390	38.224	0.00520	1170	0.005125	-2.290	0.00098	-3.0101	663	0.00151	0.761	-0.0005	0.8418	1.8106	29.1434	0.0523	1.5083	0.6281
410	39.58	0.00539	1230	0.005291	-2.276	0.00079	-3.1010	683	0.00146	0.734	-0.0005	0.8717	2.0119	31.8612	0.0550	1.4641	0.7657
430	40.865	0.00556	1290	0.005475	-2.262	0.00062	-3.2092	703	0.00142	0.705	-0.0004	0.9000	2.2505	34.7429	0.0583	1.4225	0.8823
450	42.283	0.00575	1350	0.005667	-2.247	0.00042	-3.3718	723	0.00138	0.666	-0.0004	0.9312	2.6067	37.7933	0.0642	1.3831	1.0191
470	43.768	0.00596	1410	0.005771	-2.239	0.00022	-3.6522	743	0.00135	0.613	-0.0004	0.9639	3.2148	41.0172	0.0756	1.3459	1.1975
490	44.602	0.00607	1470	0.005838	-2.234	0.00011	-3.9616	763	0.00131	0.564	-0.0003	0.9823	3.8765	44.4195	0.0857	1.3106	1.3073
510	45.139	0.00614	1530	0.005871	-2.231	0.00004	-4.4414	783	0.00128	0.502	-0.0003	0.9941	4.8845	48.0049	0.1012	1.2771	1.4296

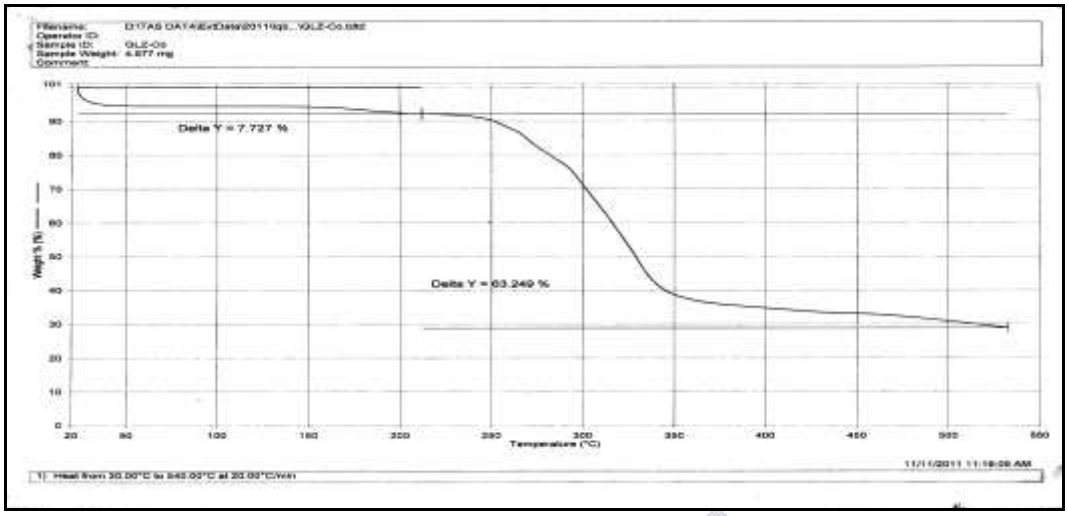


Fig 2 TGA Curve of gliclazide-cobalt complex.

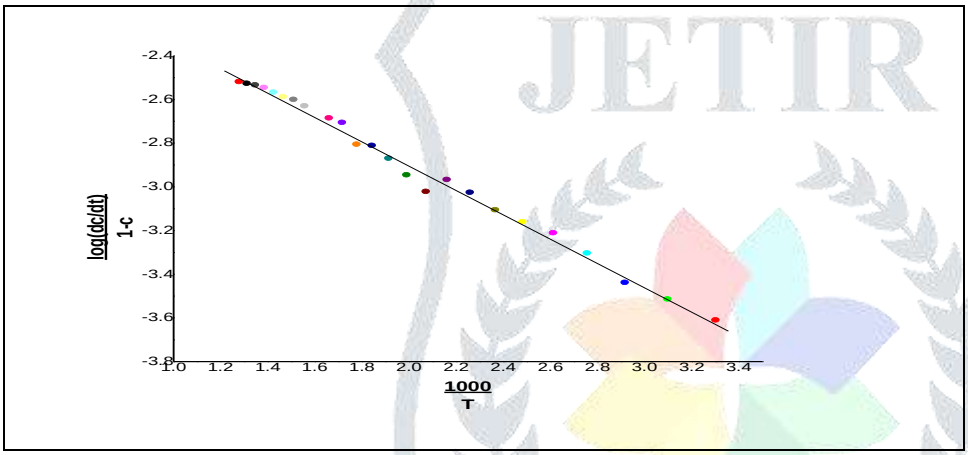


Fig. 3 Determination of activation energy by SW method

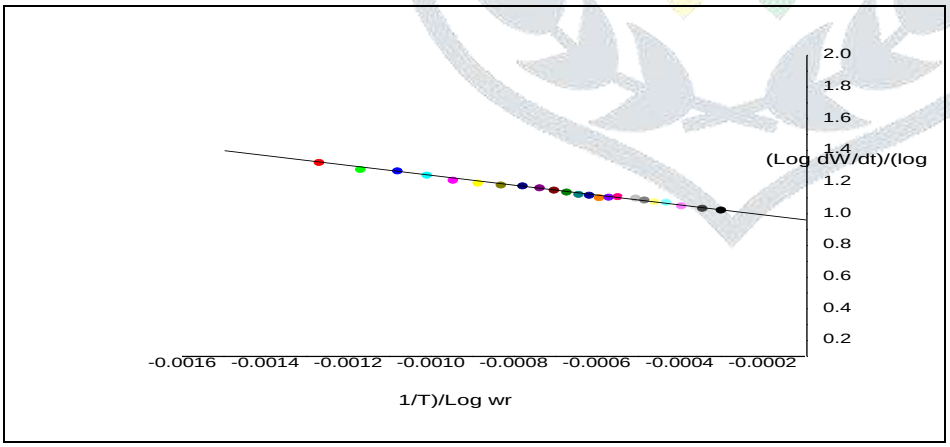


Fig.4 Determination of order of reaction and activation energy by FC method

Table 4. Thermogravimetric data of gliclazide-cobalt complex with corresponding to heating rate of 10°C/min**.**

Complexes	Decomposition Temp. (°C)	%Wt. loss	Ea(Kj/mole)		ΔS (Kj/mole)	ΔG (Kj/mole)	n
			F.C.	S.W.			
$(C_{15}H_{20}N_3O_3S)_2Co.2H_2O$	30-150	5.815	33.67	32.66	-33.98	10.26227	0.98
	150-350	51.027	55.14	54.38	-82.07	34.66047	
	350-510	69.718	109.37	108.38	-116.8	72.65703	

3.3X-Ray Diffraction Studies.

X-ray diffraction (figure. 5) studies also confirm the complex formation.16-18 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 characteristics of the various symmetry system, till a good fit was obtained between the observed and the calculated $Sin^2\theta$ value. The unit cell parameters were calculated from the indexed data, from cell data and crystal lattice parameters of $(GLZ)_2Co$, indicates complexes attributed to Monoclinic crystal system Table 5. Powder x-ray diffraction patterns indicate that the synthesized complex is monoclinic. The particle size is 9.35nm of "gliclazide"-cobalt, which is calculated from x-ray line broadening using the Scherrer formula $Dhkl = \kappa\lambda/\beta hkl \cos\theta$, where D is the particle diameter in ångstroms, κ is a coefficient and is equal to 0.89 here, β is the half-maximum line width, and λ is the wavelength of x-rays, porosity is 0.0283 calculated by formula $\frac{d_{true} - d_{obs} \times 100}{d_{tru}}$ and volume of the unit cell is Volume(abc sinβ)Å= 13880.931 where a, b and c are lattice parameters . Density = $\frac{Weight}{Volume}$ is found = 0.05329 g/cm^3. Space group is P_{mmm} and α=90°, β=89.4°, γ=90°.

Table 5 Cell data and crystal parameter of gliclazide-cobalt complex

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

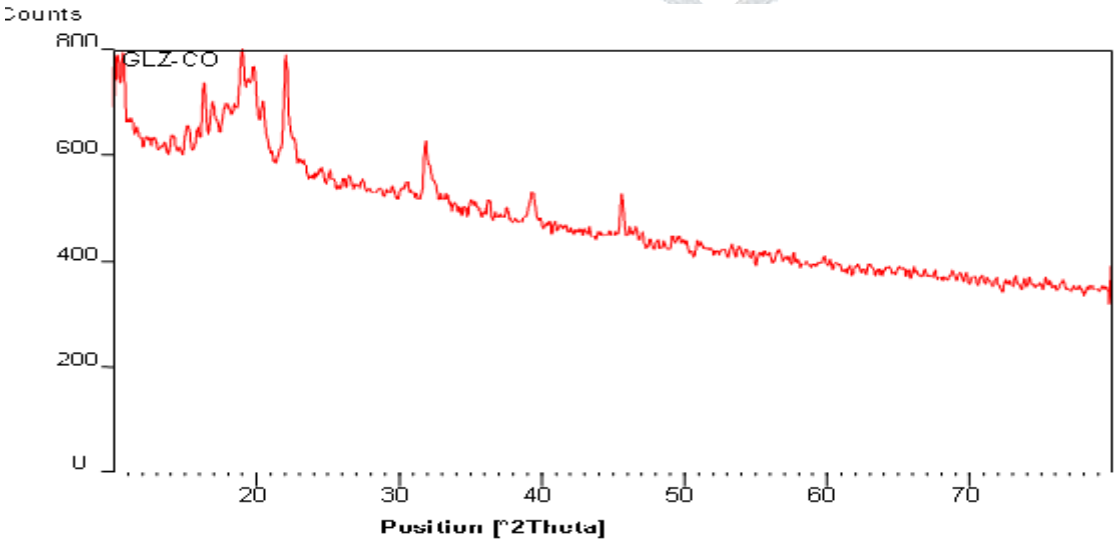


Fig 5 X-ray difractogram of gliclazide-cobalt complex.

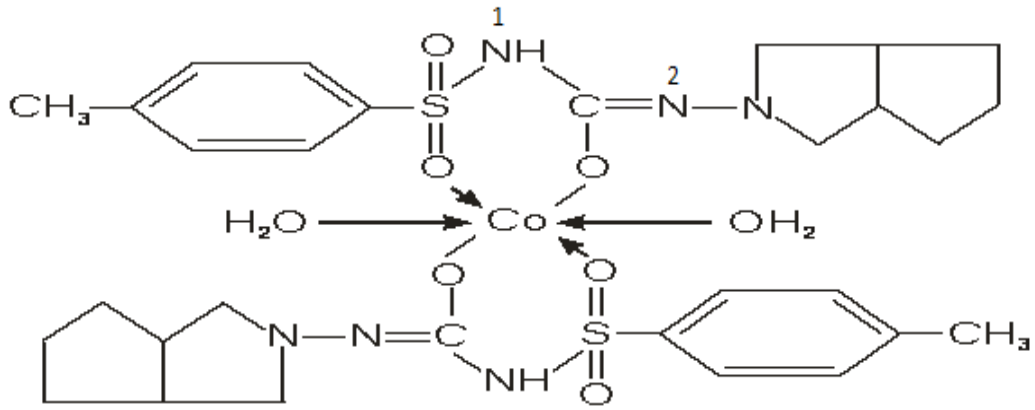
Discussion

For supporting the proposed structure (scheme-2) of cobalt-gliclazide complex, initially Job's method of continuous variation as modified by Turner and Anderson was conducted which indicate 2:1 ligand:metal ratio of the complex , moreover stability constant and free energy change was also calculated. Analytical data agrees to the molecular formula $(C_{15}H_{20}N_3O_3S)_2Co.2H_2O$ (L_2M) .

A detailed study of X-Ray (Table-5) also supports the complex formation and the various values like particle size, porosity, volume of unit cell, density as well as crystal system was evaluated. Thermal analysis has been studied by applying the Sharp-Wentworth and Freeman–Corrol methods: energy of activation (E_a), Kinetic parameter viz. -ΔS, -ΔF and order of reaction (n) were determined by applying Freeman-Corroll method. The complex formation is a first order reaction as indicated by value of n=0.98

CONCLUSION

In present paper we have syntheses complex of antidiabetic drug with cobalt and have given its spectral characterization and kinetics parameters which gives the detailed information of the new structure for coordination chemistry. Moreover we have carried out comparative study for hypoglycemic activity of parent drug and its complex with transition metals and found that the complex are more potent than pure drug. If by doing some clinical action it can be used for clinical point of view and it will be a new drug for diabetes patients.



Scheme-2 Proposed structure of cobalt complex with gliclazide

Declaration-No conflict of interests regarding the publication of this paper

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References

1] Underwood, E. J. 1971. Trace Element in Human and Animal Nutrition, Academic Press, New York, NY, USA, 3rd edition.

[2] Sadilot S. M. and Pathak, R. B. 1992. Journal of Diabetes Association, 32(4).

[3] Bloomgarden, Z. N. 91999 American Diabetes Association Consensus Statement on pharmacologic treatment, Diabetes Care, 22, S1–S114.

[4] Sanger, F and Thompson, E. O. L. 1951. The Biochemical Journal, 53, 366–535.

[5] Yoshinaga I. and Yamamotto, Y. 1966a. Endocrinologie, 50(3).

[6] Yoshinaga I. and Yamamotto, Y. 1966b. J.Osaka. 1(3).

[7] Iqbal, S. A., Jose, S.and Zaafarany, I. 2012. Synthesis, physicochemical and spectral studies of mercury complex of glibenclamide: an oral antidiabetic drug, Oriental Journal of Chemistry, 28(1), 613–618.

[8] Veer, D. and Baboo, M. 2012. Synthesis and spectral studies of some transition metal complexes, Oriental Journal of Chemistry, 28(4), 1797–1802.

[9] Job, P. 1928. Formation and stability of inorganic complexes in solution," Annales de Chimie, 10, 113.

[10] Turner, S. E. and Anderson, R. C. 1949. Spectrophotometric studies on complex formation with sulfosalicylic acid. III. with Copper(II)," Journal of the American Chemical Society, 71(3), 912–914.

[11] Singru, R. N. 2011. synthesis, characterization and thermal degradation studies of copolymer resin p-cresol-oxamideformaldhyde, Archives of Applied Science Research, 3(5), 309–325.

[12] Rahangdale, S. S. and Gurnule, W. B. 2013. Thermal and electrical properties of 2,2-HBBR Copolymer Resin, Chemical Science Transactions, 2(1), 287–293.

[13] Duval, D. 1983. Inorganic Thermogravimetric Analysis, Elsevier, London, UK.

[14] Freeman E. S. and Carroll, B. 1958. The application of thermoanalytical techniques to reaction kinetics: the thermogravimetric evaluation of the kinetics of the decomposition of calcium oxalatemonohydrate, The Journal of Physical Chemistry, 62(4), 394–397.

[15] Sharp J. H. and Wentworth, S. A. 1969. Kinetic analysis of thermogravimetric ata, Analytical Chemistry, 41(14), 2060–2062.

[16] Bragg W. L. and Bragg, W. H. 1993. The Crystalline State: A General Survey, George Bell & Sons, London, UK.

[17] Guiner, A. 1992, X-Ray Crystallographic Technology,Hilger andWatts, London, UK.

[18] M.Henry, N. F., Lipson, H. and. Wooster, W. A. 1951 The Interpretation of X-Ray Diffraction Photographs, Macmillan, London, UK.