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X-ray powder diffraction and thermal studies on Copper (II) and Nickel (II) complexes using Schiff base derived from 2-phenyl-3-{[(1*E*)-1-(2-oxo-2*H*-benzo[*F*]chromen-3-yl) ethylidene] amino}quinazolin-4(3*H*)-one

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ABSTRACT

The Cu (II), Ni (II) complexes of Schiff bases derived from 2-Phenyl-3-{[(1*E*)-1-(3-oxo-3*H*-benzo[*h*]chromen-3-yl)ethylidene]amino}quinazolin-4(3*H*)-one have been synthesized and characterized by analytical data. The complexes are monomeric, possess in 1:2 (metal: ligand) stoichiometry and are non-electrolytic in nature. The Cu (II) complexes have been characterized by thermal analysis and X-ray diffractometry. The ligand and its Ni(II) complex have been characterized by powder X-ray diffraction. The thermal analysis of Ni (II) complex has also been carried out.

Key words: Cu (II), Ni (II) complexes, Schiff base, X-ray powder diffraction.

INTRODUCTION

Schiff bases possess strong ability to form metal complexes and they deserve a proper attention because of their Biological properties [1,2]. Metal complexes with potentially tridentate and tetra dentate ligands have evoked much interest in coordination chemistry. The synthesis, thermal and X-ray diffraction studies of Cu (II) and Ni (II) complexes with 2-Phenyl-3-{[(1*E*)-1-(3-oxo-3*H*-benzo[*h*]chromen-2-yl)ethylidene]amino}quinazolin-4(3*H*)-one (L) were under taken in the present investigation.

RESULTS AND DISCUSSION

The analytical data of Cu (II) and Ni (II) complexes suggest their general formula [M (L). 2 $Cl(H_2O)$] with their molecular formula $Cu(C_{29}H_{21}N_3O_4Cl_2)$, Ni ($C_{29}H_{21}N_3O_4Cl_2$) at respectively where L= (2-Phenyl-3-{[(1*E*)-1- (L=3-oxo-3*H*-benzo[f] chromen-3-yl) ethylidene]amino}quinazolin-4(3*H*)-one). The diffractogram of Cu (II) complex records 16 reflections are 0 to 80° (2θ) with maxima at 2θ = 25.26° corresponding to value of d = 3.522Å (Table 1). The diffractogram of Ni (II) complex consists of 10 reflections with maxima at 2θ = 26.77° corresponding to the value of d=3.3272Å (Table 2). The inter planar spacing (d) has been calculated from the positions of intense peaks using Braggs relation $n\lambda=2d \sin\theta$ (where λ = wavelength of X-ray used CuKα = 1.5406 (Å). The calculated spacing together with relative intensities with respect to most intense peaks are recorded in the Table 1 and 2. All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was found that there is a good agreement between the calculated and observed values. The unit cell calculations have been carried out for the cubic symmetry of the compound. The characteristic of cubic system is that $\sin^2\theta$

Table: 1 XRD data of Cu[(L). 2Cl (H-$_2$O)]

Peak No.	2θ Degree	θ	Sin θ	Sin² θ	h²+k²+l²	h k l	Calc. (d)	Obsr (d)	Intensity	a in Å
1.	5.69531	2.84766	0.04968	0.00247	1	1 0 0	15.50508	15.50508	19.49	16.63
2.	7.78845	3.89423	0.06791	0.00461	2	1 1 0	11.34216	11.34216	54.81	16.63
3.	11.40289	5.70144	0.09934	0.00987	5	210	7.7538	7.7538	49.29	16.63
4.	14.43785	7.21892	0.12566	0.01579	7	-	6.12999	6.12999	30.82	16.63
5.	17.05353	8.52676	0.14827	0.02198	10	3 1 0	5.1952	5.1952	22.48	16.63
6.	18.0304	9.0152	0.1567	0.02455	11	3 1 1	4.91587	4.91587	61.15	16.63
7.	19.76616	9.88308	0.17164	0.02946	14	3 2 1	4.48793	4.48793	15.93	16.63
8.	22.77078	11.38539	0.19741	0.03897	18	4 1 1	3.90208	3.90208	39.18	16.63
9.	25.26503	12.63252	0.2187	0.04783	22	3 3 2	3.52222	3.52222	100.00	16.63
10.	26.87552	13.43776	0.23239	0.054	25	5 0 0	3.3147	3.3147	70.12	16.63
11.	27.13857	13.56929	0.23462	0.05505	26	5 1 0	3.28317	3.28317	96.38	16.63
12.	31.1606	15.5803	0.26859	0.07214	34	5 3 0	2.86795	2.86795	16.57	16.63
13.	39.15031	19.57515	0.33504	0.11225	52	6 4 0	2.29911	2.29911	6.97	16.63
14.	50.27675	25.13838	0.42481	0.18046	84	8 4 2	1.8133	1.8133	4.22	16.63
15.	52.29385	26.14693	0.44067	0.19419	90	9 3 0	1.8133	1.8133	5.42	16.63
16.	67.39997	33.69998	0.55484	0.30785	143	-	1.38832	1.38832	3.79	16.63

Table 2: XRD data of Ni [(L). 2Cl (H_2O)]

Peak No.	2θ Degree	θ	Sin θ	$Sin^2\theta$	$h^2+k^2+l^2$	h k l	Calc.(d)	Obsrv. (d)	Intensity	a in Å
1.	7.70637	3.85318	0.0672	0.00452	1	1 0 0	11.46278	11.4624	15.19	11.46
2.	14.56021	7.2801	0.12672	0.01606	4	2 0 0	6.07875	6.07859	5.36	11.46
3.	19.74739	9.87369	0.17148	0.0294	7	—	4.49215	4.49203	15.69	11.46
4.	25.39494	12.69747	0.2198	0.04831	11	3 1 1	3.5045	3.50441	25.44	11.46
5.	26.77186	13.38593	0.23151	0.0536	12	2 2 2	3.3273	3.32721	100	11.46
6.	28.27355	14.13678	0.24424	0.05965	13	3 2 0	3.1539	3.15382	47.89	11.46
7.	32.5986	16.2993	0.28065	0.07877	17	4 1 0	2.74465	2.74455	9.481	11.46
8.	36.42658	18.21329	0.31256	0.09769	21	4 2 1	2.46452	2.46442	8.245	11.46
9.	64.38817	32.19409	0.53279	0.28386	63	—	1.44579	1.44575	5.78	11.46
10.	74.92013	37.46007	0.60821	0.36992	82	8 3 3	1.26651	1.26647	4.22	11.46

values have been a common factor. The cell parameters has been calculated by using the equation for cubic system, $Sin^2\theta = \lambda^2/4a^2 (h^2+k^2+l^2)$, where $\lambda^2/4a^2$ is common factor. In the present case Cu for (II) complex $\lambda^2/4a^2$ =0.0021454. The experimental values of $Sin^2\theta$ / common factor are recorded in the each peak Table 1. The $(h^2+k^2+l^2)$ values are 1,2,5,7,10,11,14, 18,22,25,26,34, 52,84,90 and 143. The presence of forbidden numbers 7 and 143 indicates that the compound may belong to tetragonal or hexagonal system[5].

The present case Ni (II) complex $\lambda^2/4a^2$ = 0.0045179. The experimental values of $Sin^2\theta$/ common factor are recorded in the each peak Table 2. The $(h^2+k^2+l^2)$ values are 1,4,7,11, 12,13, 17,21,63 and 82. The presence of forbidden numbers 7 and 63 indicates that the compound may belong to tetragonal or hexagonal system[5].

Thermal analysis

The Cu (II) complex is characterized by the thermal analysis[6]. The Cu (II) complex is stable up to 234.90 °C as revealed from the plateau on TG curve, which indicates the presence of water in the co-ordinated form and absence of water molecule out side the sphere. The complex underwent degradation and gave break at 325 °C with weight loss of 39%, which corresponds to the decomposition of the ligand unit. The complex underwent further degradation and gave another break at 460 °C with a weight loss of 56.63 %, which corresponds to the loss of ligand unit. Further the complex is decomposing continuously. The final weight of residue corresponds to the formation of CuO.

EXPERIMENTAL

All chemicals used were A.R. grade. The compound (2-Phenyl-3-{[(1*E*)-1-(3-oxo-3*H*-benzo[f]chromen-2-yl)ethylidene]amino}quinazolin-4(3*H*)-one) [3,4] (L), was prepared by the reported method. The metal complexes of Cu (II) and Ni (II) were prepared by refluxing 15 ml of an ethanolic solution of an appropriate metal (II) Chloride (0.01 mol) with 30 ml of an ethanolic solution of L (0.01 mol) for 2-3 hours. 0.5g of Sodium acetate was added to the reaction mixture just to adjust the pH of the solution. The content of the flask further

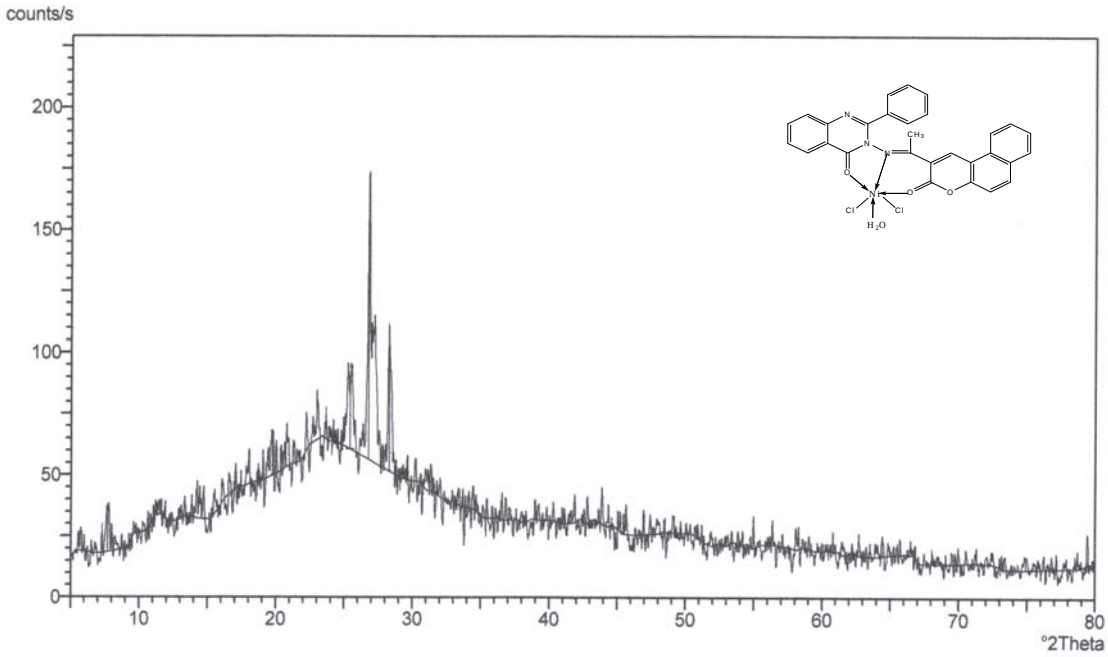


Fig. 1: Powder XRD pattern of Ni(II) complex of the ligand TL

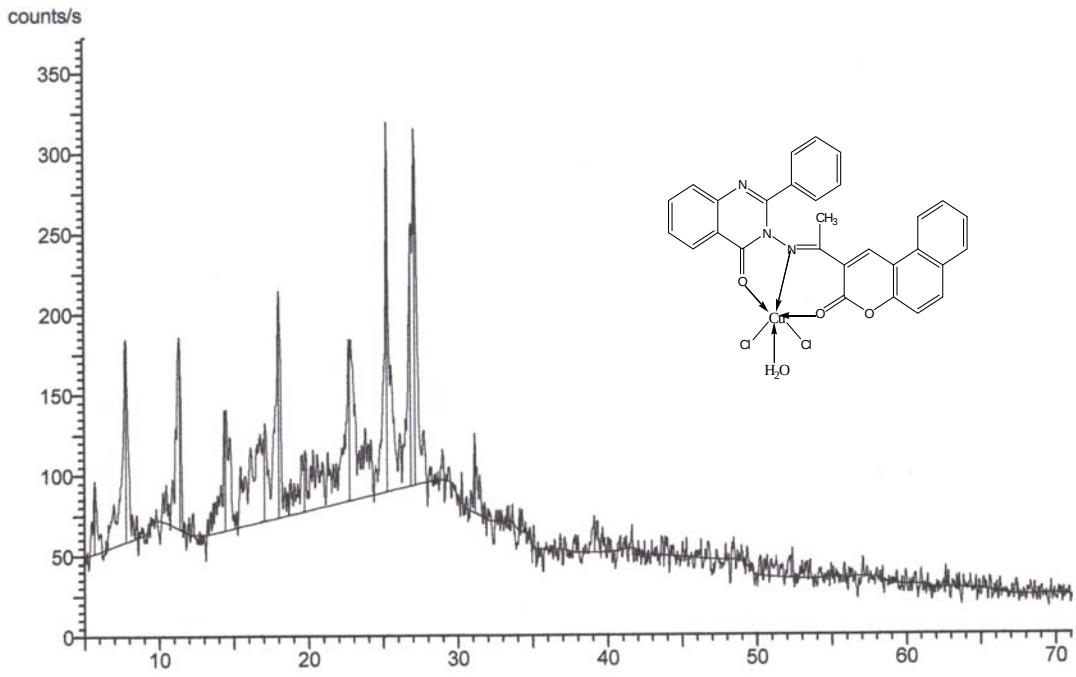
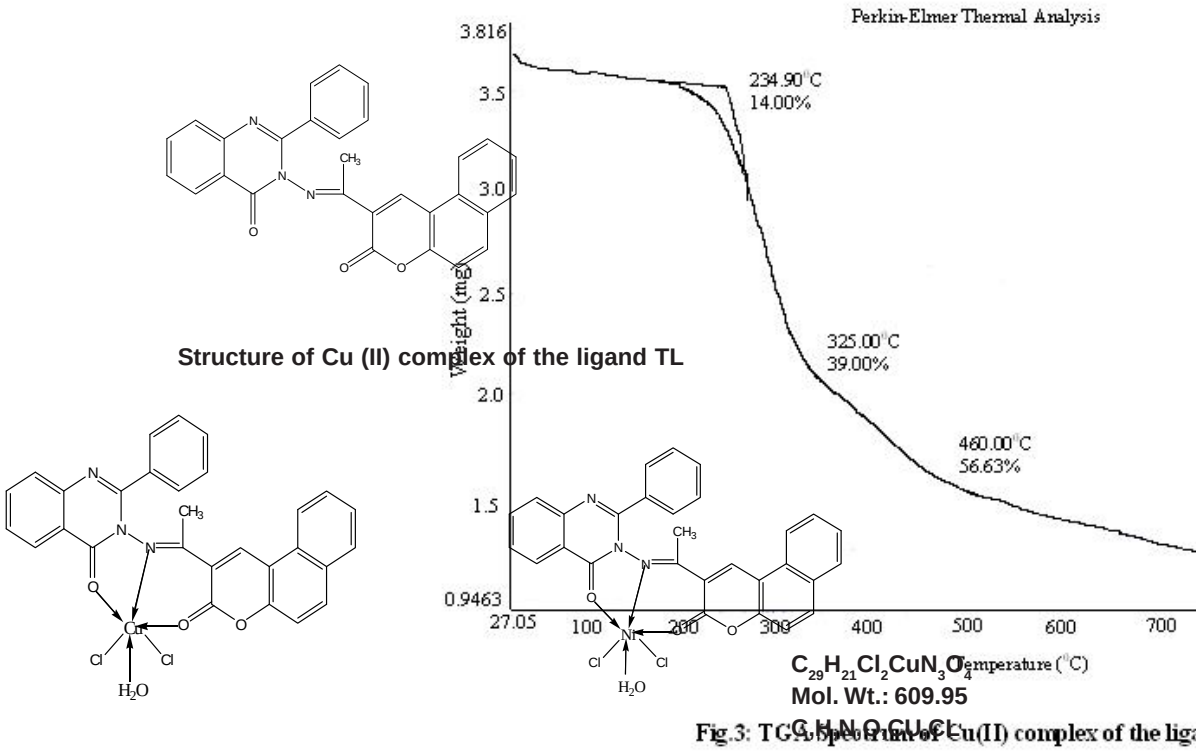


Fig. 2: Powder XRD pattern of Cu(II) complex of the ligand TL

Fig. 3: TGA spectrum of Cu(II) complex of the ligand TL



Structure of Cu (II) complex of the ligand TL

Fig. 3: TGA spectrum of Cu(II) complex of the liga

Structure of ligand TL: 2-Phenyl-2-{[(1E)1-(2-oxo-2H-benzo [f]chromen-3-yl-ethylidene]amino} quinazolin-4(3H)-one

refluxed for 2 hours more. The resulting mixture decomposed by pouring into 100 ml distilled water. Separated solid was prepared and washed with sufficient volume of distilled water and dried.

The XRD spectra were recorded on Phillips PW 3710 diffractometer attach to a digitized computer along with graphical assembly using CuKα radiation source. The X-ray tube was operated at 25 KV / 20mA. The sample were scanned in the 2θ range from 0-80° at a scanning speed of 2θ/ min.

The thermal measurements taken on PerkinElmer thermal analyzer at heating rate of 15.00 °C / min from 28.00°C to 1010.00 °C.

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