Asian Journal of Chemistry Vol. 15, No. 1 (2003), 471–474

X-ray Diffraction Studies on Mixed Ligand Complexes of Ni(II) and Cu(II)

VIVEK TIWARI* and RASHMI SINGHAI
Department of Chemistry, Dr. Hari Singh Gour University, Sagar-470 003, India
e-mail: rashmitiwari_sgr@yahoo.com

The X-ray diffraction studies of mixed ligand Ni(II) and Cu(II) terephthalate complexes with 8-hydroxyquinoline in solid state have been carried out. On the basis of XRD the crystal system of Ni(II) complex is tetragonal and Cu(II) complex is orthorhombic.

Key Words: XRD, Ni(II), Cu(II), Mixed ligand complexes , Terephthalate, 8-Hydroxyquinoline.

INTRODUCTION

Terephthalates are known in industry for their polymeric, resistant and tolerant nature. In recent years mixed ligand complexes have received special attention for their medical, biological, agricultural and industrial applicabilities. Metal terephthalate and its complexes have been studied earlier[1]. In our earlier communication[2] we reported the preparation, characterization and thermal behaviour of Ni(II) and Cu(II) terephthalate complexes with 8-hydroxy quinoline. The present work deals with the XRD studies of these complexes. For the structural characterization various techniques are used[3] and among them X-ray diffractometry (XRD) is non-destructive, non-contact, fast and sensitive. For the evaluation of various parameters and thereby deciding the structure of sample, XRD technique has been applied for the present compounds.

EXPERIMENTAL

The Ni(II) and Cu(II) mixed ligand complexes were prepared by the reported method[2]. The purity of the product was checked by elemental analysis and physico-chemical techniques. The chemicals used were of reagent grade (E. Merck/B.D.H./Loba).

The XRD spectra were recorded at RSIC Nagpur on Philips PW-1710 diffractometer using Cu-K_α-1.5418 Å at a chart speed 2° (2θ)/min in the range 5–65° on the chart speed scale, 1° = cm^{-1}.

RESULTS AND DISCUSSION

The physico-chemical characterizations of the complexes have been reported earlier[2]. The present complexes have been investigated for their crystal structure.

These compounds were subjected to X-ray powder diffraction analysis. The X-ray data of the two complexes are given in Tables 1 and 2.

TABLE-1
POWDER X-RAY DATA OF Ni(II) COMPLEX

Peak No.	d Spacing (Å)	Intensity ($I/I_0 \times 100$)	$\sin^2\theta$		hkl
			(Obs.)	(Calc.)	
1.	17.2736	2.1337	0.0019	0.0019	100
2.	12.7249	100	0.0036	0.0037	103
3.	11.1694	10.4299	0.0047	0.0047	202
4.	10.1604	2.1615	0.0057	0.0057	203
5.	9.8315	7.9299	0.0079	0.0079	220
6.	8.6291	7.3566	0.01005	0.0101	211
7.	7.6892	2.6751	0.0168	0.0167	222
8.	5.6104	6.1305	0.0208	0.0202	312
9.	5.3361	6.9904	0.0252	0.0258	320
10.	5.1248	9.8407	0.0267	0.0266	322
11.	4.7132	17.9456	0.0311	0.0308	325
12.	4.3680	3.4076	0.0327	0.0326	402
13.	4.2584	5.5414	0.0351	0.0350	404
14.	4.1123	13.8694	0.0387	0.0390	334
15.	3.9140	3.4672	0.0408	0.0401	422
16.	3.8371	4.6474	0.0457	0.0456	337
17.	3.8137	5.1910	0.0457	0.0456	426
18.	3.5532	2.0859	0.0530	0.0529	504
19.	3.1959	24.0445	0.0581	0.0555	522
20.	3.0401	4.4108	0.0643	0.0644	442
21.	3.0142	3.2006	0.0654	0.0654	443
22.	2.9750	2.1176	0.0671	0.0676	540
23.	2.8140	2.1496	0.0750	0.0754	613
24.	2.7501	2.5316	0.0785	0.0756	615

The cell parameter have been calculated[4-7] by the following equations:

For tetragonal system

$$\sin^2\theta_{(hkl)} = A(h^2 + k^2) + Cl^2$$

where $A = \lambda^2/4a^2$ and $C = \lambda^2/4c^2$

For orthorhombic system,

$$\sin^2\theta_{(hkl)} = Ah^2 + Bk^2 + Cl^2$$

where $A = \lambda^2/4a^2$, $B = \lambda^2/4b^2$ and $C = \lambda^2/4c^2$

(h, k, l are Miller indices).

TABLE-2
POWDER X-RAY DATA OF Cu(II) COMPLEX

Peak No.	d Spacing (Å)	Intensity ($I/I_0 \times 100$)	$\sin^2\theta$		hkl
			(Obs.)	(Calc.)	
1.	10.7553	13.4085	0.0051	0.0051	100
2.	7.6605	96.6139	0.0101	0.0101	030
3.	6.9648	44.1309	0.0122	0.0124	004
4.	6.5558	10.6094	0.0138	0.0135	014
5.	6.1197	9.5936	0.0158	0.0153	130
6.	5.9073	18.7358	0.0170	0.0171	033
7.	5.4514	20.5417	0.0199	0.0193	055
8.	5.2162	18.5101	0.0218	0.0216	210
9.	3.7032	22.1910	0.0433	0.0431	107
10.	3.5520	17.7200	0.0470	0.0469	301
11.	3.4020	48.6455	0.0513	0.0514	321
12.	3.3743	87.5846	0.0521	0.0519	252
13.	3.3548	100.0000	0.0528	0.0529	163
14.	3.2312	43.0020	0.0569	0.0571	331
15.	2.3114	20.4288	0.1109	0.1111	095
16.	2.2812	23.9270	0.1142	0.1141	101
17.	2.1457	15.2370	0.1290	0.1289	501
18.	2.0332	17.4943	0.1437	0.1415	532
19.	1.8749	15.6884	0.1691	0.1700	123
20.	1.7832	14.3340	0.1869	0.1866	611
21.	1.7512	14.5598	0.1937	0.1928	613
22.	1.7149	15.4627	0.2021	0.2028	640
23.	1.6569	16.7042	0.2164	0.2171	626
24.	1.5220	13.3182	0.2565	0.2559	720
25.	1.5220	12.6410	0.2671	0.2683	724
26.	1.4914	11.8570	0.3253	0.3283	800

The experimental values of $\sin^2\theta$ are in good agreement with the calculated values of $\sin^2\theta$ for tetragonal and orthorhombic cells.

The XRD pattern of Ni(II) complex consists reflections (2θ) between 5–65° with maximum at 2θ = 8° which corresponds to d = 14.2736 Å. The observed values fit well in the tetragonal system to give a unit cell with lattice constants a = b = 12.28 Å and c = 54.51 Å and cell volume = 16276.0 Å.^{3} The numbers of molecules per unit cell (n) are 20. The observed and calculated density of the complex has been found to be 1.118 g/cm^3 and 1.119 g/cm^3 respectively.

The XRD pattern of Cu(II) complex gives reflection (2θ) between 5–65° which corresponds to d = 10.7553 Å. The maximum reflection is observed at 15°. The observed values fit well in the orthorhombic system to give a unit cell with lattice

constants, a = 10.7553 Å, b = 22.90 Å and c = 17.69 Å and cell volume = 6822.9 $Å^3$. The number of molecules per unit cell (n) are 11. The observed and calculated density of the complex comes to be 1.341 g/cm^3 and 1.385 g/cm^3 respectively.

ACKNOWLEDGEMENT

The authors are thankful to the Head, RSIC, Nagpur for providing XRD analysis.

REFERENCES

1. C.L. Sharma, T.K. De. and A.K. Singh, *Indian J. Chem.*, **18A**, 183 (1979).
2. A.P. Mishra, V.K. Tiwari, and R. Singhai, *Indian J. Chem.* (in press).
3. J. Xiaoming and Wu. Zigin, *Chin. Phys. Lett.*, **7**, 356 (1991).
4. N.F.M. Henry, H. Lipson and W.A. Wooster, Interpretation of X-ray Diffraction Photographs, McMillan, London, p. 179 (1951).
5. B.D. Cullity, Elements of X-ray Diffraction, Addison-Wesley, Massachusetts (1956).
6. M.M. Woolfson, An Introduction to X-ray Crystallography, Cambridge University Press (1980).
7. H.S. Peiser, H.P. Rooksby and A.J.C. Wilson, X-ray Diffraction by Pollycrystalline Materials, The Institute of Physics, London, p. 344 (1955).

(*Received:* 13 August 2002; *Accepted*: 20 September 2002) AJC-2864