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X-RAY POWDER DIFFRACTION STUDIES OF LIGANDS AND ITS METAL COMPLESES

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ABSTRAC

X-ray powder diffraction (XRD) is a technique used to analyze the crystal structure of materials by measuring the diffraction pattern of X-rays scattered from a powdered sample, providing information about phase identification, purity, and crystallite size. XRD is a non-destructive analytical method that uses X-rays to study the arrangement of atoms within a crystalline material. A powdered sample is bombarded with X-rays. The X-rays interact with the atoms in the sample, causing them to scatter. The scattered X-rays produce a diffraction pattern, which is a series of peaks and valleys. The pattern is analyzed to determine the crystal structure of the material. X-ray diffraction (XRD) is primarily used to identify the phases and crystal structure of materials, as well as to analyze their properties like grain size, defects, and strain. X-ray powder diffraction is most widely used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds). Determination of unknown solids is critical to studies in geology, environmental science, material science, engineering and biology. X-ray powder diffraction pattern for Cu(II) complex has characterized with a view to find the type of crystal system the XRD data given the table the diffractogram of Cu(II) complex consists of seven, ten and ten reflections in the range of 10 – 22, (2θ value) with maxima at 2θ =15.94 ^{0}A, 10.78^{0}A and 10.59 ^{0}A of Cu(II) complex of ligands **L^1, L^2 and L^3**. The inter planar spacing (d) has been calculated from the position of intense peaks using Bragg's equation nλ=2dsinθ, λ = 1.5406 ^{0}A.

Keynotes: Bragg's equation nλ=2dsinθ, λ = 1.5406 ^{0}A.

INTRODUCTION:

Powder diffraction is a scientific technique using X-ray, neutron, or electron diffraction on powder or microcrystalline samples for structural characterization of materials.[2] An instrument dedicated to

performing such powder measurements is called a powder diffractometer. Powder diffraction stands in contrast to single crystal diffraction techniques, which work best with a single, well-ordered crystal. The most common type of powder diffraction is with X-rays, the focus of this article although some aspects of neutron powder diffraction are mentioned. (Powder electron diffraction is more complex due to dynamical diffraction [3] and is not discussed further herein.) Typical diffractometers use electromagnetic radiation (waves) with known wavelength and frequency, which is determined by their source. The source is often X-rays, and neutrons are also common sources, with their frequency determined by their de Broglie wavelength. When these waves reach the sample, the incoming beam is either reflected off the surface, or can enter the lattice and be diffracted by the atoms present in the sample. If the atoms are arranged symmetrically with a separation distance d, these waves will interfere constructively only where the path-length difference $2d \sin \theta$ is equal to an integer multiple of the wavelength, producing a diffraction maximum in accordance with Bragg's law. These waves interfere destructively at points between the intersections where the waves are out of phase, and do not lead to bright spots in the diffraction pattern.[4] Because the sample itself is acting as the diffraction grating, this spacing is the atomic spacing. The distinction between powder and single crystal diffraction is the degree of texturing in the sample. Single crystals have maximal texturing, and are said to be anisotropic. In contrast, in powder diffraction, every possible crystalline orientation is represented equally in a powdered sample, the isotropic case. Powder X-ray diffraction (PXRD) operates under the assumption that the sample is randomly arranged. Therefore, a statistically significant number of each plane of the crystal structure will be in the proper orientation to diffract the X-rays. Therefore, each plane will be represented in the signal. In practice, it is sometimes necessary to rotate the sample orientation to eliminate the effects of texturing and achieve true randomness. Mathematically, crystals can be described by a Bravais lattice with some regularity in the spacing between atoms. Because of this regularity, we can describe this structure in a different way using the reciprocal lattice, which is related to the original structure by a Fourier transform. This three-dimensional space can be described with reciprocal axes x^*, y^*, and z^* or alternatively in spherical coordinates q, φ^*, and χ^*. In powder diffraction, intensity is homogeneous over φ^* and χ^*, and only q remains as an important measurable quantity. This is because orientational averaging causes the three-dimensional reciprocal space that is studied in single crystal diffraction to be projected onto a single dimension. Two-dimensional powder diffraction setup with flat plate detector.[5] When the scattered

radiation is collected on a flat plate detector, the rotational averaging leads to smooth diffraction rings around the beam axis, rather than the discrete Laue spots observed in single crystal diffraction. The angle between the beam axis and the ring is called the *scattering angle* and in X-ray crystallography always denoted as 2θ (in scattering of *visible* light the convention is usually to call it θ). In accordance with Bragg's law, each ring corresponds to a particular reciprocal lattice vector G in the sample crystal. This leads to the definition of the scattering vector as in this equation, G is the reciprocal lattice vector, q is the length of the reciprocal lattice vector, k is the momentum transfer vector, θ is half of the scattering angle, and λ is the wavelength of the source. Powder diffraction data are usually presented as a diffractogram in which the diffracted intensity, I, is shown as a function either of the scattering angle 2θ or as a function of the scattering vector length q. The latter variable has the advantage that the diffractogram no longer depends on the value of the wavelength λ. The advent of synchrotron sources has widened the choice of wavelength considerably. To facilitate comparability of data obtained with different wavelengths the use of q is therefore recommended and gaining acceptability.

EXPERIMENTAL:

The X-ray tube was operated at 25 KV/20mA. The sample were scanned in the 2θ range from 0-80 ^{0} at a scanning speed of 2θ/min. Due to practical difficulties to obtain good crystals, single crystal XRD could not be scanned to confirm the structures of all the compounds.

i) Results and Dissection:

X-ray powder diffraction studies of Cu(II) complex of the ligand L^1 , L^2 and L^3.

X-ray powder diffraction pattern for Cu(II) complex has characterized with a view to find the type of crystal system the XRD data given the table the diffractogram of Cu(II) complex consists of seven, ten and ten reflections in the range of 10 – 22, (2θ value) with maxima at 2θ =15.94 ^{0}A, 10.78^{0}A and 10.59 ^{0}A of Cu(II) complex of ligands **L^1, L^2 and L^3**. The inter planar spacing (d) has been calculated from the position of intense peaks using Bragg's equation $n\lambda=2d\sin\theta$, λ = 1.5406 ^{0}A. The observed and calculated values of d are quite consistent (Table 3.12 – 3.14). The unit cell calculations have been carried out for the cubic system, the set of $h^2 + k^2 + l^2$ values of the complex were found to be 1, 2, and 4 which corresponds to the planes and absence of

forbidden number its confirms the cubic symmetry from the above results the unit cell constants for cubic system were found to be a=b=c =8.8514 ^{0}A, 8.3556 ^{0}A and 8.3548 ^{0}A for the Cu(II) complex of the ligands **L^1, L^2 and L^3**. The complex showed broad peak indicates that amorphous in nature.

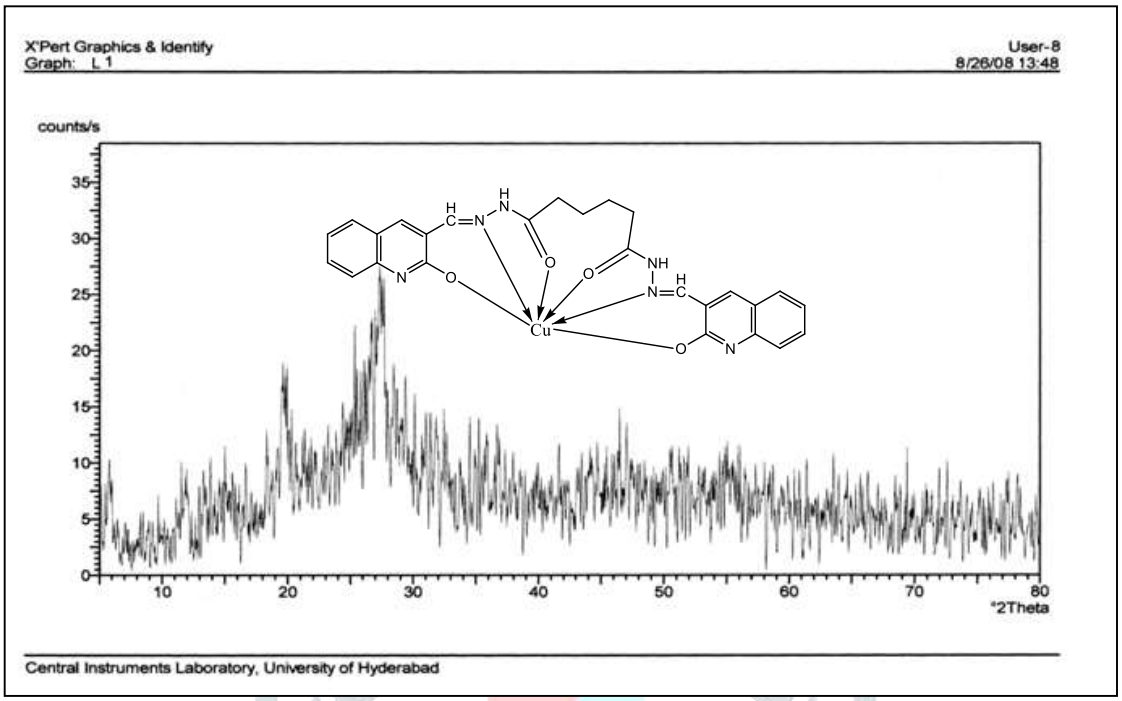
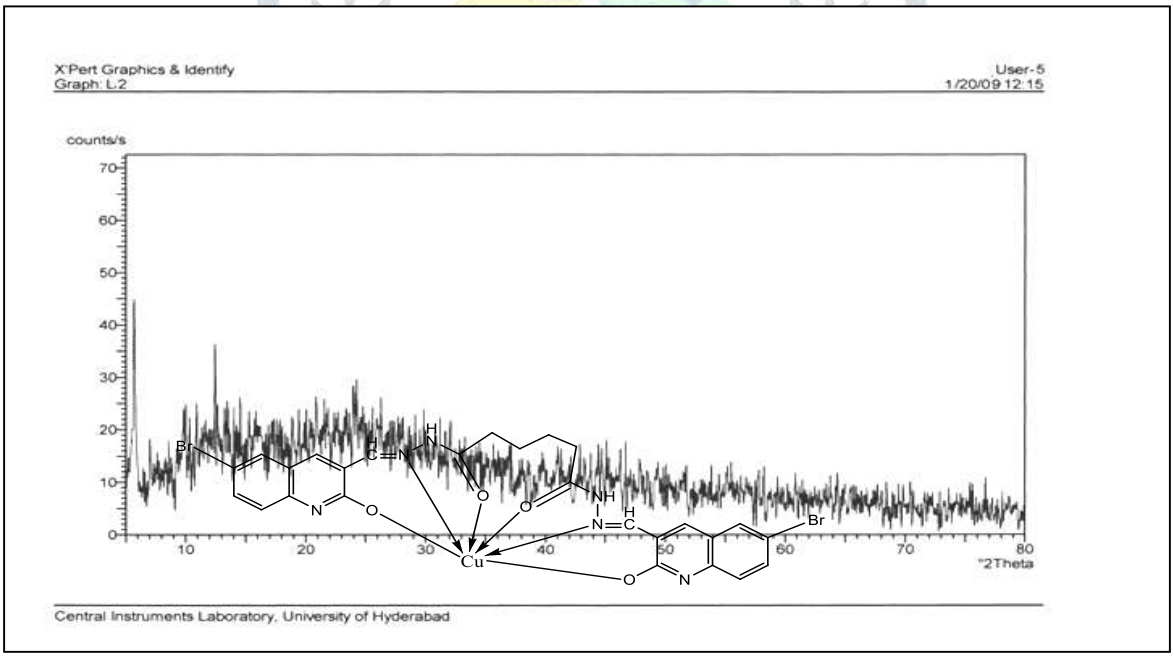


Figure-1 XRD Spectrum of Cu(II) complex of Ligand L^1 (HMOHAD)



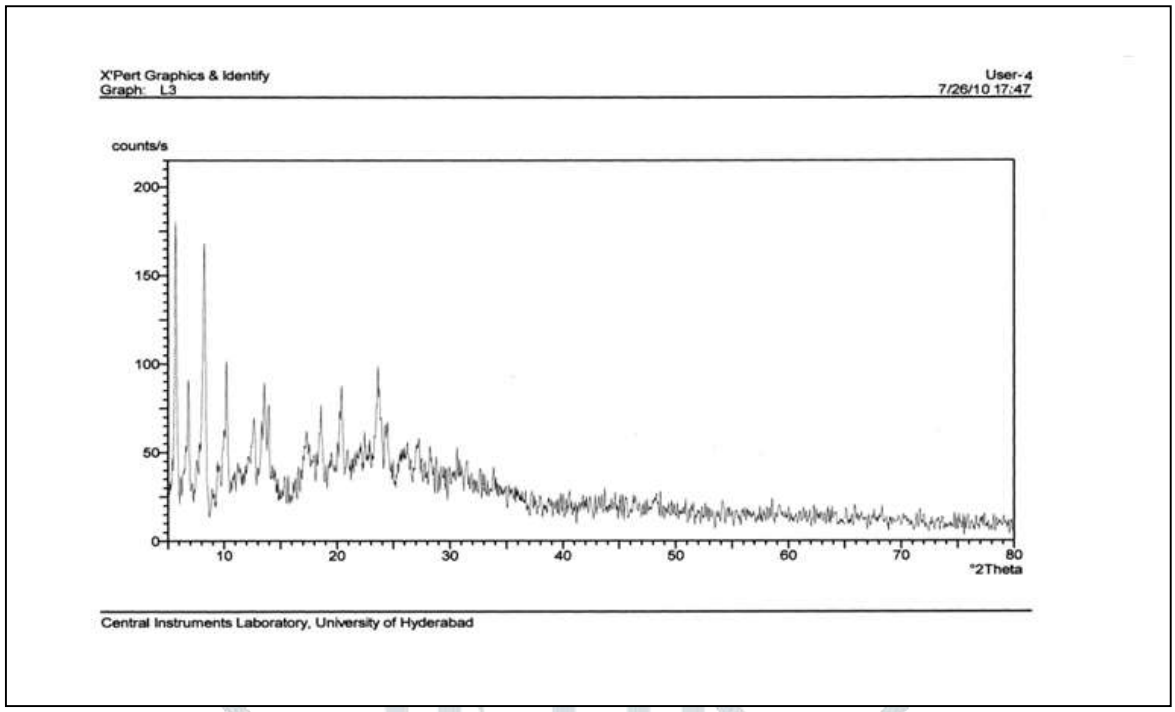


Figure-3 XRD Spectrum of Cu(II) complex of ligand L^3 (HMCHAD)

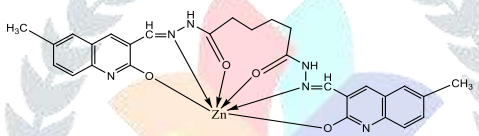


Table 1. X- ray powder diffraction data of Cu(II) complex of ligand L^1

2θ	θ	sinθ	$Sin^2θ$	$h^2+k^2+l^2$ (a)	$h^2+k^2+l^2$ (b)	h k l	d-spacing		Relative intensity (%)	a (A°)
							Cal.	Abs.		
10.93	5.46	0.0951	0.0090	1	1	1 0 0	8.346	8.332	64.84	8.3546
14.66	7.33	0.1275	0.0162	2.173	2	1 1 0	5.661	5.675	44.61	8.0032
15.94	7.97	0.1386	0.0192	2.275	2	1 1 0	5.542	5.536	100	8.8514
16.18	8.09	0.1407	0.0197	2.319	2	1 1 0	5.484	5.473	57.25	8.7625
16.88	8.44	0.1467	0.0215	2.507	2	1 1 0	5.278	5.277	47.55	8.4636
21.80	10.90	0.1890	0.0357	4.203	4	2 0 0	4.073	4.380	51.84	8.1638
22.60	11.30	0.1959	0.0383	4.116	4	2 0 0	4.116	4.114	55.61	8.2357

Table 2. X- ray powder diffraction data of Cu(II) complex of ligand L^2

2θ	θ	sinθ	$Sin^2θ$	$h^2+k^2+l^2$ (a)	$h^2+k^2+l^2$ (b)	h k l	d-spacing		Relative intensity (%)	a (A°)
							Cal.	Abs.		
10.78	5.39	0.0921	0.0083	1	1	1 0 0	8.356	8.342	95.84	8.3556
15.65	7.82	0.1363	0.0186	2.174	2	1 1 0	5.651	5.665	44.61	8.0031
15.97	7.98	0.1391	0.0191	2.271	2	1 1 0	5.542	5.536	54.63	8.8414
16.16	8.08	0.1407	0.0193	2.313	2	1 1 0	5.494	5.483	57.25	8.7615
16.81	8.40	0.1462	0.0219	2.509	2	1 1 0	5.275	5.277	47.55	8.4632
20.78	10.39	0.1891	0.0356	4.201	4	2 0 0	4.073	4.392	51.84	8.1538
21.60	10.30	0.1877	0.0355	4.118	4	2 0 0	4.117	4.114	55.61	8.2347
21.77	10.88	0.1884	0.0354	4.174	4	2 0 0	4.088	4.085	56.76	8.1751
22.25	11.12	0.1925	0.0376	4.369	4	2 0 0	3.992	3.994	61.73	8.0009
22.38	11.14	0.1943	0.0375	4.421	4	2 0 0	3.967	3.966	59.74	8.0195

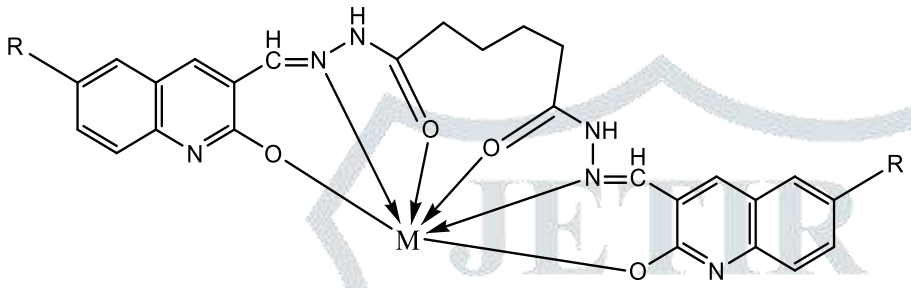
Table 3.14 X- ray powder diffraction data of Cu(II) complex of ligand L^3

2θ	θ	sinθ	$Sin^2θ$	$h^2+k^2+l^2$ (a)	$h^2+k^2+l^2$ (b)	h k l	d-spacing		Relative intensity (%)	a (A°)
							Cal.	Abs		
10.59	5.29	0.0922	0.0085	1	1	1 0 0	8.354	8.347	95.88	8.3548
15.64	7.82	0.1361	0.0185	2.176	2	1 1 0	5.659	5.661	44.60	8.0039
15.99	7.99	0.1390	0.0193	2.270	2	1 1 0	5.541	5.538	54.60	8.8412
16.80	8.40	0.1460	0.0213	2.505	2	1 1 0	5.275	5.273	47.50	8.4639
20.18	10.90	0.1890	0.0357	4.200	4	2 0 0	4.075	4.396	51.80	8.1534
20.31	10.15	0.1762	0.0310	3.647	4	2 0 0	4.371	4.369	52.30	8.7497
21.59	10.79	0.1872	0.0350	4.117	4	2 0 0	4.114	4.112	55.60	8.2345
21.75	10.87	0.1885	0.0355	4.176	4	2 0 0	4.086	4.083	56.70	8.1754
22.23	11.11	0.1926	0.0371	4.364	4	2 0 0	3.999	3.995	61.70	8.0001
22.39	11.19	0.1941	0.0376	4.423	4	2 0 0	3.968	3.967	59.70	8.0191

CONCLUSTION:

X-ray powder diffraction studies observations projects the following structures for these complexes are Cu(II), Co(II), Ni(II), Zn(II), Cd(II), Hg(II) and Mn(II) are exhibit six coordinated octahedral geometry.

Proposed structure for the metal complexes of the Schiff's base ligands L^1, L^2 and L^3



Where M = Cu(II), Co(II), Ni(II) , Zn(II), Cd(II), Hg(II) and Mn(II) **R= H (L^1), Br (L^2) and CH_3 (L^3)**

REFERENCES:

1. G. Bringmann, Y. Reichert, V. Kane, *Tetrahedron.*, **60,** 3539, (2004).
2. V. V. Kournetsov, L. Y. V. Mendez, C. M. M. Gomez, *Curr. Org. Chem*., **9,** 141, (2005).
3. Antimalarial Drugs II; W. Peters, W. H. G. Richards, Eds., Springer Verlag, Berlin Heidelberg, New York,Tokyo. (1984).
4. P. Muthumani, S. Venkataraman, R. Meera1, N. Govind Nayak, P. Chidambaranathan, B. Devi, Kameswari, *Der Pharma Chemica*., **2(1),** 385, (2010).
5. K. R. Gupta, A. Gupta, S. Paul, P. L. Kachroo, *Ind. J. Chem*., **37B,** 1211, (1998).
6. S. Goel, Ritu, *Ind. J. Hetero. Chem*., **15,** 401, (2006).
7. H. Konda, F. Sakamoto, K. Kawakami, G. Tsukamoto, *J. Med. Chem*., **31**, 221, (1998).
8. R. W. Baker, S. Cai, M. Dimitroff, L. Fang, H. Joseph, Therrien. *J. Med. Chem*., **47,** 4693, (2004).
9. P. Shaha, *Ind. J. Hetero. Chem*., **10,** 13, (1998).
10. M. Charles Hall, G. Herbert, Johnson, B. John Wright, *J. Med. Chem*., **1,** 17, (1974).

11. A. Abdulqader Alhaider Atef, M. Abdelkader, J. Eric Lein, *J. Med. Chem.*, **28,** 1394, (1985).

12. V. Nadaraj, S. Thamaraj selvi, R. Sasi. *Arkivoc*, **10,** 82, (2006).

13. B. John Sherrill, P. Ally Liou, L. Jennifer, L. Weisman Joseph, De. Risib, R Kiplin Guy, *Bio. Org. Med. Chem. Lett*, **15,** 1015, (2005).

14. R. Vlahov, S. T. Parushev, J. VlahovNickel, G. Snatzke , *Pure and Appl. Chem.*, **62(7)**, 1303, (1990).

15. M. Navarro, F. Vasquez and A. Roverto, S. Delgad,. *J. Med. Chem.*, **47,** 5204, (2004).

16. V. K. Pandey, Menal Tandon, *Ind. J. Chem.*, **40B,** 527, (2001).

17. S. Venkataraman , *Der Pharma Chemica*, **2**, 385, (2010).

18. Y.Takeuchi, M. R. Oda, T.Chang, Y. Okamoto J. Ono Oda, M.Yamato , *Chem. Pharm. Bull* (Tokyo)., **38,** 3048, (1990).

19. E. Lukevics, I. Sega, A. Zablotskayn, S. Germane, *Molecules.*, **2,** 180, (1997).

20. Y. Sawada, H. Kayakiri, Y. Abe, H. Tanaka, *J. Med. Chem.*, **47,** 2853, (2004).

21. A. Cappelli, L. Pericot Mohr, A. Gallelli, M. Rizzo, G. Giorgi, *J. Med. Chem.*, **47,** 2574, (2004).