Synthesis and X-ray diffraction study of new copper (II) complexes of α-aminonitrile derived from P- methoxybenzaldehyde with aromatic amine.

Pradeep Sharma¹, Jaishree Bhale²,⁵, Ashutosh Mishra³ and Pramod Malviya⁴
¹Govt. Holkar Science College, Indore (M.P), India
²Shree Cloth Market Institute of Professional Studies, Indore (M.P), India
³School of Physics, Devi Ahilya University, Indore (M.P), India
⁴Govt. College, Nagda (M.P), India
Email: bhalejaishree@gmail.com

Abstract: New metal complexes of transition metal ion Cu (II) of the ligand HL = (P-methoxy anilino)-P- methoxy phenyl acetonitrile were synthesized. The ligand was prepared by chemical root procedure which included the reaction of P-methoxybenzaldehyde with P-anisidine. The ligand HL act as primary ligand and sulphate, chloride, nitrate and acetate act as secondary ligand. The data obtained has been preceding using XRD data analysis program Origin 6.0 Professional. From the experimental measurements, various parameters- lattice parameter and particle size have been estimated. Particle size is found to be in the range of nanometer. The XRD analysis revealed the crystalline nature of all the complexes.

1. Introduction:
The chemistry of nitrile and α-aminonitrile compounds and their derivatives has received special attention because of their application as potential ligands for a large number of metal ions (1). Nitriles and α-aminonitrile derivatives had a biological activities (2) as herbicides (3), pharmacological agents (4) and biological synthesis of chemical compounds by it’s microbial metabolism in some organisms (5). Complexes containing more than a metal centre represent the synthetic models of ferromagnetic interaction between the metal centers which can explain oxidation-reduction processes in biological systems in addition to their catalytic and biological activities (6). Beside that, some aminonitriles were used to prepare racemic compounds (7). A search through literature reveals that there is no work has been done on the XRD of transition metal complexes of the HL. Keeping this in view, the present paper describes the results of the synthesis and the characterization- XRD of new metal complexes of some transition metal ions Cu(II) of HL [(P-methoxy anilino) –p- methoxy phenyl aceto nitrile].

2. Experimental
2.1 Materials
All reagent used were analytical grade purity. All metal salts of Cu(II) 99% (Merck), p-methoxy
benzaldehyde 99%(Merck), glacial acetic acid 99%(Merck), concentrated H2SO4 99%(Merck), ethanol absolute 99%(Fluka) were used as received from the suppliers. P-anisidine and p-anisidine sulfonic acid were supplied by Prashant group of industries. All chemicals are prepared by chemical root method.

2.2 Preparation of α-aminonitriles (HL): The aldehyde p-methoxy benzaldehyde 0.05 mole was dissolved in 50 ml of glacial acetic acid, p-anisidine sulfonic acid was added in small portions to bring the pH to 2, followed by the additions of 0.05 mole of the amine. The pH was adjusted to 3-4 by adding concentrated H2SO4 drop wise. KSCN 0.05 mole was added to the mixture which was kept stirring. The end of the reaction was checked by the disappearance of the starting material (the amine) and development of a higher spot on T.L.C. The reaction mixture was poured on ice and made slightly alkaline with ammonia. The solid product was filtered, washed with water and dried.

2.3 Preparation of Cu(II) complexes of HL: A solution of a salt of Cu(II) in absolute ethanol was added to ethanolic solution of the ligand with a continuous stirring. The molar ratio of the reactants was 2:2. Precipitation of complex took place immediately. The product was filtered off, washed several times with ethanol and dried under vacuum.

2.4 X-ray diffraction
The sample is irradiated with a beam of monochromatic x-rays over a variable incident angle range. The X-rays were produced using a sealed tube and the wavelength of X-ray as 0.154nm (Cu Kα). The X-rays were detected using a fast counting detector based on silicon strip technology (Bruker LynxEye detector). Interaction with atoms in the sample results in diffracted x-rays when the Bragg equation is satisfied. The X-rays were detected using a fast counting detector based on silicon strip technology (Bruker LynxEye detector). X-ray diffraction pattern have been recorded by Bruker D8 advance diffractometer at IUC, Indore.

3. Results and discussion:
XRD patterns are shown in ‘figures 1’. All the samples are characterized at room temperature by X-ray diffraction using Cu Kα radiation. The diffraction pattern of complexes are recorded between 2θ ranging from 10° to 80°. The particle size of the samples is estimated using the Scherrer’s formula. According to Scherrer’s equation, the particle size is given by t = 0.9 λ/Bcosθ, where t is the crystal thickness (in nm), B is half width (in radians), θ is the Bragg angle and λ is the wavelength. The particle size corresponding to each diffraction maxima are determined from the measurement of the half width of the diffraction peak. Lattice parameter for simple cubic crystal structure is determined by a²= λ²(h²+k²+l²)/4sin²θ. The value of Lattice parameter and the particle size are shown in ‘table 1’ for all the four complexes. The particle size was found to be within in the range 11.43-51.58 nm for all the complexes.

Table-1. Lattice parameter and particle size

| Complexes                                           | Lattice parameter (Å) | Particle size (nm) |
|------------------------------------------------------|-----------------------|--------------------|
| [Cu₂(HL)₂(H₂O)₈]SO₄                           | 9.36                  | 11.43              |
| [Cu₂(HL)₂(H₂O)₈]Cl₂                               | 9.33                  | 26.92              |
| [Cu₂(HL)₂(H₂O)₈]NO₃                                | 9.22                  | 30.24              |
| [Cu₂(HL)₂(H₂O)₈](CH₃COO)₂                         | 10.08                 | 51.58              |
4. Conclusion
The XRD pattern is indicative of their crystalline in nature which is confirmed by the main peaks positioned. The X-ray analysis reveals that the sample is cubic in phase as seen from the presence of extra peaks in XRD pattern. All the peaks match with the soft wear JCPDF.

![XRD patterns](image)

**Figure 1:** XRD of Cu(II) complexes of HL

Acknowledgment
We are grateful to P. Sharma and Dr. Mukul Gupta, IUC, Indore for XRD studies of prepared complexes. We are also grateful to Mr. Nihar Patel (Prashant group of industries) for his valuable support. Thanks are also due to Dr. V. Mishra, SCMIPS, Indore for his valuable support during this research work.

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