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Determination of Bond Lengths and Theoretical and Experimental Analysis of EXAFS Data

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Abstract: The present paper deals with the comparative study of experimental and theoretical EXAFS data analysis. The X-ray absorption measurements have been performed at the recently developed BL-8 dispersive EXAFS beam line at 2.5 GeV Indus-2 Synchrotron Source at RRCAT, Indore, India. The data obtained has been processed using EXAFS data analysis program Athena. The Lytle, Sayers and Stern (LSS) method and Fourier Transform method have been used for determination of bond lengths of the studied complexes. The theoretical EXAFS data of the same complexes has been generated and Fourier transformed using MathCad software. The theoretically calculated bond length has been compared with experimentally obtained bond length using Lytle, Sayers and Stern(LSS) method and Fourier Transform method.

INTRODUCTION: Schiff's bases of o-phenylenediamine and its complexes have a variety of applications including biological, analytical nd calinical [1, 2]. Metal complexes of Schiff base are studied due to synthetic flexibility and sensitivity toward a variety of metal atoms[3].They are found useful in catalysis, in medicine as antibiotics and anti-inflammatory agent and in the industry as anti-corrosion [4-7]. The copper complexes have been prepared by chemical root method.

Extended X-ray absorption fine structure (EXAFS) spectroscopy provides structural information about a sample by analysis of its X-ray absorption spectrum. In order to extract structural information from experimental spectra, a simple analytical expression that relates the EXAFS signal to the structural parameters is required. An EXAFS analytical expression is like the one suggested by Stern [8]. Various structural parameters can be extracted by an approximate Fourier analysis of the normalized EXAFS term [9]. EXAFS analytical expression can be fourier transformed to obtain a radial function [10].

TABLE 1. molecular formula of the studied complexes

S. No.	Symbol	Abbreviation
1	Cu1	2 (C34H32N8O2Cl2Cu)
2	Cu 2	(C32H26N8Cl4)

METHODOLOGY: X-ray absorption spectra of the complexes have been recorded at the K-edge of copper using the energy dispersive EXAFS beam line at 2.5 GeV Indus-2 Synchrotron radiation source (SRS) [11-12]. The complexes are given in table 1. The data obtained has been processed using EXAFS data analysis program Athena. Firstly, the normalized μ (E) versus E spectra are obtained, then χ(k) versus k spectra are obtained and finally the Fourier transforms of the later are obtained.

The Fourier transform peaks at the radial distances of the neighboring atoms from the absorbing atoms. The distances found in Fourier Transform are, However, shorter by 0.2- 0.5Å than the actual distances due to energy dependence of the phase factors in sine function of the theoretical expression for EXAFS, known as EXAFS equation.

In the LSS method, the nearest neighbor distance can be determined by

$(1/2+n)\,\pi = 2k\,(R_1-\alpha_1) + 2\beta_1,$

Where R_1 is the bond length. $(R_1-\alpha_1)$ is the phase uncorrected bond length.

Both the LSS method and the Fourier transform method give the value of $(R_1-\alpha_1)$, i.e., both the methods give the values of bond lengths which have not been corrected for the phase shifts.

The data obtained is used to determine the metal-ligand bond length by the methods namely Lytle, Sayers and Stern (LSS) method [13-14] and Fourier Transform method. Theoretical **EXAFS** data has been generated and Fourier transformed using standard **EXAFS** equation employing the software "**Mathcad**,". The bond length so obtained from the analysis has been compared with bond length obtained using Lytle, Sayers and Stern (LSS) method and Fourier Transform method.

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RESULTS AND DISCUSSION:

Thetheoretical **EXAFS** data and its Fourier transform agree well with the experimental results. The results obtained are tabulated in table 2. The χ (k) versus k spectra of the data obtained experiment ally of the studied complexes have been shown in figure 1. The magnitude of Fourier Transform of χ (k) versus k Curves of figure 1 have been shown in figure 2. The theoretically generated EXAFS spectra and its Fourier Transform of the same complexes have been given in figure 3 and 4 respectively.

Table-2 Average values of metal-ligand bond length for copper complexes

Complexes	L.S.S. method $R_1-\alpha_1$	F.T. method R	Calculated values by Mathcad
Cu1	**1.106**	**1.11**	**1.04**
Cu2	**1.32**	**1.30**	**1.29**

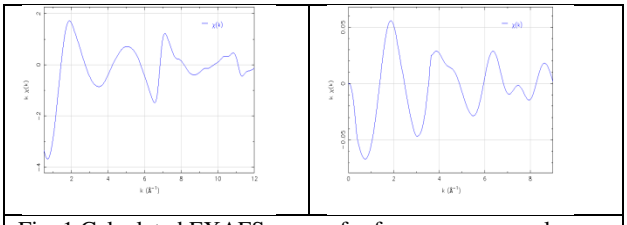

Fig. 1 Calculated EXAFS curves for four copper complexes obtained from FEFFIT program

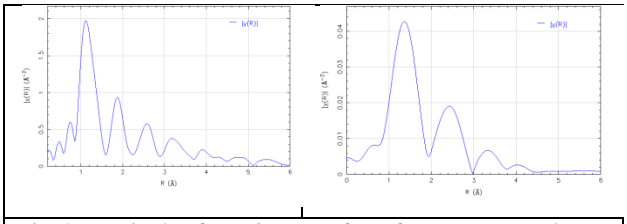

Fig. 2Magnitude of Fourier Transform for copper complexes obtained from FEFFIT program.

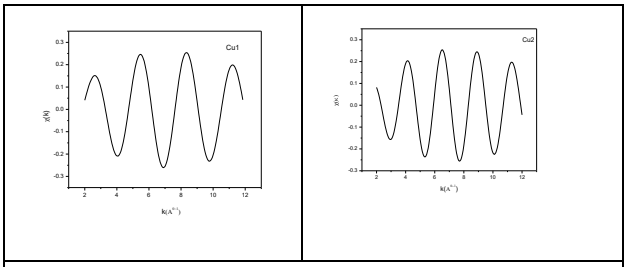

Fig. 3 Calculated EXAFS curves for copper complexes obtained from MathCAD software.

To determine the radial distance using LSS method n Vs k curve have been plotted as shown in figure 5. $(R_1-\alpha_1)$ can be obtained from the slope and β_1 can be obtained from the intercept of the curve. $(R_1-\alpha_1)$ gives the phase uncorrected bond length.

The bond length obtained from LSS method, Fourier Transform using Athena and Fourier Transform using MathCAD agree well with each other.

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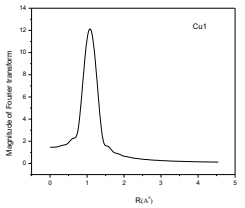


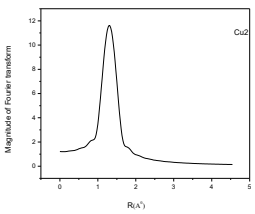
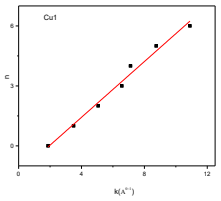


Fig. 4 Magnitude of Fourier Transform for copper complexes obtained from MathCAD software.



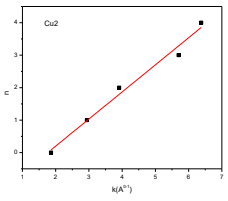


Fig. 5 n Vs k plot for four copper complexes

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