# An X-ray diffraction study of oxaloanilic acid hydrazone

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**Abstract** : X-ray diffraction study has been carried out for oxaloanilic acid hydrazone for both the structural characterization and the accountability of strain broadening effects with respect to the natural particle size. The structure of the sample has emerged as orthorhombic base centred system. The same has been tested for its antibacterial and antifungal activities.

**Keywords** : Oxaloanilic acid hydrazone, homogeneity, X-ray diffraction

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## 1. Introduction

The study of hydrazones (Ligands) and its complexes in recent years have received the special attention for their medical and biological applicabilities. Such ligands when tested for antibacterial and antifungal activities [1–4] seem to be promising. Hence, one such ligand oxaloanilic acid hydrazone has been undertaken in the present investigation for the detailed study of structural properties.

## 2. Experimental

The present ligand has been prepared by using the reported method elsewhere [5]. The EMARCK and BDH make reagent grade chemicals were used in the preparation and was purified by repetitive recrystallization process with ethanol. The purity of the product was checked by elemental analysis and physicochemical techniques.

Structure of the molecule was tentatively fixed as given in Figure 1, on the basis of electrical conductance, EPR, IR and Laser Raman studies.

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The XRD spectra were recorded in the range from 10° to 80° (2θ) on Philips make PW-1710 diffractometer attached to digitized computer along with graphical assembly in which CuKα radiation source connected with the tube of Cu-NF 2KV/20 mA was used.
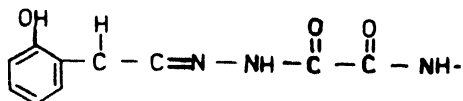
**Figure 1.** Structure of ligand.

## 3. The Results

The diffractogramme (Figure 2) consists of eleven reflections between 14° and 30° (2θ) with maximum reflection at $2\theta = 27.249°$, corresponding to $d = 3.15Å$.
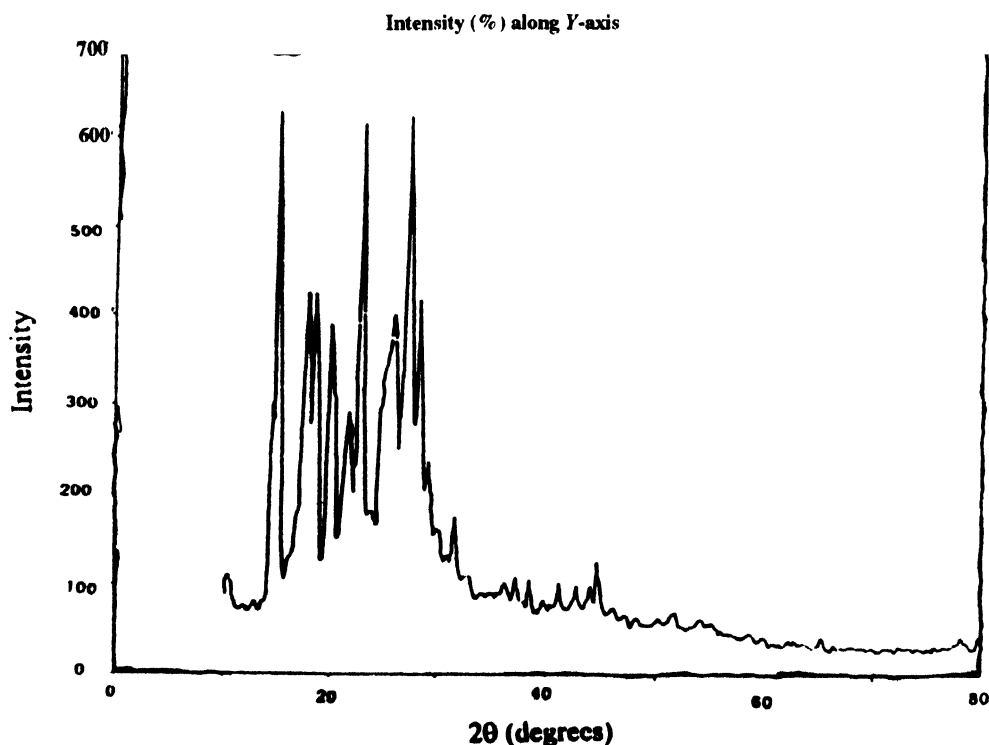


**Figure 2.** X-ray diffractogramme of ligand.

The observed 2θ's prominent peaks and corresponding observed $d$'s of the ligand were used for evaluating the calculated 2θ's and $d$'s. These are in-turn used for calculating $hkl$, miller indices and $d(Q)$, the standard deviation. All such data are presented in Table 1.

The experimental density was determined by the standard specific gravity method. Using this density and the values of molecular weight and volume of unit cell, the number of atoms $n$ ($\rho = nM/NV$) per unit cell thus emerged, was approximated to 2. With this number, theoretical density was fixed. Later the other parameters such as packing fraction, pore fraction, particle size, radius of the atom were then evaluated. Space group and point

group of the ligand were noted from International Table for X-ray crystallography [6]. All these values are entered in Table 2.

**Table 1.** Data on powder X-ray diffraction of oxaloanilic acid hydrazone.

| Peak No | $h\,k\,l$ | $2\theta_{obs}$ deg | $2\theta_{cal}$ | delta | $d_{obs}$ Å | $d_{cal}$ Å | $RI$ | delta $(Q)$ |
|---|---|---|---|---|---|---|---|---|
| 1. | 002 | 14.877 | 14.882 | 0.0053 | 5.9500 | 5.9479 | 62 3 | −0.1189E-04 |
| 2. | 301 | 17.905 | 17.894 | 0.0103 | 4.9500 | 4.9528 | 40.1 | 0.2767E-04 |
| 3. | 202 | 18.430 | 18.435 | −0.0046 | 4.8100 | 4.8088 | 43.9 | −0.1270E-04 |
| 4. | 301 | 20.258 | 20.283 | −0.0254 | 4.3800 | 4.3746 | 39.0 | −0.7694E-04 |
| 5. | 400 | 21.765 | 21.735 | 0.0301 | 4.0800 | 4.0856 | 31.5 | 0.9728E-04 |
| 6. | 401 | 22.962 | 22.997 | −0.0359 | 3.8700 | 3.8640 | 87.7 | −0.1224E-03 |
| 7. | 120 | 24.850 | 24.841 | 0.0089 | 3.5800 | 3 5813 | 36.9 | 0.3248E-04 |
| 8. | 121 | 25.955 | 25.961 | -0.0060 | 3.4300 | 3.4292 | 57.1 | −0.2278E-04 |
| 9. | 500 | 27.249 | 27.262 | −0.0131 | 3.2700 | 3 2685 | 100.0 | -0.5218E-04 |
| 10 | 501 | 28.309 | 28 293 | 0 0153 | 3.1500 | 3.1517 | 67.0 | 0.6320E-04 |
| 11. | 412 | 29.159 | 29.150 | 0.0090 | 3.0600 | 3.0609 | 28.3 | 0.3815E-04 |

**Table 2.** Evaluated parameters for oxaloanilic acid hydrazone (ligand) using XRD.

| | |
|---|---|
| Structure | Orthorhombic |
| Space group | *C222* |
| Laue group | *mmm* |
| Point group | 222 |
| Packing fraction | 53.49% |
| Symmetry of lattice | Base centered *C* |
| Vol of unit cell | 1427.11 Å$^3$ |
| Bond angles | $\alpha = \beta = \gamma = 90^\circ$ |
| Density ρ (Experimental) | 0.845 gr/cc |
| (Theoretical) | 0.659 gr/cc |
| Radius of atom | 4 5 Å |
| Vol. of atom | 381.7 Å$^3$ |
| Structure of molecule | Tetrahedral-class, Rhombic prism |
| Pore fraction | 28.2% |
| Thickness of particle | 175 Å |
| Lattice parameter | 6.3423 Å |
| | 7.3409 Å |
| | 11.8958 Å |

## 4. Discussion

The X-ray pattern was indexed using software by trial and error method. Keeping in mind the characteristics of various symmetry systems, the processing was done till a good fit was

obtained between the observed and calculated $2\theta$'s and $d$'s. It was tentatively arrived at the structure of the sample fixing it to be orthorhombic. The conditions [7,8] required for the sample to be orthorhombic such as $a \neq b \neq c$ and $\alpha = \beta = \gamma = 90°$ were tested and were found to be satisfied. The lattice parameters [$a$, $b$, $c$] of unit cell and the cell volume of the sample were calculated. These were further refined by weight fraction method. Such refined parameters were used for finding out space group and Laue group. All such calculated parameters are given in Table 1.

Particle size was determined by using the formula $t = 0.9\lambda/B \cos\theta$. As this parameter makes possible the distinguishibility between the naturel particle size and the particle size due to strain broadening effect hence it was sought of doing the analysis of XRD pattern in greater detail [9] This was done by calculating fullwidth at half maximum ($B$) corresponding to its Bragg's $\theta$ as against each peak and thereby computing cos and sin values. The nature and behaviour of these values for the present ligand is shown graphically in Figure 3.
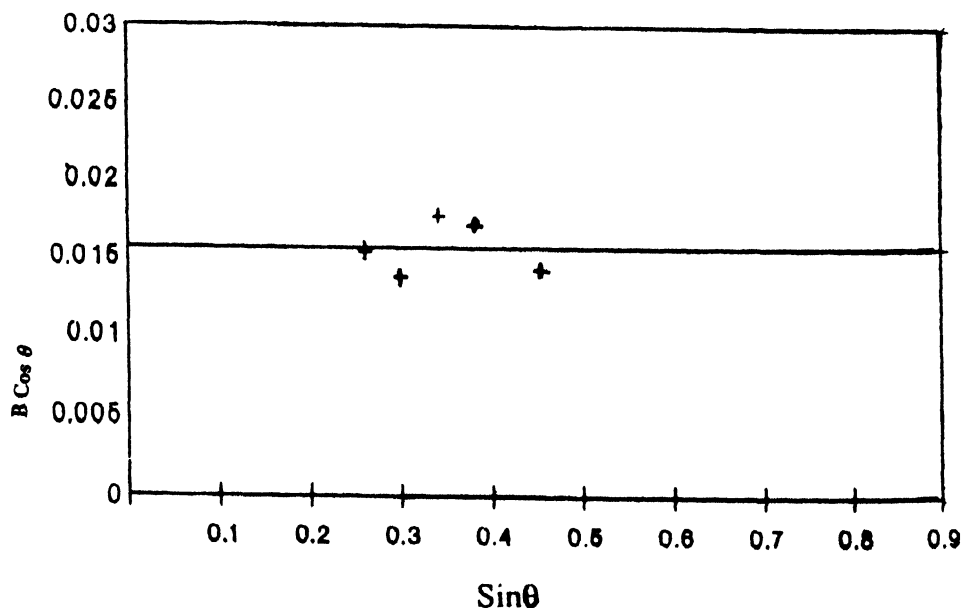


**Figure 3. Analysis of inhomogeneity.**

Zero slope (Figure 3) indicates non-presence of strains caused by inhomogeneous lattice distortions and/or compositional fluctuations. This trend may be attributed to the property of homogeneity of the present sample with respect to the particle size. Hence the estimated natural particle size as 175 Å, in the present finding may be more meaningful for the present ligand.

**References**

[1]  S R Girish, V K Revankar and V B Mahale *Transition Met. Chem.*, **21** 1 (1996)

[2]  I A Jahagirdar and B R Havinale *Transition Met. Chem*, **17** 539 (1992)

[3]  J A Jahagirdar and B R Havinale *Transition Met. Chem* **A30** 471 (1991)

[4]  N M Salunke, V K Revankar and V B Mahale *Transition Met. Chem.* **19** 53 (1994)

[5]  J A Jahagirdar, B G Patil and B R Havinale *Indian J. Chem.* **29(A)** 924 (1990)

[6]  J D H Donney and David Haker *Tables of space group criteria, crystallographiques, Naturaliste Canadien,* **67** 33 160 (1940)

[7]  B D Cullity *Elements of X-ray Diffraction* (Massachusetts : Addison-Wesley) (1956)

[8]  M J Burger *X-Ray Crystallography* (New York  Wiley) (1953)

[9]  N P Tendolkar *PhD Thesis* (Mumbai University, India) (1996)