



Powder x-ray diffraction pattern of 4-aminopyridine based copper (II) acetate crystals

¹ V. Ragavi ² P. Lalitha and ³ A. Sinthiya

^{1&2} Research Scholar ³ Assistant Professor

PG and Research Dept. of Physics, Srimad Andavan Arts and Science College, Trichy-5.

Corresponding Author: sinths@andavacollege.ac.in

ABSTRACT

The copper (II) acetate monohydrate reacts with 4-aminopyridine and crystallized in orthorhombic crystal system with lattice parameter $a = 7.5191 \text{ \AA}$, $b = 4.5842 \text{ \AA}$ and $c = 7.9624 \text{ \AA}$. The as grown crystals were crystallized using water and ethanol as the solvent by the slow evaporation method. There was change in planes (4 -2 1), (4 -1 0), and (4 -2 1) for the crystal which were grown in ethanol environment compared to the water environment.

KEY WORDS: 4-aminopyridine, powder XRD, particle size, orthorhombic

1 INTRODUCTION

Copper acetate have been used as fungicides, green pigments and used as reagents for the synthesis of various inorganic and organic compounds. The copper acetoarsenite is a powerful insecticide and fungicide termed as Paris green [1]. For treating Lambert-Eaton myasthenic syndrome and multiple sclerosis the 4-aminopyridine is used because by blocking the potassium channels. This increase transmitter release at the neuromuscular junction [2 - 10].

2 MATERIALS AND METHODS

2.1 PREPARATION

Solutions of 4-aminopyridine and copper (II) acetate monohydrate in water (20 ml) each are mixed in molar ratio of one is to one. Blue colour crystals were obtained by slow evaporation after a period of two weeks. Similarly the experiment was carried out using ethanol as a solvent and by the slow evaporation method after two weeks blue colour crystals were harvested.

2.2 POWDER X-RAY DIFFRACTION

Powder x-ray diffraction pattern were collected from diffracto-meter XPERT-PRO, with starting position $2\theta = 10.0231^\circ$, end position $2\theta = 80.9231^\circ$, step size $2\theta = 0.0500^\circ$, specimen

length = 10.00 mm, measurement temperature = 25°C, Cu as anode material and K-Alpha = 1.54060 Å.

3 RESULT AND DISCUSSION

Earlier report show that the copper (II) carboxylates reacts with a 4-aminopyridine and results in unstable violet colour crystal and stable blue colour crystal. The violet colour crystals were reported^[10] in tetragonal crystal system with $a = 10.6062 \text{ \AA}$, $b = 10.6062 \text{ \AA}$, $c = 31.1002 \text{ \AA}$, $\alpha = \beta = \gamma = 90^\circ$. But our work report that the 4-aminopyridine based copper (II) acetate monohydrate crystals crystallized in orthorhombic crystal system. The experiment were carried with two different solvents namely water and ethanol. The powder x-ray diffraction pattern for both the solvents are shown in figure 1 and they were indexed and tabulated in Table I.

The growth in two different solvent shows that there was change in planes (4 -2 1), (4 -1 0), and (4 -2 1) for the crystal which was grown in ethanol solvent compared to water environment. But both were crystallized in orthorhombic crystal system which varies from earlier reported work. Figure 2 shows the fill width half maximum value for the one peak. The Scherrer formula ($t = 0.9 \lambda / B \cos \theta_B$) is used to estimate the particle size of very small crystals.

The calculated breadth B of $2\theta^\circ$ due to small crystal effect alone of powder pattern line of particle is shown in table II. From this table we could confirm that the Breadth decreases with increase in particle size (t) with mosaic defect.

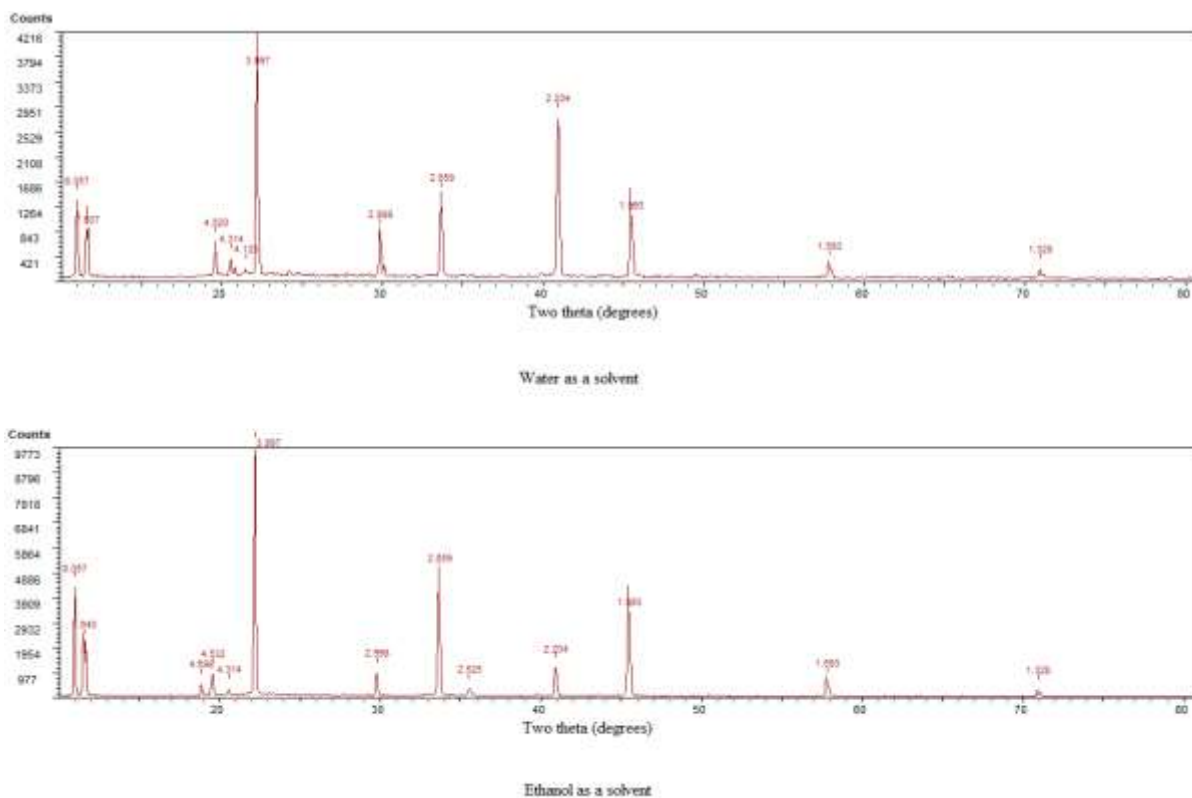


Fig 1: Powder x-ray diffraction pattern:

Pos. [$^{\circ}$ 2Th.]	Height [cts]	FWHM Left [$^{\circ}$ 2Th.]	d-spacing [\AA]	Rel. Int. [%]	h k l
11.0016	1106.57	0.1476	8.04237	29.44	
11.6284	670.50	0.1476	7.61022	17.84	2 -1 0
19.6013	642.58	0.1476	4.52905	17.10	1 1 0
20.5563	326.44	0.1476	4.32074	8.69	4 -2 1
21.4989	156.84	0.1476	4.13339	4.17	
22.2032	3758.61	0.1476	4.00385	100.00	0 0 2
24.1881	115.21	0.2952	3.67959	3.07	2 1 -1
29.8577	777.46	0.1476	2.99254	20.68	4 -1 0
33.6615	1396.65	0.1968	2.66258	37.16	1 0 2
40.9435	2713.95	0.1476	2.20428	72.21	4 -2 1
41.9935	72.67	0.1476	2.15156	1.93	4 0 -1
43.8340	53.54	0.5904	2.06540	1.42	2 1 0
45.4761	874.75	0.2460	1.99457	23.27	1 0 3
49.5191	67.29	0.3936	1.84077	1.79	2 2 0
57.7872	286.52	0.1968	1.59553	7.62	2 0 1

62.0478	41.16	0.2952	1.49582	1.10	0 2 2
70.9036	154.05	0.1476	1.32916	4.10	1 2 2

Table I: Indexing of powder x-ray diffraction pattern (water as a solvent)

Crystal data:

$a = 7.5191 \text{ \AA}$

$b = 4.5842 \text{ \AA}$

$c = 7.9624 \text{ \AA}$

$\alpha = \beta = \gamma = 90^\circ$

Crystal system: Orthorhombic

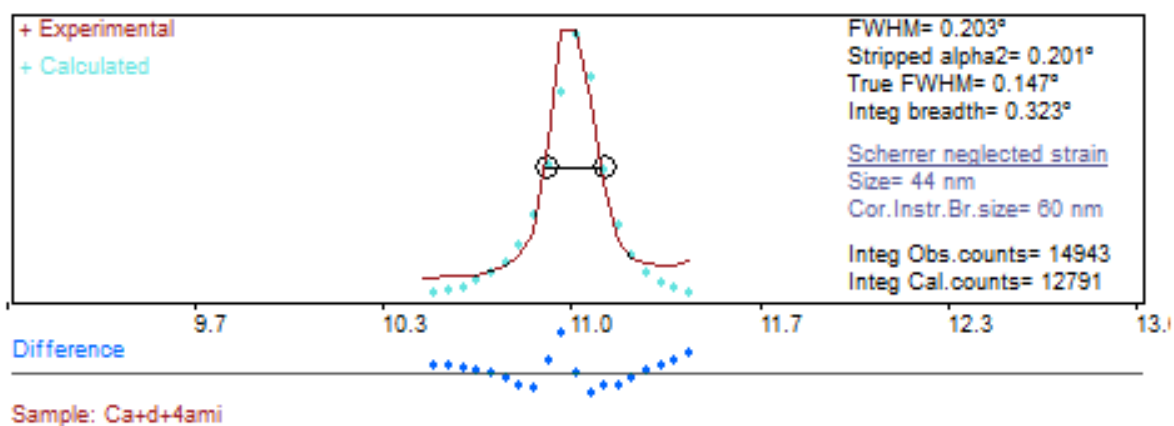


Fig 2 FWHM peak for selected hkl plane (for the crystals with water as a solvent).

S.No	Calculated t Å	B ° = 01- 02	0B°
1	170.45	1.16	45.4761
2	185.08	0.9	33.6615
3	223.84	0.82	40.9435
4	234.30	0.59	11.6284
5	241.53	0.62	22.2032
6	266.49	0.7	41.9935

Table II: Calculated (B) and (t) values (for the crystals with water as a solvent):

CONCLUSION

Selection of solvent will yield good and stable crystals in normal room temperature. Based on this the experiment were carried out and the blue colour crystal which was stable in air was obtained in both water and ethanol environment and powder x-ray diffraction pattern was used for crystal analysis.

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REFERENCE

- [1] https://www.copper.org/resources/properties/compounds/other_compounds.html
- [2] S. Alfred Cecil Raj, A. Sinthiya, Babu varghees, 4-aminopyridinium 5-carboxypentanoate monohydrate, Acta Cryst., E68,o2181 (2012)
- [3] Keith C. Hayes, The Use of 4-Aminopyridine (Fampridine) in Demyelinating Disorders, CNS Drug Reviews, Vol. 10, No. 4, pp. 295–316(2004).
- [4] Taccola, G. and Nistri, A., Acta Neurochir, 93 : 151-154 (2005).
- [5] P. Pohanish, Stanley A, Wiley guide to chemical incompatibilities, Greene, John Wiley and sons, (2009), P 64 [second edition]
- [6] Jude S. and Bever C., Pharmacol., 111 : 224 – 259 (2006).
- [7] Strupp, M., Malla, R., Dichgan, M., Fraitingner, T., Glasaner, S. and Brandt, T., Neurology, 62 : 1623-1625 (2004).
- [8] Schwid, S. B., Petrie, M. D., Mc Dermott, M.P, Tierney. D.S., Mason. D.H and Goodman A.D, Neurology, 48: 817-821 (1997).
- [9] Andersonn et al., Actacryst, E61, o1350-o1353 (2005).
- [10] Nina Lah, Joze Koller, Gerald Giester, Primoz Segedin and Ivan Leban . Copper (II) carboxylates with 4-aminopyridine: neutral mononuclear structures, isomerism of aceto compounds and a novel tetra nuclear structure. *New J. Chem.*, 26, 933-938 (2002) DOI: 10.1039/B110051C.