



## POWDER X-RAY DIFFRACTION PATTERN OF 4-AMINOPYRIDINE BASED COPPER (II) SULPHATE CRYSTALS

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### Abstract

The 4-aminopyridine based copper (II) sulphate crystals were crystallised in triclinic crystal system with cell parameter  $a = 8.4311 \text{ \AA}$ ,  $b = 7.4878 \text{ \AA}$ ,  $c = 9.0807 \text{ \AA}$ ,  $\alpha = 116.6^\circ$ ,  $\beta = 138.1^\circ$  and  $\gamma = 101^\circ$ . From the powder x-ray data we confirm that the breadth decreases with increase in particle size (t) with mosaic defect.

### Keywords

*4-aminopyridine, powder x-ray diffraction, copper (II) sulphate, triclinic system.*

### 1. Introduction

4-aminopyridine is a potassium channel-blocking agent, used as a bird repellent, and also it increases calcium influx at presynaptic terminals thereby enabling improvement of neuromuscular transmission in myelinated neurons [1-7]. Copper Sulphate is particularly useful in demanding applications, such as copper plating and electro less copper plating.

### 2 Materials and Methods

#### 2.1 Preparation

Solutions of 4-aminopyridine and copper (II) sulphate 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 three weeks.

#### 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 =  $25^\circ\text{C}$ , Cu as anode material and  $K_\alpha = 1.54060 \text{ \AA}$ .

### 3. Result and Discussion

Powder x-ray diffraction data were used to investigate the crystal system and crystalline nature of the crystals. X-ray diffraction pattern of the 4-aminopyridine based copper II sulphate has been shown in figure 1. All the sharp peaks corresponding to the different scale particles confirms the crystalline nature of the compound. The pure complex were crystallised in triclinic crystal system with cell parameter ' $a$ ' = 8.4311 Å, ' $b$ ' = 7.4878 Å, ' $c$ ' = 9.0807 Å,  $\alpha$  = 116.6°,  $\beta$  = 138.1° and  $\gamma$  = 101°. The powder x-ray diffraction pattern were indexed and tabulated in table 1.

Good crystal growth were identified in the direction of plane (-2 0 2) for the crystal which was grown in water environment. Figure 2 shows the full width half maximum value for the one peak. The Scherrer formula is used to estimate the particle size of very small crystals. The calculated breadth  $B$  of  $2\theta$  ° due to small crystal effect alone of powder pattern line of particle is shown in table 2. From this table we could confirm that the Breadth decreases with increase in particle size ( $t$ ) with mosaic defect.

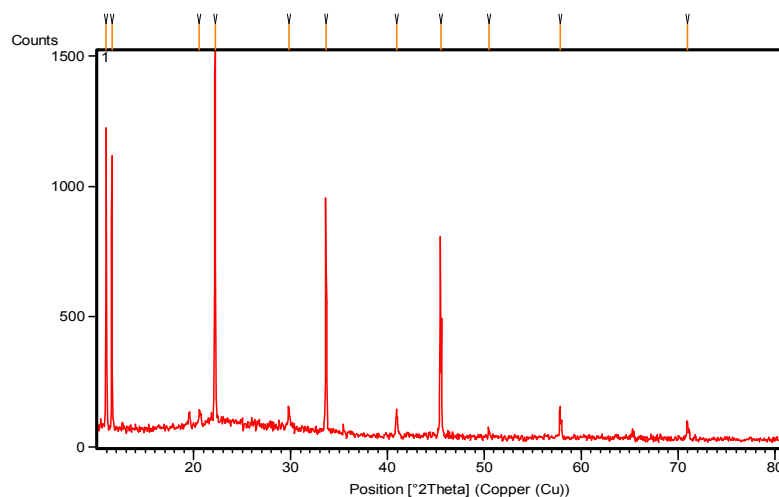


Figure 1 Powder x-ray diffraction pattern of the complex

Table 1 Indexing of powder x-ray diffraction pattern

| Pos. [°2Th.] | Height [cts] | FWHM Left [°2Th.] | d-spacing [Å] | Rel. Int. [%] | h k l  |
|--------------|--------------|-------------------|---------------|---------------|--------|
| 10.9621      | 1205.72      | 0.1476            | 8.07121       | 76.39         | 1 0 0  |
| 11.5598      | 1142.60      | 0.1476            | 7.65523       | 72.40         | 0 0 1  |
| 29.8281      | 116.16       | 0.1968            | 3.99545       | 7.36          | -2 0 2 |

| Pos. [°2Th.] | Height [cts] | FWHM Left | d-spacing | Rel. | h k l |
|--------------|--------------|-----------|-----------|------|-------|
|--------------|--------------|-----------|-----------|------|-------|

|         |        | [°2Th.] | [Å]     | Int.<br>[%] |        |
|---------|--------|---------|---------|-------------|--------|
| 33.6672 | 699.99 | 0.1476  | 2.99314 | 44.35       | 0 -2 1 |
| 40.9564 | 89.61  | 0.1476  | 2.0561  | 5.68        | 3 0 0  |
| 45.4864 | 691.34 | 0.1968  | 1.99414 | 43.80       | 2 -3 0 |
| 50.4711 | 28.86  | 0.1476  | 1.80826 | 1.83        | 1 1 0  |
| 57.8141 | 122.91 | 0.2460  | 1.59486 | 7.79        | 0 2 0  |
| 70.9336 | 73.81  | 0.1476  | 1.32867 | 4.68        |        |

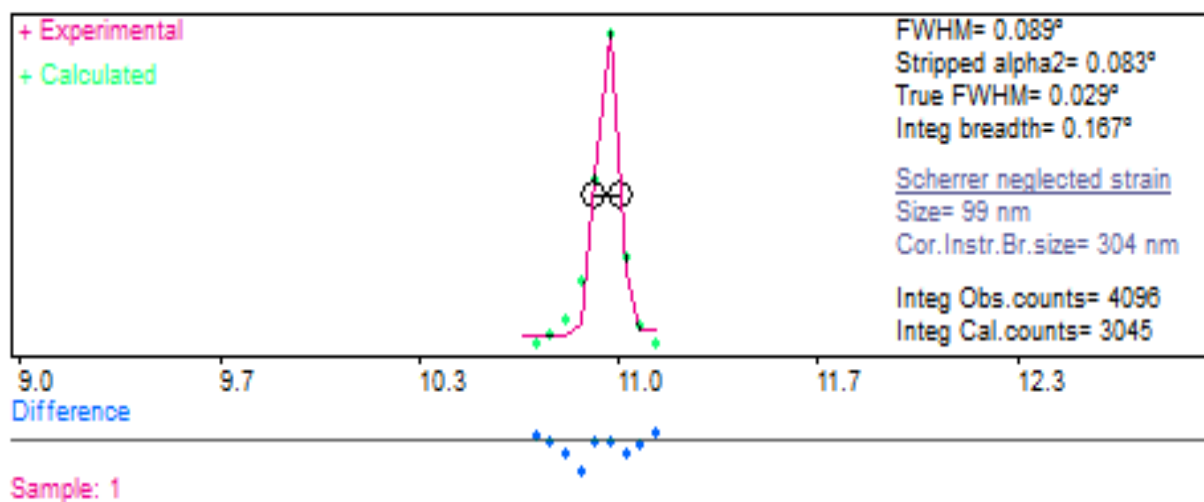


Figure 2 FWHM plot for one peak

Table 2: Calculated B and t values:

| S.No | Calculated t Å | $B^{\circ} = \theta_1 - \theta_2$ | $\Theta_B^{\circ}$ |
|------|----------------|-----------------------------------|--------------------|
| 1    | 173.03         | 0.81                              | 10.98              |
| 2    | 262.4          | 0.7                               | 40.99              |
| 3    | 284.45         | 0.5266                            | 22.24              |
| 4    | 350.83         | 0.7435                            | 57.89              |

|   |        |      |       |
|---|--------|------|-------|
| 5 | 565.02 | 0.35 | 45.48 |
|---|--------|------|-------|

#### 4. Conclusion

The 4-aminopyridine interact with copper II sulphate in water, and blue coloured crystals were harvested using slow evaporation method and the crystal system and the particle size of the as grown crystals were calculated using the powder X-ray diffraction pattern.

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