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# Process Development and Characterization of 2-Aminopyridine Potassium Di-Hydrogen Phosphate Cadmium Chloride (2APKDPC) Crystal

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***ABSTRACT:*** *A good optical quality crystal of 2-aminopyridine potassium dihydrogen phosphate cadmium chloride (2APKDPC) was grown by slow evaporation technique at room temperature. The transparent and defect less bulk crystal of 2APKDPC with dimension 23 x 10 x 5 mm$^3$ have been grown by solution growth method in the period of 90 to 95 days. From the single crystal X-ray diffraction study the measured lattice parameters values are a = 7.49 Å, b = 7.49 Å, c = 7.03 Å, α = β = γ = 90° and volume V= 394 Å$^3$, which shows that the grown crystal 2APKDPC belongs to tetragonal crystal system. The crystalline nature of the grown crystal was confirmed by powder X-ray diffraction analysis. The metal coordination and the presence of various functional groups present in 2APKDPC crystal are identified by FTIR analysis. In the recorded optical transmission spectrum, the UV cut-off wavelength was found to be at 237 nm and the band gap value also found from the absorption studies. The photoluminescence study reveals the emission of blue fluorescence from 2APKDPC crystal. The optical second harmonic generation efficiency of 2APKDPC crystal was found to be 1.23 times that of standard KDP.*

***Keywords :*** *XRD, FTIR, UV, Optical band gap, Photoluminescence.*

## I. INTRODUCTION

The search for new materials with high optical non-linearity's has captured attention, because of their practical applications in higher harmonic generation, amplitude and phase modulation, laser technology, switching and other signal processing device. Non-linear optical (NLO) crystals with high conversion efficiency for second harmonic generation (SHG) and transparent in the visible and ultra violet ranges are required for various devices in field of optoelectronics and photonics [1-3]. Some complexes of the amino acids with simple organic and inorganic salts appear to be promising for optical second harmonic generation (SHG). The crystal display interesting physical and chemical properties, exhibiting phase transition with ferroelectric, antiferroelectric and ferroelastic behavior as well as phase with commensurate and incommensurate superstructures [4,5]. But possess relatively optical non-linearity [6,7]. So the semiorganic single crystals are attracting great attention in the field of non-linear optics because of their high optical non-linearity, chemical flexibility of ions, thermal stability and excellent transmittance in the UV visible region[8,9]. Among the various property of semiorganic non-linear optical materials, metal complexes have received potentials interest, because they can be effectively used as the better alternatives for KDP crystals in the frequency doubling process and laser fusion experiments [10, 11]. Materials of amino groups possess particular feature, such as week vanderwaals and hydrogen bonds, wide transparency range in the visible region and zwritterionic nature of the molecules [12, 13]. In this present work we focused on studies of spectral and physicochemical properties of a new semiorganic crystal 2-aminopyridine potassium di-hydrogen phosphate cadmium chloride (2APKDPC) for the first time.

## II. EXPERIMENTAL PROCEDURE

### 2.1 Synthesis of 2APKDPC

The raw material 2APKDPC compounds are synthesized by using analytical reagent grade of 2-aminopyridine, Potassium dihydrogen phosphate and Cadmium chloride in equimolar ratio 1:1:1 using de-ionized water as a solvent at room temperature by slow evaporation solution growth technique. The solution was stirred for more than 8 hours using magnetic stirrer to get homogeneous solution. The chemical equation of the synthesized product is shown below.

$$C_5H_6N_2 + KH_2PO_4 + Cd\,Cl_2 \rightarrow Cd\,(C_5H_6N_2\,KH_2PO_4)\,Cl_2$$

**2.2 Crystal Growth of 2APKDPC**

The saturated solution of 2APKDPC was filtered using good quality micro filter paper to remove the insoluble impurities. The filtered solution transferred into beaker and sealed with a polythene sheet with few holes. The beaker was kept in an undisturbed and dust free environment. After the time period of 35 days the tiny seed crystals were observed. Macroscopic defect free transparent crystals were selected as seed for growing large size single crystal of 2APKDPC. As grown seed crystal was introduced into the mother solution. 2APKDPC seed crystal was grown as a bulk crystal with dimension 23 x 10 x 5 $mm^3$ in the period of 90-95 days. The photography of as grown crystal of 2APKDPC is shown in the Figure 1.

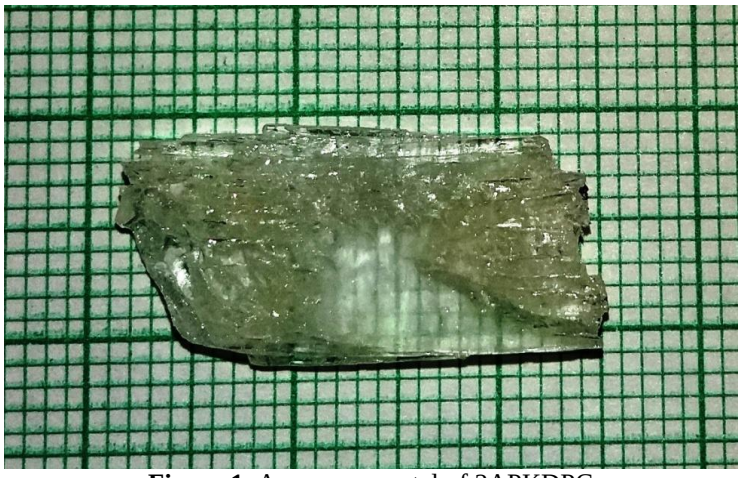

**Figure 1.** As grown crystal of 2APKDPC

## III. CHARACTERIZATION

**3.1. Single crystal X-ray diffraction Analysis**

2APKDPC crystal was analyzed by single crystal XRD method by ENRAF NONIUS CAD4-F single X-ray diffractometer with $MOK_{\alpha}$($\lambda$=0.717Å) radiation. The calculated lattice parameter values are, a = 7.49 Å, b = 7.49 Å, c = 7.03 Å, $\alpha = \beta = \gamma = 90^{o}$ and volume V= 394 $Å^3$, which shows that the grown crystal 2APKDPC belongs to tetragonal crystal system.

**3.2. Powder X-ray diffraction analysis**

The powder sample of 2APKDPC have been analyzed by using BRUCKER, Germany (model D8 Advance) X-ray diffractometer with $CUK_{alpha}$ (wavelength = 1.5405Å) radiation. The powder sample was scanned in the range of 10-$80^{o}$ at the rate of $1^{o}$ per minute. The powder XRD pattern of 2APKDPC crystal is shown in the Figure 2. The presence of sharp well-defined Bragg's peaks confirms the good crystalline nature of the grown crystal 2APKDPC.

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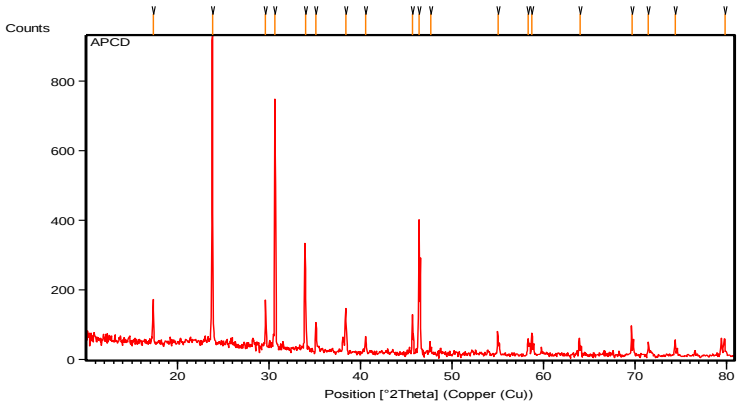


**Figure 2.** Powder XRD pattern of 2APKDPC crystal

### 3.3. FTIR spectral analysis

The FTIR spectroscopy studies are effectively used to identify the functional groups, presents in the grown crystal and to determine the molecular structure. In order to analyze qualitatively the presence of functional groups, freshly crushed powder of 2APKDPC crystal was subjected to FTIR studies using thermo Nicolet v-200 FTIR spectrometer by KBr pellet method in the range 500-4000 $cm^{-1}$. Figure 3 shows FTIR spectrum of 2APKDPC crystal. The absorbed frequencies and their assignment of 2APKDPC crystals are shown in the Table1.

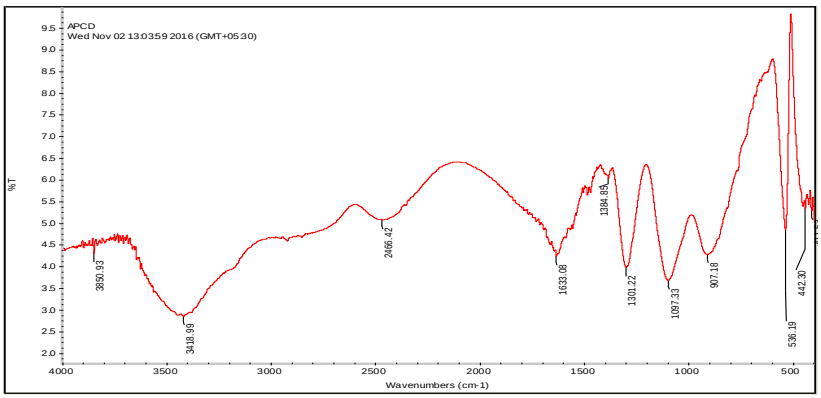


**Figure 3.** FTIR spectrum of 2APKDPC crystal

**Table 1.** Wavenumber assignments of 2APKDPC crystal

| Wavenumber $cm^{-1}$ | Assignments |
|---|---|
| 3418 | N-H stretching |
| 2466 | O-H bending |
| 1633 | P-O-H stretching |
| 1384 | C=O symmetric stretching |
| 1301 | P=O stretching |
| 1097 | CCN asymmetric stretching |
| 907 | $CH_2$ rocking |
| 536 | P-O-H deformation / K-O stretching |
| 442 | presence of metal atom |

### 3.4. Linear optical study

The UV-visible spectrum of the 2APKDPC crystal has been recorded using DOUBLE BEAM UV-Vis spectrophotometer: 2202 in the region 200- 1000 nm and the spectrum shown in Figure 4. Optically polished 2APKDPC single crystal of thickness 2 mm was used for this study. It is inferred from the spectrum that large transmission window in the entire visible region. Interestingly, the UV cut-off wavelength of the grown crystal occurs below 237 nm. It is well known that efficient NLO crystal has an optical transparency at lower cut-off wavelength between 200 and 400 nm. In the entire visible region, the optical absorption spectrum is flat and constant. The wide transmission range in the entire visible region is a desirous useful property for optoelectronic applications.

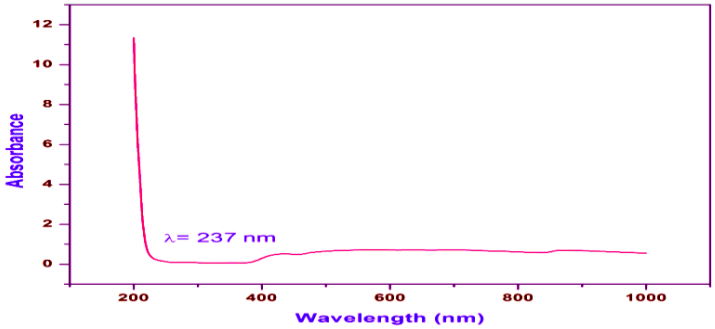


**Figure 4.** Optical absorption spectrum of 2APKDPC

The optical band gap of the 2APKDPC crystal was calculated using the Tauc's relation. A graph has been drawn between $(\alpha h\nu)^2$ versus $h\nu$ , which is shown in Figure 5 and the estimated optical band-gap is 5.1 eV.

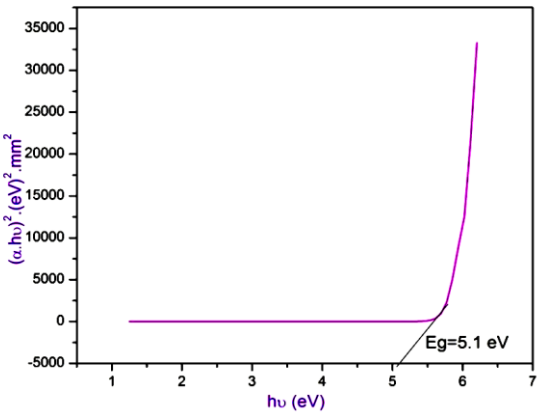


**Figure 5. Tauc's plot of 2APKDPC crystal**

### 3.5. Photoluminescence (PL) study

The emission spectrum of 2APKDPC was recorded using Cary Eclipse spectrophotometer. The PL study finds wide applications in the field of medical, biochemical and chemical research fields for analyzing compounds. Photoluminescence in solids is the phenomenon in which electronic states of solids are excited by light of particular energy and the excitation energy is released as light. The photon energies reflect the variety of energy states that are present in the material. Figure 6 shows PL emission spectrum recorded in the range of 250–500 nm with an excitation wavelength of 260 nm. The highest emission peak from the spectrum was observed to be at 484 nm. Other peaks 357 nm and 419 nm are due to anionic and cationic nature of the sample. From this wavelength it is concluded that 2APKDPC crystal emits blue fluorescence.

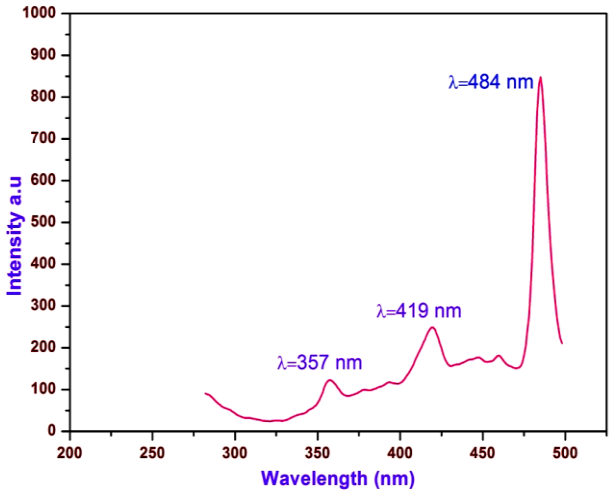


**Figure 6.** Photoluminescence emission spectrum of 2APKDPC crystal

### 3.6. Nonlinear Optical Study of 2APKDPC Crystal

Second harmonic generation test was performed by powder technique developed by Kurtz and Perry. The powder sample of 2APKDPC crystal was illuminated using the fundamental beam of 1064 nm from Q-switched ND:YAG laser. The powder SHG efficiency of 2APKDPC crystal was about 1.23 times greater than that of standard potassium Dihydrogen phosphate (KDP). The good second harmonic generation efficiency indicates that the grown crystals can be used as a suitable material for optical devices.

## IV. CONCLUSION

Good quality transparent crystal of 2APKDPC was grown successfully by slow evaporation technique. The crystallinity and crystal system confirm by X-ray diffraction studies. The grown 2APKDPC crystal belongs to tetragonal crystal system. The crystalline nature was confirmed by powder XRD analysis. The various functional groups presence in the grown crystal was identified by FTIR studies. The UV cut off wavelength of 2APKDPC crystal is found to be around 237 nm, which reveals grown crystal is potential candidate for NLO applications. The band gap was found to be 5.1 eV using Tauc's plot. The emission of blue fluorescence from the 2APKDPC crystal was confirmed by photoluminescence study. SHG efficiency was found to be 1.23 times greater than that of KDP.

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