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# Structural and Spectral Studies of Sulphamic Acid NLO Single Crystal

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**Abstract :** We report here the growth of sulphamic acid NLO single crystals by slow evaporation technique. Sulphamic acid single crystals of dimension 10×12×10 $mm^3$ were harvested in a period of a month. The as grown sulphamic acid NLO single crystals were characterized by XRPD, FTIR, UV-vis-NIR spectral analyses and dielectric studies. The second harmonic generation was confirmed by Kurtz - Perry powder test.

**Key words :** NLO, slow evaporation, sulphamic acid, XRPD, FTIR, UV-vis-NIR

## 1. Introduction

In the last ten years, investigation on new laser crystalline hosts wants for middle infrared application has been preceding apace [1-4]. Hence the synthesis of organic molecules with large hyper polarizabilities is under extensive investigation for their possible non linear

(NLO) application such as second harmonic generation and electroptic modulation; for future information technology and telecommunication [5]. Therefore the reference or organic materials in this interesting context is because the delocalized electronic structure of Π-conjugated organic compounds offers a number of tempting opportunities is application as NLO materials [6]. In particular, Π-conjugated systems linking a donor and acceptor show a large NLO response and hence have been well studied. In addition to the advantage in synthesis of organic materials have ultrafast response time photo stability and large first order hyperpolarizability values. Hence organic materials are projected as fore front candidates for fundamental and applied investing actions [7]. Triketohydrindane hydrate [8], Benzoyl Glycine [9] and N-Methyl Urea [10] are a few such compounds reported by us. Therefore efforts have been made to develop ultraviolet lasers for photonics, optoelcetronics, infrared and medical applications [11-13]. In this context sulphamic acid, an organic NLO material which crystallizes in the non-centro symmetric space group is our interest. Sulphamic acid is highly crystallize, transparent and colorless crystal. The crystal structure was solved on single crystals growth from deionized water by R.Valluvan et al [14], who interpreted XRD measurements with an orthorhombic space group $P_{bca}$. In this report the growth of sulphamic acid along with XRPD, FTIR and UV-is-NIR analyses is reported.

## 2. Experimental Techniques

### 2.1. Growth of Sulphamic acid single crystal

Appropriate stoichiometric ratio of sulphamic acid (E-mark) was taken and dissolved in reionized water. The solution prepared was kept as such for a period of thirty days using a 250 ml borosil beaker. The beaker was closed using a peaforated cover having ting holes, so as to facilitate the slow evaporation process at room temperature. Optical defect free single crystals of dimension 12 x 10 x 15 $mm^3$ were harvested .The photograph of as grown sulphamic acid single crystal is shown in Fig.1.

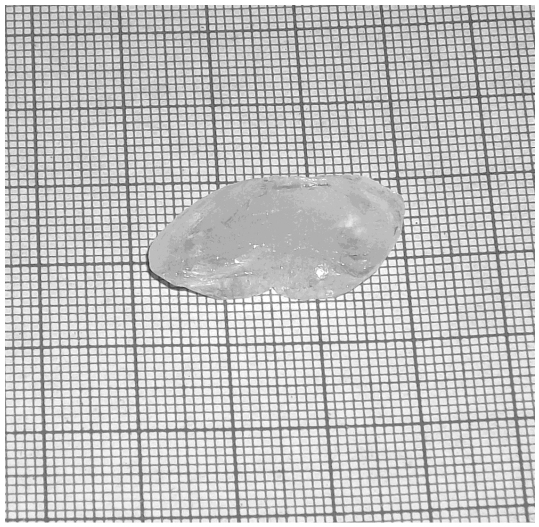

Fig.1. Photograph of as grown crystal of sulphamic acid

## 3. Characterization Techniques

### 3.1. XRPD analysis

X-ray pattern of the as grown crystal of sulphamic acid was recorded using XPERT Powder diffractometer with **CuK**$_\alpha$ ($\lambda$=1.541 Å) radiation. The lattice parameters were calculated using X'Pert Software. The results obtained are good agreement with the report values [14-15]. The XRPD diffractogram of sulphamic acid single crystal is shown in Fig.2. Form X-ray diffractogram, it is well clear that the grown sulphamic acid crystallize in a material is free from impurity.

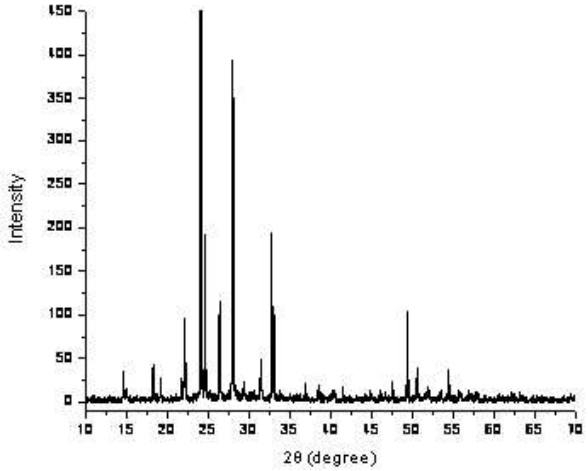


Fig.2. XRPD spectrum of sulphamic acid single crystal

## 3.2. FTIR spectral analysis

The FTIR spectrum of the as grown sulphamic acid was recorded in the frequency region 400-4000 $cm^{-1}$ .The instrument used was Perkin - Elemer lamda -35 spectrophotometer with a scanning speed of 2mm/s in KBr phase. The recorded FTIR spectra were compared with standard spectra of financial groups [16-17]. The FTIR spectrum of sulphamic acid single crystal is shown in Fig.3. The FTIR spectrum of a sulphamic acid shows a peak at 3000 $cm^{-1}$ corresponds O-H stretching frequency. The sharp at peak at 1455 $cm^{1}$ is attributed to O-H bending. A peak at 1069 $cm^{-1}$ is due to $SO_3$ deformation. The peaks assignable at 687 and 539 $cm^{-1}$ are due to N-H sketching and $SO^{3-}$ deformation. Thus the functional groups are confirmed.

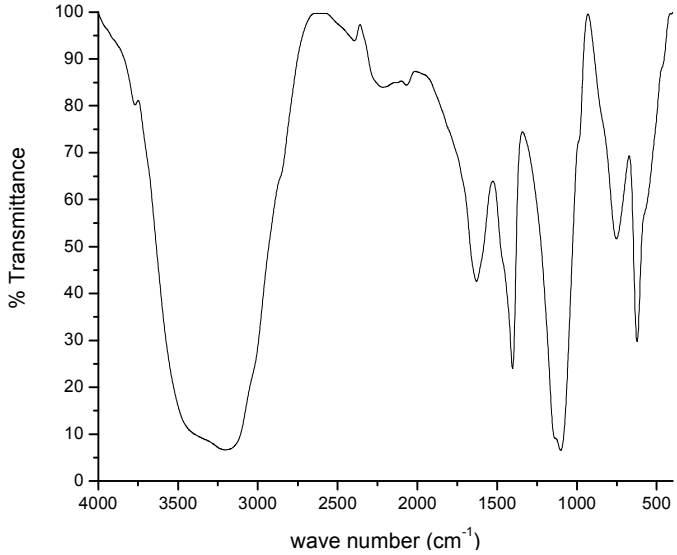


Fig.3. FTIR spectrum of sulphamic acid single crystal

## 3.3 UV-vis- NIR analysis

The UV visible absorption spectrum of sulphamic acid crystal was recorded in the wavelength region 190 nm to 1100 nm, using a Varian Carry 5E model spectrophotometer and the obtained spectrum is shown in Fig. 4. When the absorption is monitored from longer to shorter wavelength, the absorption is found to be moderately low in the visible region and near to the IR region of the spectrum. This is the most desirable property of material processing activity. The octal is

highly transparent to the entire w, visible and near IR region. The cut off wavelength is which the transmittance fails to zero is found to be 190 nm.

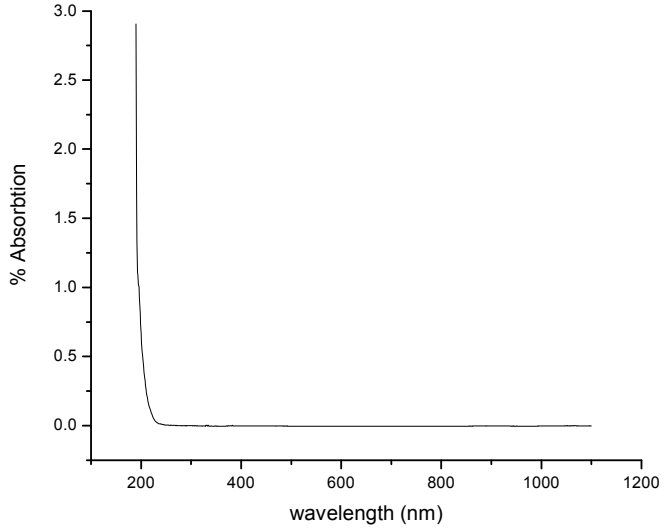


Fig.4. UV-vis-NIR spectrum of sulphamic acid single crystal

## 3.4. NLO Test

The first and the most widely used technique for confirming SHG from prospective SONLO materials is the Kurtz-Perry test. The frequency of 1064 nm from Q-switched Nd:YAG laser was used to test the SHG property of sulphamic acid crystal by using Kurtz test. The output from the Q-switched laser was focused into the crystals. The emission of green radiation from crystal confirms the SHG of the crystal.

## 4. Conclusion

An organic sulphamic acid NLO crystal was grown by slow evaporation method. XRPD analysis confirms the purity of the crystal. The functional groups in the molecule were identified by FTIR analysis. The cut-off wavelength of the crystal was evaluated from UV-vis-NIR analysis.

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