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Synthesis, growth and characterization Studies of Semi organic NLO L-Valine Calcium nitrate single crystals

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Abstract : Single crystals of semi organic non-linear L-Valine Calcium nitrate grown by slow evaporation method using water as a solvent. The L-Valine phase was confirmed by single crystal powder X-ray diffraction analysis. Presence of various functional groups of L-Valine was characterized by Fourier transform infra-red spectrum (FT-IR) and the non-linear optical property is analysed by Kurtz powder technique. The optical behavior was examined by Ultra violet –vis spectrum and found that the crystal is transparent in the region between the 245-1100nm. Hence it may be very much useful for the second harmonic generation (SHG) applications.
Keywords : Semi-organic nonlinear single crystal, L-Valine, Slow evaporation method, Second harmonic generation.

1. Introduction

In recent years, organo-inorganic hybrid materials have attracted considerable attention. In particular, the inorganic derivatives of protein amino acids are often attributed to symmetric groups without an inversion Centre mostly the polar symmetry groups. The crystals have properties whose symmetry is described by odd - rank tensors such as pyro-electric effect, spontaneous electric polarization, piezoelectric effect, generation of second optical harmonics, etc. Moreover crystals that belong to the eleven enantiomorphic point groups, having no mirrorreflection planes exhibit optical activity, which is described in terms of the axial generation tensors. While the structures of most amino acids are well defined, the structures of the derivatives of the protein amino acids with inorganic components are not. This paper defines the crystal structure of L-valine Calcium chloride [VCC]. The Functional groups are analysed by the FTIR studies, its crystalline nature is studied by the powder XRD, the transmittance and absorbance of electromagnetic radiation is studied through UV-Visible spectrum.

2. Experimental

2.1. Synthesis and crystal growth

L-valine Calcium nitrate were received from Sisco Research Laboratories PVT. Ltd (India).The present wave is the long recrystallization processes and available raw material is used one after purification. L-valine and Calcium nitrate is taken in a particular molar ratio and added 20 ml of double distilled water and stirring till dissolved the mixture. After dissolving the above mixture is transferred in to 100 ml beaker. The mixing solution kept in slow evaporation, the beakers are covered with aluminum foil sheet at room temperature. The grown crystal was colourless, good transparent crystals were obtained by 3 to 4 weeks.

Chemical reaction

$C_5H_{11}NO_2 + CaNO_3$----------->$C_5H_{11}NO_2$.$CaNO_3$

3.Characterization.

3.1.Powder X-ray diffraction

The grown single crystal of L-Valine potassium nitrate and L-Valine Lithium nitrate has been subjected to powder X-ray diffraction. Powder form of the crystal is taken for the analysed by XPERT PRO diffractometer. The indexed powder x-ray diffraction pattern of the grown crystal is presented in fig 3. The lattice parameters obtained from the data of powder XRD pattern using UNITCELL software package are a = 9.768 Å, b = 6.832 Å and c = 12.020372 Ácellvol=433.11Å3 and are found to be in good agreement with the literature.

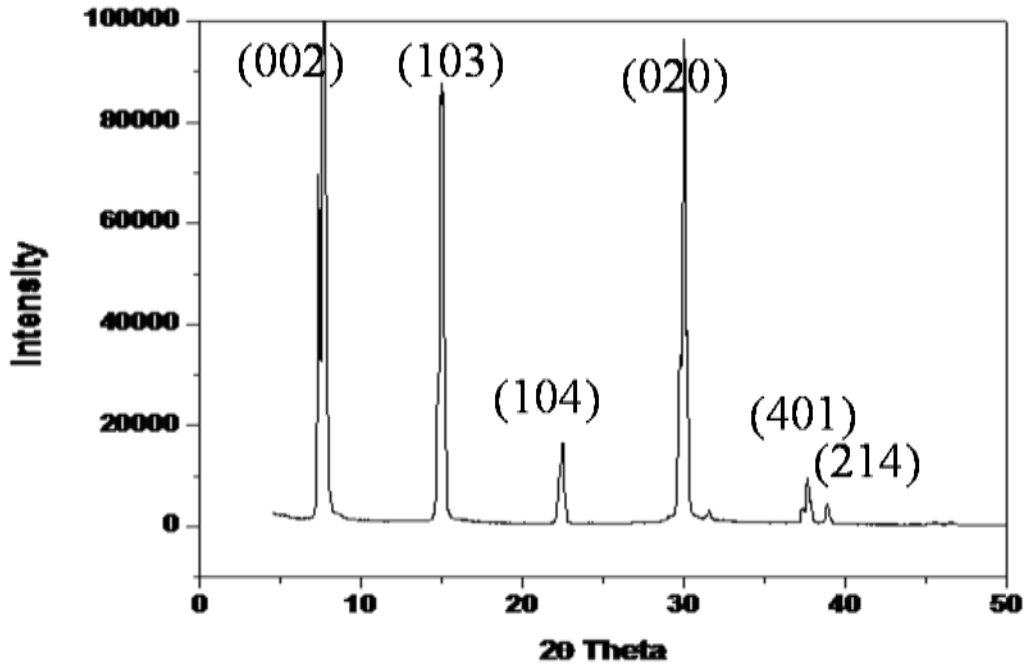

Fig.1. XRD pattern of L-Valine with Calcium nitrate

Table .1 XRD values of L-Valine with Calcium nitrate

Parameters	Present study	Reported velues
A	9.768	9.9714
B	6.832	6.2930
C	12.020372	12.6480
V	433.11	434.22
System	monoclinic	Monoclinic
Space group		P2

3.2.FTIR spectral analysis

The FTIR spectrum of VCN crystals was recorded in the range 400-4000cm-1 by employing a Perkin-Elmer spectrometer using KBr pellet method to study the metal organic coordination. Fig.2 shows the recorded

FTIR spectrum of the grown crystal of VCN. The vibrational frequency of various functional groups of VCN tentative frequency assignment are presented in Table.2.

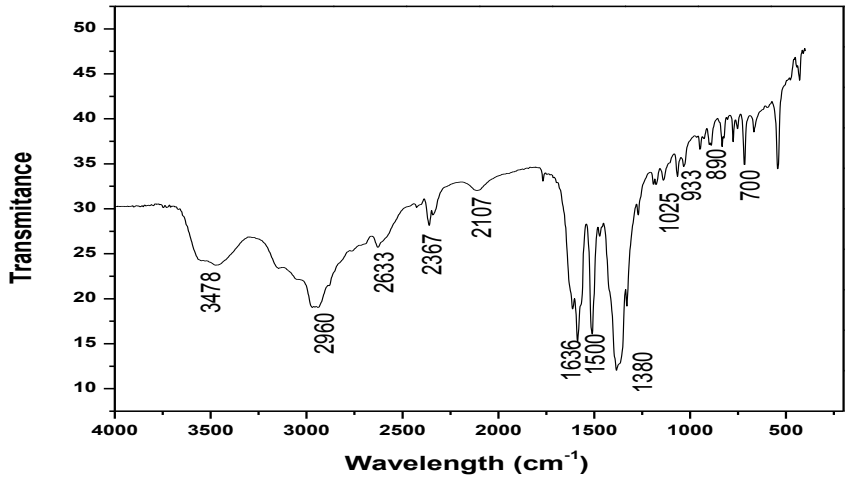

Fig.2. FT-IR of of L-Valine with Calcium nitrate

Table-2: Frequencies of the fundamental vibrations of VCN

IR peak	Assignments	Frequencies
		VCN
1	NH_3 asymmetric stretching	1630
2	NH_3 symmetric stretching	1510
3	CH_3 symmetric stretching	1390
4	$(CH_3)_2$symmetricstretching	1030
5	C-C symmetric stretching	955
6	C-C-N symmetric stretching	915
7	CH_3 rocking	703

3.4. Optical transmission spectra

A transmission spectrum is very important for NLO materials, because a nonlinear optical material for any practical use if it has a wide transparency window. In the present study, we have recorded the UV-Vis NIR transmission spectrum in the range of 200nm-1100nm is shown in fig.3.From the spectrum, it is seen that the crystal has a lower cut-off wavelength of 272nm.The crystal is transparent in the visible and infrared spectral regions. Optical transmittance of about 100%is observed for 1.5mm plates of L-Valine Calcium nitrate crystals and is sufficiently good for SHG.

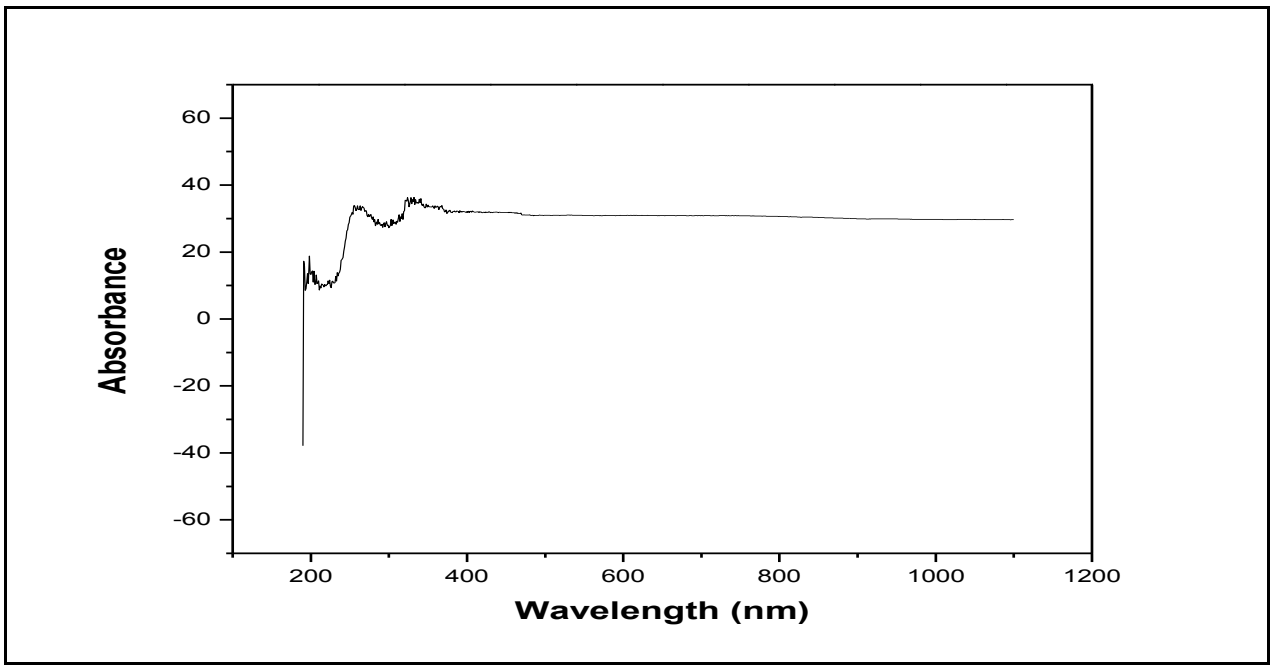

Fig.3. UV-Vis spectra of L-Valine with Calcium nitrate

3.4. Second Harmonic Generation Test

The second harmonic generation (SHG) test on the VLN and VPN crystal was performed by Kurtz powder SHG method [18].The powdered sample of crystal was illuminated using the fundamental beam of 1064 nm from Q-switched Nd:YAG laser. Pulse energy 4ml/pulse and pulse width of 6 ns and repetition rate of 10Hz were used. The second harmonic signal generated in the crystalline sample was confirmed from the emission of green radiation of wavelength 532 nm collected a monochromator after separating the 1064 nm pump beam with an IR-blocking filter. A photomultiplier tube is used as a detector. It is observed that the measured SHG efficiency of VCN crystal was 0.8times that of potassium dihydrogen phosphate (KDP).

4. Conclusion

Single crystals of L- valine Calcium nitrate was successfully Synthesized by solution growth technique. Its lattice dimensions have been determined from the powder X-ray diffraction analysis. The various functional groups have been identified from the Fourier transform infra-red (FT-IR) analysis. The grown crystal has good transmission window in the visible region between(270 nm and 280 nm) .It was suitable for NLO applications. The powder second harmonic generation efficiency measurement shows the grown VCN crystal having 0.5 times higher nonlinear optical efficiency than potassium dihydrogen phosphate.

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