International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 04 Issue: 08 | Aug -2017 www.irjet.net p-ISSN: 2395-0072

INVESTIGATIONS ON THE GROWTH AND STRUCTURAL ANALYSIS OF L-ASPARAGINE L-TARTRATE (LAT) SINGLE CRYSTALS

JEYALAXMI M[1], Dr P SHARMILA[2], MAHALAKSHMI G[3], SUGANYA V[4]

[1,2,3,4] *Assistant Professor, Department of Physics, Prince Shri Venkateshwara Padmavathy Engineering college*

INTRODUCTION:

Single crystals either in bulk form or as thin films are required for the applications of non-linear optics. All the crystalline materials can be described by 32 point groups of which 21 are non-centro symmetric; these exhibits II order non-linear optical effects. In order to grow single crystals which can be used for non-linear optical devices, it is necessary that it should be crystallized non-Centro symmetrically so as to produce bulk crystals with higher efficiencies.

SYNTHESIS AND GROWTH OF LAT:

The compound chosen for the present study belong to non-centrosymmetry category. LAT is an organic NLO material. In LAT the proton of L-Tartaric acid is deprotonated to the L-Asparagine thereby L-Asparaginium tartrate crystalline compound is formed. The grown crystal belongs to the monoclinic crystal system with space group $P2_1$.

The compound LAT has been synthesized by dissolving L-Asparagine in water with one equivalent of L-Tartaric acid. The precipitate obtained was washed and purified by recrystallization. Since LAT has good solubility in water, attempt was made to grow it from aqueous solution. Necessary care was taken to clean the vessel with Millipore water. The solution was prepared using deionized water as solvent and the excess of solute were stirred with a magnetic stirrer about 5 hours to mix the sample thoroughly to attain homogeneity. The saturated solution was allowed to evaporate at room temperature (°C). Large number of crystals has been obtained due to spontaneous nucleation. From the nucleated small crystals, well-defined seed crystal was placed in the saturated solution. By continuous slow evaporation, crystals have been obtained within 15 days. The as-grown single crystal of LAT is shown in Figure 1. The crystals were optically clear with rod like morphology. Since water holds a high solubility and also produces a transparent and bulky crystal, it is chosen as the suitable solvent to carry out the crystal growth.

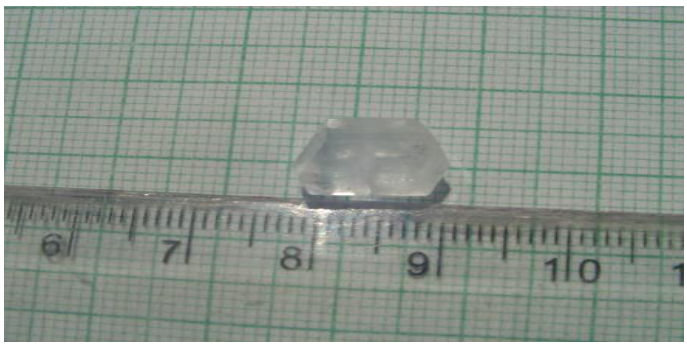

Figure. 1. As grown LAT single crystals

POWDER AND SINGLE CRYSTAL X-RAY DIFFRACTION STUDY:

The powder X-ray diffraction analysis is used to determine the particle size and structural parameters of the material. It is observed from the XRD pattern shown in Figure 2 that the LAT single crystals have prominent intensity peaks at 6.2, 8.8, 9.0, 13.5 and 43 degrees. However the entire pattern shows sharp and intense peaks ensuring crystalline nature of LAT single crystals. The lattice parameters calculated are in agreement with reported results. The important crystallographic data of LAT obtained from single crystal X-ray diffraction analysis is presented in Table 1.

Table 1 Crystallographic data of LAT

LAT	DATA
Space group	$P2_1$
Crystal system	Monoclinic
Lattice parameters	a = 5.085(3) Å b= 9.690(6) Å c = 11.863(6) Å
Cell volume	V = 584.53 (6) $Å^3$
Molecules per unit cell	Z = 4

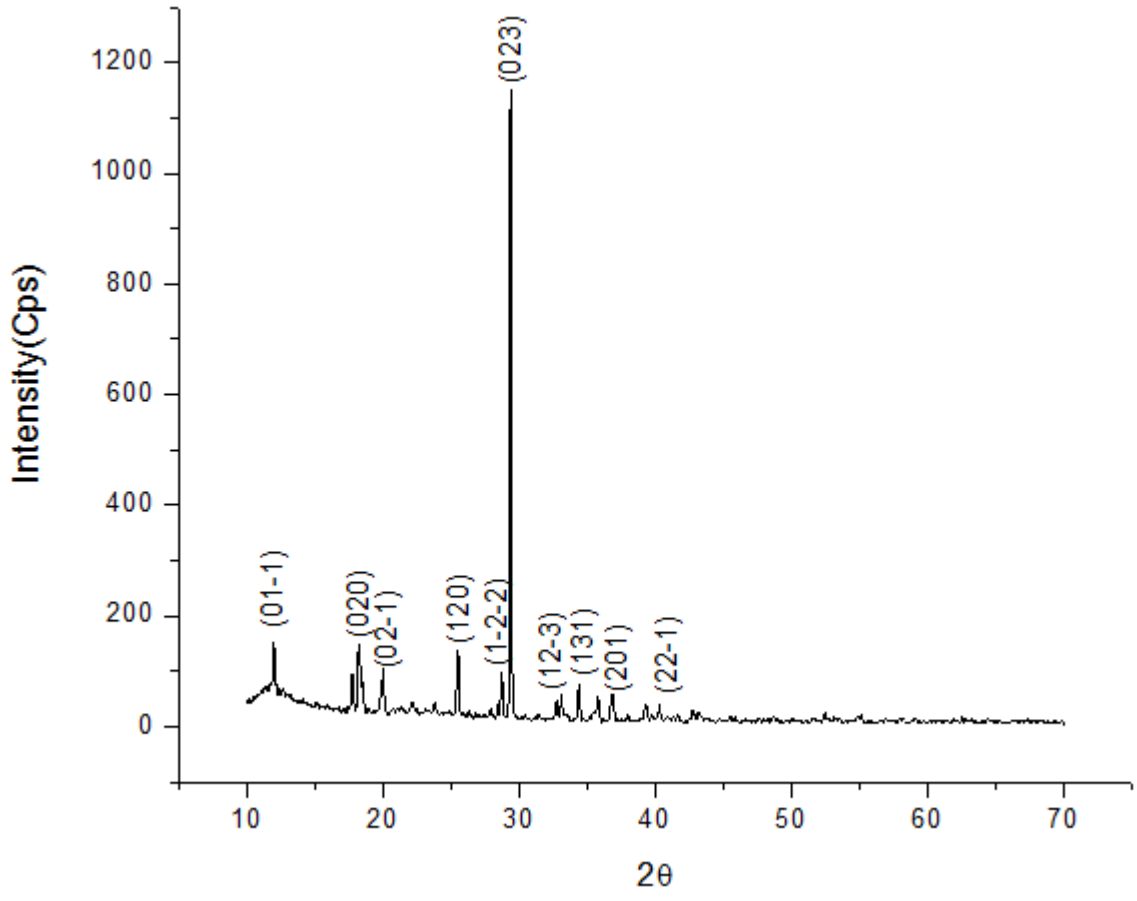

Figure 2. Powder X-ray diffraction pattern of LAT

SPECTRAL ANALYSIS:

The FTIR spectrum of LAT is shown in Figure .3. The intense broad band envelope between 2200 and 3800 cm^{-1} is due to OH stretching vibration, NH stretching vibration and CH_2 symmetry and asymmetry vibrations. The fine structure in the lower energy portion of the envelope indicates hydrogen bonding. The C=O vibration occurs at 1642 cm^{-1} and the NH bending vibration is assigned to 1523 cm^{-1}. The bending vibrations are at 1360 and 1425 cm^{-1}. The C-N vibration is

observed at 1232 cm^{-1}. The peaks at 1143 and1082 cm^{-1} are assigned to C-O vibration of L-Tartaric acid. The peak at 515 cm^{-1} is due to torsional oscillation of NH_2. As the spectrum carries the peaks due to NH vibration, C=O vibration and C-O vibration, the crystal must be made up of both L-Asparagine and L-Tartaric acid. Moreover the spectrum is not super imposable with the spectra of the two L-Tartaric acid and L-Asparagine.

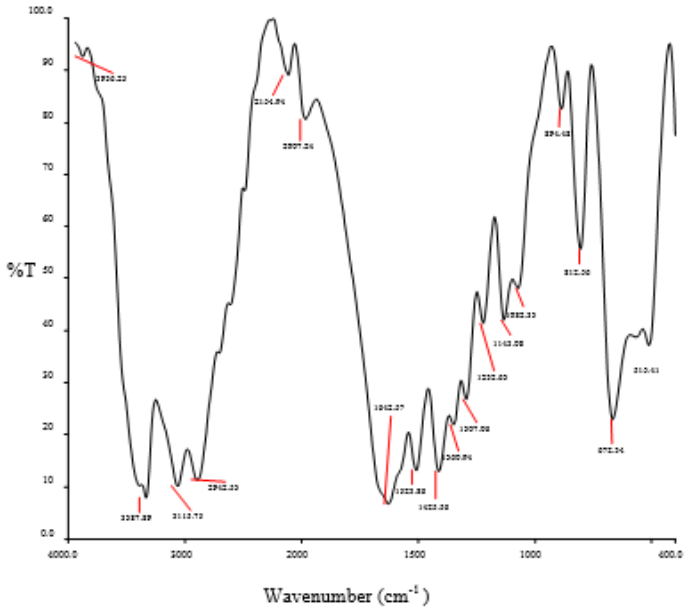

Figure 3. FTIR Spectrum of LAT

CONCLUSION:

Good optical quality LAT bulk single crystal has been grown by slow evaporation technique. Single crystal X-ray and powder diffraction studies confirm the unit cell parameters and LAT crystal belong to monoclinic crystal structure. The presence of various functional groups was confirmed by FT-IR spectrum.

REFERENCES:

[1] S. Natarajan, V. Hema, J. Kalyana Sundar, J. Suresh and P. L. Nilantha Lakshman, Acta Cryst. E, Volume 66, Part 9, September 2010, 2239.

[2] Colthup N.B., Daly L.H., and Wiberely S.E., (1964), 'Introduction to Infrared & Raman Spectroscopy', Academic Press, New York.