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Synthesis and Characterization of Magnesium Tartarate Single Crystals

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ABSTRACT

NLO organic crystals of magnesium tartarate single crystals were grown after 30 days by slow evaporation method with dimensions 28mm X 28mm X 2mm using water as a solvent. Powder X-ray diffraction predicts the lattice parameters with the use of a computer program. Fourier Transform Infra Red spectral analysis employment confirms the presence of various functional groups in magnesium tartarate. The optical transparency was determined by UV-Vis-NIR spectrum. The thermogravimetry and differential scanning calorimetry analyses gave a fixation about the thermal stability of the grown crystals. Second harmonic generation studies were found to be noted that the grown crystals NLO efficiency is higher than that of potassium dihydrogen phosphate.

1. Introduction

Optical device applications such as optical modulators, optical switches, electro optic devices(1-4) were improved for the past two decades due to the high potential NLO materials which have a great deal of attraction among Scientists. Lot of investigations proved that the organic NLO materials have high optical nonlinearities because of its highly conjugated electron system and polar nature. Polarized π-conjugation system organic compounds were found to have a potential to overcome the inorganic compounds optical behavior (5-6). On comparing with inorganic materials nonlinear susceptibilities of organic materials shows good impact with low damage threshold value. Hence for the satisfaction of day to day technological requirements it is a must to concentrate on new NLO materials. L-tartaric acid belongs to monoclinic system is a very good organic nonlinear material in optics (7). Literature shows the growth and optical studies of L-tartaric acid (8-10).From the technological point of view and from the solid state science the doping effect creates various property changes which attract great interest to proceed. Influence of doping in morphology, growth kinetics, SHG efficiency, thermal stability and dielectric properties gives us a fruitful result for device fabrications (11-14). The present work shows the growth and characterization of L-tartaric acid mixed with magnesium sulphate by slow evaporation solution growth technique with various characterizations.

2. Experimental Technique

After recrystallization, single crystal of magnesium tartarate was grown from aqueous solution by mixing analytical reagent grade sample L-tartaric acid with magnesium sulphate taking double distilled water as solvent by slow evaporation solution growth method. A sizable crystal was harvested after 25 days when an equimolar concentration of L-tartaric acid and magnesium sulphate mixed together with a magnetic stirrer under a constant temperature. Fig.1 shows the morphology of the grown crystal as 28mmX28mmX2mm.

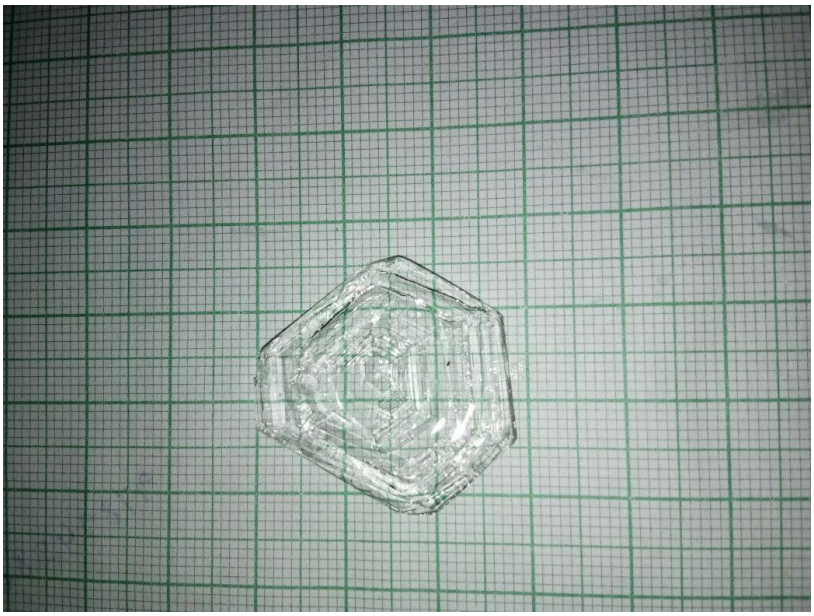

Fig.1 Morphology of Magnesium tartarate crystal

2.1. UV-Vis-NIR analysis

The recorded UV-Vis-NIR absorption spectra of the grown magnesium tartarate crystal show the suitability of the crystal in optical applications. This analysis is used to determine the transmission coverage area of the grown crystal which will fix the optical behavior quality. It was found to be noted that magnesium tartarate crystal has a cut off wavelength of 200.78 nm. Also the transparency of the magnesium tartarate crystal lies in between 200.78 to 800 nm. From the very wide transmission range an idea was framed that the grown crystal will have second harmonic behavior.

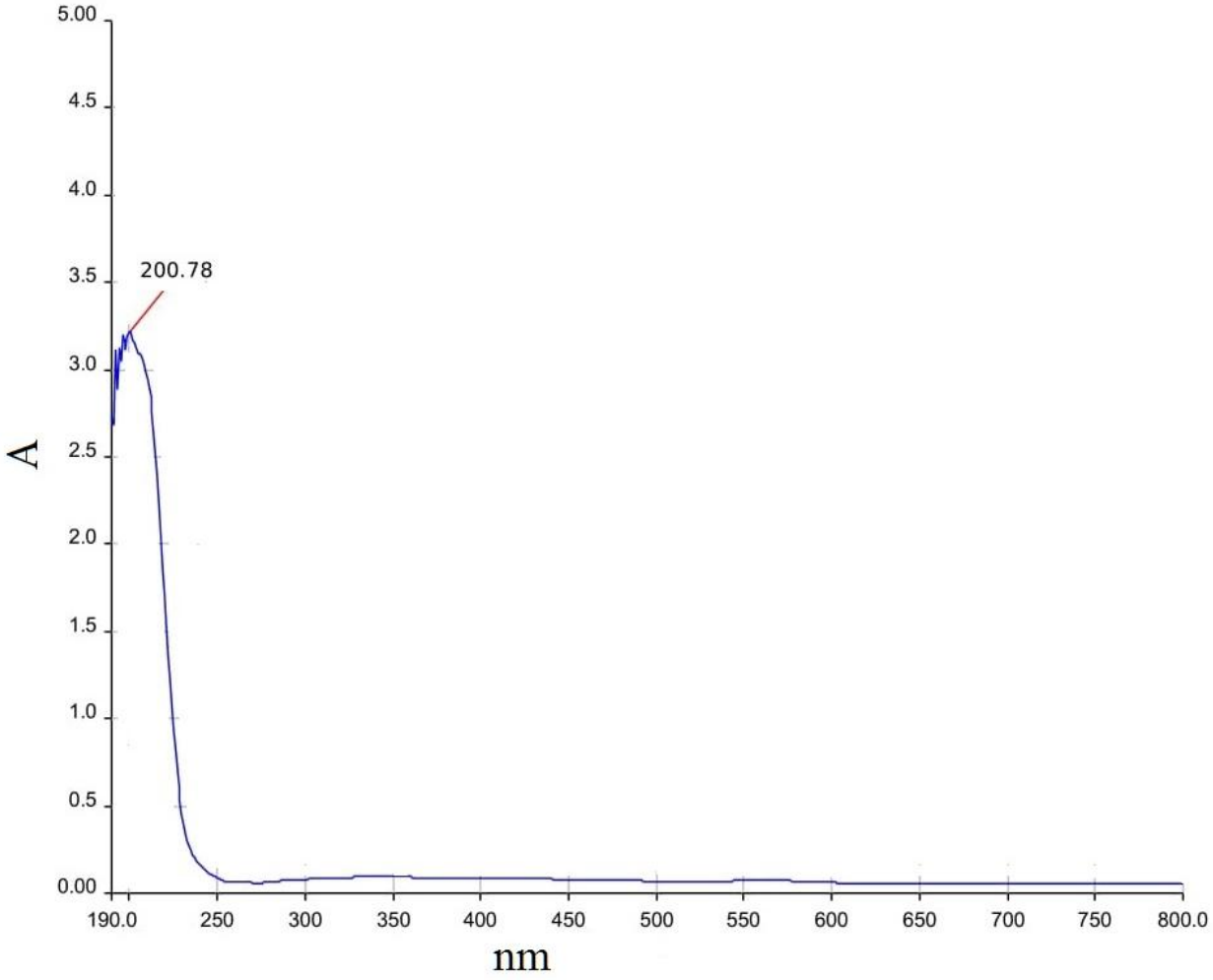

Fig.2 UV-Visible spectrum of Magnesium tartarate crystal

2.2. FT-IR Analysis

Fig.3 shows the FTIR spectrum of magnesium tartarate crystal. The obtained spectra was recorded using Perkin – Elmer FTIR spectrum RX1 spectrometer by KBr pellet technique maintained under a range 4000-400cm^{-1}. The functional groups present in the magnesium tartarate were identified as follows. The sharp and intense peak found at 3435cm^{-1} in the magnesium tartarate spectrum is due to the presence of hydroxyl group corresponding OH stretching. The >C=O stretching of magnesium tartarate is shifted to 1660 cm^{-1} on compared with tartaric acid (15). At 1100cm^{-1} the C-O stretching of magnesium tartarate occurs and the plane bending >C-H found at 618 cm^{-1}. Slight deviations were observed from the earlier report of L-tartaric acid with the magnesium tartarate.

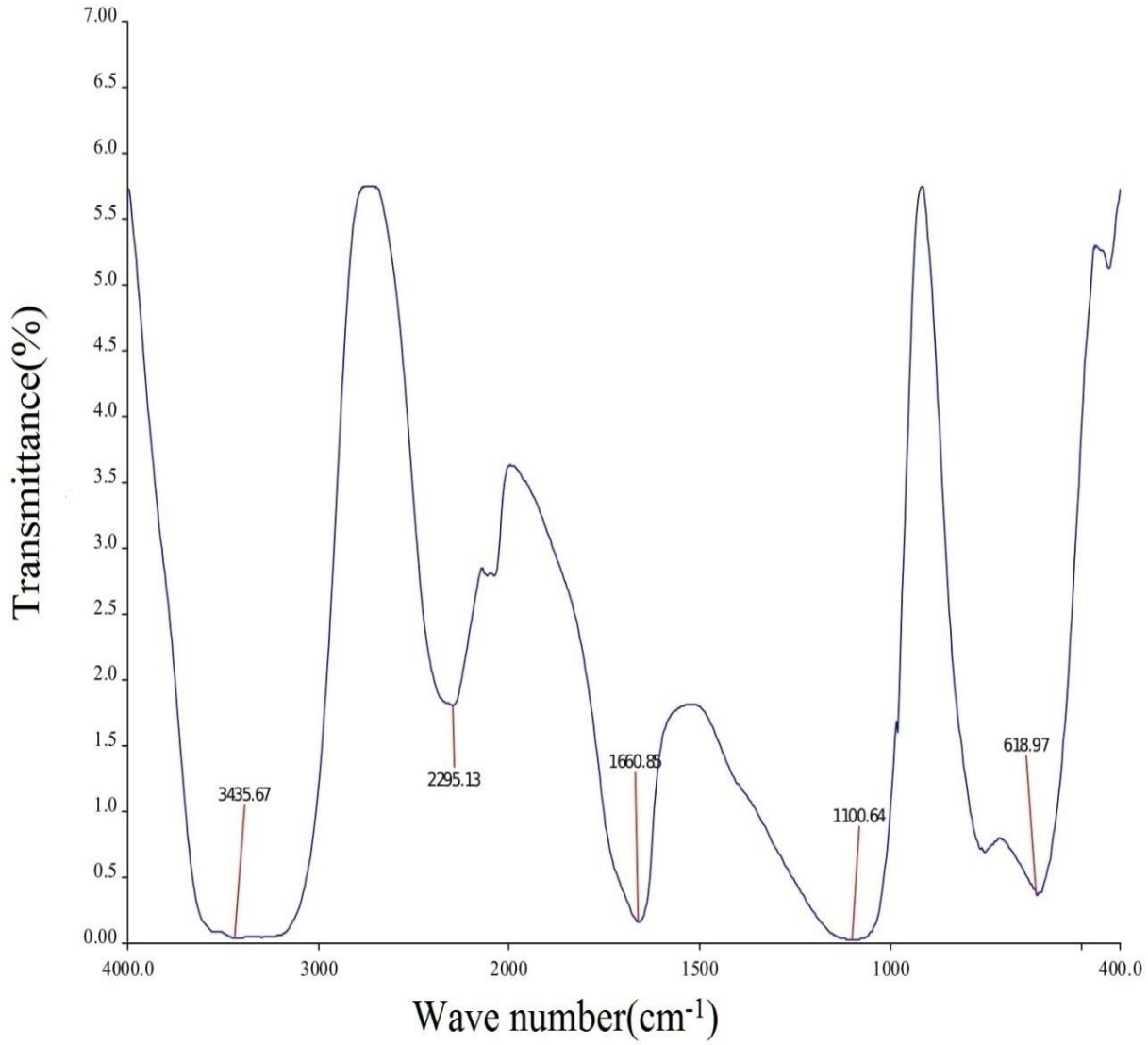

Fig. 3 FT-IR Spectrum of Magnesium tartarate crystal

Table 1: FT-IR

Wavelength	Stretching
3435.67	O-H stretching
2295.13	N=
1660.85	>C=O stretching
1100.64	C-O stretching
618.97	>C-H plane bending

2.3. Thermal Analysis

Fig. 4, 5 shows the TGA-DSC spectrum of magnesium tartarate. A single step weight loss was noted from the TGA curve on the heating the grown crystal between 0-1000°C. CO, H2 and H2O molecules were lost on heating the compound above 70° - 90° C on the account of 58.52% weight loss in the TGA curve. The decomposition pattern of the grown magnesium tartarate seems entirely different with the reported L-tartaric acid which gives confirmation about the formation of magnesium tartarate. The predicted endothermic peak at 134.07 °C resembles the decomposition of magnesium tartarate in the DSC curve.

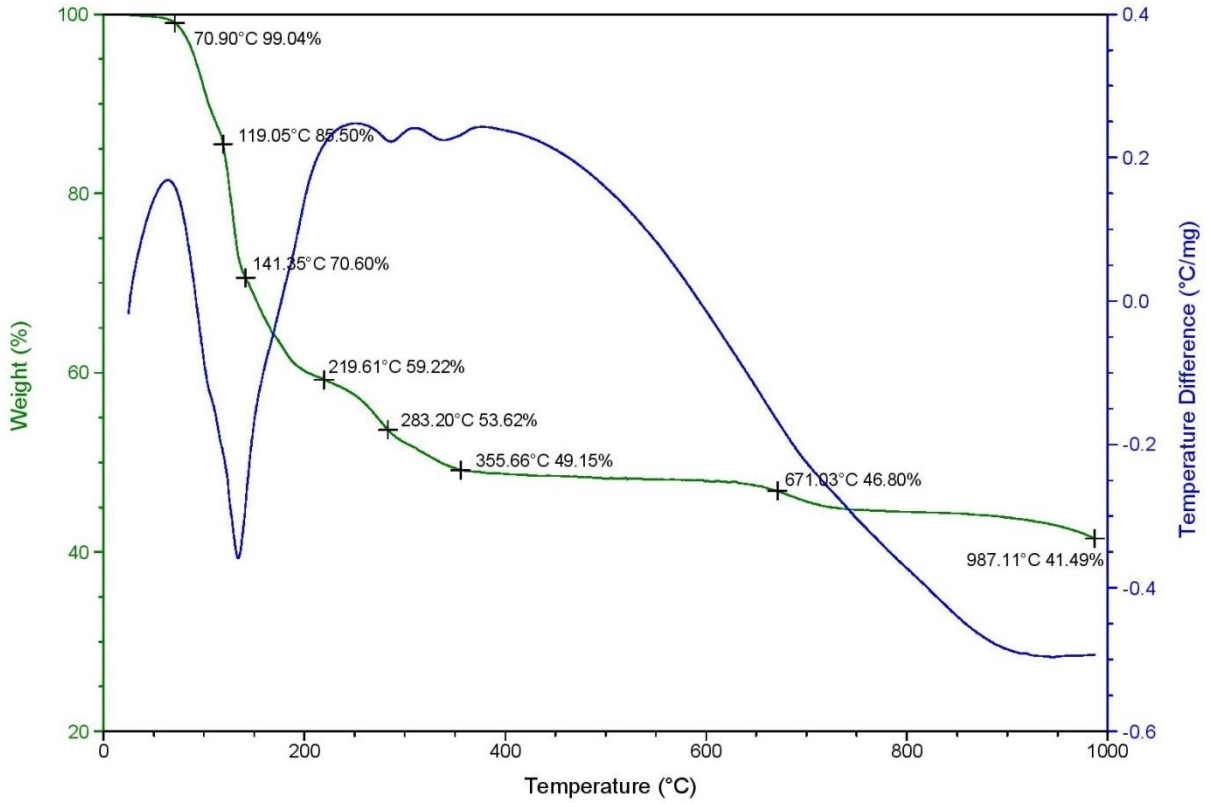

Fig.4 TGA curve of Magnesium tartarate crystal

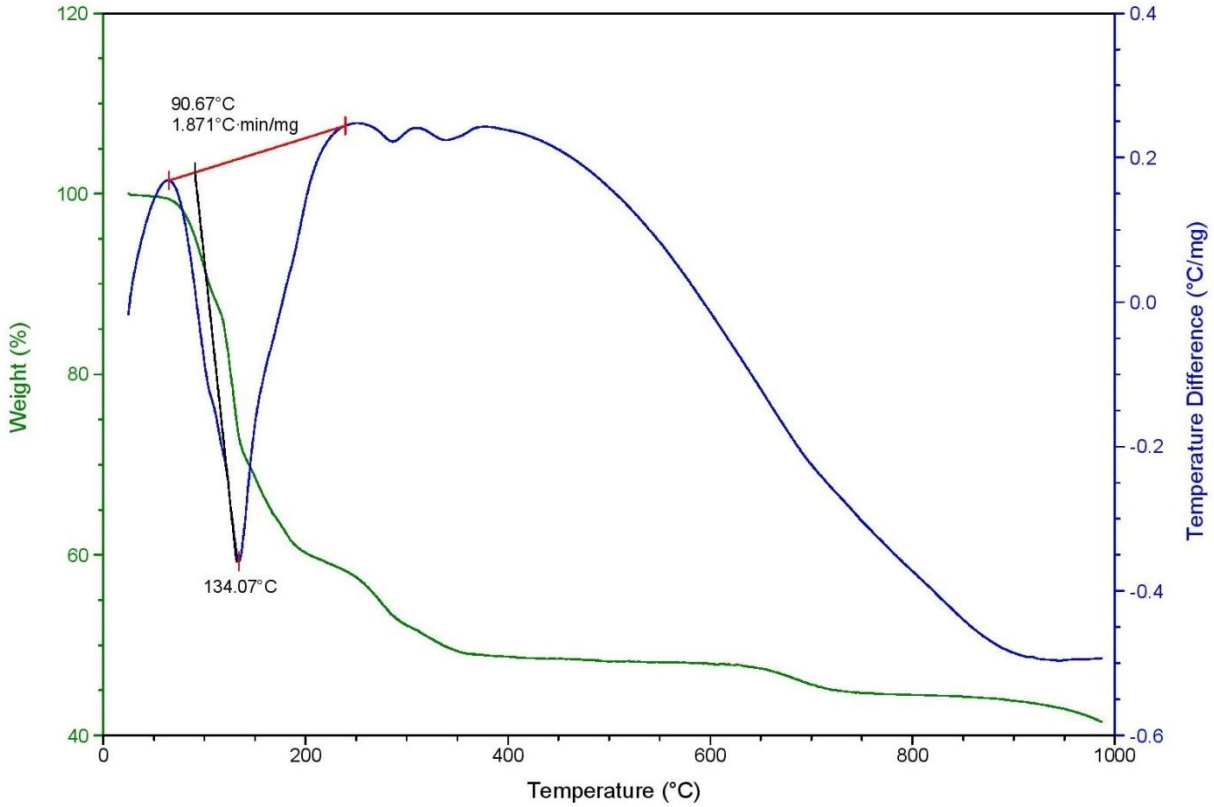

Fig. 5 DSC curve of Magnesium tartarate crystal

2.4. X-ray Diffraction Studies

Grown crystal of magnesium tartarate is taken in powdered form to determine XRD analysis by utilizing Bruker D8 advance X-ray powder diffractometer of wavelength λ=1.5406 with a characteristic CuKα radiation with a scanning rate of 1 per minute from 20° to 80°C. Fig.6 shows the powder

XRD pattern of the grown magnesium tartarate crystal. The good crystalline nature of the grown crystal was confirmed by the observation of strong and sharp peaks in the diffraction pattern. Reitveld index software package used to determine the lattice parameters and the hkl values using 2θ. Table.2 gives the calculated hkl values using 2θ and d-spacing.

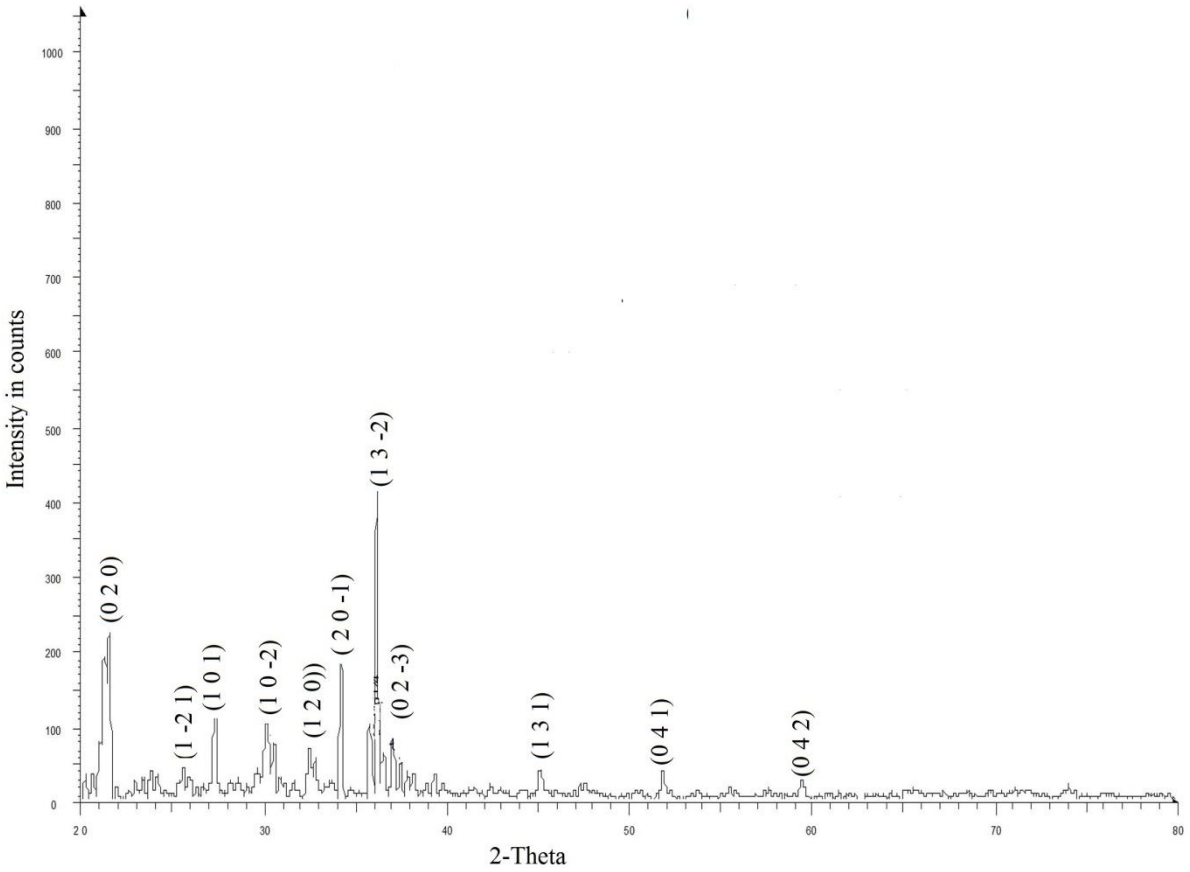

Fig. 6 XRD pattern of Magnesium tartarate crystal

Table 2: XRD values

2-Theta	d-spacing	hkl
21.395	4.14457	0 2 0
25.120	3.75347	1 -2 1
26.728	3.27416	1 0 1
30.171	2.96736	1 0 -2
32.674	2.93268	1 2 0
34.170	2.62494	2 0 -1
36.327	2.46124	1 3 -2
37.087	2.70151	0 2 -3
45.669	2.00962	1 3 1
51.959	-	0 4 1

2.5. Second Harmonic efficiency measurements

Capillary tube packed fully with the grinded powder of grown crystals were made to exposed in the path of Laser beam of wavelength λ=1064nm. The entire setup was developed by Kurtz and Perry to check the second harmonic generation efficiency of the grown crystals (16). A 10cm lens is used to focus the laser beam emitted from the Nd:YAG CD(R11) source. Perpendicularly scattered beam from the specimen is collected and the green light emission records a confirmation that the magnesium tartarate crystal has second harmonic generation efficiency. The output efficiency was found to be 72 percent of KDP.

3. Conclusion

Synthesizing of the title compound was successfully carried out and single crystals have been grown by solution growth slow evaporation technique. The crystal system of the grown crystal belongs to monoclinic was confirmed by XRD measurements along with the unit cell parameters. The FTIR analysis gives a definite exposure about the presence of functional groups. An endothermic peak found at 134.07° C confirms the melting point of the grown crystal which may lay a foundation to fabricate the NLO devices operating under the temperature134.07°C.

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