



Growth and characterization of L-histidinium 2-nitrobenzoate [LH2NB(I)]—A promising non linear optical single crystal



A. Zamara, K. Rajesh, A. Thirugnanam, P. Praveen Kumar*

PG and Research Department of Physics, Presidency College, Chennai 600005, India

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ABSTRACT

Single crystals of L-histidinium 2-nitrobenzoate [LH2NB(I)] have been successfully grown by slow evaporation method. The grown crystals have been subjected to powder X-ray diffraction studies and the cell parameters are calculated. The structural and optical properties of the grown crystals were characterized by FT-IR and UV–VIS–NIR studies. Photoconductivity and dielectric studies were also carried out for the grown crystals. Vickers microhardness test was also carried out to elucidate the mechanical behavior of the grown crystals. TG–DTG studies were carried out to determine the thermal stability of the crystals. The second harmonic generation behavior of the grown crystals was tested by Kurtz–Perry powder technique.

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1. Introduction

The search and design of highly efficient NLO crystals for visible and UV region are very important for laser processing and photonic devices. Organic NLO materials possess large NLO coefficient with lower cut-off wavelength. In the recent years, amino acid family crystals have been subjected to extensive investigation by several researchers for their NLO properties [1–3]. Crystalline salts of amino acids are the suitable candidates for searching new NLO materials in which much attracted were salts of L-arginine, L-histidine, and L-threonine.

L-Histidine compounds have gained importance because a number of non-linear L-histidine optical crystals have SHG efficiencies comparable to that of KDP. These histidine salts possess the high nonlinearity and fast optical response characteristics with better physico-chemical stability. A number of L-histidine compounds namely L-histidine acetate [4], L-histidine chloride monohydrate [5], L-histidine tetrafluoroborate [6] and L-histidinium trichloroacetate [7] exhibiting the NLO behavior were reported earlier.

The crystal growth and characterization of L-histidinium trifluoroacetate [8] and L-histidine nitrate [9] crystals were also reported.

In the present study, the title compound L-histidinium 2-nitrobenzoate [LH2NB(I)] crystals were successfully grown by slow evaporation technique. Characterization studies such as powder XRD, FT-IR, UV–VIS–NIR, TG–DTA, microhardness, dielectric studies

and Photoconductivity study have been carried out for the grown crystal. The NLO property of the grown crystal has also been studied by Kurtz–Perry powder technique.

2. Materials and methods

L-histidinium 2-nitrobenzoate [LH2NB(I)] crystals were grown from an aqueous solution by slow evaporation technique. L-Histidine (99% pure) and 2 nitrobenzoic acid were taken in the stoichiometric ratio 1:1 and dissolved in double distilled water. The reaction is as follows,



The resultant mixture was stirred continuously to obtain a homogenous solution, filtered and kept undisturbed for crystallization. Good quality single crystals of the title compound were obtained after about 2 weeks time. The photograph of the as grown crystal is shown in Fig. 1.

3. Characterization

3.1. Powder X-ray diffraction

Powder X-ray diffraction studies of the grown crystal were carried out using Siemens D500 X-ray diffractometer with $\text{CuK}\alpha$ ($\lambda = 1.5418 \text{ \AA}$) radiation. The sample was scanned for 2θ values from 10° to 70° at the rate of $2^\circ/\text{min}$ (Fig. 2). Hence the lattice parameter values for the grown crystal were calculated. It is observed that the grown crystals possess monoclinic structure with a space

* Corresponding author. Tel.: +91 9444446272.

E-mail address: papkpresidency@gmail.com (P. Praveen Kumar).



Fig. 1. Photograph of the grown LH2NB(1) crystal.

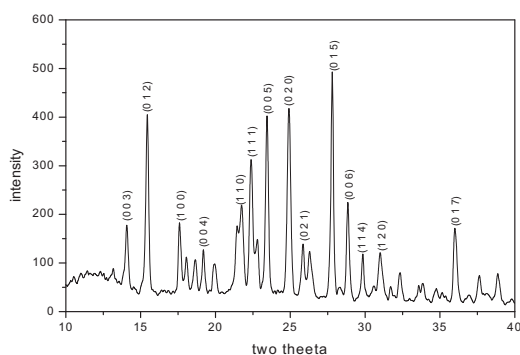


Fig. 2. Powder X-ray diffraction pattern of LH2NB(1).

group $P2_1$. The calculated lattice parameter values of the grown crystal, $a = 5.147 \text{ \AA}$, $b = 7.228 \text{ \AA}$, and $c = 18.887 \text{ \AA}$ are in good agreement with the reported values. This confirms that the grown crystal is LH2NB(1). The calculated values and the reported values [10] are presented in Table 1. The powder XRD pattern shows the good crystallinity of the grown crystal.

3.2. FT-IR spectroscopic analysis

The Fourier transform infrared spectrum [11] was recorded for the sample in the range 400 cm^{-1} to 4000 cm^{-1} using the instrument FT-IR 4100 type A spectrometer. The FT-IR spectrum is shown in Fig. 3. The assignments for the absorption peaks in the spectrum are given using the available infrared spectral data and the corresponding vibrational group assignments are given in Table 2.

Table 1

Lattice parameter values of LH2NB(1).

| Lattice parameters | Reported values | LH2NB(1) |
|--------------------|-----------------------|-----------------------|
| $a(\text{\AA})$ | 5.089 | 5.147 |
| $b(\text{\AA})$ | 7.064 | 7.228 |
| $c(\text{\AA})$ | 18.689 | 18.887 |
| β | 93.55° | 92.72° |
| Crystal system | Monoclinic | Monoclinic |
| Space group | $P2_1$ | $P2_1$ |
| Volume | 670.6 \AA^3 | 701.8 \AA^3 |

Table 2

Spectroscopic assignments of LH2NB(1).

| Wave number (cm^{-1}) | Spectroscopic assignments |
|----------------------------------|-------------------------------------------|
| 3173, 3137, 2930, 2562 | O–H stretch, carboxylic acids |
| 2098, 1950 | $\text{C}\equiv\text{C}$ stretch, alkynes |
| 1578 | N–H bend, primary amines |
| 1443 | C–C stretch, aromatics |
| 1348 | N–O symmetric stretch, nitro compounds |
| 1198 | C–O stretch, alcohols, carboxylic acids |
| 905, 838, 696 | N–H wagging, primary, sec amines |

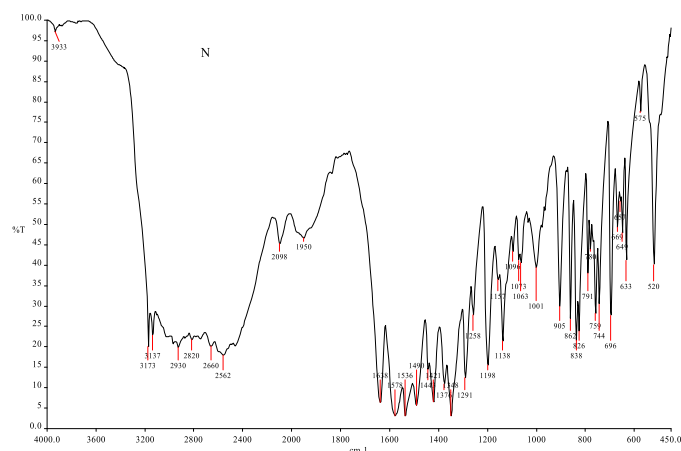


Fig. 3. FT-IR spectrum of LH2NB(1).

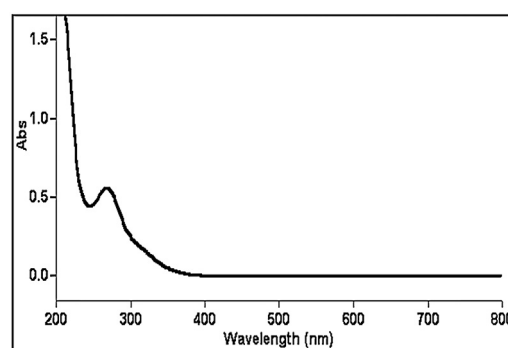


Fig. 4. UV-VIS-NIR spectrum of LH2NB(1).

The absorption peaks from 2562 cm^{-1} to 3173 cm^{-1} include overlap of peaks due to O–H stretch of carboxylic acids. The peaks at 1950 cm^{-1} and 2098 cm^{-1} represent $\text{C}\equiv\text{C}$ stretch of alkynes. The peak at 1715 cm^{-1} indicates the presence of carboxylic group of primary amines. The peaks at 1578 cm^{-1} and 1443 cm^{-1} represent N–H bend due to primary amines and C–C stretch due to aromatic compounds. The N–O symmetric stretch of nitro compounds is observed at 1348 cm^{-1} . The C–O of alcohols lies at 1198 cm^{-1} . The peaks from 696 cm^{-1} to 905 cm^{-1} represent N–H wagging of primary or secondary amines.

3.3. UV-VIS-NIR studies

In order to find the absorption range for the suitability in optical applications, the UV-VIS-NIR spectrum was recorded in the range 200–800 nm, using a Varian Carry 5E dual beam spectrophotometer. The spectrum is as shown in Fig. 4. It is observed that the crystal shows good absorption in the entire visible and IR regions. The lower cut-off wavelength is at 232 nm. The absence of absorbance of light in the visible region is an intrinsic property of all aminoacids [12,13]. Low absorbance in the region between 232 nm and 800 nm shows that this crystal could be used for optoelectronic applications and second harmonic generation (frequency conversion) or generally optical window applications [14].

3.4. NLO studies

The second harmonic generation efficiency of the powdered sample was measured using Kurtz–Perry method [15] with a Nd-YAG Q switched laser beam. The sample was illuminated with a laser input pulse of 6.2 mJ, and the pulse width of 8 ns, and a repeti-

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