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RESEARCH ARTICLE

Crystal growth, optical, thermal and analysis of β-p-aminobenzoic acid single crystal

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Abstract

β-paminobenzoic acid single crystal was grown by slow evaporation technique at 28 °C. Crystal structure of title compound was confirmed by single crystal X-ray diffraction analysis and it shows that the grown crystal has β-p-aminobenzoic acid [β-PABA] crystal structure and the lattice parameters are a=6.30 Å, b= 8.61 Å, c= 12.43 Å, α=90°, β=100.20° and γ =90°. The optical absorbance spectrum shows that the grown crystal has a characteristic absorbance in ultraviolet region. TG-DTA studies exhibits that there is no weight loss upto 187 °C. Laser damage threshold indicates that the grown crystal has good laser damage stability.

Keywords

Organic compounds

X-ray diffraction

Thermal stability

Ultraviolet

Introduction

Crystals are important in the areas of advanced applied science, detectors, optical, lasers and sensors [1]. Recently, researchers are showing their keen interest in synthesis and growth of novel organic substances due to their efficient properties [1-2]. P-aminobenzoic acid (PABA) is a biological molecule and it is a member of vitamin Bx and it acts as a bacterial cofactor involved in the synthesis of folic acid [3]. It is used in pharmaceuticals, dyes, sunscreen agents and feedstock additives [4]. P-aminobenzoic acid is the simplest aromatic carboxylic acid and the derivatives of PABA are one of the interesting materials due its polymorphic structure. PABA exhibits two kinds of polymorphs: (i) α-PABA (needle shape) (ii) β-PABA form (prism shape). Formations of these polymorphic shapes are depends on the solvent used [3, 5]. Gracin et al reported that many organic solvents forms only needles but water forms needle and prism shapes [6]. Initially, the crystal structure of β-PABA is determined by Alleaume et al. But, the precision structure of the β-polymorph of PABA redetermined by Gracin *et al.* [4]. β-PABA molecules that contains amino (NH_2) donor group and the carboxyl acid (COO^-) acceptor. It has hydrogen bonded three-dimensional network [2, 4]. The hydrogen bonding is important in molecular recognition and crystal

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engineering research. Search for organic single crystals have been substantially increased because of their efficient physical and chemical properties such as high thermal, low dielectric constant, high optical damage threshold, ultrafast response with better processability, ease of fabrication, possible integration into devices and so on [2]. Based on the above aspects, the present paper describes and discusses crystal growth, single crystal XRD, optical, thermal and laser damage threshold properties of organic β-p-aminobenzoic acid crystal.

Experimental

The commercially available p-aminobenzoic acid was purified by repeated recrystallization using deionised water as solvent which has resistivity of 18.2 MΩ cm. The solution was stirred well for 12 h until homogeneous mixture of clear solution is obtained and the solution was filtered using filter paper and transferred to a glass vessel and evaporation was allowed to take place at room temperature. After two weeks 1 mm^3 seed crystals were obtained and defect free single crystals are selected as seed for growing large size single crystals. The seed crystals were located at the bottom of the beaker containing the supersaturated solution. The beaker was kept in a constant temperature bath at 28 °C. Prismatic single crystals have been harvested after 45 days. The grown crystals of β-PABA is shown in **Fig 1**.

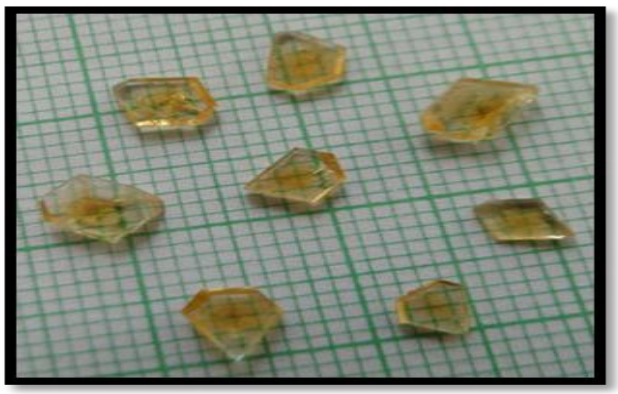

Fig 1 β-PABA single crystals.

The single crystal XRD diffraction studies of β-PABA crystal was carried out using Bruker AXS Kappa APEX II CCD diffractometer equipped with graphite-monochromated MoKα radiation (λ=0.71073 Å). The absorption spectrum of the grown crystal was made in the region 200–1100 nm using Perkin Elmer Lambda 35 spectrometer. The surface Laser damage threshold measurements has been carried out using the high-power Q-switched Nd:YAG laser with 10 Hz pulse repetition rate and the pulse width of the laser is 7 ns for 532 nm. The thermal stability of title compound was studied by TG-DTA using Perkin Elmer instrument the temperature range between 34 - 300 °C at a heating rate of 10 °C/min.

Results and discussion

From the single crystal XRD the title compound belongs to monoclinic system with space group P21/n. Lattice parameters are a=6.30 Å, b= 8.61 Å, c= 12.43 Å α=90°, β=100.20° and γ =90°. The present result is in good agreement with the reported values [4, 5]. The absorption spectrum is shown in **Fig 2**. It is seen that there is a characteristic absorbance in the UV region may be due to the electronic transitions occurring in the carboxylate (COO^-) and amino (NH_2^+) bands. After that there is no considerable absorbance in the visible and NIR region [6]. The title compound can be used for various photonic and optical applications due to its wide transparency window in the visible and NIR regions [2, 6]. Thermal analysis provides useful information about phase transition, thermal stability and different stages of decomposition of the crystal. The TG-DTA curve of the grown crystal is shown in **Fig 3**.

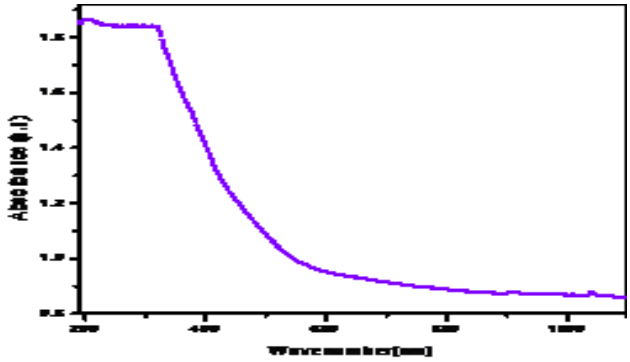

Fig 2 The UV-Vis-NIR absorption spectrum of β-PABA single crystal.

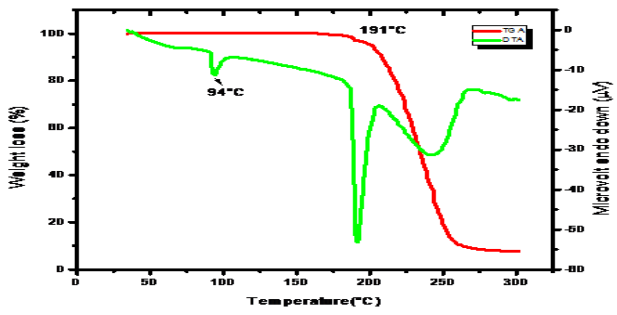

Fig 3 TG-DTA of β-PABA single crystal.

The TGA curve indicates that there is no weight loss up to 187 °C. After that, weight loss in a single step between 191 - 256 °C and gradually decreases to zero

In differential thermal analysis curve shows two remarkable thermal results. The material undergoes an endothermic transition at 94 °C, which may be due to the solid phase transition of β form. The second endothermic peak at 191 °C (α form) [5] is attributed to the melting point of PABA. Laser damage threshold noted in the crystals when exposed to high intensities of laser light. LDT of materials depends upon several factors such as dielectric breakdown, thermal absorptions, electron avalanche and so on. The most important considerations for crystal to perform as a device, is that it withstands high laser power [6, 7]. Single shot laser beams was passed along the surface of the prepared β-PABA sample. Initially 5 mJ was applied on the top surface of the sample and further it is increased to 10, 15, 20 and 30 mJ successively in steps of 5 mJ. There was no damage observed on surface of the sample for 20 s. For 35 mJ, the small spot appeared after 20 s and finally surface damage was seen when applying 45 mJ for 15 s. The experiment was executed for the different pieces of the same crystal and the similar result was recorded. The power density was calculated using the formula, $P_d = \frac{E}{\tau A}$

where E is the energy, τ is the pulse width (7 ns) and A is the area of the circular spot (4.153×10^{-4} cm^2). The damage threshold of title compound has been found to be 10.03 Gw cm^{-2}. From the thermal stability and laser damage threshold studies, it was observed that β-PABA crystal can be used for laser related experiments [6-8].

Conclusions

Prismatic single crystals of β-p-aminobenzoic acid were grown from solution growth technique. Single crystal XRD analysis confirmed that the grown crystal belongs to monoclinic system with space group P21/n. The absorbance spectrum shows that the grown crystal has no absorbance in the entire visible and NIR regions. This material can use in photonic applications. DTA shows that first endothermic transition at 94 °C may be due to the solid phase transition of β form. The surface laser damage threshold value for the β-PABA is found to be 10.03 Gw cm^{-2}. The grown crystal can be used for laser related applications.

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