

Crystal growth and characterization of *N*-hydroxyphthalimide ($C_8H_5NO_3$) crystal

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Abstract

Single crystals of *N*-hydroxyphthalimide (NHPH) were obtained from saturated aqueous solutions of methanol and acetonitrile by slow cooling method. The grown crystals were bright and transparent. From the crystal structure analysis it can be inferred that the crystal belongs to monoclinic system. The grown crystals were subjected to FTIR analysis for vibrational assignments. The optical transmission spectra showed excellent transmittance from 200 to 1100 nm. The thermal stability and thermal decomposition of NHPH crystal have been investigated by means of thermogravimetric analysis and differential thermal analysis.

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1. Introduction

One of the intensely studied fields in Chemistry and Physics are the optical properties of molecular crystals. The importance of those materials has been demonstrated in the recent years by the increased number of applications in nonlinear optics and in electronics. *N*-hydroxyphthalimide (*N*-hydroxy-1H-isolindole-1,3(2H)-dione) is an important molecular crystal and a versatile catalyst for oxidation of organic compound [1,2]. There are reports, which describes that it can act as herbicide [3–5] and germicide [6]. In addition to this there are some reports available on its application as a blocking agent for isocyanates [7]. The hydroxyl group of the title compound is bonded with the nitrogen of imide group, which is the main contributor of acidic character. Because of this property NHPH finds extensive applications. The growth of *N*-hydroxyphthalimide crystal is hitherto unreported in literature. Herein, we first report the crystal growth aspects of the title compound in two different solvents. The optical quality of crystal was determined by measuring the bulk optical transparency of the grown crystals to evaluate their characteristics absorption bands. Crystals were characterized for their stability by measuring the thermal behavior. Differential thermal analysis (DTA) was carried out for the reported crys-

tal to see the melting characteristics. The main objective of this study was to develop crystals, which are capable of shifting the wavelength of laser light over the range of 2–12 μm . This study was designed to achieve this objective and experiments were carried out for purification, single crystal growth, crystal characterization and optical quality evaluation of the title crystal. In this direction, the present work describes the growth and characterization studies of the grown NHPH crystal.

2. Experimental

2.1. Solubility and crystal growth

The compound NHPH was obtained as such from Lancaster Chemical Company, UK. The compound is further recrystallized several times in organic solvents for purification. The purified compound was used for further growth process.

Selection of suitable solvent is very definitive for the growth of good quality single crystals [8]. The equilibrium solubility is essential for aqueous solution growth [9]. As a first step in the direction of crystal growth, solubility was assessed in different organic solvents such as ethanol, methanol, acetonitrile and DMSO. The title compound was found to be soluble in two different solvents such as acetonitrile and methanol. The magnitude of the solubility of the title compound NHPH was determined

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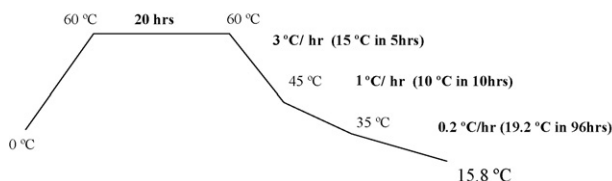


Fig. 1. Cooling curve of NHPH.

at room temperature (26 °C). The solubility was found to be 0.09 and 0.62 g in 100 ml acetonitrile and methanol, respectively.

The solution growth process was used with the technique of temperature lowering for growing crystal in solution and this technique can be applied to wide range of compounds for producing high quality crystals [10,11]. Hence the crystal growth experiment performed was slow cooling. A calculated amount of the title compound is dissolved thoroughly in the 100 ml of acetonitrile and methanol. The solution is stirred well using a magnetic stirrer to yield a saturated homogenous solution. The solution was then filtered twice to remove the suspended impurity and allowed to crystallize by slow cooling. A Teflon vessel with a volume of 250 ml was used with a use of a water bath containing approximately 100 ml of water for the purpose of controlling the temperature in the vessel and with the help of a programmable controller. The temperature adjustment accuracy in the water bath is kept at 0.2 °C/h. The cooling curve was presented in Fig. 1. The solution was obtained by adding 3 g of NHPH to 100 ml methanol as well as 100 ml of acetonitrile and 200 ml of the saturated solution of NHPH was used for growth. Initially the solution temperature was increased to 60 °C to remove any undissolved nuclei then slow cooling was applied so that spontaneously nucleation of NHPH crystal took place was at a temperature ranging from 45 to 35 °C. The cooling rate applied was 1 °C/h. Again cooling was carried out down to 15.8 °C at a rate of 0.2 °C/h. After growth period of 3 weeks, transparent, needle shaped single crystal of size 6.54 mm × 0.5 mm × 0.399 mm were obtained. The dimension measurements were carried out in digital micrometer screw gauge. The grown crystal was viewed under microscope for defect location and the photograph of the defect free crystal grown in methanol and acetonitrile are depicted in Fig. 2(a) and (b), respectively. The habit and the transparency are quite good for the crystals grown in two different solvents.

2.2. Spectral measurements

2.2.1. Powder X-ray diffraction studies

The structural properties of single crystals of NHPH have been studied by X-ray powder diffraction technique. The X-ray diffraction studies were carried out using SEIFERT diffractometer with Cu K α_1 ($\lambda = 1.5406 \text{ \AA}$) radiation. The powdered samples were scanned over the range 10–70° at a rate of 1° per minute. From the powdered X-ray data, the various planes of reflections were indexed using XRDA 3.1 program and the lattice parameters were evaluated. The indexed X-ray diffraction pattern is shown in Fig. 3. The observed 'd' values for different

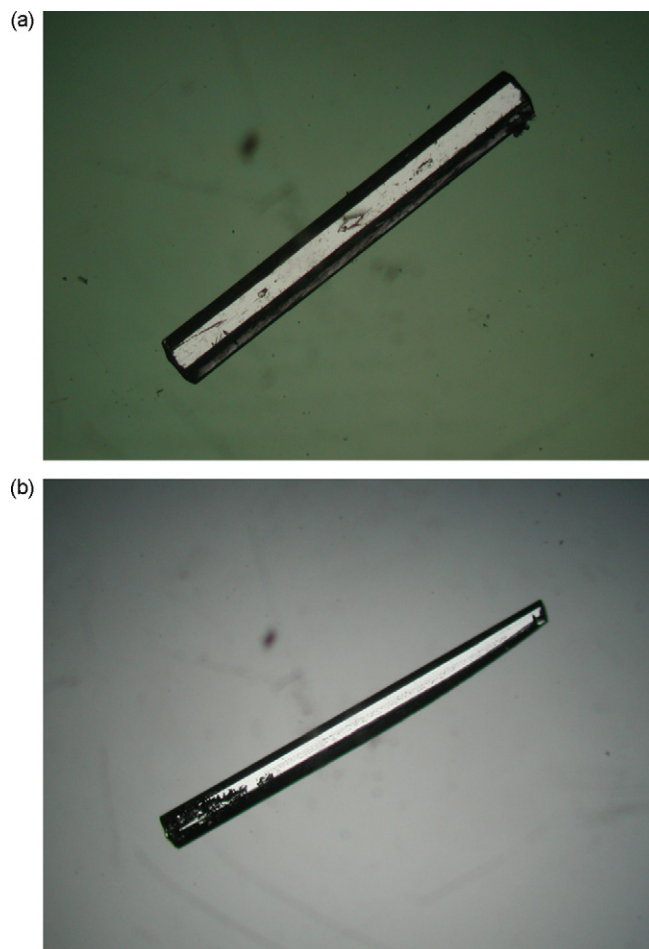


Fig. 2. (a) Photograph of the crystal grown in methanol. (b) Photograph of the crystal grown in acetonitrile.

2θ with hkl indices of the corresponding planes for the crystal are given in Table 1. From the studies it is found that the structure of the grown crystal is monoclinic and the crystallographic data is given in Table 2. The six and five membered rings in the solid space structure of the NHPH are highly symmetrical allowing the structure to crystallize in a centrosymmetric space groups.

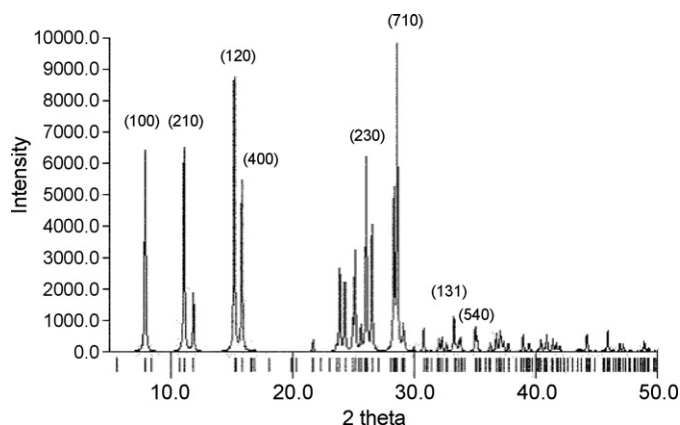


Fig. 3. The indexed X-ray diffraction pattern of NHPH.

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