Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 125 (2014) 335–344

Contents lists available at ScienceDirect

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy

journal homepage: www.elsevier.com/locate/saa

Growth and characterization of an organic single crystal: 2-[2-(4-Diethylamino-phenyl)-vinyl]-1-methyl-pyridinium iodide

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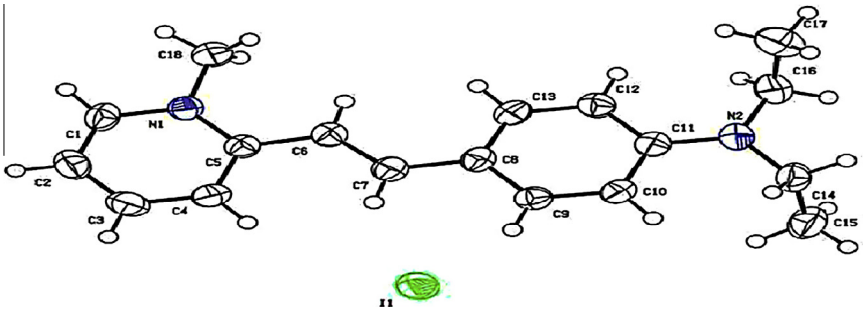
HIGHLIGHTS

- Highly good transparent single crystals with size up to $10 \times 7 \times 3$ mm^3 have been harvested by slow evaporation technique.
- ^{1}H and ^{13}C NMR confirms the molecular structure of the DEASI crystal.
- Crystal has good transparency in visible–NIR region.
- From the TGA curve of this sample shows that material is highly stable up to 262 °C.
- Refractive index, mechanical, dielectric, etching studies were carried out.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 20 November 2013
Received in revised form 13 January 2014
Accepted 22 January 2014
Available online 5 February 2014

Keywords:
Chemical synthesis
Optical materials
FT-IR
Crystal growth
Mechanical studies

ABSTRACT

Optically transparent crystal of the organic salt DEASI (2-[2-(4-Diethylamino-phenyl)-vinyl]-1-methyl-pyridinium iodide) has been synthesized by using knoevenagel condensation reaction method. The synthesized material has been purified by successfully recrystallization process. Single crystals of DEASI have been grown by slow evaporation technique at room temperature. The solubility of the title material has been determined at different temperature in acetonitrile/methanol mixture. The cell parameters and crystallinity of the title crystal were determined by single crystal XRD. The powder diffraction was carried out to study the reflection plane of the grown crystal and diffraction peaks were indexed. The presence of different functional groups in the crystal was confirmed by Fourier transform infrared (FTIR) analysis. ^{1}H NMR spectrum was recorded to confirm the presence of hydrogen nuclei in the synthesized material. The optical property of the title crystal was studied by UV–Vis–NIR spectroscopic analysis. The melting point and thermal property of DEASI were studied using TGA/DSC technique. The Vicker's hardness (H_v) was carried out to know the category. The dielectric constant and dielectric loss of the compound decreases with an increase in frequencies. Chemical etching studies showed that the DEASI grows in the two dimensional growth mechanisms. The Kurtz–Perry powder second harmonic generation (SHG) test has done for title crystal.

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Introduction

The wide range of organic nonlinear optical crystals has been investigated due to of their potential applications in signal processing, optoelectronics, laser technology, bioimaging, optical data

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http://dx.doi.org/10.1016/j.saa.2014.01.109
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storage for the development of technologies in telecommunication and data processing [1–4]. Organic materials exhibit large nonlinear response when compared to inorganic materials because of the presence of π-conjugated system linked by appropriate electron releasing groups (ERGs) and electron withdrawing groups (EWGs) at their two ends, which can enhance the asymmetric electronic charge distributions in the system. The asymmetric charge distributions between such groups leading to capable of generating second harmonic frequency, and which is playing the key step in the field of photonics [5–7]. In recent years, the efforts have been investigated towards the synthesis of novel π conjugated organic molecules because of their second and third-order nonlinear optical activities, large optical damage threshold, low frequency dispersion and fast response time [8–10]. Organic stilbazolium crystals have a relatively high probability because of their highly aligned and stable orientation of NLO chromospheres in the crystal system [11]. For the past few years, the ionic organic material is a particular interest, because of their thermal stability and photochemical stability, and its mechanical properties in the field of photonics [12,13]. The development of organic highly polar ionic stilbazolium crystals is based on the presence of strong coulomb interactions [13]. Recently stilbazolium family crystals are of considerable attention due to their attractive large nonlinear optical properties, a large electrooptic coefficient and low dielectric constant. One of the main advantages of organic materials is that the structure can be altered by suitable substituted electron donor and electron acceptor groups in their π-conjugated system compared to inorganic materials [8,14]. There are different methods for crystal growth. It is known that the slow evaporation solution growth technique is commonly used for the growth of organic and inorganic crystals.

In the present investigation, we report the synthesis, growth of a stilbazolium derivative, 2-[2-(4-Diethylamino-phenyl)-vinyl]-1-methyl-pyridinium iodide single crystals. These crystals were successfully grown by the slow evaporation technique. The grown title crystal has been subjected to different characterizations such as single crystal X-ray diffraction studies, powder XRD, FTIR, ^{1}H NMR, ^{13}C NMR, UV–Vis NIR, refractive index, thermal, dielectric studies, chemical etching and Vicker's microhardness measurements.

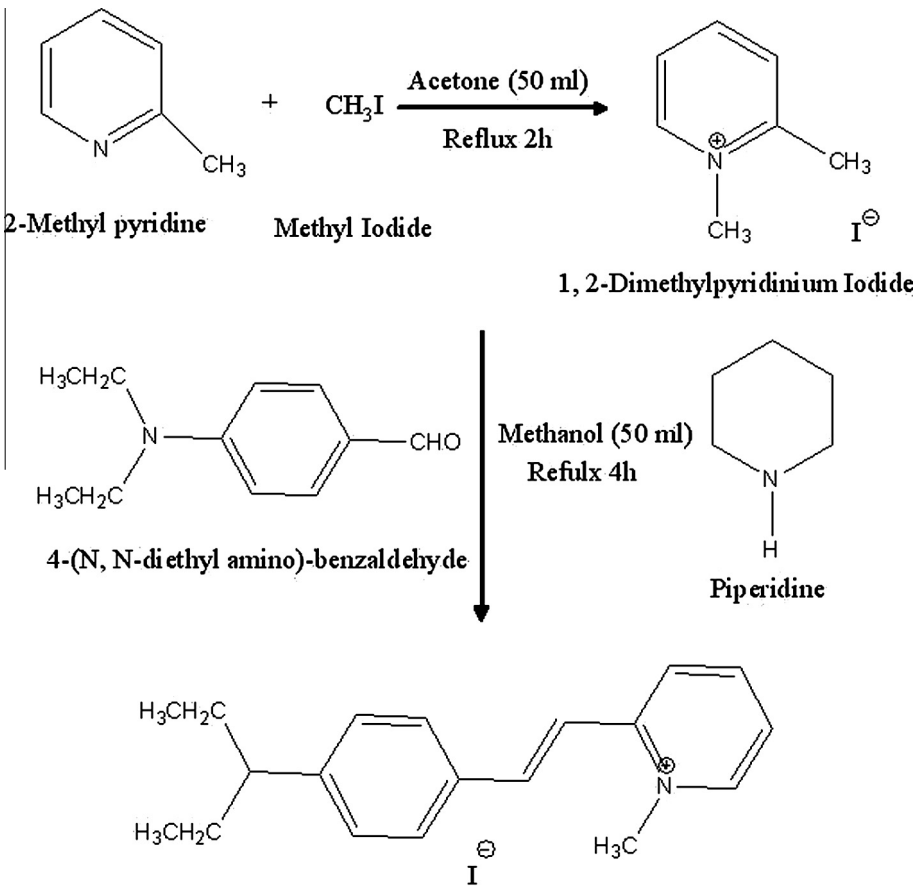
Experimental procedure

Material synthesis

The title material was synthesized by Knoevenagel condensation of 1,2-Dimethylpyridinium iodide, which was prepared from 2-methylpyridine and methyl iodide and 4-(N, N-diethyl amino)-benzaldehyde in the presence of piperidine. The overall synthesis process and molecular structures are given in Scheme 1.

*1,2-Dimethylpyridinium iodide (**A**)*

The equimolar ratio of 2-methylpyridine (8 ml, 80 mmol) and methyl iodide (5.2 ml, 80 mmol) was added in dried acetone (50 ml). The whole mixture was taken in a two-neck flask (250 ml) fitted with thermometer and condenser. Then the mixture was refluxed at 60 °C for 2 h until it pale white precipitate crystallized. The resulting salts were collected by filtration, and the salt was washed with diethyl ether to removing of unreacted starting



Scheme 1. Synthesis process of DEASI.

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