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Characterization of Sulphamic Acid Single Crystal by Optical Techniques

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Abstract- The formation of crystal in nature, like snowflakes and minerals, as well as the preparation of the crystal in laboratories and factories made for technical application is called crystal growth. The most frequently used and most important method of producing single crystals of a given material is by solidification of its own melt called 'crystal growth from the melt' or just 'melt growth'. Its technical and economic importance is due to the fact that large single crystals can be grown very effectively and with a high yield at a relatively high growth rate. This growth rate is not limited by the transport of crystal species but only by the removal of the heat of crystallization. In the last decade single crystalline subject of increasing interest, because of their remarkable characteristics. Due to the large length-to-diameter ratio composed with perfect single crystal structure and chemical homogeneity, the mechanical properties approach theoretical value.

In this work we are interested in growing single crystal for nonlinear optical applications. Beside this, we are trying to develop new materials that have specific thermo-mechanical properties to be used as a suspension for the mirror in advance gravitational waves detector in order to decrease low thermal noise.

A monocrystal known as single crystal of sulphamic acid (H2NSO3H) is an inorganic nonlinear optical material was grown by slow evaporation solution growth technique. We have performed studies characterization of Sulphamic acid single crystal by X-ray diffraction and FTIR studies. The crystals are extremely good of the orthorhombic structure. We also study the other properties like viscosity, dielectric constant, melting point, PH scale and surface tension etc.

Index Terms- Growth from solution, coefficient of viscosity, dielectric constant, melting point, PH scale and surface tension, inorganic compound.

I. INTRODUCTION

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Sulphamic acid (H2NSO3H) is an important Industrial chemical compound, which is strongly soluble in water. The molecular weight of this compound is 97.09. The growth of the single crystal in both science and technology important application of such crystal. The growth and size of any crystal depends on the condition of its formation. Temperature, pressure, presence of impurities etc will affect the size perfection of crystal.. In the recent years, several studies dealing with organic, inorganic and semi organic molecules and materials for non linear optics (NLO) reported, due to the increasing need for cheap and easily process able material. Crystals take variety of shapes, depending on the internal factors. Both internal and external factors influence the growth rates, and therefore, they modified the crystal morphology. Crystal grown from conventional solution growth method was used as a seed. The plane which has fast growth rate has chosen for this study. The growth of the face depends on the external factors such as temperature and pressure. The concentration of solution increases. In crystal growth literature, the recently discovered uniaxially solutions – crystallization method of Shankar Narayan – Ramasamy (SR) is a suitable method. To effectively control the orientation of molecule during the bulk crystal growth from solution at room temperature with 100% solute – crystal conversion efficiency. Non-linear optical (NLO) single crystal are used in the area of fiber optics, communication, optical frequency conversion, optical data storage etc. In the present investigation sulphamic acid single crystal or monocrystal grown by three distinct methods are as follows:

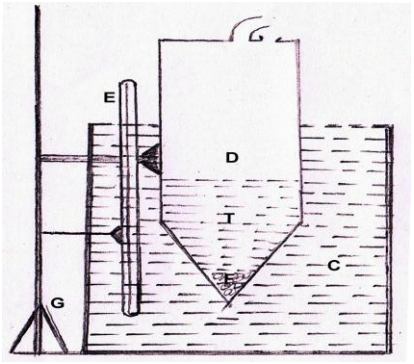
1. **Growth from solution**
2. **Growth from melt**
3. **Growth from vapor**

II. EXPERIMENTAL DETAILS

We are using the growth from solution method to grow the crystal .We prepared 26 % solution of sulphamic acid at about 30^0 to 45^0 temperature. Growth of sulphamic acid crystal was carried out by S. R. method by slow evaporation. In this method of growth assembly used 'L' bend ampoule made by borosilicate glass with seed mounting pad which is parallel to that used by S. R. method. The selection of the solvent is an important step in the bulk growth of crystal from solution by slow evaporation method. The size of the crystal depends on the amount of material available in the solution, which in turn is decided by the solubility of the material in that solvent. Seed crystal were harvested within 2 to 4 days.

As shown in fig. 1 consist of glass ampoule with seed mounting pad. A resistive ring heater (80W) of diameter 10 mm provides the necessary temperature around the growth ampoule for solvent evaporation. A ring heater positioned at the top of the growth ampoule was connected to the solvent of evaporation. In this experiment, the temperature around the growth region is mentioned at 65^0 C with ± 0.05^0 C accuracy depending on the evaporation rate of the growth crystal. The most important requirement for the growth of crystal from the vapor is sufficiently high saturated pressure P of the source to obtained an

acceptable growth rate. The rate of the crystal growth from the vapor method is 1 mm per day = 1.16 X 10^{-6}

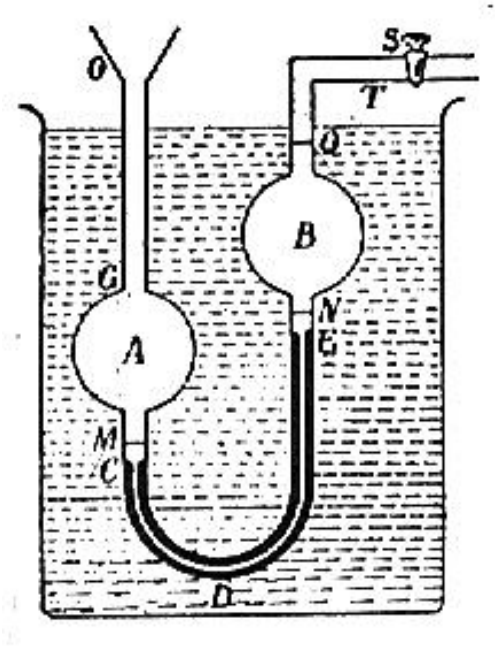


D = Glass Ampoule
T = Sulphamic Acid Saturated Solution
C = Water Bath
E = Thermometer
F = Seed Crystal of S.A.
G = Fixed Stand
L = L bend tube

Fig.1: Schematic Diagram of the experimental setup **S. A. Crystal**

Viscosity:-We used Ostwald Viscometer. The viscosities of two liquid, or the same liquid a different temperature, may be compared with the help of simple apparatus known as Ostwald Viscometer. As shown in fig.2 viscometer consists of U shape tube with two bulb A & B. A capillary position CDE a side tube T, fitted with a Tap S, & with three marks engraved on it, at M, N, & Q respectively. Prepare the saturated solution at a normal temperature of 1% sample in 100 ml distilled water.

This solution has been taken in four different concentration i e 0.4%, 0.6%, 0.8% & 1.0%. From this solution fill 10 ml in Ostwald Viscometer apparatus and measure time in different position of the liquid. If the flow of liquid increase to increase the specific coefficient of viscosity. The temperature of liquid increases to decrease the viscosity of the given liquid.



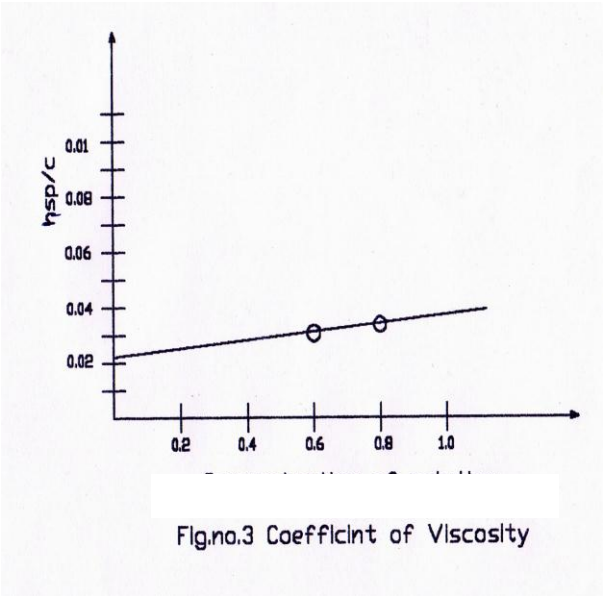


Fig. 2: Ostwal Viscometer Apparatus **Fig. 3: Coefficient of Viscosity Graph**

Dielectric constant:- To study the dielectric constant we made a very simple technique in the laboratory. The single crystal of Sulphamic Acid crushed into powder form. In this method the capacitance of a capacitor increases when it is filled with an insulating medium. The increasing capacitance depends on the property of the medium, called dielectric constant. Dielectric constant method which we set up in the lab as shown in fig 4 .Both stripping copper plate varnished. Fine powder of this sample kept between two plate & packed with the rubber band.. At that time do not pass the air into the plate through the rubber band. After that to measure the capacitance of this sample with the help of millimeter & calculate the dielectric constant.. The maximum dielectric constant response revealed may be of great interest in application. The sulphamic acid (H_2NSO_3H) Material with high dielectric constant values are quite good. Which can be used in the thin film transistor tunneling devices,

insulator, fabricating capacitors and other electric field devices. Sulphamic acid crystal is an insulating material it gets higher energy and higher dielectric constant.

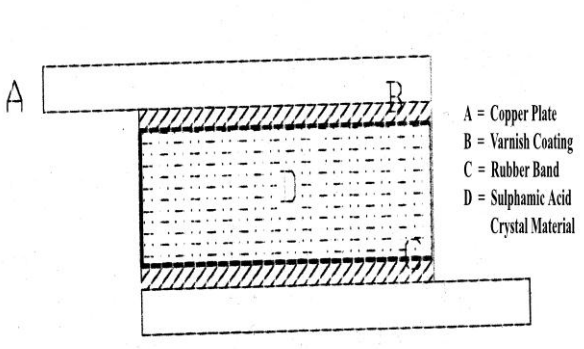
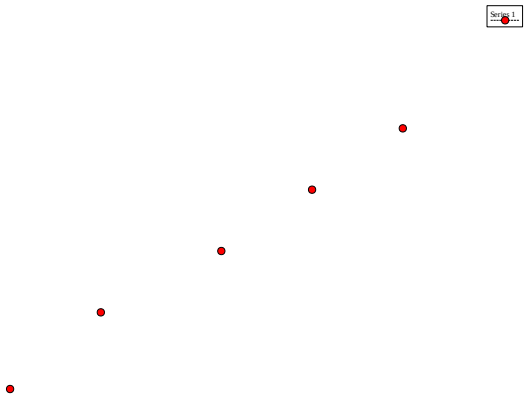


Fig.4 Dielectric Constant Method



Melting Point:- We study the capillary melting method by using the one side open capillary tube and to measure the melting point of the sample by using least capillary melting method. Crushed the SA crystal sample and fill the capillary tube and used the melting point instrument by measuring the melting point with the help of thermometer. The melting point of the crystal growth of sulphamic acid is 206˚C. The high melting point of the material contains strong bond and get the more energy of heat. Such type of material are used in the fire retardants for paper & other cellulosic's, stabilizing agent, for chlorine and hypochloride in swimming pool closed hydrogen system, bleaching paper pulp and textile catalyst for urea formaldehyde – resins.

PH Meter:- PH Scale should be clear that every acquire solution whether acidic, alkaline & neutral contain both hydrogen (H^+) and hydroxyl (OH^-) ions. There product always constant and equal to 1 x 10^{-14} at normal temperature at about 25˚C. The solution are acidic or alkaline depends upon the two ions is present in greater concentration then the other. The sulphamic acid growth crystal is an acidic medium and its PH value of preparing 1% of solution is 1.14 by using model CL-46, Sr. No.4944, Power 230 v ac PH meter about 25˚C normal temperature This sulphamic acid are used in the operation of to remove the excess nitrus Acid from the preparing diazo in the dyes plant. The sulphamic acid is less PH i.e. more acidic than the formic and phosphoric acid and acitic acid. Such type of material are used in metal and ceramic cleaning and nitrite removal in azodize operation.

Surface Tension :- To determine the surface tension ,we use capillary rise method. This method is based on the rise of liquid in a capillary tube. Consider a capillary tube lowered in a liquid that wets its surface. The contact angle between the glass & liquid is now" 0" the rising position of the liquid high depends upon the surface tension and density. The column of the liquid in the capillary is evidently being by the supported by the force acting along the surface of water, is known as force of surface tension. Measure the height through which the liquid rise and radius of capillary tube in centimeter. The height is measure by cathetometer and radius of the capillary tube measure by the traveling microscope. The rise or fall of a liquid in a capillary can be better understood by involving the concept of cohesion and adhesion position. In this method the liquid solution is depressed in the capillary tube. We found surface tension of the growth crystal of sulphamic acid is 116.41d cm-1.

III. Result & Discussion

From study of study FTIR and X-ray diffraction it is confirmed that vibration frequency of sulphamic acid. We found the dielectric constant value much higher, those reported by earlier workers. It is observed sulphamic acid is more acidic. The coefficient of viscosity is found to be 0.022dyne.sec.cm^{-2}. which is very less .and the Surface tension of sulphamic acid is more. We have observed that crystals are extremely good of orthorhombic structure. We introduced a new method to study dielectric constant. Further study on the crystal growth shows that it is more acidic, and coefficient of viscosity is found to be 0.022 d sec /cm which is very less.

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