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Growth and characterization of L- Proline dimercury chloride: NLO single crystal

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ABSTRACT

Optically cleared single crystal of L-proline dimercury chloride (LPDMC) has been successfully grown from aqueous solution by slow evaporation solution growth technique. Single crystal X-ray diffraction analysis confirms that the crystal is found to be a crystallized complex crystallizes in triclinic system with space group P_1. Optical transmittance studies on this sample shows the minimum absorption region which is well suited for optical applications. The mechanical strength of the grown crystal has been studied using Vicker's microhardness tester. The melting point is reported to be 101.8º C using thermal analysis.

KEY WORDS: L-Proline dimercury chloride, crystal.

1. INTRODUCTION

Crystal growth from solution in particular, the slow solvent evaporation technique has been widely used to grow nonlinear-optical quality single crystals. A new approach to high efficiency, low angular sensitivity organic based non-linear optical materials is to consider compounds in which a high polarizable organic molecule can be bonded to an inorganic host to form semi-organic crystals, as they have large nonlinearity, high resistance to laser-induced damage, low angular sensitivity and good mechanical hardness. In the present investigations, an attempt has been made to grow single crystals of semi organic material by slow evaporation solution growth technique at room temperature. This paper reports the synthesis, growth aspect and characteristic studies of the L-proline dimercury chloride. The grown crystals are subjected to X-ray analysis, optical, hardness and thermal studies presented and discussed.

2. MATERIAL SYNTHESIS

Slow evaporation technique was used for the growth of the title compound. The L-proline and mercury chloride were taken in the molar ratio 1:2, this has been further purified by repeated re-crystallization process to improve the optical quality. The reaction of synthesis is given by

$$C_5H_9NO_2 + 2HgCl_2 \rightarrow C_5H_9NO_2 2HgCl_2 \quad (1)$$

The saturated solution of L- proline dimercury chloride ($C_5H_9NO_2$ 2Hg Cl_2) was obtained by dissolving the recrystallized salt in double distilled water with continuous stirring using magnetic stirrer, the solution was kept at room temperature for evaporation of the excess amount of water. The crystal to a size of 17 × 4 × 2 mm^3, the crystal was harvested within 3 weeks (Figure 1).

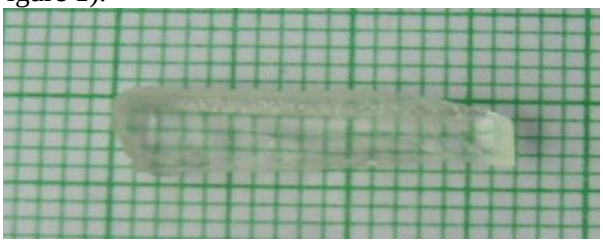

Fig.1.Photograph of the LPDMC single crystal

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction analysis: Single crystal X-ray diffraction analysis for the grown L-proline dimercurychloride (LPDMC) crystal has been carried out to confirm the crystallinity and also to identify the unit cell parameters using Bruker Kappa APEX-2 diffractometer with MoKα (λ = 0.71073Å) radiation. Triclinic crystal system and space group is P1 Unit cell dimensions are a=7.2709(4) Å, b = 9.4533(5) Å c = 10.4885(5) Å.

3.2. Optical transmittance studies: The transmittance spectrum of grown single crystal was recorded using Varian Cary 5E UV–vis–NIR spectrometer in the range of 200–1400 nm with a crystal of 2mm thickness as shown in Figure 2. From the transmission spectrum, it is observed that the maximum transparency of 85% and UV cut-off wavelength 330nm were observed for the LPDMC single crystal. The very high transmission in the entire visible and near IR region and short cut-off wavelength facilitates it to be a potential NLO material for second harmonic frequency doubling.

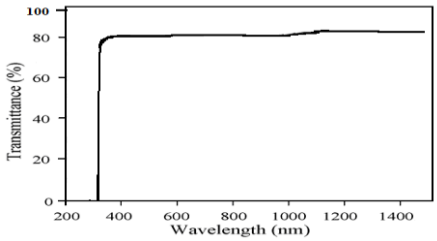

Fig.2.UV-transmittance spectrum of LPDMC

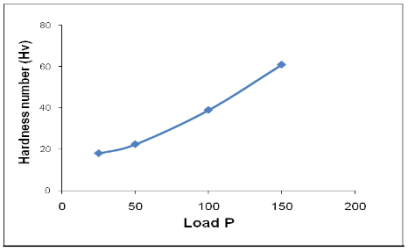

Figure.3.Plot of P vs Hv for LPDMC crystal

3.3. Microhardness studies: The hardness tests for LPDMC crystal were carried out by Leitz micro hardness tester with a diamond pyramidal indenter. The diagonal lengths of the indentation for various applied loads in kg are measured for a constant indentation period of 15 seconds. The Vickers's hardness number (H_v) is calculated using the relation,

$$H_v = \frac{1.8544\,P}{d^2} \quad \text{Kg/mm}^2 \qquad (2)$$

Where, P is the applied load in kg and d is the diagonal length in mm. The variation of H_v with the applied load P is shown in Figure 3. From the graph, the hardness value increases with increase in the load, thus satisfying the reverse indentation size effect (RISE). The hardness is 61.2 kg/mm^2.

3.4. Thermal analysis: Themogravimetry (Figure 5) and differential thermal analysis (DTA) for L-proline dimercurychloride was done using NETSZCH STA 409/C/Cd system by heating the sample of about 39.98 mg in a crucible between 30˚C to 820˚C at a rate of 20 ˚C/10 min in nitrogen atmosphere. The TG results reveal no weight loss below 200˚C and hence the crystal is found to be freed from physically absorbed and lattice entrapped water. At 200˚C, major weight loss started and goes up to 390˚C, which is followed by minor weight loss. The first major weight loss is due to the decomposition of the compounds and later, the weight losses are due to the decomposition of the residues. The total weight loss corresponds to 98%. The DTA results are illustrated in figure 6, showing there is a sharp endotherm with a maximum at 188˚C which is assigned to melting. The endotherm immediately followed by an exotherm closed at 190˚C coinciding with the first major decomposition in the TGA.

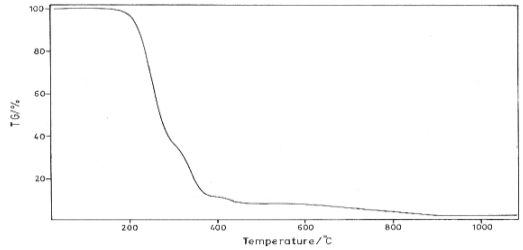

Fig.4.TGA spectrum of LPDMC

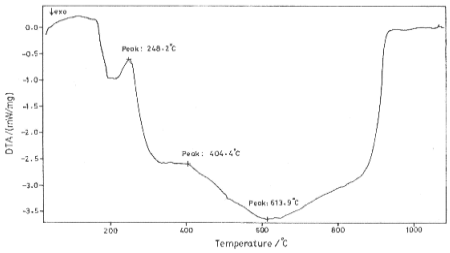

Fig.5.DTA spectrum of LPDMC

4. CONCLUSIONS

Good quality single crystals of L-proline mercury chloride (LPMC) were grown by slow evaporation technique. Single crystal X-ray analysis, confirmed the L-proline dimercury chloride (LPDMC) is triclinic system and space group P_1. Optical transmittance studies on these samples reveal minimum absorption in the region 500 nm and 800 nm. Mechanical hardness studies reveal that Vicker's hardness number decreases as the load increases satisfying reverse indentation size effect (RISE). Thermal analysis taken on the title compound reveals that there is no weight loss below 200˚C hence the crystal is free of lattice entrapped water up to 200˚C and the melting point of the compound is found to be 188˚C. These qualities advocate that this material is well suitable for device fabrication.

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