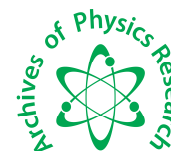




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Studies on thermal behaviour of Lead Iodide single crystals grown by gel method

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ABSTRACT

Lead Iodide have been grown by gel method using single diffusion method. Optimum conditions to grow these crystals have been obtained. Crystals of Lead Iodide have been withdrawn from the test tube in which the crystals have grown. After careful withdrawal of Lead Iodide, they are washed with acetone and kept under light for whole night. Then they are crushed of constant size (150 mesh). These grown crystals were characterized by XRD and thermal analysis. XRD confirms the structure of Lead Iodide as hexagonal, also the lattice parameters are closely matching with the ASTM data of Lead Iodide. Thermal analysis infers that Lead Iodide is thermally stable crystals and does not affect by the temperature. The results of these observations are described and discussed.

Keyword: Gel method; XRD; Thermal analysis.

INTRODUCTION

Lead Iodide is a member of IV-VII semiconducting compounds having band gap of 2.55eV, though the way synthesization is different. The details of synthesization and applications of Lead Iodide were given in different papers (1,2,3). No. of researchers worked on different properties of Lead Iodide like photodecomposition, image recording capability, electrodeposition, polytypism, photoluminescence etc. (4). Though lot of work have done on various properties of Lead Iodide, less work on thermal properties of Lead Iodide found.

In the present course investigation, the main aim is towards the thermal properties of Lead Iodide. The different properties of thermal like TGA, DTA and DSC have been studied and reported. Lead Iodide is found to be thermally stable material.

MATERIALS AND METHODS

Gel technique is very simple and cheap. In the present investigation, a solution of Sodium Silicate (sp.gr. 1.04 g cm⁻³) is mixed with Acetic Acid (1 N) and Lead Acetate (0.5 N) and

allowed to set the gel in constant temperature bath at 30⁰ C. After setting the gel Potassium Iodide (1 N) is then placed on the top of the gel. As the time lapses crystals seems to grow inside the gel. About 12 days are needed for the complete growth of the Lead Iodide crystals. Carefully they are taken out, washed and kept in light for one night and used for the characterization.

The X-ray diffraction patterns (XRD) were obtained by a diffractometer (Philips PW-1730) using CuK α radiation with Ni filter (1.5418Å) at North Maharashtra University, Jalgaon. The thermal analysis (TGA, DTA/DSC) of gel grown, doped and undoped Lead Iodide crystals have been carried out at CMET, Pune and Pratap College, Amalner.

RESULTS AND DISCUSSION

The diffractometer recordings for gel grown Lead Iodide is given in fig.1. Lattice parameters such as 'a' and 'c' were calculated from the 'd' values by method of successive refinement with the help of computer programme. The lattice parameters of gel grown Lead Iodide closely matches with ASTM parameters of Lead Iodide. Mean values of lattice parameters are tabulated elsewhere (5).

Thermal analysis

The thermal analysis (TGA, DTA/DSC) of gel grown, doped and undoped Lead Iodide crystals have been carried out in the present work. The TGA curve of Lead Iodide crystals, fig.1, were recorded as function of temperature and % weight loss of substance. The DTA and DSC of Lead Iodide crystals were recorded and are given in fig. 3 and fig. 4 respectively.

Thermograms of Lead Iodide crystals shows that there is no loss in weight upto 390⁰C, hence the material is thermally stable, which indicates no possibility of co-ordinated water molecules or any water of crystallization. Lead Iodide crystals melts at around 400⁰C and slow and gradual weight loss is observed. Then after, slow decomposition is observed from 420⁰C to 480⁰C, and then sudden loss in weight is observed from 600⁰C. The DSC curve of undoped Lead Iodide crystals is represented in fig.3, there is an endothermic peak at 405⁰C and an exothermic peak at 423⁰C. Hence, it is inferred that an endothermic peak must have been caused by a phase transformation.

Thermogravimetric analysis provides information about the gross composition of polymer formulations. Using programmed heating and a change of atmosphere (pure gas) over the sample, it is easy to determine the weight losses associated with either light or heavy oils, bulk polymer and carbon black or other reinforcing agents.

With the help of DTA and DSC it is possible to measure,

- i) Physical changes and measurements, such as melting, crystalline phase change, changes in liquid and liquid crystalline states and in polymer, phase diagrams, heat capacity, and glass transitions, thermal conductivity and diffusivity and emissivity and
- ii) Chemical reaction such as dehydration, decompositions polymer curing, glass formation and oxidative attack.

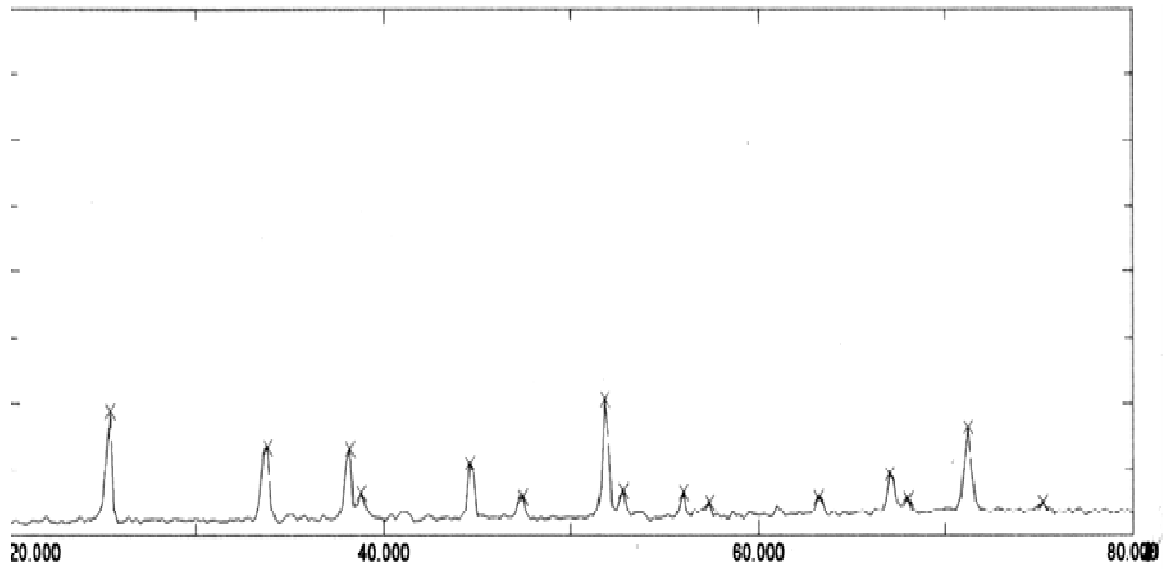


Fig.1. XRD of Lead Iodide crystals

Sample: No.3
Size: 12.4690 mg
Method: TGA
Comment: Range 20°To 400° Rate 10°/min. ATM.N2

TGA

File: C: E24TGA2K.004
Run Date: 15-Nov-00 14: 45

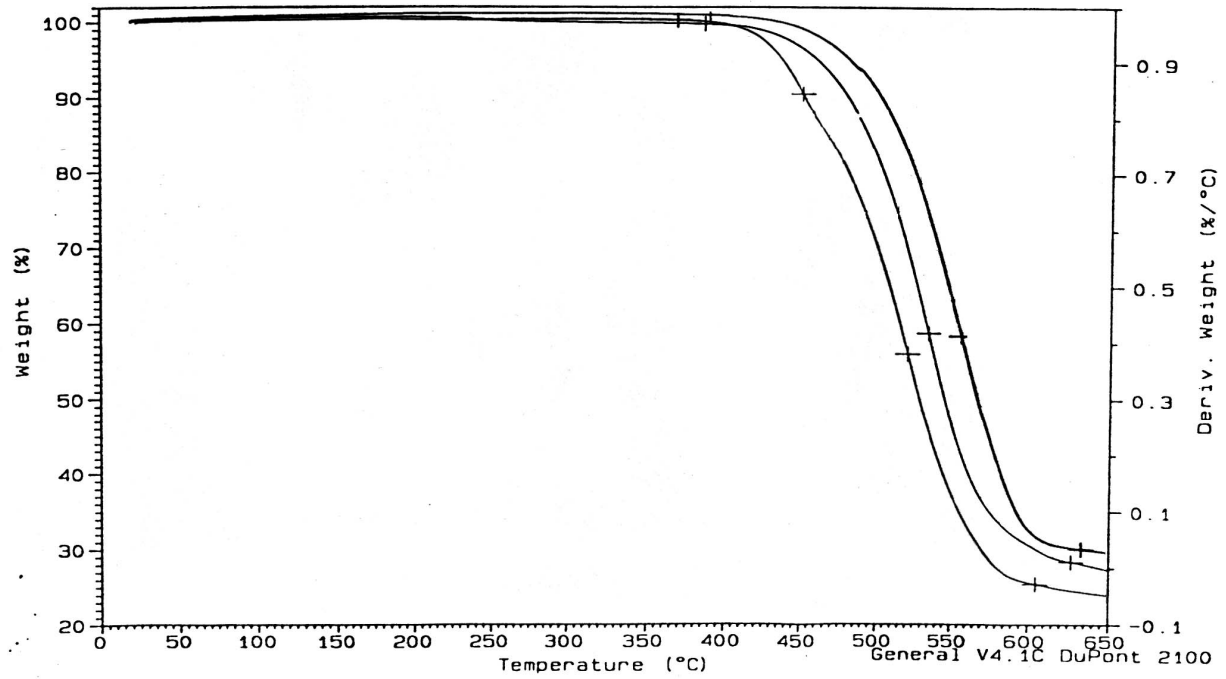


Fig.2. TGA curve for different concentrations

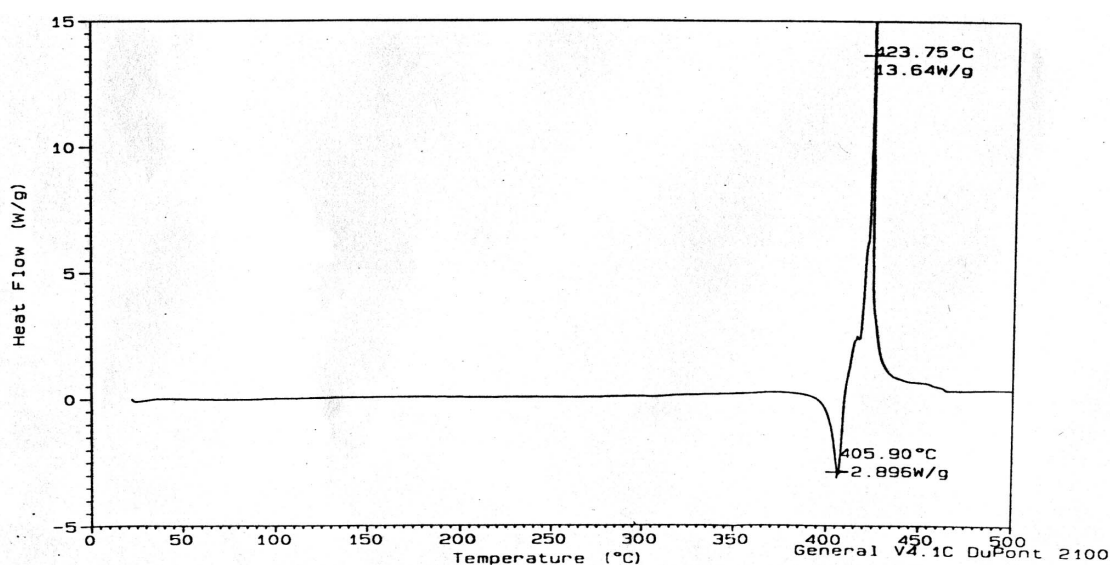


Fig. 3 DTA curve for undoped Lead Iodide crystals

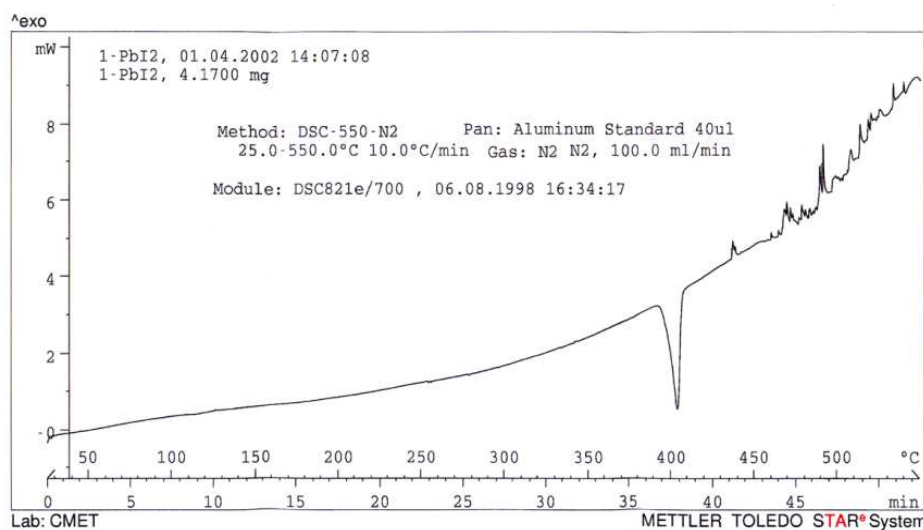


Fig. 4 DSC curve for undoped Lead Iodide crystals

CONCLUSION

1. Hexagonal structure confirmed by XRD
2. Lattice parameters are closely matching with the ASTM data of Lead Iodide
3. Thermal analysis shows that the grown crystals of Lead Iodide is thermally stable
4. It is established that, by Thermal Analysis, there is no co-ordinated nor water of crystallization present in the crystals.

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