

XRD STUDIES OF LEAD IODIDE AND LEAD IODATE CRYSTALS

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ABSTRACT

The single crystal of lead Iodide and lead iodate has been synthesized by gel method. The single diffusion method at room temperature has been used for synthesis. In this paper, 'd' values, lattice parameters 'a' and 'c', grain size were determined by XRD. The study shows hexagonal and orthorhombic structure for lead iodide and iodate respectively.

KEYWORDS: Gel method, XRD

INTRODUCTION

Very few literatures are available on the study of lead iodide and lead iodate crystals. Most of the iodide and iodates exhibits prominent non linear optics (NLO) behavior. Most of the iodate and iodide compounds are insoluble in water and decompose before melting. Hence, crystals of such type of compounds cannot be grown by either slow evaporation or melt techniques. Here we report the relationship between structure properties of lead iodide and iodate crystals.

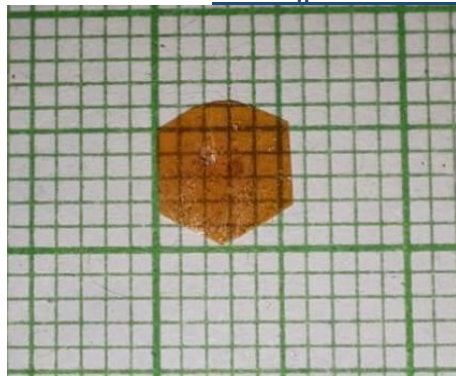
MATERIALS AND METHODS

The single diffusion technique was used to grow the crystals of Lead Iodide and Iodate. Crystals were grown at room temperature. The A.R. grade materials were used for synthesis.

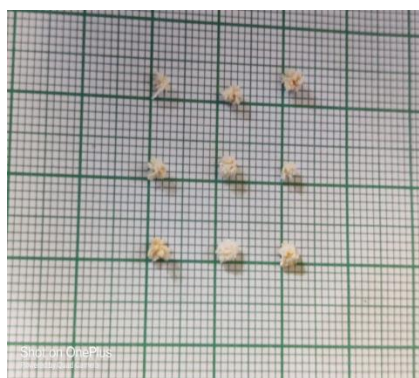
Preparation of Lead iodide and iodate crystals by gel method:

Gel Method

The growth of crystal recorded by diffusion gel method in which solute is supplied to grow crystals. Once a new solute has been brought to the surface by diffusion, growth takes place by two dimensional surface nucleation mechanisms. Gel was prepared at constant room temperature by adding the Sodium Metasilicate solution in to the solution of acetic acid (5ml, 2N) drop by drop with constant stirring till 4.1 to 4.4 pH of the mixture obtained. Mixture was then transferred into the test tube of length 15cm and 2.5cm diameter. The mouth of the test tube was covered by cotton to keep the solution free from dust and impurities. After setting the gel nearly in 10 days, it was again kept for aging for 2 days. Then slowly pour over the set gel Lead Acetate (5ml,1N) so that the gel should not disturb. The growth of Lead Iodide and iodate can be seen Day by day. After completion of growth crystals were taken out from the gel. Then they are washed with double distilled water and acetone and kept under light. The hexagonal and orthorhombic shaped crystals were formed by reaction of Lead nitrate, Potassium Iodide and iodate in gel medium.



Lead iodide crystal



Lead iodate crystal

Structural Analysis of the crystals:

X-ray diffraction patterns of the crystals were recorded with the Phillips X-ray diffractometer model using CuK α radiations with Ni filter ($\lambda = 1.5418\text{\AA}$). The lattice constant were determined.

Result and Discussion:

Structural Investigation of Lead Iodide and Lead Iodate.

X-diffraction analysis of crystals were carried out with view to gain information about the aspect interplaner spacing, lattice parameters a and c, identification of crystal structure, to determine the particle size of material under investigation.

X-Ray Diffractogram of lead iodide and lead iodate:

Figure 1 represent XRD pattern of lead iodide. It shows very sharp peaks having high intensity, which leads to perfect crystallization and larger particles and figure 2 represent XRD pattern of lead iodate crystal.

XRD pattern shows hexagonal and orthorhombic structure. It is represented in fig 1 and fig 2 the observed peaks are matching well with JCPDS (73-1754 and 82-0477) data.

composition	Interplaner distance(d) \AA
Lead iodide	2.13 \AA
Lead iodate	2.83 \AA

Table no. 1

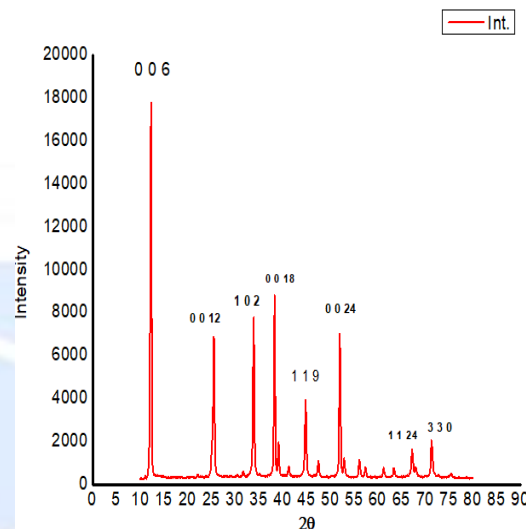


Fig.1.XRD of lead iodide

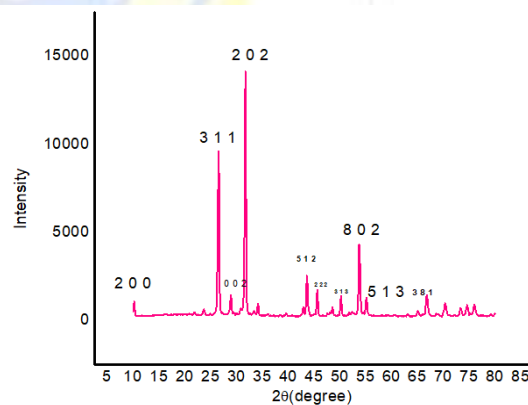


Fig.2. XRD of lead iodate

Lattice parameters:

The parameters ‘a’ and ‘c’ of the hexagonal and orthorhombic structure are computed from observed d values by a method of successive refinement. Table present values of ‘a’ and ‘c’

Composition	Lattice parameters	
	a Å	c Å
Lead iodide	4.557 Å	41.87 Å
Lead iodate	16.70 Å	6.083 Å

Table no. 2

Grain Size

The grain size was determined using Scherrer formula which gives an average grain size. The grain size was estimated by measuring the full width at half maximum in case of the most intense peak and represented in table.

Composition	Grain size (nm)
Lead iodide	27.24 nm
Lead iodate	28.14 nm

Table no.3

CONCLUSION:

1. Lead iodide and lead iodate crystals are synthesized at room temperature by gel technique. Lead iodide crystal exhibits hexagonal structure while lead iodate crystal exhibits orthorhombic structure.
2. d values, lattice parameters `a` and `c` are very closed to the reported values.

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