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Optimized growth conditions of some gel grown second group iodate crystals

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Abstract— *Single Crystals of Strontium iodate, Zinc iodate and Mercuric iodate were grown by gel technique using diffusion method. The optimum growth conditions were established by varying various parameters such as pH of the gel solution, gel concentration, gel setting time, concentrations of reactants etc.*

Keywords— Gel technique, Single diffusion method, Strontium iodate crystals, Zinc iodate crystals, Mercuric iodate crystals, Optimized growth conditions.

1. INTRODUCTION

The advances in the science of the solid state and material science depends upon the availability of good quality single crystals Consequently, tremendous amount of efforts has been made on the development of crystal growth techniques, each having its own importance and potentiality.

The new rapidly developing branches of science and technology, such as quantum electronics, quantum and non linear optics, semiconductor instrumentation, lasers and masers etc all involves the use of single crystals and their singular properties. So several techniques have been developed and are still being developed in rapid succession to synthesize better and better quality of crystals which are rare in nature or not yet grown in laboratory.

Now-a-days crystals have become the pillar of modern technology in all the respects with this aspects in the mind and ever increasing the demand of single crystals in the variety of field in science and technology, the work of the growth and the study of some important crystals have been under taken in the laboratory.

Today with modern technology, with sophisticated instrument many national laboratories and university are growing varieties of crystals, which have the utility in day to day life, still the growth of some crystals by using simple equipment by utilizing fundamental properties of material much of the research work have been carried out and it not less important by considering all aspect and available facilities in laboratories the work of the growth of iodated single crystals by Gel technique which is one of the simplest technique have been undertaken in the present work.

Growth of crystals by gel method is promising techniques for growing single crystals of substances which are sparingly soluble in water and decompose before their melting point, according to thermodynamics consideration since the growth proceeds at near ambient temperature, the grown crystal would contain relatively lesser concentration of non-equilibrium defects. The growing crystals are held in the gel in a strain free manner, thus limiting effects due to impact of the bottom or the sides of the container. In addition in this method almost complete suppression of large scale movements like convection is achieved which otherwise can be harmful to the crystals perfection. In gel method, the rate of diffusion of reactants can be controlled, since the gels are network of cavities of several tens to several thousand of angstroms in diameter, communicating through slightly smaller orifices. The gel growth technique appeared quite attractive for growing crystals, on account of its unique advantages in terms of crystals produced and the simplicity of the process moreover the method is inexpensive and within the scope of the Laboratory.

In the present work, crystals of strontium iodate, zinc iodate and mercuric iodate were grown. However, there are very few reports in the literature on the growth of these iodate crystals by gel method. These three types of crystals were grown by gel method by using single diffusion techniques. Whiskers (2 to 4 cm), Dendritic (curved) and cubical (2mm x 2mm x 2mm) of strontium iodate were obtained. Most of the strontium iodate crystals were transparent, shining, well isolated and very few of them were opaque. In case of zinc iodate crystals, various shaped such as hexagonal, star shaped and micro crystals were obtained. These crystals were found to be grown near the gel interface. Most of them were opaque and very few of them were transparent crystals.

Single diffusion technique proved to be suitable for growth.

Mercuric iodate crystals of type spherulitic (1 to 3 or 4 mm in diameter) were obtained. It was found that as the concentration of the reactant $Hg(NO_3)_2$ in the gel is increased, the size of the spherulites is also increased. Single diffusion method is found more suitable for growth of these crystals too. The crystals of strontium iodate shows better habits, possesses appreciable size and better transparency among the grown crystals. Better transparency of strontium iodate may be due to presence of strontium.

Optimum growth conditions for gel grown crystals established by varying various parameters such as gel density, pH of gel, gel setting time, gel aging time, etc.

2. Materials and Method:- Test tubes were used as crystallizing vessels. The silica gel was used as a growth media. Gel was prepared from aqueous solution of sodium meta silicate. The gel was acidified by acetic acid. The chemicals used for growth of single crystals of these iodates were CH_3COOH, $Na_2SiO_3.5H_2O$, KIO_3 , Na IO_3 , Sr Cl_2 , Zn Cl_2 , Hg Cl_2 . All chemicals were of A.R. grade.

A set of experiments using different pH values for the gel and the different concentrations for reactants were carried out. Different molar masses were tried to determine the optimum growth conditions. Out of the two reactants, one of the reactant such as $Sr(NO_3)_2$ or $Zn(NO_3)_2$ or $Hg(NO_3)_2$ was incorporated in gel and KIO_3 was used as the supernatant over the set gel.

Experiments were also carried out by interchanging the position of reactants. These experiments do not yield any crystal at all. $Sr(NO_3)_2$ or $Zn(NO_3)_2$ or $Hg(NO_3)_2$ having different concentrations was incorporated into the gel. This solution was then transferred to borosil glass tube of diameter 2.5 cm and 25 cm in height. The mouth of the test tube was then covered by cotton plug. After setting of the gel, it was left for aging for different periods of time. Another reactant having different concentrations was then added as supernatant over the set gel. A set of experiments were carried out by changing different concentrations of the reactants. The chemical reaction inside the gel can be expressed as :

$$XCl_2 + 2YIO_3 \rightarrow X(IO_3)_2 + 2YCl$$

OR

$$X(NO_3)_2 + 2YIO_3 \rightarrow X(IO_3)_2 + 2YNO_3$$

Where X =Sr or Zn or Hg and Y = K or Na

3. Results and discussions: Whiskers (2 to 4 cm), Dendritic (curved) and cubical (2mm x 2mm x 2mm) of strontium iodate were obtained. Most of the strontium iodate crystals were transparent, shining, well isolated and very few of them were opaque. In case of zinc iodate crystals, various shaped such as hexagonal, star shaped and micro crystals were obtained. These crystals were found to be grown near the gel interface. Most of them were opaque and very few of them were transparent crystals. Mercuric iodate crystals of type spherulitic (1 to 3 or 4 mm in diameter) were obtained.

Table 1 : Crystals of iodates.

Type	Method	Chemicals used	Crystal habits	Quality	Size
Strontium iodate $[Sr(IO_3)_2]$	Gel method by using single diffusion techniques	Na_2SiO_3 CH_3COOH $SrCl_2$ KIO_3	Whisker	Opaque, brittle	2-4 cm
			Dendritic	transparent, few opaque	curved
			Cubical	Good, transparent	2 x 2 x 2 $(mm)^3$
Zinc iodate $[Zn(IO_3)_2]$	Gel method by using single diffusion techniques	Na_2SiO_3 CH_3COOH $ZnCl_2$ KIO_3	Hexagonal	Transparent, few opaque.	----
			Star shaped	Opaque at the centre, transparent at the edges	----
			Micro crystals	Without shape	----
Mercuric iodate $[Hg(IO_3)_2]$	Gel method by using single diffusion techniques	Na_2SiO_3 CH_3COOH $Hg(NO_3)_2$ KIO_3	Only Spherulitic	Opaque	1 to 4 mm in diameter
				Opaque	
				Opaque	

Table 1 gives details about method and chemicals used, different habits of crystals obtained, their transparency, etc.

The crystals of strontium iodate shows better habits, possesses appreciable size and better transparency among the grown crystals. Better transparency of strontium iodate may be due to presence of strontium. These crystals inside the test tube and outside on scaling graph paper are as shown in the figure 1.

In case of zinc iodate crystals, various shaped such as hexagonal, star shaped and micro crystals were obtained. These crystals were found to be grown near the gel interface. Most of them were opaque and very few of them were transparent crystals. Single diffusion technique proved to be suitable for growth. These crystals inside the test tube and outside on scaling graph paper are as shown in the figure 2.

Mercuric iodate crystals of type spherulitic (1 to 3 or 4 mm in diameter) were obtained. It was found that as the concentration of the reactant $Hg(NO_3)_2$ in the gel is increased, the size of the spherulites is also increased. Single diffusion method is found more suitable for growth of these crystals too. These crystals inside the test tube and outside on scaling graph paper are as shown in the figure 3.

Fig.1: Few cubical transparent crystals of strontium iodate inside and outside the test tube.

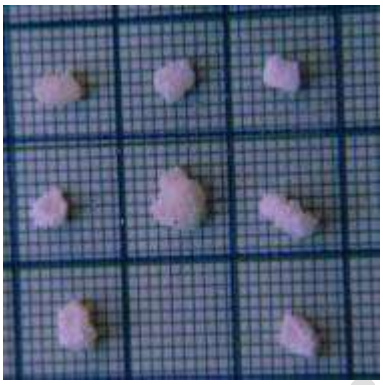

Fig.2: Few cubical transparent crystals of Zinc iodate inside and outside the test tube.

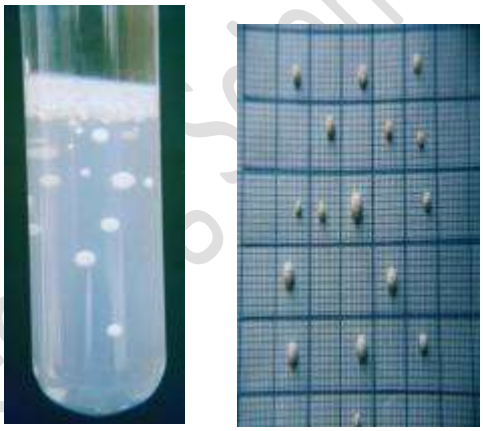

Fig.3: Few cubical transparent crystals of Mercuric iodate inside and outside the test tube.

Optimum growth conditions for gel grown crystals established by varying various parameters such as gel density, pH of gel, gel setting time, gel aging time, etc. are reported in Table 2.

Table 2 : Optimum growth condition for gel grown single iodate crystals.

Parameters	Strontium iodate	Zinc iodate	Mercuric iodate
Density of sodium meta silicate solution	1.04 gm/cm^3	1.04 gm/cm^3	1.04 gm/cm^3
Amount of acetic acid	2N, 5 cc	2N, 5 cc	1N, 5 cc
pH of mixture	4.4	4.4	4.2

Temperature	Room temperature	Room temperature	Room temperature
Gel setting time	12 days	12 days	14 days
Gel aging time	120 hours	120 hours	144 hours
Period of growth	5 weeks	3 weeks	3 weeks

For all these three crystals, suitable value of density of sodium meta silicate solution is found to be 1.04 gm/cc, pH value for strontium iodate and zinc iodate is found to be 4.4 and for mercuric iodate, it is 4.2. For pH 4.4, gel took 12 days to set and this gel was allowed to age for 5 days, while gel of pH 4.2 took 14 days to set and this gel was allowed to age for 6 days. Crystals were removed from test tubes after 5 weeks. After 5 week period, further growth was not noticed. Sometimes crystal became opaque or transluscent due to inclusion of silica in them. Reason may be the unnecessary exposure to silica gel. Various concentrations of reactants were tried. Experiments by changing the positions of reactants were also carried out. Once the optimum values of concentration of reactants were obtained, experiments of concentration programming were also carried out. All these parameters have more or less effect on growth and habit of these crystals.

4. Conclusion:

1. Gel method can be used at room temperature so that it is in the scope of laboratory.
2. This method is simple and less expensive.
3. In fact this method is alternative for the growth of crystals, which decomposes below their melting point.
4. It is also useful for those not having suitable solvents for recrystallization.
5. The crystals can be observed easily in all stages of growth.
6. A gel acts as three dimensional crucible which support the crystals and at the same time yields to it's growth without exerting major forces on it.
7. The gel medium prevents convection currents and turbulence and also helps in the formation of the good crystals by providing a frame work of nucleation sites.
8. A three dimensional crucible frame work of the gel remains chemically inert and harmless, in which the crystal nuclei are delicately held in the position of their formation and growth thereby preventing damage, if any, due to impact with either bottom or walls of the container.
9. Nucleation can be controlled by concentration programming by dummy gel.
10. The gel is soft and yields mechanically to the growing crystals.
11. A gel provides an ideal medium for the diffusion of reacting ions and can be used to keep the reacting ions separated until reaction is desired.
12. As the gel is chemically inert, it does not involve the chemical reactions between the reactants during crystallization.
13. It permits the reactants to diffuse through the gel within a controllable slow rate.
14. Thermodynamic considerations reveal that since the growth proceeds at near ambient temperature, the grown crystals contain relatively lesser concentration of defects.
15. Since the gel reduces the in effect the speed of chemical reaction, crystals would be made to grow much larger than those grown by similar reaction in water or in molten state by double decomposition process.
16. Gel does not obviate enough supply of reactants.
17. Gel media provide excellent opportunity for growing good mixed crystals.
18. Defect free which are not very large but appreciable size of crystals can be grown using this method.
19. Last but not the least, the growth procedure is highly economical, it yields a good crop of crystals with the simple and almost inexpensive equipment. The method can be exploited even in smaller laboratories which do not possess sophisticated equipments to grow perfect crystals.

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