



Single Crystal Growth and Study of Lithium Tartrate in Gel Method

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ABSTRACT

Single crystals of Lithium Tartrate were grown by single diffusion gel technique. Methanol used as solvent for lithium chloride. Optimum conditions were established for growth of good quality pure crystals. Effect of various growth parameters such as PH of the gel, concentration of inner reactants, concentration of upper reactants, gel density etc. The lattice parameters of lithium tartrate are almost matching with the JCPDS data. The crystals studied using XRD, FT-IR, and thermal analysis, (TGA & DTA) scanning electron microscope(SEM), AES Were studied. Needle shaped and whitish colour, dendritic crystals were grown. [1-10]
Keywords : Gel Technique, Lithium Tartrate Crystals, Lithium Chloride.

I. INTRODUCTION

Gel technique is the best technique for growing single crystals at ambient temperature, the compounds which are insoluble in water and decompose before melting can be easily grown by this technique. Due to very slow controlled rate of crystallization and non-turbulence during growth, good quality single crystals are obtained from this technique. The aim of the present work is to grow good quality single crystals of pure lithium tartrate in sodium metasilicate gel. Many researchers have grown the tartrate crystals of different elements having potential applications. The tartrate crystals have potentials applications such as dielectric, ferroelectric and piezoelectric.

1.1 CRYSTAL GROWTH

The growth of crystal in the gel media is based on the diffusion of the reactant i.e. supernatant. Two techniques can be used for the process of diffusion of reactant in gel media.

1) Single diffusion technique and 2) Double diffusion

technique

The single diffusion technique was found to be most suitable for the growth of good quality crystals in present work. To grow the lithium tartrate crystals, we prepared different concentrations of LiCl solution in methanol or ethanol and used as a Supernatant solution.

1.2 APPARATUS USED

1. Borosil glass test tube (25 cm in length and 2.5cm in diameter)
2. Magnetic stirrer
3. Various size beakers
4. Burettes and pipettes
5. Digital pH meter
6. Specific gravity bottle
7. Cotton

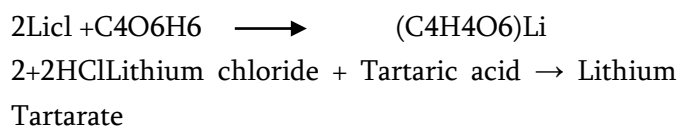
1.3 CHEMICALS Used –

The chemicals used for the growth of lithium tartrate crystals are commercial grade

1. Sodium metasilicate-(Na₂SiO₃.9H₂O) (Loba chemicals)
2. Tartaric acid - A.R grade (C₄H₆O₆) (Loba chemicals)
3. Lithium chloride -A.R grade 99.9% pure [LiCl]
4. Double distilled water for dilution
5. Methanol and ethanol

1.4 CHEMICAL REACTION –

The chemical reactions inside the gel can be expressed as



II. EXPERIMENTAL WORK

Gel was prepared by using tartaric acid and sodium metasilicate having different pH values. AR grade lithium chloride and tartaric acid were used as received. A 7ml of tartaric acid (1M) was taken in a small beaker. Sodium metasilicate solution of (1M) concentration was added drop by drop with constant stirring. The pH of solution was maintained between 4 to 4.5. The solution was then transferred in the test tube (2.5cm diameter and 25cm length) then covered its mouth with cotton plug. It was transparent initially, after 2/3 days, it turns milky and gel converted into semisolid with little amount of water on the top of the surface.

After setting of hydro silica gel, allow the aging of the gel. Aging makes the gel harder and reduces the diameter of the capillaries present in the gel. A 1M concentration of LiCl solution was made by dissolving LiCl in methanol or ethanol. The solution was often poured slowly with the help of pipette. The height of the supernatant component should be 60-70% of the gel height. With time, lithium chloride diffuses in the

gel and the crystals of lithium tartrate appears in the gel. This is called “single diffusion method”. In present work, lithium tartrate crystals were grown by single gel diffusion technique at various parameters. The crystal growth parameters were adjusted at optimum conditions to obtain full grown crystals. The solvents used to grow crystals played a vitally important role. In the present study, we used methanol or ethanol solvents to grow well-defined crystal in gel medium. An experimental set-up with working is shown in fig 1.

Fig. 1 Experimental set-up with working reaction during crystal growth in test tube.



PREPARATION OF GEL –

The various concentrations of tartaric acid and those of sodium metasilicate solutions were tried. The solution was added drop by drop with continuous stirring by using a magnetic stirrer till pH is reached in between 3.8 to 4.4. The acid was used for setting of the gel.

Table 1. Optimum conditions for growth of lithium tartrate crystals.

Sr.No	Various Process Parameter	Optimum conditions
1	Density of sodium meta silicate solution	1.04 g/cm ³
2	Volume of sodium meta silicate solution	18ml
3	Volume of tartaric acid	7 ml
4	Concentration lithium chloride	0.2 to 1M
5	pH	4.2
6	Concentration of tartaric acid	1 M
7	Environment temperature	25 to 30 °C
8	Solvent used	Ethanol or Methanol

III. MORPHOLOGY

Growth rate of the crystals is affected by parameters like pH and purity of reactance, gel setting time and aging time. Crystals of lithium tartrate are whitish, semitransparent and star shaped. Various types of crystals sizes with respect to supernatant concentration were obtained, which is tabulated in Table 2. We have also taken the photographs of the grown crystals in test tube as well as on the graph paper to measure the dimension of the grown crystal and it is represented in Fig 42. The largest crystal size of 3 x 1 x 1 mm³ was obtained at 0.8 M concentration of LiCl supernatant solution. It is important to note that the obtained crystals are of good quality, semitransparent, and well developed. The optimum conditions for lithium tartrate crystals grown in silica gel are summarized in Table 2.

Table 2 Effect of habit, quality and size of lithium tartrate.

Lower reactant C ₄ O ₆ H ₆ (ml)	Conc. of LiCl solution (1M)	pH	Habit	Quality	Size (mm)
7	0.4	4.2	Dendrite	Opaque	1 X 1 X1
7	0.6	4.2	Dendrite	Opaque	2 X 1 X1
7	0.8	4.2	Star shaped	Transparent, white, Good	3 X 1 X 1

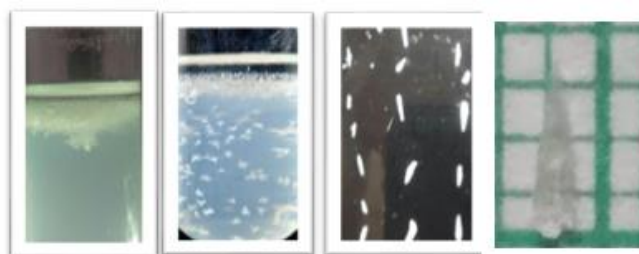


Fig 2 Growth of lithium tartrate crystal in silica gel at various concentration of supernatants solution of (a) 0.4 M, (b) 0.6 M, (c) 0.8 M, and (d) & (e), Photographs of the grown crystals.

IV. RESULT AND DISCUSSION

Crystals of lithium tartrate are whitish, semitransparent and star shaped. Crystals having size 2.5 mm x 4.5mm and thickness of about 2.5 to 3 mm are obtained. Different parameters such as concentration of reactants, pH of gel, impurities in the solvent, gel setting time, gel aging time etc. have considerable effect on growth rate. However as the reactants percolates through the gel, the controlled reaction occurs below, at the depth of 3 to 4 cm. Hence good quality, semitransparent, well developed faces of crystals are observed.

V. CONCLUSION

1. The grown lithium tartrate crystal are white

color good size of crystals are grown.

2. The supernatant solution prepared in methanol and ethanol gives good quality crystals.
3. Gel method is very simple and the colour is white.
4. within the scope of the laboratory, method be utilized to synthesize crystals, if optimum conditions are established.

VI. REFERENCES

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