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Synthesis and Characterization of Lithium Tartrate Single Crystals by Gel Technique

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ABSTRACT

In the present investigation, Lithium Tartrate crystals were grown in silica gel at ambient temperature. Methanol used as solvent for lithium chloride .Optimum conditions were established by varying various parameters such as pH of the gel solution, gel concentration, gel setting time, concentration of upper reactants, gel density etc. Crystals having different morphology were obtained. Whitish, shining, semitransparent, star shape, Needle shaped of lithium tartrate were obtained. The lattice parameters of lithium tartrate are almost matching with the JCPDS data. The crystal structure of compound was confirmed by powder X-ray powder diffraction, Atomic emission spectroscopy(AES) The crystals studied using XRD, FT-IR, and thermal analysis, (TGA & DTA), scanning electron microscope(SEM), Chemical analysis Were studied .Needle shaped and whitish colour, dentritic crystals were grown. [1-11]

Keywords : Gel technique, Lithium tartrate crystals, XRD & AES

I. INTRODUCTION

The Gel technique is the good technique for growing single crystals at room 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. CHEMICAL REACTION

Growth of lithium tartrate crystals is gained by reacting the components lithium chloride (LiCl), and tartaric acid. The expected reaction taking place in this work is as below:

$$2LiCl + C_4H_6O_6 \longrightarrow C_4H_4O_6Li_2 + 2HCl$$

II. CHARACTERIZATION OF LITHIUM TARTRATE

The features of the crystal are important components of the study of crystals. This helps the crystal grower to assess the quality, nature and property of the crystals. A large number of experimental techniques exist to assess the composition quality and the presence of its constituent elements.

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2.1. X-RAY DIFFRACTOMETRY (XRD)

The powder x-ray diffraction patterns for pure gel grown lithium tartrate crystals were recorded using powder method by Miniflex Rigaku, X-ray diffractometer at SAIF, IIT Powai. Computer program POWD was used to calculate (hkl) and'd' values of the recorded pattern. Calculated (hkl) and'd' values indicate orthorhombic crystals structure of lithium titrate crystals having lattice parameters of a = 6.7942 Å, b = 7.9807 Å and c = 5.2386 Å and volume (V) = 284.05 (Å)^3.

The unit cell parameters of the lithium tartrate crystals calculated by the computer program are shown in the table 1. The peaks obtained at 17.13, 20.26, and 21.40 corresponds to (110), (011), and (101) planes. The parameters fulfill the condition for orthorhombic system i.e. $a \neq b \neq c$ and $\alpha = \beta = \gamma = 90^o$. The recorded peaks are well matched to the JCPDS data card No. **01-0321** of lithium tartrate confirming the orthorhombic system .

Table 1. Unit cell parameters

Sr.No.	Lattice Parameters	Lithium Tartrate
1	System	Orthorhombic
2	a	6.7942 Å
3	b	7.9807 Å
4	c	5.2386 Å
5	V	284.05 (Å)^3

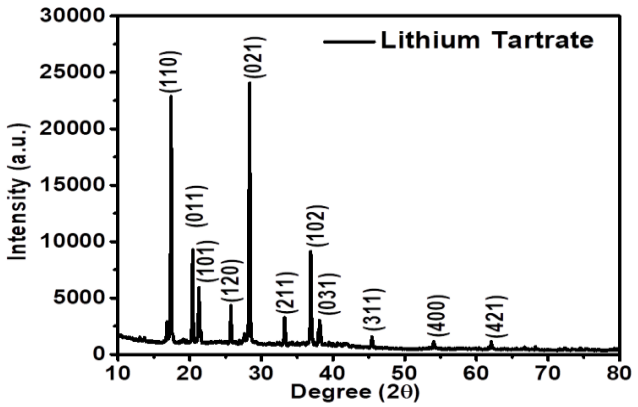

Fig 1. X-Ray diffraction pattern of lithium tartrate crystals grown by gel method

Table 2 Calculated and observed value of d-spacing

Peak no.	d-Spacing		Indices	2 θDegree	
	Obs.	Cal.	(h k l)	Obs.	Cal.
1	5.17	5.17	(1 1 0)	17.13	17.13
2	4.37	4.37	(0 1 1)	20.26	20.26
3	4.14	4.14	(1 0 1)	21.40	21.41
4	3.46	3.44	(1 2 0)	25.67	25.69
5	3.15	3.17	(0 2 1)	28.24	28.21
6	2.70	2.68	(2 1 1)	33.08	33.10
7	2.44	2.44	(1 0 2)	36.79	36.71
8	2.37	2.37	(0 3 1)	37.93	37.88
9	2.01	2.01	(3 1 1)	45.05	45.05
10	1.69	1.69	(4 0 0)	53.89	53.90
11	1.49	1.49	(4 2 1)	61.87	61.84

III. ELEMENTAL ANALYSIS

3.1. ATOMIC EMISSION SPECTROSCOPY (AES)

The peak corresponding to lithium could not be detected with the help of EDAX technique, as it has a low atomic number. So atomic emission spectroscopy (AES) technique was used to determine the presence of lithium in lithium tartrate crystals. By using this technique, the presence of lithium (Li) in the sample was confirmed[11].Analytical reports obtained for lithium tartrate crystals via Atomic Emission Spectroscopy is shown in table 3.s

Table 3. Percentage of sample (AES)

Sample	Li (%)
Li	2.95

IV. RESULT AND DISCUSSION

The crystals of lithium tartrate were characterized by XRD analysis. From these diffractogram, 'd' values were computed. From the XRD pattern it is noticed that the peaks obtained. We also found (101), (021),

and (102) lattice planes of lithium tartrate crystals at 2θ of 21.40, 28.81 and 36.51° , respectively. Moreover, apart from the individual lattice planes, we also obtained new peaks at 18.83, 23.11, 26.53, 34.51, 40.78, 48.19, 51.90 and 55.03 degree (2θ) exhibiting the planes (201), (141), (151), (171), (352), (313), (211) and (850), respectively. Calculated (hkl) and 'd' values indicate orthorhombic crystals structure of lithium tartrate crystals and having lattice parameters of a = 6.7942 Å, b = 7.9804 Å and c = 5.2386 Å and volume of unit cell, V = 284.05 $(Å)^3$.

V. CONCLUSION

There has been always a keen and competitive race to grow perfect single crystal with purity and tranceparancy. The good quality single lithium crystals of (3x1x1) and the atomic spectrum limited sample is used to determined it lelemetal composition. AES. The dopant concentration of lithium in the grown crystals is 29.8 ppm. Needle shaped and whitish colour, dentritic crystals were grown

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