

# Nucleation and Growth of Potassium Hydrogen Tartrate Single Crystals

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**ABSTRACT:** We report nucleation and growth of potassium hydrogen tartrate crystals by silica gel technique. Potassium hydrogen tartrate crystals were grown by single diffusion technique in a sodium meta silicate gel medium. The effect of various parameters like pH of gel, gel setting, gel aging, gel density and concentration of reactants and the environmental conditions on the nucleation and growth of these crystals were studied and optimum conditions for superior quality of the crystals were found out. The crystals of pure potassium hydrogen tartrate are of white colour without transparency. Depending on the environmental growth conditions, pure crystals have regular and elongated shape. The grown crystals of potassium hydrogen tartrates are of larger size – 10 mm × 6 mm × 5 mm but less transparent.

**KEYWORDS:** Potassium hydrogen tartrate, silica gel technique, single crystal growth

## I. INTRODUCTION

During last century the technology evolved rapidly and the single crystals have played a vital role in the advancements: starting from the single crystal silicon used in microelectronics to the nonlinear crystals used in lasers and optics. Crystal is a backbone of modern technology and also is receiving increasing importance due to their various applications in solid state technology. Advances in modern solid state technology depends on the availability of good quality crystals defect free crystalline materials. Single crystals are the fundamental building blocks for modern technology. A wide range of crystals can be grown in silica gel medium which prevents turbulence and as the silica gels are chemically inert these can provide a high control on the reagents diffusion rate. The process of growing crystals in gel is simple, inexpensive and the crystals can be grown at ambient temperature. Single crystals of tartrate compounds are one of such class exhibiting optical, electrical, dielectric, piezoelectric, and ferroelectric properties. Because of such versatility the tartrate compound crystals have received a worldwide attention for many years. The applications of the crystals exploiting the ferroelectric [1,2,3], piezoelectric [4] properties have been demonstrated. The optical nonlinearities such as the second harmonic generation have been extensively used for controlling and modifying laser radiation [5].

One of such metal tartrate compounds is Potassium Hydrogen Tartrate (KHT) that has the applications in the food and health industry also. KHT has been used medicinally as a laxative. It is an ingredient of Fehling's solution, formerly used in the determination of reducing sugars in solutions. In organic synthesis, it is used in aqueous workups to break up emulsions, particularly for reactions in which an aluminium-based hydride reagent was used. It is a common precipitant in protein crystallography and is also an ingredient in the Biuret reagent which is used to measure protein concentration. This ingredient maintains cupric ions in solution at an alkaline pH. It has also been used in the process of silvering mirrors.

The crystals of Strontium tartrate tetrahydrate, Potassium hydrogen tartrate, Iron tartrate, Ammonium hydrogen tartrate, Rubidium hydrogen tartrate, and Cadmium tartrate were grown in pure form have already been reported. Many tartrates salts with monovalent cations; such as rubidium hydrogen tartrate [6,7], sodium tartrate [8], and ammonium tartrate [9] and divalent cations; such as calcium tartrate [10], cadmium tartrate [11,12], manganese tartrate [13], zinc tartrate [14] and strontium tartrate [15,16,17] have been grown in pure form and studied for their dielectric and thermal properties.

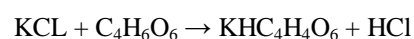
It is well known that the gel method is capable of yielding crystals of substantial size and a very high degree of optical perfection. In the sol gel technique, the major role of the gel is to suppress the nucleation. In fact, the crystals in any one system compete with one another for solute and their ultimate size is directly related to the probability of the nucleation. Nucleation is thus one of the key issues, and the extent to which nucleation can be controlled will ultimately govern the practical utility of the gel technique as a whole. Whether the gel technique actually controls the nucleation process in any particular case depends on the various parameters chosen for that particular system. Therefore, the optimization of the process parameters is a vital requirement to grow superior quality crystals.

Here, we report nucleation and growth of potassium hydrogen tartrate crystals by silica gel technique. A systematic study of the effect of various parameters like pH of gel, gel setting, gel aging, gel density and concentration of reactants and the environmental conditions like temperature and humidity of the surrounding on the growth of these crystals was carried out for which number of experiments were performed in a number of test tubes and over a span of time. The nucleation process i.e. the number of nucleations, and the growth parameters like size, colour (transparency) of the crystals were set measures to determine the optimum conditions.

## II. EXPERIMENTAL PROCEDURE

The borosilicate glass tubes of length 20 cm and diameter 2.5 cm were placed vertically on the stand. The crystals were grown in silica gel filled in number of different borosilicate tubes of length 20 cm and diameter 2.5 cm placed in aluminium stand. Silica gel was prepared by addition of sodium meta-silicate to tartaric acid. To avoid any local ion concentration causing premature local gelling that makes the final solution inhomogeneous, both the solutions are added in each other slowly and gently with continuous stirring. To prevent fast evaporation and contamination of gel, test tubes were sealed with cotton or rubber caps. The tubes were kept in stand for gel setting depending on pH of solution and environmental conditions. In summer the gel setting time is comparatively smaller than winter. The rainy season is not so good for formation and growth of crystals while summer and month of October is the best season for formation and growth of crystals.

After confirmation of gel setting, an aqueous solution of potassium chloride of particular molarity was poured over the gel surface along the walls of the test tube gently and carefully so that gel does not break. The  $K^{1+}$  ion diffuse through the pours of the gel forming the lower reactant due to the reaction between ions of  $K^{1+}$  and  $HC_4H_4O_6^-$  in gel. The following action takes place leading to the formation of pure potassium hydrogen tartrate crystal.



## III. RESULTS AND DISCUSSION

After filling the tubes for gel formation and after that by addition of proper solution to gel, tiny, small, silver white dull colour crystals of potassium hydrogen tartrate began to appear after 24 hours. In 8-10 days the crystals increased in size and numbers. Crystals in the middle region of the gel medium are well developed in size in 2-3 weeks and are perfect in nature. Complete growth of crystal requires 1-2 months. The effect of various parameters like pH of gel, gel setting, gel aging, gel density and concentration of reactants on the growth of these crystals were studied.

One of the most effective methods of nucleation control (and perhaps the most important) is the adjustment of pH which increases the solubility of nuclei and thus diminishes the probability of their large-scale survival. In our gel system increasing the pH in the range we studied caused the reduction in the perfection of the crystals.

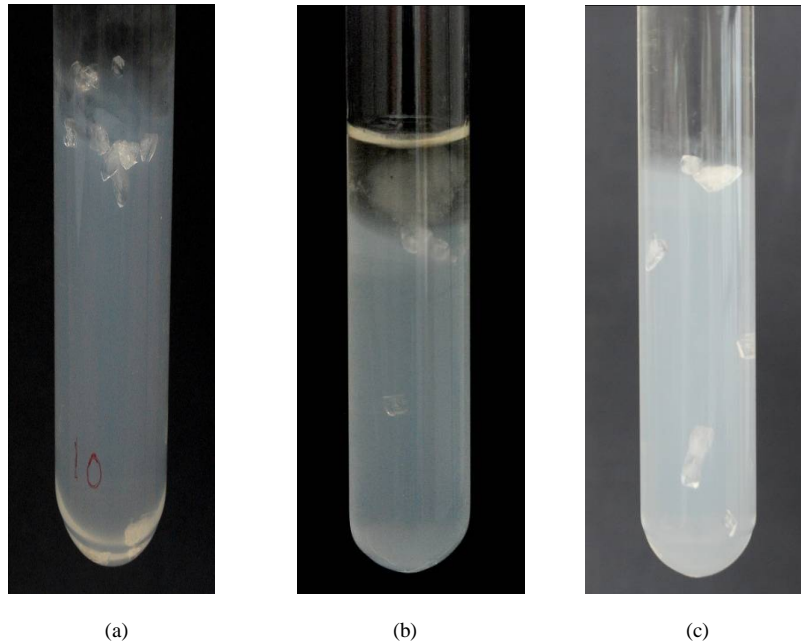


Fig 1 Crystals of Potassium Hydrogen Tartrate grown in the silica gel at various pH (a) pH =4; (b) pH = 4.2; (c)pH = 4.4

Environmental conditions	Temperature	Gel setting time(days)	Gel ageing time	Nature of crystal
Summer	35-40 °C	1	72 hrs	Good
Winter	10-15 °C	14	75 hrs	Satisfactory
Rainy Season	18-20 °C	More than 20	80-90 hrs	Not so satisfactory

**Table 1. Effect of the environmental conditions**

Test tube number	Concentration of reactant above gel	Nature of crystals formed 1
1	0.2	Few crystals of small size and not well develop and defined
2	0.4	Few crystals of increased size and well develop
3	0.6	Sufficient number of isolated crystals of increased size and well develop, various shaped
4	0.8	Large number of isolated transparent crystals of good size and well develop
5	1.0	Large number of isolated transparent crystals of large size and well develop

**Table 2. Effect of Concentration of reactant above gel**



(a)



(b)

Fig 2 Normal and elongated shaped crystals of Potassium Hydrogen Tartrate on graph paper (a) Normal shaped b) Elongated shaped

The environmental factors like ambient temperature, humidity also affect the nucleation and the growth process. Table 1 shows the details of the range of these parameters over which we studied the crystal growth process. In summer the gel setting time is comparatively smaller than winter. The rainy season is not so good for formation and growth of crystals while summer and month of October is the best season for formation and growth of crystals.

As the concentration of reactant above gel is increased the number of the nucleations, the crystal size and quality of the crystals improves. The gel aging reduces the cell size and consequently the rate of diffusion of ions into the gel. Therefore, as the aging time of the gel increased the number of the crystals as well as their size and quality was degraded. Table 2 shows the detailed analysis of the results obtained.

Optimum conditions are obtained by performing a number of experiments in a number of tubes to form well developed, large size crystals of potassium hydrogen tartrate. The crystals of pure potassium hydrogen tartrate are some times of regular shaped as in Fig. 2(a) or elongated shaped as in the Fig. 2 (b). This is due to the variations in concentration of reactant and environmental conditions. Crystals of potassium hydrogen tartrate were found to be less perfect i.e. were dull whitish silver in colour without any transparency compared to due to higher reactive nature. The grown crystals of potassium hydrogen tartrates are of larger size – 10 mm × 6 mm × 5 mm but less transparent. This is due to the higher reactivity of  $K^{1+}$  ions. Perfection of crystal depends on rate of reaction between metal ions and tartrate ions. Slow rate of reaction leads to greater perfection. 60% crystals formed of potassium hydrogen tartrate are of big size.

#### IV. CONCLUSIONS

Here, we report nucleation and growth of potassium hydrogen tartrate by silica gel technique. The effect of various parameters like pH of gel, gel setting, gel aging, gel density and concentration of reactants on the growth of these crystals were studied. From our experiments we conclude that in optimum conditions good quality of pure potassium hydrogen tartrate crystals grow as single crystals in silica gel medium. Optimum conditions for growing the crystals of larger size – 10 mm × 6 mm × 5 mm and superior quality of crystals are: Gel pH = 4; Gel age = 75 h; Gel concentration = 0.5 M; Upper reactant concentration = 1 M; Lower reactant concentration = 0.5 M. The crystals of potassium hydrogen tartrate are of white colour without transparency. Depending on the environmental growth conditions, crystals have regular and elongated shape.

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