CHAPTER - 4

CHARACTERIZATION OF GEL GRWON KDP CRYSTALS BY PHYSICAL AND CHEMICAL METHODS

4.1 INTRODUCTION

It is well known that crystals are grown for two main reasons: to understand how crystals grow and for their utility-scientific or technological-of the grown crystals. For either of these purpose, good quality crystals must be grown. Either to understand how growth processes influence perfection or to produce crystals of the perfection required for the end use, one must determine the nature and number of imperfections in the grown crystals. Thus the crystal grower is or should be intimately involved with the assessment of his grown crystals. The assessment of the physical and chemical perfection of materials is called characterization. A crystal is fully characterized when we known the identity, concentration and position of all its constituent ion Thus no crystal has ever been fully characterized; however many crystals have been characterized to a point where relationship between their properties and the location and concentration of their constituent ions could be made. The preceeding chapter (chapter-3) described the successful growth of KDP and KDP : Ni, KDP : Co crystals by changing some of the growth parameters. The present chapter describes the characterization of KPD crystals by physical and chemical methods.

4.2 EXPERIMENTAL

In this section experiment procedures for X-ray diffraction analysis and chemical analysis will be presented.

4.2.1 X-RAY DIFFRACTION ANALYSIS:

The single crystals of doped potassium dihydrogen phosphate grown in silica gel were subjected to X-ray analysis for identification.

The size of the crystals selected for X-ray analysis was about 5x3x4 mm^3. A beam of white radiation, the continous spectrum from an X-ray tube, was allowed to fall on the KDP single crystal, which was mounted on a goniometer. Since heavy metal target was needed, copper target was selected. Each crystal was oriented alongwith the crystallographic axis.

Nonicus CAD-4F-11M single crystal X-ray diffractometer was used for the analysis. The X-ray analysis involved the transmission Laue method. The Laue photographic films for doped KDP crystals were obtained as shown in fig. 8.1, 8.2, 8.3. These films were developed.

4.2.2 CHEMICAL ANALYSIS:

The presence of impurity was conformed by chemical analysis. For this analysis atomic absorption spectroscopy method was used.

Atomic absorption spectroscopy was proved itself to be the most powerful instrumental technique for the quantitative determination of trace metals in liquids. This method provides total metal content of the sample. By this technique, the determination can be made in the presence of many other elements.

The lamp was used, the light of which was able to emit the spectral lines corresponding to the energy required for an electronic transition from the ground state to an excited state. The light

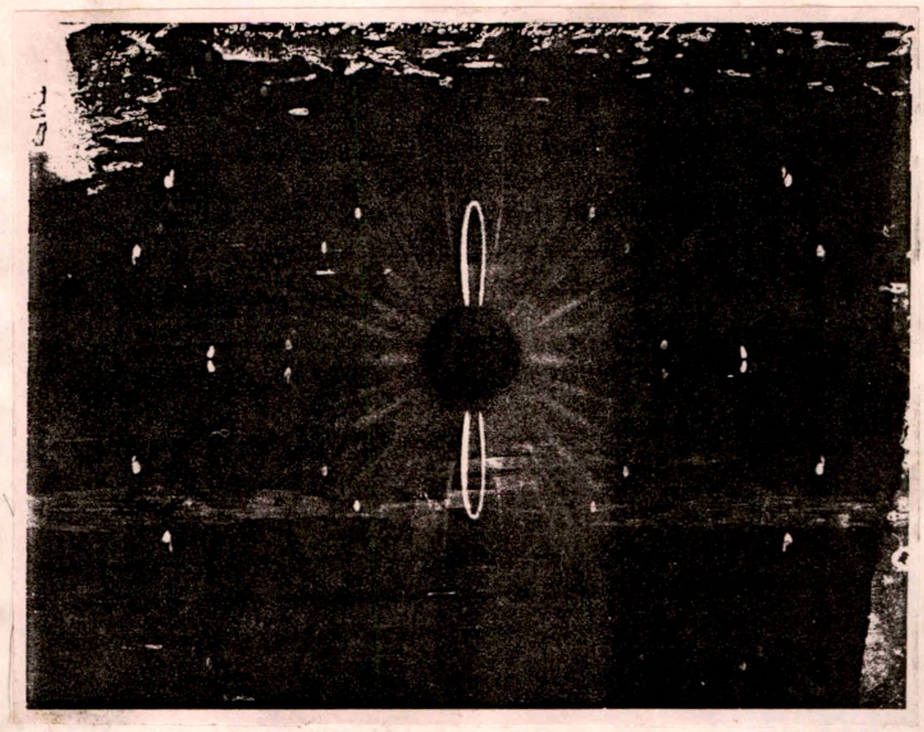

Fig. 8·1

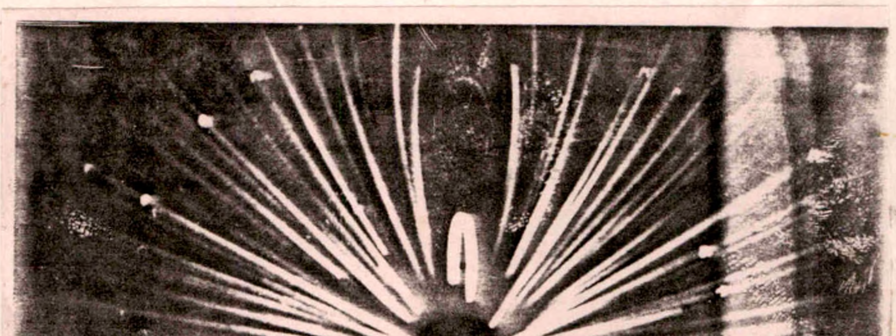

Fig. 8·2

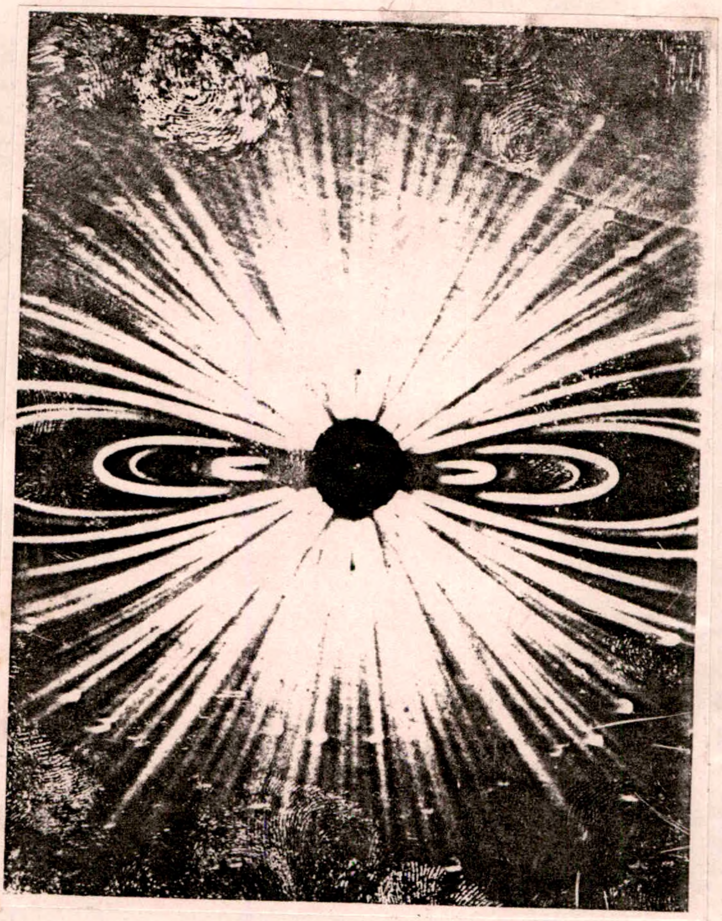

Fig. 8·3

was allowed to pass through the flame. Meanwhile the sample solution was aspirated into the flame. Before entering the flame, the solution got dispersed into a mist of very small droplets which evaporated in the flame to give first the dry salt, and then the vapour of the salt. Part of the vapour was dissociated into atoms of the element to be measured. Thus, the flame was found to be possessing free unexcited atoms which were capable of absorbing radiation from external source when the radiation corresponds exactly to the energy required for a transition element from the ground electronic state to upper excited state. Then the unabsorbed radiation from the flame was allowed to pass through a monochromator which isolated the exciting spectral lines of light source, from the monochromator the unabsorbed radiation was led into the detector which was then registered by a detector, the output of which was amplified and measured on a recorder. Absorption was measured by the difference in transmitted signal in the presence and asbsence of test element.

4.3 RESULT AND DISCUSSION:-

In this section results on X-ray analysis and chemical analysis will be presented and discussed.

4.3.1 RESULT ON X-RAY ANALYSIS:

The Laue diffraction pattern contained sharp spots, which indicate single crystallanity of the doped KDP crystals.

According to the obtained computerized data, the lattice parameters for the potassium dihydrogen phosphate crystals doped with cobalt nitrate as an impurity are given in the following table 4.1.

TABLE NO. 4.1

Concentration of the impurity by weight percent	Lattice parameters obtained in A^{O}.		
	$a(A^{O})$	$b(A^{O})$	$c(A^{O})$
0.3%	7.4494	10.1971	6.9693
0.5%	7.4407	10.1841	6.9563
1.0%	7.4403	10.1979	6.9663

Lattice parameters indicate that the unit cell for these doped KDP crystals is monoclinic.

The unit cell parameters for pure KDP cyrstal in the monoclinic phase (II), according to ASTM data are given in table 4.2.

TABLE NO. 4.2

a (A^{O})	b (A^{O})	c (A^{O})
7.47	14.49	7.43

(unit cell parameters in monoclinic phase)

The pattern confirmed to the pattern in the ASTM file indicating the crystals are potassium dihydrogen phosphate (KDP) crystals.

Some discripancy in the Lattice parameters b and c may be due to presence of impurities.

4.3.2 RESULT ON CHEMICAL ANALYSIS:

Using atomic absorption spectroscopy for chemical analysis of the doped samples, the percentage of impurities present in the host lattice was obtained.

The percentage of impurity actually doped and the percentage of impurity observed by the chemical analysis are given in table 4.3.

TABLE NO. 4.3

Type of impurity added to the pure KDP crystal	The percentage of impurity actually added as a dopant (by wt.)	percentage of impurity metal ion detected by chemical analysis (by wt.)
Cobalt nitrate	0.3%	0.003%
Cobalt nitrate	0.5%	0.0064%
Cobalt nitrate	1.0%	0.022%
Nickel Chloride	8%	0.038%
Nickel Chloride	10%	0.15%

From the atomic absorption spectroscopy, it has been found that only a fraction of added impurity entered into the crystal lattice. This may be due to the fact that during crystallization process, the impurity atoms are generally rejected by the solute of the material to be grown.

4.4 CONCLUSION:

The single crystallanity of the grown crystals was conformed with the help of transmission laue diffraction photographs.

From the X-ray diffraction pattern lattice parameters were obtained, which approximately agreed with the value reported in the literature.

The silica gel grown crystals exhibited monoclinic habit.

Chemical analysis showed that impurities were present in the crystals and only a very small fraction of impurity actually found to be entered in host crystal lattice.

From laue photographs, it was observed that as the concentration of impurity increased, sharpness of the laue spots found to be decreasing.