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Growth and structural studies of calcium doped triglycine sulphate (TGS) single crystals

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ABSTRACT

Pure and Calcium doped Triglycine Sulphate (TGS) salts were synthesized and single crystals of the salts were grown from aqueous solutions by slow evaporation technique. The structural studies on the grown crystals were carried out by powder XRD analysis and found that the grown crystal crystallize in monoclinic structure. The FTIR spectra have been recorded to identify the functional groups present.

Keywords: TGS; Crystal growth; Doping; Rare earth; XRD; FTIR

INTRODUCTION

Triglycine Sulphate (TGS) belongs to the biaxial monoclinic system associated with the polar space group $P2_1$ in the ferroelectric phase and it crystallizes in $P2_1/m$ space group in the paraelectric phase . The monoclinic b-axis is known to be parallel to spontaneous polarization direction. Therefore the b-cut / (010) crystals are technologically important for many device applications [1-3]. Pure TGS crystal have a tendency to polarization reversal, in order to overcome this difficulty dopant has been added. Even though many study have been performed with different amino acid doping [4-6] and metal ion doping [7-9]. There are only very few report on rare-earth doped TGS crystals [10,11].

Doping of heavy rare-earth ions like Ho,Tm and Yb creates structural and chemical defects in the crystal and also defects density depends on the electronic configuration of the rare-earth ions [10]. In the case of light rare-earth ion doping, the crystal structure remains unaltered [11]. Crystals of triglycine sulphate (TGS), a well known ferroelectric material, find wide application as room temperature IR detectors [12]. In this present work systematic study on the Growth and structural Characterization of TGS crystals doped with the light rare earth ion Calcium**.**

MATERIAL AND METHODS

Growth Procedure

TGS salts where synthesized from the following reaction

$3(NH_2CH_2COOH)+H_2SO_4 \rightarrow (NH_2CH_2COOH)_3(H_2SO_4)$

AnalaR Grade Glycine and Sulphuric acid were used for the synthesize of TGS salt, after successive recrystalization processes the purified salt were used for the preparation of the solution. Calcium carbonate was added in the ratio viz. 1:0.000 , 1:0.002, 1:0.004 , 1:0.006 , 1:0.008 and 1:0.010 to the TGS solution and saturated at 45°C Growth was initiated by a temperature reduction of the solution and slow cooling was employed. After a few days of growth the crystals were harvested after a typical growth period of 10 days.

The Grown crystals were characterized by taking powder X-ray diffraction data using PANalytical diffractometer with copper K-Alpha (1.5406A°) radiation. The calcium presence in the impurity added crystals were confirmed by carried out Atomic Absorption Spectroscopic study.

FTIR spectra were recorded by using 'SHIMADZU' FTIR 8400S model spectrometer through KBr method.

RESULTS AND DISCUSSION

Growth

Figure.1 shows the grown crystals in the present study. It is found that the grown crystals are transparent and optically good quality crystals.

FTIR (Fourier transform infrared spectroscopy analysis)

The NH , OH and CH absorption are occur at high frequency 3164cm^{-1}. The frequency at 1600cm^{-1} is assigned for NH_2 bending. The absorption in the range 1700-1865cm^{-1} is assigned to C=O stretching of carboxylic acid.

The strong absorption in the range 1020 - 1128cm^{-1} is evidently due to the sulphate part of the molecule. The broad band around 1100cm^{-1} is assigned to C-N asymmetric stretch of SO_4. The peak absorption at 615cm^{-1} and 501 cm^{-1} are due to NH_3 oscillation . Since the functional group of pure and doped TGS crystals are identical. The FTIR spectra of pure and 1:0.002 calcium doped TGS crystals are given in Fig.2 and Fig.3 respectively as an illustration.

Lattice Parameter

Analysis of X-ray diffraction data shows that the grown crystals are monoclinic in structure. The lattice parameter thus calculated are given in table.1 along with Calcium concentration. It is found that the lattice parameter observed in the present study coincide with the reported [13] value given in the bracket. The addition of dopant has no influence on the lattice parameter. The XRD pattern of pure TGS and 1:0.002 calcium doped crystals are given in the Fig.4 and Fig.5 respectively as an illustration

Table.1 Values of lattice parameter along with calcium concentration

System	Calcium concentration (ppm)	Lattice Parameter			
		a (nm)	b (nm)	c (nm)	β
TGS	-	9.5487 [9.38]	12.6524 [12.634]	5.392 [5.734]	110°36´ [109°55´]
1:0.002	290.184	9.5247	12.6477	5.2726	110°38´
1:0.004	304.839	9.5115	12.797	5.4378	110°46´
1:0.006	439.842	9.5545	12.6171	5.3496	110°46´
1:0.008	614.827	9.499	12.5554	5.3643	110°46´
1:0.010	1802.242	9.624	12.5517	5.444	110°46´

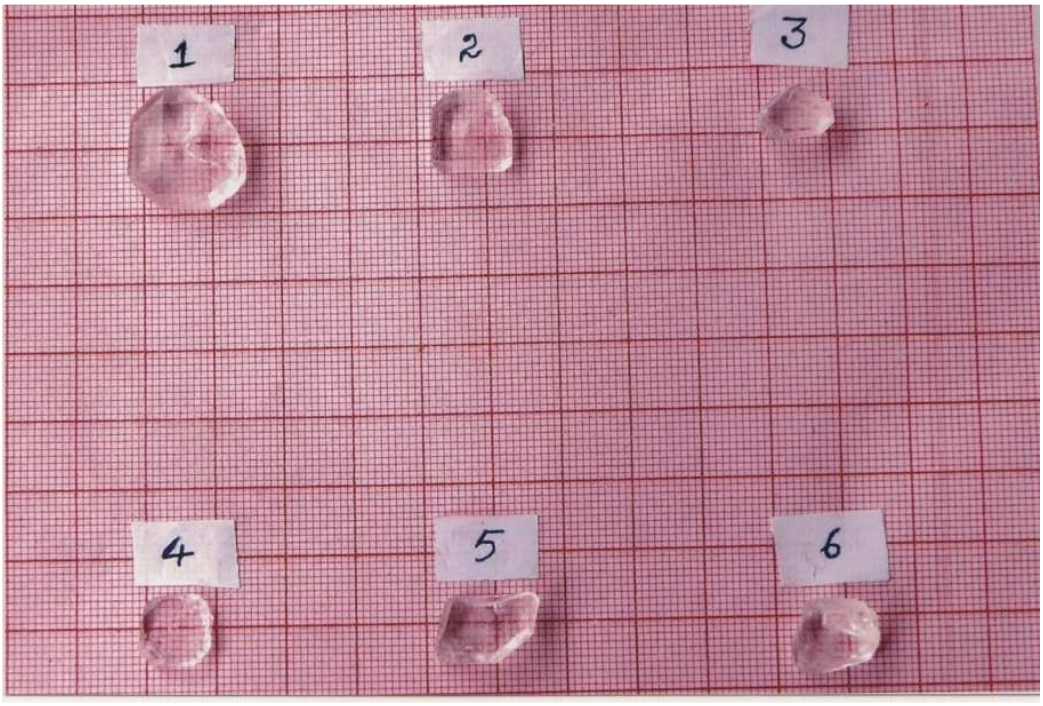

Figure.1 Photograph of grown crystals
1 - Pure TGS 2 - 1:0.002 3 - 1:0.004 4 - 1:0.006 5 - 1:0.008 6 - 1:0.010

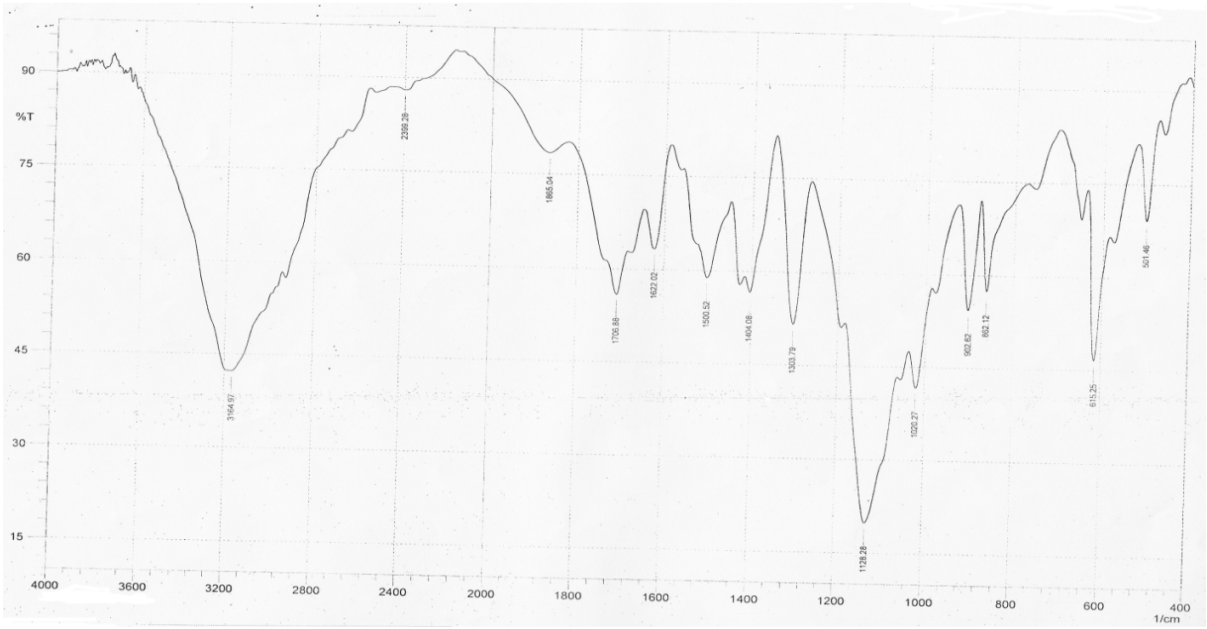

Figure.2 FTIR Spectrum for Pure TGS

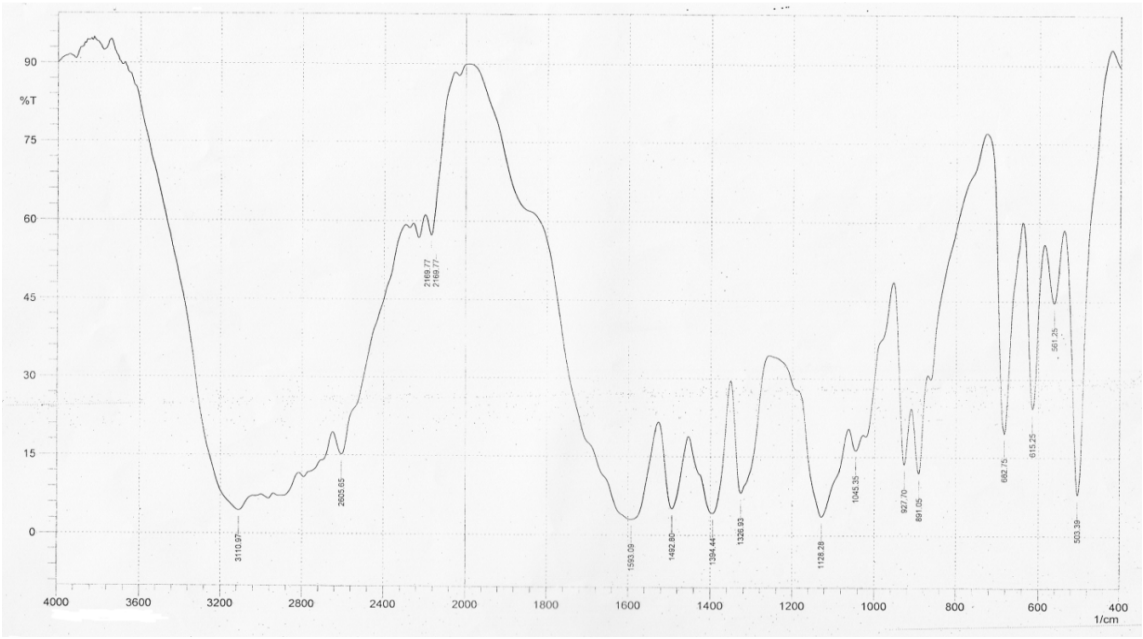

Figure.3 FTIR Spectrum for 1:0.002 Ca doped TGS

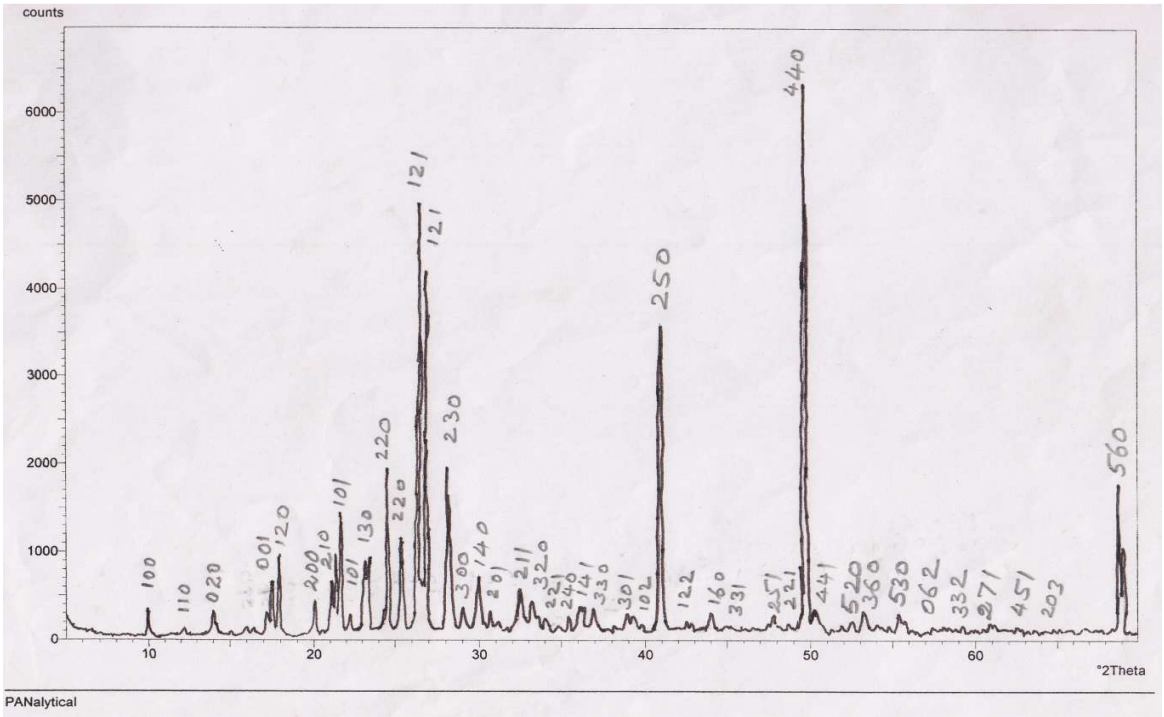

Figure.4 XRD pattern of Pure TGS

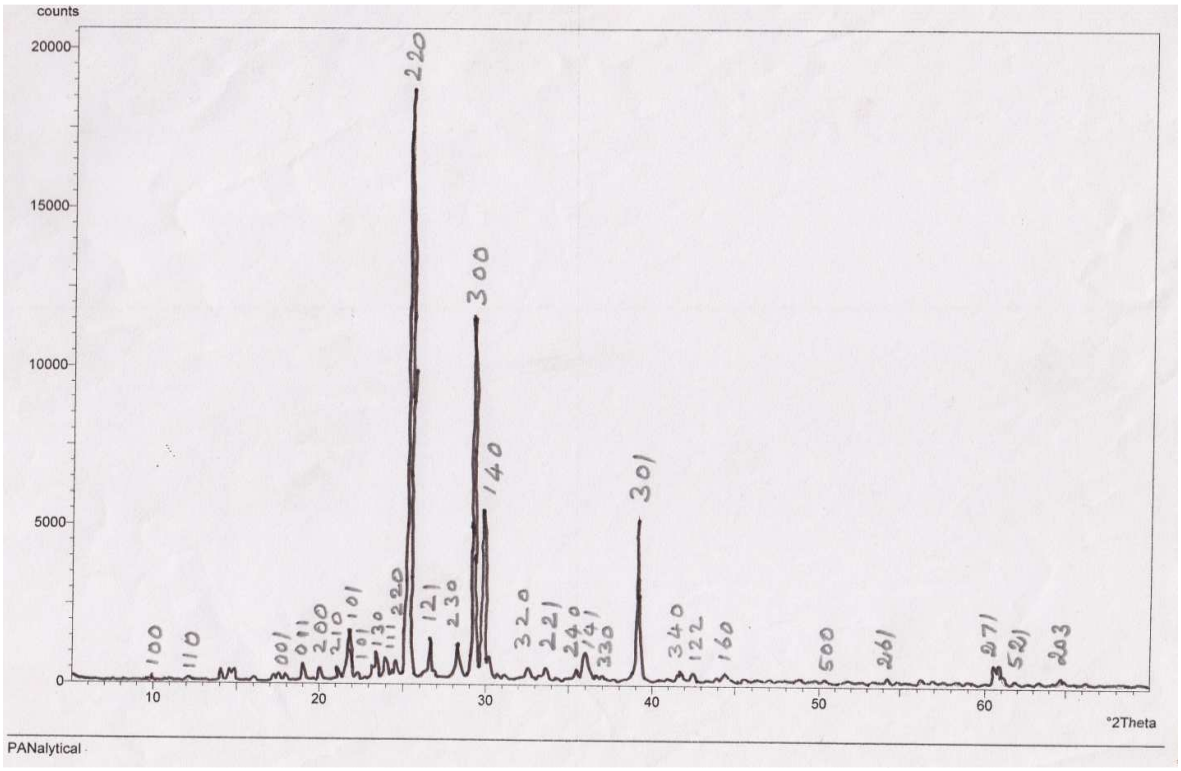

Figure.5 XRD pattern of 1:0.002 Ca doped TGS

CONCLUSION

Pure and Calcium doped TGS Crystals grown in the present study are good quality ,transparent crystals. The XRD data shows the grown crystals are monoclinic in structure . The FTIR spectra confirm all the functional groups present in the grown crystals.

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