Growth And Characterization Of Sulphanillic Acid Single Crystal From Vapor

Dr. Rita A. Gharde**, Divakar T. Chunarkar*

Abstract: - There have been many contributors to the art of growing single crystals and varied ingenious techniques have been applied to the problem. Despite the substantial advance in the theory of crystal growth, in practice the growth of a large single crystal must still be approached from the empirical viewpoint. Crystal growth is seldom susceptible to observation during growth. This generally means that the proper growth conditions must be determine by trial, error and once determined means must be available for reproducing these optimum conditions whenever desired. The system must be stable since there is seldom any convenient away for the operator to observe the growing process and compensate for deviation from the ideal growth conditions. The shape of the grown crystal is a result of contributions from lattice force, surface tension, pull rate and temperature gradients in the crystal. The shape of the cross section perpendicular to the growth direction is determined by a balance between lattice force which tends to form equilibrium faces and surface tension which tends to make the cross section circular. The average diameter of the crystal is determined by the rate of pull and thermal gradient in the crystal. Growth from vapor has many experimental advantages to recommend it. If a suitable solvent can be found, crystal growing can be performed at temperatures well below the melting point of the crystal often at room temperature. These low temperature place much less demand on furnaces and power supplies. In addition the reactivity of the solute may be greatly moderate not only by the low temperature but also by the dilution with the solvent. The three techniques usually employed are change in temperature, evaporation of solvent, and addition of solute. The difficulty of controlling the temperature gradients in solution also means that a large volume of solution surrounding the crystal will be supersaturated and labile with respect to growth. The great caution is required in assuring that no spurious nuclei are present. In all subsequent show our growth the main features and study on crystal of this materials.

Keyword: - Growth from vapor, FTIR, X Ray diffraction, DSC etc.

Introduction:-

Sulfanillic acid ($NH_3^+C_6H_4SO_3^-$) is an important and interesting compound , which find a number of application including non linear optics (2).Crystallization is an important process because of the number of martial can be marketed in the form of crystals. Its wide use is probably due to the high purified and attractive form of a chemical solid, which can be obtained from relatively impure solution in a single processing step. The growth and size of crystal depends on the condition of its formation. Temperature, pressure, presence of impurities etc. will affect the size perfection of crystal. In the recent year, several study dealing with organic, inorganic and semi organic molecule and material for non linear optics reported, due to the increasing need for cheap and easily process able materials. Crystal take verity of shape, depending on the internal factor. Both internal and external factors influence of the growth rate, and there they modified the crystal morphology. Crystal grown from conventional solution growth method was used as a seed. The growth of the face depends on the external factors such as temperature and pressure. Because of the temperature difference, concentration of solution increases. On linear optical single crystal are used in the area of fiber optics, communication, optical frequency conversion, optical data storage etc. In crystal growth technique, the recently discovered uniaxially solution crystallization method of Shankar Narayan-Ramsamy (S.R.) is a suitable method.

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To effectively control the orientation of molecule during bulk crystal growth from solution at room temperature with 100% solute-crystal conversion efficiency. In the present investigation sulphanillic acid single crystal grown by three distinct methods are as follows.

1) Growth from solution
2) Growth from melt
3) Growth from vapor

Experimental set up:-

It consist of growth ampoule made out of glass with seed mounting pad .An outer glass shield tube protect and hold the inner growth ampoule. A ring heater positioned at the top of the growth ampoule was connected to the temperature controller and it provides the necessary temperature for solvent evaporation. The temperature around the growth ampoule was selected based on the solvent used and was controlled with the aid of the temperature controller depending on the growth rate of crystal, The ring heater was moved during a translation mechanism.

Fig.1. Sulfanillic Acid crystal

Characterization:-

Result and discussions:-

X-Ray diffraction:-The powder x-ray diffraction pattern was recorded for the grown sulfanilic acid using a cuka. The single crystal XRD reveals that the sulfanillic acid crystal belongs to mono hydrates flat crystal. The obtained cell parameters are in good agreement with earlier repot. The different faces (planes) of the seed crystals were identified from single crystal XRD and the morphology has been drawn as shown in fig. 1).The morphology of sulfanillic acid single crystal is not yet reported. The powder XRD pattern of sulfanillic acid is shown in (fig.2).We have collected X-ray diffraction data of the single crystal using Enraf- Nnius CAD-4 diffractometer, graphite monochromated CuKa radiation. The powder of the grown crystals by analyzed by powder X- Ray diffraction using richseifert diffraction with Cuka radiation (wave length=1.5406) and scan rate of 0.30mm. X-ray studies are carried out at room temperature.The calculated (h l k) planes satisfy the general reflection condition of space group observed from the structure determination of the crystal. It is display an anionic part and a cationic part indicative of the zwitter ionic structure. Sulfanilic acid crystallizes in the orthorhombic structure with space group of as shown in Table 1

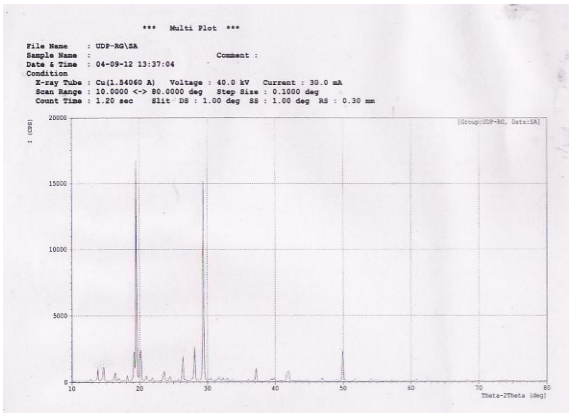

Fig.2.XRD Analysis for S. A.

# Strongest 3 peaks							
no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	5	19.4910	4.55066	100	0.25920	7926	22842
2	10	29.3904	3.03654	52	0.26430	4101	11539
3	9	28.0932	3.17373	17	0.26130	1323	3904

# Peak Data List						
peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	13.7964	6.41352	5	0.25300	413	1103
2	14.6344	6.04810	7	0.27710	528	1665
3	16.3358	5.42181	4	0.28380	302	1053
4	19.0000	4.66714	4	0.16020	322	498
5	19.4910	4.55066	100	0.25920	7926	22842
6	20.1345	4.40665	15	0.27980	1177	3586
7	23.5956	3.76751	5	0.31220	433	1972
8	26.3000	3.38592	9	0.15600	704	1030
9	28.0932	3.17373	17	0.26130	1323	3904
10	29.3904	3.03654	52	0.26430	4101	11539
11	37.1466	2.41839	6	0.26900	480	1458
12	41.8331	2.15766	6	0.44300	470	2298
13	49.8984	1.82616	16	0.24520	1244	3302

Table 1 Data for XRD

FTIR:-

The recorded FTIR spectra as shown by fig. compare with the previous spectra of sulfanillic acid in FTIR analysis by SHIMADZU IR Prestige 21 model. Graph is very sharp contain the range 400 cm $^{-1}$ to 4000 cm^{-1} on X- axis and Y-axis 40 ^{O}C TO 100 ^{O}C respectively. Intensity of the sulphanillic acid decreases to increase the area of sample contains. The infrared spectroscopy is an important technique to investigate the functional group of the crystals. When irradiated with infrared light the molecule in the crystal absorbs energy and begins to vibrates in different form with respect to the different chemical bonds. Present among the atom as a result are vibration spectrums is obtained it gives information about certain group of atoms and functional group such as SO2, SO3 present in sample. In the present study the FTIR spectra of pure material the peaks is wave number relative intensities and the peak assignments of the grown crystals as shown in fig.3. and Table2.

	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	487.99	79.78911	6.555784	557.43	470.63	6.038936	1.162588
2	599.86	81.58934	6.906239	607.58	586.36	1.451736	0.321299
3	623.01	84.64072	4.458698	632.65	615.29	1.026049	0.156183
4	696.3	83.73912	4.490018	704.02	669.3	1.765771	0.085928
5	719.45	73.65411	6.934614	727.16	704.02	2.22506	0.242074
6	761.88	67.73561	17.74447	783.1	750.31	3.234649	1.014133
7	800.46	67.38643	14.07646	808.17	783.1	2.735882	0.656671
8	823.6	68.02384	6.881262	829.39	808.17	2.929421	0.434572
9	869.9	67.05725	9.025543	877.61	860.25	2.384132	0.349399
10	921.97	69.03999	6.780652	931.62	900.76	4.109856	0.49987
11	960.55	56.60649	8.525677	968.27	945.12	4.781087	0.69042
12	1004.91	49.94351	7.604927	1020.34	970.19	13.35361	1.887198
13	1028.06	52.55967	4.531073	1049.28	1022.27	6.256442	0.259512
14	1083.99	65.69842	3.827162	1095.57	1074.35	3.556603	0.217813
15	1134.14	58.72343	1.515662	1138	1126.43	2.568014	0.077764
16	1172.72	43.61282	17.92969	1207.44	1147.65	16.52691	3.942968
17	1215.15	59.12207	3.557628	1226.73	1209.37	3.713751	0.225835
18	1253.73	59.04431	7.026321	1278.81	1242.16	6.802982	0.474268
19	1323.17	63.58437	7.216386	1340.53	1315.45	4.09815	0.521072
20	1365.6	59.89608	3.168012	1367.53	1346.31	3.466411	0.120888
21	1375.25	57.12796	8.51692	1404.18	1369.46	6.032979	0.937469
22	1415.75	72.98509	5.280875	1421.54	1404.18	1.972062	0.193219
23	1454.33	57.95233	1.364942	1456.26	1436.97	4.081122	0.059571
24	1463.97	52.59903	9.506414	1496.76	1458.18	6.318545	0.738556
25	1668.43	78.71788	3.870936	1676.14	1653	2.101025	0.263622
26	1735.93	43.70193	38.66240	1772.58	1699.29	12.18431	5.978387
27	2943.37	34.33023	13.49719	3028.24	2914.44	36.67965	5.674339

Table 2 Data for FTIR

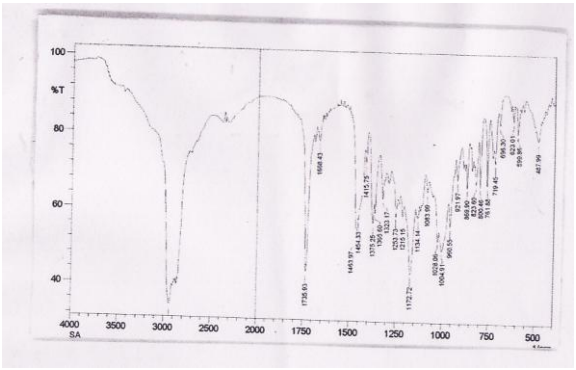

Fig. 3 FTIR Analysis for S. A.

(DSC) Differential scanning calorimetric:-

In this analysis the energy necessary to establish a zero temperature difference between the sample and a reference material is measure as a function of temperature or time when an endothermic transition occur. The energy absorbed by the sample is compensated by an increased energy input to the sample in order to maintain a zero temperature difference on a DSC chart recording the abscissa indicates the transition temperature and the peak area measure the total energy transfer to form the sample [1]. These methods are used to study the number of transition range of polymorphous. Each polymorphic transition causes an energy change that is detected by

DSC. Various forms of dioxins have been investigated. In this sample the rage of temp 100 ^{0}C to 400 ^{0}C which stat forms 35^{0}C and end in 400 ^{0}C. DSC curves used to determine the purity of drug sample as shown in fig.4. From DSC plot we observed peak at 55.84 ^{o}c, 74.40 ^{o}c, 123.09 ^{o}c. The melting point of sulphanillic acid is 173 ^{o} c. Cooling curve is straight line and lightly curve in both end and heating high curve in both side form 35^{0}C to 300^{0}C. In this analysis nitrogen gas are used for cooling of atmosphere.

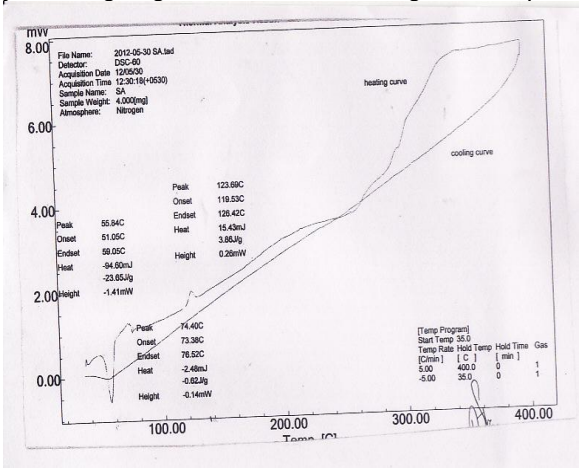

Fig. 4 for DSC Analysis

Dielectric constant:-

The single crystals of sulfanillic acid crushed in to powder form there are no any shape and size. Such type of crushed material filled in the assembly of dielectric constant instrument and found the capacitance with the help of multi meter. The increasing capacitance depends on the properties of medium called dielectric constant. The maximum dielectric constant response revealed may be of great interest in application. The dielectric constant of sulfanillic acid shows 4.23 at normal temperature. Dielectric materials are used in detecting nitrites, antibacterial and directed dyes, food color reactive dyes and metal complex dyes.

Surface Tension:-

Number of method for the measurement of surface tension. But in this sample use the capillary rise method. The contact angle between the glass and liquid material. The liquid will rise almost instantaneously up to a certain height depending up on a surface tension and density. The column of the liquid in the capillary is evidently being supported by some force acting along the surface of the material of liquid. In this sample the surface tension of materials found 2.172 d/cm.

Conclusion:-

From the above analysis, we are able to draw the conclusion that the relative size of given crystal was grown by S.R. Method. The effectiveness of this method was shown by the growth of large size sulfanillic acid single crystals. The single crystal XRD shows that the sulfanillic acid belongs to orthorhombic crystals. The FTIR studies revealed the presence of functional groups. Dielectric values and surface tension values are quite good.

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Reference:-

1) Crystal Research Technology xx (2006) xxx-xxx M. lenin, N. Balamurugan,P. Ramasamy.

2) Principesl of Physical Chemistry, Puri, Sharma Pathania, Panjab University Chandigarth.

3) P. C. Jain, Monika Jain ,Farmly faculty member of Delhi college of Engg. Delhi.

4) IJAMTES Volume 1,Issue 10 July 2012 ISSN 2249-7455

5) Rita A. Gharde, Divakar T. Chunarkar Dept. of Physics Mumbai University Mumbai -98.

6) J.C .Brice Crystal Growth Process ,Wiley, New York 1986.

7) K.ShankarNarayanan, J.Crystal Growth 284 (2005)203 Anna University Chennai.

8) G. Bhagavannaarayana, N.Vijayan, Material Charectrization Division, Dr. K. S. Krishnan Marg New Dehli.

9) R. Ramesh Babu N. Vijayan,R. Gopalakrishan and P. Ramasamy, Crystal Growth Centre , Anna University Chennai.

10) B. W. Betterman and H. Cole, Rev.Mod. Physics ,36,681.

11) Rita A. Gharde,DivakarT. Chunarkar IJSRP ISSN 2250-3153 June 2012,vol. 2 issue 6.