GROWTH AND CHARACTERIZATION OF PURE THIOUREA DOPED WITH L- VALINE CRYSTAL

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ABSTRACT

Poly crystals of pure thiourea doped with L-valine an organic material: have been grown by slow evaporation technique at room temperature .The crystalline nature of grown crystal was confirmed by power x ray diffraction analysis (XRD).The functional group of the grown crystals was found by FTIR analysis. The spectral bands have been compared with similar thiourea complexes using FTIR spectrum in the range 1000- 3500 cm^{-1} .The UV- Vis study was performed to know optical behaviour of the grown crystals.

I.INTRODUCTION

The search for new organic nonlinear optical crystals has been of great interest because of their wide range of potential applications such as frequency doubling, optical switching, optical disk data storage, optical modulation, laser remote sensing and medical diagnostics. These materials attracting significant attention as they posses large nonlinear optical susceptibilities, ultrafast nonlinear response time, high laser damage threshold and scope for introducing desirable characteristics by multifunctional substitutions [1-3]. For NLO device applications still there is a demand for good quality crystals with higher efficiency. For the past ten years amino acids are playing a vital role in the nonlinear optical crystal growth. They individually exhibit nonlinear properties as they posses proton donor carboxyl acid (COO^-) and proton acceptor amino (NH_3) group also in solid state they exist as zwitterions. Amino acid crystals play a major role in NLO applications such as L-valine, L-alanine,L-proline, L-leucine, L-histidine, L-arginine and L-phenylalanine. Worldwide urea and its derivatives are extensively used in crystal engineering and supramolecular chemistry for their flexibility in the synthesis of functional materials. Also for a long time it is used as a model system for crystals from solution growth and reference material in Powder SHG .

In the present work urea and L-valine single crystals were grown by slow evaporation solution growth technique. The grown crystal was characterized by various characterization techniques such as CHN analysis; powder XRD, FT-IR,UV- Vis analysis.

II . METHODS

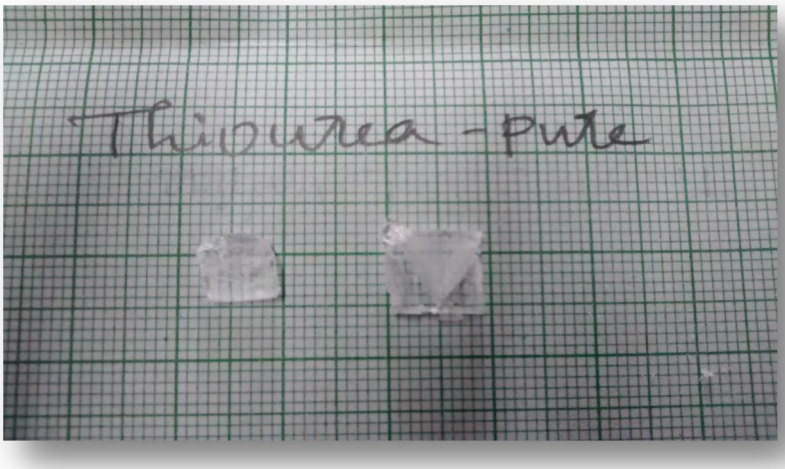
(1) Slow Evaporation Method:

In this method, an excess of a given solute is established by utilizing the difference between the rates of evaporation of the system remains constant in the solvent

evaporation method. The solution loses particles, which are weakly bound to other components and therefore the volume of the solution decreases. In almost all cases, the vapour pressure of the solute and therefore the solvent evaporates more rapidly and the solution becomes supersaturated usually it is sufficient to allow the vapour format above the solution to escapes freely into the atmosphere. This is the oldest method of crystal grown by 15 days and technologically. It is very simple typical growth conditions involve temperature stabilization to about 0.005^0C and rates of evaporation of a few mm^3/hr.

5. GROWTH OF PURE THIOUREA CRYSTAL:

The 10ml double distilled water is taken in the 50ml beaker. The addition of 0.8909g of Thiourea with constant stirring by stirrer. After one hour stirrer by the solution get saturated. Then solution is filtered by filter paper. Here the work is done in room temperature itself. This saturated solution is taken in a 50ml beaker and the beaker is closed with the Aluminium foil sheet. Some tiny holes are made on the foil sheet. So the creation of a few small seed crystals at bottom of beaker containing the mother solution within 15 days. The grown crystal of pure Thiourea is showed in the below figure.



X-RAY DIFFRACTION (XRD) TECHNIQUE:

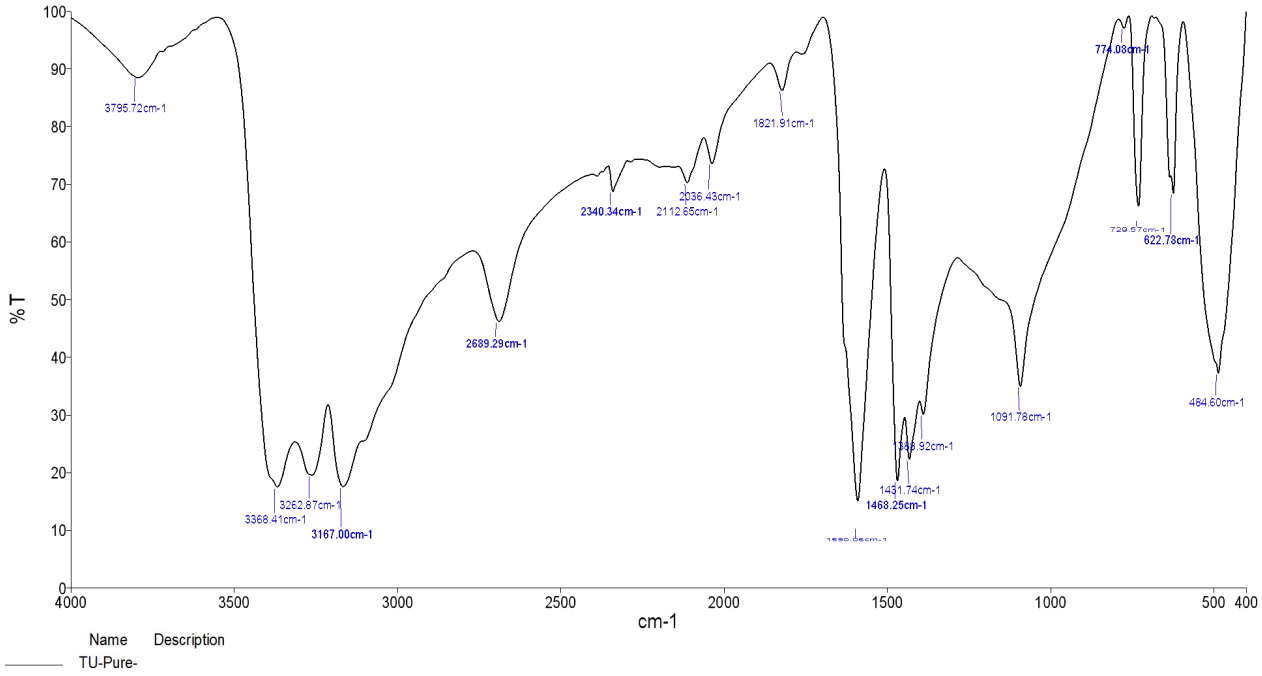
X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized and average bulk composition is determined.

X-rays were accidentally discovered by Rontgen (professor from German) in 1895When he was studying the phenomenon of electricity through rarefied gases at very low pressures. After performing a series of experiments, Rontgen concluded that when a beam of fast moving electrons strike a solid target an invisible high penetrating radiation

is produced. Because of their unknown nature, Rontgen called these radiations as X-rays. They are also called Rontgen rays. Actually X-rays are electromagnetic waves like visible light, but of very short 5wave length in the range of $0.5A^0$to $10A^0$.

The FTIR spectra of as prepared pure Thiourea crystal sample:

The graph is plotted between % of transmittance and wave number (cm^{-1}).In this graph different peaks formed at different wave number. It is observed that the graph have various frequency vibrations which are shown by different peaks formed.

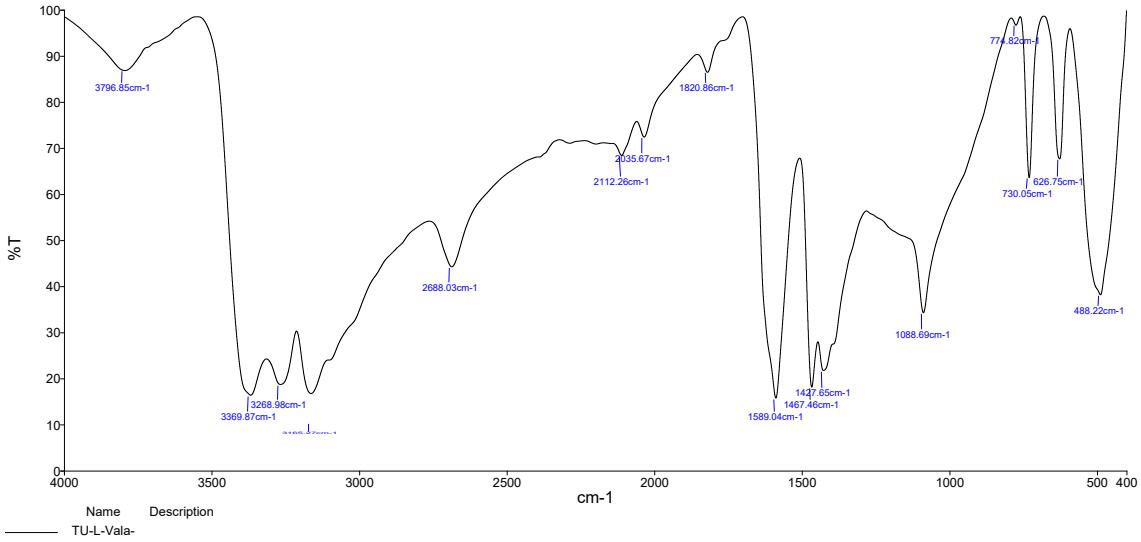


The graph is plotted between % transmittance and wave number (cm-1).In this graph different peaks formed at different intensity (a.u). It is observed in the graph that pureThiourea have various frequency vibrations which are shown by different peaks . and the

Frequency(cm^{-1})	Nature of the peak	Assignment
3262.87	Strong	N-H Amine Stretch
2689.29	strong	O=HCarboxylic acid
2036.43	Medium	Nitrite medium
1821.91	Weak	-C-H(Aromatic)
1091.78	Strong	C-I stretchAlkyl Halid

The FTIR spectra of as prepared pure Thiourea doped l-valine sample:

The graph is plotted between % transmittance and wave number (cm^{-1}).In this graph different peaks formed at different wave number. It is observed in the graph that have various frequency vibrations which are shown by different peaks formed.



The graph is plotted between % transmittance and wave number (cm-1).In this graph different peaks formed at different intensity (a.u). It is observed in the graph that Thiourea doped L-valine have various frequency vibrations which are shown by different peaks formed and the x axis shows the 2T (Degree).

Frequency(cm^{-1})	Nature of the peak	Assignment
3369.85	Medium	O=H(Carboxylic acid)
3268.98	Strong	N-H (Amine)
2688.03	Strong	O=H(carboxylic acid)
1088.69	Two bands or more	C-C-O Stretching
730.05	Strong	C-C stretching

The X-ray diffraction of as prepared pure Thiourea sample:

The pure Thiourea crystals were crystallized by powder X-ray diffraction to identify the structure and lattice parameters .XRD techniques is used to investigate inner arrangement of atoms or molecules in a crystalline material. The sample was scanned for 2Ɵ values o^0 to 100^0.

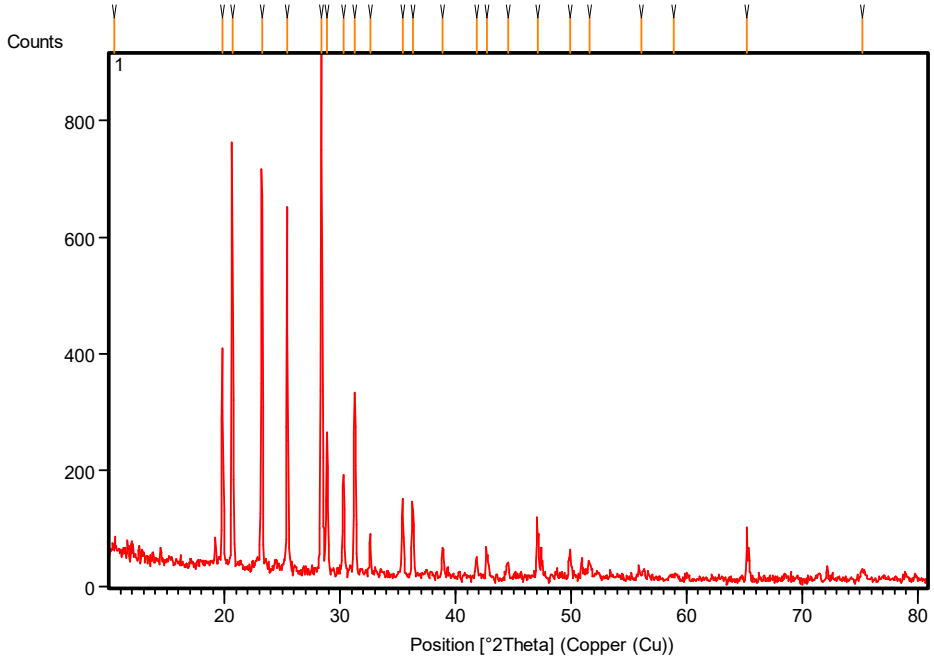


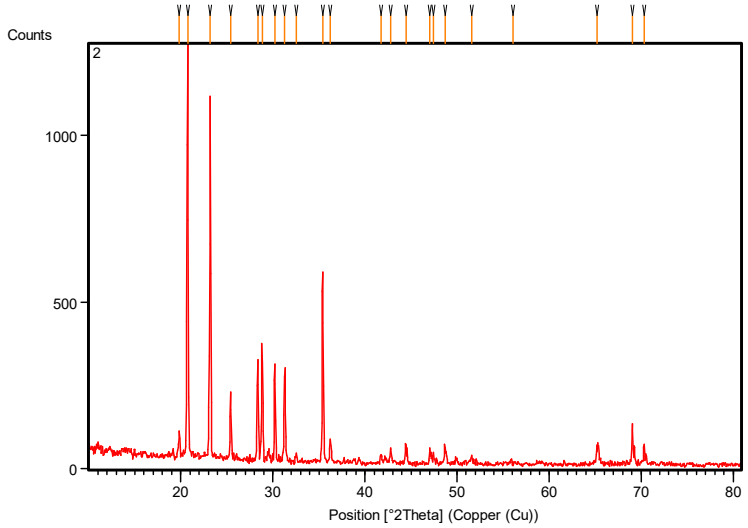
Figure: The X-ray diffraction of pure Thiourea sample.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
10.4517	63.22	0.5904	8.46426	7.90
19.8412	387.05	0.1476	4.47482	48.37
20.6912	734.33	0.1476	4.29287	91.78
23.2438	746.73	0.1476	3.82689	93.33
25.4451	625.36	0.1476	3.50060	78.16
28.4076	800.11	0.1476	3.14191	100.00
28.8824	258.87	0.1476	3.09133	32.35
30.3129	185.21	0.1476	2.94864	23.15
31.2819	322.54	0.1968	2.85947	40.31
32.6290	79.51	0.1476	2.74443	9.94
35.4236	141.78	0.1968	2.53407	17.72
36.2994	130.47	0.1968	2.47491	16.31

38.8930	59.15	0.1476	2.31564	7.39
41.8063	41.02	0.1476	2.16076	5.13
42.6928	56.55	0.2460	2.11792	7.07
44.5427	29.03	0.2952	2.03417	3.63
47.0984	81.86	0.1476	1.92958	10.23
49.9025	40.92	0.2952	1.82753	5.11
51.6207	30.14	0.2952	1.77067	3.77
56.0603	15.04	0.5904	1.64051	1.88
58.8775	9.60	0.5904	1.56856	1.20
65.1934	70.25	0.1968	1.43105	8.78
75.2447	18.50	0.3936	1.26289	2.31

The X-ray diffraction of as prepared Thiourea Doped L-valine sample:

The pure Thiourea doped l-valine crystals were crystallized by powder X-ray diffraction to identify the structure and lattice parameters .XRD techniques is used to investigate inner arrangement of atoms or molecules in a crystalline material. The sample was scanned for 2Θ values o^0 to 100^0.



Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
19.8615	73.05	0.1476	4.47029	5.86
20.7607	1246.23	0.1476	4.27866	100.00
23.2166	1081.09	0.1476	3.83132	86.75
25.4306	198.25	0.1476	3.50257	15.91
28.3809	296.35	0.1476	3.14481	23.78
28.8586	314.43	0.1476	3.09383	25.23
30.2320	284.87	0.1476	2.95634	22.86
31.3061	288.80	0.1476	2.85732	23.17
32.5224	22.18	0.1968	2.75318	1.78
35.4295	558.27	0.1968	2.53366	44.80
36.2625	71.51	0.1476	2.47735	5.74
41.7413	24.74	0.1968	2.16397	1.99
42.7964	47.60	0.1476	2.11304	3.82
44.4604	59.32	0.2460	2.03774	4.76
47.0714	47.02	0.1968	1.93062	3.77
47.4649	37.27	0.1476	1.91553	2.99
48.7274	45.65	0.2460	1.86881	3.66
51.5976	19.75	0.2952	1.77140	1.59
56.0824	3.80	1.1808	1.63992	0.30
65.2404	43.04	0.1968	1.43013	3.45
69.0335	127.81	0.1476	1.36052	10.26
70.2933	60.77	0.1476	1.33920	4.88

The UV spectra of as pure Thiourea crystal sample:

The optical transmission studies were recorded for the sample obtained for the crystal. The sample is prepared by dissolving in the water. To find the transmission range of pure Thiourea crystal the optical transmission spectrum was recorded using UV –Vis Nir spectrometer.

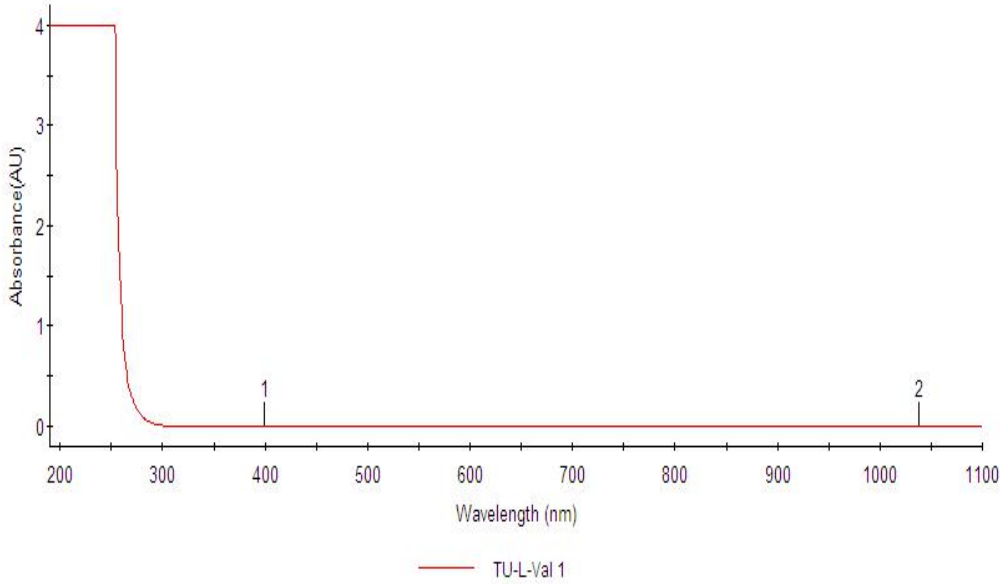
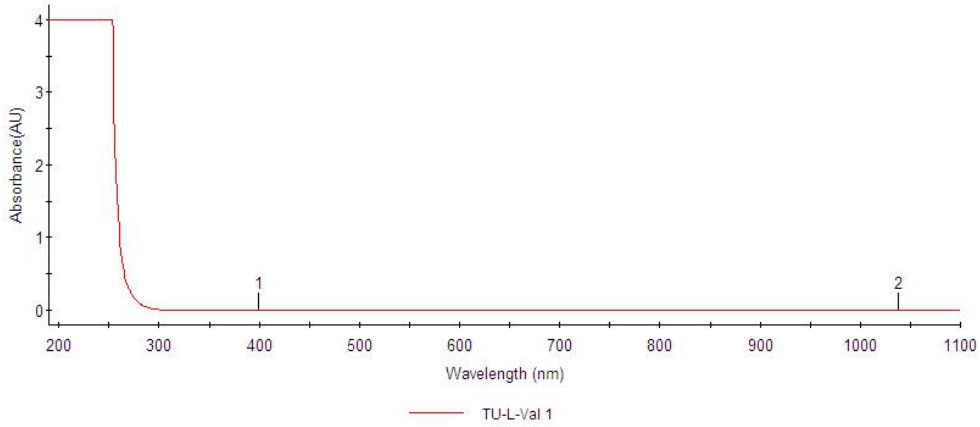


Figure: UV –Visible spectrum of pure Thiourea crystal.

The UV spectra of as pure Thiourea doped l-valine crystal sample:

The optical transmission studies were recorded for the sample obtained for the crystal. The sample is prepared by dissolving in the water. To find the transmission range of pure Thiourea doped l-valine crystal the optical transmission spectrum was recorded using UV –Vis Nir spectrometer.



Name	No.	Peak (nm)	Peak (AU)
TU-L-Va	1	1398.8	0.001233348
	2	1,038.0	0.002547054

Figure: UV –Visible spectrum of pure Thiourea doped l-valine crystal.

Conclusion

Thiourea and L-valine has been growing by slow evaporation method:

The growth of pure *Thiourea* and L-valine doped crystal were grown by solution method under slow evaporation technique. The spectacular growth of solid state electronics is critically dependent on crystal quality. The most versatile technique of commercial importance is solution technique.

The structure and morphology of the crystal was analyzed to x-ray diffraction and it is found to be the crystals are of orthorhombic shape.

The FTIR studies of samples the characteristics absorption of the bands.

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