



## CRYSTAL GROWTH AND CHARACTERIZATION OF CADMIUM TRIS THIOUREA CHLORIDE DOPED WITH L- ARGININE CRYSTALS (CTTCDA)

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### Abstract

The Tris Thiourea cadmium chloride doped with L-Arginine (CTTCDA) was successfully grown into crystals using slow evaporation method. The grown crystals were the subject of numerous investigation, including UV-Visible spectra, FTIR, thermal analysis (TG-DTA), SEM-EDAX spectra, and powder X-ray diffraction. Spectral analyses in the UV-Visible region were used to determine the optical and electronic properties of (CTTCDA). The calculated band gap energy is 4.57 eV. By using FTIR analysis, the functional group of CTTCDA crystals doped with L-Tryptophan was observed. The CTTCDA is thermally stable up to 240 °C, according to TG-DTA studies. The powder X-ray diffraction pattern confirms the crystalline type and purity of the grown crystals. An analysis of grown crystals using a Scanning Electron Microscope reveals that they are of good quality and have a layer-like structure. The characterization studies have shown that synthesized crystals exhibit favorable properties for nonlinear optical applications.

**Keywords:** CTTCDA Crystal, UV-Visible Spectrophotometer, Fourier Transform – Infra Red Spectrophotometer, SEM-EDAX, TG-DTA, P-XRD

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### 1. INTRODUCTION:

Growth is an essential and fundamental part of material science and engineering because crystals of suitable size and perfection are required for fundamental data acquisition as well as practical devices such as detectors, integrated circuits, plethora of other applications [1-11]. In addition to the fact that crystals have been marvels for many thousands of years, the high demand for crystals to meet the needs of the information age and newly emerging fields of study, as well as the fact that crystals have been wonders for more than a century, have been the focus of research on a variety of techniques for the growth of crystals. Doped NLO crystals may be more useful for optoelectronic devices than pure NLO crystals because doping has reported to change a variety of physical and chemical properties [12-16]. The good mechanical and thermal properties of the ionic liquid tend to interact with the good optical non linearity of purely organic amino acids, resulting in organic molecules with inorganic components that are suitable materials for nonlinear optical applications. The building blocks of the protein structure are amino acids, which mostly comprise a carboxylic group and an amino group. The organic ligand typically has a stronger influence on the NLO effect in metal organic complexes based on amino acids. The prospective NLO materials

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have been researched, including bis thiourea cadmium chloride and E-allyl thiourea cadmium chloride. The production of salts with various organic and inorganic salts have made use of amino acids like L-arginine and L-histidine. Non-linear optical materials have several uses in a variety of industries, including optical modulation, frequency doubling technology, color displays, optical data storage, and submarine communication. The mechanical and thermal properties of the organic NLO are poor, and they have low laser damage thresholds and high second order optical non-linearity. Despite having excellent mechanical and thermal qualities, inorganic materials exhibit poor optical non-linearity. The combination of organic and inorganic components makes up semi-organic materials. This substance exhibits strong mechanical, thermal, and nonlinear coefficients. The good mechanical and thermal properties of the ionic liquid tend to interact with the good optical non linearity of purely organic amino acids, resulting in organic molecules with inorganic components that are suitable materials for nonlinear optical applications. In recent years the metal complexes of thiourea, allylthiourea, and thiocyanate explore strong second order nonlinear optical properties for laser application. In recent years the metal complexes of thiourea, allylthiourea, and thiocyanate explore strong second order nonlinear optical properties for laser application. Slow evaporation solution growth is widely used techniques to grow crystals CTTCDA family [17-20]. In this present communication, the impact of L-Tryptophan dopant on changing the crystal morphology, lattice parameters, mechanical, and optical properties of CTTCDA has been reported. Results of powder X-ray analysis, optical transmittance, SEM, TG-DTA, and other experimental evidences are presented. Because they can generate higher original frequency values, non linear (NLO) materials are essential for optical modulation, fibre optic communication, and optoelectronics. Growing organic, inorganic, and semiorganic materials that are suitable for device fabrication and have high damage thresholds, broad transparency ranges, and high nonlinear coefficients are ongoing efforts. Tris thiourea cadmium chloride is a semi-organic compound that shows both semi-organic and organic materials have greater thermal stability.[21-25] They are recognised as valuable crystals for nonlinear optical applications in electro-optical modulation, optical switching, optical data storage, optical information processing, optical communication, and optical switching. A brand-new class of materials for frequency conversion applications has emerged: semi organic crystals. Such materials, which are created when a polarizable organic molecule is joined to an inorganic host to form a salt or a coordination complex, include compounds like L-arginine phosphate and zinc tris thiourea sulphate. A desirable combination of nonlinear optical, mechanical, and growth properties can be obtained using molecular engineering because both components have a wide range of possible configurations. Samples with good optical quality and size can be produced at comparatively low cost because many of these crystals can be grown from aqueous solutions.

## 2. MATERIAL AND METHODS

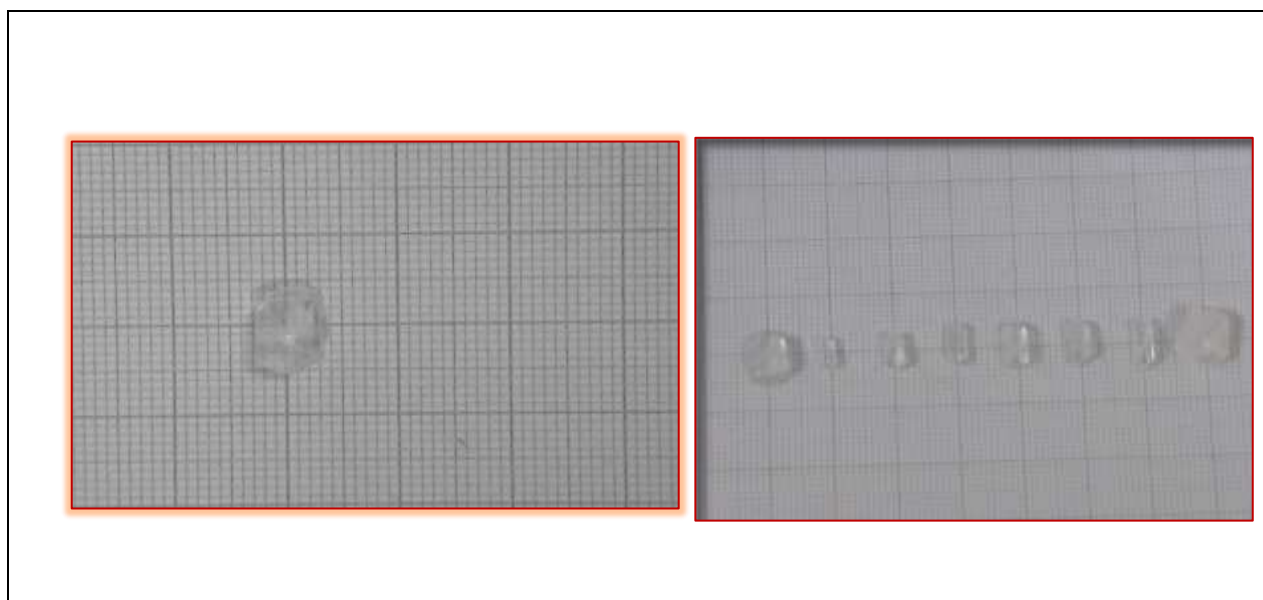
One of a very few numbers of studies that used slow evaporation techniques to investigate the synthesis, growth, and characterisation of zinc tris thiourea doped L-arginine crystals. This method of crystal growth results in crystals with exceptional optical transmission. The NLO property was also researched. Thiourea, Cadmium chloride, and L-Arginine were combined in the amounts 2:1:1 according to the following chemical reaction to produce crystals of Cadmium tris thiourea chloride doped with L-Arginine (CTTCDA).



### 2.1 Synthesis and crystal growth techniques

For the current work, the AR grade of L-Arginine, Cadmium chloride, and Thio Urea was used in a 1:1:3 ratio. For this synthesis, 16.1 g of Zinc sulphate dissolved in 100 ml of water and 15.22 g of Thio urea dissolved in 100 ml of water were used. After thoroughly mixing the two solutions, the solution was heated in a magnetic stirrer at 50° C until a white crystal of CCTTDA solution been obtained. The solution was ordered separately to obtain doped salt at a concentration of L-Arginine of 1 mole percent. Solution growth and slow evaporation techniques were used to grow crystals of L-Tryptophan doped CTTDA at room temperature. CCTTDA crystal is synthesized by dissolving AR grade of thiourea and AR grade of cadmium chloride doped with L-Arginine in the molar ratio 3:1:1 in distilled water. The saturated solution of cadmium chloride was slowly added to the saturated solution of thiourea this was stirred well to get a clear solution. CCTTDA crystal was synthesised according to the reaction. The saturated solution was kept in a beaker covered with filter paper.

#### CTTCDA \ CRYSTAL



**Figure 1 shows Grown crystal of cadmium Tris thiourea chloride doped with l-arginine crystals (CTTCDA)**

### 2.2 Characterization

The FT-IR of spectra of CTTCDA was acquired in the 4000-600 cm<sup>-1</sup> region using an Agilent Technologies Cary 630 FT-IR Spectrophotometer utilising the K Br pellet technique. The UV-Visible near analysis was carried out using a VARIAN COPY 5E spectrophotometer between 200 and 800 nm. The thermal behaviour of the grown crystal was investigated using simultaneous TGDTA analyses in the temperature range 100 to 1200 in a nitrogen atmosphere using a model ZETZSCH STA 449F3 analyzer at a heating rate of 20 K/min. Powder XRD studies were carried out using the circle Rigaku Mini Flex 600 to support the formed crystal. To analyse the observation, the SEM spectrum of as grown crystals was analysed using a CARL ZEISS FESEM with a scanning image range of SED 200 KV

### 3. RESULTS AND DISCUSSION

The computed band gap energy is 4.78 eV. The functional group of CTTCDA crystals doped with L-Arginine was discovered via FTIR analysis. According to TG-DTA tests, the CTTCDA is thermally stable up to 240°C. UV-Visible spectroscopic, Band gap energy, FT-IR Spectroscopy, TG-DTA, SEM with EDAX for morphological and elemental analysis, Powder XRD analysis for crystallography and lattice parameter studies are discussed briefly.

#### 3.1 UV-Visible Spectroscopic Studies

The optical absorption spectrum of CTTCDA crystal was recorded in the wavelength region 200-800 nm using VARIAN COPY 5E SPECTROPHOTOMETER and is shown in the table Fig 2. The UV absorption edge of the grown crystal was found to be approximately 250 nm long. It is determined that the lower cutoff wavelength is 218 nm, shown in table 1 & Fig 1. The near UV region absorption is induced by an electronic transition within the thio urea units of CTTCDA, which is in good accordance with the reported value. The material is a viable target for optoelectronic applications due to the wide range of transparency in the entire visible region [4]. The  $\pi$ - $\pi^*$  conjugation shown Table 2 by the peak at about 218 nm. The peak intensity typically rises with the presence of additive. The exhibits greater depth at 250 nm, which is conducive to more non-linear interaction. In order to understand the relationship between optical absorption coefficient and photon energy location, it is necessary to understand band structure and electron transmission types. The absence of absorption in the range 242-800 nm good optical transmission of the crystal favors the SHG.

The crystal shows a good transmittance in the visible region which enables it to be a good material for optoelectronic applications. The lower cut off wavelength for CTTCDA is found at 213 nm. The wide range of transparency suggests that the crystals are good candidates for non linear applications. The shift of lower cut off wavelength in UV region is due to doping of L-arginine and is desirable for optoelectronic applications.

#### 3.2 Band gap energy

The band gap measurement was also carried out doped CTTCDA crystals. The energy band gap of rough mixed crystal of CTTCDA are indicated an extended trend line shown in Figure 2. The band gap of rough mixed crystal with a Thiourea surface is assumed to be 2 eV indicating the line crossed the X-axis. When L-arginine doped with cadmium Tris thiourea Chloride crystal. The band gap cadmium tris thiourea chloride doped L-arginine are estimated to 4.75 eV

#### 3.3 FT-IR Spectral analysis

Using the Agilent Technologies Cary 630 FT-IR spectrophotometer [24-27], the FT-IR spectrum from 600  $\text{cm}^{-1}$  to 4000  $\text{cm}^{-1}$  was detected [6]. The FT-IR spectrum for the grown L-Tryptophan doped with Zinc Bis thiourea Sulphate crystals is presented in the Figure 4. To record FT-IR spectra of TDZBTS crystal Perkin Elmer spectrometer was used in the range 700- 4000  $\text{cm}^{-1}$  which was in the form of solid dispersed K Br pellet. The asymmetric stretching vibration of the NH symmetry is shown by the peak at 3465  $\text{cm}^{-1}$ . The absorption at 3362  $\text{cm}^{-1}$  is due to the presence of O-H stretching & CO stretching, carboxylic acid also presents in this compound. The peak at 3176  $\text{cm}^{-1}$  is due to NH stretching vibration. The peak at 3105  $\text{cm}^{-1}$  is due to the formation of NH stretching. Secondary amide present in this

compound. The absorption at  $1601\text{ cm}^{-1}$  is due to the existence of NH plane in bending vibration, free primary amide presents in this compound. The absorption at  $1438\text{ cm}^{-1}$  is due to the existence of C=S Stretching vibration, free primary amide presents in this compound. This is also confirmed to the primary thio amide group present in this compound. The absorption peak at  $3176\text{ cm}^{-1}$  is due to the presence of NH stretching frequency Indole group present in this compound. Depending upon coordinate compound which is a derivative of CTTCDA can be produced.

### 3.4 TG-DTA analysis

The CISL Laboratory was used to perform the TG-DTA analysis on the CTTCDA. Instrumentation for thermal analyzers that is placed in a nitrogen atmosphere has the potential to quickly heat up to  $1200^{\circ}\text{C}$  from ambient temperature at a rate of  $20^{\circ}\text{C}$  per minute. The TG-DTA curve of L-Arginine doped with crystals of CTTCDA is shown in the Fig 4. The peak of both DTA and TGA curves at  $100^{\circ}\text{C}$  shows that the material starts to degrade roughly  $250^{\circ}\text{C}$ . The TGA curve demonstrates the CTTCDA good thermal stability up to  $240^{\circ}\text{C}$  with the greatest weight loss (nearly 95%) occurring in the temperature ranges of  $210^{\circ}\text{C}$  to  $320^{\circ}\text{C}$  [8]. A first endothermic transition, which occurs at  $270^{\circ}\text{C}$  and is caused by the volatile compounds like Sulphur oxide, nitrogen and it is followed by a second endothermic transition, which occurs at  $300^{\circ}\text{C}$ , as shown by the DTA curve. The sharpness of the melting curve implies that the sample is perfectly crystalline.

### 3.5 SEM and EDAX analysis

The SEM of the L-arginine doped cadmium tris thiourea chloride crystal is shown in Fig.5 widely varying morphology absorb particle appeared as aggregates of tiny crystal. They were particle of nano and bulk dimension with bulk dimension the average diameter of the particle  $10\mu\text{m}$ . In micrographs clearly show that at first spherical nanoparticles formed under experimental condition undergoes extensive agglomeration. Reduction of particle size causes the enhancement of surface activity that leads to larger agglomerated particles. Figure.5. shows the particles size distribution of processed crystals The EDAX spectra for the CTTCDA crystal is shown in Figure 5. The weight % of each element derived from the EDAX spectra is illustrated in Figure 2. From the weight % bar chart it was clearly evident that the major component of the crystal mixture was CTTCDA. The peak show the presence of Sulphur, Zinc in the crystal. It was observed that the atomic % of Zn, S and other impurities of Cd, Ni 25.27, 74.43, 0.09, 0.21 respectively, shown in table 3 and fig 5.

### 3.6 Powder XRD analysis

The powder X-ray diffractometers was used to determine the sample crystallinity type the compounds degree of crystalline nature is indicated by the sharp intensity peak [16]. The observed CTTCDA crystal powder XRD pattern, which not only verifies the crystals strongly defined crystalline character but also provides additional information are depicted in Figure 2. The alterations that could result in patterns for crystals observed over the course of two hours using the (hkl) indices. The peaks were identified using powder XRD. The observation that the hkl peaks and intensities for the simulated and empirically acquired PXRD patterns coincide each other clearly shows this. The d-spacing for the matching (h k l) values was used to index the diffraction peaks in the PXRD pattern [26]. X-ray powder diffraction pattern of CTTCDA was recorded on a Rigaku Mini Flex 600 diffractometer. The powder sample was scanned over the range  $10-40$  at a scan rate  $1/\text{min}$ . The PXRD pattern of the crystal is shown in fig.7. the unit cell parameters  $a=18.9652$   $b=10.5996$ .

$c=17.2446$   $V=2781.36$ , which are in good agreement with the results reported. The powder X-ray diffractometer was used to determine the sample crystallinity type the compounds degree of crystalline nature is indicated by the sharp intensity peak [11]. The monoclinic structure of the crystals was confirmed by powder X-ray diffraction, and the lattice parameters were found to be reasonably matched to the reported values. It confirmed that the Cadmium Tris thiourea doped L-Arginine that had been grown from melt had crystallized in a Triclinic system and had a strong diffraction peak. The  $2\theta$  value shows excellent crystalline quality and purity that can be used in NLO applications. The XRD pattern for L-Tryptophan doped CTTCDA are shown in the table 4. [26-28].

#### 4. SUMMARY AND CONCLUSION

The crystals of were successfully grown using slow evaporation techniques. The crystal that comprise of thiourea and doped with L-Arginine of equimolar concentration. The aforementioned crystal exhibits 100% transmission in the UV spectrum, with a cut off wavelength of 250 nm. The band gap energy found to be 4.75 eV. It indicates that it can be applied for laser application in the Nano technology. FT-IR analysis confirm the presence of functional groups stable up to 240° C. P-XRD was used to confirm the grown crystals crystalline nature and structural composition. It is also study the lattice parameters value. According to the finding of these studies, L-Argine doped cadmium tris thiourea chloride crystals are a viable option for use in the production of laser techniques. Further research is needed to investigation the synthesis and characterization of a number of other novel crystals using various methods and application such as gas storage and separation, catalysis, and sensors. The wide bandgap and the optical transparency of the crystal, especially suited for nonlinear optics, organic superconductor, optical sensors, optical computers and display system.

**Table 1 :** UV absorption zone of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)

Max value (nm)	Transition
218	$\pi-\pi^*$
250	$n-\pi^*$

**Table 2:** FT-IR Spectrum of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)

Wave number (Cm-1)	Mode	Assignment
3465	NH asymmetric stretching	Secondary amine
3362	OH stretching	Carboxylic acid group
3176	NH stretching vibration	Secondary amide
3105	NH stretching	Secondary amide
1601	NH plane in bending	Primary amide
1438	C=S stretching	Amide I band

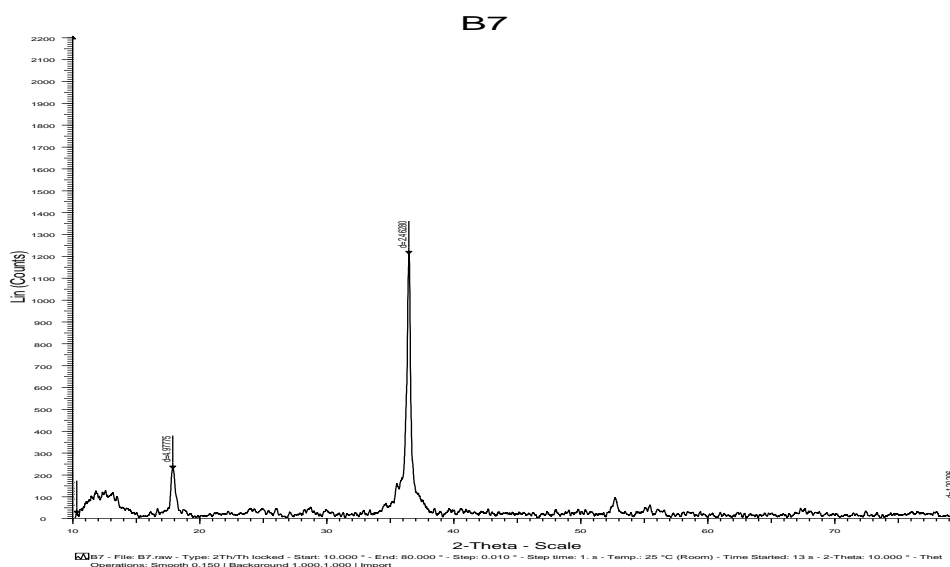
1385	C=S stretching vibration	Thio amide
3176	NH symmetric stretching	Primary thio amide
706	NH out of plane bending	Aliphatic amine

**Table 3:** SEM-EDAX Spectrum of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)

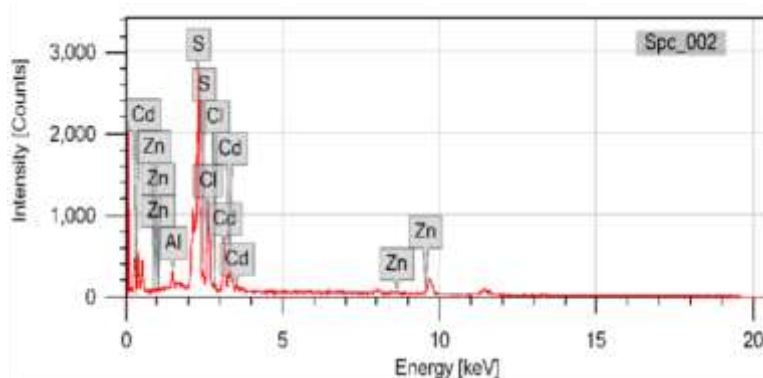
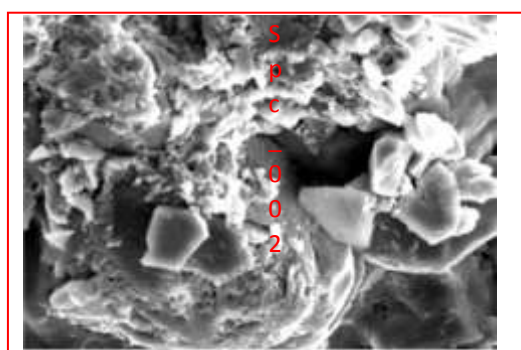
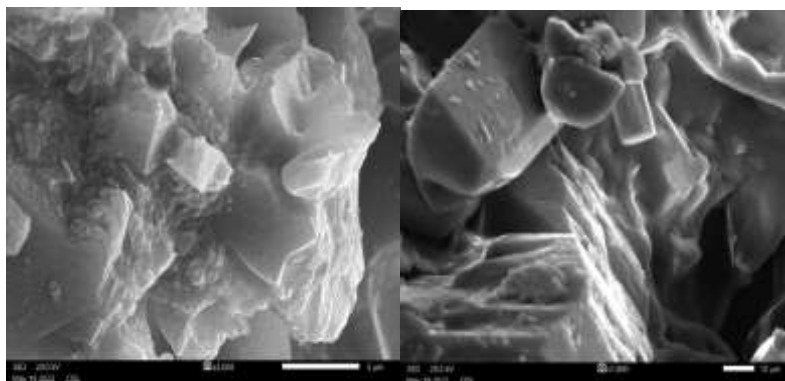
Element	Line	Mass%	Atom%
Al	K	2.55±0.11	4.25±0.18
S	K	36.86±0.32	51.74±0.45
Cl	K	22.61±0.30	28.71±0.38
Zn	K	0.33±0.24	0.23±0.16
Cd	L	37.66±0.57	15.08±0.23
Total		100.00	100.00
Spc_002			Fittingratio0.4486

**Table 4:** Crystal structure of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)

Crystal system	A	B	C	Alpha	Beta	Gamma	Volume
Triclinic	18.9652	10.5996	17.2446	90°	10.740°	90°	2781.36



**Figure 1:** Powder XRD of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)



**Figure 2:** SEM-EDAX spectrum of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)

Element	Line	Mass%	Atom%
Al	K	2.55±0.11	4.25±0.18
S	K	36.86±0.32	51.74±0.45
Cl	K	22.61±0.30	28.71±0.38
Zn	K	0.33±0.24	0.23±0.16
Cd	L	37.66±0.57	15.08±0.23
Total		100.00	100.00
Spc_002			Fittingratio0.4486



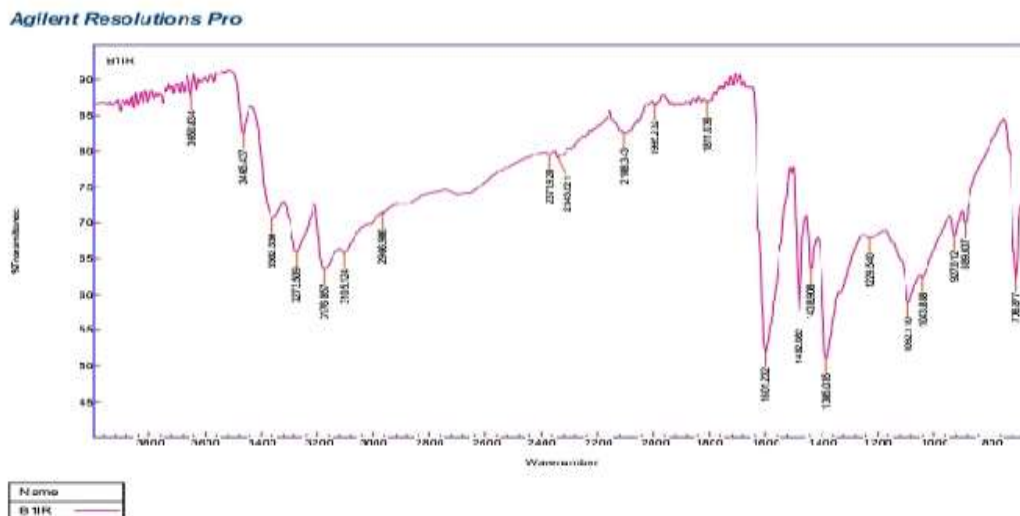


Figure 3: FT-IR Spectrum of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)

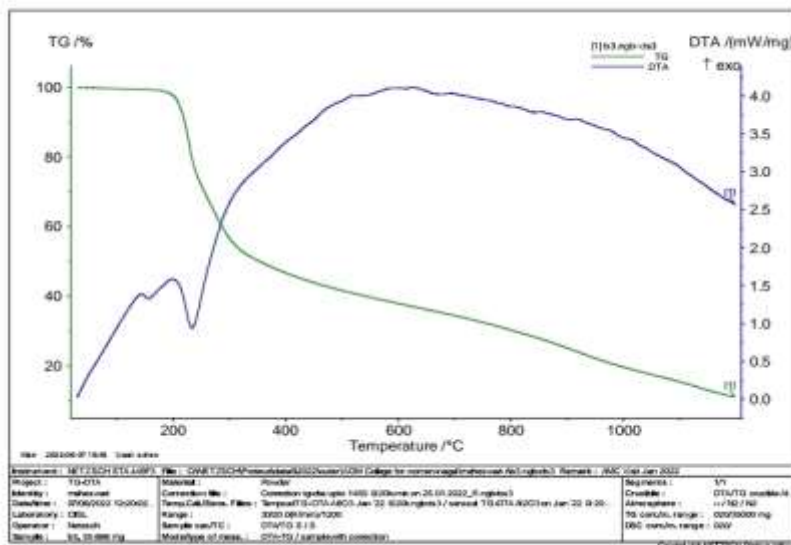
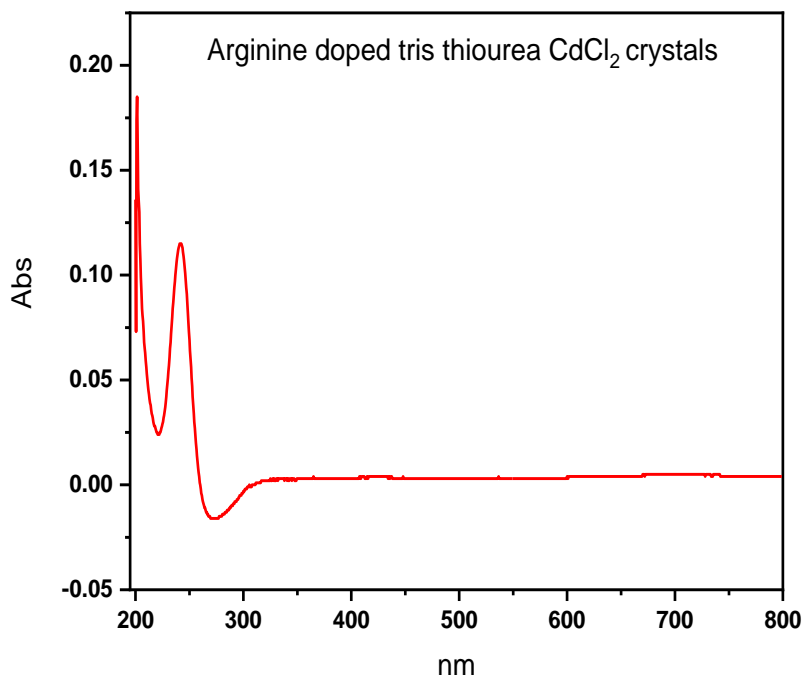
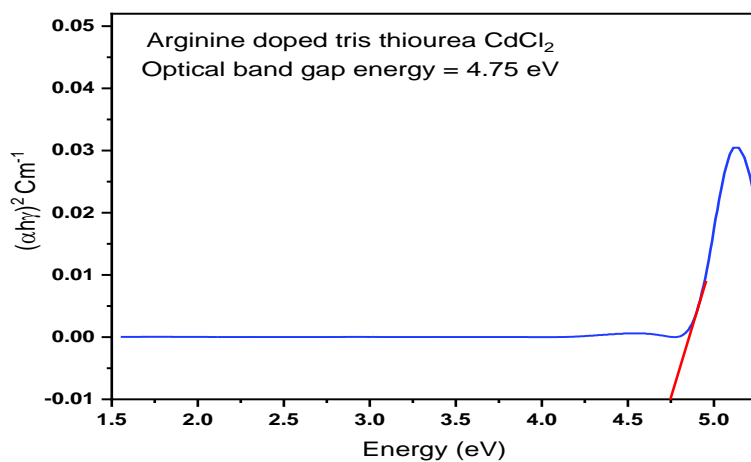


Figure 4: TG-DTA Spectrum of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)



**Figure 5:** UV-Visible Spectrum of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)



**Figure 6:** Band gap energy of cadmium Tris thiourea chloride doped with L-arginine crystals (CTTCDA)

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