

## EDAX, FTIR, Powder XRD and Thermal studies of pure and mixed levo-tartrate crystals cobalt cadmium

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**Abstract:** Metal tartrate compounds like cadmium tartrate and cobalt tartrate find piezoelectric and electro-chemical applications, respectively. In the present work, the growth of pure and mixed crystals of cobalt and cadmium levotartrate is accomplished by gel method. Spherulitic and dark brown crystals are obtained. The metallic element content in the crystals is estimated by EDAX. The analysis of FTIR spectroscopy data confirms the presence of various functional groups. The powder XRD suggests the mixed phase nature of the grown crystals. By applying the Rietveld analysis the two phases, i.e. orthorhombic phase of cobalt levo-tartrate and monoclinic phase of cadmium levo-tartrate were identified. The thermo-grams confirm the conversion of crystals into oxide form after removal of water molecules. DTA curves show the peaks corresponding to related reactions.

**Keywords:** cobalt and cadmium mixed levotartrate crystals, gel method, FTIR, Powder XRD, TGA, DTA.

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### I. Introduction:

Tartrate crystals have attracted the researchers due to their ferroelectric and piezoelectric properties and usefulness as transducer and linear and non-linear mechanical devices [1-5]. For monovalent and bivalent alkali metals, the general formula of tartrates can be written as  $A_2C_4H_4O_6$  and  $BC_4H_4O_6$ , respectively. Some tartrate molecules may be hydrated also and contain one, two, three or more number of water molecules. Tartrates are the materials, which have the lowered solubility in water and decompose before melting, the gel growth technique under the air temperature of an environment is found to be suitable [6]. Tartrate compounds of cobalt and cadmium find important applications, for example, cobalt solutions containing cobalt (II) tartrate complexes find application in an electrochemical procedure of anodic deposition of cobalt oxyhydroxide film on a glassy carbon substrate in an alkali medium [7], as a precursor cobalt tartrate is used to synthesize cobalt nano ferrites [8] and piezoelectric application of cadmium tartrate [9]. There are several attempts have been made by different workers to grow cadmium tartrate [4,5,10,11] and cobalt tartrate [12-14] crystals by the gel growth technique. Moreover, 2-dimensional cobalt (III) tartrate hydrated coordination polymer crystallization is reported recently by Khunar et al [15]. Also, a three dimensional chiral structure of cadmium tartrate is reported by Peng and Jiang [16]. Altogether, attempts have been made earlier to grow certain mixed tartrates by the gel method, such as lead-cadmium tartrate [17], manganese-copper mixed levo-tartrate [18], calcium-cadmium tartrate [19], iron-manganese-cobalt ternary levo-tartrate [20]. Further, it is interesting to note that only few attempts have been made to synthesize metal tartrate nano-particles such as calcium tartrate nano-particles [21] and recently iron tartrate nano particles [22]. Considering the applications of cobalt tartrate and cadmium tartrate, the authors have made an effort to accomplish the growth of mixed levotartrate crystals of cobalt and cadmium of different volume concentrations by gel method and characterize them by EDAX, FTIR, Powder XRD and TGA-DTA.

**Experimental:** The single diffusion gel method was used to grow the pure and mixed levotartrate crystals of cobalt and cadmium. Silica hydro gel was used as a growth medium having definite specific gravity and pH value. The gel solution was poured into glass test tubes having proper dimensions and left for some days so that can convert into gel form. The solutions used have the below commendation compositions and were slowly discharged on the set gel.

(I) 10 ml, 1M,  $Co(NO_3)_2 \cdot 6H_2O$  (sample-1)

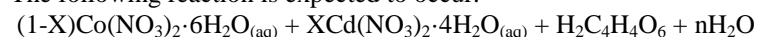
(II) 8 ml, 1M,  $Co(NO_3)_2 \cdot 6H_2O$  + 2 ml, 1M,  $Cd(NO_3)_2 \cdot 4H_2O$  (sample-2)

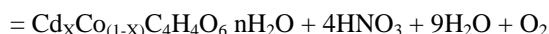
(III) 6 ml, 1M,  $Co(NO_3)_2 \cdot 6H_2O$  + 4 ml, 1M,  $Cd(NO_3)_2 \cdot 4H_2O$  (sample-3)

(IV) 4 ml, 1M,  $Co(NO_3)_2 \cdot 6H_2O$  + 6 ml, 1M,  $Cd(NO_3)_2 \cdot 4H_2O$  (sample-4)

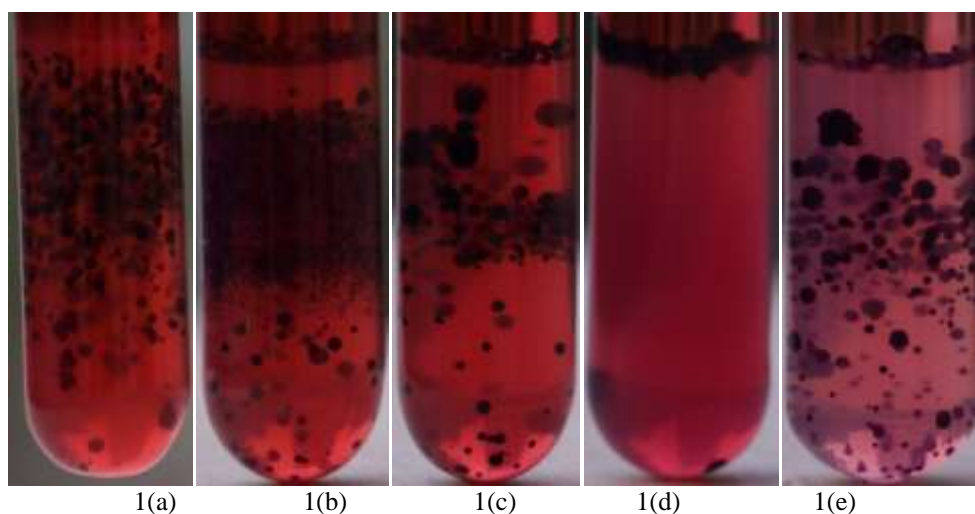
(V) 2 ml, 1M,  $Co(NO_3)_2 \cdot 6H_2O$  + 8 ml, 1M,  $Cd(NO_3)_2 \cdot 4H_2O$  (sample-5)

The following reaction is expected to occur.





The value of X that has been set for the experiment is 0.2, 0.4, 0.6 and 0.8. Its exact value is determined from EDAX analysis. The nitric acid produced during the reaction is so dilute that shows no major effect on the growth process of crystals [4,12]. The growth was completed in nearly twenty days. The nature of the grown crystals was spherulitic type for all the five samples. The images of the growth of crystals inside test tubes are shown in the figure 1(a-e) for the solution I, II, III, IV and V, respectively.



**Figure 1:** Growth of crystals in test tubes

The grown crystals were characterized by EDAX, FTIR, Powder XRD and thermal analysis. The EDAX was carried out by using the set up of the instrument Philips XL-30. The data of %T were recorded on Perkin Elmer Spectrum GX spectrometer in the range from 400–4000  $\text{cm}^{-1}$  in KBr medium. The powder XRD patterns were recorded on Bruker AXS D8 Advance Diffractometer by using  $\text{CuK}_\alpha$  radiation. The Rietveld analysis of the soft data was done by Full Prof Program at IIT Kanpur. The TGA and DTA were conducted on Perkin Elmer, Diamond TG/DTA within 40 to 800  $^\circ\text{C}$  range of temperature having rate of heating 10  $^\circ\text{C}/\text{min}$  in the atmosphere of air.

## II. Result and Discussion:

**EDAX Analysis:** The composition of the grown crystals was determined by EDAX and listed in Table 1.

**Table 1:** Atomic weight % of Co and Cd

Sample No.	Sample	Expected Atomic Weight (%)		Observed Atomic Weight (%) (From EDAX)	
		Element		Element	
		Co	Cd	Co	Cd
1.	$\text{CoC}_4\text{H}_4\text{O}_6 \cdot n\text{H}_2\text{O}$	100	0	-	-
2.	$\text{Co}_{0.8}\text{Cd}_{0.2}\text{C}_4\text{H}_4\text{O}_6 \cdot n\text{H}_2\text{O}$	80	20	77.66	22.34
3.	$\text{Co}_{0.6}\text{Cd}_{0.4}\text{C}_4\text{H}_4\text{O}_6 \cdot n\text{H}_2\text{O}$	60	40	43.68	56.32
4.	$\text{Co}_{0.4}\text{Cd}_{0.6}\text{C}_4\text{H}_4\text{O}_6 \cdot n\text{H}_2\text{O}$	40	60	31.65	68.35
5.	$\text{Co}_{0.2}\text{Cd}_{0.8}\text{C}_4\text{H}_4\text{O}_6 \cdot n\text{H}_2\text{O}$	20	80	15.98	84.02

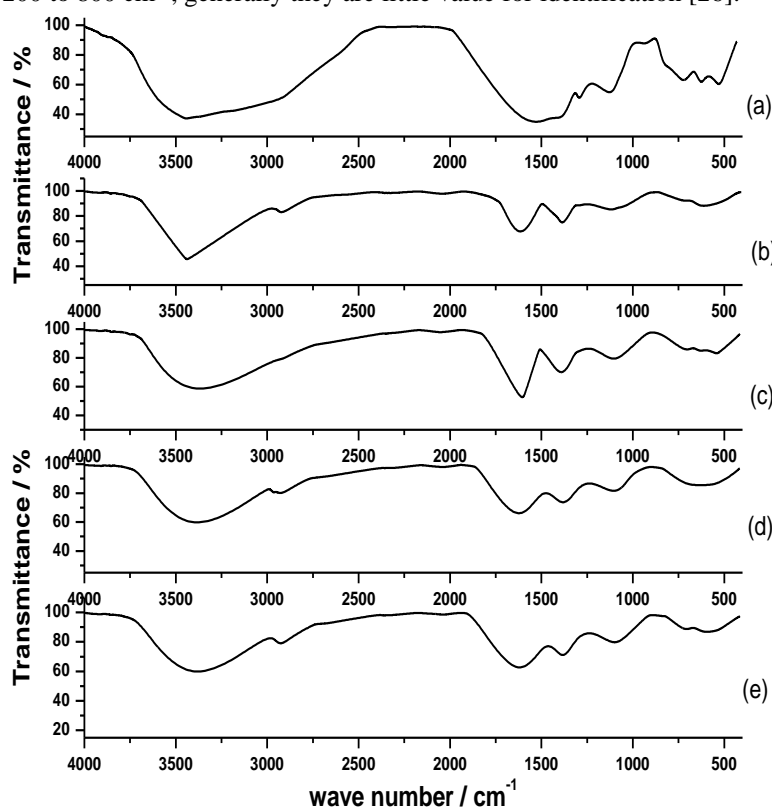
From table 1, one can find that the atomic weight percentage of cadmium in the crystalline lattice is higher than cobalt in the samples 3 to 5, while the atomic weight percentage of cobalt in the crystalline lattice is higher than cadmium in the sample 2.

The reason of higher atomic weight percentage of cadmium in the samples 3 to 5 may be due to the smaller hydrated radii of  $\text{Cd}^{+2}$  ions compared to  $\text{Co}^{+2}$  ions. In the solution,  $\text{Co}^{+2}$  and  $\text{Cd}^{+2}$  ions possess a primary hydration shell, which results into the formation of hydrated radius for those ions. The smaller ions having greater ionic potential attract more water molecules. In the present study, the non-hydrated radius of  $\text{Co}^{+2}$  is 0.65 Å and second ionization potential is 17.06 eV, while the same for  $\text{Cd}^{+2}$  are 0.97 Å and 16.1908 eV, respectively. Therefore, the reverse nature can be observed for hydrated radii of  $\text{Co}^{+2}$  and  $\text{Cd}^{+2}$  than that for the non-hydrated ones [23,24], resulting into higher concentration of Cd in grown crystals than Co.

The reason of higher atomic weight percentage of cobalt in sample 2 may be due to the highest volume concentration of cobalt nitrate tetrahydrate solution, which results into the very high concentration of  $\text{Co}^{+2}$  ion

and the electronic configuration of the element used. Cadmium ion has completely filled d orbital, therefore, it is a stable ion, while, cobalt ion has more than half filled d orbital and due to this it is unstable and possess the higher tendency to form compound. The highest concentration and higher tendency to form compound of  $\text{Co}^{+2}$  ion compared to  $\text{Cd}^{+2}$  ion result into the higher concentration of Co in grown crystals of sample 2 than Cd.

**FTIR Analysis:** The FTIR spectra are shown in the figure 2(a-e) for the samples 1 to 5, respectively. The allocation of functional groups corresponding to the absorption region is shown in the table 2. The absorption observed between  $3420$  to  $3445\text{ cm}^{-1}$  corresponds to hydroxy group, H-bonded O – H stretching vibrations [14,25] due to water of crystallization associated with the crystals, which confirms the hydrous nature of all the compounds. The bands observed nearly  $2960\text{ cm}^{-1}$  can be attributed to the asymmetrical C – H stretching vibrations of tartaric acid [13,25]. The C = O group stretching vibrations are observed between  $1603$  to  $1621\text{ cm}^{-1}$  [12,14,25]. The O – H bending vibrations are observed between  $1382$  to  $1385\text{ cm}^{-1}$  [14,25]. The bands observed between  $1046$  to  $1118\text{ cm}^{-1}$  are corresponding to the stretching vibrations of C – O bonding in the tartrate of the compound [14,25]. The out of plane bending vibrations of O – H are observed between  $710$  to  $720\text{ cm}^{-1}$  [25]. Absorption wave numbers between  $532$  to  $545\text{ cm}^{-1}$  are assigned for metal-oxygen vibrations. This analysis confirms the presence of the above mentioned functional groups and the vibrations corresponding to metal oxygen bonding. Absorption wave numbers below  $590\text{ cm}^{-1}$  are assigned for metal-oxygen vibrations [12,14]. The pure and mixed metal tartrate crystals also possess the C – C group and therefore, one can assign the C – C stretching vibrations also. But, the bands assigned to C – C stretching vibrations are weak and appear in the broad region of  $1200$  to  $800\text{ cm}^{-1}$ ; generally they are little value for identification [26].



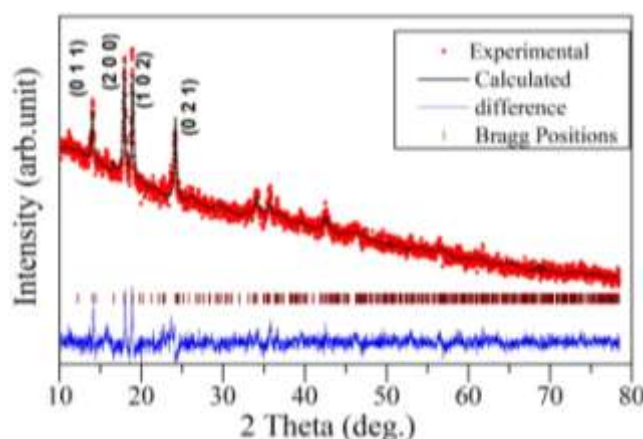
**Figure 2.** FTIR spectra of samples (1-5)

Comparing the different absorptions of mixed Co-Cd *levo*-tartrate crystals in FTIR spectra with the pure cobalt *levo*-tartrate crystals, one can conclude that there is no major effect of cadmium on the different absorptions in FTIR spectra. Atomic mass number and ionic radius of cobalt are 59 u and  $0.65\text{ \AA}$ , respectively, while, the same quantities of cadmium are 112 u and  $0.97\text{ \AA}$ , respectively. From table 2, it is observed that as the cadmium is added into the cobalt, the shifting of metal-oxygen stretching vibrations is found towards higher frequency, indicating high energy is required for vibrations. The presence of the more massive atom and larger ion is expected to cause alternations in the molecular geometry, bond length and mechanical vibrations and as a result, the absorption spectra are slightly altered. The variation in O – H stretching vibrations indicates that the metal ion is coordinated with water molecule through O – H bonding and having metal-oxygen bonding.

**Table 2:** Assignments of functional groups with frequency

Assignments	Wave number (cm <sup>-1</sup> )				
	Sample (1)	Sample (2)	Sample (3)	Sample (4)	Sample (5)
O – H Stretching	3443.73	3425.18	3439.73	3439.82	3424.11
Asymmetrical C – H Stretching	2962.22	2959.97	2961.71	2963.55	2958.94
C = O Stretching	1603.90	1616.29	1621.01	1616.27	1615.30
O – H banding In-plane	1382.55	1384.46	1385.20	1384.78	1384.54
C – O Stretching	1046.75, 1118.43	1046.36, 1084.54	1054.16, 1116.49	1055.29, 1115.47	1057.23, 1115.63
O – H bending out-of-plane	717.05	719.14	710.68	711.28	711.37
Co – O and Cd – O Stretching	532.82	539.12	543.37	543.50	544.21

**Powder XRD Analysis:** The Powder XRD data for the samples 1 to 5 were collected at room temperature using a Bruker AXS D8 Advance X-ray powder Diffractometer with a Si(Li) PSD detector for CuK<sub>α</sub> radiation  $\lambda = 1.5406 \text{ \AA}$ . The angular range of  $2\theta$  scan was from  $10^\circ$  to  $80^\circ$  with a step size of  $0.02^\circ$ . To elucidate the structure of all the samples 1 to 5, Rietveld refinement of powder diffraction pattern was carried out at IIT Kanpur using FULLPROF program. Figure 3(a) and 3(b) shows Rietveld refined experimental powder diffraction patterns of pure cobalt levo-tartrate crystals of sample 1 and cadmium mixed cobalt levo-tartrate crystals of samples 2 to 5. The same program was also used as a profile matching tool. The space group and the lattice parameters reported for the cadmium tartrate [7] and cobalt tartrate [12] and were used as starting lattice parameters. The difference profile shown in the figures after the Rietveld refinement indicates a quite reasonable fit of the XRD patterns, which is also confirmed by the lower values of  $\chi^2$  varying between 1.22 and 1.92 for all the samples. The pure crystals of cobalt levo-tartrate of sample 1 crystallize in orthorhombic structure with  $P2_12_12_1$  space group, while pure crystals of cadmium tartrate crystallize in monoclinic structure with space group  $P2_1$  [9]. When cadmium is added into the pure crystals of cobalt levo-tartrate, the formed mixed levo-tartrate crystals of cobalt and cadmium of samples 2 to 5 show the biphasic nature. In the figure 3, I and II indicate the peaks belong to orthorhombic phase of cobalt tartrate and monoclinic phase of cadmium tartrate, respectively. The lattice parameter values of mixed cobalt cadmium levo-tartrate crystals are listed in table 3.

**Figure 3(a).** Rietveld refined XRD pattern of sample 1

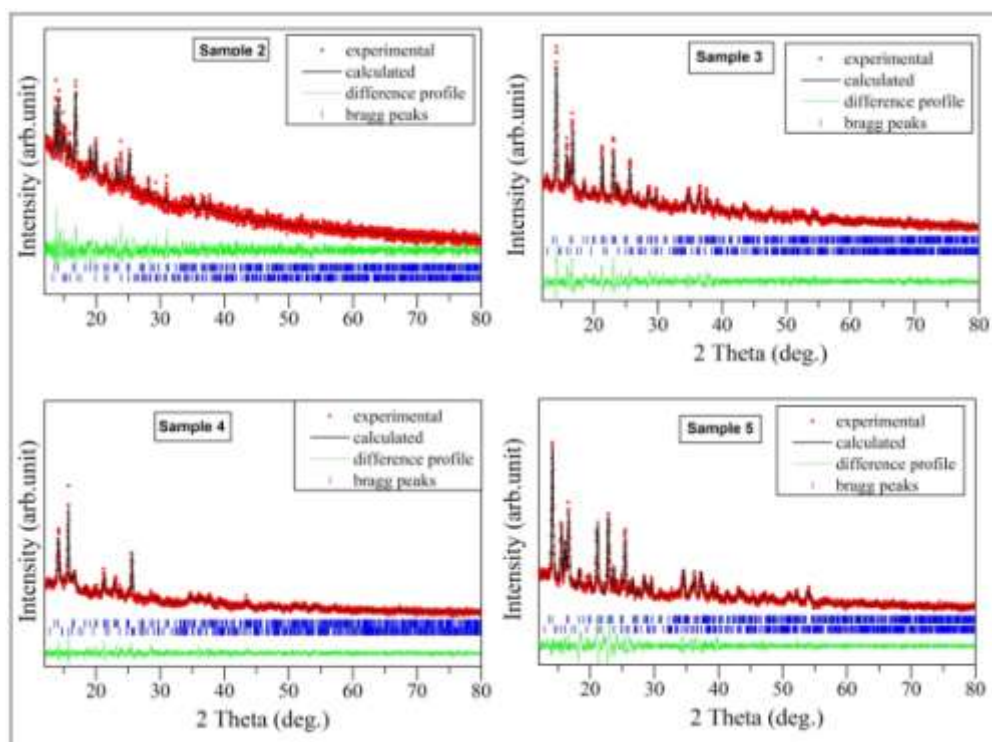


Figure 3(b). Rietveld refined XRD patterns of samples 2 to 5

Table 3: Lattice parameters, phase and system

Sample No	Unit cell parameters and crystal system						
	Phase I, Orthorhombic $\alpha = \beta = \gamma = 90^\circ$			Phase II, Monoclinic $\alpha = \gamma = 90^\circ$			
	a(Å)	b(Å)	c(Å)	a(Å)	b(Å)	c(Å)	$\beta$
1	9.8882	7.8445	10.6358	---	---	---	---
2	9.6866	8.2428	10.6251	6.2729	11.9513	7.5381	117.40
3	9.6478	8.2711	10.6298	6.0345	12.0715	7.6444	115.86
4	9.6896	8.2525	10.6477	6.0482	12.0793	7.6835	115.87
5	9.7601	8.2225	10.6791	6.1134	12.2103	7.6501	116.38

The change in diffraction peaks intensity indicates the presence of cadmium in the cobalt levo-tartrate. The peak intensity reflects the total scattering from each crystal planes and depends on the distribution of atoms in the crystal structure [27]. In general, if crystalline nature of the sample increases, then it results into the increase in number of planes orientated in particular direction. This results into the increase in multiplicity and lead to increase in intensity of diffraction peaks. In the present case, due to addition of cadmium the peak intensity is found to be decreased, indicating the loss of crystallinity due to lattice distortion. The effect of the presence of large size ion of cadmium compared to cobalt is that it not only causes the lattice distortion up to certain minor extent, but disturbs the entire lattice structure of cobalt which is reflected by change in the unit cell parameters of pure cobalt levo-tartrate as well as the biphasic nature of the mixed crystals of cobalt and cadmium levo-tartrate of samples 2 to 5. Further, it is observed that the peaks (200) and (021) of pure cobalt levo-tartrate are shifting slightly due to the addition of cadmium and also the FWHM increases due to the addition of cadmium, which may be due to the defect and generation of strain in the lattice of pure cobalt levo-tartrate.

**TG Analysis:** TG curves of all the samples 1 to 5 are shown in the figure 4(a-e), respectively.

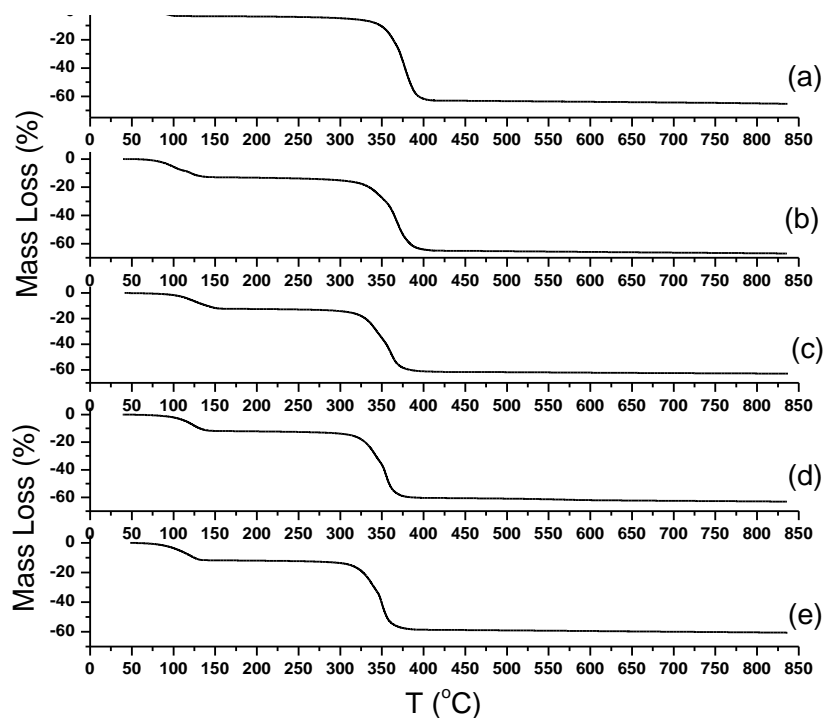


Figure 4. TG curves of samples (1-5)

The theoretical and experimental mass loss values with the reactions involved at each stage of decomposition of the samples are shown in the table 4.

Table 4: Theoretical and experimental mass loss (%)

Sample	Range of T in (°C)	Expected Reaction within temperature range shown	mass loss (%) Theoretical	mass loss (%) Experimental
Sample 1	40 to 76	Stable, No reaction	00	00
	76 to 92	$\text{CoC}_4\text{H}_4\text{O}_6 \cdot 0.35\text{H}_2\text{O} \rightarrow \text{CoC}_4\text{H}_4\text{O}_6 + 0.35\text{H}_2\text{O}$	03.00	2.70
	92 to 287	Stable, No reaction	00	00
	287 to 413	$\text{CoC}_4\text{H}_4\text{O}_6 \rightarrow \text{CoO} + \text{CH}_4 + 3\text{CO} + \text{O}_2$	64.86	63.00
Sample 2	40 to 67	Stable, No reaction	00	00
	67 to 134	$\text{Co}_{0.78}\text{Cd}_{0.22}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.8\text{H}_2\text{O} \rightarrow \text{Co}_{0.78}\text{Cd}_{0.22}\text{C}_4\text{H}_4\text{O}_6 + 1.8\text{H}_2\text{O}$	13.00	12.30
	134 to 285	Stable, No reaction	00	00
	285 to 410	$\text{Co}_{0.78}\text{Cd}_{0.22}\text{C}_4\text{H}_4\text{O}_6 \rightarrow \text{Co}_{0.78}\text{Cd}_{0.22}\text{O} + \text{CH}_4 + 3\text{CO} + \text{O}_2$	65.52	64.87
Sample 3	40 to 90	Stable, No reaction	00	00
	90 to 140	$\text{Co}_{0.44}\text{Cd}_{0.56}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.6\text{H}_2\text{O} \rightarrow \text{Co}_{0.44}\text{Cd}_{0.56}\text{C}_4\text{H}_4\text{O}_6 + 1.6\text{H}_2\text{O}$	11.00	10.07
	140 to 284	Stable, No reaction	00	00
	284 to 390	$\text{Co}_{0.44}\text{Cd}_{0.56}\text{C}_4\text{H}_4\text{O}_6 \rightarrow \text{Co}_{0.44}\text{Cd}_{0.56}\text{O} + \text{CH}_4 + 3\text{CO} + \text{O}_2$	60.64	60.54
Sample 4	40 to 81	Stable, No reaction	00	00
	81 to 133	$\text{Co}_{0.32}\text{Cd}_{0.68}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.5\text{H}_2\text{O} \rightarrow \text{Co}_{0.32}\text{Cd}_{0.68}\text{C}_4\text{H}_4\text{O}_6 + 1.5\text{H}_2\text{O}$	10.00	10.33
	133 to 288	Stable, No reaction	00	00
	288 to 377	$\text{Co}_{0.32}\text{Cd}_{0.68}\text{C}_4\text{H}_4\text{O}_6 \rightarrow \text{Co}_{0.32}\text{Cd}_{0.68}\text{O} + \text{CH}_4 + 3\text{CO} + \text{O}_2$	58.89	59.32
Sample 5	40 to 73	Stable, No reaction	00	00
	73 to 130	$\text{Co}_{0.16}\text{Cd}_{0.84}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.4\text{H}_2\text{O} \rightarrow \text{Co}_{0.16}\text{Cd}_{0.84}\text{C}_4\text{H}_4\text{O}_6 + 1.4\text{H}_2\text{O}$	09.00	09.77
	130 to 283	Stable, No reaction	00	00
	283 to 390	$\text{Co}_{0.16}\text{Cd}_{0.84}\text{C}_4\text{H}_4\text{O}_6 \rightarrow \text{Co}_{0.16}\text{Cd}_{0.84}\text{O} + \text{CH}_4 + 3\text{CO} + \text{O}_2$	56.80	58.80

All the samples 1 to 5 are water coordinated compounds; they first may lose the water molecules while heating. TG curves of figure (4 a to e) clearly show two stages, namely, the dehydration stage and the oxide stage. At the end of the first stage, all the samples 1 to 5 convert into anhydrous form and at the end of the second stage, they convert into oxide form. In the following paragraphs, both the stages are described.

Figure (4a) shows the thermo-gram of the pure cobalt levo-tartrate, i.e. sample-1, which indicates that the compound remains stable up to nearly 76°C and then slowly starts losing crystalline water and becomes anhydrous at nearly 92°C after losing approximately 03 % weight of its original weight. The anhydrous sample remains stable up to nearly 300°C and then start to decompose. This process of decomposition lasts up to nearly 413°C. During this stage of decomposition, the anhydrous sample is converted into oxide form with the loss of nearly 64 % of its original weight. The oxide sample remains stable up to the upper limit of the temperature under study, i.e. 837°C. From the analysis, it is found that 0.35 water molecules are associated with the crystal.

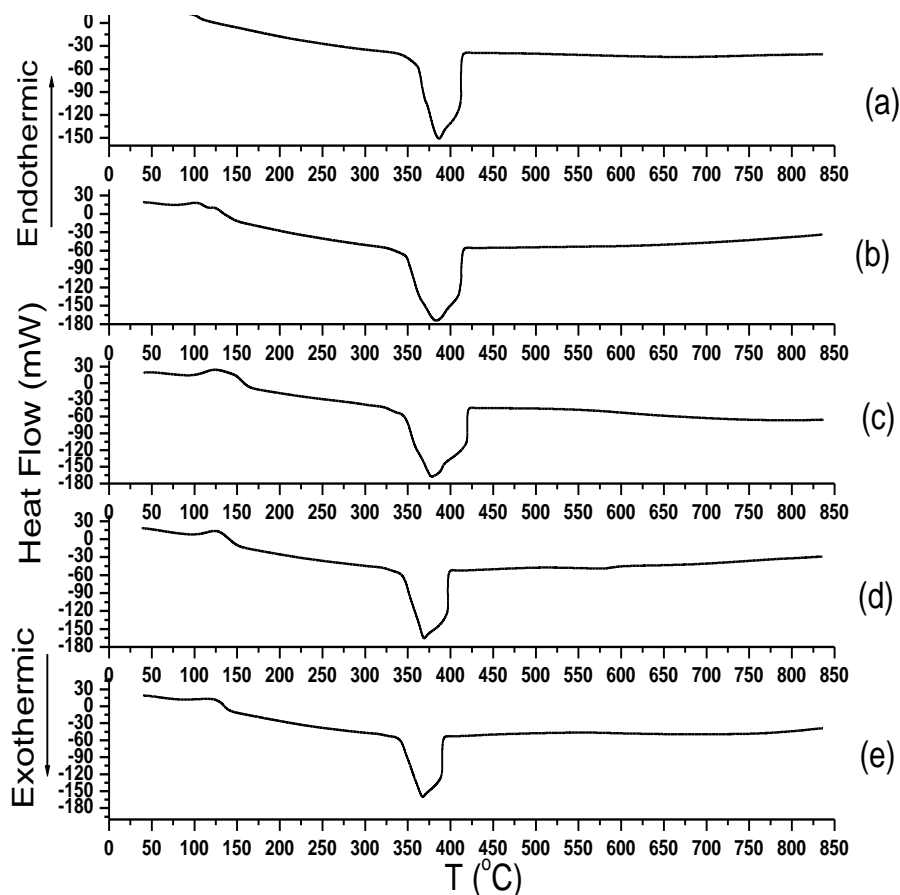
Figures (4 b to e) show the thermo-grams of the samples (2 to 5), respectively. The samples 2 to 5 remain stable up to the range of temperature 67°C to 90°C from the initial temperature, i.e., room temperature. Then all the samples 2 to 5 slowly start losing crystalline water and become anhydrous within the range of temperature 130°C to 140°C after losing approximately 13%, 11%, 10% and 9% weight of its original weight, respectively. All the anhydrous samples 2 to 5 remain stable up to the range of temperature 283°C to 285°C then start to decompose. During this stage of decomposition, all the anhydrous samples 2 to 5 are converted into oxide form with the loss of nearly 66%, 61%, 59% and 59% of its original weight, respectively. Then, all the oxide samples 2 to 5 remain stable up to the upper limit of the temperature under study, i.e. 837°C. The water molecules that are bounded with samples 2 to 5 have been calculated and found to be 1.8, 1.6, 1.5 and 1.4, respectively.

The correct stoichiometric formulas for all the samples 1 to 5 are listed in the table 5 after the EDAX and TGA study.

**Table 5:** The correct stoichiometric formula

Sample	The correct stiochiometric formula from TGA
Sample 1	$\text{CoC}_4\text{H}_4\text{O}_6 \cdot 0.35\text{H}_2\text{O}$
Sample 2	$\text{Co}_{0.78}\text{Cd}_{0.22}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.8\text{H}_2\text{O}$
Sample 3	$\text{Co}_{0.44}\text{Cd}_{0.56}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.6\text{H}_2\text{O}$
Sample 4	$\text{Co}_{0.32}\text{Cd}_{0.68}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.5\text{H}_2\text{O}$
Sample 5	$\text{Co}_{0.16}\text{Cd}_{0.84}\text{C}_4\text{H}_4\text{O}_6 \cdot 1.4\text{H}_2\text{O}$

**Differential Thermal Analysis:** DTA curves of all the samples 1 to 5 are shown in the figure 5(a-e), respectively, which show that the decomposition process of all the samples occurs through one endothermic reaction and one exothermic reaction. The DTA plot of figure (5a) indicates that the decomposition process of pure cobalt levo-tartrate (sample-1) occurs through one minor endothermic reaction at 92°C. When cadmium is added, the decomposition process still occurs through one endothermic reaction but at higher temperatures, i.e. at 104.83°C for sample-2, 129.80°C for sample-3, 126.92°C for sample-4 and 123.22°C for sample-5. The energy required to knock out the water molecules associated with the samples depends upon how strongly they are bounded in the lattice [28]. The destruction of water molecules at a higher temperature for cobalt cadmium mixed levo-tartrate crystals of samples 2 to 5 shows that water molecules are tightly bounded in the lattice of the mixed crystals compared to pure cobalt levo-tartrate crystals. Generally, broad endotherms signify dehydration reactions [29]. Here, endothermic peaks associated with the above mentioned temperatures for pure and mixed cobalt cadmium levo-tartrate crystals of samples 1 to 5 are attributed to the elimination of water molecules and conversion of samples into anhydrous form through the dehydration reactions.



**Figure 5.** DTA curves of samples (1-5)

Further, figure (5a) shows the decomposition process of pure cobalt levo-tartrate (sample-1) through one exothermic reaction at  $386.77^{\circ}\text{C}$ . When cadmium is added, the decomposition process still occurs through one exothermic reaction but at slightly lower temperatures, i.e., at  $383.46^{\circ}\text{C}$  for sample-2,  $378.03^{\circ}\text{C}$  for sample-3,  $369.39^{\circ}\text{C}$  for sample-4 and  $367.61^{\circ}\text{C}$  for sample-5. Generally, chemical reactions, particularly those of an oxidative nature give rise to exothermic peaks [29]. Here, exothermic peaks associated with the above mentioned temperatures for pure and mixed cobalt cadmium levo-tartrate crystals of samples 1 to 5 are attributed to the conversion of samples into oxide form. No additional peaks are observed in the DTA plot of figure (5 a to e) other than the peaks associated with the weight losses in the TGA curve of figure (4 a to e). So, there are no structural changes around the peaks [28]. Arora et al [4] has reported the strong exothermic peak of oxidative process at  $370^{\circ}\text{C}$  for the pure cadmium tartrate crystals. In the present case, when weight% of cadmium is increased in the grown crystals moving from sample-2 to sample-5 in the table (3.1), the temperature of exothermic reaction of oxidative process starts to decrease towards  $370^{\circ}\text{C}$  from  $386.77^{\circ}\text{C}$  of pure cobalt levo-tartrate. This shows the effect of cadmium in the pure cobalt levo-tartrate crystals.

### III. Conclusion:

Pure and mixed levo-tartrate crystals of cobalt and cadmium were successfully grown by single diffusion gel technique in silica hydro gel medium. The spherulitic and brown coloured crystals were grown in the gel medium. The EDAX analysis suggested that the atomic weight percentage of cadmium in the crystalline lattice is higher than cobalt in the samples 3 to 5, which is due to lower hydrated radius of  $\text{Cd}^{+2}$  ions compared to  $\text{Co}^{+2}$  ions, while the highest concentration and higher tendency to form compound of  $\text{Co}^{+2}$  ion compared to  $\text{Cd}^{+2}$  ion resulted into the higher concentration of Co in grown crystals of sample-2 than Cd. The FTIR spectra of the grown crystals indicated the presence of O-H, C-H, C-O, C=O functional groups with metal-oxygen vibrations. By applying the Rietveld analysis to the powder XRD patterns it is found that cobalt and cadmium



mixed levo-tartrate crystals possessed bi-phasic nature, i.e. orthorhombic phase of cobalt levo-tartrate and monoclinic phase of cadmium levo-tartrate. The introduction of cadmium in cobalt levo-tartrate not only produced the separate phase but produced strain in cobalt levo-tartrate and resulted into shifting in the major XRD reflections of cobalt levo-tartrate. From the thermo-grams, it was found that the crystals were thermally unstable. On heating, they became anhydrous and decomposed into metal oxides without passing through any intermediate stage of carbonate or oxalate. The presence of water molecules was detected and calculated. The exact stoichiometric formulations for mixed crystals were suggested. The DTA suggested the decomposition process of all the samples followed by one endothermic reaction and one exothermic reaction, representing dehydration and decomposition reactions, respectively.

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