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Synthesis and Characterisation of Teterkis (Hydrazine) Cadmium(II) Chloride Monohydrate Crystals

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Abstract

Single crystals of Tetrakis(hydrazine) cadmium(II) chloride monohydrate (TCCM) were synthesised and characterised. The crystals were synthesised by solvent evaporation technique at ambient temperature. The presence of different elements in the compound and their composition were confirmed by energy dispersive analysis of X-ray (EDAX) and elemental analysis (CHN). FTIR spectroscopy confirms the presence of functional groups and molecular structure has been confirmed by ^{1}H NMR spectrum of the grown crystal. The sharp Bragg peaks from powder X-ray diffraction pattern shows crystalline nature of the compound. Thermal properties of TCCM were examined by thermogravimetry (TG), differential thermal analysis (DTA) and low temperature differential scanning calorimetry (DSC). Second-harmonic generation (SHG) measurement on the powder samples does not exhibit SHG efficiency.

Keywords: *FT-IR; SHG.; Solvent evaporation; TCCM; Thermogravimetry.*

1. INTRODUCTION

Hydarazines are compounds of interest in both the chemical and pharmaceutical industry. Its derivatives can act as ligands for a number of metal ions and the complexes formed have a variety of applications. Now a days it has been used as new class of materials of solar cell applications (Ravindra *et al.* 1999; Serov and kwak, 2010; Schaffer *et al.* 2010). In this paper, we report the growth and characterization of a new inorganic material, tetrakis(hydrazine) cadmium(II) chloride monohydrate crystals (TCCM).

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2. EXPERIMENTAL DETAILS

Hydrazine hydrochloride and cadmium nitrate taken in equimolar ratio was used to synthesize tetrakis(hydrazine) cadmium(II) chloride monohydrate complex. Under the experimental condition, bright, transparent and colourless tetrakis(hydrazine) cadmium(II) chloride monohydratecrystals were obtained within 10-15 days as shown in Fig.1. The net chemical reaction is as follows:

$$4\,(N_2H_4.2HCl) + Cd(NO_3) \xrightarrow[\text{Medium}]{H_2O} [Cd\,(N_2H_4)_4]\,Cl_2 .H_2O + 2HNO_3 + 6HCl$$

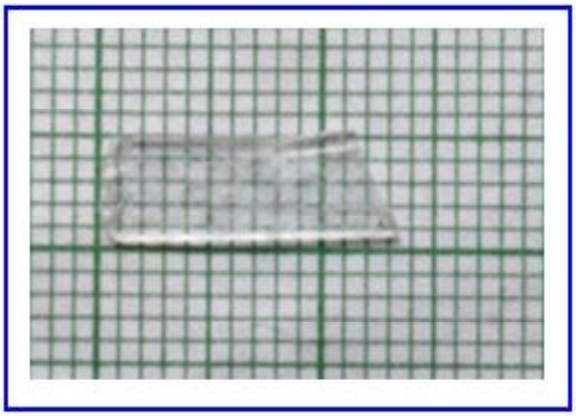


Fig. 1: Photograph of TCCM crystal

3. RESULTS & DISCUSSION

3.1 Elemental Analysis

The elemental analysis shows that the compound contains nitrogen: 33.99% (33.60) and hydrogen: 5.46% (6.32). Theoretical values are given in brackets.

3.2 Energy Dispersive X-Ray Spectroscopy

The EDX pattern of TCCM crystals which reveals the presence of cadmium, nitrogen, chlorine and oxygen. The presence of hydrogen was also detected.

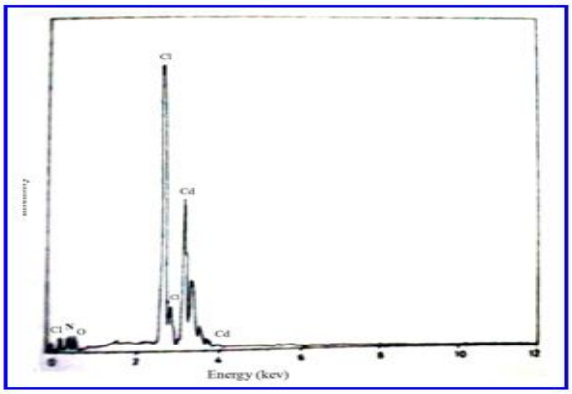


Fig. 2: EDX pattern of TCCM

3.3 Infrared spectroscopy

The FT-IR spectrum of TCCM crystalsis shown in the Fig.3. The frequency observed at 3519 cm^{-1} is due to the intermolecular hydrogen bonding as well as asymmetric O-H stretching vibration present inthe compound. The absorption frequency at 3321 cm^{-1} is due to symmetric O-H stretching vibration mode as well as N-H asymmetric stretching present in the compound (Vikram and Sivasankar, 2010).

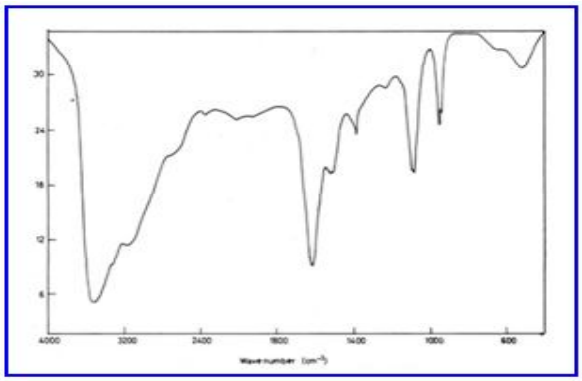


Fig. 3: FT-IR spectrum of TCCM

The intermolecular hydrogen bonding in the compound might be due to the presence of N-group in the compound as well as the presence of water of hydration in the crystal. The absorption frequency at 3173 cm^{-1} is due to the N-H symmetric stretching present in the compound. The frequency at 1611 cm^{-1} is due to NH_2 deformation in the compound. The frequency at 1085 cm^{-1} is due to H-O-H deformation. The absorption frequencies at 954 cm^{-1} is due to N-N stretching in the compound [4]. The absorption frequency at 511 cm^{-1} is due to Cd –N bonding. The absorption frequencies at 2360, 1381 and 1226 cm^{-1} could not be accounted for (Heaton *et al.* 1996).

3.4 NMR spectroscopy

The ^1H NMR spectrum of TCCM is shown in the Fig.4. The chemical formula of the compound is

$[Cd(N_2H_4)_4]Cl_2.H_2O$. The ^1H NMR spectrum exhibits a single proton signal at δ 4.74 ppm indicating the presence of N-H proton in the compound. In general, hydrazine complexes are expected to exhibit two signals one at 2 ppm range and the other one at 4ppm range in $CDCl_2$ and methanol solvents.

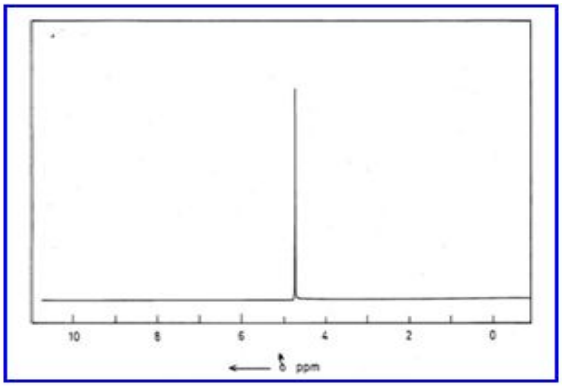


Fig. 4 : ^1H spectrum of TCCM

3.5 Low Temperature Digital Scanning Calorimetry

Low Temperature DSC curve is shown in the Fig.5. The curve recorded in the cooling run from RT to -70 °C. The cooling run from -70 °C to RT shows that there is no phase transition in the compound.

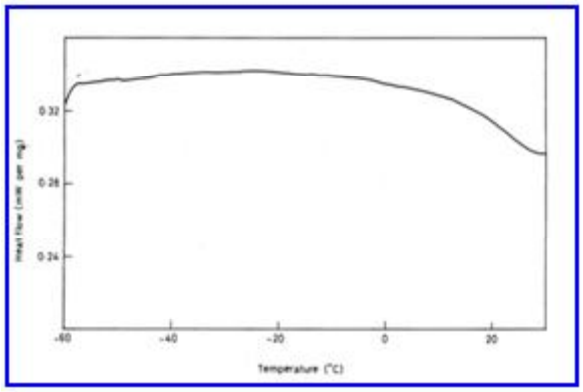
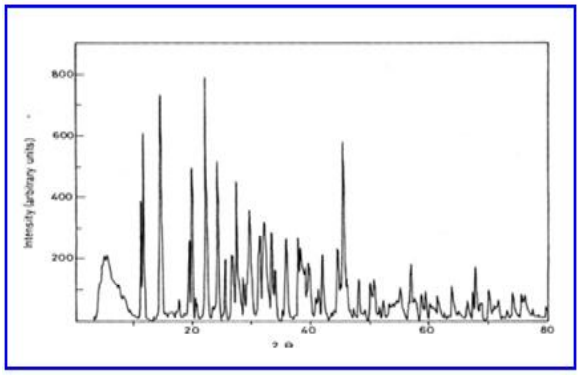


Fig. 5: Low temperature DSC of TCCM

3.6 Powder X-ray diffraction

The powder X-ray diffraction pattern of the TCCM crystal is shown in the Fig.6. The sharp and well defined Bragg peaks in the powder XRD pattern confirm the crystalline nature of the compound (Cheetham, 1987). The compound crystallizes in tetragonal system. The unit cell parameters are a=17.980070 Å, b=17.980070Å and c=10.806860Å. The values of $\alpha = \beta = \gamma = 90^o$. Volume of the unit cell is 3493.67$Å^3$.



Fi. 6: Powder X-ray diffraction of TCCM

3.7 Non Linear Optical Property

The TCCM crystal in the powder form when tested with Nd: YAG laser of wavelength 1.06 µm did not give any green signal. Hence the compound does not possess any SHG property (Balasubramaniam *et al*. 2010).

4. CONCLUSION

Single crystals of TCCM were grown from saturated solutions by slow evaporation method at room temperature. The crystals are characterised through Elemental analysis, EDAX, FT-IR, ^1H, low temperature DSC, powder XRD and NLO testing.Elemental analysis and EDX of the compounds confirm the proposed

chemical makeup of the compounds. The presence of one water molecule and characteristic chemical bonds present in the compounds are confirmed through various absorption frequencies in FTIR spectra. Presence of protons present in the crystals was confirmed by proton nuclear resonance spectroscopy. There was no phase transition observed in TCCM and there is no SHG effect in TCCM crystals. The sharp and well defined Bragg peaks obtained with specific 2θ angles for both crystals confirm the crystalline nature of the compound.

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