**Special Issue**
**Journal Of Basic And Applied Sciences**

# XRD And TGA-DSC as an Effective tools of Research in Physical Science

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**ABSTRACT**

X-ray diffraction (XRD) is an effective method for determination of the phase composition of unknown crystalline and amorphous materials. The scattering of X-rays by crystal atoms, produce a diffraction pattern that yields information about the structure of the crystal. This data is represented in a collection of single-phase X-ray powder diffraction are used for detarination of interplanar spacings (D), relative intensities (I/Io), particle size and shape, crystal structure of an unknown material. The TGA can be used to obtain the difference of the mass during this process is measured. The DSC can be used to obtain the thermal critical points like melting point, enthalpy specific heat or glass transition temperature of substances

## INTRODUCTION

The techniques of X-ray diffraction analysis are used to study, for example, metals, alloys, minerals, inorganic and organic compounds, polymers, amorphous materials, liquids, gases, and the molecules of proteins and nucleic acids [1-5] The X-ray diffractogram is an important tool for determination of the crystal structure of a compound. Diffraction pattern can be described in terms of three dimensional arrangement of lattice points. The smallest three dimensional geometric array of points from which a crystal can be created is called a unit cell. It is possible to diffract X-rays by means of crystals because the wavelength of X-rays is of about the same order as the inter-atomic distances in a crystal. The X-rays interact with inner most electronic cloud in the atoms and provides information about internal arrangement of the atoms in the crystal. [6-7] The intensity of diffracted beam is related to the structure factor. The atomic distribution in the sample can be determined by interpreting the XRD patterns and it is a direct tool for assigning the crystal structure to the complexes.

Differential thermal analysis (DTA) are widely used in qualitative evaluation of various compounds. In view of the possibilities offered by such methods in the investigation of both physical phenomena (melting, sublimation, adsorption, change in crystallographic properties etc.) and chemical phenomena (dehydration, decomposition, oxidation, etc.) method of thermal analysis have found applications in almost all the natural sciences. The DSC can be used to obtain the thermal critical points like melting point, enthalpy specific heat or glass transition temperature of substances.

### Powder X-ray diffraction:

X-ray diffraction is now a common technique for the study of crystal structures and atomic spacing. X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced.

When the geometry of the incident X-rays impinging the sample satisfies the Bragg Equation, $n\lambda = 2d\sin\theta$ constructive interference occurs and a peak in intensity occurs. This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of $2\theta$ angles,

all possible diffraction directions of the lattice should be attained due to the random orientation of the powdered material. Conversion of the diffraction peaks to d-spacing allows identification of the mineral because each mineral has a set of unique d-spacing. Typically, this is achieved by comparison of d-spacing with standard reference patterns.

The X-ray diffraction is a powerful method to understand the structure of the compound.29 X-ray method, can determine the structure and symmetry properties of complexes. It gives information of inter atomic distance, bond angles and electronic arrangement in a complex.

**Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA)**

The thermal gravimetric analysis (TGA) and the differential scanning calorimetry (DSC) are methods to investigate the thermal characteristics of substances like polymers.

**TGA:**

The schematic principle of the TGA measurement is shown in Figure 1. The sample is heated under nitrogen or synthetic air with constant heat rate while the difference of the mass during this process is measured. A mass loss indicates that a degradation of the measured substance takes place. The reaction with oxygen from the synthetic air for example could lead to an increase of mass.

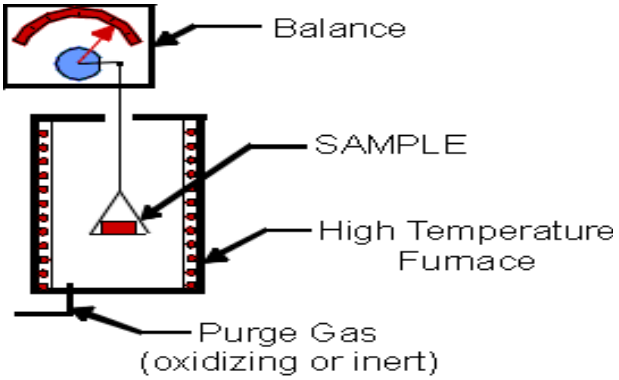


**Fig 1: Schematic principle of TGA measurement.**

**DSC:**

The DSC can be used to obtain the thermal critical points like melting point, enthalpy specific heat or glass transition temperature of substances. The schematic principle of the DSC is described in Figure 2. The sample and an empty reference crucible is heated at constant heat flow. A difference of the temperature of both crucibles is caused by the thermal critical points of the sample and can be detected.

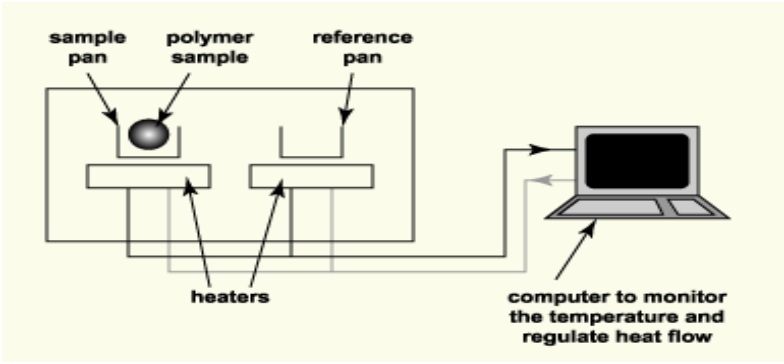


**Fig 2: Schematic principle of DSC measurement.**

The information obtained from DTA and DSC is very similar. However DTA can be used up to higher temperatures and the quantitative data obtained from DSC are more reliable. Both DTA and DSC are often used for fingerprint comparison of the results obtained from a sample with those of reference material

**4.0 XRD Studies of Sm(III) Complex**

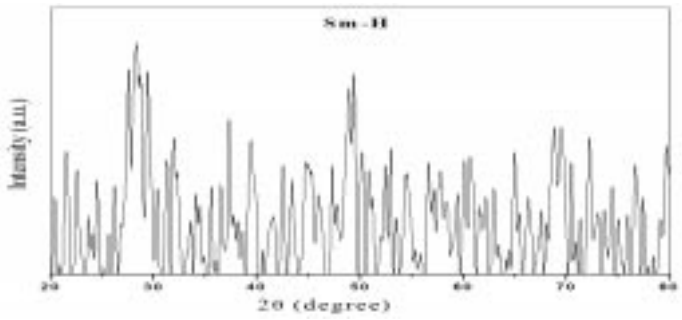


**Fig.3: X-ray diffraction of Sm(III) Complex**

The X-ray diffraction of representative metal complexes was scanned in the range 20-80º at wave length 1.540Å. The diffractogram and associated data depict the 2θ value for each peak, relative intensity and inter-planar spacing (d-values). The diffractogram of Sm(III) complex had 22 reflections with maxima at 2θ= 49.372º and its intensity 113.72 a.u. corresponding to d value 1.844 Å. The X-ray diffraction pattern of these complexes with respect to major peaks having relative intensity greater than 10% has been indexed by using computer programme. The above indexing method also yields Miller indices (hkl), unit cell parameters and unit cell volume. The unit cell of Sm(III) complex yielded values of lattice constants, a=8.21 Å, b = 8.21 Å, c = 6.47Å and unit cell volume V= 436.104Å3 In concurrence with these unit cell parameters, the conditions such as a = b≠ c and $\alpha = \beta = \gamma = 90$ required for sample to be tetragonal were tested and found to be satisfactory. Hence it can be concluded that Sm(III) complex have tetragonal crystal system. [9]

**4.0 TGA- DSC Studies of Sm(III) Complex**

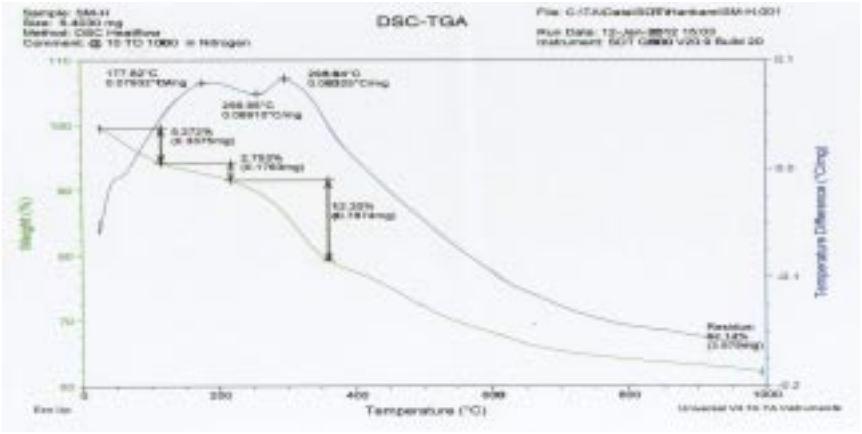


**Fig.4: TGA- DSC analysis of Sm(III) Complex**

The simultaneous TGA-DSC analysis of metal complexes was studied from ambient temperature to 1000 ºC in nitrogen atmosphere using α-Al2O3 as reference. The lattice water is removed in the temperature range of 55-110 ºC and the ionic nitrates are removed in the 178-360 ºC range. The macrocycle is lost in the temperature range of 361- 825 ºC along with the coordinated nitrate [8]. Samarium(III) complexes in which three lattice water and one coordinated water molecules are removed with mass lose of 7.5 % (calcd.7.15%) between 50-190 ºC and one ionic nitrate ion is removed with loss of 6.9% (calcd.6.24%) between 190-298 ºC. An endothermic peak in the range 170-180 ºC (λmax 177.8 ºC) on the DSC curve corresponds to the dehydration step and second endothermic peak in the range 295-305 ºC (λmax 298 ºC) corresponds to the denitration step. The

macrocycle is lost in the temperature range of 298-820 ºC along with the coordinated nitrate. The mass of final residue corresponds to stable Sm2O3, 62.14% (calcd. 59.17%).[9]

## CONCLUSION

XRD Technique is Non-destructive, fast, easy sample preparation,We can determine interplanar spacings (D), relative intensities (I/Io), particle size and shape, crystal structure of an unknown material This Technique is used Single crystal, poly, and amorphous materials. The information obtained from DTA and DSC is very similar. However DTA can be used up to higher temperatures and the quantitative data obtained from DSC are more reliable. Both DTA and DSC are often used for fingerprint comparison of the results obtained from a sample with those of reference material and determination of water molecules, Nitrate,Sulphate,Carbonate ions according to their percentage mass loss.

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