

# 1: Thermal Analysis

## Learning Objectives

After studying this chapter, you should be able to:

- List the different thermal methods of analysis.
- Define the major methods of thermal analysis.
- Identify endothermic and exothermic transitions.

Thermal analysis refers to the group of methods in which some physical property of the sample is continuously measured as a function of temperature, whilst the sample is subjected to a controlled temperature change.



### Brain Teaser

Can you write down one similar method, used to characterize solid organic compounds in particular, which depends upon a thermal technique?

Have you guessed it? It is Melting point determination.

The effect of heat can be wide ranging and cause changes in many properties of a sample. In thermal analysis, changes in weight form the basis of Thermogravimetry (TG) while measurement of energy changes form the basis of Differential Thermal Analysis (DTA) and of Differential Scanning Calorimetry (DSC). These techniques are the most important in thermal analysis. Thus, for example, TG tells us when a sample is losing weight (and how much) while DTA or DSC will tell us if that reaction is exothermic or endothermic (and often capable of measuring heat change). These important techniques can be applied to the study of almost any substance. Apart from these, there are some other thermal methods which are listed below:-

**Thermomechanical Analysis (TMA):** Dimensional changes as a function of temperature.

**Thermoacoustimetry:** Characteristics of imposed sound waves produced as the material being heated.

**Thermopotometry:** Study of an optical characteristic of a sample as it undergoes a thermal programme.

**Electrothermal analysis:** Study of electrical conductivity as a function of temperature.

**Thermomagnetometry:** Study of variation in a magnetic property of a material with temperature.

**Activity 1.1:** It is said that ‘**immediate recall, aids retention**’, so

We have briefly discussed about eight kinds of thermal effects which can occur when a sample is subjected to a controlled temperature programme. Write down as many of these eight effects as you can in the left hand column opposite the name of the corresponding thermal method given in the right hand column.

Fill in the thermal effect/change that is measured in the methods mentioned.

Thermal effect/change	Name of thermal method
	TG
	TMA
	DTA
	Thermoacoustimetry
	Thermopotometry
	Electrothermal Analysis
	Thermomagnetometry

(Dodd & Tonge, 2008)

**Activity 1.2:** Match the following:

Technique	Quantity Measured
1)DSC	a) Weight change
2)DTA	b) Heat and temperature of transition and reactions.
3)EGA	c)Temperature of transitions and reactions.
4)TG	d) Amount of gaseous products of thermally induced reactions.

**Answers:** 1)\_\_\_\_ 2)\_\_\_\_ 3)\_\_\_\_ 4) \_\_\_\_

(Dodd &Tonge, 2008)

Let us now learn in detail about some of the frequently used thermal methods. These are listed below:

**Thermogravimetry (TG/TGA):** TG or TGA is a technique in which the weight of a sample is measured as a function of temperature, whilst it is subjected to a controlled heating programme.

**Derivative Thermogravimetry (DTG):** DTG is a method of expressing the results of TG by giving the first derivative curve as a function of temperature or time.

**Differential Thermal Analysis (DTA):** DTA is a technique in which the difference in temperature ( $\Delta T$ ) between the sample and an inert reference material, is measured as a function of temperature under controlled heating.

**Differential Scanning Calorimetry (DSC):** DSC is very similar to DTA and gives much the same sort of information but DSC is more often used for quantitative measurement of energy changes.

**Evolved Gas Detection (EGD):** EGD is a technique in which the evolution of gas from a sample is detected, as a function of temperature, whilst the sample is subjected to controlled thermal programme.

**Evolved Gas Analysis (EGA):** EGA is a technique whereby the volatile products, released by a sample on decomposition, may be analysed as the sample is heated according to controlled thermal programme. (Dodd & Tonge, 2008)

**Pre-requisite:**

Before we deep dive into some thermal methods it is essential to check our understanding about the concept of exothermic/endothermic and different transitions accompanied by weight loss or weight gain.

Let us consider an example of different states of water and their inter-conversion,

Ice<sub>(s)</sub> ↔ Water<sub>(l)</sub> ↔ Steam<sub>(g)</sub>

In each of the above transitions can you tell which one is an endo/exothermic transition? And is there any change in weight during these transitions?

For further explanation please see Introductory Chemistry in *Libretexts*

[https://chem.libretexts.org/Bookshelves/Introductory\\_Chemistry/Introductory\\_Chemistry/03%3A\\_Matter\\_and\\_Energy/3.09%3A\\_Energy\\_and\\_Chemical\\_and\\_Physical\\_Change](https://chem.libretexts.org/Bookshelves/Introductory_Chemistry/Introductory_Chemistry/03%3A_Matter_and_Energy/3.09%3A_Energy_and_Chemical_and_Physical_Change)

Now that you have some idea about endothermic and exothermic transitions, try activity 1.4

**Activity 1.4:** Can you assign which of these processes will be endothermic/exothermic?

**Endothermic / Exothermic transitions (please tick)**

Phenomenon	Exothermic	Endothermic
Adsorption		
Desorption		
Fusion (melting)		
Vaporization		

Decomposition		
Dehydration		

(Dodd & Tonge, 2008)

**Activity 1.5:** Can you assign which of these processes will be endothermic/exothermic and accompanied by weight loss or weight gain. **(Please tick)**

Phenomenon	Weight gain	Weight loss	Endothermic	Exothermic
Melting				
Adsorption of gas				
Desorption of gas				
Vaporisation				
Dehydration				
Decomposition				
Sublimation				

(Dodd & Tonge, 2008)

Now that we have an idea about the exothermic, endothermic, weight gain and weight loss process it is important to note that these transitions can be either physical or chemical transitions.



#### Brain Teaser:

How might you study a sample which, on heating, first melts and then at a later stage decomposes with loss of weight?

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