6: Self Assessment Activities- Answer Key

Activity 1.1:

Thermal effect/change	Name of thermal method
Weight	TG
Dimension	TMA
Energy (difference in temperature)	DTA
Acoustic property	Thermoacoustimery
Optical property	Thermoptometry
Electrical conductivity	Electrothermal Analysis
Magnetic property	Thermomagnetometry

Activity 1.2:

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

Activity 1.4:

Phenomenon	Exothermic	Endothermic
Adsorption	✔	
Desorption		✔
Fusion (melting)		✔
Vaporization		✔
Decomposition	✔	✔
Dehydration		✔

Activity 1.5:

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

Activity 2.1:

Phenomenon	Physical	Chemical
Adsorption	✔	
Dehydration		✔
Desorption	✔	
Fusion (melting)	✔	
Chemisorption		✔
Vaporization	✔	
Decomposition		✔
Redox reactions		✔
Reduction in gaseous atmosphere		✔

Activity 2.2:

a. The part of the TG curve where the mass is essentially constant – Plateau AB

b. The temperature at which cumulative mass change reaches a magnitude that the

thermobalance can detect -Point B

c. The temperature at which the cumulative mass change reaches a maximum-Point C

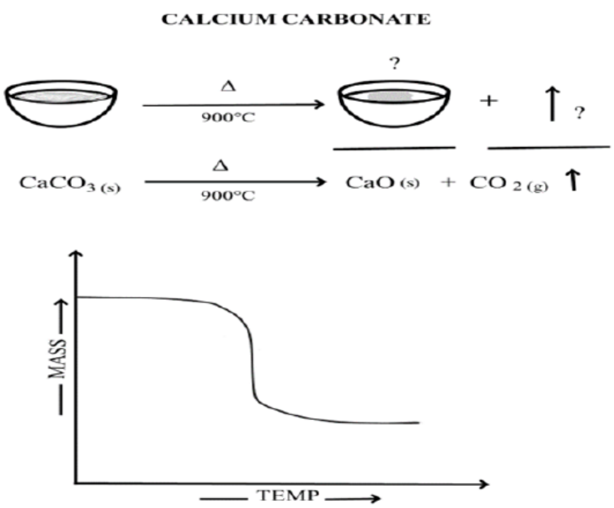
Activity 2.3:

Phenomenon	Type
Sublimation	Y
Adsorption of gas	X
Desorption of gas	Y
Vaporisation	Y
Dehydration	Y
Decomposition	Y
Melting	None

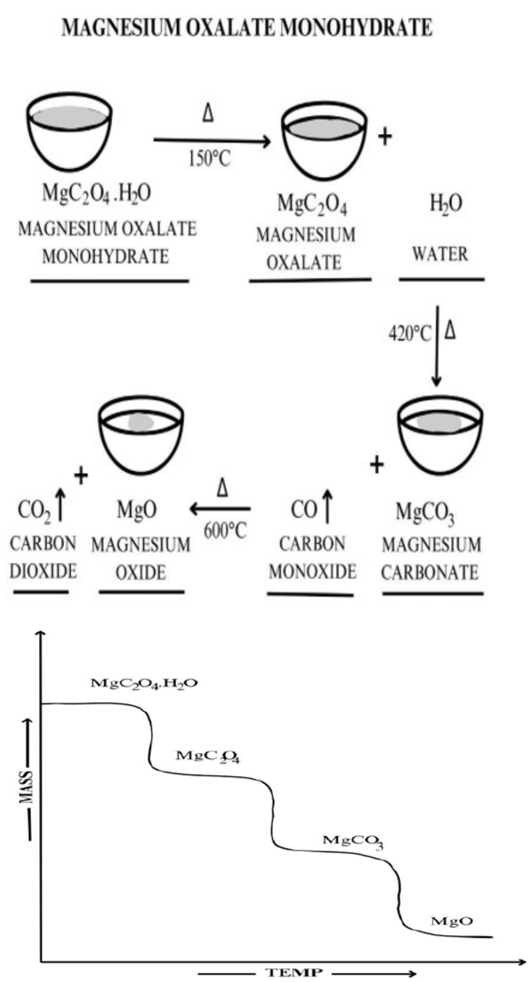
Activity 2.4:

1. A derivative thermogravimetric (DTG) curve represents a plot of mass of sample, as a function of temperature. **F**
2. A point of inflection on a thermogravimetric (TG) curve will correspond to a minimum on the DTG curve for a given chemical decomposition. **T**
3. A ‘plateau’ on a TG curve will not necessarily correspond to a zero ordinate value of the DTG curve. **F**
4. Thermal decomposition which overlap are often indicated more clearly by DTG curves than by the corresponding TG curves. **T**

Activity 2.5:

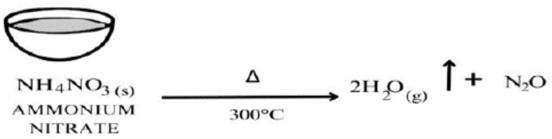


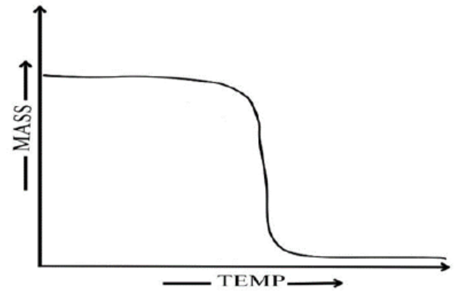
Activity 2.6:



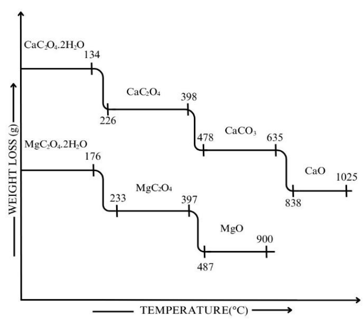
Activity 2.7:

AMMONIUM NITRATE





Activity 2.8: Will you be able to construct separate decomposition curves for calcium oxalate dihydrate and magnesium oxalate dihydrate based on the above application.



Activity: 2.10

1. Ans: a

$Ca(OH)_{2(s)} \rightarrow CaO_{(S)} + H_2O_{(g)}$

74.1g 56.1g 18g

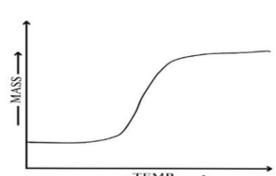
% weight loss = $\frac{74.1 - 56.1}{74.1}$ x 100 = **24.3%**

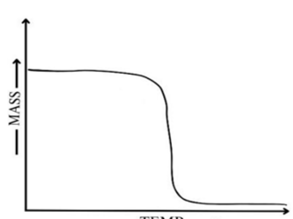
Ans: b

$6PbO_{(s)} + O_{2(g)} \rightarrow 2Pb_3O_{4(S)}$

1339.2 g 1371.2 g

% weight gain = $\frac{1371.2 - 1339.2}{1339.2}$ x 100 = **2.4%**





TG for ‘b’ TG for ‘a’

2. Ans:

The decrease in weight corresponds to the amount of carbon dioxide lost due to the decomposition of calcium carbonate present in the mixture as per the following reaction:

$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)}\uparrow$

Weight loss = 250.6-190.8 = 59.8 mg

1mol of $CaCO_3$ ≡ 1 mol of CO_2

100.1mg of $CaCO_3$≡ 44mg of CO_2

? ≡ 59.8 mg of CO_2

$= \frac{100.1 \times 59.8}{44}$ = 136.05 mg of $CaCO_3$

Weight of the sample = 250.6 mg = mixture of $CaCO_3$ &CaO

250.6 mg of mixture = 136.05 mg of $CaCO_3$

100 mg of mixture = $\frac{136.05 \times 100}{250.6}$

= **54.29%** of $CaCO_3$

3.Ans: $CaC_2O_4.H_2O_{(s)} \rightarrow CaC_2O_{4(s)} + H_2O\uparrow$

$CaC_2O_{4(s)} \rightarrow CaCO_{3(s)} + CO_{(g)}\uparrow$

$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)}\uparrow$

ii. Calculate the % weight loss at each step.

% weight loss = $\frac{initial\ weight - final\ weight}{initial\ weight}$ x 100

% weight loss at step 1 = $\frac{17.61 - 15.44}{17.61} x\ 100$ = **12.32**

% weight loss at step 2 = $\frac{15.44 - 12.06}{15.44} x\ 100$ = **21.89**

% weight loss at step 3 = $\frac{12.06 - 6.76}{12.06} x\ 100$ = **43.95**

Activity 3.1:

The record shown is that of a **DTA** experiment since the **ordinate** plot ΔT which is a **differential** temperature. The **downward** direction of the peak indicates that a **endothermic** reaction has occurred. This in turn implies that the corresponding **enthalpy** change (ΔH) must have been **positive** ie the value of enthalpy **after** the thermal effect was **greater** than its value **before**. This means that the sample **took in** heat during the reaction. Furthermore, there is evidence of a change in the **heat capacity** as the temperature is increased beyond the thermal transition. This is shown by the **displacement** of the **baseline** just beyond the end.

Answers: Select from the following list

[upward/**downward**, free energy/**heat capacity**, **greater**/less, DTG/**DTA**, **base-line**/background, derivative/**differential**, **took in**/gave out, negative/**positive**, **enthalpy**/entropy, **before/after**/during, exothermic/**endothermic**/isothermal, abscissa/**ordinate**, distortion/**displacement**.]

Activity 3.2:

Yes, they do offer an explanation. The curve for polyester shows that it melts at 255°C. Molten polymer in contact with the skin is a real hazard. Cotton, on the other hand, does not melt. It decomposes at 345°C.

Activity 4.1: Read each of the statements and mark them as **True or False**

(i) In DSC the sample and reference positions are provided with their own separate heating sources, so that the assembly may be operated on a ‘null balance’ basis. **T**

(ii) In DTA the equipment is so designed that the temperature of the sample is equal to that of the reference material at every point in the heating programme. **F**

(iii) Chemical decompositions which give rise to weight changes may be detected by DTA and DSC. **T**

(iv) The main components of a conventional differential thermal analyser consist of following: **F (programmer missing)**

a) The sample/reference holder

b) The thermocouple

c) The furnace

d) The amplifier

e) The recorder

Activity 4.2:

If $\Delta H < 0$, the system has undergone an endothermic/ **exothermic** change which means

$T_s > T_R$ [Choose the correct option: =, <, >]

Conversely $\Delta H > 0$ means **endothermic** change and $T_s < T_R$ [Choose the correct option: =, <, >]

In order to keep $\Delta T = 0$ [$\Delta T = T_s - T_R$]

● In case of an endothermic reaction we must provide heat to **sample**/reference.

● In case of an exothermic reaction we must provide heat to sample/**reference**

Activity 4.3: Observe the DSC curve given in 'figure **4.3a**.' and answer the following questions.

➢ How many transitions are seen in the above diagram?

Ans: Two

➢ Do you see any endotherm or exotherm? If yes, how many?

Ans: One endotherm and one exotherm.

➢ What does the endo or exothermic nature of the transition tell you about transition or ΔH value?

Ans: Endotherm: + ve ΔH (heat is absorbed) Exotherm: -ve ΔH (heat is given out)

➢ Do you see a shift in the baseline post glass transition temperature?

Ans: Yes, due to change in heat capacity

➢ Is the DSC curve of PET useful in predicting stability of the polymer? Justify your answer.

Ans: Yes, the melting point of any polymer is an important characteristic. It is the minimum temperature for processing the polymer and maximum temperature for using it. In addition, the glass transition temperature is useful since beyond this there is change in some of the physical properties of the polymer.
