

# Thermo Gravimetric Analysis - Instrumentation

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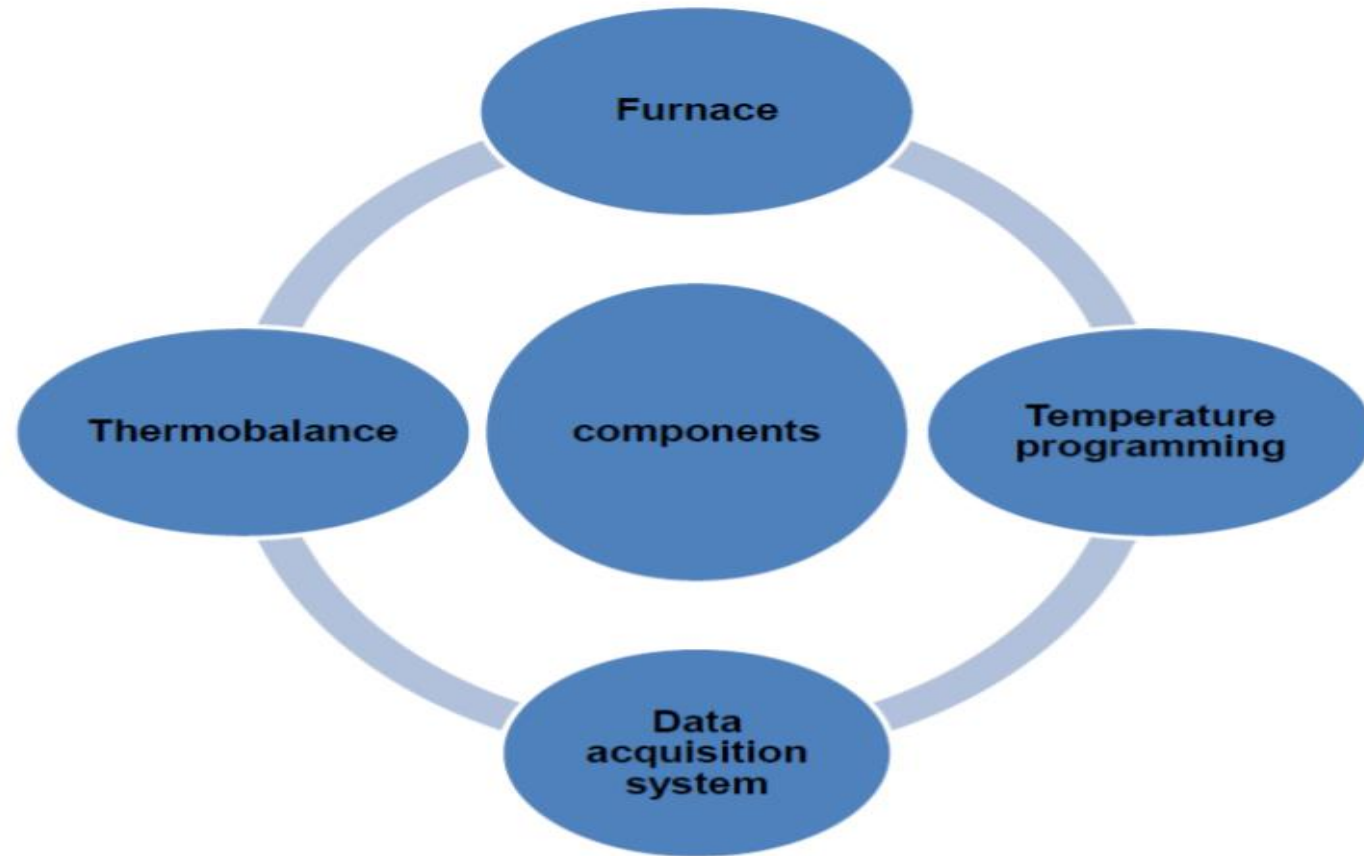
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# Contents...

- TGA - Instrumentation
- Components of a TGA instrument
- TGA Analyzer functions
- TGA – Experimental procedure
- Factors affecting TGA curve
- TGA - Calibration

# TGA Components

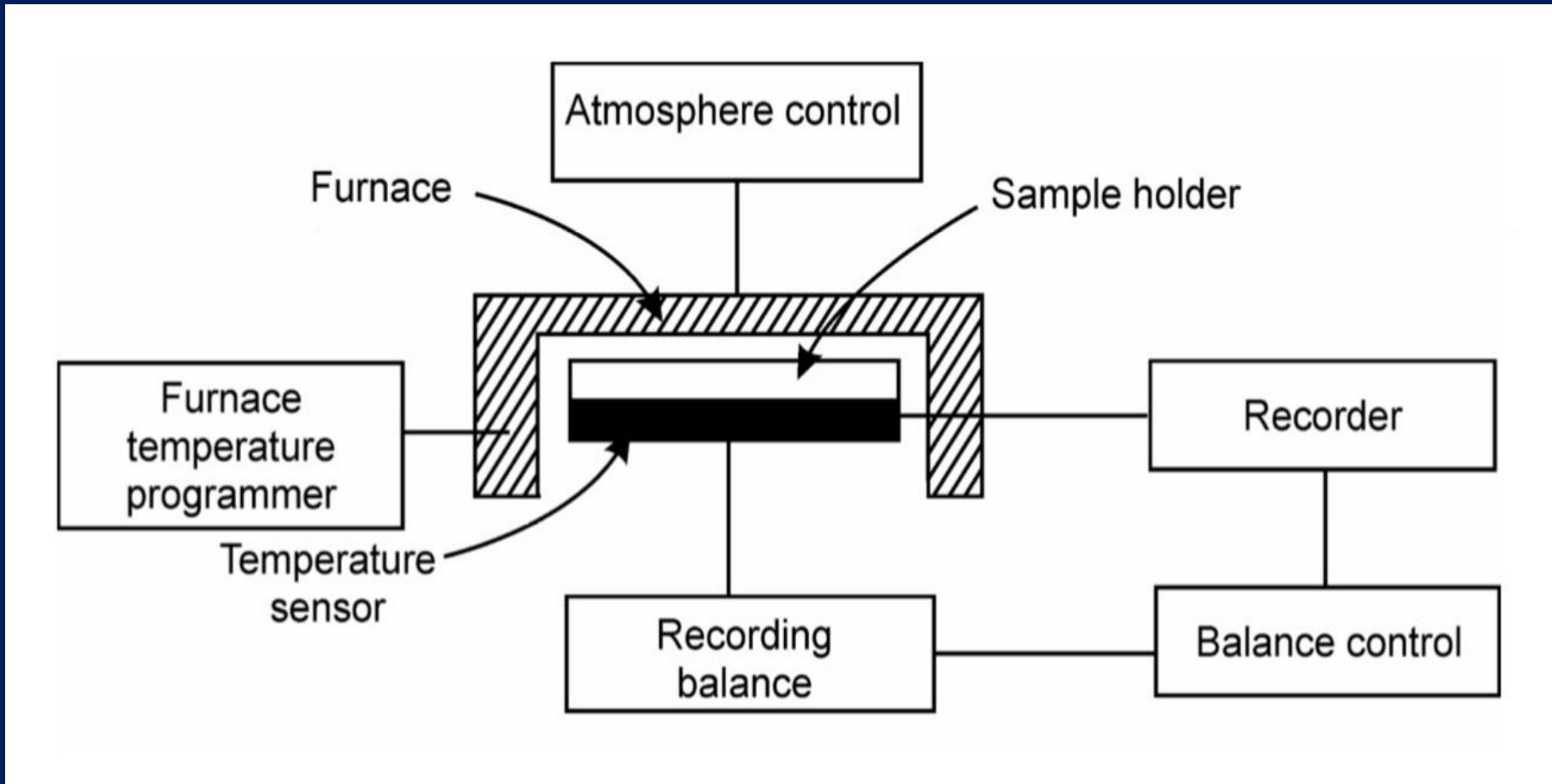


# Components of TGA...

A TGA instrument consists of,

1. A Thermo balance
2. A Sample container
3. A Furnace
4. Temperature programmer
5. Data Acquisition system

# Block diagram of TGA



# TGA Analyzer - Functions

A TGA analyzer must accurately,

- Control heating rate
- Measure the change in temperature
- Measure the change in mass of a sample

# Thermobalance...

A Thermobalance consists of ,

- an electronic micro balance
- a furnace
- a temperature programmer and
- a recorder

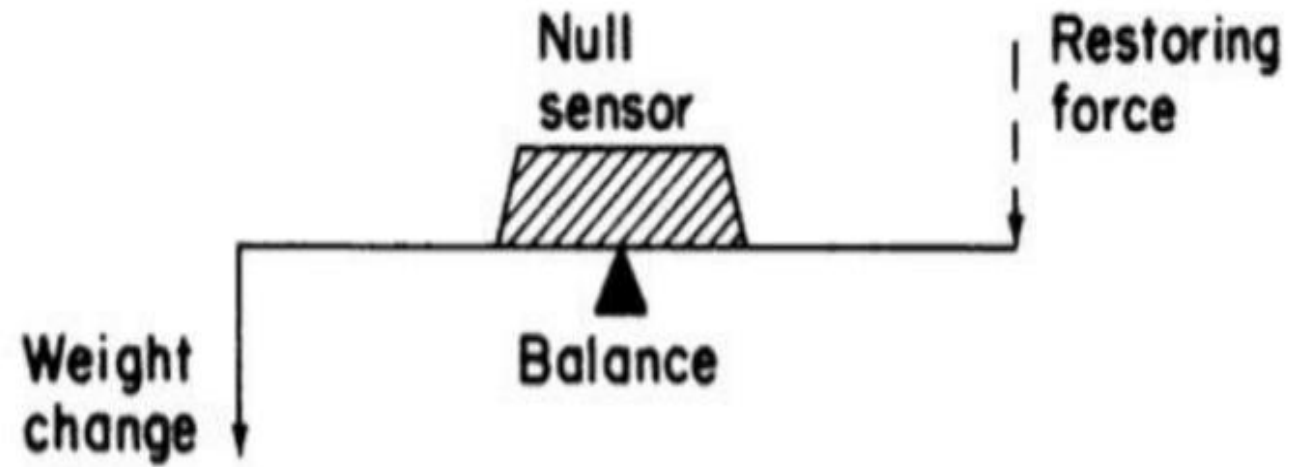
## TGA :How the Balance works ?

The balance operates on a null-balance principle.

In the null point balance system, when there is a change in weight, the balance beam will deviate from its usual position.



# Null point Balance



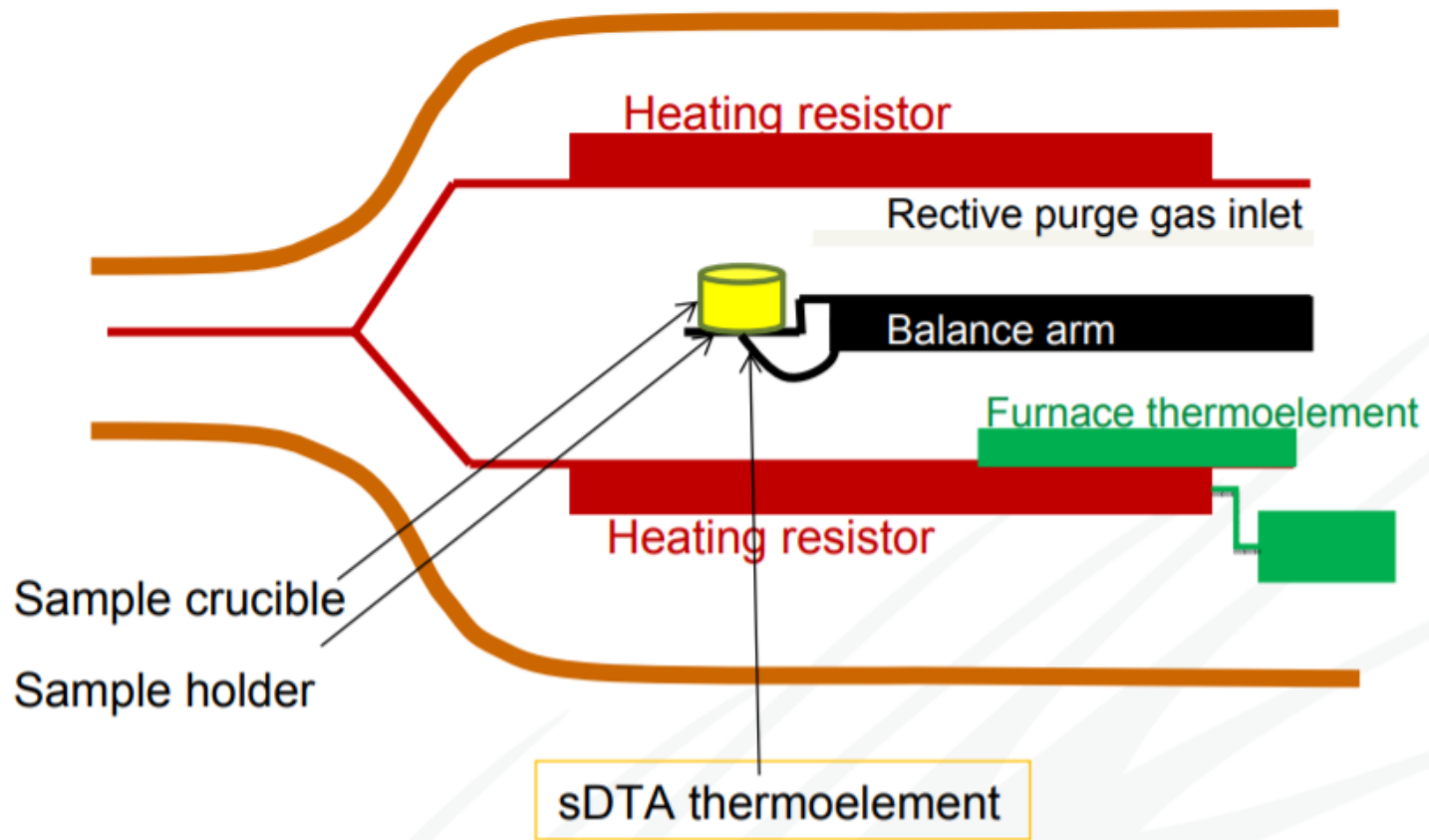
*A 'Null point' balance*

# Balance Principle

A sensor detects this deviation and initiates a force that will restore the balance to the null position.

This restoring force is proportional to the change in weight.

# Furnace components



# TGA : Experimental Procedure...

- The samples are placed in a shallow platinum crucible
- The sample container is connected to an automatic recording microbalance.
- A null point type balance system is employed in TGA for measuring the weight change.
- Which connected directly to the y- axis of the recorder

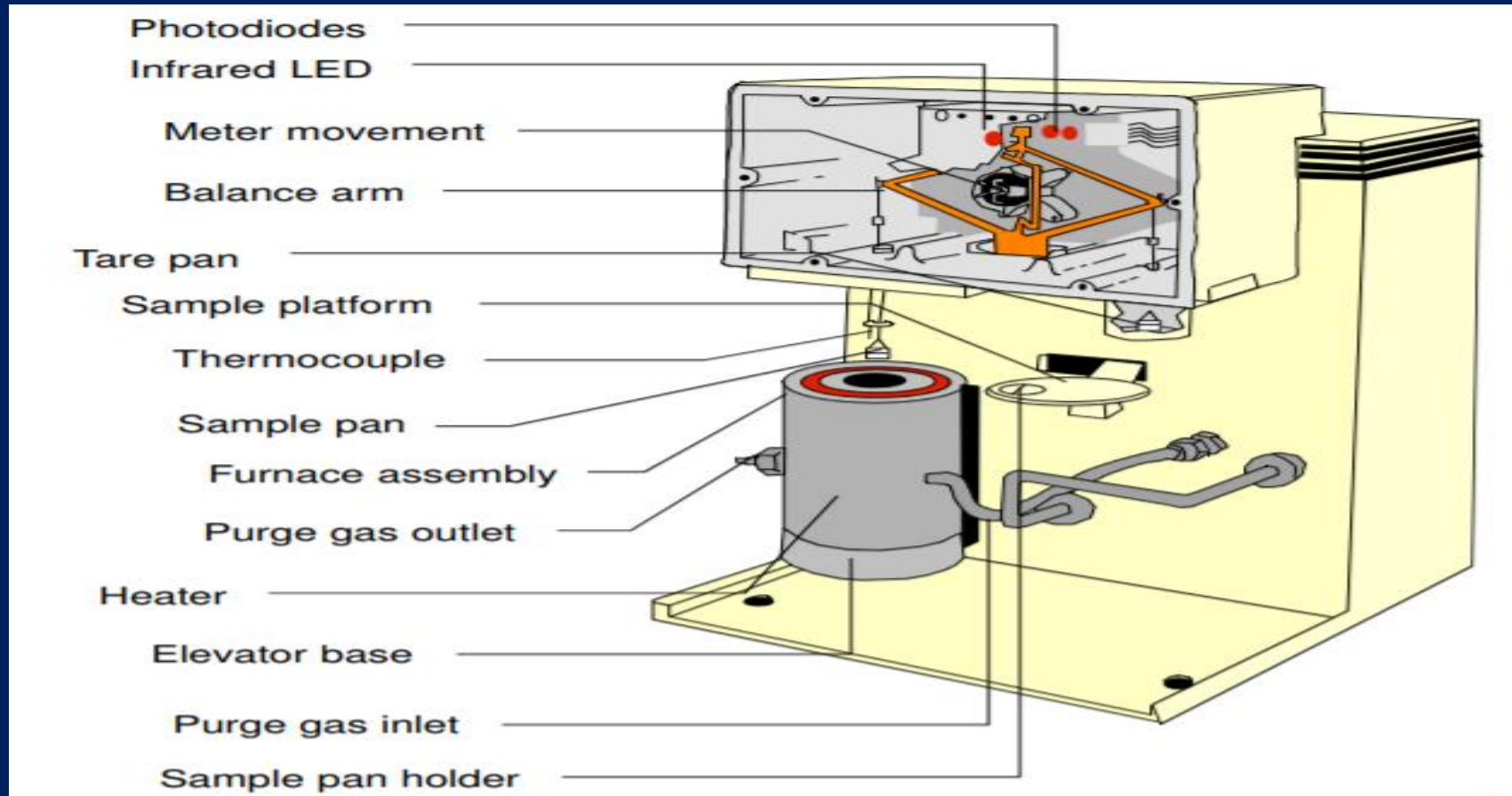
# TGA : Experimental Procedure...

- The sample container is placed in a Quartz housing located within the furnace.
- A thermocouple, located immediately below the sample container, is used to monitor the furnace temperature.
- The resulting signal is connected directly to the x – axis of the recorder.

# TGA Instrument



# Modern TGA instrument (TGA 2950)



# Factors Affecting TGA Curve

1. Instrumental Factors
2. Sample Characteristics
3. Geometry of the sample container

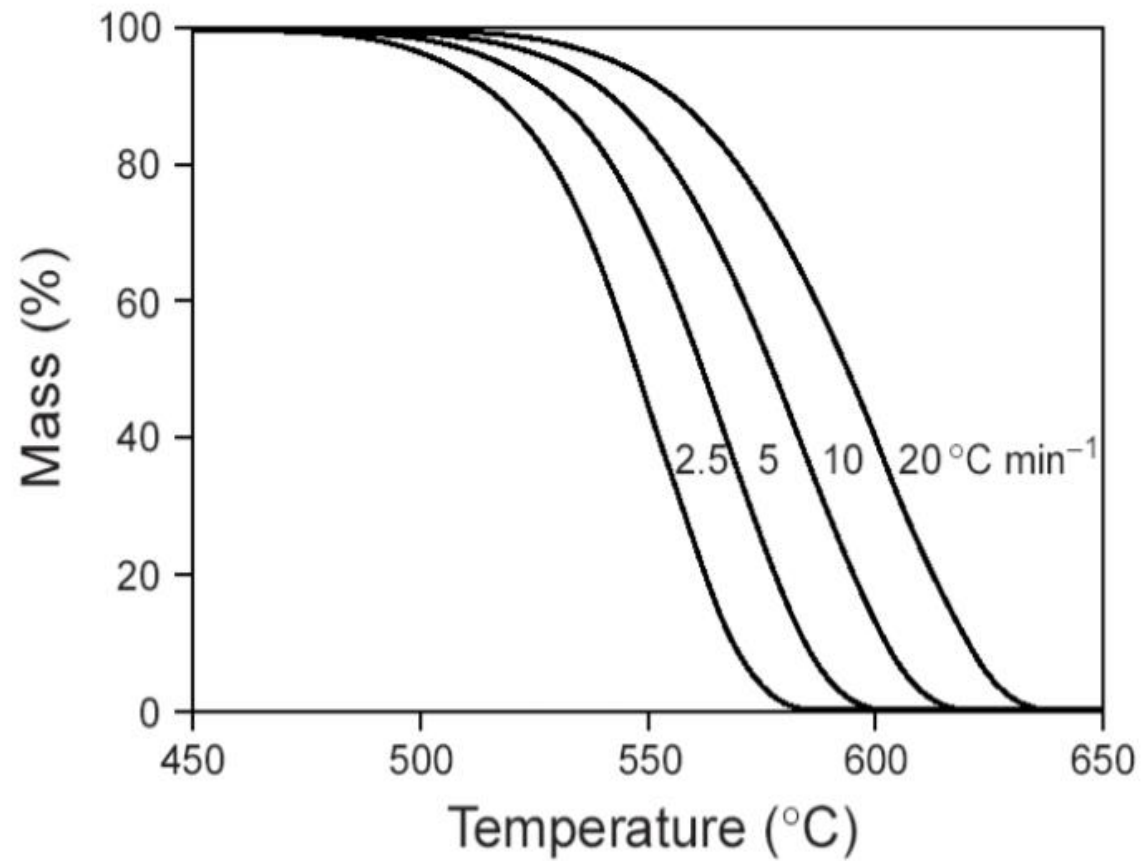


## Instrumental factors

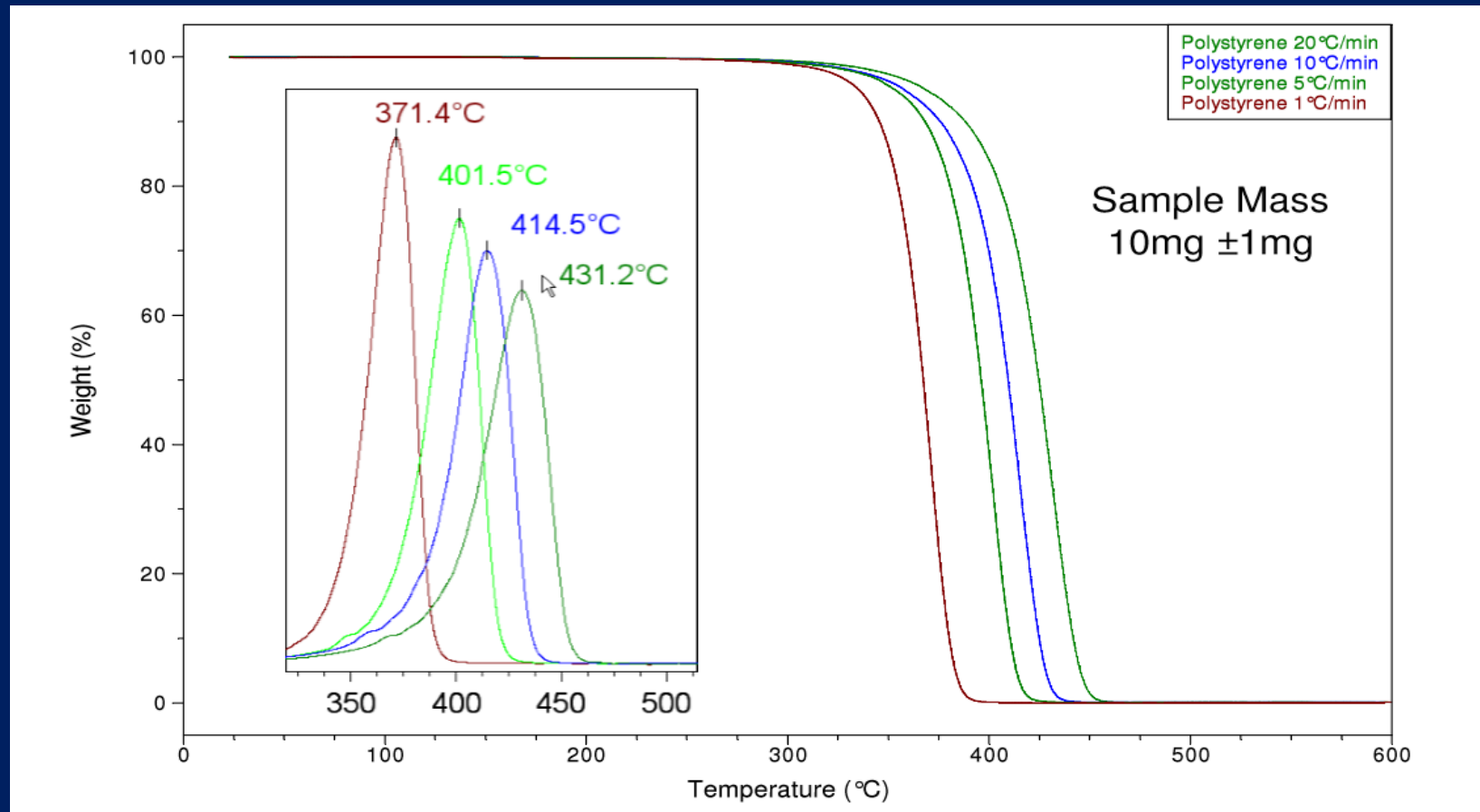
**Furnace Heating rate:** The temperature at which the sample compound decompose is depends upon the heating rate of furnace employed in the analysis.

# Effect of Heating rate

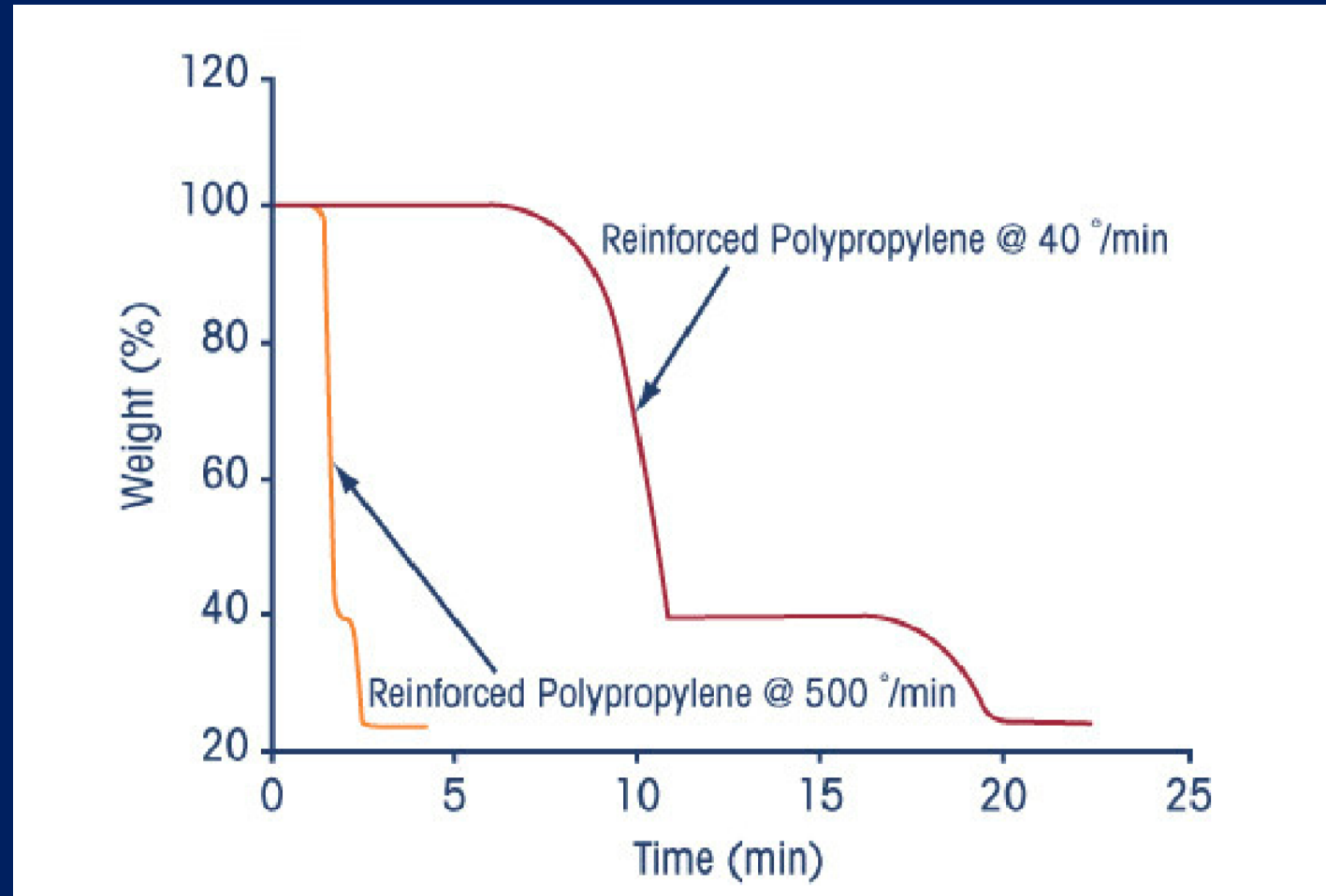
10 mg samples of PTFE, heated at 2.5, 5, 10 and 20 °C/min



# Higher heating rates increases the observed decomposition temperature



# Influence of heating rate on resolution



# Effect of Furnace atmosphere

The nature of the surrounding atmosphere inside the furnace has a significant affect upon the decomposition temperature.

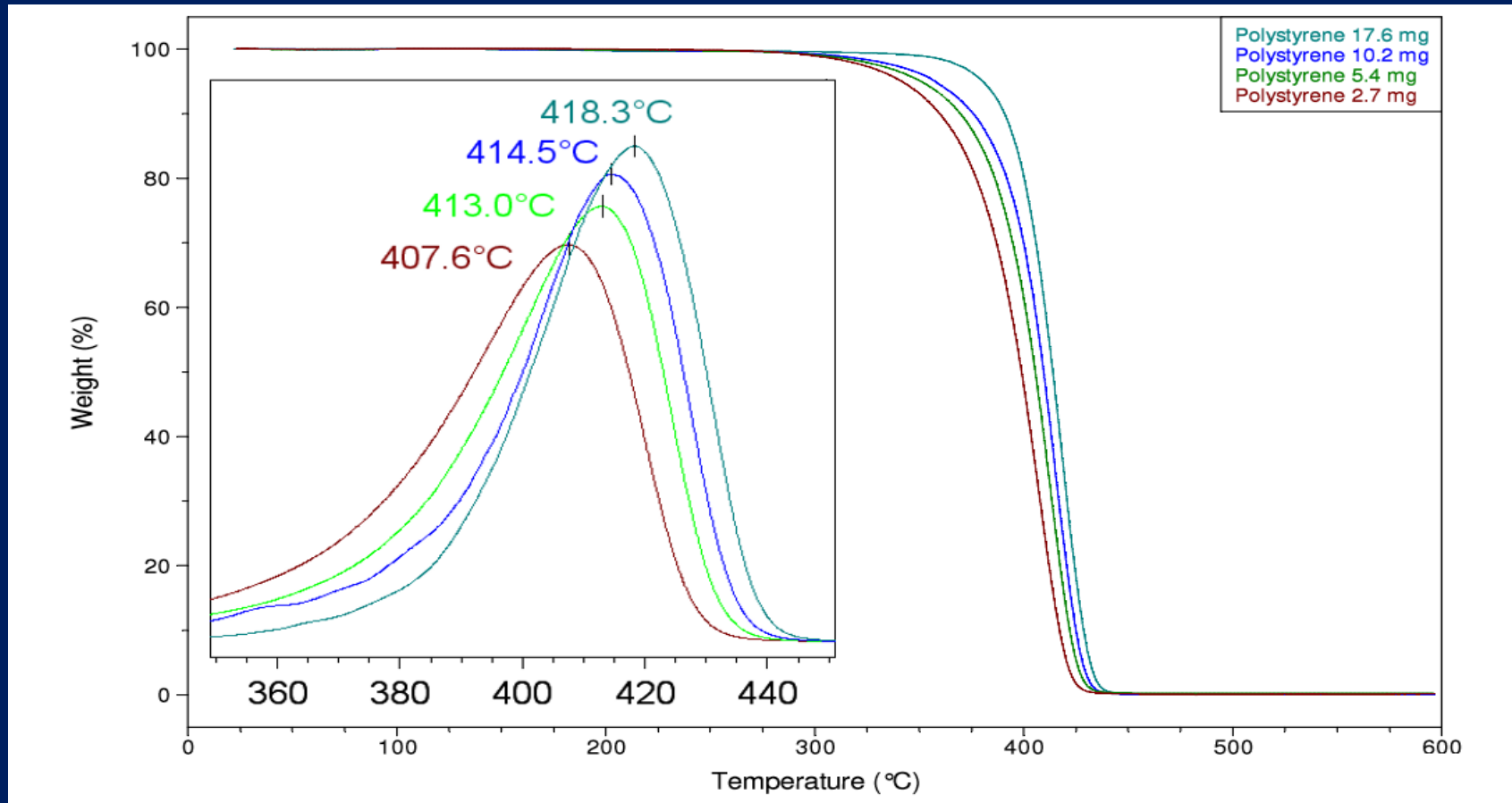
# Sample Characteristics

Mode of preparation of a sample

Weight of the sample

Particle size of the sample

# Larger sample mass increases the observed decomposition temperature



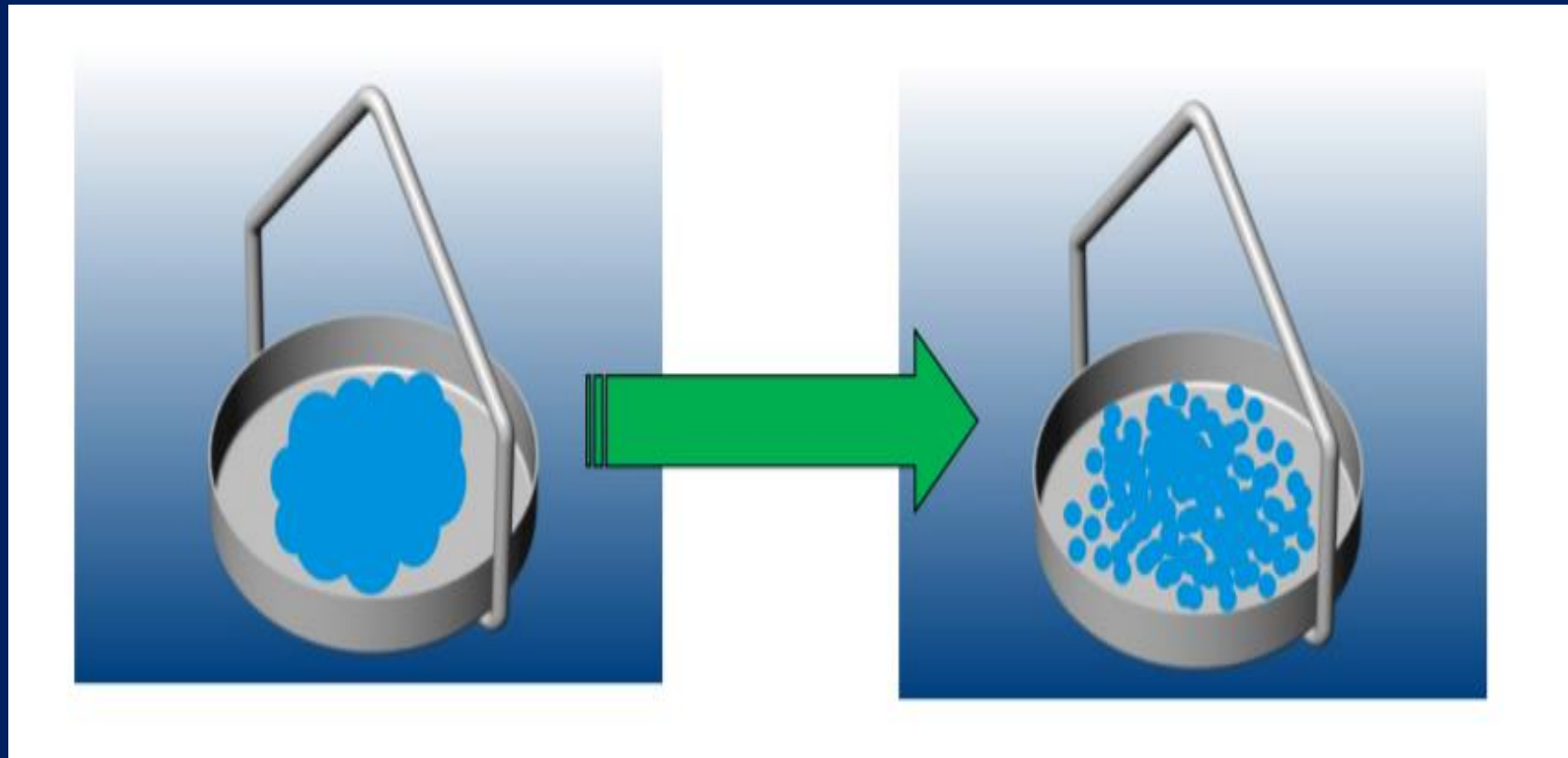
# Sample preparation

Sample mass having,

- 10-20 mg is used for most applications
- 50-100 mg is used for measuring volatile compounds



# TGA : Sample preparation



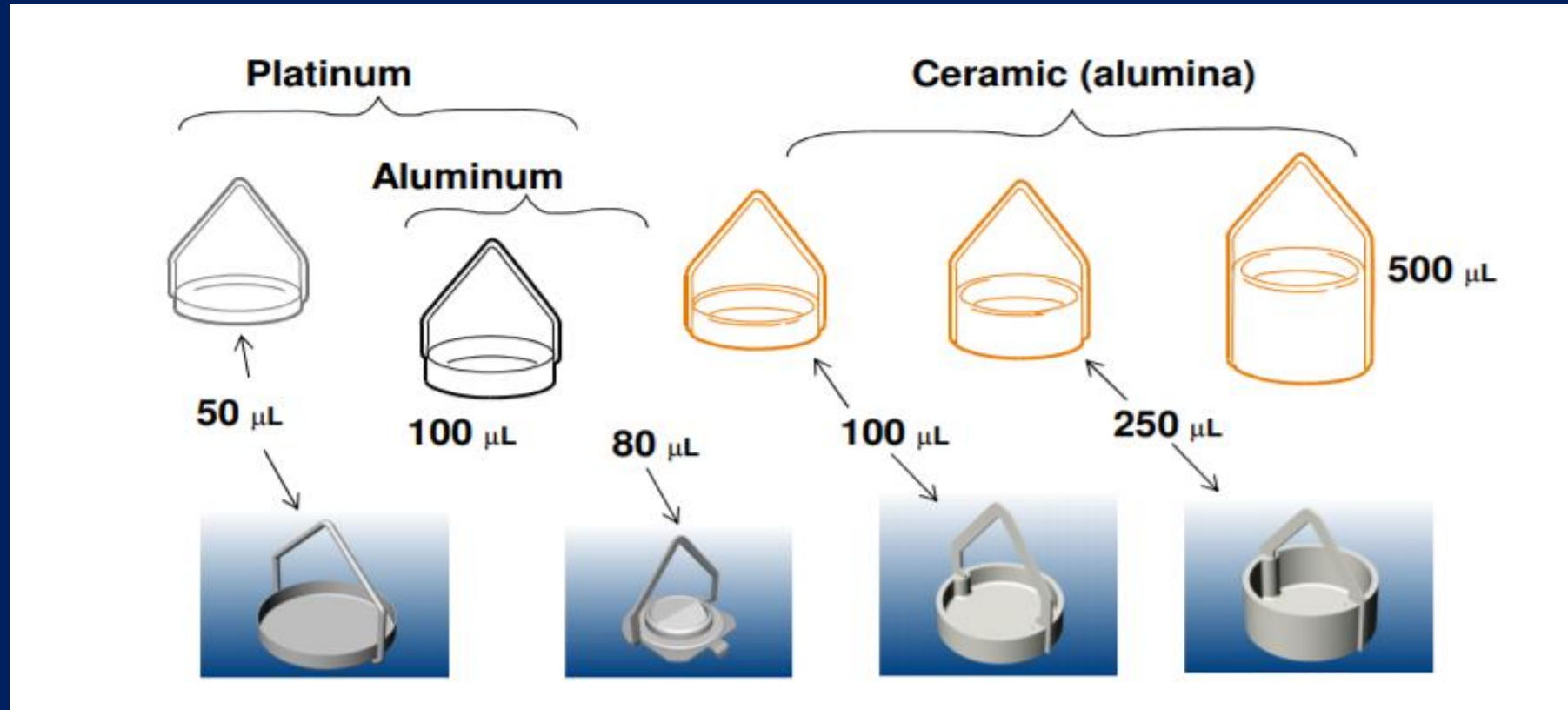
## Geometry of the sample container

A flat, plate shaped crucibles are generally preferred for better results.

**Alumina Pans:** Inert till 1700 °C

**Platinum Pans:** Having Good thermal conductivity.

# Different types of Sample Pans



# TGA : Calibration...

A TG analyzer should be calibrated for,

- mass using standard substances
- temperature using high pure reference materials and
- gas flow rate control

# Improving resolution in TGA experiments

- Slower heating rate
- Reduced sample size
- Self-generating atmosphere

Thank You.....

