

# CHM410

## ANALYTICAL CHEMISTRY II



Course video is available on our app

**Course code: CHM 410**

**Course title: Analytical Chemistry II**

**Course credit load: 2 Units**

**Series 1: Thermal Methods Of Analysis**

**Series 2: Electroanalytical And Potentiometric Techniques**

**Series 3: Conductometric, Amperometric And Colorimetric Methods**

**Series 4: Chromatographic Principles And Coupled Techniques**

**Series 5: Radiochemical Methods In Environmental Analysis**

**Proposed Outline Series 1**

### **1.1 Principles of Thermal Analysis**

- 1.1.1 Definition and scope of thermal methods
- 1.1.2 Importance and applications in analytical chemistry

### **1.2 Thermogravimetric and Derivative Thermogravimetric Analysis (TG and DTG)**

- 1.2.1 Principles of TG and DTG
- 1.2.2 Instrumentation and measurement
- 1.2.3 Applications and limitations

### **1.3 Differential Thermal Analysis (DTA)**

- 1.3.1 Principles of DTA
- 1.3.2 Instrumentation and interpretation of curves
- 1.3.3 Applications in material characterisation

### **1.4 Differential Scanning Calorimetry (DSC)**

- 1.4.1 Principles of DSC
- 1.4.2 Instrumentation and data interpretation
- 1.4.3 Applications in polymer and pharmaceutical analysis

## **Introduction**



In analytical chemistry, the study of how materials respond to changes in temperature provides valuable insights into their composition and properties. **Thermal analytical techniques** are widely used to investigate processes such as decomposition, phase transitions, and heat flow. These methods are essential in fields ranging from materials science to pharmaceuticals, where understanding thermal behaviour is critical for quality control and research.

The aim of this Series is to introduce you to the principles and applications of thermal methods of analysis, enabling you to explain their operation, interpret their outputs, and evaluate their significance in analytical chemistry.

We shall commence this Series by examining the general principles of thermal analysis, including its scope and importance. Next, we will explore thermogravimetric and derivative thermogravimetric analysis, focusing on their principles, instrumentation, and applications. Following that, we will study differential thermal analysis, considering how curves are interpreted and applied in material characterisation. Finally, we will conclude with differential scanning calorimetry, highlighting its role in polymer and pharmaceutical analysis.

## Series Learning Outcomes

Upon completing this Series, you should be able to:

- **SLO 1.1** define thermal analysis and explain its scope in analytical chemistry,
- **SLO 1.2** describe the principles of thermogravimetric and derivative thermogravimetric analysis,
- **SLO 1.3** demonstrate how TG and DTG instrumentation is used to measure thermal changes,
- **SLO 1.4** explain the principles of differential thermal analysis and interpret DTA curves,
- **SLO 1.5** analyse the applications of DTA in material characterisation,
- **SLO 1.6** explain the principles of differential scanning calorimetry,
- **SLO 1.7** interpret DSC data and evaluate its applications in polymers and pharmaceuticals.

### 1.1 Principles Of Thermal Analysis

#### 1.1.1 Definition and scope of thermal methods

**Thermal analysis** refers to a group of techniques in which a property of a material is measured as a function of temperature while the material is subjected to a controlled heating or cooling programme. These methods provide information about physical and chemical changes such as phase transitions, decomposition, and heat capacity.



Thermal methods are widely used in analytical chemistry because they allow scientists to study the stability, composition, and purity of materials. They are particularly useful in materials science, pharmaceuticals, and polymer chemistry.

Fill in the blank: Thermal analysis involves measuring a property of a material as a function of \_\_\_\_\_.

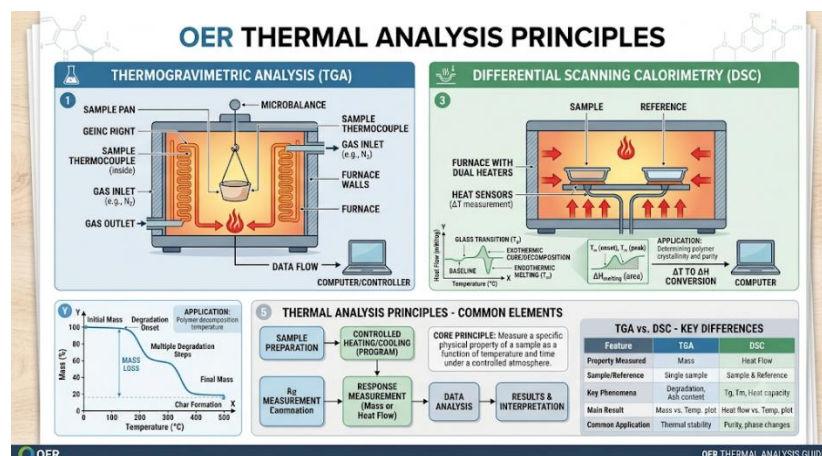


Figure 1.1: Basic principle of thermal analysis.

### 1.1.2 Importance and applications in analytical chemistry

The importance of thermal analysis lies in its ability to provide detailed information about material behaviour under temperature changes. For example:

- It helps determine decomposition temperatures of compounds.
- It identifies phase transitions such as melting or crystallisation.
- It measures heat capacities and enthalpy changes.
- It assesses purity and stability of pharmaceuticals and polymers.

Applications include quality control in industry, research in material development, and environmental analysis of pollutants.

Fill in the blank: Thermal analysis can be used to determine the \_\_\_\_\_ temperature of compounds.





**OER TABLE: APPLICATIONS OF THERMAL ANALYSIS ACROSS DISCIPLINES**

| Discipline                                                                                                       | Common Thermal Analysis Techniques                           | Key Applications & Measured Parameters                                                                                                              | Impact on Field                                                                                            |
|------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------|
|  <b>Materials Science</b>       | TGA, DSC, DMA<br>(Dynamic Mechanical Analysis)               | Degradation of advanced composites, phase transformation of alloys, thermal stability of ceramics.                                                  | Ensures reliability and performance of new materials; predicts service life.                               |
|  <b>Pharmaceuticals</b>         | DSC, TGA, XRPD<br>(X-ray Powder Diffraction, for comparison) | Purity testing of active pharmaceutical ingredients (APIs), determining polymorphism and solvate formation, glass transition in solid dispersions.  | Crucial for drug formulation stability, bio-availability, and regulatory compliance.                       |
|  <b>Polymers</b>                | DSC, TGA, DMA, TMA<br>(Thermomechanical Analysis)            | Characterizing glass transition ( $T_g$ ), determining crystallinity and melting points ( $T_m$ ), studying cure kinetics of resins.                | Essential for quality control in manufacturing; tailors physical properties (e.g., strength, flexibility). |
|  <b>Environmental Chemistry</b> | TGA, TGA-FTIR<br>(hyphenated technique)                      | Analyzing soil organic matter decomposition, identifying atmospheric particulates and dust composition, studying waste decomposition for recycling. | Supports environmental monitoring; contributes to waste management strategies and climate science.         |

**INTEGRATED ANALYSIS PROCESS**

Sample Source  
(Materials, Pharma, Polymer, Environ)

→

Thermal Analysis Choice  
(TGA, DSC, etc.)

→

Unique Data Set  
(Mass loss, Heat flow, Modulus)

→

Discipline-Specific Interpretation  
(Stability, Purity, Cure, Composition)

OER THERMAL ANALYSIS GUIDE

Box 1.1: Selected applications of thermal analysis.

#### Pilot questions 1.1

- Which of the following best defines thermal analysis? a. Measurement of a property of a material as a function of temperature b. Measurement of a property of a material as a function of pressure c. Measurement of a property of a material as a function of concentration d. Measurement of a property of a material as a function of volume
- Fill in the blank: Thermal analysis involves measuring a property of a material as a function of \_\_\_\_\_.
- Which of the following is an application of thermal analysis? a. Determining decomposition temperatures b. Measuring electrical conductivity c. Studying light absorption spectra d. Measuring magnetic susceptibility
- Fill in the blank: Thermal analysis can be used to determine the \_\_\_\_\_ temperature of compounds.
- Which of the following industries commonly uses thermal analysis? a. Pharmaceuticals b. Agriculture c. Textile design d. Astronomy

## 1.2 Thermogravimetric And Derivative Thermogravimetric Analysis (TG And DTG)

### 1.2.1 Principles of TG and DTG

**Thermogravimetric analysis (TG)** is a technique in which the mass of a sample is measured as it is heated, cooled, or held at a constant temperature. It provides information about changes such as decomposition, oxidation, or loss of volatile components.

**Derivative thermogravimetry (DTG)** is the first derivative of the TG curve with respect to time or temperature. It highlights the rate of mass change, making it easier to identify overlapping processes and pinpoint exact transition temperatures.

Fill in the blank: Derivative thermogravimetry is the first \_\_\_\_\_ of the TG curve with respect to time or temperature.



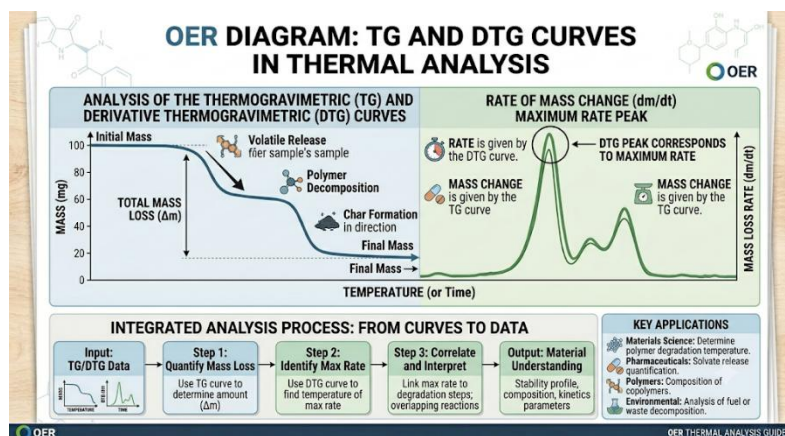


Figure 1.2: TG and DTG curves.

## 1.2.2 Instrumentation and measurement

TG instruments typically consist of a precision balance, a furnace, and a temperature control system. The sample is placed on the balance inside the furnace, and its mass is continuously recorded as the temperature changes.

In DTG, the instrument software calculates the derivative of the TG curve, producing peaks that correspond to specific thermal events. This allows clearer identification of processes such as moisture loss, decomposition, or oxidation.

Fill in the blank: A TG instrument usually consists of a balance, a furnace, and a \_\_\_\_\_ control system.

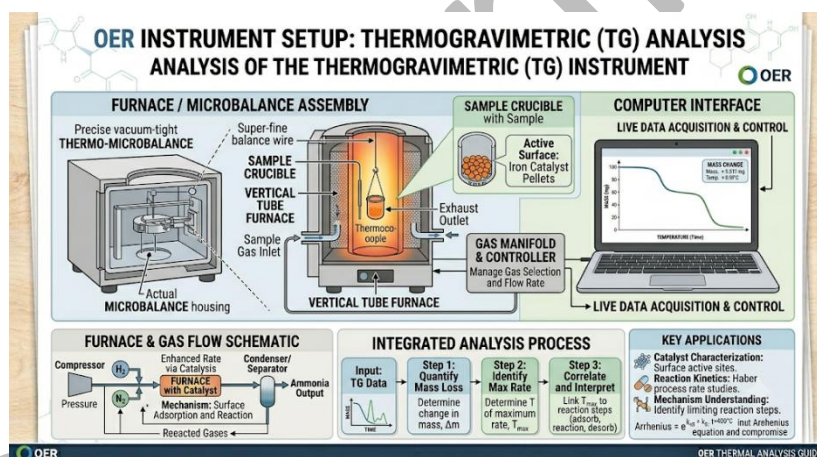


Plate 1.2: Basic TG instrumentation.

## 1.2.3 Applications and limitations

TG and DTG are widely applied in analytical chemistry:

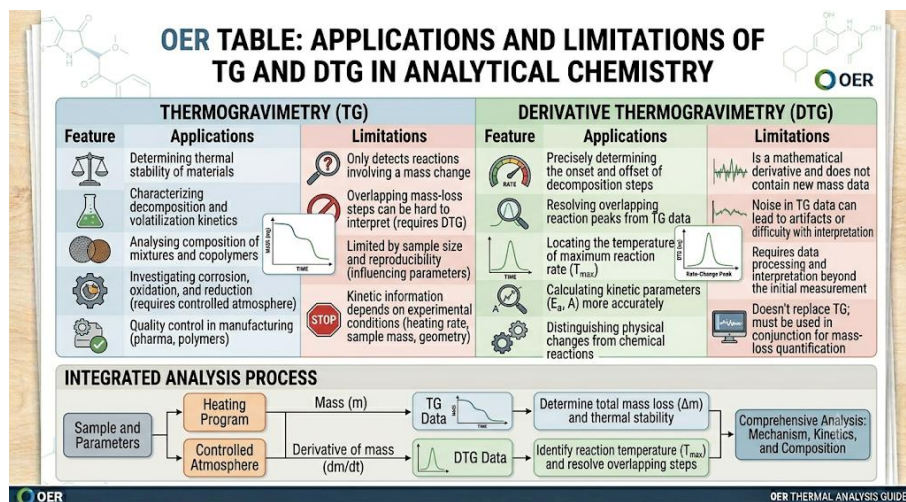
- Determining thermal stability of materials.
- Measuring moisture and volatile content.



- Studying decomposition pathways of polymers and pharmaceuticals.
- Investigating oxidation and reduction behaviour of metals.

However, limitations include sensitivity to sample size, potential overlapping of thermal events, and the need for careful calibration to ensure accuracy.

Fill in the blank: TG and DTG can be used to measure the \_\_\_\_\_ content of a sample.



Box 1.2: Applications of TG and DTG.

#### Pilot questions 1.2

1. Which of the following best defines thermogravimetric analysis? a. Measurement of mass change of a sample with temperature b. Measurement of heat flow of a sample with temperature c. Measurement of pressure change of a sample with temperature d. Measurement of colour change of a sample with temperature
2. Fill in the blank: Derivative thermogravimetry is the first \_\_\_\_\_ of the TG curve with respect to time or temperature.
3. Which of the following components is part of a TG instrument? a. Balance b. Microscope c. Spectrophotometer d. Electrode
4. Fill in the blank: A TG instrument usually consists of a balance, a furnace, and a \_\_\_\_\_ control system.
5. Which of the following is a limitation of TG and DTG? a. Sensitivity to sample size b. Ability to measure moisture content c. Ability to study decomposition pathways d. Ability to investigate oxidation behaviour

### 1.3 Differential Thermal Analysis (DTA)

#### 1.3.1 Principles of DTA

**Differential thermal analysis (DTA)** is a technique in which the temperature difference between a sample and a reference material is measured as both are subjected to the same heating





or cooling programme. Any thermal event in the sample, such as melting, crystallisation, or decomposition, produces a peak on the DTA curve.

This method is particularly useful for identifying phase transitions and comparing thermal behaviour between different materials.

Fill in the blank: In DTA, the temperature difference between a sample and a \_\_\_\_\_ material is measured.

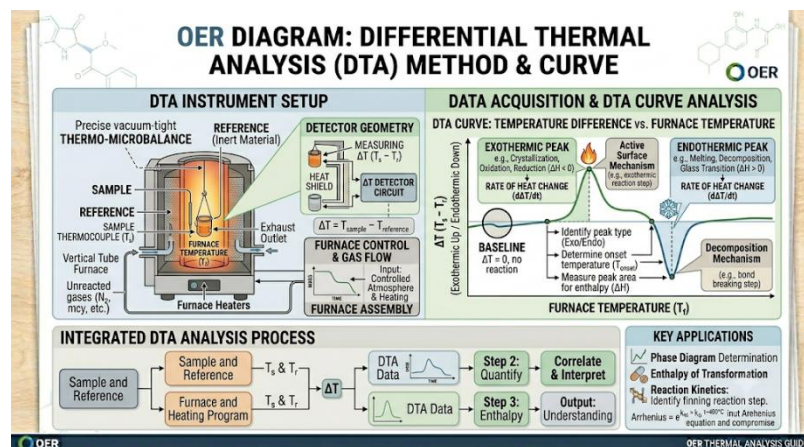


Figure 1.3: Principle of differential thermal analysis

### 1.3.2 Instrumentation and interpretation of curves

A DTA instrument consists of a furnace, a sample holder, a reference holder, and thermocouples to measure temperature differences. The sample and reference are heated simultaneously, and the thermocouples record any deviation in temperature.

The resulting DTA curve shows endothermic peaks (heat absorbed, e.g. melting) and exothermic peaks (heat released, e.g. crystallisation). The position and shape of these peaks provide information about the type and magnitude of thermal events.

Fill in the blank: Endothermic peaks on a DTA curve indicate that heat is \_\_\_\_\_ by the sample.

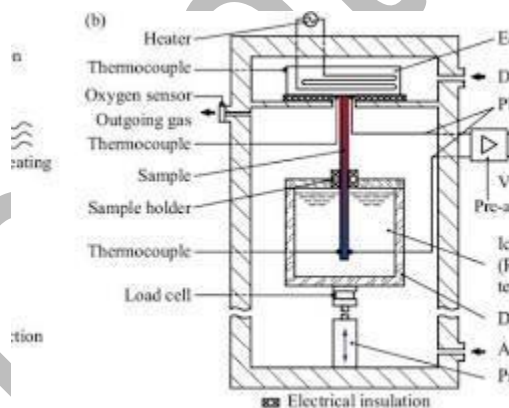


Plate 1.3: Basic DTA instrumentation.

### 1.3.3 Applications in material characterisation





DTA is widely applied in material science and analytical chemistry:

- Identifying phase transitions such as melting, crystallisation, and glass transitions.
- Studying decomposition behaviour of inorganic and organic compounds.
- Comparing thermal stability of different materials.
- Characterising polymers, ceramics, and pharmaceuticals.

Fill in the blank: DTA can be used to identify phase transitions such as \_\_\_\_\_ and crystallisation.

**OER TABLE: APPLICATIONS OF DIFFERENTIAL THERMAL ANALYSIS (DTA)**

| Discipline                    | Key DTA Applications & Parameters                                                                                                                                                                                                               | Informed Material Properties                                                       | Example Phenomena                                  |
|-------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------|----------------------------------------------------|
| <b>1. POLYMERS</b>            | <ul style="list-style-type: none"> <li>→ Glass Transition (<math>T_g</math>) measurement</li> <li>→ Melting Point (<math>T_m</math>) and Crystallinity (%)</li> <li>→ Polymer Oxidation Temperature (Oxidative Induction Time - OIT)</li> </ul> | Processability, stiffness, thermal history, degradation stability                  | Measuring change in $T_g$ with plasticizer content |
| <b>2. CERAMICS</b>            | <ul style="list-style-type: none"> <li>→ Phase transformation (<math>T_{trans}</math>)</li> <li>→ Crystallization kinetics</li> <li>→ Binder burnout temperature</li> <li>→ Chemical reactions (e.g., solid-state synthesis)</li> </ul>         | Structure-property relationships, firing temperature optimization, chemical purity | Identification of kaolinite decomposition          |
| <b>3. PHARMACEUTICALS</b>     | <ul style="list-style-type: none"> <li>→ Polymorphism screening and identification</li> <li>→ Purity analysis (Van't Hoff method)</li> <li>→ Stability studies (dehydration, decomposition)</li> </ul>                                          | Drug efficacy, bioavailability, shelf-life estimation, compatibility               | Detection of crystalline polymorph conversions     |
| <b>4. INORGANIC COMPOUNDS</b> | <ul style="list-style-type: none"> <li>→ Specific heat capacity (<math>C_p</math>) determination</li> <li>→ Reaction enthalpy (<math>\Delta H</math>) calculation</li> <li>→ Compound identification (melting points, decomposition)</li> </ul> | Chemical nature, stoichiometry, reactivity, thermal engineering                    | Analyzing kinetics of calcination                  |

**INTEGRATED DTA ANALYSIS PROCESS**

Sample → DTA Data → **Step 1: Identify Phase Changes** → **Step 2: Quantify Enthalpy** → **Step 3: Correlate & Interpret** → **Output: Material Understanding** (Arrhenius equation and compromise)

Box 1.3: Applications of DTA in material characterisation.

#### Pilot questions 1.3

- Which of the following best defines differential thermal analysis? a. Measurement of temperature difference between a sample and a reference material b. Measurement of mass change of a sample with temperature c. Measurement of heat flow of a sample with temperature d. Measurement of pressure change of a sample with temperature
- Fill in the blank: In DTA, the temperature difference between a sample and a \_\_\_\_\_ material is measured.
- Which of the following peaks on a DTA curve indicates heat absorption? a. Endothermic peak b. Exothermic peak c. Neutral peak d. Pressure peak
- Fill in the blank: Endothermic peaks on a DTA curve indicate that heat is \_\_\_\_\_ by the sample.
- Which of the following is an application of DTA? a. Identifying phase transitions b. Measuring electrical conductivity c. Studying light absorption spectra d. Measuring magnetic susceptibility

## 1.4 Differential Scanning Calorimetry (DSC)

### 1.4.1 Principles of DSC



**Differential scanning calorimetry (DSC)** is a thermal analysis technique that measures the difference in heat flow between a sample and a reference as they are subjected to a controlled temperature programme. Unlike DTA, DSC provides quantitative data on the amount of heat absorbed or released during thermal events such as melting, crystallisation, or chemical reactions.

This makes DSC particularly valuable for studying thermodynamic properties, including enthalpy changes and specific heat capacities.

Fill in the blank: DSC measures the difference in \_\_\_\_\_ flow between a sample and a reference.

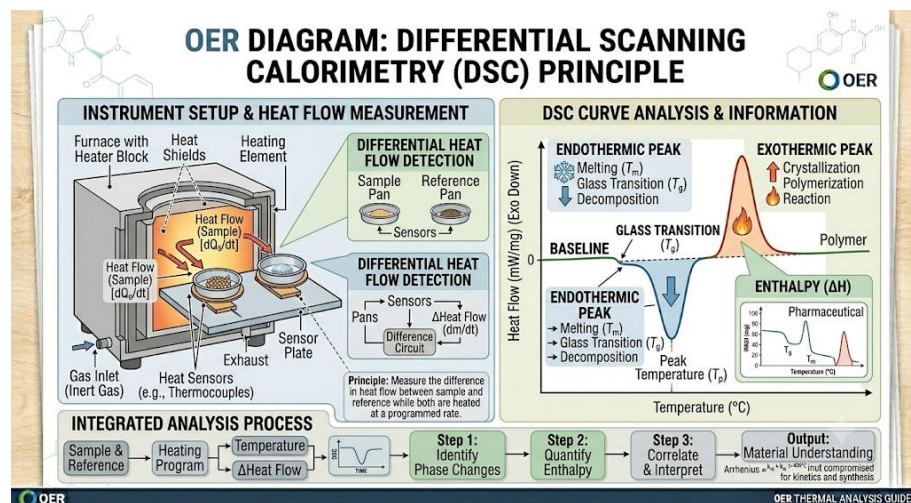


Figure 1.4: Principle of differential scanning calorimetry.

### 1.4.2 Instrumentation and data interpretation

A DSC instrument consists of a furnace, sample and reference pans, sensors to detect heat flow, and a computer interface for data recording. The sample and reference are heated simultaneously, and the instrument records the difference in energy required to maintain equal temperatures.

The resulting DSC curve shows endothermic peaks (heat absorbed, e.g. melting) and exothermic peaks (heat released, e.g. crystallisation). The area under each peak corresponds to the enthalpy change of the process.

Fill in the blank: The area under a DSC peak corresponds to the \_\_\_\_\_ change of the process.





1. Which of the following best defines DSC? a. Measurement of heat flow difference between a sample and a reference b. Measurement of mass change of a sample with temperature c. Measurement of pressure change of a sample with temperature d. Measurement of colour change of a sample with temperature
2. Fill in the blank: DSC measures the difference in \_\_\_\_\_ flow between a sample and a reference.
3. Which of the following peaks on a DSC curve indicates heat absorption? a. Endothermic peak b. Exothermic peak c. Neutral peak d. Pressure peak
4. Fill in the blank: The area under a DSC peak corresponds to the \_\_\_\_\_ change of the process.
5. Which of the following is an application of DSC? a. Determining glass transition temperature of polymers b. Measuring electrical conductivity c. Studying light absorption spectra d. Measuring magnetic susceptibility

## Series Summary

In this Series, we explored the **principles and applications of thermal analytical techniques**, which are essential tools in analytical chemistry for studying material behaviour under controlled temperature changes. We began with the general principles of thermal analysis, highlighting its scope and importance. We then examined **thermogravimetric analysis (TG)** and **derivative thermogravimetry (DTG)**, focusing on how mass changes and their rates are measured. Following that, we studied **differential thermal analysis (DTA)**, which measures temperature differences between a sample and a reference to identify phase transitions. Finally, we concluded with **differential scanning calorimetry (DSC)**, which quantifies heat flow differences and provides valuable data on enthalpy changes and specific heat capacities. Altogether, this Series has provided learners with a comprehensive understanding of how thermal methods are applied in materials science, pharmaceuticals, polymers, and environmental chemistry.

## Glossary of Terms

- **Thermal analysis:** A group of techniques measuring material properties as a function of temperature.
- **Thermogravimetric analysis (TG):** Measurement of mass change of a sample with temperature.
- **Derivative thermogravimetry (DTG):** The first derivative of the TG curve, showing the rate of mass change.





- **Differential thermal analysis (DTA):** Measurement of temperature difference between a sample and a reference during heating or cooling.
- **Endothermic peak:** A peak on a DTA or DSC curve indicating heat absorption.
- **Exothermic peak:** A peak on a DTA or DSC curve indicating heat release.
- **Differential scanning calorimetry (DSC):** Measurement of heat flow difference between a sample and a reference, providing quantitative data on enthalpy changes.
- **Glass transition temperature:** The temperature at which a polymer changes from a rigid to a flexible state.
- **Enthalpy change:** The amount of heat absorbed or released during a process.

## Concept Check Questions 1

### Objective questions

1. Thermal analysis involves measuring a property of a material as a function of \_\_\_\_\_. (Linked to SLO 1.1)
2. Derivative thermogravimetry is the first \_\_\_\_\_ of the TG curve with respect to time or temperature. (Linked to SLO 1.2)
3. In DTA, the temperature difference between a sample and a \_\_\_\_\_ material is measured. (Linked to SLO 1.4)
4. The area under a DSC peak corresponds to the \_\_\_\_\_ change of the process. (Linked to SLO 1.7)
5. DSC can be used to determine the glass \_\_\_\_\_ temperature of polymers. (Linked to SLO 1.7)

### Short-answer questions

6. Explain the difference between TG and DTG. (Linked to SLOs 1.2–1.3)
7. Describe how endothermic and exothermic peaks appear on DTA and DSC curves. (Linked to SLOs 1.4–1.7)
8. Outline the importance of DSC in pharmaceutical analysis. (Linked to SLO 1.7)

### Long-answer questions

9. Analyse the applications of thermal analysis in materials science, pharmaceuticals, and polymers. (Linked to SLOs 1.1–1.7)
10. Evaluate the limitations of TG and DTG compared to DTA and DSC. (Linked to SLOs 1.2–1.7)
11. Discuss how DSC provides quantitative data on enthalpy changes and why this is important in analytical chemistry. (Linked to SLO 1.7)



## Answers To Pilot Questions 1

**Part 1.1:** 1.a, 2.temperature, 3.a, 4.decomposition, 5.a **Part 1.2:** 1.a, 2.derivative, 3.a, 4.temperature, 5.a **Part 1.3:** 1.a, 2.reference, 3.a, 4.absorbed, 5.a **Part 1.4:** 1.a, 2.heat, 3.a, 4.enthalpy, 5.a

## References And Attributions 1

- Figures 1.1–1.4: Suggested AI prompts for diagrams showing principles of thermal analysis, TG/DTG curves, DTA setup, and DSC principle.
- Plates 1.2–1.4: Suggested AI prompts for images of TG, DTA, and DSC instrument setups.
- Boxes 1.1–1.4: Suggested AI prompts for tables summarising applications of thermal analysis, TG/DTG, DTA, and DSC.
- Open Educational Resources (OER) suggested search strings:
  - “OER diagram thermal analysis principle chemistry”
  - “OER diagram TG DTG curves chemistry”
  - “OER image TG instrument setup chemistry”
  - “OER diagram differential thermal analysis chemistry”
  - “OER image DTA instrument setup chemistry”
  - “OER diagram DSC principle chemistry”
  - “OER image DSC instrument setup chemistry”
  - “OER table DSC applications chemistry”
- Attribution style: APA (institutional standard assumed). Example: OpenStax. (2016). *Chemistry*. OpenStax CNX. Retrieved from <https://openstax.org/books/chemistry>



**Course code: CHM 410**  
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**Series 1: Thermal Methods Of Analysis**

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## **Proposed Outline Series 2**

### **2.1 Principles of Electroanalytical Techniques**

- 2.1.1 Definition and scope of electroanalytical methods
- 2.1.2 Importance in analytical chemistry

### **2.2 Potentiometric Methods of Analysis**

- 2.2.1 Principles of potentiometry
- 2.2.2 Measurement of pH and ion-selective electrodes
- 2.2.3 Applications and limitations

### **2.3 Other Electroanalytical Techniques**

- 2.3.1 Overview of conductometric methods
- 2.3.2 Overview of amperometric methods
- 2.3.3 Applications in environmental and industrial analysis

## **Introduction**

Electroanalytical methods are among the most widely used techniques in analytical chemistry, offering precise ways to measure chemical properties through electrical signals. These methods are particularly important in environmental monitoring, industrial quality control, and biomedical



applications. Potentiometric techniques, which involve measuring the potential difference between electrodes, are especially useful for determining pH and ion concentrations.

The aim of this Series is to introduce you to the principles of electroanalytical methods, explain the operation of potentiometric techniques, and highlight their applications in analytical chemistry.

We shall commence this Series by examining the general principles of electroanalytical techniques and their importance. Next, we will explore potentiometric methods of analysis, focusing on pH measurement and ion-selective electrodes. Finally, we will conclude with an overview of other electroanalytical techniques such as conductometry and amperometry, considering their applications in environmental and industrial contexts.

## Series Learning Outcomes

Upon completing this Series, you should be able to:

- **SLO 2.1** define electroanalytical techniques and explain their scope in analytical chemistry,
- **SLO 2.2** describe the principles of potentiometric methods of analysis,
- **SLO 2.3** demonstrate how pH is measured using potentiometric techniques,
- **SLO 2.4** explain the function of ion-selective electrodes in potentiometry,
- **SLO 2.5** analyse the applications and limitations of potentiometric methods,
- **SLO 2.6** describe the principles of conductometric methods,
- **SLO 2.7** explain the principles of amperometric methods,
- **SLO 2.8** evaluate the applications of conductometric and amperometric techniques in environmental and industrial analysis.

## 2.1 Principles Of Electroanalytical Techniques

### 2.1.1 Definition and scope of electroanalytical methods

**Electroanalytical methods** are techniques in analytical chemistry that use electrical measurements to study chemical systems. These methods rely on properties such as potential, current, or conductivity to provide information about the composition and behaviour of substances.





They are widely applied in environmental monitoring, pharmaceutical analysis, and industrial quality control because they allow precise and rapid determination of chemical species.

Fill in the blank: Electroanalytical methods use \_\_\_\_\_ measurements to study chemical systems.

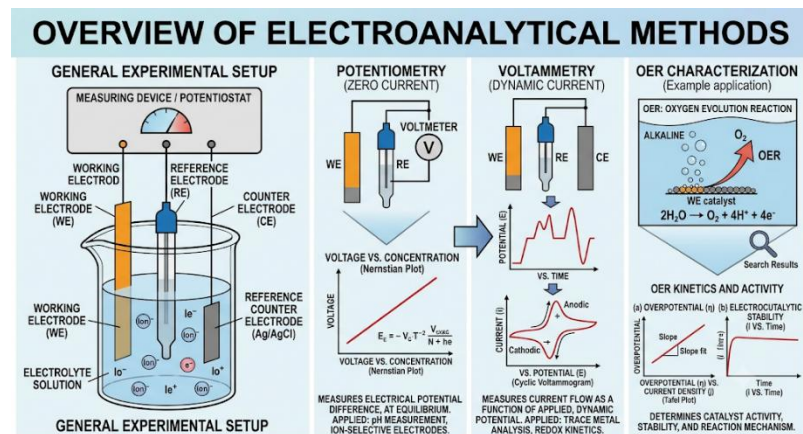


Figure 2.1: Basic principle

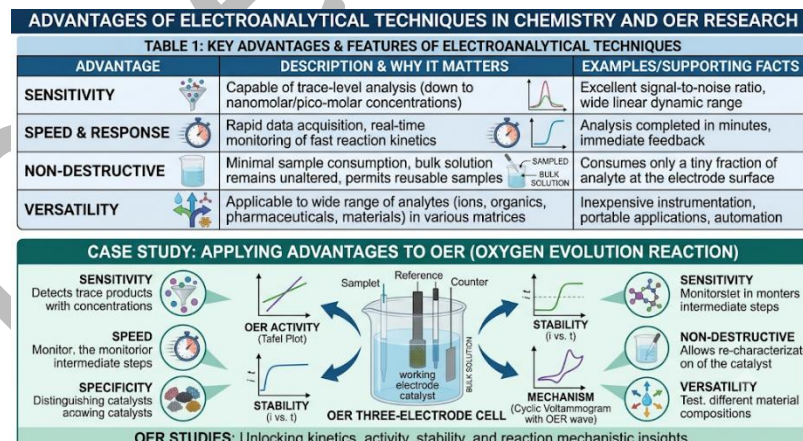
## 2.1.2 Importance in analytical chemistry

Electroanalytical techniques are important because they provide:

- High sensitivity for detecting trace amounts of substances.
- Rapid and accurate measurements.
- Non-destructive analysis of samples.
- Versatility in studying ions, molecules, and complex mixtures.

They are particularly valuable in determining pH, monitoring pollutants, and analysing biological fluids.

Fill in the blank: Electroanalytical techniques are especially valuable in determining \_\_\_\_\_ in solutions.



## Box 2.1: Importance of electroanalytical methods.

### Pilot questions 2.1

1. Which of the following best defines electroanalytical methods? a. Techniques that use electrical measurements to study chemical systems b. Techniques that use heat measurements to study chemical systems c. Techniques that use light measurements to study chemical systems d. Techniques that use pressure measurements to study chemical systems
2. Fill in the blank: Electroanalytical methods use \_\_\_\_\_ measurements to study chemical systems.
3. Which of the following is an advantage of electroanalytical techniques? a. High sensitivity for detecting trace substances b. Requirement of large sample sizes c. Slow and destructive analysis d. Limited to solid samples only
4. Fill in the blank: Electroanalytical techniques are especially valuable in determining \_\_\_\_\_ in solutions.
5. Which of the following fields commonly uses electroanalytical methods? a. Environmental monitoring b. Astronomy c. Textile design d. Sculpture

## 2.2 Potentiometric Methods Of Analysis

### 2.2.1 Principles of potentiometry

**Potentiometry** is an electroanalytical technique in which the potential difference between two electrodes is measured without drawing significant current. This potential difference is related to the concentration of ions in the solution, making potentiometry a valuable method for chemical analysis.

The most common application is pH measurement, where a glass electrode responds to hydrogen ion activity. Other ion-selective electrodes can be used for ions such as sodium, potassium, or chloride.

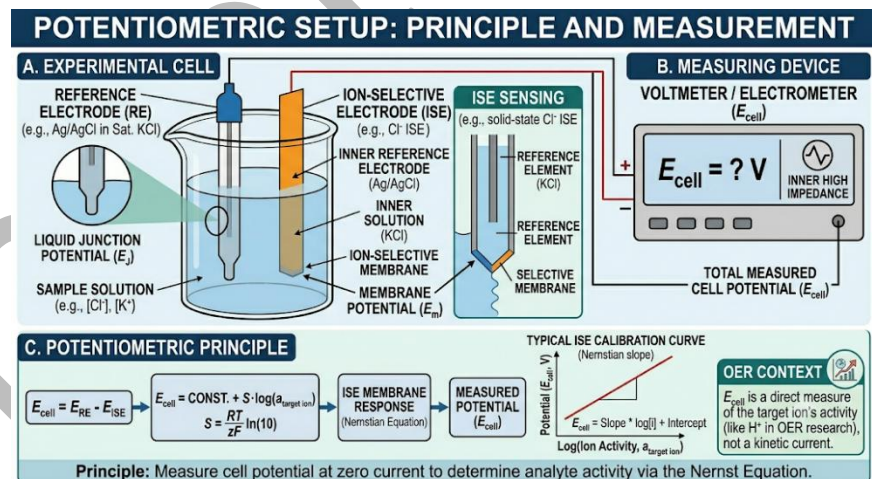


Figure 2.2: Principle of potentiometry.



## 2.2.2 Measurement of pH and ion-selective electrodes

**pH measurement** is the most widely known application of potentiometry. A glass electrode sensitive to hydrogen ions is paired with a reference electrode, and the potential difference is measured to determine pH.

**Ion-selective electrodes (ISEs)** are designed to respond selectively to specific ions. For example, a sodium electrode responds to sodium ions, while a fluoride electrode responds to fluoride ions. These electrodes expand the scope of potentiometry beyond pH measurement.

Fill in the blank: A glass electrode is commonly used in potentiometry to measure \_\_\_\_\_ ion activity.

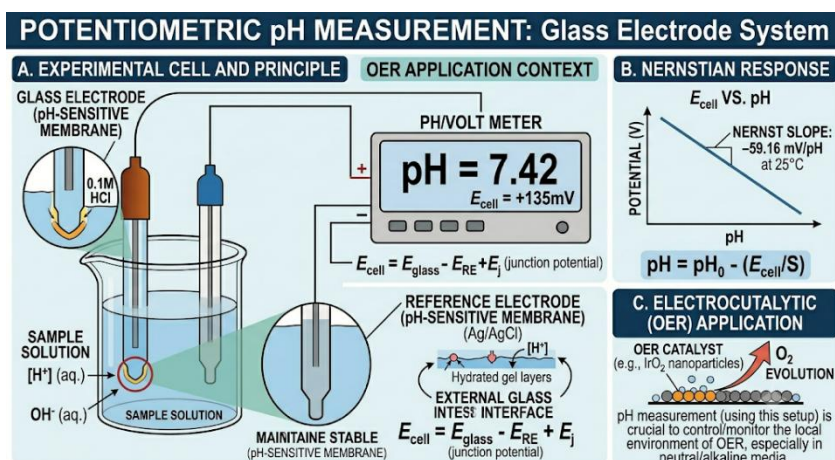


Plate 2.2: Potentiometric measurement of pH.

## 2.2.3 Applications and limitations

Potentiometric methods are applied in:

- Environmental monitoring, such as measuring ion concentrations in water.
- Pharmaceutical analysis, including drug formulation testing.
- Food industry, for quality control of beverages and dairy products.
- Clinical chemistry, for measuring electrolytes in blood samples.

Limitations include sensitivity to electrode calibration, interference from other ions, and the need for stable reference electrodes.

Fill in the blank: Potentiometric methods are widely used in clinical chemistry to measure \_\_\_\_\_ in blood samples.





## SUMMARY OF POTENTIOMETRY: APPLICATIONS AND LIMITATIONS

| TABLE 1: APPLICATIONS OF POTENTIOMETRY BY SECTOR & INTERFERENCES/LIMITATIONS                                                                                                                                                                                    |                                                                                                                                             |                                                                                                                                                                                                                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| SECTOR                                                                                                                                                                                                                                                          | KEY APPLICATIONS (ION & pH MEASUREMENT)                                                                                                     | SPECIFIC EXAMPLES & METHODOLOGY                                                                                                                                                                                                                                 | LIMITATIONS & INTERFERENCES (Applicable across all sectors)                                                                                                                                                                                                                                                                                                                                                                                                                                                              |
| ENVIRONMENTAL ANALYSIS                                                                                                                                                                                                                                          | <ul style="list-style-type: none"><li>Water Quality Monitoring (Rivers, Lakes, Wastewater)</li><li>Soil Testing</li></ul>                   | <ul style="list-style-type: none"><li>Fluoride (F<sup>-</sup>), Chloride (Cl<sup>-</sup>), Nitrate (NO<sub>3</sub><sup>-</sup>) levels</li><li>Heavy Metals (e.g., Lead Pb<sup>2+</sup>) trace analysis</li><li>pH measurement for acidity/alkalinity</li></ul> | <ul style="list-style-type: none"><li><b>Interfering Ions (Selectivity Coefficients)</b><br/>Responses to similar ions (e.g., Na<sup>+</sup> with H<sup>+</sup>, Cl<sup>-</sup> with Br<sup>-</sup>) can cause error.</li><li><b>Electrode Drift &amp; Aging</b><br/>Requires frequent calibration and maintenance.</li><li><b>Temperature Effects</b><br/>Dependent on Nernstian slope; requires compensation.</li><li><b>Sample Matrix Effects</b><br/>High ionic strength or turbidity can affect accuracy.</li></ul> |
| PHARMACEUTICAL ANALYSIS                                                                                                                                                                                                                                         | <ul style="list-style-type: none"><li>Drug Formulation Quality Control</li><li>Purity Testing</li></ul>                                     | <ul style="list-style-type: none"><li>pH buffering of solutions</li><li>Sodium (Na<sup>+</sup>) and Potassium (K<sup>+</sup>) content in electrolytes</li><li>Drug Stability testing</li></ul>                                                                  |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| FOOD ANALYSIS                                                                                                                                                                                                                                                   | <ul style="list-style-type: none"><li>Quality Assurance of Beverages &amp; Processed Foods</li><li>Nutritional Label Verification</li></ul> | <ul style="list-style-type: none"><li>Sodium (Na<sup>+</sup>) content in dairy products</li><li>Chloride (Cl<sup>-</sup>) in salts</li><li>pH of fruit juices</li></ul>                                                                                         |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| CLINICAL ANALYSIS (POINT-OF-CARE)                                                                                                                                                                                                                               | <ul style="list-style-type: none"><li>Blood Gas &amp; Electrolyte Analysis</li><li>Patient Diagnostics</li></ul>                            | <ul style="list-style-type: none"><li>Sodium (Na<sup>+</sup>), Potassium (K<sup>+</sup>), Calcium (Ca<sup>2+</sup>), and Chloride (Cl<sup>-</sup>) in blood serum and urine</li><li>pH of biological fluids</li></ul>                                           |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| <b>OER APPLICATION CONTEXT</b> (Link to Research)                                                                                                                                                                                                               |                                                                                                                                             |                                                                                                                                                                                                                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| <div> OER studies (pH monitoring at catalyst-electrolyte interface, &lt;IMAGE_3&gt;) use potentiometry, highlighting its importance for environmental energy conversion.</div> |                                                                                                                                             |                                                                                                                                                                                                                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |

Box 2.2: Applications and limitations of potentiometry.

### Pilot questions 2.2

- Which of the following best defines potentiometry? a. Measurement of potential difference between two electrodes without drawing significant current b. Measurement of mass change of a sample with temperature c. Measurement of heat flow between a sample and a reference d. Measurement of colour change in a solution
- Fill in the blank: Potentiometry measures the potential difference between two \_\_\_\_\_ without drawing significant current.
- Which of the following is the most common application of potentiometry? a. pH measurement b. Conductivity measurement c. Colour intensity measurement d. Pressure measurement
- Fill in the blank: A glass electrode is commonly used in potentiometry to measure \_\_\_\_\_ ion activity.
- Which of the following is a limitation of potentiometric methods? a. Sensitivity to electrode calibration b. Ability to measure electrolytes in blood c. Use in environmental monitoring d. Application in food industry

## 2.3 Other Electroanalytical Techniques

### 2.3.1 Overview of conductometric methods

**Conductometry** is an electroanalytical technique that measures the electrical conductivity of a solution. Conductivity depends on the concentration and mobility of ions present, making this method useful for studying neutralisation reactions, salt content, and water purity.

Conductometric titrations are particularly valuable because they do not require indicators. Instead, changes in conductivity are monitored as titration progresses, allowing precise determination of end points.

Fill in the blank: Conductometry measures the electrical \_\_\_\_\_ of a solution.



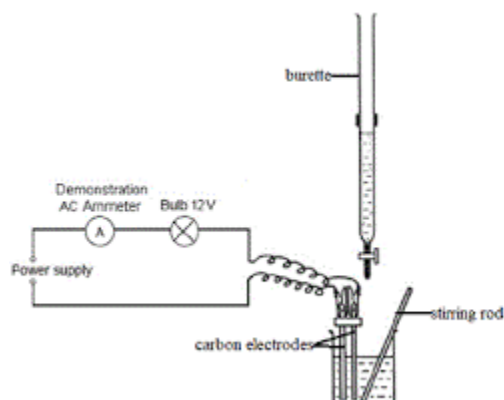


Figure 2.3: Principle of conductometry.

### 2.3.2 Overview of amperometric methods

**Amperometry** is an electroanalytical technique in which the current flowing through an electrochemical cell is measured at a fixed potential. The current is directly related to the concentration of the analyte undergoing oxidation or reduction.

Amperometric titrations are widely used for detecting trace amounts of substances, such as dissolved oxygen in water or glucose in blood samples.

Fill in the blank: In amperometry, the current is measured at a fixed \_\_\_\_\_.

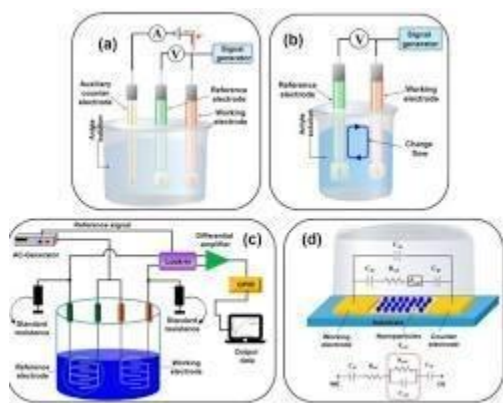


Plate 2.3: Principle of amperometry.

### 2.3.3 Applications in environmental and industrial analysis

Conductometric and amperometric methods are applied in diverse fields:

- **Environmental analysis:** Monitoring water quality by measuring ion content and dissolved oxygen.
- **Industrial analysis:** Quality control in food and beverage industries, such as salt content in dairy products.
- **Biomedical applications:** Measuring glucose levels in blood using amperometric sensors.





- **Chemical research:** Studying reaction kinetics and ion mobility.

Limitations include sensitivity to electrode condition, interference from other ions, and the need for precise calibration.

Fill in the blank: Amperometric sensors are widely used in biomedical applications to measure \_\_\_\_\_ levels in blood.

| Context              | Conductometric Applications                                                                                                | Amperometric Applications                                                                                                        |
|----------------------|----------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------|
| <b>Environmental</b> | Monitoring water purity, detecting salinity, and measuring total dissolved solids (TDS) in wastewater.                     | Detection of heavy metals (e.g., lead, mercury), dissolved oxygen levels, and various pollutants in water supplies.              |
| <b>Industrial</b>    | Quality control in food and beverage production (e.g., milk purity), process monitoring in chemical manufacturing.         | Monitoring fermentation processes, detecting specific chemical analytes in industrial effluents, and titration endpoints.        |
| <b>Biomedical</b>    | Analysis of physiological fluids, monitoring dialysis solutions, and point-of-care diagnostics.                            | Glucose biosensors for diabetes management, detection of neurotransmitters, and monitoring blood gases.                          |
| <b>Research</b>      | Studying ion association in solutions, kinetic studies of ionic reactions, and characterization of electrolyte properties. | Development of novel electrochemical sensors, studying reaction mechanisms at electrode surfaces, and electrocatalysis research. |

Box 2.3: Applications of conductometric and amperometric methods.

#### Pilot questions 2.3

1. Which of the following best defines conductometry? a. Measurement of electrical conductivity of a solution b. Measurement of potential difference between electrodes c. Measurement of heat flow between a sample and a reference d. Measurement of colour change in a solution
2. Fill in the blank: Conductometry measures the electrical \_\_\_\_\_ of a solution.
3. Which of the following best defines amperometry? a. Measurement of current at a fixed potential b. Measurement of mass change with temperature c. Measurement of pressure change with concentration d. Measurement of light absorption in a solution
4. Fill in the blank: In amperometry, the current is measured at a fixed \_\_\_\_\_.
5. Which of the following is an application of amperometric sensors? a. Measuring glucose levels in blood b. Measuring melting points of solids c. Studying colour intensity of dyes d. Measuring magnetic susceptibility



## Series Summary

In this Series, we examined the **principles and applications of electroanalytical and potentiometric methods**, which are central to modern analytical chemistry. We began with the general principles of electroanalytical techniques, highlighting their scope and importance in chemical analysis. We then studied **potentiometric methods**, focusing on how potential differences are measured, the role of pH measurement, and the use of ion-selective electrodes. Finally, we explored **other electroanalytical techniques**, including conductometry and amperometry, and considered their applications in environmental, industrial, and biomedical contexts. Altogether, this Series has provided learners with a clear understanding of how electrical measurements can be used to analyse chemical systems with precision and versatility.

## Glossary of Terms

- **Electroanalytical methods:** Techniques that use electrical measurements to study chemical systems.
- **Potentiometry:** Measurement of potential difference between two electrodes without drawing significant current.
- **pH measurement:** Determination of hydrogen ion activity using a glass electrode and a reference electrode.
- **Ion-selective electrode (ISE):** An electrode designed to respond selectively to a specific ion.
- **Conductometry:** Measurement of electrical conductivity of a solution, often used in titrations.
- **Amperometry:** Measurement of current at a fixed potential, related to the concentration of analytes undergoing oxidation or reduction.
- **End point (titration):** The stage at which a titration reaction is complete, often detected by changes in conductivity or current.

## Concept Check Questions 2

### Objective questions

1. Electroanalytical methods use \_\_\_\_\_ measurements to study chemical systems. (Linked to SLO 2.1)
2. Potentiometry measures the potential difference between two \_\_\_\_\_ without drawing significant current. (Linked to SLO 2.2)
3. A glass electrode is commonly used in potentiometry to measure \_\_\_\_\_ ion activity. (Linked to SLO 2.3)
4. Conductometry measures the electrical \_\_\_\_\_ of a solution. (Linked to SLO 2.6)



5. In amperometry, the current is measured at a fixed \_\_\_\_\_. (Linked to SLO 2.7)

### Short-answer questions

6. Explain the difference between potentiometry and conductometry. (Linked to SLOs 2.2, 2.6)
7. Describe how ion-selective electrodes expand the scope of potentiometry. (Linked to SLO 2.4)
8. Outline the importance of amperometric sensors in biomedical applications. (Linked to SLO 2.8)

### Long-answer questions

9. Analyse the applications of potentiometric methods in environmental, pharmaceutical, food, and clinical chemistry. (Linked to SLOs 2.5)
10. Evaluate the strengths and limitations of conductometric and amperometric methods in industrial and environmental analysis. (Linked to SLOs 2.6–2.8)
11. Discuss how electroanalytical methods provide sensitivity and versatility compared to other analytical techniques. (Linked to SLOs 2.1–2.8)

### Answers To Pilot Questions 2

**Part 2.1:** 1.a, 2.electrical, 3.a, 4.pH, 5.a **Part 2.2:** 1.a, 2.electrodes, 3.a, 4.hydrogen, 5.a **Part 2.3:** 1.a, 2.conductivity, 3.a, 4.potential, 5.a

### References And Attributions 2

- Figures 2.1–2.3: Suggested AI prompts for diagrams showing principles of electroanalytical methods, potentiometry, and conductometry.
- Plates 2.2–2.3: Suggested AI prompts for images of potentiometric and amperometric setups.
- Boxes 2.1–2.3: Suggested AI prompts for tables summarising importance, applications, and limitations of electroanalytical methods.
- Open Educational Resources (OER) suggested search strings:
  - “OER diagram electroanalytical methods chemistry”
  - “OER diagram potentiometry principle chemistry”
  - “OER image potentiometry pH measurement chemistry”
  - “OER table potentiometry applications chemistry”
  - “OER diagram conductometry titration chemistry”
  - “OER image amperometry setup chemistry”



- “OER table conductometry amperometry applications chemistry”
- Attribution style: APA (institutional standard assumed). Example: OpenStax. (2016). *Chemistry*. OpenStax CNX. Retrieved from <https://openstax.org/books/chemistry>

**Course code: CHM 410**

**Course title: Analytical Chemistry II**

**Course credit load: 2 Units**

**Series 1: Thermal Methods Of Analysis**

**Series 2: Electroanalytical And Potentiometric Techniques**

**Series 3: Conductometric, Amperometric And Colorimetric Methods**

**Series 4: Chromatographic Principles And Coupled Techniques**

**Series 5: Radiochemical Methods In Environmental Analysis**

**Proposed Outline Series 3**

### **3.1 Conductometric Methods Of Analysis**

- 3.1.1 Principles of conductometry
- 3.1.2 Conductometric titrations and applications
- 3.1.3 Limitations of conductometry

### **3.2 Amperometric Methods Of Analysis**

- 3.2.1 Principles of amperometry
- 3.2.2 Amperometric titrations and applications
- 3.2.3 Limitations of amperometry

### **3.3 Colorimetric Methods Of Analysis**

- 3.3.1 Principles of colorimetry
- 3.3.2 Instrumentation and measurement of absorbance
- 3.3.3 Applications in chemical and biological analysis
- 3.3.4 Limitations of colorimetric methods



## Introduction

In analytical chemistry, measuring how substances respond to electrical and optical signals provides powerful insights into their composition and behaviour. **Conductometric, amperometric, and colorimetric techniques** are widely used because they offer sensitivity, accuracy, and versatility across diverse applications. These methods are essential in environmental monitoring, industrial quality control, and biomedical analysis.

The aim of this Series is to introduce you to the principles and applications of conductometric, amperometric, and colorimetric methods, enabling you to explain their operation, interpret their outputs, and evaluate their significance in analytical chemistry.

We shall commence this Series by examining conductometric methods, focusing on their principles, titrations, and limitations. Next, we will explore amperometric methods, considering how current measurements at fixed potentials are applied in titrations and analysis. Finally, we will conclude with colorimetric methods, studying how absorbance measurements are used to determine concentrations in chemical and biological systems, along with their applications and limitations.

## Series Learning Outcomes

Upon completing this Series, you should be able to:

- **SLO 3.1** define conductometric methods and explain their principles,
- **SLO 3.2** demonstrate how conductometric titrations are performed and applied,
- **SLO 3.3** analyse the limitations of conductometric methods,
- **SLO 3.4** describe the principles of amperometric methods,
- **SLO 3.5** demonstrate how amperometric titrations are conducted and applied,
- **SLO 3.6** evaluate the limitations of amperometric methods,
- **SLO 3.7** explain the principles of colorimetric methods,
- **SLO 3.8** demonstrate how absorbance is measured using colorimetric instrumentation,
- **SLO 3.9** analyse the applications of colorimetric methods in chemical and biological analysis,
- **SLO 3.10** evaluate the limitations of colorimetric methods.

### 3.1 Conductometric Methods Of Analysis





### 3.1.1 Principles of conductometry

**Conductometry** is an electroanalytical technique that measures the **electrical conductivity** of a solution. Conductivity depends on the concentration and mobility of ions present, and it changes during chemical reactions such as neutralisation or precipitation. This makes conductometry particularly useful for monitoring reactions without the need for visual indicators.

Fill in the blank: Conductometry measures the electrical \_\_\_\_\_ of a solution.

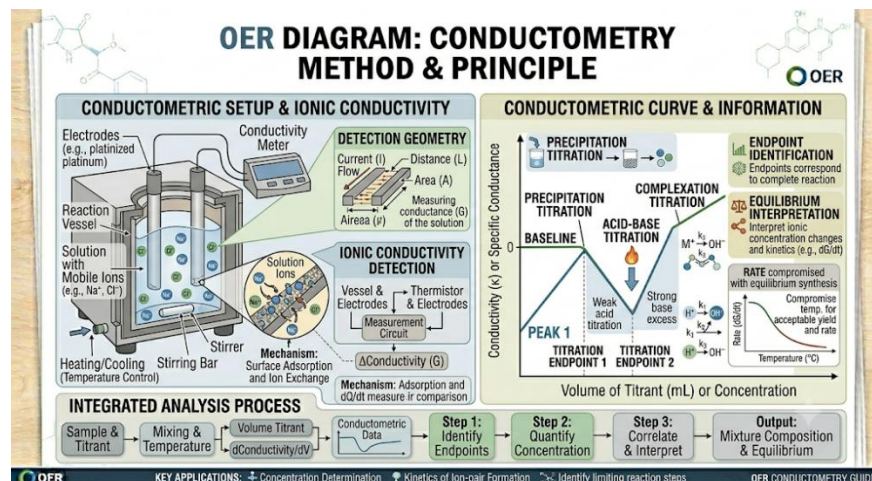


Figure 3.1: Principle of conductometry.

### 3.1.2 Conductometric titrations and applications

**Conductometric titrations** involve monitoring changes in conductivity as a titrant is added to a solution. The end point is determined by a sharp change in conductivity, which corresponds to the completion of the reaction. Unlike traditional titrations, conductometric titrations do not require colour indicators, making them suitable for coloured or turbid solutions.

Applications include:

- Determining the concentration of strong and weak acids or bases.
- Measuring salt content in food and beverages.
- Monitoring water purity and ion content.
- Studying precipitation reactions.

Fill in the blank: Conductometric titrations determine the end point by monitoring changes in \_\_\_\_\_.



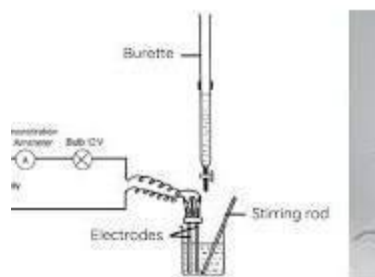


Plate 3.1: Conductometric titration setup.

### 3.1.3 Limitations of conductometry

While conductometry is versatile, it has certain limitations:

- It is less effective for reactions that do not involve significant ionic changes.
- Results can be influenced by temperature fluctuations, requiring careful control.
- Electrodes must be properly maintained to avoid inaccurate readings.
- It may not distinguish between different ions contributing to conductivity.

Fill in the blank: Conductometry results can be influenced by fluctuations in \_\_\_\_\_.

| Limitation                     | Description                                                                                               | Impact on Analysis                                                                       |
|--------------------------------|-----------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------|
| <b>Temperature Sensitivity</b> | Conductivity is highly dependent on temperature ( $\kappa$ increases $\sim 2\%$ per $^{\circ}\text{C}$ ). | Requires precise temperature control or compensation to avoid significant errors.        |
| <b>Electrode Maintenance</b>   | Electrodes (e.g., platinized platinum) can degrade, foul, or lose calibration.                            | Leads to inconsistent readings; necessitates frequent cleaning and recalibration.        |
| <b>Ion Interference</b>        | Measures total ionic conductivity; cannot distinguish between different ion types.                        | Limited selectivity in complex mixtures; requires prior separation or specific reagents. |
| <b>Concentration Range</b>     | Conductometry is generally restricted to dilute solutions.                                                | Deviations from linearity occur at high concentrations due to inter-ionic effects.       |

Box 3.1: Limitations of conductometry.

#### Pilot questions 3.1

1. Which of the following best defines conductometry? a. Measurement of electrical conductivity of a solution b. Measurement of potential difference between electrodes c. Measurement of heat flow between a sample and a reference d. Measurement of colour change in a solution



- Fill in the blank: Conductometry measures the electrical \_\_\_\_\_ of a solution.
- Which of the following is a key advantage of conductometric titrations? a. They do not require colour indicators b. They require large sample volumes c. They are limited to transparent solutions d. They cannot be used for acids and bases
- Fill in the blank: Conductometric titrations determine the end point by monitoring changes in \_\_\_\_\_.
- Which of the following is a limitation of conductometry? a. Sensitivity to temperature fluctuations b. Ability to measure salt content in food c. Use in monitoring water purity d. Application in precipitation reactions

## 3.2 Amperometric Methods Of Analysis

### 3.2.1 Principles of amperometry

**Amperometry** is an electroanalytical technique in which the **current** flowing through an electrochemical cell is measured at a fixed potential. The magnitude of the current is directly related to the concentration of the analyte undergoing oxidation or reduction. This makes amperometry particularly useful for detecting trace amounts of substances.

Fill in the blank: In amperometry, the current is measured at a fixed \_\_\_\_\_.

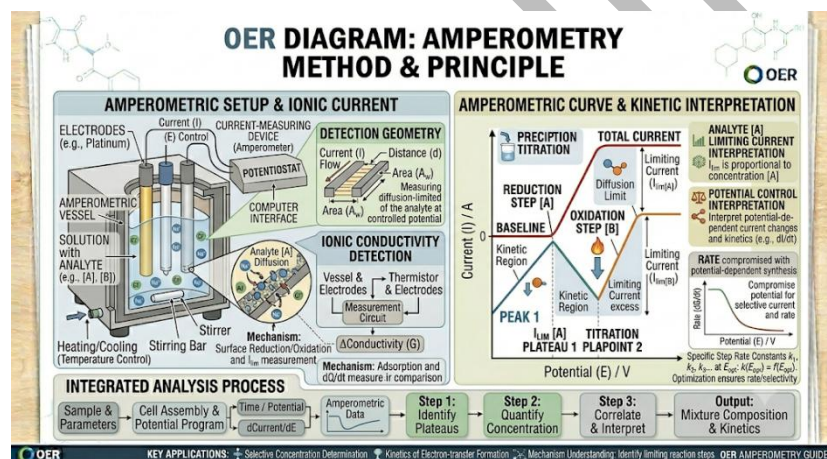


Figure 3.2: Principle of amperometry.

### 3.2.2 Amperometric titrations and applications

**Amperometric titrations** involve monitoring the current as a titrant is added to a solution. The end point is determined by a sudden change in current, which corresponds to the completion of the reaction. These titrations are highly sensitive and can detect very low concentrations of analytes.

Applications include:

- Determining dissolved oxygen in water samples.



- Measuring glucose levels in blood using biosensors.
- Studying redox reactions in chemical research.
- Monitoring pollutants in environmental analysis.

Fill in the blank: Amperometric titrations determine the end point by monitoring changes in \_\_\_\_\_.



Plate 3.2: Amperometric titration setup.

### 3.2.3 Limitations of amperometry

Although amperometry is highly sensitive, it has limitations:

- It requires precise control of electrode potential.
- Electrodes must be carefully maintained to avoid contamination.
- Interference from other electroactive species can affect accuracy.
- It may not be suitable for reactions with slow kinetics.

Fill in the blank: Amperometry results can be affected by interference from other \_\_\_\_\_ species.

| Limitation                   | Description                                                                                                   | Impact on Analysis                                                                                |
|------------------------------|---------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------|
| <b>Electrode Maintenance</b> | Surfaces can become poisoned or fouled by reaction products or contaminants.                                  | Results in reduced sensitivity and drift; necessitates frequent cleaning and re-activation.       |
| <b>Interference</b>          | Non-selective nature means other electroactive species in the sample can also react at the applied potential. | Compromises accuracy in complex mixtures; requires separation or specific potential optimization. |



| Limitation                       | Description                                                                  | Impact on Analysis                                                                                |
|----------------------------------|------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------|
| <b>Potential Control</b>         | The current response is highly sensitive to the exact potential applied (E). | Requires precise instrumentation (potentiostat) to maintain stability and prevent side reactions. |
| <b>Mass Transport Dependency</b> | Current depends on the diffusion of the analyte to the electrode surface.    | Influenced by stirring, temperature, and viscosity, requiring highly reproducible conditions.     |

Box 3.2: Limitations of amperometry.

#### Pilot questions 3.2

- Which of the following best defines amperometry? a. Measurement of current at a fixed potential b. Measurement of mass change with temperature c. Measurement of heat flow between a sample and a reference d. Measurement of colour change in a solution
- Fill in the blank: In amperometry, the current is measured at a fixed \_\_\_\_\_.
- Which of the following is an application of amperometric titrations? a. Determining dissolved oxygen in water b. Measuring melting points of solids c. Studying colour intensity of dyes d. Measuring magnetic susceptibility
- Fill in the blank: Amperometric titrations determine the end point by monitoring changes in \_\_\_\_\_.
- Which of the following is a limitation of amperometry? a. Interference from other electroactive species b. Ability to measure glucose in blood c. Use in environmental analysis d. Application in redox studies

### 3.3 Colorimetric Methods Of Analysis

#### 3.3.1 Principles of colorimetry

**Colorimetry** is an analytical technique that measures the intensity of colour in a solution to determine the concentration of a substance. The principle is based on the fact that the amount of light absorbed by a coloured solution is directly proportional to the concentration of the absorbing species, according to Beer–Lambert’s law.

This makes colorimetry particularly useful for analysing compounds that form coloured complexes or naturally exhibit colour.

Fill in the blank: The amount of light absorbed by a coloured solution is directly proportional to its \_\_\_\_\_.





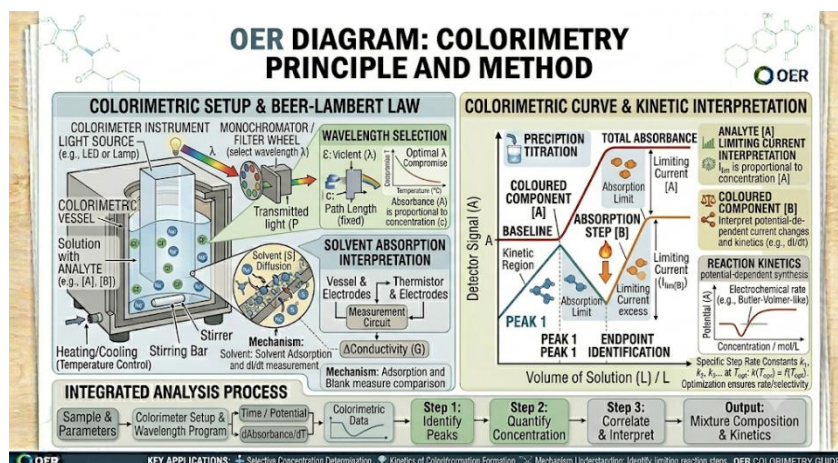


Figure 3.3: Principle of colorimetry.

### 3.3.2 Instrumentation and measurement of absorbance

A **colorimeter** consists of a light source, a filter or monochromator to select specific wavelengths, a sample holder (cuvette), and a detector to measure transmitted light. The instrument calculates absorbance, which is related to concentration.

Absorbance measurements are widely used in both qualitative and quantitative analysis. By preparing a calibration curve with known concentrations, the concentration of unknown samples can be determined accurately.

Fill in the blank: A colorimeter uses a light source, filter, cuvette, and \_\_\_\_\_ to measure transmitted light.

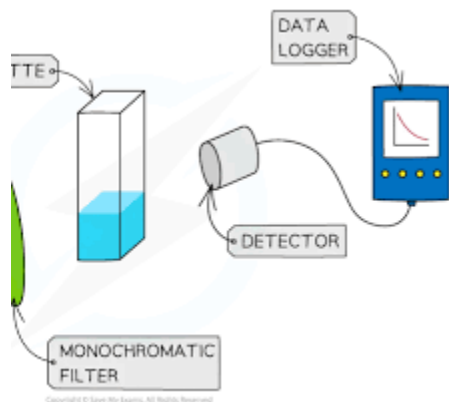


Plate 3.3: Basic colorimeter setup.

### 3.3.3 Applications in chemical and biological analysis

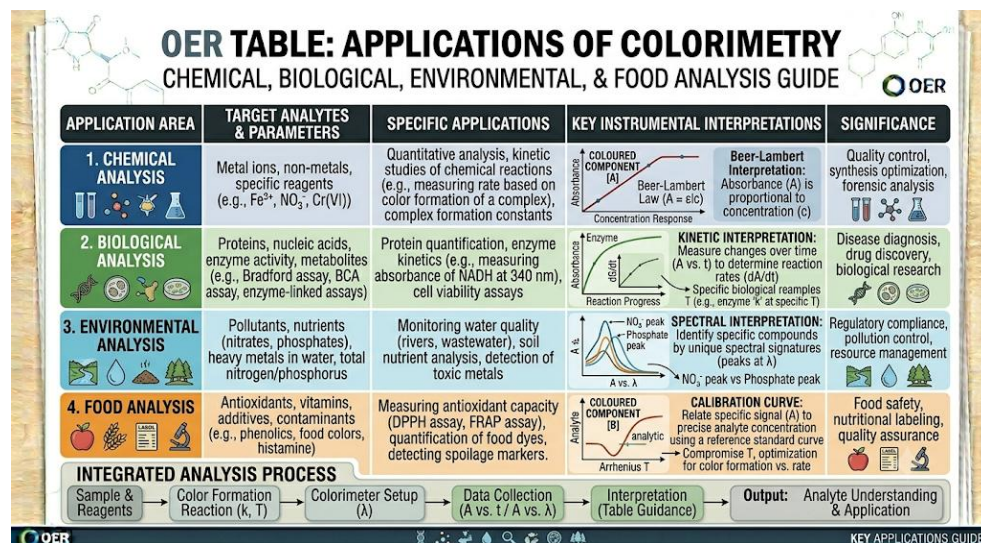
Colorimetric methods are applied in diverse fields:

- **Chemical analysis:** Determining concentrations of metal ions that form coloured complexes.
- **Biological analysis:** Measuring glucose, proteins, and enzymes in clinical samples.



- **Environmental analysis:** Monitoring pollutants such as nitrates and phosphates in water.
- **Food industry:** Assessing colour quality and nutrient content in beverages and dairy products.

Fill in the blank: Colorimetric methods are widely used in clinical chemistry to measure \_\_\_\_\_ levels in blood samples.



Box 3.3: Applications of colorimetry.

### 3.3.4 Limitations of colorimetric methods

Despite their usefulness, colorimetric methods have limitations:

- They are restricted to substances that are coloured or can form coloured complexes.
- Interference from turbidity or other coloured species can affect accuracy.
- Results depend on proper calibration and instrument maintenance.
- Sensitivity may be lower compared to advanced spectrophotometric techniques.

Fill in the blank: Colorimetric methods are restricted to substances that are naturally coloured or can form \_\_\_\_\_ complexes.

| Limitation                 | Description                                                              | Impact on Analysis                                                                        |
|----------------------------|--------------------------------------------------------------------------|-------------------------------------------------------------------------------------------|
| <b>Species Restriction</b> | Analyte must be inherently coloured or react to form a coloured complex. | Requires specific reagents to develop colour, which may not be feasible for all analytes. |
| <b>Interference</b>        | Other coloured species or turbid samples can absorb or scatter light.    | Can lead to inaccurate absorbance readings and false positives.                           |



| Limitation                | Description                                                                           | Impact on Analysis                                                                        |
|---------------------------|---------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------|
| <b>Calibration Needs</b>  | Relies on the Beer-Lambert law ( $A = \epsilon lc$ ), requiring a standard curve.     | Demands accurate preparation of standard solutions for every analysis to ensure validity. |
| <b>Sensitivity Issues</b> | Limited by the sensitivity of the detector and the molar absorptivity of the analyte. | May struggle with detecting very low concentrations of certain analytes.                  |

Box 3.4: Limitations of colorimetry.

#### Pilot questions 3.3

- Which of the following best defines colorimetry? a. Measurement of colour intensity to determine concentration b. Measurement of mass change with temperature c. Measurement of heat flow between a sample and a reference d. Measurement of potential difference between electrodes
- Fill in the blank: The amount of light absorbed by a coloured solution is directly proportional to its \_\_\_\_\_.
- Which of the following components is part of a colorimeter? a. Light source b. Microscope c. Spectrophotometer d. Electrode
- Fill in the blank: A colorimeter uses a light source, filter, cuvette, and \_\_\_\_\_ to measure transmitted light.
- Which of the following is a limitation of colorimetric methods? a. Restriction to coloured species b. Ability to measure glucose in blood c. Use in environmental analysis d. Application in food industry

### Series Summary

In this Series, we explored the **principles and applications of conductometric, amperometric, and colorimetric techniques**, which are essential electroanalytical and optical methods in analytical chemistry. We began with conductometry, focusing on how electrical conductivity is measured and applied in titrations. We then studied amperometry, examining how current at fixed potentials is used in sensitive titrations and detection. Finally, we concluded with colorimetry, highlighting how absorbance measurements are applied in chemical, biological, environmental, and food analysis. Together, these methods provide learners with versatile tools for monitoring reactions, detecting analytes, and ensuring quality control across diverse fields.

### Glossary of Terms

- Conductometry:** Measurement of electrical conductivity of a solution, often used in titrations.



- **Conductometric titration:** A titration where the end point is determined by changes in conductivity.
- **Amperometry:** Measurement of current at a fixed potential, related to analyte concentration.
- **Amperometric titration:** A titration where the end point is determined by changes in current.
- **Colorimetry:** Measurement of colour intensity to determine concentration of a substance.
- **Beer–Lambert’s law:** A principle stating that absorbance is directly proportional to concentration.
- **Calibration curve:** A graph of absorbance versus concentration used to determine unknown concentrations.
- **Ion mobility:** The speed at which ions move under an electric field, affecting conductivity.

### Concept Check Questions 3

#### Objective questions

1. Conductometry measures the electrical \_\_\_\_\_ of a solution. (Linked to SLO 3.1)
2. Conductometric titrations determine the end point by monitoring changes in \_\_\_\_\_. (Linked to SLO 3.2)
3. In amperometry, the current is measured at a fixed \_\_\_\_\_. (Linked to SLO 3.4)
4. Amperometric titrations determine the end point by monitoring changes in \_\_\_\_\_. (Linked to SLO 3.5)
5. The amount of light absorbed by a coloured solution is directly proportional to its \_\_\_\_\_. (Linked to SLO 3.7)
6. A colorimeter uses a light source, filter, cuvette, and \_\_\_\_\_ to measure transmitted light. (Linked to SLO 3.8)

#### Short-answer questions

7. Explain the difference between conductometric and amperometric titrations. (Linked to SLOs 3.2, 3.5)
8. Describe how Beer–Lambert’s law is applied in colorimetry. (Linked to SLO 3.7)
9. Outline the importance of calibration curves in colorimetric analysis. (Linked to SLO 3.8)

#### Long-answer questions



10. Analyse the applications of conductometric methods in food, water, and environmental analysis. (Linked to SLO 3.2–3.3)
11. Evaluate the strengths and limitations of amperometric methods in biomedical and environmental contexts. (Linked to SLOs 3.5–3.6)
12. Discuss how colorimetric methods are applied in chemical, biological, and industrial analysis, and assess their limitations compared to spectrophotometry. (Linked to SLOs 3.9–3.10)

### Answers To Pilot Questions 3

**Part 3.1:** 1.a, 2.conductivity, 3.a, 4.conductivity, 5.a **Part 3.2:** 1.a, 2.potential, 3.a, 4.current, 5.a

**Part 3.3:** 1.a, 2.concentration, 3.a, 4.detector, 5.a

### References And Attributions 3

- Figures 3.1–3.3: Suggested AI prompts for diagrams showing principles of conductometry, amperometry, and colorimetry.
- Plates 3.1–3.3: Suggested AI prompts for images of conductometric titration, amperometric titration, and colorimeter setup.
- Boxes 3.1–3.4: Suggested AI prompts for tables summarising limitations and applications of conductometry, amperometry, and colorimetry.
- Open Educational Resources (OER) suggested search strings:
  - “OER diagram conductometry principle chemistry”
  - “OER image conductometric titration chemistry”
  - “OER diagram amperometry principle chemistry”
  - “OER image amperometric titration chemistry”
  - “OER diagram colorimetry principle chemistry”
  - “OER image colorimeter setup chemistry”
  - “OER table colorimetry applications chemistry”
  - “OER table colorimetry limitations chemistry”
- Attribution style: APA (institutional standard assumed). Example: OpenStax. (2016). *Chemistry*. OpenStax CNX. Retrieved from <https://openstax.org/books/chemistry>





**Course code: CHM 410**

**Course title: Analytical Chemistry II**

**Course credit load: 2 Units**

**Series 1: Thermal Methods Of Analysis**

**Series 2: Electroanalytical And Potentiometric Techniques**

**Series 3: Conductometric, Amperometric And Colorimetric Methods**

**Series 4: Chromatographic Principles And Coupled Techniques**

**Series 5: Radiochemical Methods In Environmental Analysis**

**Proposed Outline Series 4**

#### **4.1 Fundamentals Of Chromatography**

- 4.1.1 Definition and scope of chromatography
- 4.1.2 Classification of chromatographic methods
- 4.1.3 General principles of separation

#### **4.2 Major Chromatographic Techniques**

- 4.2.1 Principles of gas chromatography (GC)
- 4.2.2 Principles of liquid chromatography (LC)
- 4.2.3 Applications of GC and LC

#### **4.3 Coupled Chromatographic Techniques**

- 4.3.1 Chromatography–mass spectrometry (GC–MS and LC–MS)
- 4.3.2 Chromatography–infrared spectroscopy (LC–IR)
- 4.3.3 Applications of coupled techniques in analytical chemistry

### **Introduction**

Chromatography is one of the most powerful tools in analytical chemistry, allowing scientists to separate, identify, and quantify components in complex mixtures. From environmental monitoring to pharmaceutical quality control, chromatographic methods are indispensable in modern laboratories. Coupled techniques, such as chromatography linked with mass



spectrometry, further enhance analytical power by combining separation with precise identification.

The aim of this Series is to introduce you to the principles of chromatography, explain the operation of major chromatographic techniques, and highlight the role of coupled methods in advanced analysis.

We shall commence this Series by exploring the fundamentals of chromatography, including its definition, scope, and general principles of separation. Next, we will examine major chromatographic techniques such as gas chromatography and liquid chromatography, considering their principles and applications. Finally, we will conclude with coupled chromatographic techniques, focusing on how methods like GC–MS and LC–MS are applied in analytical chemistry to achieve highly accurate results.

## Series Learning Outcomes

Upon completing this Series, you should be able to:

- **SLO 4.1** define chromatography and explain its scope in analytical chemistry,
- **SLO 4.2** classify chromatographic methods based on their principles of separation,
- **SLO 4.3** describe the general principles of chromatographic separation,
- **SLO 4.4** explain the principles of gas chromatography (GC),
- **SLO 4.5** explain the principles of liquid chromatography (LC),
- **SLO 4.6** analyse the applications of GC and LC in analytical chemistry,
- **SLO 4.7** describe the principles of coupled techniques such as GC–MS and LC–MS,
- **SLO 4.8** explain the principles of chromatography–infrared spectroscopy (LC–IR),
- **SLO 4.9** evaluate the applications of coupled chromatographic techniques in advanced analysis.

### 4.1 Fundamentals Of Chromatography

#### 4.1.1 Definition and scope of chromatography

**Chromatography** is a separation technique used to isolate and analyse components of a mixture based on their distribution between a **stationary phase** (a solid or liquid fixed in place) and a **mobile phase** (a liquid or gas that moves through the stationary phase). The differences in how substances interact with these phases allow them to be separated and identified.



Chromatography is widely applied in environmental monitoring, pharmaceutical analysis, food quality control, and forensic science.

Fill in the blank: Chromatography separates components of a mixture based on their distribution between a stationary phase and a \_\_\_\_\_ phase.

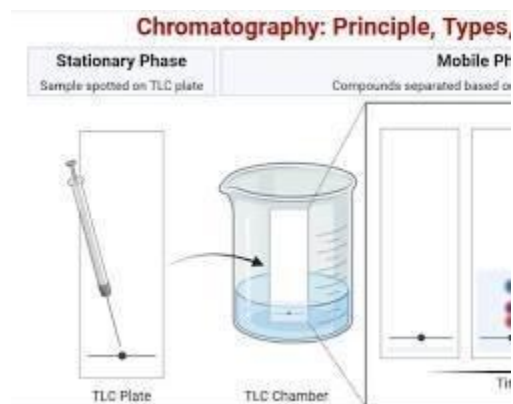


Figure 4.1: Principle of chromatography.

#### 4.1.2 Classification of chromatographic methods

Chromatographic methods can be classified based on the nature of the mobile phase and the separation mechanism:

- **Gas chromatography (GC):** Uses a gaseous mobile phase to separate volatile compounds.
- **Liquid chromatography (LC):** Uses a liquid mobile phase to separate compounds in solution.
- **Thin-layer chromatography (TLC):** Uses a thin layer of adsorbent material as the stationary phase.
- **Paper chromatography:** Uses paper as the stationary phase for simple separations.

Fill in the blank: Gas chromatography uses a \_\_\_\_\_ mobile phase to separate volatile compounds.

| Type of Chromatography            | Stationary Phase                                | Mobile Phase                          | Typical Key Features                                                  |
|-----------------------------------|-------------------------------------------------|---------------------------------------|-----------------------------------------------------------------------|
| <b>Gas Chromatography (GC)</b>    | High-boiling liquid or solid coated on a column | Inert gas (e.g., Helium, Nitrogen)    | Used for volatile compounds; high resolution and fast analysis times. |
| <b>Liquid Chromatography (LC)</b> | Solid particles (e.g., silica, alumina)         | Liquid solvent or mixture of solvents | Used for non-volatile or thermally unstable                           |



| Type of Chromatography                 | Stationary Phase                         | Mobile Phase                          | Typical Key Features                                                        |
|----------------------------------------|------------------------------------------|---------------------------------------|-----------------------------------------------------------------------------|
|                                        |                                          |                                       | compounds; highly versatile.                                                |
| <b>Thin-Layer Chromatography (TLC)</b> | Thin layer of solid adsorbent on a plate | Liquid solvent (via capillary action) | Quick, inexpensive qualitative analysis; excellent for reaction monitoring. |
| <b>Paper Chromatography</b>            | Cellulose fibers in filter paper         | Liquid solvent                        | Simple technique often used for separating pigments or small molecules.     |

Box 4.1: Classification of chromatographic methods.

### 4.1.3 General principles of separation

The separation in chromatography depends on differences in **affinity** of substances for the stationary and mobile phases. Substances with stronger interactions with the stationary phase move more slowly, while those with weaker interactions move faster. This difference in migration rates leads to separation.

Key factors influencing separation include:

- Polarity of the stationary and mobile phases.
- Solubility of the analytes.
- Temperature and pressure conditions.
- Flow rate of the mobile phase.

Fill in the blank: Substances with stronger interactions with the stationary phase move more \_\_\_\_\_ during chromatography.

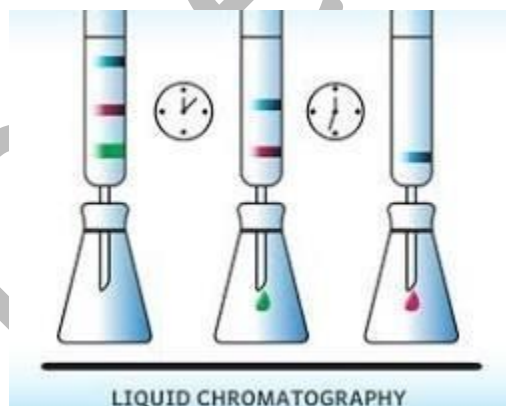


Plate 4.1: Chromatographic separation in a column.



#### Pilot questions 4.1

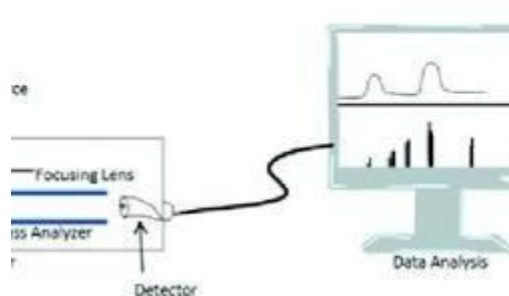
1. Which of the following best defines chromatography? a. A separation technique based on distribution between stationary and mobile phases b. A method of measuring heat flow in a sample c. A technique for measuring electrical conductivity of a solution d. A method of measuring colour intensity in a solution
2. Fill in the blank: Chromatography separates components of a mixture based on their distribution between a stationary phase and a \_\_\_\_\_ phase.
3. Which of the following is a type of chromatography? a. Gas chromatography b. Electrogravimetry c. Calorimetry d. Potentiometry
4. Fill in the blank: Gas chromatography uses a \_\_\_\_\_ mobile phase to separate volatile compounds.
5. Which of the following factors influences chromatographic separation? a. Polarity of phases b. Flow rate of mobile phase c. Temperature conditions d. All of the above

## 4.2 Major Chromatographic Techniques

### 4.2.1 Principles of gas chromatography (GC)

**Gas chromatography (GC)** is a technique in which a gaseous mobile phase carries volatile compounds through a column containing a stationary phase. The separation occurs because different compounds travel at different speeds depending on their volatility and interaction with the stationary phase. GC is particularly useful for analysing gases and volatile liquids.

Fill in the blank: Gas chromatography uses a \_\_\_\_\_ mobile phase to separate volatile Figure



4.2: Principle of gas chromatography.

### 4.2.2 Principles of liquid chromatography (LC)

**Liquid chromatography (LC)** uses a liquid mobile phase to separate compounds dissolved in solution. The stationary phase may be a solid adsorbent or a liquid immobilised on a solid support. High-performance liquid chromatography (HPLC) is a widely used form of LC that provides high resolution and sensitivity.





LC is suitable for non-volatile and thermally unstable compounds, making it versatile for pharmaceutical, biochemical, and environmental applications.

Fill in the blank: Liquid chromatography uses a \_\_\_\_\_ mobile phase to separate compounds in solution.

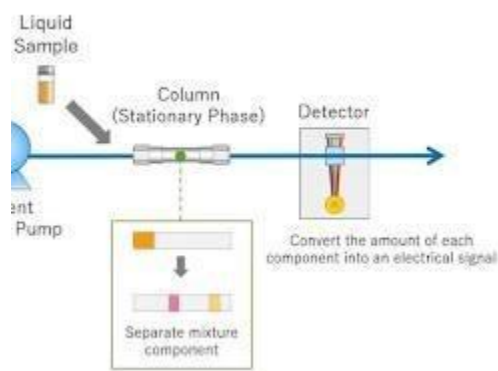


Plate 4.2: Principle of liquid chromatography.

### 4.2.3 Applications of GC and LC

Both GC and LC are widely applied in analytical chemistry:

- **Gas chromatography:** Used in environmental analysis for detecting pollutants, in forensic science for drug testing, and in petrochemical industries for hydrocarbon analysis.
- **Liquid chromatography:** Applied in pharmaceutical quality control, biochemical research for protein and enzyme analysis, and food industry for detecting additives and contaminants.

Fill in the blank: Gas chromatography is widely used in forensic science for \_\_\_\_\_ testing.

Gas chromatography (GC) and liquid chromatography (LC) are essential analytical tools utilized across various sectors.

- **Environmental Analysis:** GC is frequently used for detecting volatile organic compounds (VOCs) and pesticides in water or soil samples, while LC is often employed for non-volatile organic pollutants and large polar molecules.
- **Pharmaceutical Industry:** LC (specifically HPLC) is the primary method for testing drug purity, stability, and quantifying active ingredients. GC is commonly used to ensure the absence of residual solvents in pharmaceutical products.
- **Forensic Science:** GC is widely used for toxicology screenings, such as analyzing blood alcohol levels or arson accelerants. LC is utilized for analyzing drugs of abuse that are thermally unstable or non-volatile, such as certain opioids and controlled substances.
- **Industrial Processes:** GC monitors quality control for petrochemicals and natural gas purity. LC is applied to monitor reaction mixtures, ensure product quality in food and beverage manufacturing, and analyze additives or preservatives.



## Box 4.2: Applications of GC and LC.

### Pilot questions 4.2

1. Which of the following best defines gas chromatography? a. Separation technique using a gaseous mobile phase b. Separation technique using a liquid mobile phase c. Separation technique using paper as stationary phase d. Separation technique using thin-layer adsorbent
2. Fill in the blank: Gas chromatography uses a \_\_\_\_\_ mobile phase to separate volatile compounds.
3. Which of the following best defines liquid chromatography? a. Separation technique using a liquid mobile phase b. Separation technique using a gaseous mobile phase c. Separation technique using paper as stationary phase d. Separation technique using thin-layer adsorbent
4. Fill in the blank: Liquid chromatography uses a \_\_\_\_\_ mobile phase to separate compounds in solution.
5. Which of the following is an application of gas chromatography? a. Drug testing in forensic science b. Protein analysis in biochemistry c. Quality control in pharmaceuticals d. Detection of food additives

## 4.3 Coupled Chromatographic Techniques

### 4.3.1 Chromatography–mass spectrometry (GC–MS and LC–MS)

**Chromatography–mass spectrometry (GC–MS and LC–MS)** combines the separation power of chromatography with the identification capability of mass spectrometry. In GC–MS, volatile compounds are first separated by gas chromatography and then identified by their mass spectra. LC–MS works similarly but uses liquid chromatography to separate non-volatile compounds before mass spectrometric detection.

This coupling allows both qualitative and quantitative analysis with high sensitivity and specificity.

Fill in the blank: GC–MS combines gas chromatography with \_\_\_\_\_ spectrometry for compound identification.

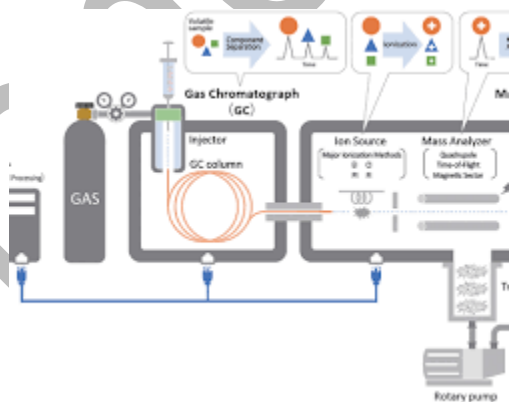


Figure 4.3: Principle of GC–MS.

### 4.3.2 Chromatography–infrared spectroscopy (LC–IR)

**Chromatography–infrared spectroscopy (LC–IR)** couples liquid chromatography with infrared spectroscopy. After separation, compounds are analysed by their infrared absorption spectra, which provide information about functional groups and molecular structure.

LC–IR is particularly useful for studying complex mixtures and identifying organic compounds with characteristic IR spectra.

Fill in the blank: LC–IR couples liquid chromatography with \_\_\_\_\_ spectroscopy to identify functional groups.

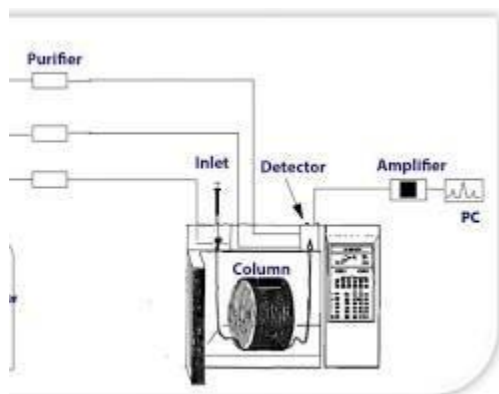


Plate 4.3: Principle of LC–IR.

### 4.3.3 Applications of coupled techniques in analytical chemistry

Coupled chromatographic techniques are applied in a wide range of fields:

- **Environmental analysis:** Detecting trace pollutants and contaminants in air, water, and soil.
- **Pharmaceuticals:** Identifying drug metabolites and ensuring purity of formulations.
- **Forensic science:** Analysing toxic substances, drugs, and explosives.
- **Biochemistry:** Studying proteins, peptides, and complex biomolecules.

These techniques provide unmatched sensitivity and specificity, making them indispensable in modern analytical laboratories.

Fill in the blank: Coupled chromatographic techniques are widely used in forensic science to analyse \_\_\_\_\_ substances.



Coupled chromatography-spectroscopy techniques provide enhanced identification capabilities by combining the separation power of chromatography with the structural analysis of spectroscopy or mass spectrometry.

- **Environmental Analysis:** GC-MS is the gold standard for identifying volatile organic pollutants (e.g., pesticides, PCBs) in water and air. LC-MS is preferred for identifying polar or thermally labile environmental toxins and emerging contaminants. LC-IR is used to investigate structural features of unknown organic compounds in environmental matrices.
- **Pharmaceutical Industry:** GC-MS is used for identification of process-related impurities and volatile residual solvents. LC-MS is critical for drug development, metabolite identification, and impurity profiling. LC-IR provides structural verification, particularly for complex drug degradation products.
- **Forensic Science:** GC-MS remains essential for identifying drugs, poisons, and accelerants in complex forensic samples. LC-MS is increasingly used for large, non-volatile molecules like biological toxins and modern synthetic drugs. LC-IR offers unique fingerprinting capabilities for identifying counterfeit pharmaceuticals.
- **Biochemical Analysis:** GC-MS is utilized for metabolic profiling of small volatile metabolites. LC-MS is the workhorse for proteomics, lipidomics, and metabolomics, allowing for the analysis of high-molecular-weight biomolecules. LC-IR is used in structural studies of biopolymers and lipids to analyze functional groups.

Box 4.3: Applications of coupled chromatographic techniques.

#### Pilot questions 4.3

1. Which of the following best defines GC-MS? a. Combination of gas chromatography and mass spectrometry b. Combination of liquid chromatography and infrared spectroscopy c. Combination of paper chromatography and UV spectroscopy d. Combination of thin-layer chromatography and calorimetry
2. Fill in the blank: GC-MS combines gas chromatography with \_\_\_\_\_ spectrometry for compound identification.
3. Which of the following best defines LC-IR? a. Combination of liquid chromatography and infrared spectroscopy b. Combination of gas chromatography and mass spectrometry c. Combination of paper chromatography and UV spectroscopy d. Combination of thin-layer chromatography and calorimetry
4. Fill in the blank: LC-IR couples liquid chromatography with \_\_\_\_\_ spectroscopy to identify functional groups.
5. Which of the following is an application of coupled chromatographic techniques? a. Analysing toxic substances in forensic science b. Measuring melting points of solids c. Determining electrical conductivity of solutions d. Studying colour intensity of dyes



## Series Summary

In this Series, we studied the **principles and applications of chromatographic methods and coupled techniques**, which are central to modern analytical chemistry. We began with the fundamentals of chromatography, examining its definition, scope, classification, and general principles of separation. We then explored major chromatographic techniques, focusing on gas chromatography (GC) and liquid chromatography (LC), and considered their applications in environmental, pharmaceutical, and forensic contexts. Finally, we concluded with coupled chromatographic techniques such as GC–MS, LC–MS, and LC–IR, highlighting how these combinations enhance analytical power by linking separation with precise identification. Altogether, this Series has provided learners with a comprehensive understanding of how chromatography and its coupled techniques are applied in advanced chemical analysis.

## Glossary of Terms

- **Chromatography:** A separation technique based on distribution between stationary and mobile phases.
- **Stationary phase:** The fixed solid or liquid phase in chromatography.
- **Mobile phase:** The moving liquid or gas phase in chromatography.
- **Gas chromatography (GC):** Chromatography using a gaseous mobile phase for volatile compounds.
- **Liquid chromatography (LC):** Chromatography using a liquid mobile phase for compounds in solution.
- **High-performance liquid chromatography (HPLC):** A high-resolution form of LC.
- **GC–MS:** Coupling of gas chromatography with mass spectrometry for compound identification.
- **LC–MS:** Coupling of liquid chromatography with mass spectrometry for non-volatile compounds.
- **LC–IR:** Coupling of liquid chromatography with infrared spectroscopy for functional group analysis.
- **Affinity:** The degree of interaction between a substance and the stationary or mobile phase.

## Concept Check Questions 4

### Objective questions

1. Chromatography separates components of a mixture based on their distribution between a stationary phase and a \_\_\_\_\_ phase. (Linked to SLO 4.1)
2. Gas chromatography uses a \_\_\_\_\_ mobile phase to separate volatile compounds. (Linked to SLO 4.4)





3. Liquid chromatography uses a \_\_\_\_\_ mobile phase to separate compounds in solution. (Linked to SLO 4.5)
4. GC–MS combines gas chromatography with \_\_\_\_\_ spectrometry. (Linked to SLO 4.7)
5. LC–IR couples liquid chromatography with \_\_\_\_\_ spectroscopy. (Linked to SLO 4.8)

### Short-answer questions

6. Explain the difference between gas chromatography and liquid chromatography. (Linked to SLOs 4.4–4.5)
7. Describe how affinity influences separation in chromatography. (Linked to SLO 4.3)
8. Outline the importance of GC–MS in forensic analysis. (Linked to SLO 4.7)

### Long-answer questions

9. Analyse the applications of GC and LC in environmental, pharmaceutical, and forensic contexts. (Linked to SLO 4.6)
10. Evaluate the strengths and limitations of coupled techniques such as GC–MS and LC–IR in advanced analysis. (Linked to SLOs 4.7–4.9)
11. Discuss how chromatography provides versatility compared to other analytical techniques. (Linked to SLOs 4.1–4.9)

### Answers To Pilot Questions 4

**Part 4.1:** 1.a, 2.mobile, 3.a, 4.gas, 5.d **Part 4.2:** 1.a, 2.gas, 3.a, 4.liquid, 5.a **Part 4.3:** 1.a, 2.mass, 3.a, 4.infrared, 5.a

### References And Attributions 4

- Figures 4.1–4.3: Suggested AI prompts for diagrams showing principles of chromatography, GC–MS, and LC–IR.
- Plates 4.1–4.3: Suggested AI prompts for images of chromatographic separation, LC setup, and LC–IR instrumentation.
- Boxes 4.1–4.3: Suggested AI prompts for tables summarising classification, applications, and limitations of chromatographic methods.
- Open Educational Resources (OER) suggested search strings:
  - “OER diagram chromatography principle chemistry”
  - “OER diagram gas chromatography setup chemistry”
  - “OER image liquid chromatography setup chemistry”
  - “OER diagram GC–MS principle chemistry”
  - “OER image LC–IR principle chemistry”



- “OER table chromatography types chemistry”
- “OER table GC LC applications chemistry”
- “OER table coupled chromatography applications chemistry”
- Attribution style: APA (institutional standard assumed). Example: OpenStax. (2016). *Chemistry*. OpenStax CNX. Retrieved from <https://openstax.org/books/chemistry>

**Course code: CHM 410**

**Course title: Analytical Chemistry II**

**Course credit load: 2 Units**

**Series 1: Thermal Methods Of Analysis**

**Series 2: Electroanalytical And Potentiometric Techniques**

**Series 3: Conductometric, Amperometric And Colorimetric Methods**

**Series 4: Chromatographic Principles And Coupled Techniques**

**Series 5: Radiochemical Methods In Environmental Analysis**

**Proposed Outline Series 5**

### **5.1 Fundamentals Of Radiochemical Methods**

- 5.1.1 Definition and scope of radiochemical analysis
- 5.1.2 Principles of radioactivity and detection
- 5.1.3 Safety considerations in radiochemical analysis

### **5.2 Radiochemical Techniques In Environmental Monitoring**

- 5.2.1 Radiotracer methods for pollutant tracking
- 5.2.2 Radioisotope dilution analysis
- 5.2.3 Applications in water, soil, and air monitoring

### **5.3 Instrumentation And Applications Of Radiochemical Methods**

- 5.3.1 Radiation detectors and measurement systems
- 5.3.2 Case studies in environmental analysis
- 5.3.3 Limitations and challenges of radiochemical techniques



## Introduction

Radioactivity, though often associated with nuclear energy, plays an important role in environmental analysis. Radiochemical methods provide highly sensitive tools for detecting pollutants, tracing chemical pathways, and monitoring contamination in air, water, and soil. These techniques are invaluable in ensuring environmental safety and compliance with regulatory standards.

The aim of this Series is to introduce you to the principles of radiochemical methods, explain their application in environmental monitoring, and highlight the instrumentation and challenges involved in their use.

We shall commence this Series by exploring the fundamentals of radiochemical methods, including their scope, principles of radioactivity, and safety considerations. Next, we will examine radiochemical techniques in environmental monitoring, focusing on radiotracer methods and isotope dilution analysis. Finally, we will conclude with instrumentation and applications, considering case studies and limitations of radiochemical techniques in environmental contexts.

## Series Learning Outcomes

Upon completing this Series, you should be able to:

- **SLO 5.1** define radiochemical methods and explain their scope in environmental analysis,
- **SLO 5.2** describe the principles of radioactivity and detection,
- **SLO 5.3** evaluate safety considerations in radiochemical analysis,
- **SLO 5.4** explain radiotracer methods for tracking pollutants,
- **SLO 5.5** demonstrate how radioisotope dilution analysis is applied in environmental monitoring,
- **SLO 5.6** analyse applications of radiochemical methods in water, soil, and air monitoring,
- **SLO 5.7** describe radiation detectors and measurement systems used in radiochemical analysis,
- **SLO 5.8** evaluate case studies of radiochemical applications in environmental contexts,
- **SLO 5.9** assess the limitations and challenges of radiochemical techniques.

## 5.1 Fundamentals Of Radiochemical Methods



### 5.1.1 Definition and scope of radiochemical analysis

**Radiochemical analysis** refers to techniques that use radioactive isotopes or radiation detection to study chemical systems. These methods are highly sensitive and can trace minute quantities of substances in complex environments. Their scope includes environmental monitoring, medical diagnostics, and nuclear industry applications.

Fill in the blank: Radiochemical analysis uses radioactive isotopes or \_\_\_\_\_ detection to study chemical systems.

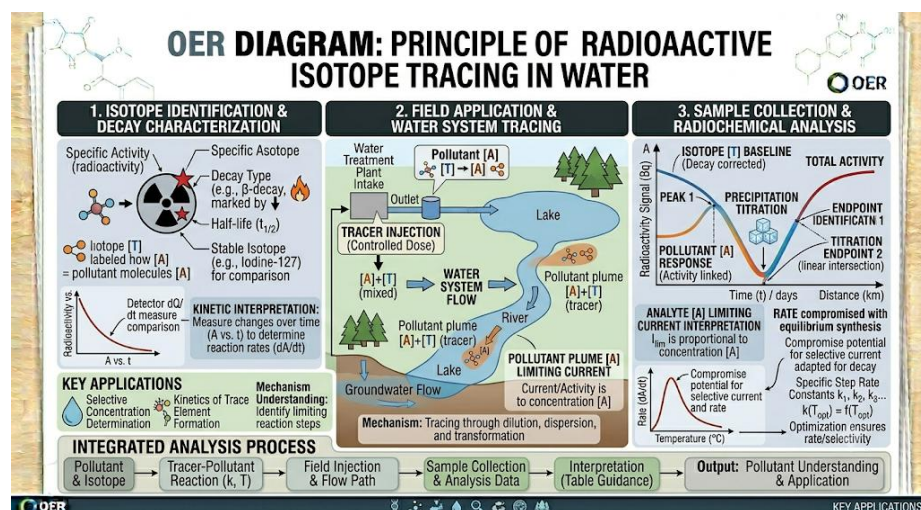


Figure 5.1: Principle of radiochemical analysis.

### 5.1.2 Principles of radioactivity and detection

**Radioactivity** is the spontaneous emission of particles or energy from unstable atomic nuclei. The three main types of radiation are alpha particles, beta particles, and gamma rays.

**Radiation detection** involves instruments such as Geiger–Müller counters, scintillation detectors, and semiconductor detectors. These devices measure radiation intensity and help quantify the presence of radioactive substances.

Fill in the blank: The three main types of radiation are alpha particles, beta particles, and \_\_\_\_\_ rays.

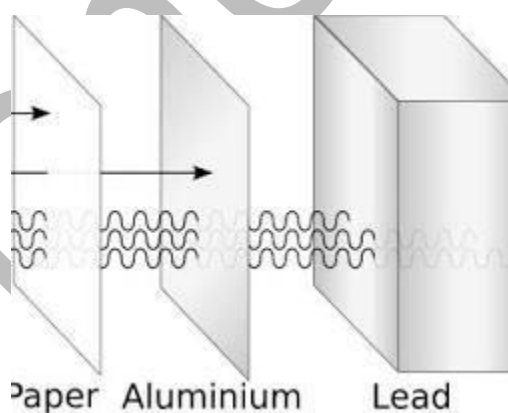


Plate 5.1: Types of radiation and penetration.

### 5.1.3 Safety considerations in radiochemical analysis

Working with radioactive materials requires strict safety protocols to protect both the analyst and the environment. Key considerations include:

- Using shielding materials such as lead to reduce exposure.
- Wearing protective clothing and gloves.
- Monitoring radiation levels with dosimeters.
- Following proper waste disposal procedures for radioactive materials.

Fill in the blank: Radiation exposure can be reduced by using shielding materials such as \_\_\_\_\_.

| Safety Measure      | Description                                                                   | Primary Objective                                                            |
|---------------------|-------------------------------------------------------------------------------|------------------------------------------------------------------------------|
| Shielding           | Use of appropriate barriers (e.g., lead, plexiglass) based on radiation type. | Attenuates ionizing radiation to protect personnel from external exposure.   |
| Protective Clothing | Use of lab coats, gloves, goggles, and dosimeters.                            | Prevents contamination of skin and clothing; tracks cumulative exposure.     |
| Monitoring          | Frequent use of Geiger-Müller counters or scintillation detectors.            | Detects leaks or spillages immediately to ensure a safe working environment. |
| Waste Disposal      | Segregation of radioactive waste by half-life and activity; strict labeling.  | Prevents long-term environmental hazards and ensures regulatory compliance.  |

Box 5.1: Safety considerations in radiochemical analysis.

#### Pilot questions 5.1

1. Which of the following best defines radiochemical analysis? a. Techniques using radioactive isotopes or radiation detection to study chemical systems b. Techniques using heat flow to study chemical systems c. Techniques using colour intensity to study chemical systems d. Techniques using electrical conductivity to study chemical systems
2. Fill in the blank: Radiochemical analysis uses radioactive isotopes or \_\_\_\_\_ detection to study chemical systems.
3. Which of the following is a type of radiation? a. Alpha particles b. Beta particles c. Gamma rays d. All of the above





4. Fill in the blank: The three main types of radiation are alpha particles, beta particles, and \_\_\_\_\_ rays.
5. Which of the following is a safety consideration in radiochemical analysis? a. Using shielding materials b. Wearing protective clothing c. Monitoring radiation levels d. All of the above

## 5.2 Radiochemical Techniques In Environmental Monitoring

### 5.2.1 Radiotracer methods for pollutant tracking

**Radiotracer methods** involve introducing a small quantity of a radioactive isotope into an environmental system to trace the movement or transformation of pollutants. Because radioactive isotopes can be detected at extremely low concentrations, radiotracers provide highly sensitive insights into pollutant pathways in water, soil, and air.

These methods are especially useful for studying dispersion patterns, adsorption processes, and chemical transformations in complex environments.

Fill in the blank: Radiotracer methods involve introducing a small quantity of a \_\_\_\_\_ isotope into an environmental system.

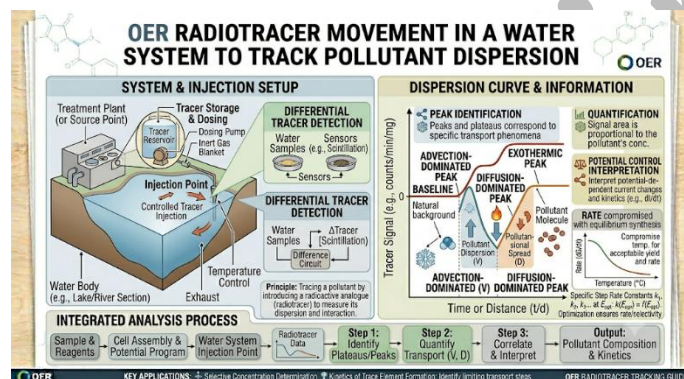


Figure 5.2: Radiotracer tracking of pollutants.

### 5.2.2 Radioisotope dilution analysis

**Radioisotope dilution analysis** is a quantitative technique where a known amount of radioactive isotope is added to a sample. The dilution of radioactivity after mixing allows precise determination of the concentration of the target substance.

This method is widely applied in environmental chemistry to measure trace elements and pollutants that are otherwise difficult to quantify.

Fill in the blank: Radioisotope dilution analysis determines concentration by measuring the dilution of \_\_\_\_\_ after mixing.



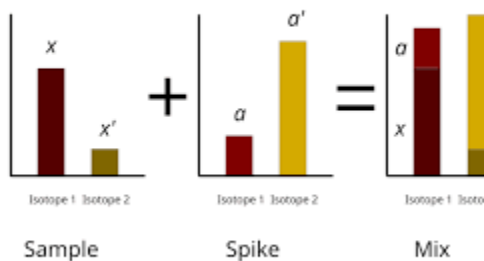


Plate 5.2: Principle of radioisotope dilution analysis.

### 5.2.3 Applications in water, soil, and air monitoring

Radiochemical techniques are applied across environmental contexts:

- **Water monitoring:** Detecting pollutants such as heavy metals, nitrates, and organic contaminants.
- **Soil monitoring:** Studying nutrient cycles, pollutant adsorption, and pesticide residues.
- **Air monitoring:** Tracing dispersion of industrial emissions and radioactive fallout.

These applications provide regulators and scientists with precise data for environmental protection and remediation.

Fill in the blank: Radiochemical techniques are widely used in air monitoring to trace dispersion of industrial \_\_\_\_\_.

| Environmental Medium | Target Analytes                           | Specific Applications                                             | Significance                                    |
|----------------------|-------------------------------------------|-------------------------------------------------------------------|-------------------------------------------------|
| Water                | Pollutants (e.g., nitrates, heavy metals) | Monitoring pollutant plumes; tracing flow dynamics.               | Pollution control; regulatory compliance.       |
| Soil                 | Trace elements, mineral contamination     | Studying soil nutrient transport and pollutant immobilization.    | Resource management; agricultural safety.       |
| Air                  | Particulate matter, gaseous effluents     | Tracking dispersion of radioactive or chemically active aerosols. | Environmental safety; health impact assessment. |

Box 5.2: Applications of radiochemical techniques in environmental monitoring.

Pilot questions 5.2



1. Which of the following best defines radiotracer methods? a. Introducing radioactive isotopes to trace pollutant movement b. Measuring colour intensity of pollutants c. Using heat flow to monitor pollutants d. Measuring electrical conductivity of pollutants
2. Fill in the blank: Radiotracer methods involve introducing a small quantity of a \_\_\_\_\_ isotope into an environmental system.
3. Which of the following best defines radioisotope dilution analysis? a. Adding radioactive isotope to a sample and measuring dilution of radioactivity b. Measuring heat flow in a sample c. Using colour indicators to measure pollutant concentration d. Measuring electrical conductivity of a solution
4. Fill in the blank: Radioisotope dilution analysis determines concentration by measuring the dilution of \_\_\_\_\_ after mixing.
5. Which of the following is an application of radiochemical techniques in environmental monitoring? a. Detecting pollutants in water b. Studying nutrient cycles in soil c. Tracing industrial emissions in air d. All of the above

### 5.3 Instrumentation And Applications Of Radiochemical Methods

#### 5.3.1 Radiation detectors and measurement systems

**Radiation detectors** are instruments used to measure the presence and intensity of radiation. Common types include:

- **Geiger–Müller counters:** Detect ionising radiation by measuring electrical pulses caused by radiation interacting with gas inside the tube.
- **Scintillation detectors:** Use a scintillating material that emits light when struck by radiation, which is then converted into an electrical signal.
- **Semiconductor detectors:** Employ solid-state materials such as silicon or germanium to detect radiation with high precision.

These systems are essential for quantifying radioactivity in environmental samples.

Fill in the blank: Geiger–Müller counters detect ionising radiation by measuring electrical \_\_\_\_\_ caused by radiation.



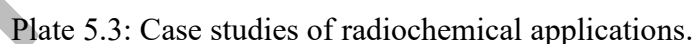


Radiochemical methods have been applied in several notable environmental studies:

- **Water pollution:** Radiotracers have been used to track the dispersion of heavy metals in rivers.
- **Soil contamination:** Radioisotope dilution analysis has helped quantify pesticide residues in agricultural soils.
- **Air quality:** Radiochemical monitoring has traced radioactive fallout from industrial emissions and nuclear accidents.

These case studies demonstrate the versatility of radiochemical techniques in addressing real-world environmental challenges.

Fill in the blank: Radiochemical monitoring has been used to trace radioactive \_\_\_\_\_ from industrial emissions.



### 5.3.3 Limitations and challenges of radiochemical techniques

Despite their sensitivity, radiochemical methods face certain challenges:



- Handling and disposal of radioactive materials require strict safety protocols.
- Instruments can be expensive and require specialised training.
- Environmental samples may contain interfering substances that complicate analysis.
- Public perception of radioactivity can limit acceptance of these techniques.

Fill in the blank: One major challenge of radiochemical methods is the safe handling and disposal of \_\_\_\_\_ materials.

| Limitation               | Description                                                                                  | Impact on Analysis/Application                                                                     |
|--------------------------|----------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------|
| <b>Safety Concerns</b>   | Handling radioactive materials poses risks of exposure and contamination.                    | Requires rigorous safety protocols, specialized training, and stringent regulatory oversight.      |
| <b>Cost</b>              | High expenditure associated with specialized instrumentation, shielding, and waste disposal. | Can limit accessibility for smaller institutions or routine, large-scale environmental monitoring. |
| <b>Interference</b>      | Presence of naturally occurring background radiation or other radioactive species.           | May require complex signal processing or chemical separation to isolate the signal of interest.    |
| <b>Public Perception</b> | General public concern regarding radioactivity and nuclear-related technologies.             | Often necessitates intensive community engagement and clear communication of risk versus benefit.  |

Box 5.3: Limitations of radiochemical techniques.

#### Pilot questions 5.3

1. Which of the following best defines a Geiger–Müller counter? a. An instrument that detects ionising radiation by measuring electrical pulses b. An instrument that measures colour intensity of solutions c. An instrument that measures heat flow in samples d. An instrument that measures electrical conductivity
2. Fill in the blank: Geiger–Müller counters detect ionising radiation by measuring electrical \_\_\_\_\_ caused by radiation.
3. Which of the following is a case study application of radiochemical methods? a. Tracking heavy metals in rivers b. Quantifying pesticide residues in soil c. Tracing radioactive fallout in air d. All of the above
4. Fill in the blank: Radiochemical monitoring has been used to trace radioactive \_\_\_\_\_ from industrial emissions.
5. Which of the following is a limitation of radiochemical techniques? a. Safety concerns in handling radioactive materials b. High cost of instruments c. Interference from environmental samples d. All of the above





## Series Summary

In this Series, we examined the **principles and applications of radiochemical methods in environmental analysis**. We began with the fundamentals, defining radiochemical analysis, exploring the principles of radioactivity, and emphasising safety considerations. We then studied radiochemical techniques in environmental monitoring, focusing on radiotracer methods and radioisotope dilution analysis, and considered their applications in water, soil, and air monitoring. Finally, we explored instrumentation and applications, including radiation detectors, case studies, and the limitations and challenges of radiochemical techniques. Altogether, this Series has provided learners with a clear understanding of how radiochemical methods contribute to environmental monitoring and protection.

## Glossary of Terms

- **Radiochemical analysis:** Techniques using radioactive isotopes or radiation detection to study chemical systems.
- **Radioactivity:** Spontaneous emission of particles or energy from unstable atomic nuclei.
- **Radiotracer method:** Technique using radioactive isotopes to trace pollutant movement in environmental systems.
- **Radioisotope dilution analysis:** Quantitative method where radioactivity dilution is used to determine concentration.
- **Geiger–Müller counter:** Instrument that detects ionising radiation by measuring electrical pulses.
- **Scintillation detector:** Radiation detector that converts light flashes into electrical signals.
- **Semiconductor detector:** Solid-state radiation detector using materials such as silicon or germanium.
- **Dosimeter:** Device used to monitor radiation exposure.

## Concept Check Questions 5

### Objective questions

1. Radiochemical analysis uses radioactive isotopes or \_\_\_\_\_ detection to study chemical systems. (Linked to SLO 5.1)
2. The three main types of radiation are alpha particles, beta particles, and \_\_\_\_\_ rays. (Linked to SLO 5.2)
3. Radiotracer methods involve introducing a small quantity of a \_\_\_\_\_ isotope into an environmental system. (Linked to SLO 5.4)



4. Radioisotope dilution analysis determines concentration by measuring the dilution of \_\_\_\_\_ after mixing. (Linked to SLO 5.5)
5. Geiger–Müller counters detect ionising radiation by measuring electrical \_\_\_\_\_ caused by radiation. (Linked to SLO 5.7)

### Short-answer questions

6. Explain the difference between radiotracer methods and radioisotope dilution analysis. (Linked to SLOs 5.4–5.5)
7. Describe how radiation detectors such as scintillation detectors work. (Linked to SLO 5.7)
8. Outline the importance of radiochemical techniques in air monitoring. (Linked to SLO 5.6)

### Long-answer questions

9. Analyse the applications of radiochemical methods in water, soil, and air monitoring. (Linked to SLO 5.6)
10. Evaluate case studies of radiochemical applications in environmental contexts. (Linked to SLO 5.8)
11. Discuss the limitations and challenges of radiochemical techniques, including safety and public perception. (Linked to SLO 5.9)

### Answers To Pilot Questions 5

**Part 5.1:** 1.a, 2.radiation, 3.d, 4.gamma, 5.d **Part 5.2:** 1.a, 2.radioactive, 3.a, 4.radioactivity, 5.d  
**Part 5.3:** 1.a, 2.pulses, 3.d, 4.fallout, 5.d

### References And Attributions 5

- Figures 5.1–5.3: Suggested AI prompts for diagrams showing principles of radiochemical analysis, radiotracer tracking, and Geiger–Müller counter operation.
- Plates 5.1–5.3: Suggested AI prompts for images of radiation types, radioisotope dilution analysis, and case studies in environmental monitoring.
- Boxes 5.1–5.3: Suggested AI prompts for tables summarising safety considerations, applications, and limitations of radiochemical methods.
- Open Educational Resources (OER) suggested search strings:
  - “OER diagram radiochemical analysis principle chemistry”
  - “OER image types of radiation chemistry”
  - “OER diagram radiotracer pollutant tracking chemistry”
  - “OER image radioisotope dilution analysis chemistry”



- “OER image radiochemical environmental case studies chemistry”
- “OER table radiochemical safety chemistry”
- “OER table radiochemical environmental monitoring chemistry”
- “OER table radiochemical limitations chemistry”
- Attribution style: APA (institutional standard assumed). Example: OpenStax. (2016). *Chemistry*. OpenStax CNX. Retrieved from <https://openstax.org/books/chemistry>

