Analytical Chemistry (Chem2021)

➢ The nature and importance of chemical analysis

Key terms and concepts

- ❖Definition of analytical chemistry
- ❖The applications of analytical chemistry
- ❖Steps in chemical analysis
- ❖Classification of analytical techniques

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Definition:

➢Analytical chemistry is a scientific discipline used to study the chemical composition, structure and behavior of matter.

➢Analytical chemistry involves the application of various techniques and methodologies to obtain qualitative, quantitative and structural information on the nature of matter.

- ❖ **Qualitative analysis** : Identification of elements, species and/or compounds present in a sample.
- ❖ **Quantitative analysis**: Determination of the absolute or relative amounts of elements, species or compounds present in a sample.
- ❖ **Structural analysis**: Determination of the spatial arrangement of atoms in an element or molecule or the identification of characteristic groups of atoms (functional groups).

➢Unfortunately, this description ignores the unique perspective that analytical chemists bring to the study of chemistry.

➢ The craft of analytical chemistry is not in performing a routine analysis on a routine sample (which is more appropriately called chemical analysis), **but in improving established methods, extending existing methods to new types of samples, and developing new methods for measuring chemical phenomena.**

- **Analytical chemistry is thus** concerned with the development of analytical methods/techniques for **chemical analysis** which are utilized in various fields as the key to the solution of a variety of scientific problems and of industrial problems.
- An analytical **method consists of a detailed**, stepwise list of instructions to be followed in the qualitative, quantitative or structural analysis of a sample for one or more analytes and using a specified technique.

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- It will include a summary and lists of chemicals and reagents to be used, laboratory apparatus and glassware, and appropriate instrumentation.
- An analytical **technique** is a chemical or physico-chemical processes, which are characteristics of a particular substance or its constituent, that provide the basis for analytical measurements.
- There are numerous chemical or physico-chemical processes that can be used to provide the basis for analytical measurements or analytical information. Some important of these are listed in Table 1.

Table 1. Analytical techniques and principal applications

Technique	Property measured	Principal areas of application
Gravimetry	Weight of pure analyte or compound of known stoichiometry	Quantitative for major or minor components
Titrimetry	Volume of standard reagent solution reacting with the analyte	Quantitative for major or minor components
Atomic and molecular spectrometry	Wavelength and intensity of electromagnetic radiation emitted or absorbed by the analyte	Qualitative, quantitative or structural for major down to trace level components
Mass spectrometry	Mass of analyte or fragments of it	Qualitative or structural for major down to trace level components isotope ratios
Chromatography and electrophoresis	Various physico-chemical properties of separated analytes	Qualitative and quantitative separations of mixtures at major to trace levels
Thermal analysis	Chemical/physical changes in the analyte when heated or cooled	Characterization of single or mixed major/minor components
Electrochemical analysis	Electrical properties of the analyte in solution	Qualitative and quantitative for major to trace level components
Radiochemical analysis	Characteristic ionizing nuclear radiation emitted by the analyte	Qualitative and quantitative at major to trace levels

Chemical analysis

- A chemical analysis provides chemical or physical information about a sample. The component of interest in the sample is called the **analyte**, and the remainder of the sample is **the matrix**.
- In a chemical analysis we determine the identity, concentration, or properties of an analyte.
- To make this determination, we measure one or more of the analyte's chemical or physical properties like density, refractive index, color, electrical conductivity, thermal conductivity and electrical charges, etc.

- The most important aspect of an **analysis is to ensure that it will provide useful and reliable** data on the qualitative and/or quantitative composition.

- The analytical chemist must often communicate with other scientists and nonscientists to establish the amount and quality of the information required, the time-scale for the work to be completed and any budgetary constraints.

- The most appropriate analytical technique and method can then be selected from those available or new ones devised and validated by the analysis of substances of known composition and/or structure.
- It is essential for the analytical chemist to have an appreciation of the objectives of the analysis and an understanding of the capabilities of the various analytical techniques at his/her disposal without which the most appropriate and cost- effective method cannot be selected or developed.

- Selecting or **developing and validating appropriate methods of analysis to provide reliable data in a variety of contexts are the principal problems** faced by analytical chemists.

Purpose of Analytical chemistry

- The maintenance of, and improvement in, the quality of life throughout the world, and the management of resources rely heavily on the information provided by chemical analysis.

- Manufacturing industries use analytical data to monitor the quality of raw materials, intermediates and finished products.

Example: Analysis of drugs, leather, food product

- Monitoring of toxic substances in the environment is of ever increasing importance.

Example: Water quality, soil quality analysis

- Studies of biological and other complex systems are supported by the collection of large amounts of analytical data.

Example: Blood, urine, biopsy

Important areas of application of Analytical Chemistry

Quality control (QC):

- In many manufacturing industries, the chemical composition of raw materials, intermediates and finished products needs to be monitored to ensure satisfactory quality and consistency.
- Products from automobiles to clothing, pharmaceuticals and foodstuffs, electrical goods, sports equipment and horticultural products rely, in part, on chemical analysis.

Monitoring and control of pollutants:

- Presence of toxic heavy metals (e.g., lead, cadmium and mercury)
- organic chemicals (e.g., polychlorinated biphenyls and detergents)
- vehicle exhaust gases (oxides of carbon, nitrogen and sulfur, and hydrocarbons) in the environment are health hazards that need to be monitored by sensitive and accurate methods of analysis, and remedial action taken.

Clinical and biological studies:

- nutrients, trace metals (e.g., sodium, potassium, calcium and zinc)
- naturally produced chemicals, such as cholesterol, sugars and urea
- administered drugs in the body fluids of patients undergoing hospital treatment require monitoring and speed of analysis is often a crucial factor and automated procedures are designed for such analyses.

Geological assays:

- The commercial value of ores and minerals is determined by the levels of particular metals, which must be accurately estimated.

Fundamental and applied research:

- Where new drugs or materials with potential commercial value are synthesized, a complete chemical characterization may be required involving considerable analytical work.
- Combinatorial chemistry is an approach used in pharmaceutical research that generates very large numbers of new compounds requiring confirmation of identity and structure.

- In summary, quality control in manufacturing industries, the monitoring of clinical and environmental samples, the assaying of geological specimens, and the support of fundamental and applied research are the principal applications.

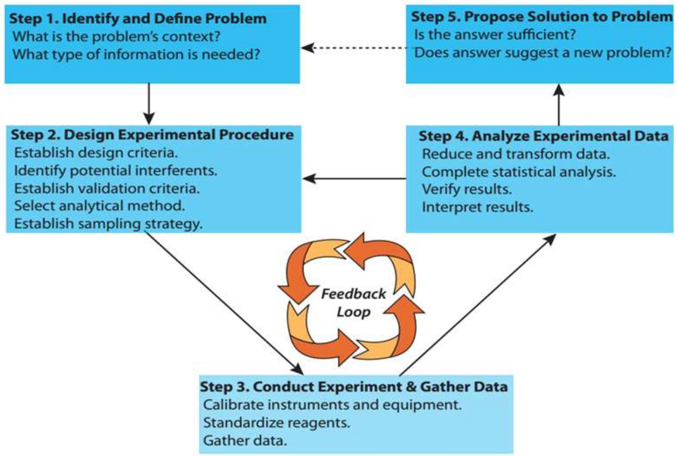
Steps in chemical analysis

- Many analytical chemists describe this chemical analysis steps as an analytical approach to solve problems. It is also known as the analytical process or perspective
- Although there are probably as many descriptions of the analytical approach as there are analytical chemists, it is convenient for our purposes to treat it as the five step process (Figure 1) and each step is crucial to get accurate and meaningful results.

Steps in chemical analysis

1. Identify and define the problem
2. Design the experimental procedure
3. Conduct an experiment, and gather data
4. Analyze the experimental data
5. Propose a solution to the problem

Figure 1: Flow diagram for the analytical approach



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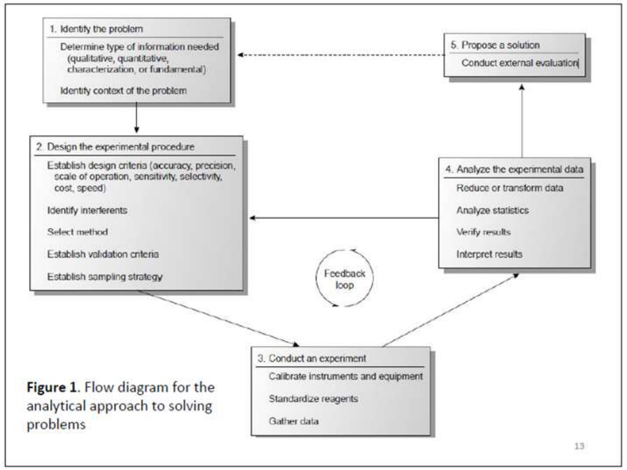

Figure 1. Flow diagram for the analytical approach to solving problems

- The **first step in** the process is **to begin with a problem**. It may involve a collaboration b/n the analytical chemist and the individual or agency working on the problem. Together they determine what information is needed to design an appropriate experimental procedure.
- **To design the experimental** procedure the analytical chemist considers criteria such as
  - the desired accuracy, precision, sensitivity, and detection limits
  - the urgency with which results are needed
  - the cost of a single analysis; the number of samples to be analyzed; and the amount of sample available for analysis
  - availability of laboratory instruments and facilities and selection of best technique such as, spectroscopic, chromatographic, electro analytical, thermal methods, etc.,
- The analytical chemist should also given consideration (sampling, sample pre-treatment or conditioning) for sample collection, storing, and preparation. For example, conversion of the sample into a form suitable for analysis, like dissolving,

or converting the analyte(s) into a specific chemical form, separate the analyte(s) from the matrix of sample to avoid interference, i.e., need to consider whether chemical or physical interferences will affect the analysis

- Finding an appropriate balance between these parameters is frequently complicated by their interdependence.

- The most visible part of the analytical approach occurs in the laboratory. As part of the validation process, appropriate chemical and physical standards are used **to calibrate any equipment** and to standardize any reagents.
- Measurement of the instrumental response for sample under the same conditions as for the standards; Replicating the analysis to improve the reliability of the data;
- The data collected during the experiment are then **analyzed.** Frequently the data is reduced or transformed to a more readily analyzable form.

- A statistical treatment of the data is used to evaluate accuracy and precision, and to validate the procedure.
- **Results are compared** to the original design criteria and the experimental design is reconsidered, additional trials are run, or a solution to the problem is proposed.

- In summary, Figure 1: Flow diagram showing one view of the analytical approach to solve problems **along with some** important considerations at each step.
- Three general features of this approach deserve attention.
- First, steps 1 and 5 provide opportunities for analytical chemists to collaborate with individuals outside the realm of analytical chemistry. In fact, many problems on which analytical chemists work originate in other fields.

- Second, the analytical approach is not linear, but incorporates a “feedback loop” consisting of steps 2, 3, and 4, **in which the outcome of one step may cause a reevaluation of the other two steps.**
- **Finally,** the solution to one problem often suggests a new problem.

Practice Exercise 1.1

As an exercise, let's adapt our model of the analytical approach to the development of a simple, inexpensive, portable device for completing bioassays in the field. Before continuing, locate and read the article

“Simple Telemedicine for Developing Regions: Camera Phones and Paper-Based Microfluidic Devices for Real-Time, Off-Site Diagnosis”

by Andres W. Martinez, Scott T. Phillips, Emanuel Carriho, Samuel W. Thomas III, Hayat Sindi, and George M. Whitesides. You will find it on pages 3699-3707 in Volume 80 of the journal *Analytical Chemistry*, which was published in 2008. As you read the article, pay particular attention to how it emulates the analytical approach. It might be helpful to consider the following questions:

What is the analytical problem and why is it important?
What criteria did the authors consider in designing their experiments?
What is the basic experimental procedure?
What interferences were considered and how did they overcome them?
How did the authors calibrate the assay?
How did the authors validate their experimental method?
Is there evidence of repeating steps 2, 3, and 4?
Was there a successful conclusion to the problem?

Don't let the technical details in the paper overwhelm you. If you skim over these you will find that the paper is well-written and accessible.

Analytical Methods

- ✓ A method is the application of a technique for a specific analyte in a specific matrix. It consists of a detailed, stepwise list of instructions to be followed in the qualitative, quantitative or structural analysis of a sample.
- ✓ It will includes a summary and lists of chemicals, reagents, apparatus, glassware instrumentation with source and specifications
- ✓ Specifications are of crucial importance in obtaining meaningful results
- ✓ The preparation or pre-treatment of the sample
- ✓ Standardization of reagents and calibration of instruments under specified conditions

Analytical Method selection

- Selection of the most appropriate analytical method should consider the following factors:
  - ❖ the purpose of the analysis, the required time scale and any cost constraints;
  - ❖ the level of analyte(s) expected and the detection limit required;
  - ❖ the nature of the sample, the amount available and the necessary sample preparation procedure; the accuracy required for a quantitative analysis;

- the availability of reference materials, standards, chemicals & solvents, instrumentation; possible interference with the detection or quantitative measurement of the analyte(s) and the possible need for sample clean-up to avoid matrix interference;
- the degree of selectivity available − methods may be selective for a small number of analytes or specific for only one;
- quality control and safety factors.

Classification of Analytical Methods

Classical methods

- Methods that rely on chemical reactions and involves separating the components in a sample by precipitation, extraction or distillation
- In **qualitative classical methods, the separated components treated with** reagents that can yield products recognized by their colors, boiling points, melting points, solubility's in a series of solvents, odors, optical activities or their refractive index

- In quantitative classical methods, the **amounts of components are determined** by gravimetry or titration methods

Instrumental methods

- Measure the physical or electrical properties of analytes such as- conductivity, electrode potential, light absorption or emission, mass-to-charge ratio, fluorescence, etc.,

- Most instrumental analysis methods can be classified in one of three general categories: **spectroscopy**, which uses instruments generally known as a spectrometers; **chromatography**, which uses instruments generally known as chromatographs; and **electroanalytical chemistry**, which uses electrodes dipped into the analyte solution.
- Spectroscopic methods involve the use of light and measure the amount of either light absorbed (absorbance) or light emitted by solutions of the analyte under certain conditions.

- Chromatographic methods involve more complex samples in which the analyte is separated from interfering substances using specific instrument components and electronically detected, with the electrical signal generated by any one of a number of detection devices.
- Electroanalytical methods involve the measurement of a voltage or current resulting from electrodes immersed into the solution. Table 1 shows few instrumental methods along with the property measured.

Table 1: Chemical and Physical properties Employed in Instrumental Methods

Characteristic property	Instrumental methods
Interaction with radiation	Spectroscopy methods (UV visible, IR, x-ray and NMR spectroscopy, -- etc.)
Electrical	Potentiometry, conductometry, - -etc.
Mass-to-charge ratio	Gravimetry, mass spectrometry, - -etc
Rate of reaction	Kinetic methods
Thermal characteristics	Thermal gravimetry TG, Differential thermal analysis DTA, differential scanning calorimetry DSC, - -etc.
Radioactivity	Activation and isotope analysis

THANK YOU!