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A brief study on various instrumental methods of analysis

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Abstract
In early year of chemistry, most analyses were carried out by separating the component of the analytic in a sample by precipitation, extraction, or distillation. Analytical chemistry studies the various instruments and methods that are used for to separation, identify, and quantify of the matter Analytical chemistry has been important since the early days of chemistry; it has providing the various methods for determining the elements and chemicals groups those are present in the sample. The main aim of the pharmaceutical Instrumental methods of analysis have become the predominant approach for performing chemical measurements. It is critical for scientists who rely on these powerful tools to understand the fundamentals and applications of analytical instruments. However chemical instruments is not entirely sufficient in intelligently solving problems. The accurate and appropriate instrumental measurement of an incorrect or irrelevant sample obtained from a poorly designed method will not solve the intended problem. A variety of instrumental techniques are available for the application to chemical problems. They include mass spectrometric, optical spectroscopic, nuclear, surface, electrochemical and separation methods. They are commonly used individually and in manners where two or more techniques are combined beneficially, creating hyphenated techniques. In general, all of these instrumental techniques function by converting information in the non-electrical domain into the electrical domain, where the information can be transformed into meaningful information, then converted into a form that is meaningful to the analyst.

Keywords: analysis, qualitative analysis, methods

1. Introduction
Analytical chemistry has been important since the early days of chemistry; it has providing the various methods for determining the elements and chemicals groups those are present in the sample. During this period significant contributions to analytical chemistry include the development of systematic elemental analysis and systematized organic analysis based on the specific reactions and functional groups.

Analytical chemistry
Studies the various instruments and methods that are used for to separation, identify, and quantify of the matter. Separation, identification or quantification may constitute the entire analysis or be combined with another method. Separation isolates analytes. Qualitative analysis identifies analytes, while quantitative analysis determines the numerical amount or concentration.

Qualitative analysis
It may be define as "which analyte present in the given sample". This has the goal to identify the various component present in the sample on the basis of physical and chemical properties such as functional group, element composition and melting point etc.

Quantitative analysis
It may be define as "How much amount of analyte present In the sample". This has the goal to determine the quantity of each component present in the sample.

Semi-quantitative analysis
It is the type of analysis which only describe whether the quantity of impurity present in the sample is blow or above the specified limit such as limit test.

Classical methods
In early year of chemistry, most analyses were carried out by separating the component of the analyte in a sample by precipitation, extraction, or distillation. For quantitative analysis, the separates components were then treated with reagent then yield products that could be recognized by their colour, boiling point, solubility.

a. Gravimetric methods
b. Titrimetric methods

Gravimetric methods
It include determination of mass of analyte or some compound produced from analyte.

Titrimetric methods
It include the determination of the volume or weight of standard reagent required to react completely with analyte.
Now a days instrumental method of analysis is widely used. These method are extremely sensitive, providing precise and detailed information from small of material.

a. Electro analytical method: it is the modification of methods in which the reaction process and endpoint are determine by an electric measurement.

b. Spectroanalytical methods: It is widely used for quantitative as well as qualitative analysis. They give result in the form of an interpredectable spectrum.

c. Chromatography methods: The method is used for the determination of complex mixture or mixture of compound by separation of individual. For ex. Gas, HPLC/TLC

Analytical determination is based on the measurement of some physical, chemical or structural properties which are related directly or indirectly to the amount of desired constituent present in the sample. Analytical chemistry can be di fine as the science and art of determining the composition of material, in term of the element or compound contained either qualitatively or quantitatively.

The following steps are involved in the analysis of sample:

1. Obtained the sample in pure form
2. Preparation of sample
3. Identification of suitable analytical procedure

- Chemical or physical method
- Condition determined either by analytical problem by the substance being investigated.
- Requirement, either fast, accurate & economical adaptable to automation
- Evolution of satisfactory result.

Classification of analytical methods

A. General Classification
B. Scientific Classification
C. Traditional Classification
D. Morden Classification

Electrogravimetry

It is the method used for the separation of quantify ion of a substance, usually a metal. In this process, the analyte solution is electrolyzed. Electrochemical reduction causes the analyte to be deposited on the cathode or anode.

- The mass of electrode analyte can be directly measured the amount of analyte.
- Not always practical numerous material can be reduced or oxidized and still not plated out on an electrode is called the generator electrode.

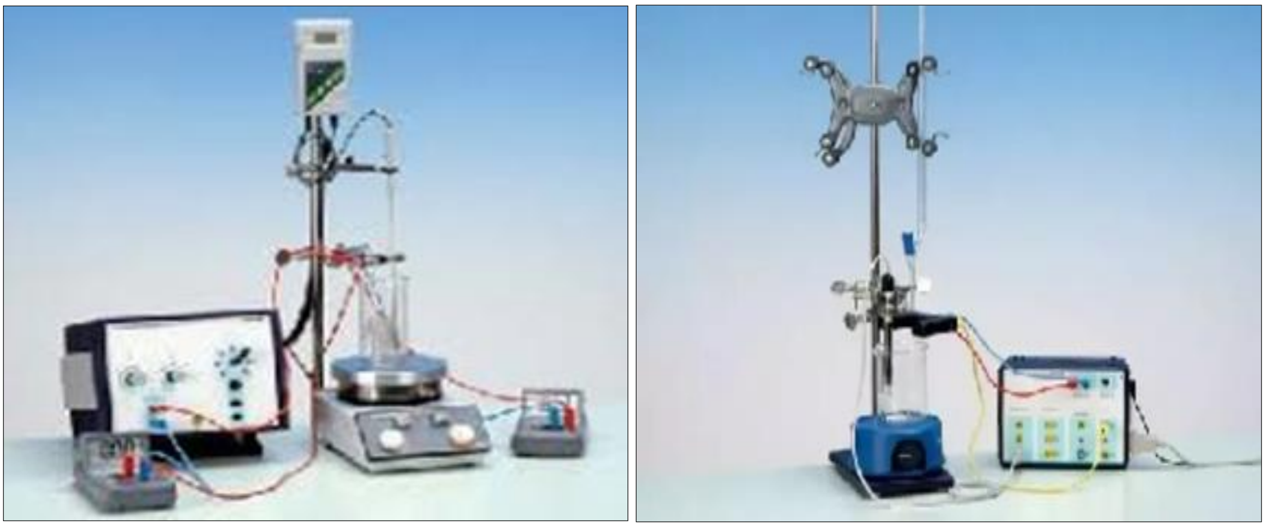

Fig 2

Instrumentation

- Controlled-current coulometry normally is carried out using a two electrode Galvanosalt consisting of a working electrode and a counter electrode.
- The working electrode-often a simple Pt. electrode

In the Electrogrvimetry there are three Electrode configurations

1. Reference electrode – maintains fixed potential depite change in solution comp.
2. Working electrode – electrode of interest which is the cathode in this system.
3. Counter electrode – third electrode taking most current flow (act as current sink).

Advantages of three electrode system

1. Change in concentration at counter electrode are not important, no effect on working electrode potential.
2. No current flow through reference -> no IR drop.

Application of Electrogravimetry

1. Quantitative analysis- the method is give to the very accurate and precise, weighing is the only measurement operation can control degree of completion for the deposition reaction by paper choice of potential.
2. Separation – separate one species from another in solution by selectively plating it out and removing it from solution this method is used to removing interference particularly in electrochemical methods.
3. Electro synthesis – used by organic chemist to perform oxidation or reduction reaction at batch scale.
4. Purification – to remove trace metals from reagent by plating them out of solution onto a large Hg pool electrode. This cell is also be used for electrosynthisis.

Flam emission Spectrocopy

A sample of a material (analyte) is brought into the flame as a gas, sprayed solution, or directly inserted into the flame by use of a small loop of wire, usually platinum. The heat from the flame and evaporates the solvent and breaks chemical bonds to create free atoms. The thermal energy also excites the atoms into excited electronic states that subsequently emit light when they return to the ground electronic state. Each element emits light at a characteristic wavelength, which is dispersed by a grating or prism and detected in the spectrometer.

Turbidimetry/nephelometry

- It is the process of measuring the loss of intensity of transmitted light due to the scattering effect of particles suspended.

- Light is passed through a filter creating a light of known wavelength:
- When it is passed through a cuvette containing a solution. A photoelectric cell collects the light which passes through the cuvette. A measurement of light is given to the amount of absorbed light:

A. Colorimeter

The colorimeter instrument is very simple, it is consisting of a light source (lamp), filter, curette and photosensitive detector to collect the transmitted light. a single detector may be used to measure incident and transmitted light, alternately.

B. Spectrophotometer

It is a more sophisticated instrument. A photometer is a device that is measuring

'light', and 'spectro' implies the whole range of continuous wavelengths that the light source is capable of producing. The detector in the photometer is generally a photo cell in which a sensitive surface receives photons; and a current is generated that is proportional to the intensity of the light beam, reaching the surface.

In these instruments for measuring the ultraviolet/visible light, two lamps are usually required:

1. One, a tungsten filament lamp which produces wavelengths in the visible region.
2. The second is a hydrogen or deuterium lamp it is suitable for the ultraviolet.

There are two kinds of optical arrangements: a single-beam or a double beam type.

Beer's Law: This law states, that the intensity of transmitted light decrease exponentially with the increase in concentration of colored substance in the solution. The amount of light absorbed by a colored solution is directly proportional to the conc. Of substance in the solution.

Lambert's law: This law states that, the intensity of transmitted light decreased exponentially with increase in length pathway. The amount of light absorbed by a vapored solution is directly proportional to the length of light path.

Chromatography

- It is a laboratory technique for the separation of a mixture.
- The mixture is dissolved in a fluid called the *mobile phase,* which carries it through a structure holding another material called the *stationary phase.*
- The various constituents of the mixture travel at different speeds, causing them to separate.
- The separation is based on differential partitioning between the mobile and stationary phases. Subtle differences in a compound's partition coefficient result in differential retention on the stationary phase and thus affect the separation.

Chromatography may be preparative or analytical. The purpose of preparative chromatography is to separate the components of a mixture for later use, and is thus a form of purification. Analytical Chromatography is done normally with smaller amounts of material.

Gas Chromatograph (GC)

A gas chromatograph (GC) is an analytical instrument that measures the content of various components in a sample. The analysis performed by a gas chromatograph is called gas chromatography.

Thin-layer chromatography

It is a technique used to separate non-volatile mixtures. Thin-layer chromatography is performed on a sheet of glass, plastic, or aluminium foil, which is coated with a thin layer of adsorbent material, usually silica gel, aluminium oxide (alumina), or cellulose. This layer of adsorbent is known as the stationary phase. Thin-layer chromatography can be used to monitor the progress of a reaction, identify compounds present in a given mixture, and determine the purity of a substance

To quantify the results, the distance travelled by the substance being considered is divided by the total distance travelled by the mobile phase. (The mobile phase must not be allowed to reach the end of the stationary phase.) This ratio is called the retardation factor (R_F).

Factors affection Rf value

- Natural adsorbent
- Mobile phase
- Thickness of layer
- Temperature
- Equilibrium
- Loading of sample
- Dipping zone
- Chromatographic techniques

HPTLC (High-performance thin-layer chromatography)

- It is an enhanced form of thin-layer chromatography (TLC). A number of enhancements can be made to the basic method of thin-layer chromatography to automate the different steps, to increase the resolution achieved and to allow more accurate quantitative measurements.
- The spot capacity (analogous to peak capacity in HPLC) can be increased by developing the plate with two different solvents, using two-dimensional chromatography.
- The procedure begins with development of sample loaded plate with first solvent. After removing it, the plate is rotated 90° and developed with a second solvent.
- Mobile phase flow by capillary action effect.
- Component move according to their affinities towards the adsorbent.
- The component with higher affinity towards adsorbent travels slowly.
- The component with lesser affinity towards the stationary face travels faster.
- The separation is based on their solubility in mobile phase.

Fig 2

Steps involving in HPLC

- Selection of chromatographic plate
- Layer pre washing
- Activation of pre coated plate
- Sample preparation and application
- Selection of mobile phase
- Pre conditioning
- Chromatographic development
- Detection
- Documentation

Gravimetric titrimetric

- To determines the quantity of analyte by a measure of the mass of a solution of known concentration. Gravimetric titrimetric arrives at the amount of analyte by measuring the duration of a given electrical current.
- A weighed sample is dissolved
- An excess of a precipitate agent is added to this solution
- From the mass and known composition of the precipitate the amount of the original ion can be determined
- The four main types of this method of analysis are

1. Precipitation
2. Volatilization
3. Electrogravimetry
4. Miscellaneous physical method. The methods involve changing the phase of the analyte to separate it in its pure form from the original mixture and are quantitative measurements.

Conclusion

The main aim of the pharmaceutical Instrumental methods of analysis have become the predominant approach for performing chemical measurements. It is critical for scientists who rely on these powerful tools to understand the fundamentals and applications of analytical instruments. However chemical instruments is not entirely sufficient in intelligently solving problems. The accurate and appropriate instrumental measurement of an incorrect or irrelevant sample obtained from a poorly designed method will not solve the intended problem. A variety of instrumental techniques are available for the application to chemical problems. They include mass spectrometric, optical spectroscopic, nuclear, surface, electrochemical and separation methods. They are commonly used individually and in manners where two or more techniques are combined beneficially, creating hyphenated techniques. In general, all of these instrumental techniques function by converting information in the non-electrical domain into the electrical domain, where the information can be transformed into meaningful information, then converted into a form that is meaningful to the analyst.

Instrumental methods of analysis have become the predominant approach for performing chemical measurements. It is critical for scientists who rely on these powerful tools to understand the fundamentals and applications of analytical instruments. However, expertise with chemical instruments is not entirely sufficient in intelligently solving problems. The accurate and appropriate instrumental measurement of an incorrect or irrelevant sample obtained from a poorly designed method will not solve the intended problem. In other words, the instrument and analyst are only as good as the sample presented for analysis. To provide results in a method that are valid, an analyst must understand the role an instrument plays in a method, making certain the appropriate sample is presented to the instrument and also applying a technique that is adequate for the desired answer. A variety of instrumental techniques are available for the application to chemical problems. They include mass spectrometric, optical spectroscopic, nuclear, surface, electrochemical and separation methods. They are commonly used individually and in manners where two or more techniques are combined beneficially, creating hyphenated techniques. In general, all of these instrumental techniques function by converting information in the non-electrical domain into the electrical domain, where the information can be transformed into meaningful information, then converted into a form that is meaningful to the analyst. The manner in which each instrument accomplishes this varies, driven by the nature of the interaction of the probe with the chemical property of the analyst, but generally the overall pathway of the flow of information is similar from instrument to instrument.

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