Analytical Techniques

Basic concepts of Laboratory Techniques (PGS 504)

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LECTURE # 02

Analytical Chemistry

Part II

Analytical Chemistry II

- Definition
- Classification
- Physical properties useful in analysis
- Methods of separation prior to analysis
- Terminologies

Definition

"Analytical Chemistry may be defined as the art and science of determining the composition of materials in terms of the elements or compounds which they contain."

Classification

Qualitative Analysis

- Informs **what** substances are present in a sample, but only very roughly their amounts

Quantitative Analysis

- Gives precise information concerning the **amount** of a specified component of the sample

Classification

Ultimate Analysis

- An analysis that permits the determination of the chemical elements of which the unknown is composed

  [N in pulses]

Proximate Analysis

- An analysis that determines the quantities of radicals, compounds, or classes of compounds

  [Protein in pulses]

Classification

Complete Analysis

- If a sample is to be analyzed for all its constituents, it is complete analysis

Partial Analysis

- If only one or two constituents are analysed in a sample, it is partial analysis

Physical Properties Useful for Analysis

Extensive Properties

- Mass (or weight)
- Volume (of a liquid or gas)

Mechanical Properties

- Specific gravity or density
- Surface tension
- Viscosity
- Velocity of sound (in a gas)

Physical Properties Useful for Analysis

Properties Involving Interaction with Radiant Energy

- Absorption of EMR (X-rays, uv-vis, IR, microwaves)
- Turbidity
- Emission of radiation (following excitation)
- Raman effect
- Rotation of the plane of polarized light
- Refractive index
- Dispersion
- Fluorescence
- Diffraction of X-rays and electrons
- Nuclear magnetic resonance

Physical Properties Useful for Analysis

Electrical Properties

- Half-cell potentials
- Decomposition potentials
- Electrical conductivity
- Dielectric constant
- Magnetic susceptibility

Physical Properties Useful for Analysis

Thermal Properties

- Transition temperatures (melting or boiling points, etc.)
- Heat of reaction (combustion, neutralization, etc.)
- Thermal conductivity (of a gas)

Nuclear Properties

- Radioactivity

General Classification of Analytical Techniques

Group	Property measured
Gravimetric	Weight of pure analyte / compound
Volumetric	Volume of standard reagent
Spectrometric	Intensity of EMR emitted / absorbed
Electrochemical	Electrical properties of analytes
Radiochemical	Intensity of radiations emitted
Mass spectrometric	Abundance of molecular fragments
Chromatographic	Physico-chemical properties
Thermal	Physico-chemical properties

Methods of Separation Before Analysis

- Precipitation
- Electrodeposition
- Formation of complexes
- Distillation
- Solvent extraction
- Partition chromatography
- Adsorption chromatography
- Ion exchange
- Electrophoresis
- Dialysis

Definitions

- **Accuracy**

  The closeness of an experimental measurement to the true or accepted value

- **Precision**

  Precision is a measure of agreement among individual measurements

Definitions

- **Analyte**

  Constituent of the sample which is to be studied by quantitative measurements or identified qualitatively

- **Sample**

  A substance or a portion of a substance about which analytical information is required

Definitions

- **Assay**

  A high accurate determination, usually of a valuable constituent in a material of large bulk, e.g. minerals and ores

- **Background**

  That proportion of a measurement which arises from sources other than the analyte itself

Definitions

- **Blank**

  A measurement in which the sample is replaced by a simulated matrix, the conditions otherwise being identical to those under which a sample would be analyzed

- **Calibration**

  A procedure which enables the response of an instrument to be related to the mass, volume or concentration of an analyte in a sample

Definitions

- **Concentration**

  The amount of a substance present in a given mass or volume of another substance [w/w, w/v and v/v]. Concentration may be expressed in several ways

- **Constituent**

  A component of a sample; may be further classified as major (>10%), minor (0.01-10%), trace (0.0001-0.01% or 1-100 ppm) and ultratrace (<1 ppm)

Definitions

- **Determination**

  A quantitative measure of an analyte with an accuracy of considerably better than 10% of the amount present

- **Estimation**

  A semi-quantitative measure of the amount of an analyte present in a sample, i.e. an approximate measure having accuracy no better than about 10% of the amount present

Definitions

- **Interference**

  An effect which alters or obscures the behavior of an analyte in analytical procedure. It may arise from the sample itself, from contaminants or reagents introduced during the procedure or from the instrumentation used for measurements

- **Masking**

  Treatment of a sample with a reagent to prevent interference with the response of the analyte by other constituents of the sample

Definitions

- **Method**

  The overall description of the instructions for a particular analysis
- **Procedure**

  A description of the practical steps involved in an analysis
- **Technique**

  The principle upon which a group of methods is based

Definitions

- **Standardization**

  Determination of the concentration of an analyte or reagent solution from its reaction with a standard or primary standard

- **Validation of Methods**

  Analyzing standards that have accepted analyte content and a matrix similar to that of the sample; to ensure that results yielded by a method are as accurate as possible [Internationally accepted standards are available]

Definitions

- **Reagent**

  A chemical used to produce a specific reaction in relation to an analytical procedure

- **Primary Standard**

  A substance whose purity and stability are particularly well established and with which other standards may be compared

Definitions

- **Detection Limit**

  The smallest amount or concentration of an analyte that can be detected by a given procedure and with a given degree of confidence

- **Sensitivity**

  The change in response from an analyte relative to a small variation in the amount being determined;

  The ability of a method to detect or determine an analyte

Methods of Expressing Concentration

- Grams per unit volume
- Percentage composition
- Specific gravity
- Volume ratios
- Molar and Formal Solutions
- Normal solutions

Grams per Unit Volume

- Concentration is expressed in terms of number of grams (or mg) of solute in each litre (or mL) of solution
- Simple & direct expression
- But not convenient for stoichiometry since substances react on molecule-to-molecule basis,not on gram-to-gram basis

Percentage Composition

- Concentration is expressed in terms of grams of solute per 100 g of solution
- A 10% solution of a given salt is made by dissolving 10 g of salt in 90 g of water

Specific Gravity

- A measure of concentration of solute in the solution
- A solution of H_2SO_4 of sp gravity 1.14 at 15 °C contains 19.6% H_2SO_4 by weight
- Not convenient, since necessitates consulting a table to determine percentage-by-weight composition of the solution

Volume Ratios

- Occasionally concentration of a mineral acid or of NH_4OH is given in terms of volume ratio of common concentrated reagent and water
- H_2SO_4 (1 : 3) signifies a solution made by mixing one volume of conc. H_2SO_4 with three volumes of water