# Analytical and Industrial Chemistry Chemistry B. Sc. (Chem. Hons. ) 3rd Year; Paper XII

#### **Analytical Chemistry Introduction**

Analytical chemistry is concerned with the chemical characterization of matter and the answer to two important questions: what is it (qualitative analysis) and how much is it (quantitative analysis). Chemicals make up everything we use or consume, and knowledge of the chemical composition of many substances is important in our daily lives. Analytical chemistry plays an important role in nearly all aspects of chemistry, for example, agricultural, clinical, environmental, forensic, manufacturing, metallurgical, and pharmaceutical chemistry. The nitrogen content of a fertilizer determines its value.

Foods must be analyzed for contaminants (e.g., pesticide residues) and for essential nutrients (e.g., vitamin content). The air we breathe must be analyzed for toxic gases (e.g., carbon monoxide). Blood glucose must be monitored in diabetics (and, in fact, most diseases are diagnosed by chemical analysis). The presence of trace elements from gun powder on a perpetrator's hand will prove a gun was fired by that hand. The quality of manufactured products often depends on proper chemical proportions, and measurement of the constituents is a necessary part of quality assurance. The carbon content of steel will influence its quality. The purity of drugs will influence their efficacy. In this text, we will describe the tools and techniques for performing these different types of analyses. There is much useful supplemental material on the text website, including Excel programs that you can use, and videos to illustrate their use. You should first read the Preface to learn what is available to you, and then take advantage of some of the tools.

### Qualitative analysis and quantitative analysis

The discipline of analytical chemistry consists of qualitative analysis and quantitative analysis. The former deals with the identification of elements, ions, or compounds present in a sample (we may be interested in whether only a given substance is present), while the latter deals with the determination of how much of one or more constituents is present. The sample may be solid, liquid, gas, or a mixture. The presence of gunpowder residue on a hand generally requires only qualitative knowledge, not of how much is there, but the price of coal will be determined by the percent of undesired sulfur impurity present. Qualitative tests may be performed by selective chemical reactions or with the use of instrumentation. The formation of a white precipitate when adding a solution of silver nitrate in dilute nitric acid to a dissolved sample indicates the presence of a halide. Certain chemical reactions will produce colors to indicate the presence of classes of organic compounds, for example, ketones. Infrared spectra will give "fingerprints" of organic compounds or their functional groups.

A clear distinction should be made between the terms selective and specific:

- A selective reaction or test is one that can occur with other substances but exhibits a degree of preference for the substance of interest.
- A specific reaction or test is one that occurs only with the substance of interest.

### **How Did Analytical Chemistry Originate?**

That is a very good question. Actually, some tools and basic chemical measurements date back to the earliest recorded history. Fire assays for gold are referred to in Zechariah 13:9, and the King of Babylon complained to the Egyptian Pharoah, Ammenophis the Fourth (1375–1350 BC), that gold he had received from the pharaoh was "less than its weight" after putting it in a furnace. The perceived value of gold, in fact, was probably a major incentive for acquiring analytical knowledge. Archimedes (287–212 BC) did non-destructive testing of the golden wreath of King Hieron II. He placed lumps of gold and silver equal in weight to the wreath in a jar full of water and measured the amount of water displaced by all three. The wreath displaced an amount between the gold and silver, proving it was not pure gold! The balance is of such early origin that it was ascribed to the gods in the earliest documents found. The Babylonians created standard weights in 2600 BC and considered them so important that their use was supervised by the priests. The alchemists accumulated the chemical knowledge that formed the basis for quantitative analysis as we know it today. Robert Boyle coined the term analyst in his 1661 book, The Sceptical Chymist. Antoine Lavoisier has been considered the "father of analytical chemistry" because of the careful quantitative experiments he performed on conservation of mass (using the analytical balance). (Lavoisier was actually a tax collector and dabbled in science on the side. He was guillotined on May 8, 1793, during the French Revolution because of his activities as a tax collector.) Antoine Lavoisier used a precision balance for quantitative experiments on the conservation of mass.

He is considered the "father of quantitative analysis." Gravimetry was developed in the seventeenth century, and titrimetry in the eighteenth and nineteenth centuries. The origin of titrimetry goes back to Geoffroy in 1729; he evaluated the quality of vinegar by noting the quantity of solid  $K_2CO_3$  that could be added before effervescence ceased. Gay-Lussac, in 1829, assayed silver by titration with 0.05% relative accuracy and precision!

## Steps in an analysis.

#### Define the Problem Prepare the Sample for Analysis Factors: What is the problem—what needs to be found? Factors: Qualitative and/or quantitative? · Solid, liquid, or gas? What will the information be used for? Who will use it? · Dissolve? When will it be needed? Ash or digest? · How accurate and precise does it have to be? · Chemical separation or masking of interferences needed? · What is the budget? · Need to concentrate the analyte? . The analyst (the problem solver) should consult with · Need to change (derivatize) the analyte for detection? the client to plan a useful and efficient analysis, including · Need to adjust solution conditions (pH, add reagents)? how to obtain a useful sample. Perform Any Necessary Chemical Separations Select a Method Factors: Distillation Sample type Precipitation · Size of sample · Solvent extraction Sample preparation needed · Solid phase extraction Concentration and range (sensitivity needed) Chromatography (may include the measurement step) · Selectivity needed (Interferences) Electrophoresis (may include the measurement step) · Accuracy/precision needed Tools/Instruments available Expertise/experience Cost Perform the Measurement Speed Factors: Does it need to be automated? Calibration Are methods available in the chemical literature? Validation/controls/blanks Are standard methods available? Replicates Are there regulations that need to be followed? Obtain a Representative Sample Calculate the Results and Report Statistical analysis (reliability) Sample type/homogeneity/size · Report results with limitations/accuracy information Sampling statistics/errors Interpret carefully for Intended audience. Critically evaluate results. Are Iterations needed?