分析化學與儀器分析
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Introduction to Analytical Chemistry

Edited from textbook of "Fundamentals of Analytical Chemistry"
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Chapter 1 - Introduction

What is Analytical Chemistry?

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What is Analytical Chemistry?

Analytical chemistry is a **measurement science** consisting of a set of powerful ideas and methods that are useful in all fields of science and medicine.

More detailed definition of "analytical chemistry" is in following page:

Analytical chemistry

Analytical chemistry is the study of the separation, identification, and quantification of the chemical components of natural and artificial materials.[1] Qualitative analysis gives an indication of the identity of the chemical species in the sample, and quantitative analysis determines the amount of certain components in the substance.
The separation of components is often performed prior to analysis.

Analytical methods can be separated into **classical and instrumental**.[2]

Classical methods (also known as **wet chemistry methods**) use separations such as precipitation, extraction, and distillation and qualitative analysis by color, odor, or melting point.
Classical quantitative analysis is achieved by measurement of weight or volume.
[Note: It is called "wet chemistry" since most analyzing is done in the liquid phase.]

Instrumental methods use an apparatus to measure physical quantities of the analyte such as light absorption, fluorescence, or conductivity.

The separation of materials is accomplished using chromatography, electrophoresis or field flow fractionation methods.

Qualitative Analysis *vs.* Quantitative Analysis

- **Qualitative** analysis reveals the identity of the elements and compounds in a sample.
- **Quantitative** analysis indicates the amount of each substance in a sample.

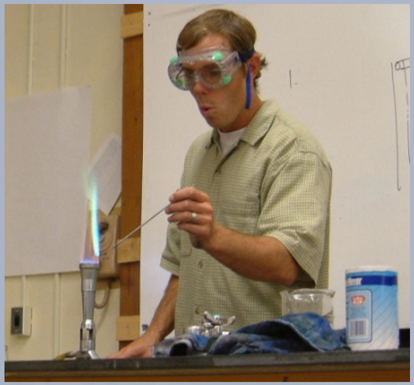
Qualitative analysis

A qualitative analysis determines the presence or absence of a particular compound, but not the mass or concentration. By definition, qualitative analyses do not measure quantity.

Chemical tests

There are numerous qualitative chemical tests, for example, the acid test for gold and the Kastle-Meyer test for the presence of blood.

Flame/color test



Blood test Kastle-Meyer test - A drop of phenolphthalin reagent is added to the sample.

The presence of copper in this qualitative analysis is indicated by the bluish-green color of the flame. Yellow flame for Na.

Analytical Chemistry vs. Instrumental Analysis

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Analytical chemistry is also focused on improvements in experimental design, chemometrics, and the creation of new measurement tools to provide better chemical information.

Classical Analytical Chem.

→ Modern Instrumentation

Although modern analytical chemistry is dominated by sophisticated instrumentation, the roots of analytical chemistry and some of the principles used in modern instruments are from traditional techniques many of which are still used today.

These techniques (traditional) also tend to form the backbone of most undergraduate analytical chemistry educational labs.

1A The Role of Analytical Chemistry

- Analytical chemistry is applied throughout **industry, medicine, and all the science**s.
- Quantitative analytical measurements also play a vital role in **chemistry, biochemistry, biology, geology, physics, and the other sciences**.
- Many scientists devote much time in the laboratory gathering quantitative information about systems that are important and interesting to them.

1A The Role of Analytical Chemistry

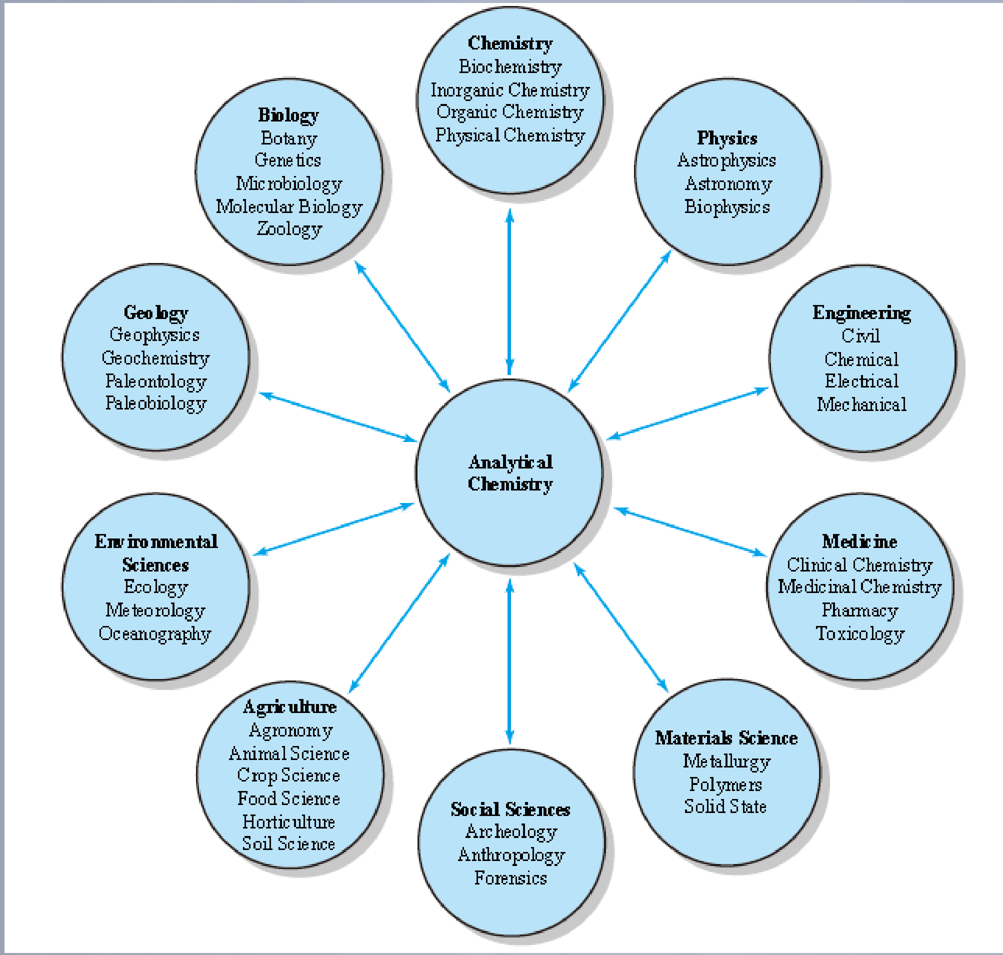


Figure 1-1

The relationship between analytical chemistry, other branches of chemistry, and the other sciences.

The central location of analytical chemistry in the diagram signifies its importance and the breadth of its interactions with many other disciplines.

1B Classifying Quantitative Analytical Methods

The results of a typical quantitative analysis are computed from two measurements:

1. One is the mass or the volume of sample to be analyzed.

2. The second is the measurement of some quantity that is proportional to the amount of analyte in the sample, such as mass, volume, intensity of light, or electrical charge.

1B Classifying Quantitative Analytical Methods

We classify analytical methods (quantitative) according to the nature of this final measurement.

1. **Gravimetric methods** determine the mass of the analyte or some compound chemically related to it.
2. **Volumetric method** determines the volume of a solution containing sufficient reagent to react completely with the analyte.

1B Classifying Quantitative Analytical Methods (cont.)

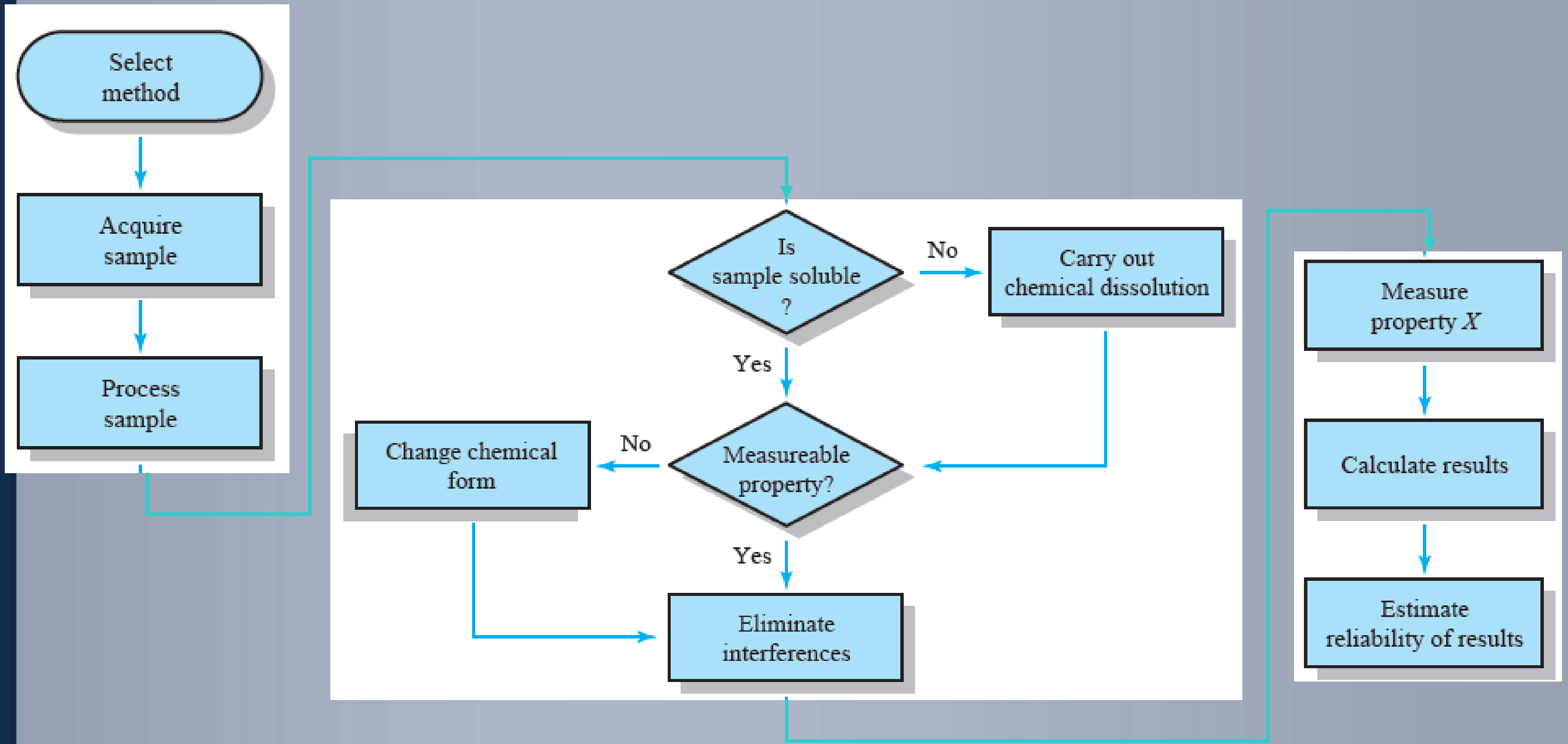
3. Electroanalytical methods involve the measurement of such electrical properties as **voltage, current, resistance**, and quantity of electrical charge.
4. Spectroscopic methods are based on measurement of the interaction between **electromagnetic radiation and analyte atoms or molecules** or on the production of such radiation by analytes.

1B Classifying Quantitative Analytical Methods (cont.)

Miscellaneous methods:

- mass-to-charge ratio
- rate of radioactive decay
- heat of reaction
- rate of reaction
- sample thermal conductivity
- optical activity
- refractive index.

Flow Diagram Showing the Steps in a Quantitative Analysis



1C-1 Picking a Method

- One of the first questions to be considered in the selection process is the level of accuracy required.
- A second consideration related to economic factors is the number of samples to be analyzed.
- The complexity of the sample and the number of components in the sample always influence the choice of method to some degree.

1C-2 Acquiring the Sample

- Sampling involves obtaining a small mass of a material whose composition accurately represents the bulk of the material being sampled.
- Sampling is frequently the most difficult step in an analysis and the source of greatest error. →The final results of an analysis will never be any more reliable than the reliability of the sampling step.

1C-2 Acquiring the Sample

- A material is *heterogeneous* if its constituent parts can be distinguished visually or with the aid of a microscope.
- An ***assay*** (化驗,分析) is the process of determining how much of a given sample is the material indicated by its name.
- We *analyze* **samples** and we determine ***substances***.

1C-3 Processing the Sample

- Under certain circumstances, no sample processing is required prior to the measurement step.
- **Under most circumstances**, we must process the sample in any of a variety of different ways.
- The first step in processing the sample is often the preparation of a laboratory sample.

1C-3 Processing the Sample

- Preparing a Laboratory Sample
  - A solid sample is ground, mixed to ensure homogeneity, and stored for various lengths of time before analysis begins.
  - Because any loss or gain of water changes the chemical composition of solids, it is a good idea to dry samples just before starting an analysis.
  - Alternatively, the moisture content of the sample can be determined at the time of the analysis in a separate analytical procedure.

1C-3 Processing the Sample

- Preparing a Laboratory Sample (Cont.)
  - Liquid samples are subject to solvent evaporation
  - If the analyte is a gas dissolved in a liquid, analyte must be kept inside a second **sealed container** to prevent contamination by atmospheric gases.
  - Extraordinary measures, including sample manipulation and measurement in an inert atmosphere, may be required to preserve the integrity of the sample.

1C-3 Processing the Sample

- **Replicate samples, or replicates**, are portions of a material of approximately the same size that are carried through an analytical procedure at the same time and in the same way.
- Replication improves the quality of the results and provides a measure of their reliability.
- Quantitative measurements on replicates are usually averaged, and various statistical tests are performed on the results to establish their reliability.

1C-3 Processing the Sample

- Preparing **Solutions**: Physical and Chemical Changes
  - Ideally, the solvent should dissolve the entire sample, including the analyte, rapidly and completely.
  - The sample may require heating with aqueous solutions of strong acids, strong bases, oxidizing agents, reducing agents, or some combination of such reagents.
  - It may be necessary to ignite the sample in air or oxygen or perform a high-temperature fusion of the sample in the presence of various fluxes.

1C-4 Eliminating Interferences

- Few chemical or physical properties of importance in chemical analysis are unique to a single chemical species.
- Species other than the analyte that affect the final measurement are called interferences, or interferents.
- An *interference* is a species that causes an error in an analysis by enhancing or attenuating (making smaller) the quantity being measured.

1C-4 Eliminating Interferences

- Techniques or reactions that work for only one analyte are said to be *specific.* Techniques or reactions that apply for only a few analytes are *selective.*

- The *matrix,* or *sample matrix,* is all of the components in the sample containing an analyte. [note: In chemical analysis, **matrix** refers to the components of a sample other than the analyte[1] of interest.]

Remain Steps of A Typical Quantitative Analysis

- **1C-5 Calibration and Measurement**
  - Ideally, the measurement of the property is directly proportional to the concentration.

$$C_A = kX$$

where *k* is a proportionality constant

- **1C-6 Calculating Results**
  - Computing analyte concentrations are based on the raw experimental data collected in the measurement step, the characteristics of the measurement instruments, and the stoichiometry of the analytical reaction.

Remain Steps of A Typical Quantitative Analysis

- **1C-7 Evaluating Results by Estimating Their Reliability**
  - Analytical results are incomplete without an estimate of their reliability.

1D An Integral Role For Chemical Analysis: Feedback Control Systems

- Chemical analysis is the measurement element in all of these examples and in many other cases.
- The process of continuous measurement and control is often referred to as a ***feedback system***, and the cycle of measurement, comparison, and control is called a ***feedback loop*** *[**see next page**]*.

Feedback Control Systems

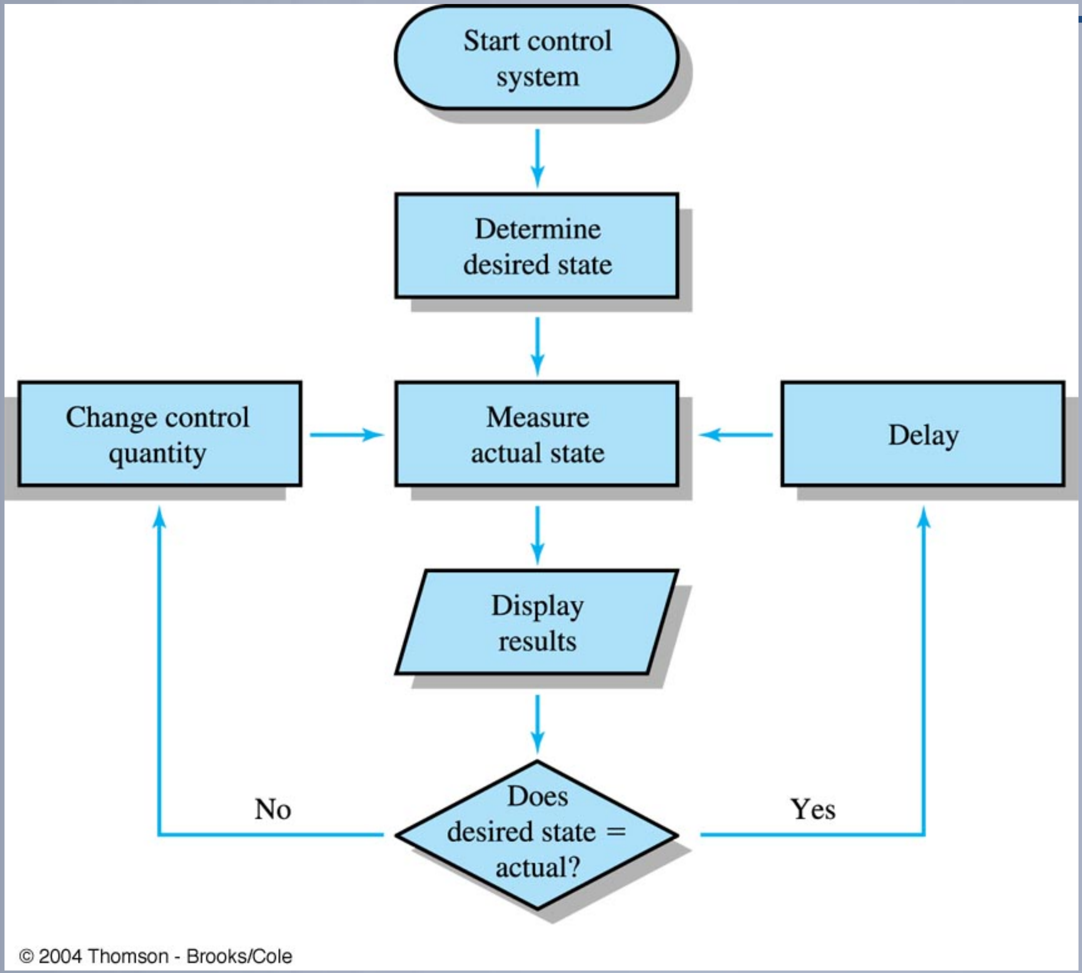


Figure 1-3 Feedback system flow diagram. The desired state is determined, the actual state of the system is measured, and the two states are compared. The difference between the two states is used to change a controllable quantity that results in a change in the state of the system. Quantitative measurements are again performed on the system, and the comparison is repeated. The new difference between the desired state and the actual state is again used to change the state of the system if necessary. The process provides continuous monitoring and feedback to maintain the controllable quantity, and thus the actual state, at the proper level. The text describes the monitoring and control of blood glucose as an example of a feedback control system.

THE END –Chap 1 - Introduction