

A BRIEF HISTORY OF ANALYTICAL CHEMISTRY

(http://www.trincoll.edu/~henderson/textfi-1/Chem%20208%20notes/introduction_to_analytical_chemi.htm)

1950's - This was a pretty dull field. pH meters and single wavelength spectrophotometers, and electrochemical techniques. Lots of titrations, gravimetric analysis. Some important work done to lay the theoretical groundwork. Data was primarily one dimensional.

Experiment → Number

1960's - Invention of Gas Chromatography and Atomic Absorption spectrophotometry make trace analysis possible and reasonably easy. Analysis of ppm and ppb levels of metals and organics in the environment begins. Text book triples in size. Scanning spectrophotometers become common. Thus data representations were now two dimensional.

Experiment → Graph

1970's - Invention of liquid chromatography and the common use of mass spectrometry for analytical chemistry begins. GC and AA reach new limits of sensitivity allowing part per trillion trace analysis. We begin to find virtually everything almost everywhere. Surface analysis of thin layers becomes common. Analytical chemistry is brought to bear on problems of the environment, energy, and biological and physiological analysis.

1980's - Continued strides in trace analysis and in identification of trace components through interfaced (hyphenated) methods eg. GC-MS, LC-MS, GC-IR, etc. Computers appear to control instruments, manipulate data, and run experiments Robots appear to conduct complete analytical schemes Multidimensional data representations add new dimensions to data interpretation It becomes possible to detect single atoms of many substances. Three dimensional data presentations become important (MS-MS and 2D NMR).

Experiment → 3D Graphic

1990'S - Detection limits continue to drop to the point that one can begin to discuss detection of single atoms and molecules. Sample preparation is also a major focus as old, time consuming methods of solvent extraction are replaced with fast, automated procedures. Multi-channel analysis becomes the major thrust (396 well plates, parallel analysis).

2000's – ??

What is Analytical Chemistry Anyway? (<http://www.sbuniv.edu/~ggray/CHE3345/chp1.html>)

Analytical chemistry deals with separating, identifying, and quantifying the relative amounts of the components of an analyte.

What is an analyte?

Analyte(s) = the thing to analyzed; the component(s) of a sample that are to be determined.

There are several different areas of analytical chemistry:

1. **Clinical analysis** - blood, urine, feces, cellular fluids, etc., for use in diagnosis.
2. **Pharmaceutical analysis** - establish the physical properties, toxicity, metabolites, quality control, etc.
3. **Environmental analysis** - pollutants, soil and water analysis, pesticides.
4. **Forensic analysis** - analysis related to criminology; DNA finger printing, finger print detection; blood analysis.
5. **Industrial quality control** - required by most companies to control product quality.
6. **Bioanalytical chemistry and analysis** - detection and/or analysis of biological components (i.e., proteins, DNA, RNA, carbohydrates, metabolites, etc.).
 - a. This often overlaps many areas.
 - b. Develop new tools for basic and clinical research.

For the first month and a half or so of this course, we will study three overall analytical methods:

1. **Gravimetric methods** - measure the mass of the analyte (or a compound chemically related to the analyte).

2. **Volumetric methods** - measure the volume of a solution containing sufficient reagent to react with the analyte (i.e., titrations or gas analysis).

3. **Spectroscopic methods** - measuring the interaction between the analyte and electromagnetic radiation (or the production of radiation by an analyte).

Then, for the remainder of the course, we will study “miscellaneous methods” (that don't fit the classifications above):

Mass spectrometry - mass-to-charge ratio of an analyte's decomposition products.

BUT THERE ARE OTHERS, that will either touch upon or you'll encounter in further courses, graduate school or your career:

Radiochemical methods - measuring rates of radioactive decay by an analyte.

Kinetics - measuring reaction rates.

Thermal conductivity.

Optical activity - measuring the interactions of an analyte with plane-polarized light.

Refractometry/Refractive index.

STEPS IN A TYPICAL QUANTITATIVE ANALYSIS (See powerpoint lecture #1)

A. Method selection.

B. Sampling. A representative sample must be obtained.

C. Preparing a laboratory sample.

D. Defining replicate samples. Replicate samples are always performed unless the quantity of the analyte, expense or other factors prohibit.

E. Preparing solutions of the sample.

F. Eliminating interferences. Interferences are substances that prevent direct measurement of the analyte and must be removed.

G. Calibration and measurement.

H. Calculating results.

I. Evaluating the results and estimating their reliability. This requires appropriate use of statistics.

SAMPLE PREPARATION

(http://www.trincoll.edu/~henderson/textfi-1/Chem%20208%20notes/sample_preparation.htm)

I. Is the sample a Solid or a Liquid?

Liquids

II. Are you interested in all sample components or only one or a few?

If only a few then separation is necessary by extraction or chromatography.

III. Is the concentration of the analytes appropriate for the measurement technique?

If not, dilute or concentrate with extraction, evaporation, lyophilization.

IV. Is sample unstable

If yes, derivatize, cool, freeze, store in dark

V. Is the liquid or solvent compatible with the analytical method?

If not, do solvent exchange with extraction, distillation, lyophilization.

Solid Samples

I. Is the sample size appropriate for dissolution or extraction?

If not, mill, grind, chop, blend etc.

II. Is the sample homogeneous?

Homogenize

Take a representative sample - sample size required is a function of particle size.

III. Are you interested in all sample components or only **one or a few**?

If only a few, are they the **volatile** components. If so use Headspace analysis, Solid Phase microextraction, or purge and trap.

If only a few **non-volatile** components then separation is necessary by boiling, soxhlet extraction, sonication, microwave digestion, supercritical fluid extraction.

IV. If **all components** must be analyzed it must be dissolved.

Dissolution - What is the sample soluble in?

Water, organics, etc.

What if the sample is insoluble? For organics this is tough - polymer analysis of intact high polymers. These may be impossible to dissolve in any solvent.

For Inorganic analysis -Ashing techniques - Wet vs Dry Ashing

Fusions with Carbonate and Borate

What are some of these Strange Techniques?

Soxhlet Extraction -

Solid Phase Extraction-

Solid Phase Microextraction-

Headspace Sampling-

Purge and Trap analysis-

Microwave methods-

Sonication Methods-