

The Holy Grail of Analytical Instrumentation

Characteristics:

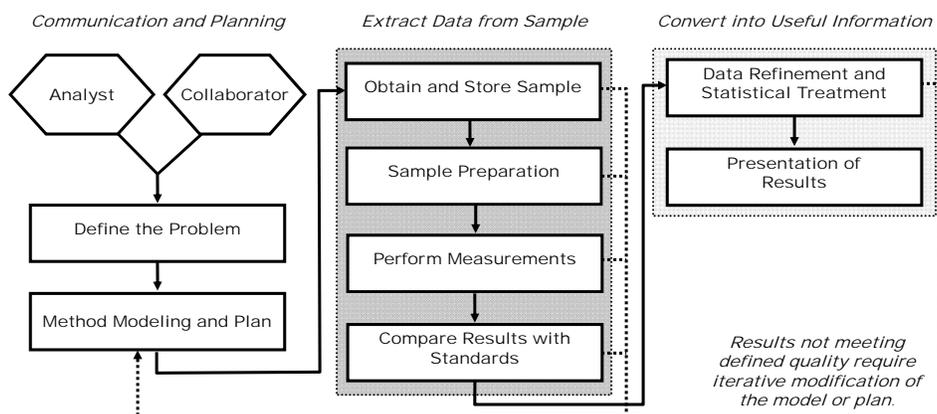
- Sensitive
- Selective
- Suitable for wide range of analytes
- Useable in a variety of environments
- Portable



Goals of CHEM 322

- Discuss factors that limit the application of instrumental methods.
- Identify some of the “tools” in the chemist’s toolbox.
- Explore principles by which these instruments operate.
- Identify advantages and limitations of each instrument.
- Gain hands-on experience using some of these tools.

The Analytical Process: “The Big Picture”



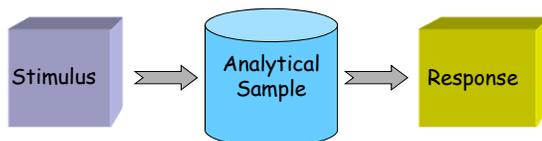
- The instrument is only a very small part of the picture.
- BUT, the choice of instrument influences each of the other steps

You Gotta Understand the Problem!

- Questions a good analytical chemist asks to define a problem:
 - What type of data is needed?
 - Qualitative: ID, Structure determination, etc.
 - Quantitative: What accuracy/precision is expected?
 - In what form is the sample?
 - How much sample will I have?
 - What other materials will likely be found in the sample?
- Based on the answers to these questions, the analysis can proceed.

Instrument Characteristics

- Converting chemical/physical properties into information



- Stimulus: chemical, optical, electrical...
- Response: detectible effect of stimulus
 - Related to quantity of analyte present
- To understand the benefits and limitations of an instrument, you must understand this relationship.

Instrument Data Flow

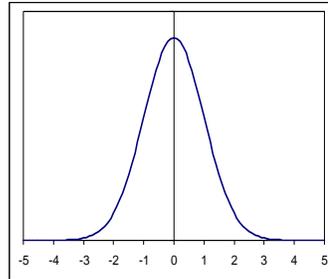
TABLE 1-2 Some Examples of Instrument Components

Instrument	Energy Source (stimulus)	Analytical Information	Information Sorter	Input Transducer	Data Domain of Transduced Information	Signal Processor/Readout
Photometer	Tungsten lamp	Attenuated light beam	Filter	Photodiode	Electrical current	Amplifier, digitizer, LED display
Atomic emission spectrometer	Inductively coupled plasma	UV or visible radiation	Monochromator	Photomultiplier tube	Electrical current	Amplifier, digitizer, digital display
Coulometer	Direct-current source	Charge required to reduce or oxidize analyte	Cell potential	Electrodes	Time	Amplifier, digital timer
pH meter	Sample/glass electrode	Hydrogen ion activity	Glass electrode	Glass-calomel electrodes	Electrical voltage	Amplifier, digitizer, digital display
Mass spectrometer	Ion source	Mass-to-charge ratio	Mass analyzer	Electron multiplier	Electrical current	Amplifier, digitizer, computer system
Gas chromatograph with flame ionization	Flame	Ion concentration vs. time	Chromatographic column	Biased electrodes	Electrical current	Electrometer, digitizer, computer system

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Quantifying Instrument Performance: Figures of Merit

- Precision:
 - Definition:
 - Random errors
 - Review Statistics!



- Know how to calculate standard deviation, RSD, variance and what these quantities describe.

Foundations: Getting our Story Straight

- Analytical Technique vs. Analytical Method
- Procedure vs. Protocol
 - Guiding forces
 - SOP's
- Instrument vs. Machine
- The importance of method validation

Quantifying Instrument Performance: Figures of Merit

- Bias:
 - Definition:

 - Systematic errors
- Sensitivity
 - Definition:
- Detection Limit or LOD:
 - Definition:

Quantifying Instrument Performance: Figures of Merit

- Dynamic Range or LDR:
 - Definition:
- Selectivity:
 - Definition:

Keys to Understanding Instruments

- No Black Boxes!
- Think of key questions:
 - What chemical or physical phenomenon is the instrument probing?
 - What types of samples is the instrument best suited to handle?
 - What detection limits are reasonable and what limits detectability?
 - What steps do I need to take to ensure the best outcome for my measurements?
 - What does the output of the instrument tell me and how to I translate that into an answer to my question?