

Process Analytical Chemistry

A Different World

Lynn A. Melton

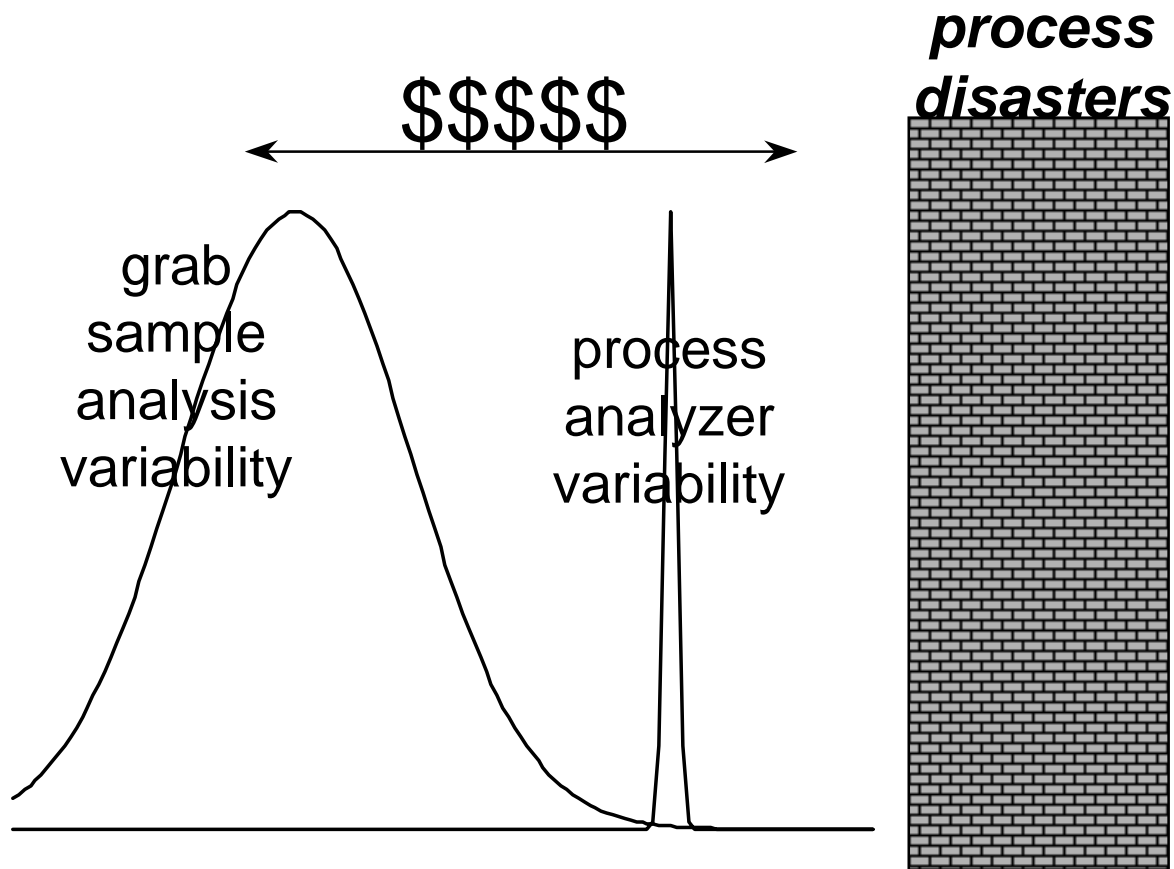
University of Texas at Dallas

melton@utdallas.edu

Process Analytical Chemistry

- PAC is the art and science of making measurements for the purpose of control of large scale chemical processes.
- In large companies, the number of “lab chemists” is decreasing and the number of “process analyzers” is increasing.
- Industry needs people with knowledge of process analytical chemistry.

Lab v. On-line Analysis



Process Analytical Chemistry

- Your task is to construct an analytical laboratory in which
 - data is available $> 98\%$ of the time for 15 years;
 - each instrument gets one hour of service/month;
 - updated analyses are available every 5 minutes;
 - the samples may be high pressure, high temperature, wet, and/or dirty;
 - the lab is in a corrosive environment.

Exterior of Analyzer House



Interior of Analyzer House



Process Analytical Chemistry

Making Money!

- Allow better process control
- Must calculate LTCO (“long term cost of ownership”)
 - an analyzer which requires a lot of maintenance can cost you money (and the engineers will stop using the data)
 - reliability is the key!

Process Analytical Chemistry

- Simplicity is the key to reliable instrument operation.
- The goal is
 - “no moving parts”
 - automated self calibration
 - failure of instrument is unambiguous.

Process Analytical Chemistry

Gas Chromatography

- Widely used in industry
- Advantages
 - multi-component analyses
 - can determine isomers
- Disadvantages
 - speed of analysis
 - the multiport valve is a “moving part”

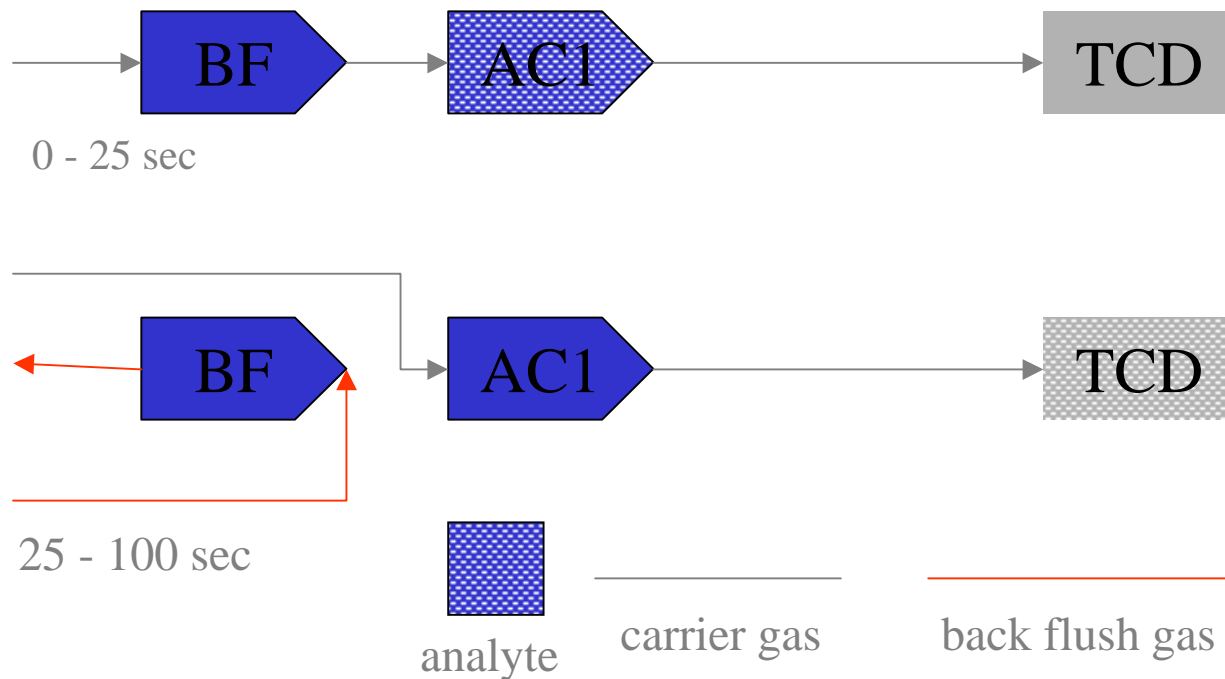
Online GC Applications

Backflush - Uses

- Used to trap “heavy” components for flushing to vent or detector
 - protects analytical column(s)
 - more robust applications
 - 6 mos - 1 year without maintenance
 - reduces analysis time
 - most online applications include a backflush operation

Online GC Applications

Backflush - Timing Diagram

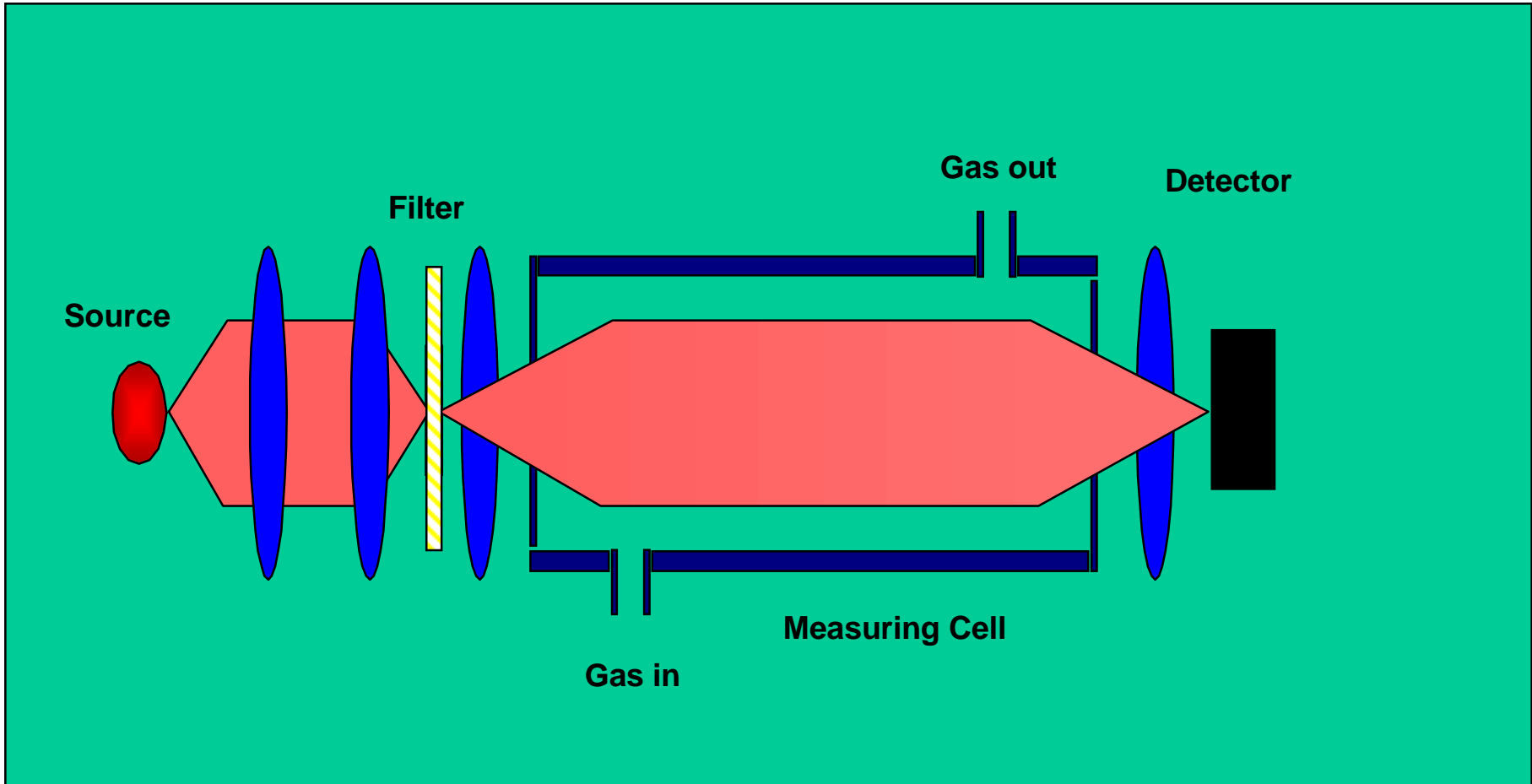


Online Spectroscopy Non-Dispersive IR Analyzers

- Filter Photometers
- Luft detection
- Gas Correlation
- Maintenance Aspects

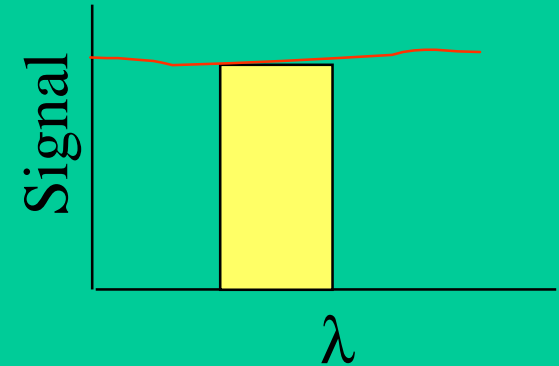
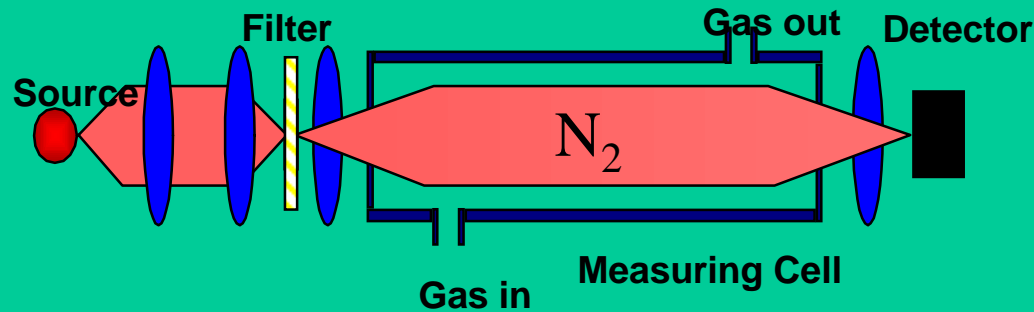
Filter Photometers

(single-beam, single- λ)

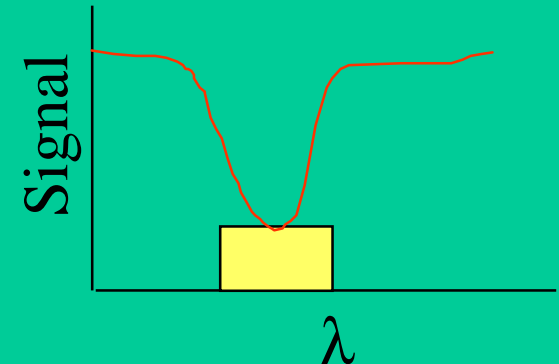
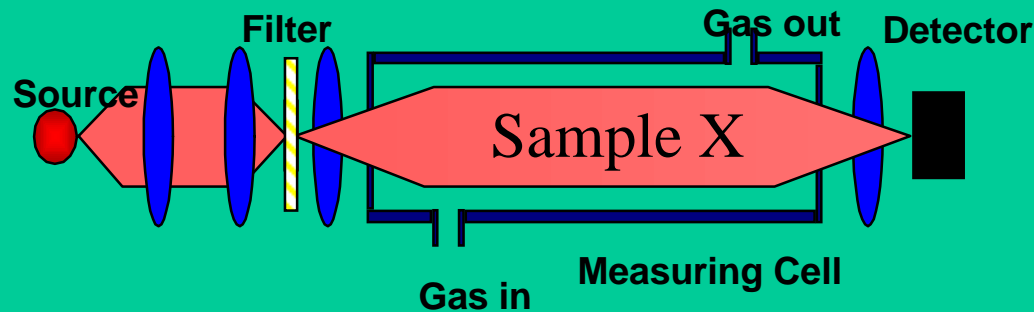


Method for Single-beam, Single- λ

Reference

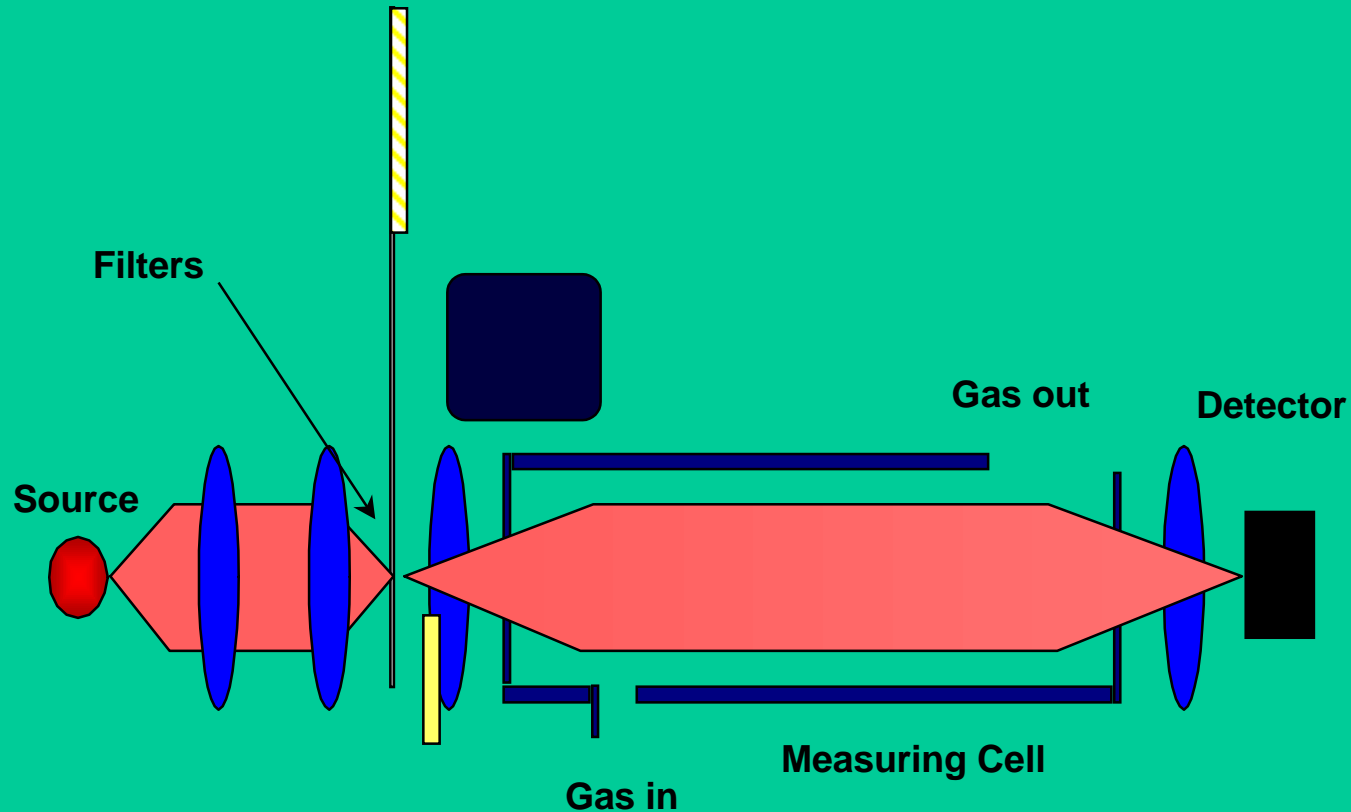


Measurement



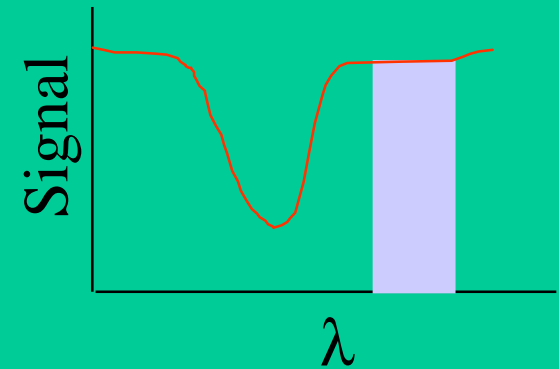
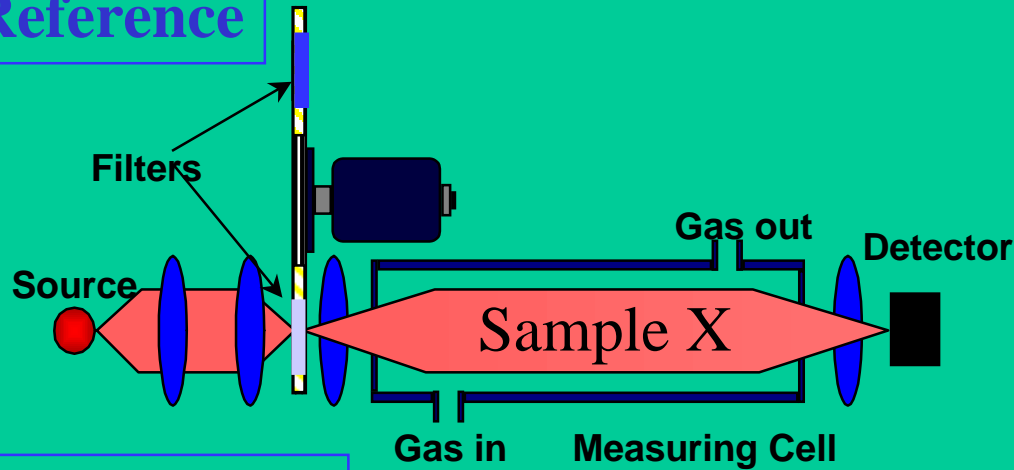
Filter Photometers

(single-beam, dual- λ)

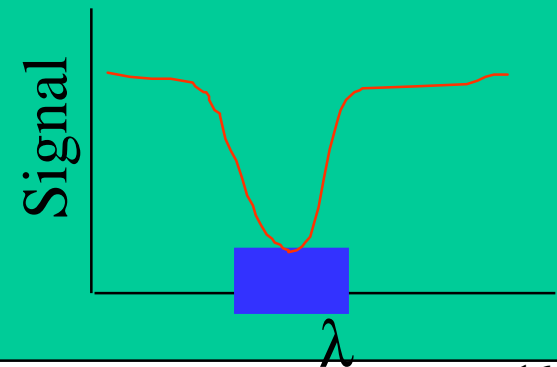
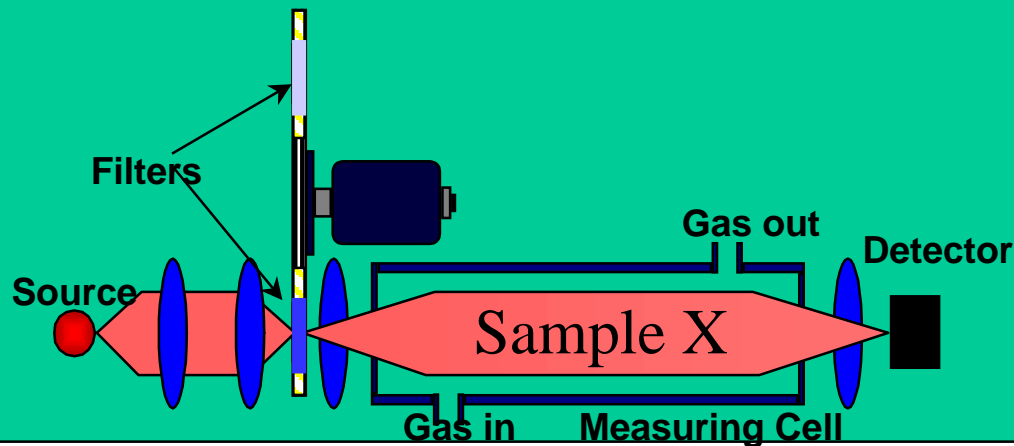


Method for Single-Beam, Dual- λ

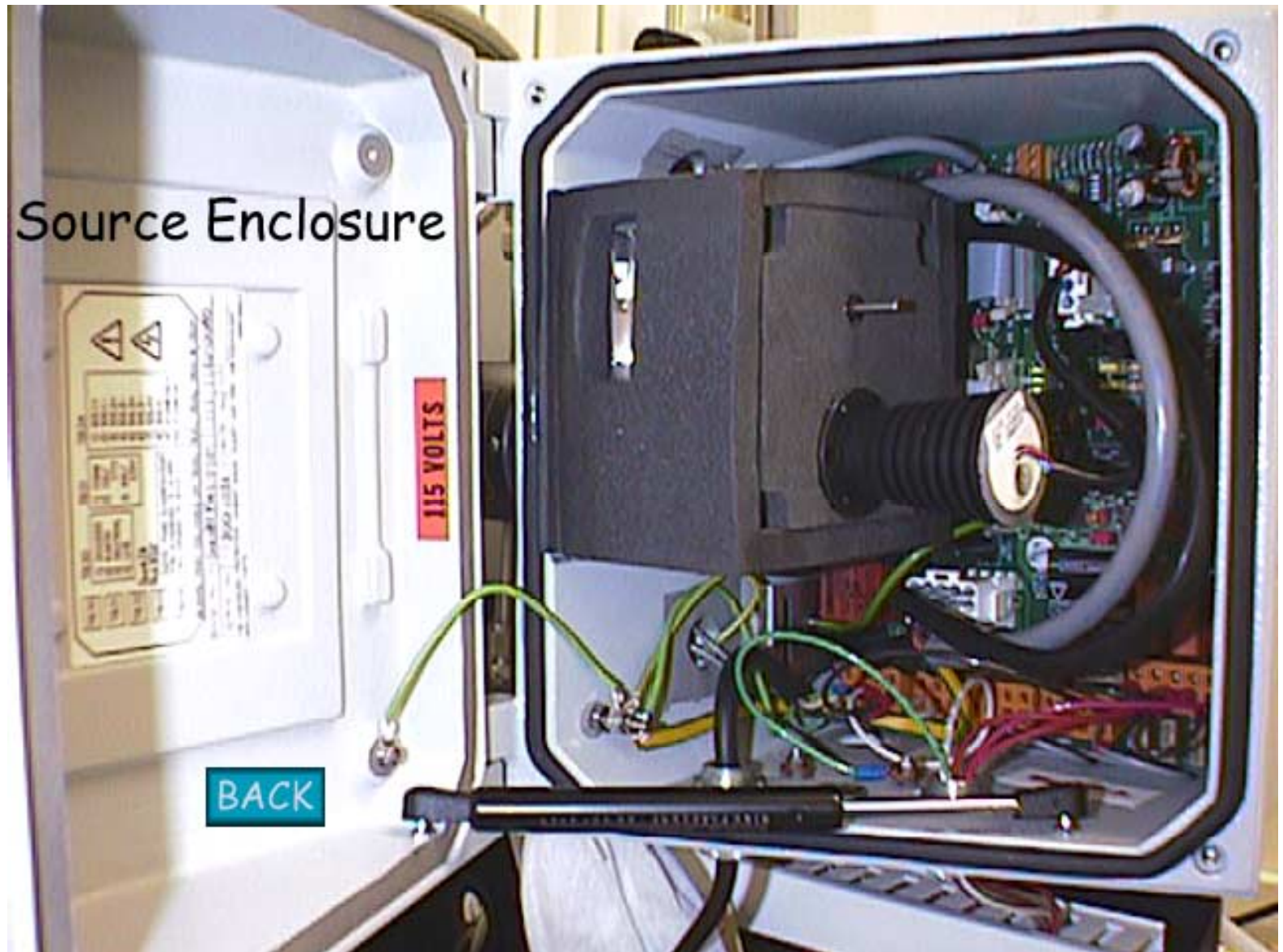
Reference



Measurement



Source Enclosure



Process Analytical Chemistry

Sampling

- Expect that the sampling system will cost 40-50% of the total project
- If the sampling system does not work right, the measurements are meaningless.
- Must transport and condition the sample so that the (one atmosphere, room temperature) instrument can make accurate, long term reliable measurements.

General Characteristics of Sample Systems

- Typically cost about as much as the analyzer
- Typically the highest maintenance part of the system
- The part of an analyzer system that is most likely to result in the failure of an analyzer project

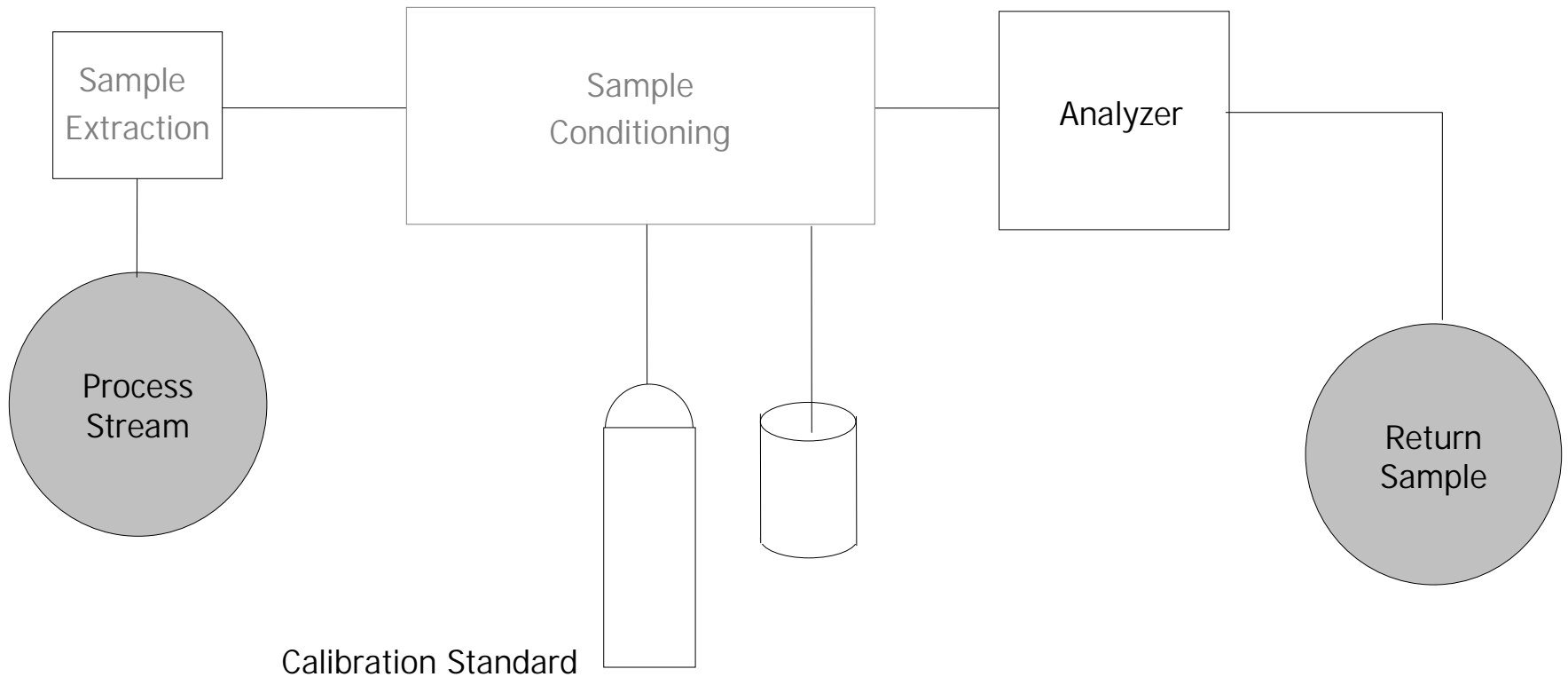
In-Situ vs. Extractive Samples

- In-situ analyzer sensor/probe is mounted directly in the process vessel
 - fiber optic spectrometer interface
 - zirconium oxide O₂ analyzer
 - conductivity probe
- Extractive sample is removed from the process pipe and delivered to the analyzer
 - gas chromatographs
 - mass spectrometers
 - paramagnetic O₂ analyzers

Sample Conditioning Systems

- Extract sample from process
- Transfer sample to analyzer
- Convert or maintain the sample state to one that is compatible with the analyzer
- Return the sample to an appropriate waste stream
- Provide additional infrastructure to support the analyzer such as calibration, lab sample facilities, etc.

Components of a Sample Conditioning System



Sample System Design Issues (1)

- Does the sample represent the portion of the process you want to analyze?
- Does the sample system provide the sample to the analyzer in a timely manner?
- Is the sample presented to the analyzer in a state that is compatible with the analyzer?

Sample System Design Issues (2)

- Is the sample system cost effective?
- Is the sample system reliable?
- Is the sample system maintainable?
- Is the sample system safe during all process conditions and during hardware failure incidents (fail-safe)?

Practical Aspects of Process Analytical Chemistry

- In 1999, when Melton was at Dow Chemical Company in Freeport, Texas, he and Dow organized a one week intensive short course, Practical Aspects of Process Analytical Chemistry.
- The course materials are now available to college instructors.

Practical Aspects of Process Analytical Chemistry

- In January 2001, UT-Dallas and Dow Chemical Company co-sponsored a one week workshop to help twelve college faculty members learn Practical Aspects of Process Analytical Chemistry.
- They will begin to incorporate the PAPAC materials into their chemistry and chemical engineering courses.

UT-Dallas

Doctor of Chemistry

- Prepares students for careers as “industrial problem solvers”
- Every student spends a year working in industrial R&D -- the Industrial Practicum
- 90% of DChem graduates go directly from campus to career R&D positions -- no low paying postdoc position.