Chemical Threats
Advanced Chemistry Course

SAND2010-3894P

Analytical Chemistry Measurements and Hardware

SAND Number: 200n-qqqq

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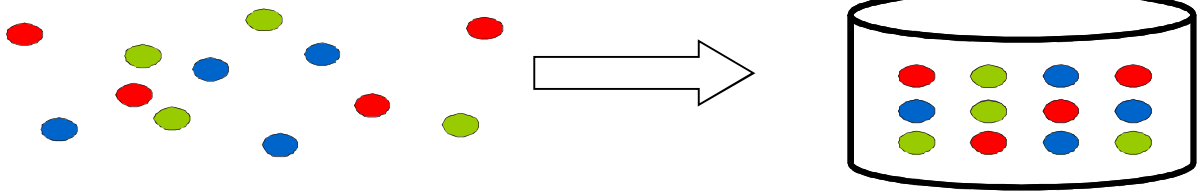
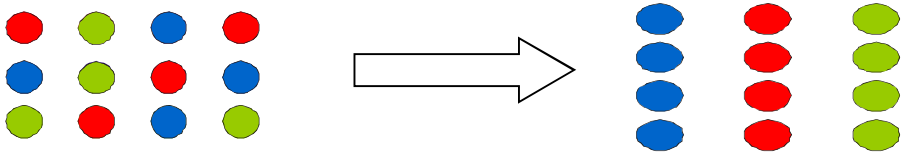
The "Three Steps" of Almost Every Analytical Chemistry Measurement

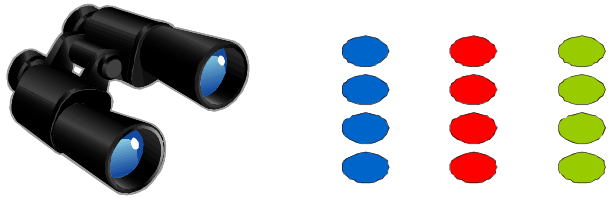
1) Get your sample (collect / preconcentrate / prepare)



2) Separate (isolate the "analyte of interest")

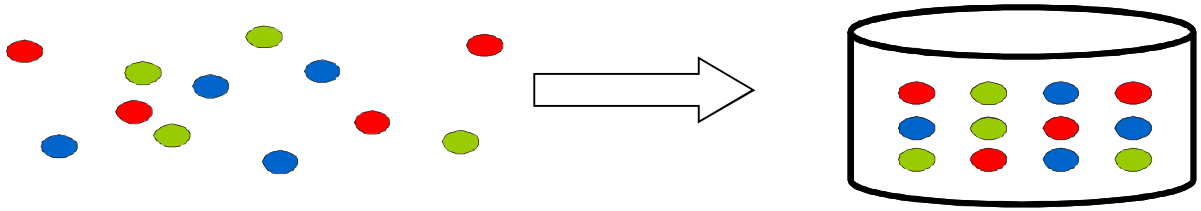


3) Detect (measure chemical or physical property)



Mix and match as time, budget, and hardware allow!

There are many ways to get and prepare your sample (which contains your analyte)



Gas
Liquid
Solid

- Containers
  - Bag, Cannister
- Immobilize
  - Solvent, Sorbent, Temp.
- React
  - Derivatize, Neutralize
- Change physical state

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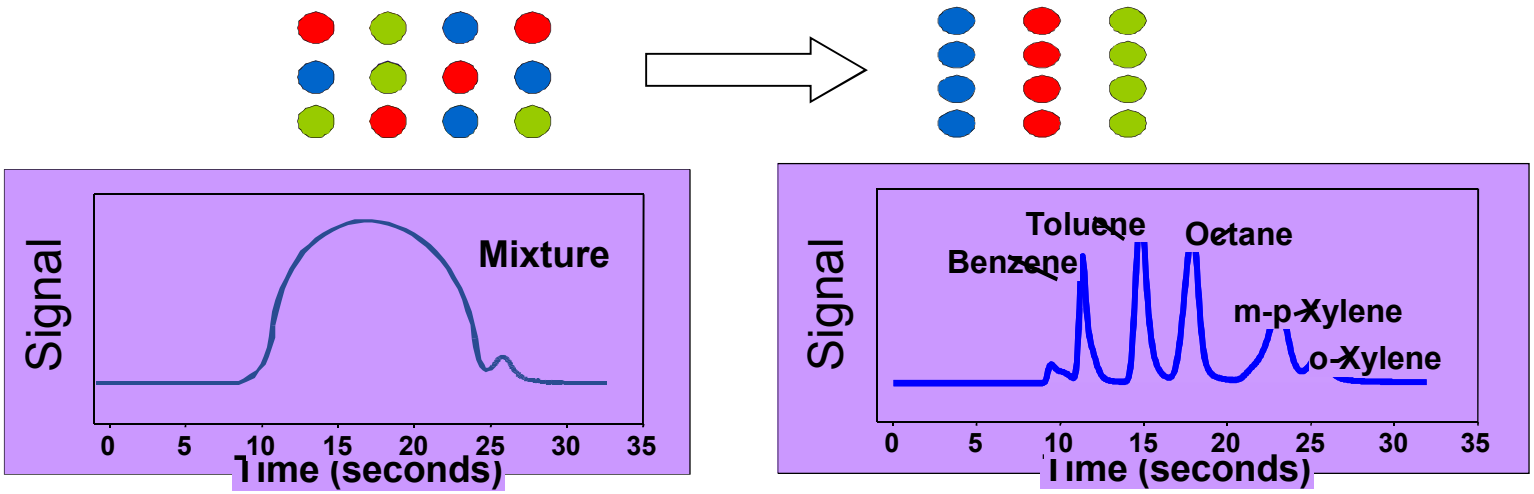
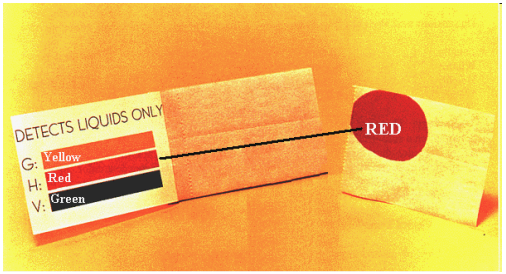
There are many ways to separate your analyte from other components of the sample



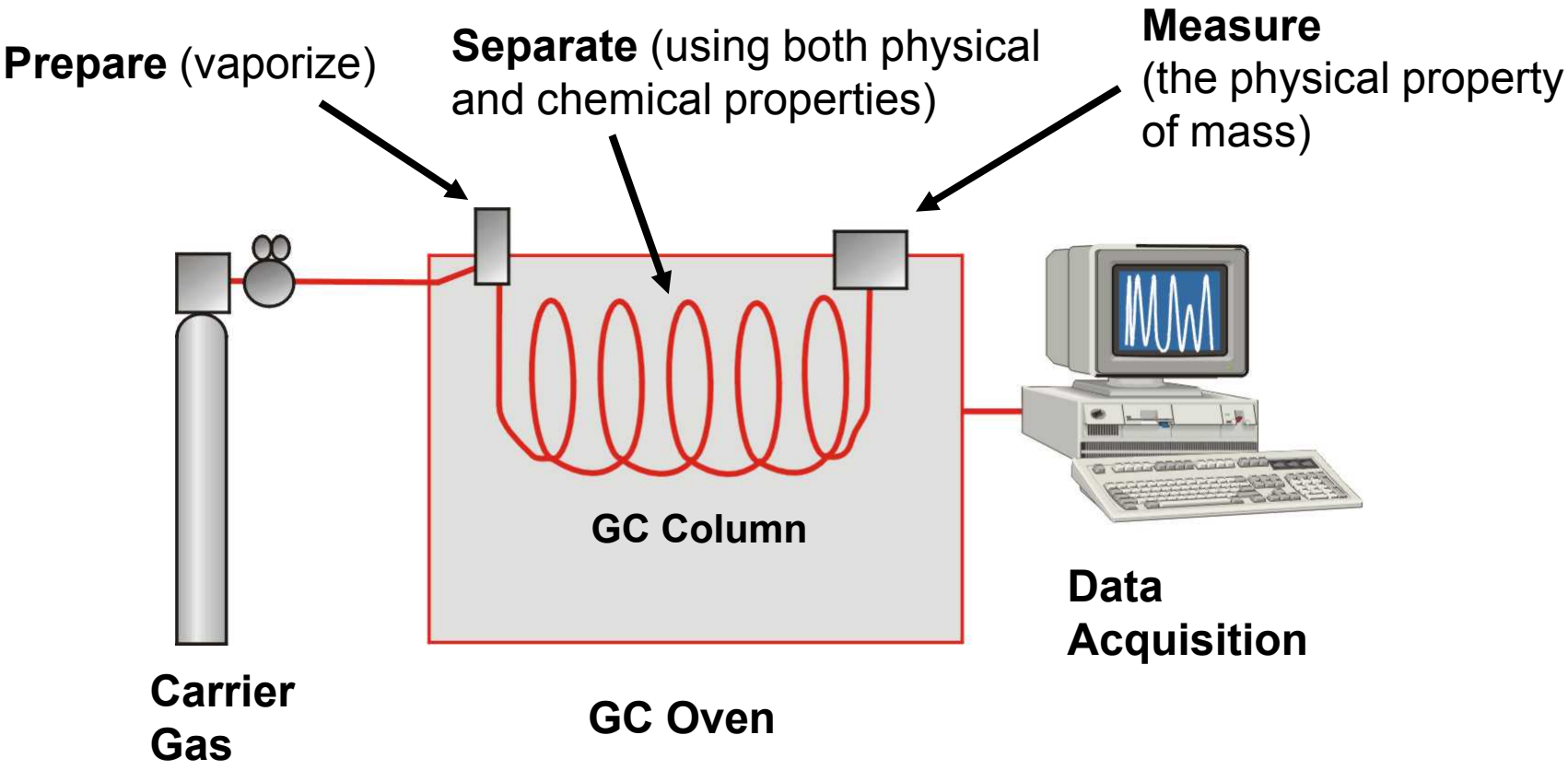
- **Improves detection confidence**
- **Can be performed during collection or even detection**
- **Capitalize on:**
  - Physical properties (b.p., m.p.)
  - Chemical properties (solubility, reactivity)

There are many ways to detect your analyte

- **Optical**
  - Absorption, emission, fluorescence
- **Mass / ion mobility**
  - Mass spectrometer, surface acoustic wave
  - Mobility spectrometer
- **Physical**
  - Chemical reaction
  - Indicator reaction

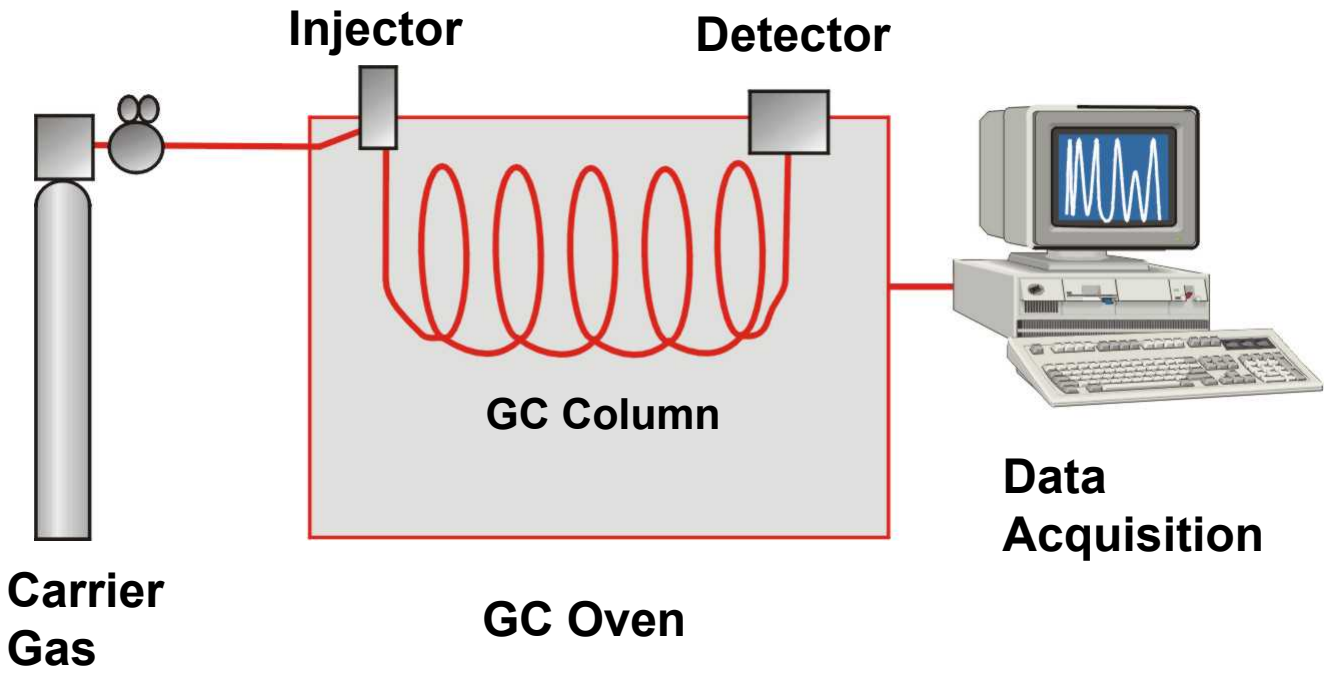


Measurements in this Course Follow the "Three Steps" of Analytical Chemistry



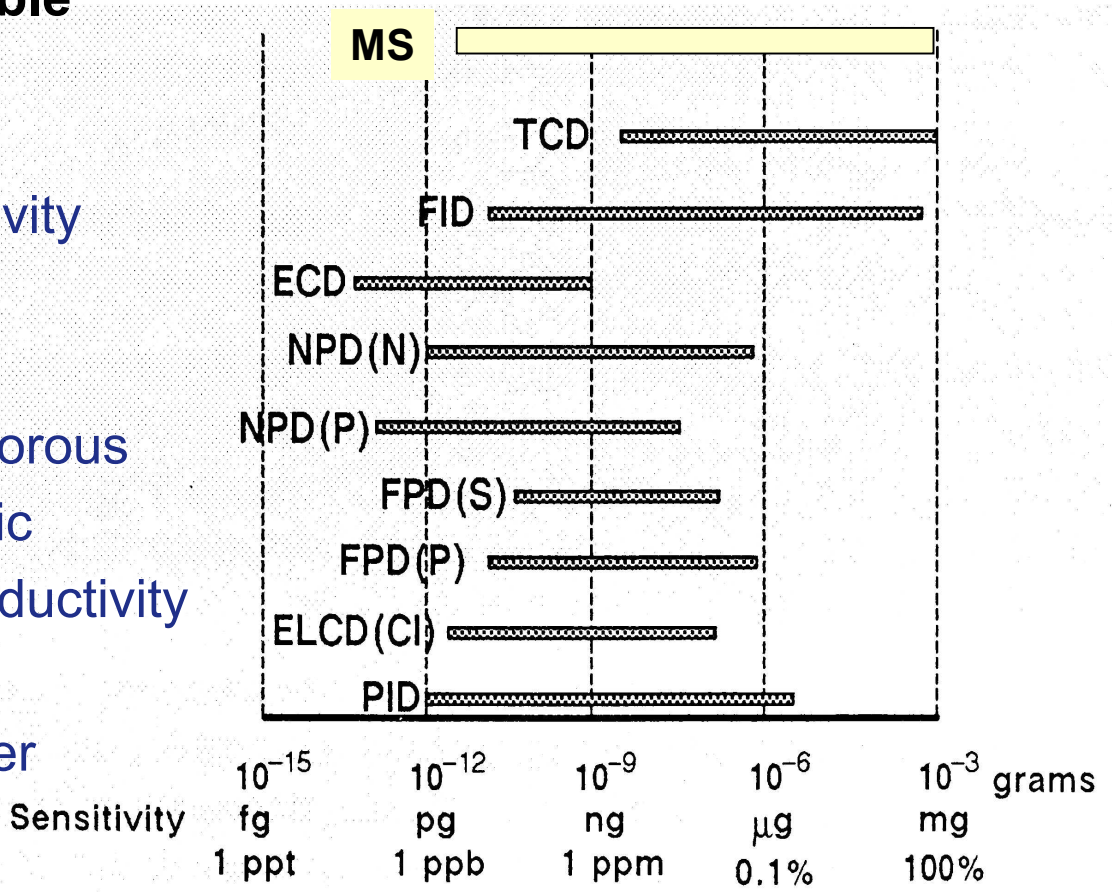
Our Measurements Utilize a Gas Chromatograph (GC) System

- **In addition to the components shown we need control of temperatures, gas flow and pressure.**



There are Many Options for Detecting a Gas Phase Molecule

- **Must have fast, predictable response to analyte**
- **Examples:**
  - TCD: thermal conductivity
  - FID: flame ionization
  - ECD: electron capture
  - NPD: nitrogen phosphorous
  - FPD: flame photometric
  - ELCD: electrolytic conductivity
  - PID: photoionization
  - MS: mass spectrometer



We choose MS….

Ref: Buffington and Wilson

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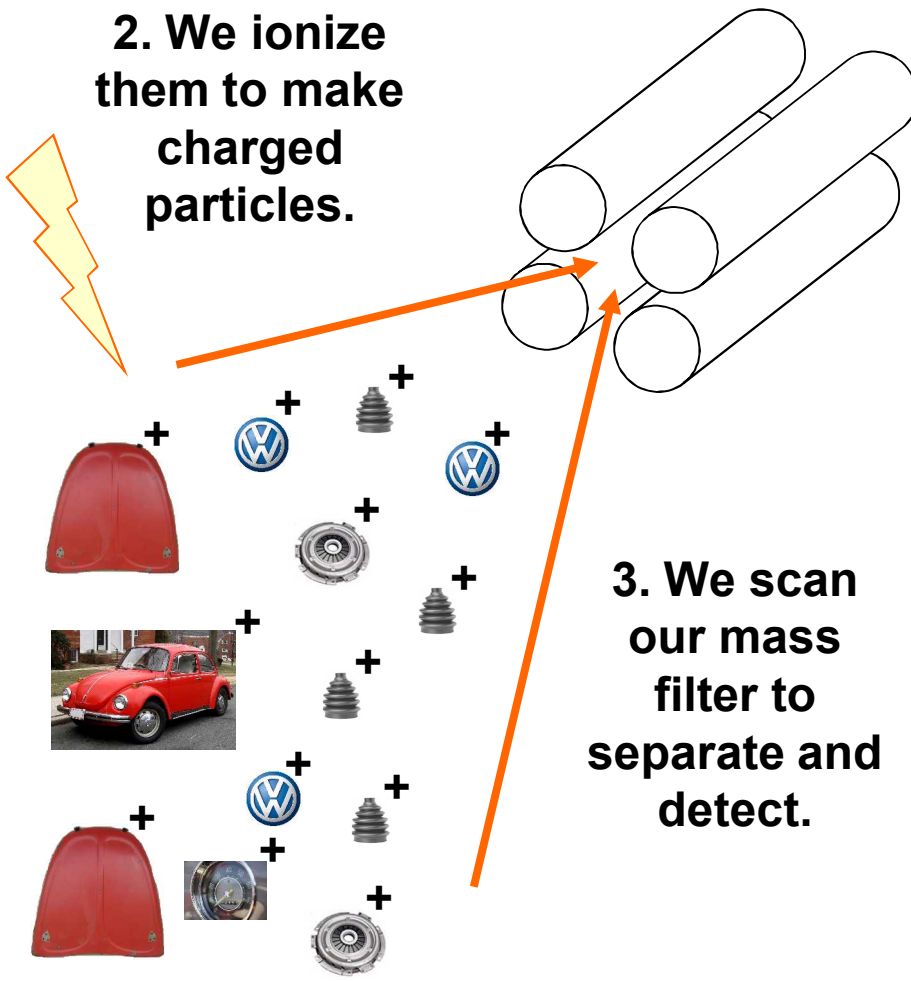
How Does Our Mass Spectrometer Work?

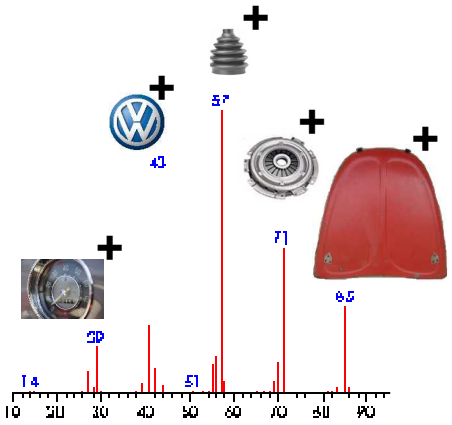
1. Molecules exit GC column.

2. We ionize them to make charged particles.



3. We scan our mass filter to separate and detect.

4. We gather and process the data for interpretation.



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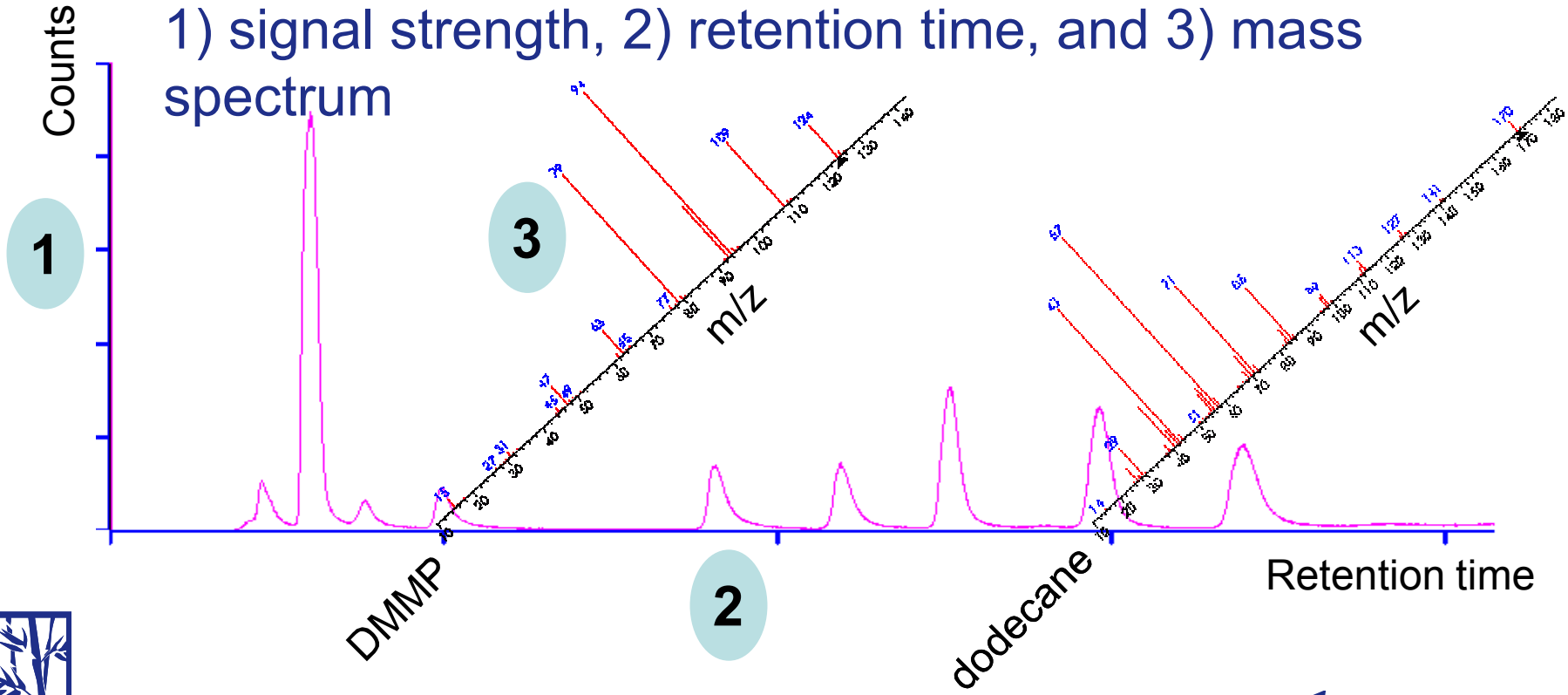
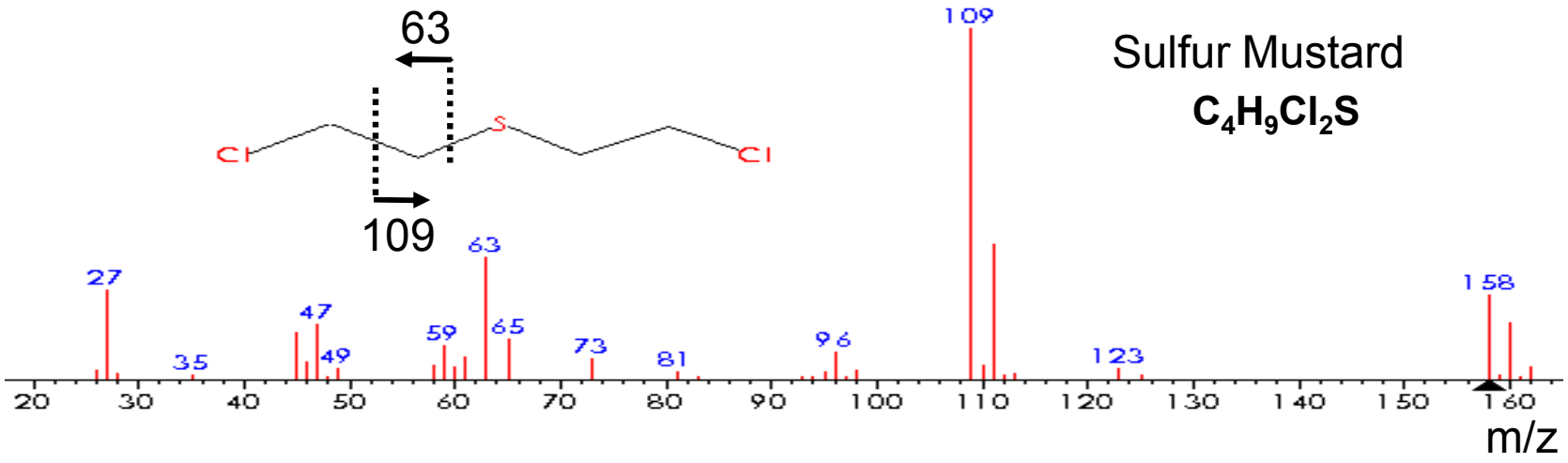
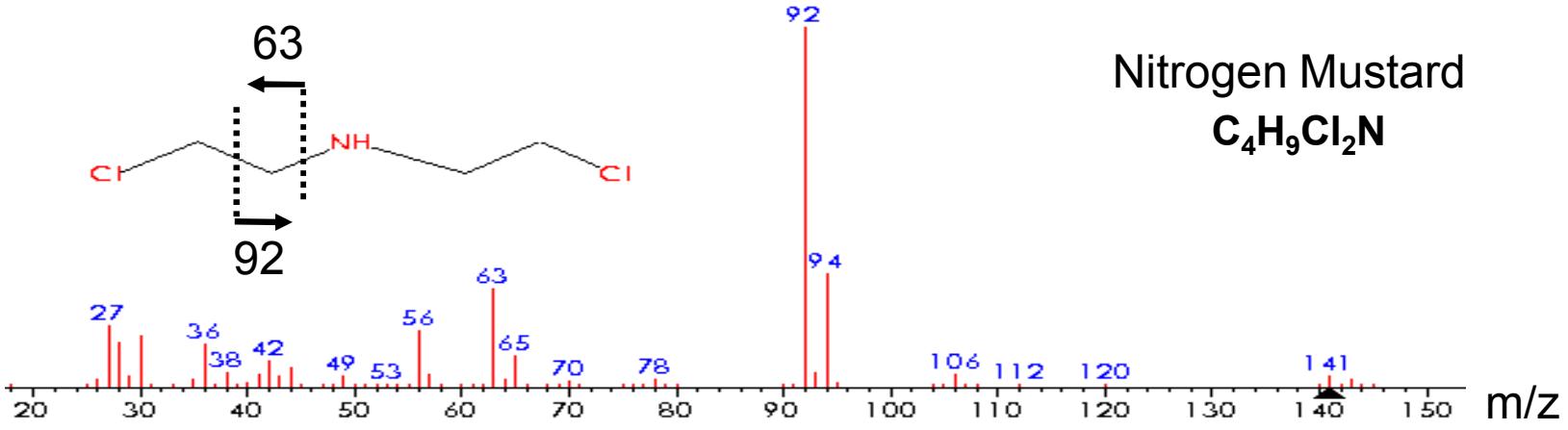
Why is Mass Spectrometry Often Called the "Gold Standard" of Measurements?

- **Its all about confidence**
  - The data is "3D" - each peak has 3 unique identifiers: 1) signal strength, 2) retention time, and 3) mass spectrum



We interpret patterns, isotope content, and relative signals to identify the analyte





Spectra from: NIST/EPA/NIH Mass Spectral Library ver. 2.0d

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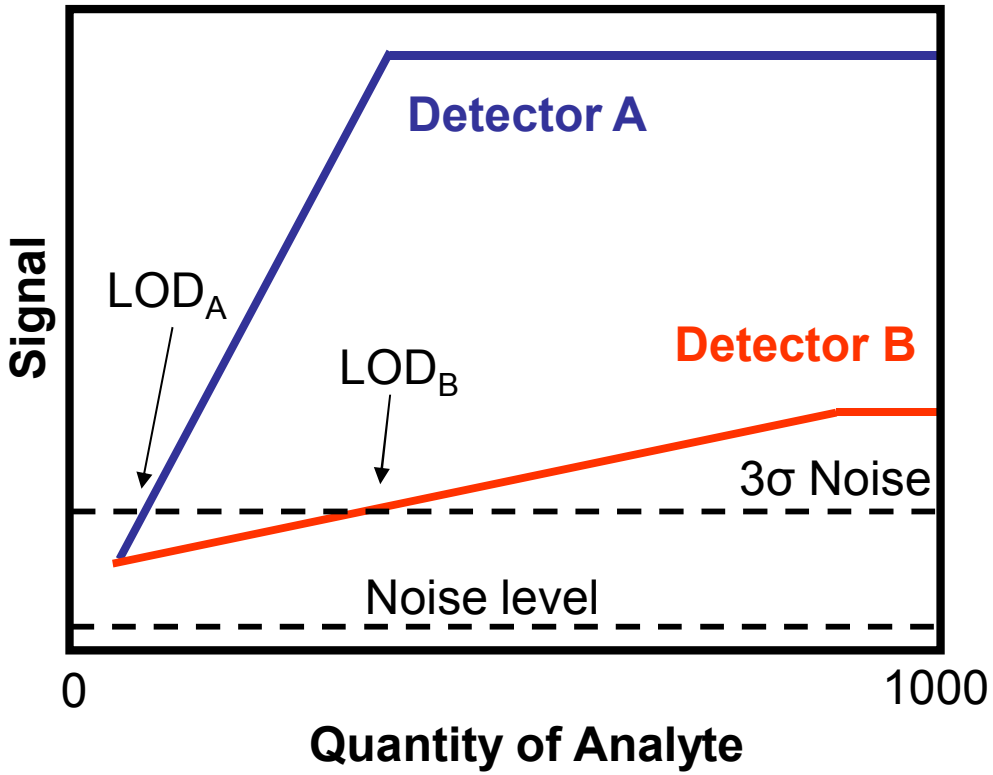
A Brief Graphical Summary of Terms for a Measurement System (includes all steps)

- **Sensitivity**
  - The slope of the response curve.
- **Limit of Detection (LOD)**
  - The lowest quantity that system can detect
- **Dynamic Range**
  - Useful signal range (before saturation)



Measurements and Hardware Summary

- **There are many options**
  - Measurement factors to consider:
    - **Speed, sensitivity, selectivity, dynamic range, analysis time, reproducibility**
    - **Sample complexity, physical state**
  - Instrumentation factors to consider:
    - **Cost, complexity, training, maintenance**
- **There is no single answer**

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