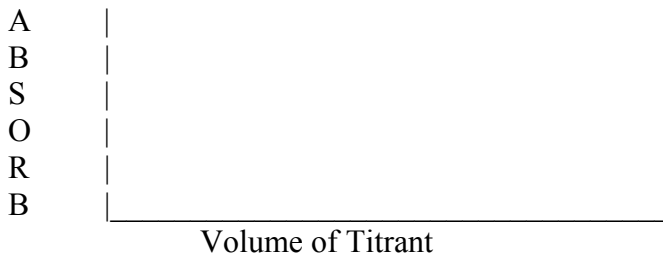


**ANALYTICAL CHEMISTRY, CHEM 465**  
**SPRING 2003, TEST 1**

Name \_\_\_\_\_

Short Answer: Be brief but thorough. (5 pts)

1. Distinguish between the three calibration methods: standard curve, standard addition and internal standard.
2. Why do quantitative and qualitative analysis often require different monochromator slit widths?
3. Explain why fluorescence measurements are usually more sensitive and more selective than absorption spectrophotometric measurements.
4. In a photometric titration the absorbance of the sample solution is monitored as titrant is added.  $\text{Titrant} + \text{Analyte} \rightarrow \text{Product}$ . Make a sketch of absorbance vs. volume of titrant for a titration in which the spectrometer is set to a wavelength where the molar absorptivity of the titrant is twice the molar absorptivity of the analyte. The product does not absorb at this wavelength.



5. Distinguish between atomic and molecular spectroscopy. How do the absorbance spectra differ?

**Problems: Show your work in a neat orderly step-by-step fashion. Include all units and maintain significant figures. Discussion: Read the question carefully. Use complete sentences and correct grammar. (15 pts each)**

1. Explain the processes of absorption, emission, and fluorescence of electromagnetic radiation on the molecular scale. Use an energy level diagram for your explanation. During absorption why are some wavelengths absorbed by a sample while others are transmitted through the sample?
  
2. Describe the differences between the following and list any particular advantages possessed by one over the other.
  - (a) filters and monochromators and wavelength selectors
  
  - (b) phototubes and photomultiplier tubes as detectors for electromagnetic radiation
  
  - (c) conventional (slewing instruments) and diode-array spectrometers for absorbance measurements
  
3. Draw a block diagram of a graphite furnace atomic absorption spectrometer. Label the major components. Describe the sequence of operation. Briefly describe the function of each component.
  
4. Draw block diagrams of the following two mass spectrometers. Label the major components. Describe the operation sequence for each spectrometer.
  - (a) quadropole
  
  - (b) time of flight

5. The following data were obtained for the calibration of an atomic absorption spectrometer for the determination of lead.

Concen. Pb (ppm)	# replicate measurements	mean signal response	standard deviation
0.000	12	0.052	0.0067
5.00	4	0.341	0.0082
10.00	4	0.585	0.0079
20.00	4	1.17	0.0089
sample	4	0.327	0.0085

Determine:

- the calibration sensitivity
- the detection limit in units of ppm
- the signal to noise ratio for the sample measurement
- the molar absorptivity at the wavelength used if the burner slot is 10 cm long
- the concentration of the lead in the sample