

Name: _____

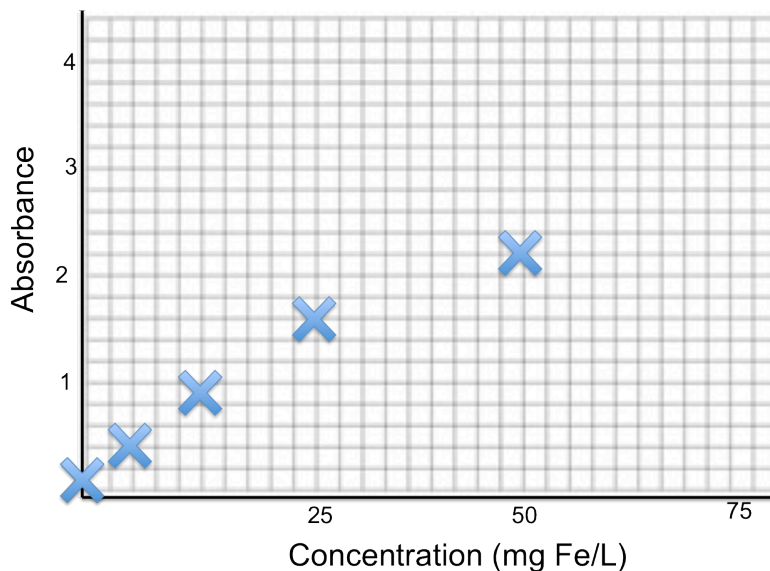
1. You are asked to measure the percent iron in a steel sample you believe to be about 50% Fe by mass. You dissolve four replicate samples in nitric acid and dilute them by mass to a concentration of 30 mg Fe L^{-1} for analysis by Flame AA. You begin by dissolving 1.0000 g of steel and dilute it to a final volume of 50 mL. Then you dilute an aliquot of your solution to a final volume of 100 mL for analysis.

(10 pts) Calculate the concentration of the initial solution and the volume of the aliquot required to make the final solution for analysis. BE SURE TO SHOW YOUR CALCULATIONS OR YOU WILL NOT RECEIVE CREDIT.

You also prepare calibration standards of 0, 5, 10, 25, and 50 mg Fe L^{-1} . You run Flame AA on your samples and standards. After the analysis, you plot your data (shown below). The average absorbance value of your samples is 2.6.

(2 pts) Draw the line of best fit through the data and estimate the concentration of Fe in your sample solution.

(3 pts) You know that it is a bad idea to extrapolate your calibration curve, so you quickly dilute a new high standard at 75 mg Fe L^{-1} and run it. The resulting absorbance measurement is 2.9. Add this value to your plot and estimate the concentration of Fe in your sample solution.



(5 pts) Describe your confidence in your measurement. If you are not 100% confident, describe what you believe to be wrong with reference to the appropriate information from class. List at least four reasons.

(5 pts) What aspects of experimental design were right in this experiment? List at least 3 things that were well-designed or turned out right.

(5 pts) If you are not confident in your measurement, list ways you would perform the measurement differently to give you more confidence. List at least 3 things you might change.

2. Consider random and systematic errors.
(3 pts) Define random error.

(2 pts) How can you quantify random error?

(3 pts) Give an example of how you would quantify random error in the experiment described in the first problem?

(3 pts) Define systematic error.

(2 pts) Compare the relative difficulty of detecting and quantifying random and systematic error.

(4 pts) List at least four ways you can assess your method for systematic error

3. (10 pts) Diagram an FT-IR instrument. Label each part and the function of each part.

(7 pts) Describe how FT-IR measurements are made by the instrument.

(3 pts) Draw an example interferogram. Be sure to label the axes.

(5 pts) Describe the information contained in the interferogram.

(5 pts) Describe how the interferogram is converted to the familiar output spectrum.

(3 pts) Draw an example output spectrum. Label the axes.

4. The following data are for a liquid chromatographic column:

length = 24.7 cm flow rate = 0.313 mL min⁻¹ V_m = 1.37 mL V_s = 0.164 mL

A chromatogram of a mixture of species A, B, C, D provided the following data:

| Species | retention time (min) | width of peak (FWHM; min) |
|----------------|----------------------|---------------------------|
| t _m | 3.1 | |
| A | 5.4 | 0.41 |
| B | 13.3 | 1.07 |
| C | 20.1 | 3.16 |
| D | 21.6 | 3.72 |

(2 pts) Calculate the amount of time species B spends in the solid phase.

(5 pts) Calculate the number of theoretical plates and plate height for Species A.

(3 pts) For species C and D, calculate the resolution. Is quantitative separation achieved?

5. (2 pts) List the four components of a mass spectrometer

(5 pts) Diagram a mass spectrometer, label the pieces and explain how they work.

(3 pts) Describe how each of the components listed above is accomplished in the diagram above.