

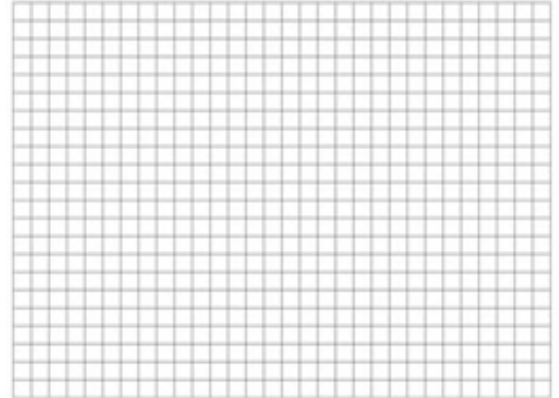
Calibration

Learning Objectives:

- Learn the relative merits of external calibration (by calibration curve or standard addition) and internal calibration.
- Identify and justify the best calibration method for a given application and be able to design a proper experiment.
- Be able to calculate the concentration of an unknown (and associated error) by each technique.

Calibration Curve

Draw an example graph of what data generated using each method would look like. Indicate on each graph where you would measure the concentration of an unknown. Be sure to label the axis.



State the equation for the line of best fit.

Describe the method of least squares analysis in words

Goodness of fit is frequently indicated using the R^2 value. What does this value mean?

State the equations needed to calculate the error associated with the concentration of an unknown.

State at least 5 requirements for a successful experiment using a calibration curve.

4-30. Using the linear calibration curve in Figure 4-13, find the quantity of unknown protein that gives a measured absorbance of 0.264 when a blank has an absorbance of 0.095.

Standard Addition

When is standard addition the preferred method?

Draw an example graph of what data generated using each method would look like. Indicate on each graph where you would measure the concentration of an unknown. Be sure to label the axis.

State the equation of a standard addition line of best fit.



State the equations needed to calculate the error associated with the concentration of an unknown.

Why is it desirable to the method of standard addition to add a small volume of concentrated standard rather than a large volume of dilute standard?

5-24. An unknown sample of Cu^{2+} gave an absorbance of 0.262 in an atomic absorption analysis. Then 1.00 mL of solution containing 100.0 ppm ($= \mu\text{g/mL}$) Cu^{2+} was mixed with 95.0 mL of unknown, and the mixture was diluted to 100.0 mL in a volumetric flask. The absorbance of the new solution was 0.500.

- (a) Denoting the initial, unknown concentration as $[\text{Cu}^{2+}]_i$, write an expression for the final concentration, $[\text{Cu}^{2+}]_f$, after dilution. Units of concentration are ppm.
- (b) In a similar manner, write the final concentration of added standard Cu^{2+} , designated as $[\text{S}]_f$.
- (c) Find $[\text{Cu}^{2+}]_i$ in the unknown.

Internal Standards

State when standard additions and internal standards, instead of a calibration curve, are desirable and why.

When preparing calibration standards for each of the following methods, which of the following are held constant?

1. Calibration Curve_____

2. Standard Addition_____

3. Internal Standard_____

A. final amount

B. amount of sample

C. amount of standard

D. amount diluent added

5-30. A solution containing 3.47 mM X (analyte) and 1.72 mM S (standard) gave peak areas of 3 473 and 10 222, respectively, in a chromatographic analysis. Then 1.00 mL of 8.47 mM S was added to 5.00 mL of unknown X, and the mixture was diluted to 10.0 mL. This solution gave peak areas of 5 428 and 4 431 for X and S, respectively.

- (a) Calculate the response factor for the analyte.
- (b) Find the concentration of S (mM) in the 10.0 mL of mixed solution.
- (c) Find the concentration of X (mM) in the 10.0 mL of mixed solution.
- (d) Find the concentration of X in the original unknown.