

CHEM312 Calibration Quiz No outside sources, no other excel files are allowed to be used; please show all work. Submit a completed spreadsheet file with worksheets for each of the 4 solved problems in Excel format with name.

Caffeine Analysis with Adenine Internal Standard

Internal standards are routinely used in analytical chemistry to measure and factor out systemic biases such as extraction efficiencies and recoveries. In chromatography, internal standard methodology provides a useful way to lessen sample injection volume errors. A GC method for caffeine analysis was developed using adenine as the internal standard. To measure the response factor, a standard analyte caffeine solution (1.0 mg/mL) was made by dissolving 25.0 mg of caffeine in 10 ml of methanol, adding this solution to a 25 mL volumetric flask, and diluting with methanol to volume. A separate internal standard solution of adenine (2.0 mg/mL) was made by dissolving 100 mg of adenine in 25 ml of DMSO, adding the solution a 50 mL volumetric flask and diluting to volume with DMSO.

1. Response Factor determination was made by combining 1.0 ml of the 1.0 mg/mL caffeine solution with 0.2 mL of the 2 mg/mL adenine internal standard solution. GC analysis of this mixture yielded peak areas of 15,867 and 10,456 for caffeine and adenine respectively.
 - a. Calculate the mg/mL concentrations of caffeine and adenine in the combined solution.
 - b. Showing all equations & their derivation, calculate the response factor F.
2. A subsequent sample analysis was conducted by combining 1.0 ml of a coffee extract with 0.2 mL of the adenine internal standard solution. GC analysis of this sample and internal standard mixture yielded peak areas of 7,644 and 9,865 for caffeine and adenine respectively.
 - a. Calculate the mg/mL concentration of the internal standard adenine in the combined sample and internal standard mixture.
 - b. Showing all equations and derivations, calculate the mg/mL concentration of caffeine in the combined solution.
 - c. Calculate the mg/mL concentration of caffeine in the coffee extract.

Calibration and Sum of Squares A set of lithium calibration standards yielded the following data:

| <u>Li Conc (ppm)</u> | <u>Line Intensity</u> |
|----------------------|-----------------------|
| 1 | 0.110 |
| 1 | 0.139 |
| 2 | 0.227 |
| 2 | 0.258 |
| 3 | 0.341 |
| 3 | 0.365 |
| 5 | 0.635 |
| 5 | 0.652 |
| 7 | 0.867 |
| 7 | 0.849 |

- a. For a first order fit of these data, identify the number of parameters, the replicate degrees of freedom, and the lack of fit degrees of freedom.
- b. Mean center the Li calibration data and conduct a first order fit; then calculate (showing all work and not using LINEST) the Total Sum of Squares, the Regression Sum of Squares, and the Residual Sum of Squares

Second Order Calibration Curve for Tap Water Pollutant

It is well known that, at moderately high absorbance values, there might be some non-linearity when using atomic absorption spectrometry. Because of this, the EPA-approved method for a certain pollutant found in tap water samples requires using a second-order polynomial calibration regression. Calibration and sample data are tabulated below.

| <u>Analyte Conc ($\mu\text{g/mL}$)</u> | <u>Absorbance</u> |
|---|-------------------|
| 3.45 | 0.162 |
| 6.41 | 0.283 |
| 10.03 | 0.391 |
| 15.11 | 0.531 |
| 18.92 | 0.587 |

| <u>Tap Water Sample</u> | <u>Absorbance</u> |
|-------------------------|-------------------|
| Unknown Conc | 0.347 |

- Conduct a second order regression of the calibration model, write out the specific equation developed, and comment on the fraction of variability accounted for by your model.
- Develop a calibration plot for these data using best practices established in this course.
- Calculate the pollutant $\mu\text{g/mL}$ concentration in the tap water sample.

Au Standard Addition Seawater Analysis

The gold content of a concentrated sea-water sample was determined by atomic absorption spectrometry using standard addition with the results in the table below.

| <u>Flask</u> | <u>ng Au added per mL of concentrated sample</u> | <u>Absorbance</u> |
|--------------|--|-------------------|
| 1 | 0 | 0.257 |
| 2 | 10 | 0.314 |
| 3 | 20 | 0.364 |
| 4 | 30 | 0.413 |
| 5 | 40 | 0.468 |
| 6 | 50 | 0.528 |
| 7 | 60 | 0.574 |
| 8 | 70 | 0.640 |

- Construct a clearly labeled standard addition calibration curve using these data.
- Estimate the concentration of the gold in the concentrated seawater.
- Outline very specifically how a set of these solutions would be prepared.
- Use propagation of error to calculate the uncertainty in your calculated Au concentration in units of ng/mL .