

Guided Inquiry for an Internal Standard Determination.

An internal standard is a compound which is different from the analyte that is added to an unknown sample. The chemical properties of the internal standard are similar to those of the analyte. However, the signals for each will occur at different wavelengths (for optical methods) or at different times (for chromatographic methods). The signal from the analyte is radioed with signal from the internal standard to quantify the analyte. An example of an internal standard/analyte pair is acetaminophen and caffeine (HPLC) or lithium and sodium in optical emission methods.

In the table we see data taken from the analysis of sodium using lithium as an internal standard.

Sodium Concentration, ppm	Sodium Emission Signal	Lithium Emission Signal	$\frac{\text{Sodium Signal}}{\text{Lithium Signal}}$
0.20	0.22	48	
0.50	0.53	47	
2.00	2.30	51	
5.00	5.00	46	

1. Transfer this data to a new EXCEL worksheet in a new workbook.
2. Use formulas to calculate the ratio of the sodium signal to the lithium signal. Show one example calculation here with units.
3. Plot this ratio versus the sodium concentration on a new worksheet. Add a trend line and write down the equation for the best fit line and the R^2 value. *This is an internal standard plot.* Make sure it labeled properly.

Other quantities from this regression are (you can easily calculate the others you need from your calculator or use the `linest()` function in EXCEL from Experiment #8)

$$s_y = 0.00087$$

$$S_{xx} = 14.4675$$

4. A measurement of an unknown gave a sodium signal of 0.88 and a lithium signal of 48, find the concentration and uncertainty of the unknown to the correct number of significant figures in the EXCEL worksheet. The equation for the uncertainty is provided. Replications should be set to one (1).

$$s_c = \frac{s_y}{|m|} \sqrt{\frac{1}{\text{Replications}} + \frac{1}{\text{Points}} + \frac{(\bar{y}_c - \bar{y})^2}{m^2 S_{xx}}}$$

5. Repeat this experiment, but plot JUST the sodium signal versus sodium concentration on a new worksheet. Add a trend line to get the slope, intercept and R^2 . Make sure the plot is appropriately labeled.

Other quantities from this regression line are (others you can calculate with your calculator)

$$s_y = 0.173879$$

$$S_{xx} = 14.4675$$

6. Using the same measurements for the unknown (sodium signal = 0.88; lithium signal = 48), find the concentration and uncertainty of the unknown to the correct number of significant figures about this regression. Note $k = 1$ here (# of replications) The equation for the uncertainty can be found above. Again, replications should be one.
7. Calculate the 95% confidence level the concentration determined from the internal standard plot and the external calibration plot. $\text{DOF} = \# \text{ Points in plot} - 2$.
8. Compare and contrast the concentrations and confidence limits in the two cases. What are your conclusions as to which method is superior – external type calibration or internal standard? Explain.
9. Let's say that the unknown you analyzed was made by adding 0.300 mL of toluene (internal standard), mixed with 15.00 mL of the original unknown and topped off with methanol in a 25 mL volumetric flask.
10. Using the concentration determined from the internal standard plot, use the dilution equation to find the concentration in the original 15.00 mL unknown (use dilution equation backwards). Show work and units here.
11. As with Experiment #8, find the uncertainty in the original unknown as well as the 95% confidence limit.