CH 2212 Lab Exploration
Standard Addition in Spectrophotometry

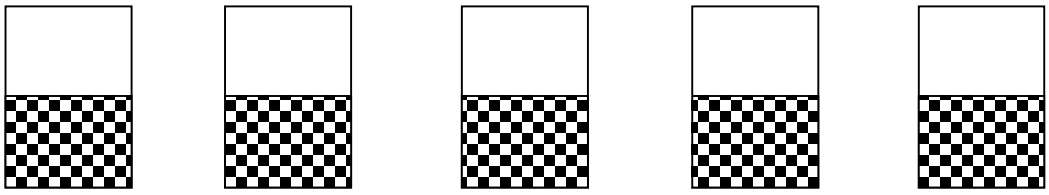
Now that you have successfully generated a linear calibration plot for MnO_4^- in the "Calibration Curves in Spectrophotometry" exploration, you have a good idea of the concentration range that will produce a linear response in the range of 0 – 1 absorbance units. You will use this information to choose initial dilutions for generating a multi-point standard additions plot. You will then use this method to determine the concentration of MnO_4^- in a solution supplied by your neighbor.

Harris has a brief introduction to the multi-point standard addition method on p. 88 of the Seventh Edition. Do <u>not</u> try and correlate Figs 5-4 and 5-5 – they are dealing with two entirely different approaches to standard additions.

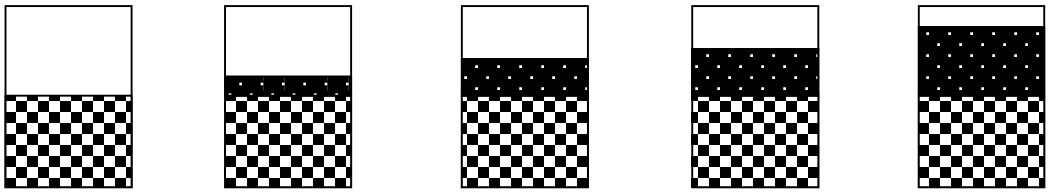
Basically, standard additions is set up in the following manner. Each box represents a volumetric flask. **NOTE: You only need one volumetric flask for this procedure. You prepare each solution in turn and then transfer the final diluted solution to a separate Erlenmeyer flask for temporary storage and analysis. The vol flask is rinsed and then the next solution is prepared.** However, for purposed of illustrating the procedure, multiple vol flasks will be represented.

The following procedure is for illustration only. Your volumes will change depending on how you set the standard additions procedure up.

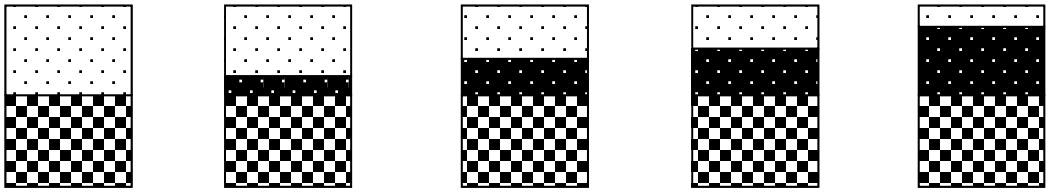
1) Quantitatively place 50 mL of the unknown solution in each of five 100-mL vol flasks.



2) Quantitatively add progressively larger amounts of a standard of known concentration.



3) Dilute each flask to the mark with distilled water. Mix thoroughly (19 inversions).



4) Determine the signal response (absorbance in our example) for each solution in turn.

5) Calculate the concentration of added standard for each flask as measured after dilution.

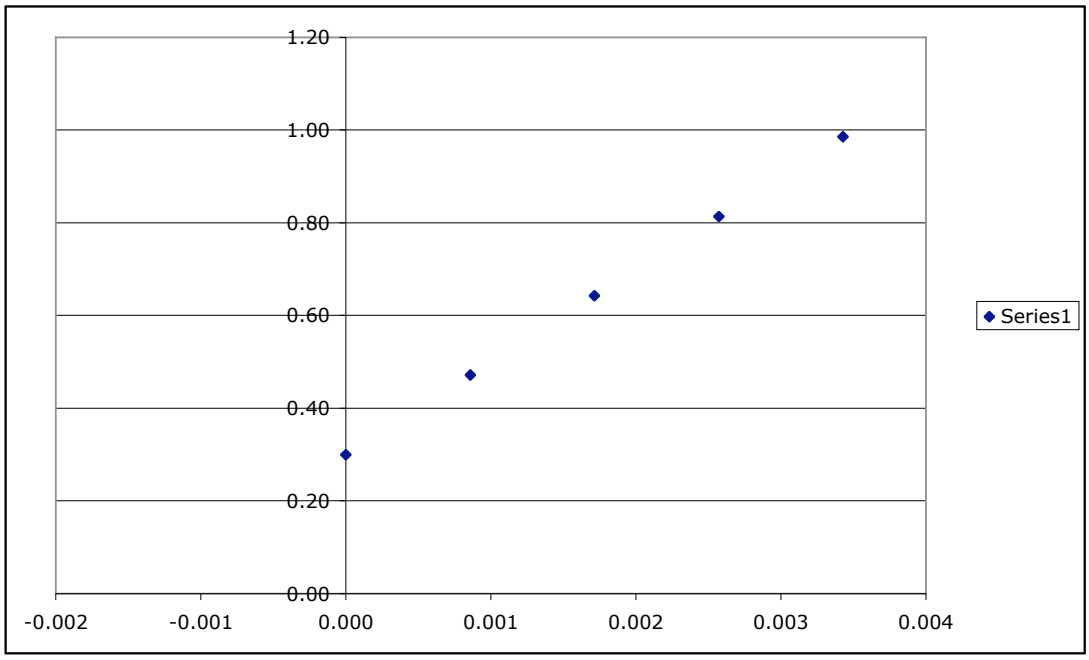
6) Plot absorbance vs. concentration of added standard. Clean up the axis numbers. Remove the colored (or gray) background.

Example of standard addition experiment

Adding successive volumes of beetlejuice standard solution to 50 mL of fruit extract

Final dilution is to 100 mL

beetlejuice std conc (M) =	8.57E-03		Vs = mL beetlejuice added	concentration of added standard	signal (AU)
Vo (mL) =	50				
Sample conc (used for calculating signal)	3.00E-03		0	0.00E+00	3.00E-01
			10	8.57E-04	4.71E-01
			20	1.71E-03	6.43E-01
beetlejuice absorptivity (used for calc signal)	200	Lmol-1cm-1	30	2.57E-03	8.14E-01
			40	3.43E-03	9.86E-01



7) Click on one of the data points. They will all be highlighted. Go to Chart, Add Trendline. Choose the linear trendline. Go to options and check "Display equation on chart."

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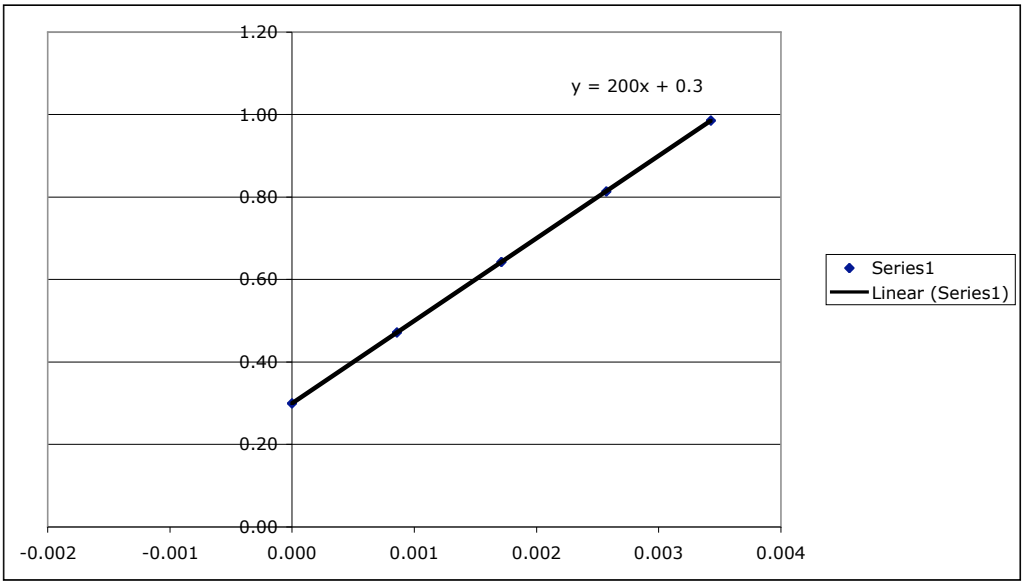

8) Double-click on the line. Choose Options, Forecast, Backward. Type in a good estimate of the distance (positive value only) from zero to where the line through your data points should intercept the x-axis. By trial and error, you should quickly get a very good approximation of this value. Note the value that you typed in.

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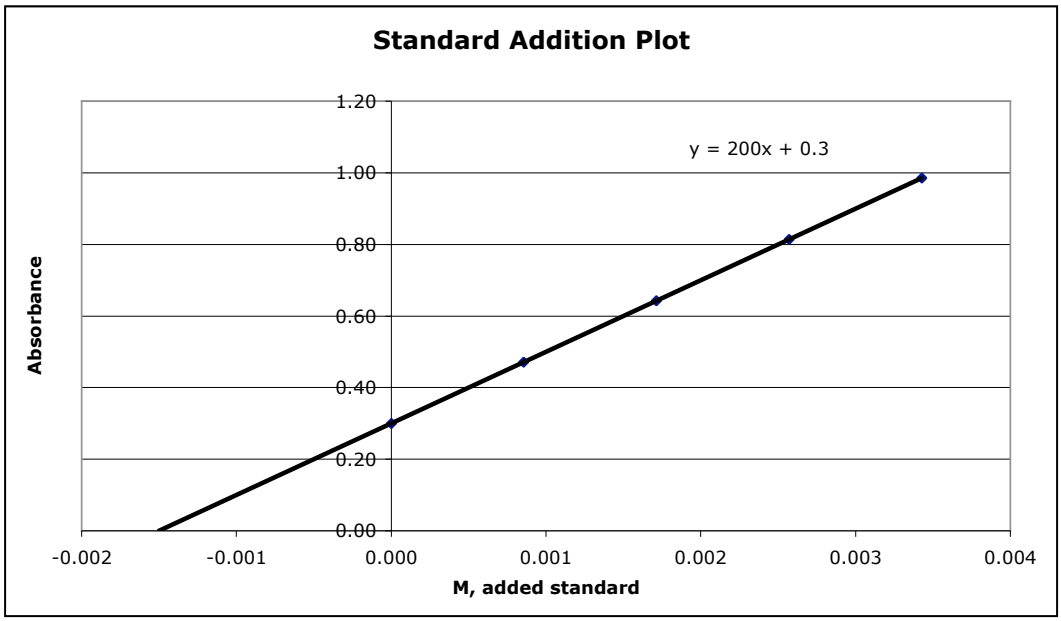

9) The value (0.0015 M) where the standard additions plot intersects the x-axis represents the concentration of beetlejuice (absolute value, of course) in the 100-mL vol flask. Remember, this sample was diluted by a factor of two (50 mL to 100 mL) so the actual concentration of the original sample is 0.0015 M x 2 = 0.003 M. And coincidentally, that is the exact concentration used in the spreadsheet to generate the absorbance data.

10) Clean up your plot. Add a title and axis labels of appropriate font size.

The lab exploration.

1) Design a standard additions analysis for MnO_4^- assuming your unknown will have an absorbance of approximately 0.6 AU **before dilution**. Use the information you have obtained from the MnO_4^- calibration plot previously obtained.

- Use a 100-mL vol flask.
- Use 50-mL sample volumes. This will provide an absorbance of about 0.3 AU for the sample with no added standard.
- Determine how many mL of one of your standard or intermediate dilutions will be needed to increase the absorbance of the diluted sample by about 0.15 AU. Add this volume (and its multiples) to the subsequent standard additions solutions.

2) Provide your neighbor with 500 mL of a solution of MnO_4^- of a molar concentration that will produce an absorption of 0.6 AU **without any dilution**. Do not disclose the concentration. Receive a like sample in return.

3) Follow your plan from 1) above. Generate a standard additions plot using Excel. Determine the concentration of your unknown. Remember to take into account the dilution of the original sample prior to analysis.

4) Record the molar concentration of your unknown sample on the board. Your neighbor will then disclose the value that they claim it is.

5) Repeat Step 5 after your neighbor has posted their result for the sample that you prepared.