Calcium Analysis by Atomic Absorption

In this experiment, your unknown calcium solution from last week's experiment will be analyzed for calcium by atomic absorption analysis and the results compared with those of the EDTA titration.

In atomic absorption, a solution containing the metal of interest is aspirated into a flame at a temperature of 2000 to 3000 ºC. The electrons in the metal (in this case calcium) are initially in their lowest energy state. Then a narrow beam of visible light is passed through the flame. The wavelength of the light is selected to correspond to an electron transition of that atom. Some of this radiation is absorbed by the metal causing electrons to move to a higher energy level. The amount of radiation absorbed can be related to the concentration of calcium ions in the solution using Beer's Law.

The light source for the atomic absorption apparatus is a hollow cathode lamp in which the cathode is made from the same metal that is being analyzed, in this case calcium. When heated, the calcium atoms are excited and their electrons go to higher energy levels. When the electrons fall back to lower levels, visible radiation is given off. The energy of the emitted photons corresponds to the energy difference of the Ca atom electron levels. We will be measuring the intensity of 423-nm radiation, which corresponds to a prominent Ca atom electron transition. The 423-nm radiation emitted by the Ca atoms in the hollow cathode lamp will be selectively absorbed by the Ca atoms in the solutions fed into the AA flame.

Using the standard calcium solution made last week; dilutions are made to prepare solutions of 1, 2, and 5 ppm of calcium. Each solution is aspirated into the flame of the AA spectrophotometer and its absorbance measured. From these data a calibration curve of absorbance versus ppm of calcium is plotted. The unknown calcium solution from last week's lab is diluted by a factor of 100 and then aspirated into the flame and its absorbance determined. The ppm of calcium in the diluted unknown solution is then read from the calibration curve. Then the concentration of calcium in the original unknown solution is calculated.

Procedure

The calcium solution prepared last week from 0.312 grams of calcium carbonate dissolved in HCl and diluted with water to a volume of 250.0 mL contains 1248 ppm of $CaCO_3$ or **500.0 ppm of calcium** ion. Two 100 mL volumetric flasks are needed to make three standard (5, 2, 1 ppm) calcium solutions and one diluted unknown calcium solution.

Pipet 10.00 mL of the **standard calcium (500.0 ppm) solution** in a 100-mL volumetric flask and fill to the mark with distilled water and mix well. Label it as **50.0 ppm** calcium solution.

5 ppm calcium: Pipet 10.0 mL of the **50.0 ppm calcium solution** into a 100 mL volumetric flask and dilute to the mark with distilled water. Mix well and pour about 50 mL into a clean, dry 100 ml flask or beaker and label it **5 ppm calcium**. Dispose of the remaining solution in the volumetric flask.

2 ppm calcium: Rinse the 100 mL volumetric flask well with distilled water. Now pipet 4 mL of the **50.0 ppm calcium solution** into the volumetric flask, dilute to the mark and mix well. Pour about 50 mL into a clean, dry container and label it as **2 ppm calcium**. Dispose of the remaining solution in the volumetric flask.

1 ppm calcium: Rinse the 100 mL volumetric flask well with distilled water. Pipet 1 mL of the **50.0 ppm calcium solution**, fill to the mark with distilled water mix well and pour 50 mL into a clean, dry container. Label this as **1 ppm calcium solution**. Dispose of the remaining solution in the flask.

Unknown solution: Rinse the 100 mL volumetric flask well with distilled water and pipet 1.00 mL of last week's **unknown calcium** solution into it. Dilute to the mark with water. Mix the solution well after dilution. Label this flask as **unknown**.

AA Analysis of the Four Solutions

Before aspiration of any sample into the AA flame, **be sure to auto zero (AZ button) on the AA spectrometer while the capillary tube is in distilled water.**

1. **Put the aspiration capillary tube in distilled water and press the AZ (auto zero) button.** This sets the instrument to zero absorbance with no calcium in the flame

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2. **Put the aspiration capillary tube in a sample. When the lamp energy reading stabilizes**, **press the READ button and read the absorbance on the display screen.**
3. **Repeat steps 1 and 2 above for each standard and the unknown solution.**

Aspirate each of the three diluted standards into the flame and record the absorbance. For the calibration curve plot a graph of absorbance versus ppm calcium ion of the three standards and evaluate the slope of the straight line. The straight line should go close to the origin, since zero calcium concentration should absorb no 423-nm radiation.

Aspirate your diluted unknown solution into the flame and record its absorbance. Determine the ppm concentration of calcium in the diluted and original unknown solution from the calibration curve.

Name: ___________________________

Data Sheet

Concentration of Calcium in the Standard Solutions (ppm)	**Measured Absorbance**
0.000	0.000
1.00	
2.00	
5.00	
//	//
Diluted Unknown Solution from the Last Lab Unknown Number:______________________	

Calculations and Report

1. For the calibration curve, make a plot of absorbance vs. ppm calcium for the 1.00, 2.00 and 5.00 calcium standards. Attach your graph to this report. Be sure to include zero ppm Ca and zero absorbance as part of the data. Use Excel or Graphical Analysis to plot the graph.

2. Calculate the slope of the calibration curve. Show your setup.
   Slope = (Change in Absorbance)/(change in concentration of Ca^{+2} in ppm)

3. What is the absorbance of the diluted unknown solution? _____________________

4. What is the ppm of calcium in the diluted unknown solution?
   Ppm Ca = Absorbance/slope of calibration curve

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5. What is the ppm of calcium in the original unknown solution ?
$(conc)_i \times (vol)_i = (conc)_f \times (vol)_f$

6. What was the ppm of $CaCO_3$ in the unknown from last week's EDTA titration ?

7. What is the ppm of calcium ion in your unknown from last week's EDTA titration?
ppm Ca^{+2} = (ppm $CaCO_3$) x (formula mass of Ca^{+2})/(formula mass of $CaCO_3$)

8. Compare the ppm of calcium from the AA analysis with the EDTA titration. Which method do you think is better and why?

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Prestudy

1. 0.156 grams of $CaCO_3$ is dissolved in aqueous HC1 and diluted to 250.00 mL in a volumetric flask. What is the ppm, mg Ca^{+2}/L, of calcium ion in the final solution?

2. A 10.0-mL volume of the above solution (C_1) is diluted to 100.0 mL in a volumetric flask to give C_2. A 5.00-mL aliquot of this solution (C_2) is diluted to 100.0 ml to give C_3 . What is the ppm of Ca^{+2} in all **three solutions**?

3. The slope of the calibration curve for a series of standard calcium solutions is 0.300/ppm Ca^{+2}. A 10.0-mL aliquot of an unknown solution (C_1) is diluted to 100.0 mL in a volumetric flask to give C_2. A 10.0-mL aliquot of this solution (C_2) is diluted to 100.0 mL to give C_3. The absorbance of this final solution (C_3) is found to be 0.155. What is the ppm of calcium ion in all **three solutions**?