Experiment 7: Interferences in Atomic Absorption Spectroscopy

Introduction:

Atomic Absorption and Atomic Emission Spectroscopies are quantitative techniques for determining elemental abundance based on either the absorption or emission of specific wavelengths of UV or visible light corresponding to the unique electronic transitions in the element. As a result of the high heat used in the technique, molecular and ionic species are vaporized and atomized, so that the spectra of individual atoms may be recorded. In absorbance mode, a lamp containing the element to be analyzed provides a spectrum of wavelengths specific to the element, while in emission mode, the elements to be analyzed are thermally energized, causing them to emit their respective spectra. Thermal atomization and excitation are accomplished in one of two ways: 1) by introducing a liquid sample into a flame, or 2) rapidly heating the sample in a small graphite tube furnace. Quantification is possible via variants of Beer’s law and standard curves derived therefrom:

- For Absorption: \( A = k \times C \)
- For Emission: \( I_e = k \times C \)

The variable 'k' encompasses a number of experimental and elementally-specific parameters which must be kept constant for the standard solutions used to make the standard curve as well as the sample. In fact, a number of factors may make it difficult to keep these parameters constant between standards and the sample—perhaps the most troublesome of which are chemical interferences. Bearing in mind that, in order to obtain a good signal from the analyte, the element of interest must be vaporized and converted to the atomic (un-ionized state), several types of interferences are possible:

1. Chemical species present in the sample form very high-boiling point compounds with the element, preventing it from being fully vaporized.
2. Chemical species present in the sample prevent the reduction of the element to its atomic state.
3. The viscosity of the solution prevents it from being well nebulized.

In this lab, we will explore several chemical species that may interfere in the analysis of calcium by flame atomic absorption, or may, in some cases, prevent interference.

Equipment: Read through the procedures and make a list of the equipment you will need.

Solutions: You will be provided with the following solutions to be used in making up samples for analysis:

- 100 ppm Ca Stock solution
- 0.01 M H₃PO₄
- 0.01 M AlCl₃
- 0.01 M KCl
- 0.05 M La₂O₃
- 95% Ethanol

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Safety Considerations: Read through the procedures and note any safety considerations.

Procedure:
I. Each group will be assigned to make solutions of Ca including one of the following: \( \text{H}_3\text{PO}_4 \), \( \text{AlCl}_3 \), \( \text{KCl} \), or ethanol. Make the ten solutions as instructed below. After making each, pour it into a labeled beaker or Erlenmeyer flask and rinse the volumetric flask for reuse.
   A. Use the 0.01 M prepared solution of your compound or the 95% ethanol to make 5 solutions (using a 50 mL volumetric flask) containing:
      i. 10 mL of Ca Stock Solution (100 ppm), measured out with a 10 mL volumetric pipette
      ii. 1, 2, 5, 10, or 20 mL of the 0.01M compound solution or 95% ethanol measured with a graduated cylinder
      iii. enough water to make up 50 mL
   B. Use the 0.01 M prepared solution of your compound and the 0.05M \( \text{La}_2\text{O}_3 \) to make 5 solutions (using a 50 mL volumetric flask) containing:
      i. 10 mL of Ca Stock Solution (100 ppm), measured out with a 10 mL volumetric pipette
      ii. 20 mL of the 0.01M compound solution or 95% ethanol, measured with a volumetric pipette
      iii. 1, 2, 5, 10, or 20 mL of 0.05M \( \text{La}_2\text{O}_3 \) solution, measured with a graduated cylinder
      iv. enough water to make up 50 mL
II. Transfer 10 mL of Ca Stock Solution (100 ppm) to a 50 mL volumetric flask with a volumetric pipette. Fill to the line with water. You may leave this solution in the flask.
III. Take your 11 solutions to the Atomic Absorption Spectrometer and measure the absorbance of each:
   A. The spectrometer should have been turned, including the Ca-lamp to be used, to warm up for 20 minutes.
   B. Turn on the flame and zero the instrument using distilled water.
   C. Using a 50 ppm Ca solution that will be prepared by your instructor, adjust the fuel mix and burner height / position to obtain the maximum absorption.
   D. Aspirate each of your 11 solutions in the following order, recording the absorbance of each:
      i. 20 ppm Ca solution
      ii. 20 ppm Ca solutions with your assigned compound from lowest to highest concentration
      iii. 20 ppm Ca solutions with 20 mL of your assigned compound and \( \text{La}_2\text{O}_3 \) from lowest to highest concentration
IV. Calculate the percent change (%), either positive or negative, for each of your 10 "compound" solutions versus the 20 ppm Ca solution. Record these percent differences and volumes of compounds added in a table on the blackboard.
V. Record every groups' data before leaving lab.
Analysis

1. Using the class' data, make plots of %Change in Absorbance vs. Volume of Compound Solution added for all solutions run (8 graphs in all). Be sure to descriptively label your graphs, including axes.

Conclusions

1. How do each of the additives, (H₃PO₄, AlCl₃, KCl, La₂O₃, and ethanol) influence the experimental determination of calcium? Discuss trends in the graphs you created in Analysis. Are there any concentrations of the additive where the influence reaches a maximum and plateaus?

2. Assume you have a sample you need to analyze for calcium, and you believe it contains one or more of the interfering species. Propose a way of making up the sample and the standards that would minimize interferences and maximize the signal of calcium in a flame Atomic Absorption Spectrometer. Be as specific as possible; from the data collected in class, can you determine specific concentrations of any species that will be most effective at reducing interferences