DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION
PE AAnalyst-100

Basis of the experiment

Flame atom absorption is used to determine the concentration of magnesium in different solutions. The effects of instrumental parameters (slit width and monochromator setting), flame conditions (fuel to oxygen ratio) and matrix substituents are examined.

Equipment

PE AAnalyst-100 Atomic Absorption Spectrophotometer, Mg hollow cathode lamp.

Reagents & Solutions

1. Atomic absorption standard solutions of $MgNO_3$, NaCl, and KCl are available in A207. Ethanol is also available in A207. Urine samples will be supplied by the student.

2. Prepare 100.0 ml of 100.0 ppm Mg stock solutions from a 1000 ppm stock available in the lab. (Please do not pipette from the reagent bottle – pour an appropriate an appropriate amount into a beaker for this)

3. From your stock solution prepare 500 ml of a 1.00 ppm Mg standard solution and 50.0 ml of the following Mg standard solutions: 0.050, 0.075, 0.100, 0.150, 0.200, 0.250, 0.500 ppm Mg.

4. Prepare three additional 1.0 ppm Mg standards: one with 200 ppm Na (using the 1000 ppm NaCl available in A207) one with 200 ppm K (using the 1000 ppm KCl available in A207), and one in 50/50 (V/V) ethanol/H_2O. Also prepare about 25 ml of a 50/50-ethanol/H_20 blank.

Waste: Sink, except urines into toilet.

Procedure

Special Precautions. Background information on AA instrumentation, theory, and techniques may be found in Skoog & Leary, or any advanced instrumental text. Before beginning this experiment, please read the following carefully.

1. Never exceed the maximum current rating of the hollow cathode. This rating is checked during the instrument set-up procedure.

2. Always turn the air on first, establish the flow, then turn on the acetylene, establish this flow and ignite the burner. To shut down the burner, reverse this operation. This instrument does this under computer control.

3. If the Mg lamp is in place, assume the burner alignment is correct. If not, do not attempt to move the burner head without first checking with the instructor.

4. If the flame wavers due to drafts in the room, very sporadic data will be obtained. If such a condition exists be sure to block out any drafts and protect the burner from these fluctuations.

5. A clogged burner slot is indicated by an uneven flame. This can be corrected by carefully scraping the burner slot with a spatula or razor blade while running air-only through the burner. Check with your instructor if you suspect that there is a problem like this.

6. Never aspirate a sample with the burner off and **always aspirate distilled water after each sample aspiration**. When aspirating a variety of samples be sure to rinse the polyethylene tubing between each aspiration with distilled water from a wash bottle and carefully wipe the tubing dry with kimwipes.

PE AAnalyst-100 Instrument Operating Procedures. Please read these instructions completely before operating the instrument or attempting any of the procedures outlined below.

1. Sign the Log Book.

2. Turn on the instrument, using the Power switch. (This is on the right side next to the power cable input)

3. Turn on the computer and printer. The user name and password are both *student*. When you have your desk top displayed start the AA program from the AA WinLab Analyst icon. From the WinLab introduction screen select the "basic system" icon. This will bring you to the "Manual Analysis" window from which you will collect the data for this lab.

4. Select your method from the top menu bar and select the method SCHA318. You will find most of your parameters set as defaults. This instrument will do an automatic optimization each time the method is closed. Also note that F1 will bring you the appropriate Help for the window that is currently active.

5. Turn on the gas valves, for air and acetylene, on the top of both the tanks.

6. Access the Flame window from the top menu bar. A switch icon in this window will start gas flow and light you burner flame when clicked.

7. Select 'continuous graphics' under the tools menu to get a window that shows the lamp signal. Start aspirating your 1.00-ppm standard. Adjust the absorbance reading to a maximum value by moving the burner assembly up and down and in and out.

Preparation of the Calibration Curve

1. Change back to the distilled water blank. Recheck you method to insure that the standards in the method table are entered in for the correct amounts for you work. If you make changes you might need to exit that window to have the changes take effect.

2. Return to the "manual analysis" mode and you will observe that there are icons for blank, standards and samples.

3. Move the aspirator hose to the "blank" solution, wait a few seconds, and click the blank button. The method should be configured to collect at least triplicates. When data is collected the green on the button will go out.

4. Aspirate Standard #1, wait a few seconds, and press the standard button. The standard number will update when the data is collected.

5. Repeat step 4 until standards are done (aspirating distilled water between and after each reading). Check to see that the readings make sense. Don't forget to use the 1.0-ppm solution. You may see your standard curve by clicking the Calib Icon in the top bar. Total results can be viewed by clicking the Results icon.

Analysis of Samples

1. AA is a method that is susceptible to drift so you should have all your solutions prepared prior to data collection. All remaining solutions should be run as samples. As a matter of Good Laboratory Practice you should make sure to appropriately label each sample in the software window prior to data collection.

2. Provide clean 50 ml volumetric flasks with stoppers (one flask for each person), a pipetter, and your stock solution to your instructor to obtain your unknown. Determine the concentration of your unknown. You may need to dilute your unknown to have its absorbance fall within the limits of your calibration curve.

3. Test a urine sample from each lab partner for Mg (the daily output of Mg for a normal individual is 100 to 300 mg). Potassium and sodium are present in relatively high concentrations in urine. Check for possible interference by these ions by measuring the 1-ppm Mg standards containing 200 ppm of Na and K respectively. Don't forget to check the zero and 1 ppm Mg standards again, too (measured as samples). Report your results in terms of both the amount of Mg in the samples and total daily output (estimated based on daily urine output of 2000 mL). Note: The urine sample should probably be diluted on the order of 1:100 or 1:200 with distilled water.

5. Measure the absorbance of the Mg in the ethanol/H_2O. Use the ethanol/H_20 blank to read a baseline value.

6. Test the effect of slit width upon the Absorbance detected by changing the slit value in the method set up window. Measure you signal while aspirating the 1 ppm Mg sample. Explain your results. Different types of flames (rich, lean, hot, etc.) may be superior for a particular analysis.

7. We wish to investigate effect of small changes in the flame on Mg analyses. Measure the 1 ppm Mg solution at settings 1.0, 1.5, and 2.0 on the instrument's fuel scale. Do this first without checking the zero and again checking the zero between each fuel setting by aspirating distilled water.

8. Due to the nature of AA the monochrometer λ setting can be very crucial for obtaining high quality data. In older and many current instruments this is an important parameter and must be set correctly. This instrument automatically tunes to the line that is encoded on the hollow cathode lamp. To observe why proper λ selection is important you will observe the lamp output. With the flame off (see instructions in the shut down procedure) go to the 'tools' menu and select 'Wavelength Scan' (this feature allows you to see the output of the hollow cathode lamp as a function of wavelength). Click 'Scan' and print the results. Next change the number of points per nm to 100 and re-scan. Plot these results also. What is the difference between these plots? Which is better at determining the maximum output wavelength? Next scan the wavelength range from 190 to 210 nm with 10 points per nm. Plot these results. Lastly select the wavelength range of 200 to 400 nm with auto points per nm and scan. Observe the spectrum as it is plotted. The instrument will re-scale the output to accommodate the larger peaks. Plot this result. The instrument suggests only two wavelengths, 202 (small peak) and 285 (large peak) for data collection. Discuss in your report why one of the other strong lines from this lamp is not used.

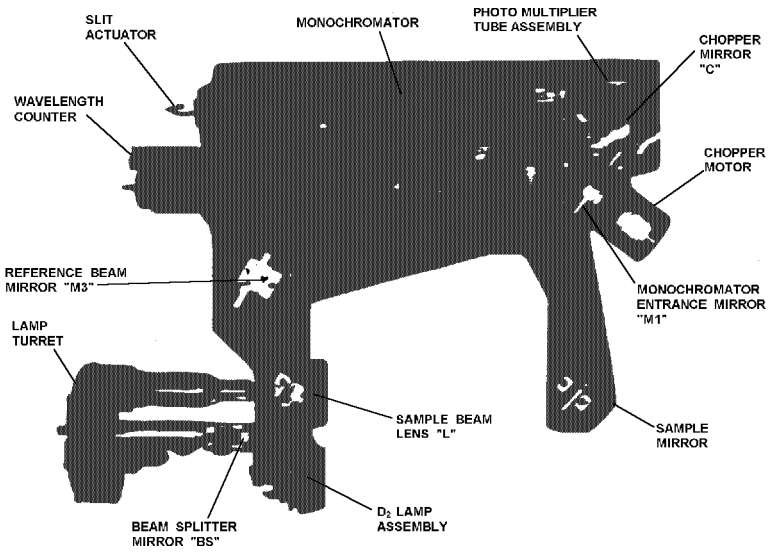
Instrument Shut-down Sequence

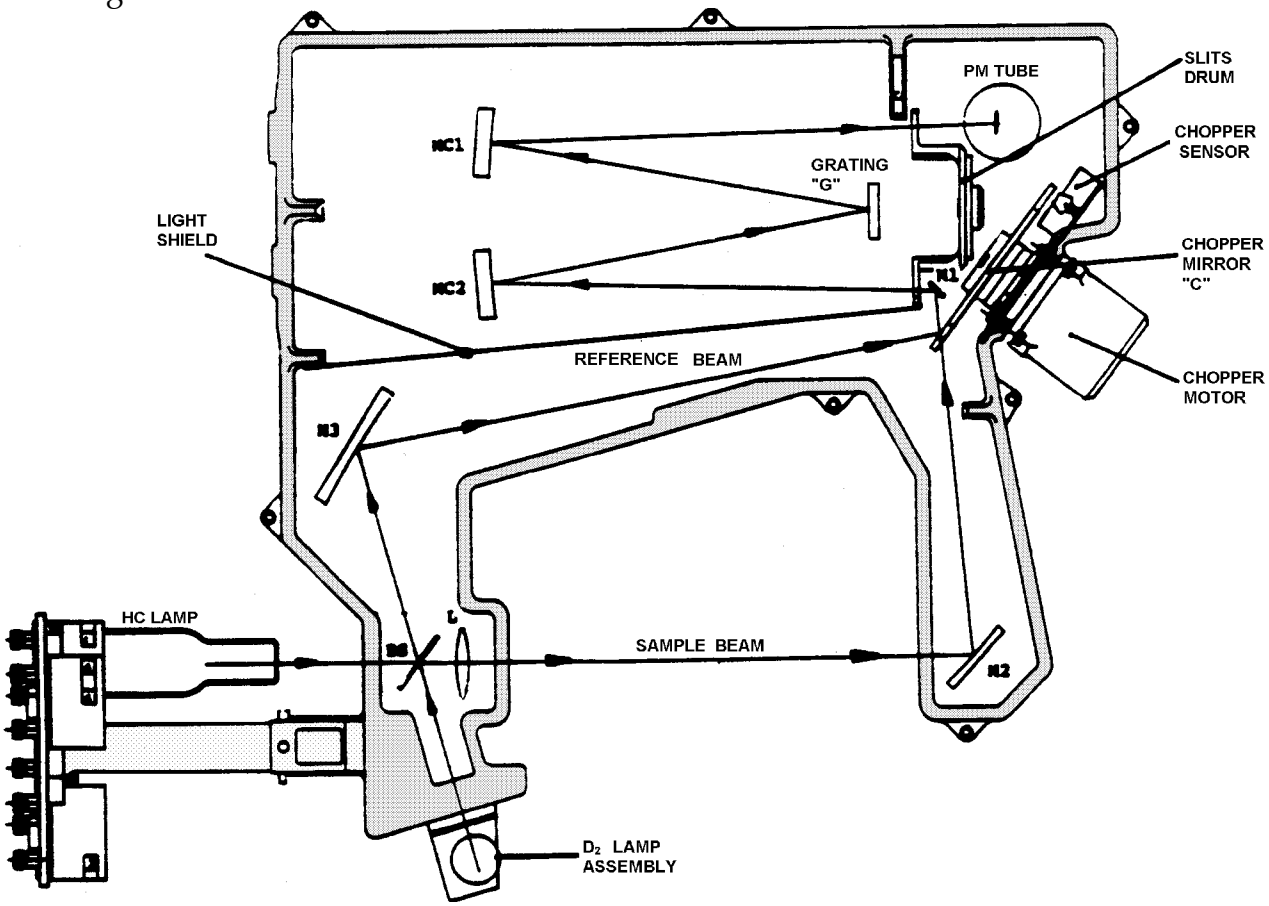
1. Aspirate air when done

2. Go to the flame window and click off. Turn the main gas tank valves off and bleed the system by hitting the bleed button. Notify the instructor if the acetylene pressure is less than 120 lb/in^2 or if the air pressure is less than 1000 lb/in^2.

5. Turn the instrument off and shut down the PC.

Instrument Drawings





Report

Tables.

1. Prepare tables of your results for the calibration curve, unknown, urine samples, and the effect of instrument adjustments on the signal (slit width, monochromator setting and fuel mixture).

2. Prepare a table to demonstrate the effect of sodium, potassium and ethanol on the readings for 1 ppm Mg.

Figure

1. Calibration curve: Plot your data and derive a best-fit Mg calibration curve by linear regression; identify the calculator or computer program used. You should not accept the calibration curve produced by the instrument, or the values it returned for your samples or unknowns.

   Include data points for the samples with Na and K and in EtOH/H_20, and your unknown on the calibration graph. Use distinct symbols for each set of conditions and clearly identify the points with labels or a figure legend. Report the concentration of Mg in the unknown.

Answer the following questions in your discussion. Use complete paragraphs and refer to tables and figures where appropriate.

1. Instrument: include a block diagram of the instrument and describe the important components in the Experimental and Instrumental section. The SpectrAA-10 is a double beam instrument: what potential problems can be compensated by this feature?

2. Na and K: do you observe any interference due to Na or K? Account for the presence or absence of spectral interference. I.e. do either Na or K have an absorption or emission at a wavelength that might interfere?

4. Solvent: explain the result you obtained for the Mg in EtOH/H_20 in terms of the processes which lead to atomization: nebulization, desolvation, and vaporization.

5. Wavelength and slit: report your results from *Analysis of Samples* of the procedure. Explain the results.

6. Flame: was there much variation in the reading as the fuel/air ratio was changed? One would not want the measurement to be very sensitive to this parameter because then small changes in the flame would result in large errors in the analysis. Describe why you might observe effects caused by different flame parameters.

7. *Abstract*: after you've written the report write an abstract (see the suggestions in the syllabus). Put the abstract at the beginning of the report.