

## V. LAB REPORT

The lab report should include an abstract and responses to the following items. All materials should be submitted by each individual, not one copy for the group. The goal for this part of the report is for each person to take the large amount of raw data generated by the commercial instruments, sort through it, and organize it into a tabulated form that is easy to reference and use. That is exactly what you would do if you were using a modern instrument on an research project. Use the table numbers and column letters given below to identify the information and give each table a title. Include a table of contents when you have completed the tables for your report, and number all pages of your report at the center bottom position of each page.

### PART I. ICP-AES (section IVA)

1. **Raw data generated by ICP-AES.** Each group member will have a copy of the Excel files generated during the run. Cut and paste from these spreadsheets to create the requested tables 1- 4 below.

**Table 1. Instrumental Method Parameters.** Include the following parameters for each element in your method. Use the following **column** designators:

- A. wavelength for the analytical line
- B. wavelength for the background correction
- C. peak integration time
- D. slit widths
- E. PMT bias voltage
- F. PMT electronic gain

**Table 2. Calibration Method Information.** Note that the program automatically determines the best calibration curve equation and that it may be a higher order polynomial fit. Include the following parameters for each element in your method. Use the following **column** designators:

- A. element
- B. nominal concentration of standard
- C. net intensities
- D. calculated concentrations of standards (must be obtained from printed output)
- E. calibration equation (e.g., slope, coefficients, intercept values) for each element

**Table 3. Analysis Results** for all the samples for each element in your method. Use the following **column** designators:

- A. sample
- B. element
- C. average net intensity
- D. average calculated concentration
- E. standard deviation
- F. relative standard deviation

**Table 4. Detection Limits.** This is based on the standard deviation for each element as determined using the 2% HNO<sub>3</sub> blank run with 15 replicates for each element in your method. For the ICP emission spectrometer, the software reports the blank standard deviation in concentrations units (i.e., it already reports  $\text{stdev}_{\text{bk}} = s_{\text{bk}}/m$ ). Hence,  $\text{DL} = 3X\text{stdev}_{\text{bk}}$  in this case. Include a sample calculation as a footnote to the table for one element. Use the following column designators:

- A. element
- B. standard deviation
- C. detection limit (ng/mL)

## 2. Presentation of Analysis Calculations.

**Table 5.** For each type of sample, report the information specified in the Table 5 template below from the ICP-AES analysis. If the measured value for the concentration of a particular element in a test solution is below the calculated detection limit, report the concentration as 'not detected' or ND for that element. Use the proper number of significant figures. For example, the number of significant figures is rarely ever greater than 3 for concentration data, and only 2 significant figures should be reported for SD, RSD, and the DL.

**Table 5. Elemental Concentrations in Test Solutions with % RSD and Detection Limits from ICPAES.**

Elements Tested	DL (ng/mL)		Samples Tested. Test Solution Results (ng/mL)						
			Syn Unk	Mixed Std- 1 µg/mL	Tap water	Multimineral tablet 1	Multimineral tablet 2	Clam or Oyster	Yogurt
Ca		Conc							
		STD							
		RSD							
Cu		Conc							
		STD							
		RSD							
continue with Fe, Mg, Pb, Zn and your group's element of choice									

**3. Final Sample Results. Prepare Tables 6- 8 as described below.** Back-calculate to relate your results to the values listed on the original containers or given on the USDA web page.

**For Table 6:** Give the initial mass in grams of the original sample used (before digestion or dilution), give the dilution factor if applicable (e.g., 1, 10, 100), and the mass of each element per gram of original sample, that is, the mass of each element per g yogurt, per g of vitamin tablet, and per g of wet oyster or wet clam. Use “typical” serving sizes listed on the container (normally 8 oz yogurt, 1 tablet of vitamin and 3 oz oyster and 2 oz for clams), and calculate the mass of each element in a typical serving, that is, give mass per 8 oz serving for the yogurt, per vitamin tablet and per 3 oz serving of oyster or 2 oz serving of clam. **Show a sample calculation all the way through and give proper units and significant figures for one element for each type of sample in Table 6.**

**Table 6. Mass in a “Typical Serving” or Tablet found from your data.** Include at least the following columns:

Sample Name	Original Sample Mass, g	Test Solution Total Volume, mL	Dilution Factor	Element Name	Test Solution Conc., ng/mL	mg Element/g Sample	Typical Sample Serving Size, g	mg Element /Serving
<b>Yogurt</b>				(make separate row for each element)				
<b>Multi vitamin 1</b>				(make separate row for each element)				
<b>Multi vitamin 2</b>				(make separate row for each element)				
<b>Oyster or Clam</b>				(make separate row for each element)				

**Table 7. Comparison to expected mass values for Vitamin Tablet.** Report in **Table 7** for each of the seven elements (Ca, Cu, Fe, Mg, Zn, Pb, + element of your choice), the mass (mg/tablet) found for multivitamin 1 and multivitamin 2 (from Table 6), the average of these two values, the absolute value of the range between these two values, the percent deviation from the mean in duplicate measurements (% deviation = (range / 2) / average x 100), the expected value for mg element/tablet listed on the container, and the percent difference between the average value you determined and the expected value for mg element/tablet on the container.

- Show a sample calculation with units and proper significant figures for one element listed in Table 7.
- Briefly discuss how well the observed mass agrees with the expected mass for each element.
- Offer some explanation for any elements that do not agree. Consider the differences between observed and expected mg/tablet to the % deviation in replicate measurements, as appropriate.

**Table 8. Comparison to “%Daily Value” or “%DV”.** Report in **Table 8** the sample name, name of element measured, the value for %DV for that element based on your results, %DV for that element listed on the sample container, and % difference between your measured value and the container value. If the element is not listed on the container show NA (not available) in the table entry.

Based on your results for mass per serving (in mg) listed in Table 6, calculate your experimental values for the %DV for each element as follows:

$$\%DV = \text{mass per serving (mg)} / \text{FLGRV daily value (mg)} \times 100$$

The FLGRV values (i.e., Food Labeling Guide Reference Values) are “daily values” recommended by the FDA. Turn to <http://www.cfsan.fda.gov/~dms/dslg-toc.html> and select the link at the bottom of the page to point 14, Appendix F. Calculate the Percent Daily Value (DV) for the Appropriate Nutrients.

- Show a sample calculation with units and proper significant figures for one element listed in Table 8.
- Briefly discuss how well the results for each of the real samples compares to what is expected for each element
- Offer some explanation for any elements that do not agree between measured %DV and reported %DV.

4. **Spectral interferences on ICPAES.** For the added element your group chose, compare the spectral profile run for the standard solution and the profile run on the vitamin tablet solution for that same element. Is there evidence in the vitamin tablet profile that suggests elemental spectral interferences in the region of the element peak? If yes, how could this affect your final concentration results for that element? (Hint: what is the wavelength for the background correction point used to determine the net peak intensities for the added element?)

**PART II. AAS (section IVB)** Work up the data according to the following instructions and turn in the results. Label each item with the numbers and titles given below. **Show a sample calculation with values and units.**

1. **Lab Notebook pages.** Turn in your duplicate pages from your lab notebook with all sample preparation information, file names, method names, and other information. These pages should be initialed by an instructor.
2. **Nonlinear calibration (Copper only).** Use the data for *all* (1, 3, 5, 20  $\mu\text{g/mL}$ ) of the calibration standards and make your own calibration graph of  $A$  vs concentration for Cu only in Excel. Use a polynomial to fit the data with "Trendline" and report the polynomial equation. Does the line deviate from linearity at higher concentrations, and, if yes, what are two possible causes of the non linearity.
3. **Linear Calibration (Copper only).** Using *only the data that yields a linear calibration*, make a calibration graph of  $A$  vs concentration for Cu only in Excel and determine the coefficients in the linear calibration equation. Report the slope in AU per  $\mu\text{g/mL}$  and report the intercept. Calculate the absorbance values expected for the standard solutions from the linear calibration equation (i.e., your results from this step should be in the same format as those from the Spreadsheet Proficiency Test). Make a **Table 9** and show the comparison for Cu calculated using Excel and the values reported by the SpectraAA software by listing the values and giving the % difference between these two values from Excel and SpectraAA, respectively.
4. **Concentrations of test solutions for Copper.** Report the concentrations for Cu only in ( $\mu\text{g/mL}$ ) based on both the calibration equation obtained in Step 3 and the SpectraAA software for the test samples: synthetic unknown, tap water, oyster or clam, VITA1, and VITA2. Include the % difference between the two reported values for Cu and present this information in **Table 10**.
5. **Characteristic Concentration for Copper.** The **characteristic concentration** ( $m_A$ ) for atomic absorption is defined as the concentration of the element that results in a transmittance of 99% T or absorbance of 0.004365. If your intercept (e.g., from least squares linear fit) is non-zero, the characteristic concentration is the concentration which yields an absorbance 0.004365 **above the absorbance at the intercept**. Hence,  $m_A = 0.004365/(\text{calibration curve slope in AU per concentration unit})$ . Calculate the characteristic concentration of Cu in ng/mL. The characteristic concentration for Cu should be less than 100 ng/mL. If the characteristic concentration value is reported in the SpectraAA software, report it also.

6. **Detection Limit for Copper and Lead.** Report the repetitive measurements of absorbance from the blank (reference) solution, plus the mean and the standard deviation (use at least 4 place precision).

$$DL = 3s_{bk}/m$$

where  $m$  is the slope of the calibration curve. The units of  $s_{bk}$  are typically signal units such as volts or absorbance units. Calculate the detection limit for Cu and Pb in ng/mL. For Cu this should be above zero but well below 100 ng/mL and it is normally higher for Pb.

### **PART III. Comparisons for results using three atomic spectroscopy techniques.**

7. **Compare FAAS, GFAA, and ICPAES for synthetic unknown and tapwater.** Prepare a summary **Table 11.** that reports the concentration ( $\mu\text{g/mL}$ ) of Cu and Pb in the synthetic unknown and in the tap water obtained by all three technique: ICPAES, FAAS and GFAA. Discuss how well the results agree and offer an explanation for any that do not agree. Note GFAA was not available for 2011.
8. **Compare FAAS, GFAA, and ICPAES for vitamin tablet and oyster or clam.** Calculate the mass of Cu and Pb in each vitamin tablet and the mean for two tablets (mg per tablet) and in the oysters or clams ( $\mu\text{g/g}$  in the original sample). Show sample calculations for both samples. Prepare a **Table 12** that compares the values for Cu and Pb determined by all three techniques (mg / tablet for the vitamin tablets, and  $\mu\text{g/g}$  for the oyster or clam). Include a percent differences between the ICPAES results and the other two techniques to show how these compare. Discuss how well the results agree and offer an explanation for any results that do not agree.
4. **Table 13.** Report the concentration ranges over which the calibration is linear for each of these techniques used. That is, what is the highest concentration and what is the lowest concentration for the standards that produce a linear response for each technique?
10. **Summary Sheet.** Complete and turn in the "SUMMARY OF MASS DATA AND RESULTS SHEET"

