NAME_______________________________ F ’11

OREGON STATE UNIVERSITY DEPARTMENT OF CHEMISTRY

Experiment 4

Integrated Laboratory Experiment - CH 461 & CH 461H

EMISSION AND ABSORPTION ATOMIC SPECTROMETRY

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EXPERIMENT 4

ICP EMISSION AND FLAME & FURNACE ABSORPTION SPECTROMETRY

I. INTRODUCTION

A. *Atomic Spectrometry*

The development of analytical **atomic spectrometry** began in the 18th century with the observation of colored radiation from flames containing alkali salts. Initially all atomic spectrometers were atomic emission spectrometers because they were based on thermal excitation of atoms to their excited states and monitoring of their characteristic emission lines. Due to the thermal nature of excitation, the magnitude of the emission signal is extremely dependent on the temperature of excitation source (flame, plasma, etc.). The first **flame atomic emission (AE) spectrometers** for quantitative analysis appeared before 1950 and improvements were made in instrumentation including higher temperature and more stable flames and improved monochromators. Emission spectrometry based on arc and spark instrumentation also flourished for analysis of metals in alloys and steel. Although flame atomic emission can be used to determine most metals at concentrations of μg/mL (and below in many cases), it did not provide adequate detection limits for many important metals because of the limited excitation energy of flames. By the 1970's, flame emission spectrometry (AES) was largely replaced by **flame atomic absorption (AA) spectrophotometry** (today AES is sometimes used for determination of alkali metals in clinical samples).

The phenomenon responsible for atomic absorption was first observed in 1802 with the discovery of the Fraunhofer lines in the sun's spectrum. It was recognized that the phenomenon of atomic absorption is responsible for non-linearity in emission methods of analysis (i.e., self-absorption and self reversal). In 1955, Walsh proposed that atomic absorption could advantageously be used for quantitative chemical analysis and the initial growth of analytical **atomic absorption spectroscopy** (AAS) was rapid as it replaced flame AES spectrometry.

It provides better detection limits than flame emission spectrometry for many elements (e.g. Zn, Be, Cd). Flame atomic absorption spectrometry is subject to the same chemical and physical interferences as flame atomic emission spectrometry; however, it is less affected by spectral overlap interferences. **Electro-thermal atomizers** are also widely used as atomization

sources for AAS and provide superior detection limits relative to flames but yield lower sample throughput. This technique is referred to as Graphite Furnace AAS (or GFAA).

Atomic absorption spectrometry is based on the absorption of radiation from an external excitation source by ground state atomic vapor. Under proper conditions, Beer's law is followed and the absorbance of the atomic vapor is directly proportional to the solution concentration of the element determined. Usually line sources such as **hollow cathode lamps** (HCL) are used for atomic absorption spectrometry. Normally a different HCL is required for each element, although some multi-element lamps are available.

In the 1980's, plasma sources for atomic emission spectrometry (AES) were refined and combined with high resolution monochromators. These plasma atomic emission spectrometers have now replaced flame atomic absorption spectrophotometers in many cases, although flame AAS is still probably the most popular single-element technique for analysis of trace amounts of metals.

The most common plasma source is an **inductively coupled plasma (ICP)** which is an argon plasma that receives its energy from a radio frequency (RF) coil. The high excitation temperature (about 6000 K) provides ng/mL detection limits for many elements and calibration linearity extends over 4 or 5 decades with a PMT detector. ICP atomic (optical) emission spectrometers (ICP-AES or OES) are considered multi- element instruments. With high-resolution, computer-controlled monochromators, instruments can perform rapid sequential determinations of many elements in a relatively short time with little operator attention. Instruments that perform simultaneous analysis instead of sequential analysis use CCD detectors.

B. *Scope of Experiment*

In this experiment, you will study:

- the emission of several elements in a plasma, including Ca, Cu, Fe, Mg, Zn, Pb, and element of your choice from the commercial standard by ICPAES
- the absorption of Ca, Cu, Zn and Pb will be measured by flame and Cu and Pb by graphite furnace .

For all elements, data are obtained to construct calibration curves, to estimate the detection limit, and to determine the analyte concentration in real samples including: tap water, a vitamin/mineral tablet, a dairy product (e.g., milk, yogurt), and a biological sample (e.g., liver, clams, or oysters).

II. Solution Preparation

A. Stock solutions and Standards.

Stocks. The following stock solutions are available in the laboratory:

1. a commercial multi-element standard containing 100 μg/mL of Ca, Cr, Cu, Fe, Mg, Pb, Zn, and other elements (As, Be, Cd, Co, Li, Mn, Mo, Ni, Sb, Se, Sr, Ti, Tl, and V).
2. various multi-element standards that are dilutions of stock solution #1 including 5000, 500, 100, 50, and 20 ng/mL levels in 2%(v/v) nitric acid
3. 100 μg/mL mixed standard for Ca, Cu, Pb and Zn (for Flame AA work).

Standards. Each team makes 4 standards - the rest are provided for you. Make your standards from stock #3 above and quantitatively prepare the following test solutions in 100-mL volumetric flasks for the Flame AAS measurements. Dilute to volume with 2%(v/v) nitric acid made in MP water (already done for you and stored in labeled carboys):

4. 20 μg/mL Ca, Cu, Pb and Zn
5. 5 μg/mL Ca, Cu, Pb and Zn
6. 3 μg/mL Ca, Cu, Pb and Zn
7. 1 μg/mL Ca, Cu, Pb and Zn

B. Synthetic unknown and Real Samples.

For the **synthetic multi-element unknown**, quantitatively transfer the contents of the vial to a 100-mL volumetric flask and dilute to volume with 2%(v/v) nitric acid. Record the number of your team's unknown in your notebook.

There are four types of real samples analyzed in this experiment. Be sure to record all relevant product information that is listed on the containers of yogurt, vitamins, clams, oysters: serving size, mass / serving and % DV (daily value) of all the metals listed on the containers.

(1) oysters or clams,

(2) Centrum multivitamin/multimineral tablet,

(3) tap water and

(4) yogurt.

Sample Preparation Protocols:

Oysters and Clams

Oysters and clams are a challenging samples for analysis. They have high mineral content and are considered a good source of Cu, as is liver and crab. The typical Cu content of oysters is 1 to 3 mg per oz (1 oz = 28.35 g) and in clams is 0.1 - 0.2 mg per 3 oz. In this experiment, the oysters and clams will be dry ashed in a muffle furnace. The first day you will just weigh them and place them in a beaker and put the beaker in a designated spot in the muffle furnace in the main lab. The next lab period you dissolve the ash that remains after heating to prepare two solutions for chemical analysis: one concentrated solution for the AAS analysis and one diluted solution for the ICPAES and GFAA analyses.

Oysters:

Take a couple of oysters out of the can and pat the oil/and liquid off with some Kimwipes. Weigh the oysters to 0.1 mg and transfer to a small beaker. The total mass should be between 3 and 6 g. If the mass is not in this range, add or remove an oyster. Somewhat chop up the oysters in the beaker (don't lose any mass). The instructor will help you put your team's beaker in a designated spot in the muffle furnace. Over the weekend the muffle furnace will be ramped up to 500 °C and the oysters roasted for at least 24 hr at to produce a white ash. At first the temperature is set to 100 °C and then increased to 500 °C so avoid excessive organic smoke at the start that could cause losses.

Clams:

Take several clams out of the can and drain and pat the oil/and liquid off with some Kimwipes. The total mass should be between 30 and 40 g. If the mass is not in this range, add or remove an clam. Weigh the clams to 0.1 mg and transfer to a medium beaker. Somewhat chop up the clams in the beaker (don't lose any mass). The instructor will help you put your team's beaker in a designated spot in the muffle furnace. Over the weekend the muffle furnace will be ramped up to 500 °C and the clams roasted for at least 24 hr at to produce a white ash. At first the temperature is set to 100 °C and then increased to 500 °C so avoid excessive organic smoke at the start that could cause losses.

For both the oysters and the clams:

The next lab period, add a few milliliters of 50% (v/v) nitric acid (i.e., 50% concentrated HNO_3, 50% water that you make yourself). Gently heat and swirl for 5 min. Do not use boiling chips. Neutralize up to between pH 2 and 7 by adding a drop of con NH_3 (aqueous) (check pH and add more base if needed. Keep a record of how much base is added.) Filter the solution directly into a 50-mL volumetric flask using Whatman Qualitative "1" filter paper, 5.5 cm. Wash the beaker with 2% (v/v) nitric acid and quantitatively transfer to the flask through the filter paper BUT DON'T exceed 50 mL of washings. Bring up to the 50 mL volume with 2% (v/v) nitric acid. This is the concentrated solution and will be analyzed by AAS. Next, quantitatively prepare a 1 to 10 dilution of this concentrated solution using 2% (v/v) nitric acid and this diluted solution will be used for the ICP experiment.

Multi-vitamin/Multi-mineral Tablet

Analyze two Centrum Multivitamin & Multi-mineral tablets as follows. You will have four solutions at the end of this procedure. Weigh each whole Centrum tablet to 0.1 mg and then crush each one in a separate mortar and pestle. Transfer about 0.15 g of each powdered tablet to weighing paper. Weigh and quantitatively transfer all the powder into a medium test tube, then re-weigh the paper to determine the mass transferred to the test tube. CAREFULLY add 5 mL of concentrated nitric acid to both tubes and heat them in the dry block heater at 60 °C for 1 hr under a hood. Quantitatively transfer the contents of each test tube into a separate 100-mL volumetric flask and bring up to volume with MP water (the solution will now be 5% (v/v) HNO_3). These solutions are denoted as vitamin sample solutions VITA1 and VITA2.

Next, make a 1 to 100 dilution of each VITA solution in the 2 % (v/v) HNO_3 stock solution and denote these solutions as VITB1 and VITB2.

Tap Water

Clean two 100-mL bottles. Note the location of the tap or water fountain, the time and date, and the sampling conditions that you choose to use (e.g., first sample of the day, sample after extensive flushing, etc.). Collect two samples by filling two bottles and stopper these (one for AAS and one for ICPAES & GFAA analysis). **Do not use DI or MP water for this part.**

Yogurt

Yogurt, a fermented, semifluid milk product, is used extensively as a health food. It is prepared from fresh whole or skim milk, boiled and concentrated by evaporation. Fermentation is caused by the addition of cultures of bacteria, usually Lactobacillus bulgaricus and Streptococcus thermophilus, and sometimes Lactobacillus acidophilus. This type of fermented milk has long been an important constituent of the diet in southeastern Europe and in Asia Minor and is popular in the U.S.

Procedure for using the CEM Microwave Digestion System

You will use the MDS 2000 microwave digestion system in GBAD 313 to digest a yogurt sample for elemental analysis with ICPAES. The total digestion time will take about 50 min so that you should set this part up to run while you work on making the other solutions for Exp. 4. **Wear eye protection and latex gloves.** Be prepared to clean up any spills immediately with sodium bicarbonate and wash with water. If you get nitric acid on your skin, flush with copious amounts of water and don't be surprised if you see yellow stains from the reaction of the acid with certain amino acids in the skin.

The dissolution process is carried out as follows.

1) Record serving size and %DV of Ca. Task is to quantitatively mass a viscous sample. One method is as follows: draw up sample in plastic pipet, weigh whole thing on analytical balance, transfer 0.15 to 0.25 g of yogurt by dropping (about 0.25 mL to 0.50 mL) directly into the bottom of the Teflon vessel (avoid splashing sample on the inside walls). Re-weigh the plastic pipet with left over sample, and determine by difference the amount of yogurt added to the Teflon vessel. If you added too much or it spilled during the transfer, wash out the bomb vessel and start over. If you have a better idea, try it! You just need to make an analytical transfer for the sample.

2) Working under the hood, carefully add 10 mL of concentrated HNO_3 (70%) so that it covers the sample. Let the vessel liner stand open in the hood for 15 min before sealing. This pre-digestion step allows the sample digestion process to begin before sealing the vessel and helps to avoid pressure overruns during the closed vessel step.

3) Put the Teflon cover on the liner and seal the cap on the threaded ring.

4) Carefully insert and seat one new rupture membrane inside the grey vent fitting. Tighten the vent fitting over the knurled outlet on the cover. The membrane is designed to blow if the pressure builds up too high in the sample vessel.

5) Place the liner in the sleeve and put the entire thing in a position of the turntable. Record the position that you are using in your notebook. After all the sample vessels are assembled and in the turntable, the turntable will be loaded in the microwave oven (instructor). At this time each vessel is fitted with a vent tube through the ferrule nut. The vent tubes go into a collection vessel in case the reaction is so vigorous that the excess pressure breaks the membrane and hot acid solution spews out.

6) The instructor will load the temperature probe into the sensor vessel and fit it with the special microwave valve. The pre-programmed pressure method called "Yogurt" below has been loaded in memory and will be used. Press the Start button on the front panel of the oven.

MicroWave Digestor File Name: Yogurt
Standard Digestion
stage (1) (2) (3) (4) (5)
POWER (% 630 W) 60% 60% 60% 60% 60%
PRESSURE (psi) 20 40 85 130 175
RUN TIME (min) 8:00 8:00 10:00 10:00 10:00
TIME @ P (min) 3:00 3:00 5:00 5:00 5:00
TEMPERATURE °C 120 140 160 180 200
FAN SPEED 100% 100% 100% 100% 100%
NUMBER OF VESSELS: 6
VOLUME PER VESSEL: 11 mL
SAMPLE WEIGHT: 0.2 TO 0.3 g
ACID: HNO3

After the digestion is finished (possibly next lab period):

7) The instructor will remove the turntable with vessels to a position under the hood. The vessels must cool down to room temperature before they are vented.

8) After the vessels have cooled to room temperature, carefully vent by slowly opening the vent fitting. Work under the hood. Wear eye protection and latex gloves. Expect a small amount of NO_2 gas to be emitted.

9) Quantitatively transfer your sample solution into a clean 50-mL beaker. Rinse out the Telfon vessel with a minimum of Millipore water (not more than about 20 mL).

10) Rinse all Teflon parts immediately with hot water. The imide cap and threaded ring should be rinsed and dried. Return all parts to the instructor immediately.

11) Quantitatively transfer the sample solution in the beaker to a 200-mL volumetric flask and carefully dilute to volume with Millipore water. Do this under the hood, wear eye protection and gloves and use caution. The digestion program is given below.

Waste Disposal. Excess solutions containing 2%(v/v) nitric acid should be neutralized with $NaHCO_3$ (baking soda) before disposal down the drain. To neutralize, transfer acidic solutions into a large beaker on ice, place in hood, and slowly add $NaHCO_3$ until evolution of CO_2 ceases.

III. Instrumentation

This experiment will be conducted with three commercial spectrometers. The flame absorption measurements for three elements will be done on the Agilent 240 Fast Sequential flame absorption spectrometer. The GFAA measurements for three elements will be done on the Agilent 240Z electrothermal absorption spectrometer. Both of these are in GBAD 314. For multi-element emission measurements, the JY 2000 ICP emission spectrometer in GBAD 313 will be used. One of the instructors will demonstrate the use of these spectrometers when you are ready to use them. An additional document with more detailed instructions about the operation of each instrument will be provided to you. The instrument manuals are usually found next to each instrument and are also available for reference. Review the pictures of the ICP torch on the course supplementals page.

IV. Experimental

A. *ICP Emission*

Use the following initial instrumental settings on the JY 2000 (in GBAD 313):

plasma power	1000 W
sample flow rate (pump speed)	20 (1 mL/min)
plasma gas (Ar) flow rate	12 L/min
auxiliary gas flow rate	0
sheath gas flow rate (gainage)	0.2 L/min
nebulizer pressure	3.2 bar
nebulizer gas flow rate	~0.7 to ~ 1 L/min (regulated by neb)
entrance slit width	20 µm (10 µm is also available)
exit slit width	15 µm (80 µm is also available)
integration time	3 s except 1 s for Mg
measurement mode	max (1/1)
no. of measurements for calibration	3
no. of measurements for recalibration	3
flush time	30 s at fast pump speed

An instructor will take you through some of the primary operations of the instrument which is computer controlled and will provide details about the software, ICP-JY v . 5.4. Use the 2% HNO_3 blank solution as the Global Rinse solution when prompted for this. You should perform the following steps:

1. **Initial Plasma Setup and Zero Order Search** - Learn about how to start the plasma and to adjust plasma conditions such as RF power, plasma gas flow rate, and sheath gas flow rate. Once the plasma has started and stabilizes, minimize the Automatism window (don't close it or the plasma will go off in 7 min!). Next run the zero order search under the Instrument-Configuration-Run- from the top menu bar to find the search zero order position for the stepper motor. Open the Communication task window on the task bar so you can watch the progress. The software automatically optimizes the search for zero order. Record the zero order position in the ICP log book and in your notebook. Select Stop when the search is done and close the Communication window.

2. **Select a Method -** You will choose a method file which has been configured for this experiment (**"CH_461_2011"**). Load the Analytical Method from the top menu bar. **Immediately save this general course method with a new name that identifies your team.** When a "method" is developed, the user specifies which elements are to be determined and the measurement conditions for each element selected (e.g., analytical wavelength, entrance and exit slit widths, PMT and gain parameters, integration time, number of repetitive measurements, measurement mode). The background-correction wavelength is set after the slow scan of the peak region or profile task is done.

3. **New Method Development and Setup-**Before a method can be applied, several steps or tasks called **peak search**, **autoattenuate**, and **profile** must be carried out for each element in the method. Peak Search counts out the steps from zero order to the element wavelength and stores it; AutoAttenuate optimizes the HV and Gain at the peak wavelength for the PMT detector for each element and stores it. Profile uses the peak search and autoattenuate results and scans through each element line and store this scan of the peak. You will use the profile scan to identify a background point as reference and store this. We have already run these tasks to prepare this method for your use for the 5 main elements, however, to illustrate what steps are involved with setting up a method, you will edit your team method and re-run these three tasks for one element, so use the additional element you chose to add to your method from the commercial 21 element standard for quality control (QC).

Edit Method to Add New Element:

A. Add your additional element of choice to the established method. To do this open your Method if not already open. (See step 2 above). Select Edit from right tool bar, then Add/Delete button at the bottom. A periodic table should open. Click on the element that you are adding to the method. Set "concentration high" to 5000 ng/mL for this element.

B. Select the analytical line for the new element by clicking on the Lines tab. There is a database in the software that will bring up the suggested lines for analysis of this new element. Select a line that has a decent LOD (limit of detection) and it is usually better to pick a line that is more toward the visible region if possible.

C. Next you will need to set the Acquisition mode. Select the new element from the Lines list and select the Modify button at the bottom of the screen. Go to Acquisition tab. Select Customised (big button); Analysis mode: Max; measure points 1, calculate points 1; Integration time, 3 s; entrance slit 20 and exit slit 15; increment 0.002 nm. Then OK.

D. Add the set of calibration standards already setup for the other elements to your new element. Click on Standards tab, select the element symbol, check all 4 standard boxes. Select the Standards button lower left, and enter the values for the standards (5000, 500, 50, 0). Select Save and Close method.

Running the Instrument:

E. Click on the Sequences & Tasks icon from the Top Menu. You need to associate your method with a Sequence. To do this, look near top of the form on the left side and select your method from the drop down list. Next, select New from the right side menu and build a sequence of the three setup tasks (or steps): **peak search**, **autoattenuate**, **and profile**. Your method name will also be listed in the middle column in the list of tasks (check this before you go on). Click on the task ICON at the start of a row and **select only the new element you added to the class method.** The other elements have already been run this year.

Read the overview below of what the three setup Tasks will do before you actually start the run. Note that two different standard test solutions are used in the steps below :

i.. **Peak search** - This task is carried out to check that the monochromator is properly calibrated to find the wavelengths for all elements selected (finds and stores the correct number of stepper motor steps). For your new element, use the **5000 ng/mL** multi-element standard for peak search.

ii. **Autoattenuate** - This task automatically adjusts the PMT bias voltage (HV) for an optimal gain and adjusts the electronic gain (1, 10, or 100) to optimize the absolute magnitudes for the peak signals so they will be on scale. For your new element, use the multi-element standard containing **5000 ng/mL**

iii. **Profile** - This task provides a scan of each element line spectrum over a very narrow wavelength range around the peak for the element. Later you will use the profile spectrum to select a background correction wavelength point for each element. For your new element, use the multi-element standard containing **500 ng/mL**. The profile is setup to run with a 0.3-s default integration time per point (don't change this). After the profile is done running, get a print out by selecting Print near the bottom of the open sequence window.

Now you are ready to run the Sequence of Tasks you just built. Select the Red arrow (top left side of menu) and when prompted give your Sequence a unique file name. Once the sequence is started you will be prompted to use a Global Rinse (use the 2% HNO_3) between samples and standards. You should always wipe the sipper tube with a kimwipe when switching test solutions to avoid carry over. To monitor the progress of the run, maximize the ICP Analyst window and click on the Instrument Status window on the task bar. To check the intensity of the signal for each element as the run proceeds, click on the lightning-bolt icon next to the element symbol.

iv **Set Background point**: After running the setup Sequence, close Sequences and go back to the Methods screen. Open the Lines tab, then select Edit (right side), then select Modify (bottom) and then Background tab. Select Profiles and pick the profile filename that you just collected for your new element. Find a point on the baseline, either to the left or to the right of the main peak where the baseline intensity is low and constant. Set this as the background point by double clicking (select only one background point). If the background point has already been assigned, you can use the pre-assigned value or set a new value yourself (you have to delete the old one). Now for all analysis runs, the software will automatically calculate the net peak intensity based on the value at the peak maximum and the value at this background point. Later you will re-run a profile scan on the same (new) element using the multivitamin sample solution to check for possible interferences from other elements in the real sample in the region of this element peak.

4. **Print a copy of the Analysis Parameters -** Print the important method parameters for all elements. **To do this, go to Method and choose Print Method BUT check only the following:**

User Acquisition Parameters, Background, and Plasma Parameters.

5. **Calibration -** Return to the Sequences screen and select the Calibration task. **<u>Run this task for all the elements in the method</u>**. Run the 2% HNO_3 blank solution as the lowest standard and the three multi-element standards as follows in this order:

STDLOW	blank (2% HNO_3) - be sure it is freshly taken from the carboy
STDA	50 ng/mL multi-element
STDB	500 ng/mL multi-element
STDHIGH	5000 ng/mL multi-element

Check List for getting a good calibration curve:

- The standards must be run in the order listed above
- Do not forget to give your Calibration task a unique group file name when you are prompted to do so.
- Look at the average intensity of the signal for each element line immediately as it is generated to confirm that the repetitive measurements are consistent within each standard and that the intensity increases with concentration. For example, the signal for 5000 ng/mL is 10 times the signal for 500 ng/mL, and the 500 ng/mL is 100 times the signal for 50 ng/mL. If not consult with the instructor before continuing. **Sometimes Cu doesn't work well on the blank and the 50 ppb standard. If these values are nonsense, rerun just Cu (ask if you can't figure out how to do this).**

After all the standards are run, the program will autoprint a calibration summary which includes the average net corrected signal and the RSD for each element in each standard. Check that the RSD values are reasonable. If these are too high, investigate the causes. The summary also includes the equation for the calibration curve based on a linear regression fit for each element in the form (the software automatically determines if more than a first order fit is required):

conc = (intercept) + (slope) Intensity + (coefficient) (Intensity)^2 + higher orders

The calculated concentrations should be near the expected values for STD A, B, and HIGH and the blank (STDLOW) should be within 10 ng/mL of zero for most elements. You may be prompted to accept the calibration. If the calibration is not good, rerun the elements and/or concentrations that don't match (you don't have to run all elements and all concentrations).

Now return to the Methods screen and select the Calibration tab to view a plot of the calibration curve for each element. If one of the standards is not reasonable it can be eliminated from the calibration, or you may choose to rerun that element calibration if it is really bad. The general course method is setup to use a weighting factor for each standard (1/square root of standard deviation) in the regression; the weighting factor helps to ensure that the low-concentration samples aren't overwhelmed by the high-concentration samples in the regression over the wide range of concentration data. The program also decides whether a polynomial fit is better than a simple linear fit. You can change this if you don't like it.

6. Analysis and Detection Limit - Return to the Sequence window, select New, and add one Analysis Task for each sample listed below (bulleted items) and save the Sequence. Edit the name for each task to indicate the sample name and put your team number in the name. Determine all the elements in the method except where indicated below. The method you are using is setup to run each element for each sample in triplicate. You should change the number of replicates for the blank measurement to 15 to get better statistics for the DL.

The analysis samples are:

- 2 % HNO_3 blank (STDLOW) - run this first and use this data later to find the detection limit for each element. You will need to change the number of measurements in the this task to15 replicates for each of the five elements: select Edit from the menu bar on the right, then left double click on the task icon and change the number of Replicates from 3 (the default) to 15.

Change the number of Replicates back to 3 (triplicate - not 15).

- tap water (all elements in your method)
- synthetic unknown (all elements in your method)
- 1 µg/mL mixed Ca, Cu, Pb, and Zn standard that you made - to test your solution preparation skills. To select only these 4 elements, (the others in the method are not in the standard solution) select Edit from the menu bar on the right, then left double click on the task icon, go to Elements (a periodic table should appear on the screen), deselect elements that you do not want to run this time for this sample.
- yogurt (all elements in your method)
- vitamin tablet solution **VITB1** filtered with 0.45 µm nylon disk filter (run all elements in your method)
- vitamin tablet solution **VITB2** filtered with 0.45 µm nylon disk filter (runa all elements in your method)
- oyster or clam - **only the diluted 1 to 10** solution and filtered with 0.45 µm nylon disk filter (run all elements in your method)
- add one more Task and make it a Profile run of the **VITB1** solution and set it only for the new element that you ran a profile on earlier using a standard. Get a hard copy of this using Print before leaving the Sequence window.

Before you start the Sequence, save it with a unique name using your group number. After each sample is run, the program will export the data to an Excel file: the mean net or baseline corrected signal, the estimated concentration in the sample based on your calibration data, the calibration fit equation for each element, the SD, and the RSD.

Now check your data against the criteria below to make sure it is consistent and if not, re-run the solution that doesn't fit:

a. For your analysis samples, the element concentrations should be within the standard range and the RSD should be reasonable (better than 5%) in most cases.

b. For your blank measurement, note that this signal for the blank and its SD (i.e., the blank noise (s_{bk})) is **reported directly in concentration units** and not in signal units. This means

that the SD reported by the program is the standard deviation of the signal automatically divided by the calibration curve slope. Later you will use the SD in concentration units to calculate the detection limit for that element. **Check that the individual peak intensities taken for the DL measurement are consistent** with each other. For example, if the first point is high and distorts the standard deviation, recalculate the blank SD yourself using the remaining points. Also check that the readout resolution is adequate. **If the standard deviation shows as zero**, you should adjust for more resolution (decimals for readout) or later calculate the standard deviation from individual results given in the spreadsheet of the exported data.

c. For the sample VITB1 profile, go back to the Method screen and look at the Profile of the VITB1 solution you just ran and compare with the original profile run on the standard solution. Note any differences. Get a hard copy of both profiles (the standard and the VITB1 before you leave).