Varian Vista MPX Inductively Coupled Plasma (ICP) Spectrophotometer

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Instrument instructions can be found at:
http://academic.bowdoin.edu/chemistry/resources/instructions.shtml

If you have any problems with the instrument or would like to get trained, please contact
Celeste Morin
(x3756 / cmorin@bowdoin.edu / Druckenmiller 243)

Be sure to read and familiarize yourself with the following before you operate or get trained on the instrument.

1. Understand how the ICP-OES works:
    a. Access Multimedia Help: Open up the instrumentation software on "simulation" mode (Left Click on Start/AllPrograms/ICPExpert/Simulation). Then access multimedia help via the drop down Help key on the top white bar: Help/Help Topics/ Multimedia Help.
    b. Review the "Starting the ICP" power point. This is located on the desktop of the ICP's computer.
    c. Read the FAQs at the end of this document.

2. **Create a folder for yourself in Program Files/Varian/ICPExpert/Run/My Results.**

3. Using the simulation mode, set up a "semi-quantitative" analysis (see FAQs #3). When you run a semi-quant you will get an idea of the identity of elements in your sample and their approximate concentrations. In turn, this will help you decide the
    a. Appropriate torch, spray chamber and nebulizer for your analyses.
    b. Appropriate standard concentrations required for your analysis. Be sure to make your standards in the same matrix as your samples.
    c. If an ion-suppressant or internal standard is required (this is particularly useful if you cannot matrix match your standard and samples, i.e., if you are analyzing seawater). If any elemental concentration is above 200 ppm (for Ca, Na, Mg) and you cannot matrix match, be sure to use the ion suppressant and an internal standard.

Note: Most semi-quants are not set up to handle Hg (this is "sticky" element, more information later).

4. Torch, Nebulizer, Spray Chamber Selection. The selection is determined by "%" solids or "salt" or "ion" concentration present in your samples. If you are analyzing DI water or fresh water, you are dealing with a "low" solids operation. If you are analyzing seawater, soil extractions, or solutions with a high ionic strength, you are dealing with a "high" solids operation. In your selection, you will weigh what is appropriate for the instrument and how close your samples are to the detection limit for your analyses. Note all high solids components cause a decrease in sensitivity as they are designed to reduce the salts reaching the cone face and detector. Use 100-200 ppm Na, Ca, Mg concentration cutoff between high and low solids operations.

High Solids: High Solids Torch (shorter than the low solids torch), Twisted Cyclonic spray chamber (this one has a baffle as opposed to the low solids), C-style or V Groove Nebulizer.

f. 373.69 is not a great peak for Ca, 234.35 line for Fe has a neighboring peak close by, 404.721 line for K is very insensitive.
a. If your

tubing from bottom, and the drain container tubing from the top. Press down the clamp and clip to engage.

4. When the pump starts (after the torch is lit), adjust the pressure knobs so the sample uptake is a smooth flow and the drain is “starved”, meaning there are air bubbles in the line because it doesn’t have enough liquid for a constant flow. Improper flow rates can extinguish the torch.

v. Autosampler

1. Yellow tinted tubing with white tabs is used for the autosampler. One end is connected to the tube that fills the container that rinses the sample probe, and is at the top of the peristaltic pump. The tubing is then fed through the peristaltic pump. The other end of the yellow tinted tubing connects to the tubing that is in a 2% HNO_3 (in DI H_2O) reservoir and is at the bottom of the peristaltic pump. There are two ports on the front of the this container. The port on the right is for filling and connects to the yellow tinted tubing. The port on the left is for draining and connects to the tubing going into the drain container. Once again, adjust the pressure knobs so water is being drawn up the tube in which the sample probe rests.

g. **Check that the instrument is turned on** (green light on lower left side of instrument is lit).
h. **Check that the laboratory exhaust system is on** (dial is turned to "OCCUP").
i. **Check that the drain tubing is in the drainage container.**
j. **Turn on the water cooler** (located behind the instrument, should go to 45psi).
k. **Open the ICP Expert 4.1.0 software.**
    i. Start > All Programs > ICPExpert > ICPExpert.
l. **First time users adjust these preferences:**
    i. Go to Options > Preferences.
        1. General
            a. QC test page visible – check
            b. On startup turn Polychromator Boost on – check
            c. Drift correct with Argon lines – check
        2. Sequence
            a. Recalibrate every – uncheck
        3. Conditions

a. Power (kW) – 1.20	e. Sample Uptake Delay - 30
b. Replicate read time (s) – 30	f. Pump Rate - 15
c. Plasma Flow - 15	g. Rinse – 15
d. Nebulizer – 0.75	h. Initial Stability Delay 15

****Note**--For 3 replicates w/30sec read time, 30sec sample uptake delay, and 15rpm pump rate = 3mls of sample**

        4. Calibration

a. Units - ppm	c. Weighted fit - check
b. Max % error - 10	d. Outside valid cal. Range - check

    ii. You can change these settings in the Conditions tab in Method Editor, but these should be the default conditions that the program uses.
    iii. Restart ICPExpert program.
m. **Polychromator boost will start when program is opened.**
n. **Click on the Instrument icon and select the <u>Status</u> tab.** Check the following:
    i. HV power supply off – this will turn on once the torch is lit.
    ii. Water Cooler – Flow Ok.
    iii. Gas Flow – Argon Ok.
    iv. Plasma Off.
    v. Poly Boost – On.
    vi. Cone Purge – Off.
    vii. Casting - ~ 35 C.
    viii. Peltier - ~ -30 C.
    ix. Hook rinse water line to pump between the white clips. Be sure of the direction.
o. **Turn the autosampler peristaltic pump on so sample probe rinse container fills with water before torch is lit.**
    i. <u>Instrument Window/Autosampler</u> tab.
        1. Click on the Rinse button.

2. Check the container where the autosampler rinse probe rests and make sure water is spilling over into the drainage section. This may take a few minutes.
3. Once water is spilling over, click the Stop rinse button.

p. **Start the plasma torch.**

i. Turn on the pump (middle pump button) for a brief period to make sure DI water can reach the torch before you turn on the plasma. Check for even flow of water to the drain as mentioned earlier. **If water cannot reach the torch when ignited, it will melt.**

ii. Turn on the plasma by clicking the Plasma On icon  (or go to Analyze > Plasma On). The peristaltic pump will be initialized and the DI water

2. Check that Mode on the left hand side is “point sum”, the Background Correction mode on the right hand side is “fitted”, and the number of points per peak is “2”.
3. For more advance corrections see below.
4. You should have one of your standards in an autosampler tube so you can test to see if there are any interferences and verify the baseline points are correctly set.
5. Click on the “Go To SPS Tube” icon  .
6. On the screen that appears there is a layout of the autosampler tubes. Click on the tube that contains your sample. If you make a mistake, click on the Rinse icon and the sample probe will go back to the rinse tube.
7. When finished, click OK.
8. When the sample has reached the torch, click on the Read Spectrum icon  .
9. When the sample has been read (spectra will appear), return the sample probe to the rinse state.
    a. Click on the “Go To SPS Tube” icon.
    b. Click on the Rinse button.
    c. Click OK.
10. On the Corrections window you will see a spectrum of your first element.
11. The red bars near the top of the peak indicate where the program is integrating the peak. Draw a line straight down from the two vertical bars and that section is what the program is integrating.
12. To move the red bars, move the cursor over to the bars and click and hold the Ctrl key. The crosshairs will change into a hand. Now click and hold the left mouse button and move the red bars so they are centered on the peak. You can also click and hold the Ctrl key and move the hand to the top of the peak and click the left mouse button once. This will center the red bars on the point at which you clicked.
13. The Background Correction Mode is set to “Fitted”. This will work 99% of the time. If you are not happy with the baseline, you can manually change it.
    a. Select “Offpeak BC – Left & Right” from the Background Correction Mode pull down menu.
    b. Move the cursor to the teal or yellow bars that are at either end of the baseline.
    c. When the cursor turns into a hand, click and hold the left mouse button.
    d. Move the bars to the endpoints you would like to use.
    e. This will change the baseline points for all the standards/samples using this line.
14. Click on each element and make the corrections.
15. When finished, click OK.

viii. Save method parameters (File > Save).

ix. Close Method Editor (File > Exit).

a. **Sequence tab**
   i. Sample source should be "Autosampler" (located on right side of screen).
   ii. Click on the **Sequence Editor Button** (located on right side of screen).
      1. Samples and Calibrations tab
         a. Enter the number of samples in the Sample count field (minimum of 2).
         b. Make sure "begin with calibration" and "include a blank in calibration are checked". You may chose a recalibration rate and to end with a calibration if you like.
      2. Rate Generated QC Tab

      (*Note: you will use either Rate Generated or Manual QC)*
         a. On the left side you will see a list of the QC tests you selected. On the right side you will see different rate blocks. They correspond to different types of QC tests.
         b. The QC tests that relate to your standards (ICV only) will be placed in the "Calibration QC Blocks" box.
            i. To add one of the QC tests to the block, click and hold the left mouse button on the test and drag it to the block. When the block is highlighted, release the mouse button and the test will appear below the block.
            ii. To change the action that is performed when the QC test fails, highlight the block by clicking once on it with the left mouse button. Do not skip this step.
            iii. Right click on the block and select Properties. In properties section, you may use **flag and continue**, else your run will be stopped if the QC fails
         c. The QC tests that relate to your samples will be placed in the "Sample QC Blocks" box. [*not typically used]*
            i. To add one of the QC tests to the block, click and hold the left mouse button on the test and drag it to the block. When the block is highlighted, release the mouse button and the test will appear below the block.
            ii. To change the action that is performed when the QC test fails, highlight the block by clicking once on it with the left mouse button. Do not skip this step.
            iii. Right click on the block and select Properties.
         d. The "Rate QC Blocks" box will let you select a rate (in number of samples) in which you want that QC test performed.
            i. To create a new rate block, right click in the "Rate QC Blocks" box and select "New Rate block".
            ii. To change the action that is performed when the QC test fails or the rate, highlight the block by clicking once on it with the left mouse button. Do not skip this step.

1. Go to View > Define Column.
2. Expand the Lines folder (click on the plus sign)
3. Click and drag the element you want the mean for and place it on the Elements folder.
4. If you expand Elements, it will now have a section titled “xx Mean”. “xx” is the symbol of the element you picked.
5. You can remove lines by right clicking on the line in the Element section and selecting “Remove”.

Note: Flagged values are not used in the mean.

vi. There are four different screens that can be viewed. You can switch between screens

certain replicate or the mean of all the replicates. This will remove it from the calculations.

1. Select the row you want to mask.
2. Click the Mask icon, then the Apply icon, and then the Recalculate icon.
3. Masked data does not go away.

a. **Create a template if you plan on using the method, sequence, or calibration worksheet again.**
   i. Method Template – will store only the method information (lines used, conditions, etc).
   ii. Sequence Template – will store everything the method template does, but also the sequence information (QC tests, sample names, etc).
   iii. Calibration Template – will store everything the method and sequence templates do, but also the calibration information. Since the calibration line is not valid for quantitative work after a few hours or so, this is more for semi-quantitative work. This is useful for screening future samples to get an idea what is in them without having to build a calibration table.

b. **To create a template based off an existing worksheet.**
   i. Go to File > Save As.
   ii.

d. **Go to Corrections in Method Editor** (Method tab).

e. **Change the mode from Point Sum to FACT. This will bring up the FACT wizard.**

Note: Before clicking the analyze button in the wizard, make sure that you are analyzing the correct solution. When analyzing multiple lines at the same time, the wizard will not advance each line, so you will need to go through for each line and click the Next button until you are at the appropriate screen.

f. **The wizard will prompt you to analyze different solutions.** The order will be:
   i. Blank
   ii. Matrix (optional)
   iii. Analyte – the solution that contains just the element you are analyzing.
   iv. Interference model – the solution that contains the element that is causing the interference. You can right click on the "Interference" and name this model when this step is complete.
   v. Sample – this is the solution that you were initially analyzing. This is how FACT will test the model it created

g. **When finished, click the Test FACT button.**

h. **A graph will be displayed that shows the analyte, the interference element, and the combination of the two.**

i. **Once FACT is finished, all the results for line that you selected and ran FACT for will be automatically recalculated.**

Note: You will need to run FACT for each analyte line that has interference.

a. **Open Report Settings** (File > Report Settings).

b. **Select options and preview report.**

c. **When finished, print the report** (File > Print).

d. **Save worksheet** (File > Save).

a. **Rinse the spray chamber by aspirating DI water for a few minutes.**

b. **If you used an IS/IS solution, be sure to rinse that tubing out by flushing it out with DI water.** Just remove it from the IS/IS solution and place it is DI water.

c. **Extinguish the plasma by clicking the O12(a b)- 222./F2 12 Tf1 0 0 1 224.21a0in iOti ic 0 612**

Note: The value you enter is how far off the sample probe is from the bottom of the sample tube in millimeters. Be sure not to enter a value that will cause the sample probe to hit the bottom of the sample tube. Doing so could bend the sample probe.

a. **If you are not sure what the optimal height is, go to the <u>Method</u> tab and click on the Method Editor button.** Go to Tools > Corrections. Click on "Go To SPS Tube". There is a "Down Height" box that will let you adjust the height of the sample probe. Test this, decreasing the height slowly, until you find the optimal height. Once you have entered a height, click on the autosampler tray display (left side of the same window) in a location that contains a sample tube.
b. **In the <u>Sequence</u> tab, click on the Autosampler Setup button.**
c. **Click on the Options button.**
d. **<u>H</u>**

a. **After analysis is complete, you can only change information on the Standards tab in the Method Editor.** You can change the concentration of the standards, which standards to use, and the MulitCal parameters. You cannot change the elements that were analyzed, the conditions in which the samples were run, the quality control settings, or the sequence table. There are a few data manipulations you can do in the analysis screen.