1.0 Purpose

To describe how to verify that magnesium sulfate heptahydrate samples are below the USP limits of 0.014% chloride, 20 ppm iron, 0.001% heavy metals, and 0.003% selenium, following USP Monograph: Magnesium Sulfate.

2.0 Scope

This procedure applies to USP lot change, stability testing, and any time USP quality needs to be verified. All USP testing is performed in the Quality Assurance laboratory.

3.0 Responsibility

QA Lab personnel are responsible for USP testing.

4.0 Safety Considerations

Safety Goggles, Chemical Resistant Gloves, and Lab Coat should be worn.

Safety is a condition of employment. Employees are not authorized to work in an unsafe manner and are prohibited from harming the environment of the facility or community.

5.0 Materials/Equipment

- Balance-Mettler Toledo X5105Du, B13929Z316
- Weigh Paper
- Eppendorf 1000-µl Adjustable Pipette
- Eppendorf 5-ml Adjustable Pipette
- 100-ml Graduated Cylinder
- 2 x 100-ml Class A Volumetric Flasks
- 2000-ml Class A Volumetric Flask
- 4 x 25-ml Beakers
- 2 x 100-ml Beakers
- 250-ml Beaker
- 7 x 5000-mL Reagent Carboys
- 15-ml Metal Free Centrifuge Tubes (One Per Sample)
- 9 x 50-ml Standard Autosampler Tubes
- 14 Position Rack for 50-ml Standard Autosampler Tubes
- 44 Position Rack for 15-ml Autosampler Tubes, With Bottom Removed

- Prodigy High Dispersion Simultaneous ICP Spectrometer with Axial Option, Halogen Option for Axial, Autosampler, Salsa Software Package, and Accessories-Teledyne Leeman Labs

Reagents:

- Nitric Acid, 70%, High Purity, Trace Metals
- Deionized H_2O (ASTM Type II or Better)
- GILES-4 Multi-element Stock Standard Solution-Inorganic Ventures
- GILES-5 Multi-element Stock Standard Solution-Inorganic Ventures
- Gallium, 1000 µg/ml ICP Standard in 5% Nitric Acid-Teledyne Leeman
- Yttrium, 1000 µg/ml ICP Standard in 5% Nitric Acid-Teledyne Leeman
- Arsenic, 1000 µg/ml ICP Standard in 5% Nitric Acid-Teledyne Leeman
- Manganese, 1000 µg/ml AA Standard in 3% Nitric Acid-Spectro Pure

6.0 Procedure

Solutions Preparation:

1. 2% Nitric Acid—Using a clean 100-ml graduated cylinder, add 57 ml of 70% nitric acid to 1000 ml of DI H_2O in a clean 2000-ml class A volumetric flask, dilute to volume, and mix. Store this solution in a 5000-ml reagent carboy.

2. Internal Standard Working Solution—Using a clean 100-ml graduated cylinder, add 57 ml of 70% Nitric Acid to 1000 ml of DI H_2O in a clean 2000-ml class A volumetric flask. Using a 100-ml beaker and the analytical balance, weigh out 61.02 g of gallium standard, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Using a 100-ml beaker and the analytical balance, weigh out 10.17 g of yttrium standard, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Dilute to volume, and mix. Store this solution in a 5000-ml reagent carboy. This Internal Standard Working Solution is 30 ppm gallium and 5 ppm yttrium in 2% nitric acid.

3. STD-1 (Blank)— Using a 250-ml beaker and the analytical balance, weigh out 201.82 g of Internal Standard Working Solution, and add it to a clean 2000-ml class A volumetric flask. Rinse the beaker twice with 2% Nitric Acid, and add the rinses to the flask. Dilute to volume with 2% Nitric Acid, and mix. Store this solution in a 5000-ml reagent carboy.

4. STD-2— Using a 250-ml beaker and the analytical balance, weigh out 201.82 g of Internal Standard Working Solution, and add it to a clean 2000-ml class A volumetric flask. Rinse the beaker twice with 2% Nitric Acid, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 2.124 g of GILES-4 Multi-element Stock Standard

Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 2.012 g of GILES-5 Multi-element Stock Standard Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Dilute to volume with 2% Nitric Acid, and mix. Store this solution in a 5000-ml reagent carboy.

5. STD-3— Using a 250-ml beaker and the analytical balance, weigh out 201.82 g of Internal Standard Working Solution, and add it to a clean 2000-ml class A volumetric flask. Rinse the beaker twice with 2% Nitric Acid, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 4.248 g of GILES-4 Multi-element Stock Standard Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 4.024 g of GILES-5 Multi-element Stock Standard Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Dilute to volume with 2% Nitric Acid, and mix. Store this solution in a 5000-ml reagent carboy.

6. STD-4— Using a 250-ml beaker and the analytical balance, weigh out 201.82 g of Internal Standard Working Solution, and add it to a clean 2000-ml class A volumetric flask. Rinse the beaker twice with 2% Nitric Acid, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 6.372 g of GILES-4 Multi-element Stock Standard Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 6.036 g of GILES-5 Multi-element Stock Standard Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Dilute to volume with 2% Nitric Acid, and mix. Store this solution in a 5000-ml reagent carboy.

7. STD-5— Using a 250-ml beaker and the analytical balance, weigh out 201.82 g of Internal Standard Working Solution, and add it to a clean 2000-ml class A volumetric flask. Rinse the beaker twice with 2% Nitric Acid, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 8.496 g of GILES-4 Multi-element Stock Standard Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Using a 25-ml beaker and the analytical balance, weigh out 8.048 g of GILES-5 Multi-element Stock Standard Solution, and add it to the flask. Rinse the beaker twice, and add the rinses to the flask. Dilute to volume with 2% Nitric Acid, and mix. Store this solution in a 5000-ml reagent carboy.

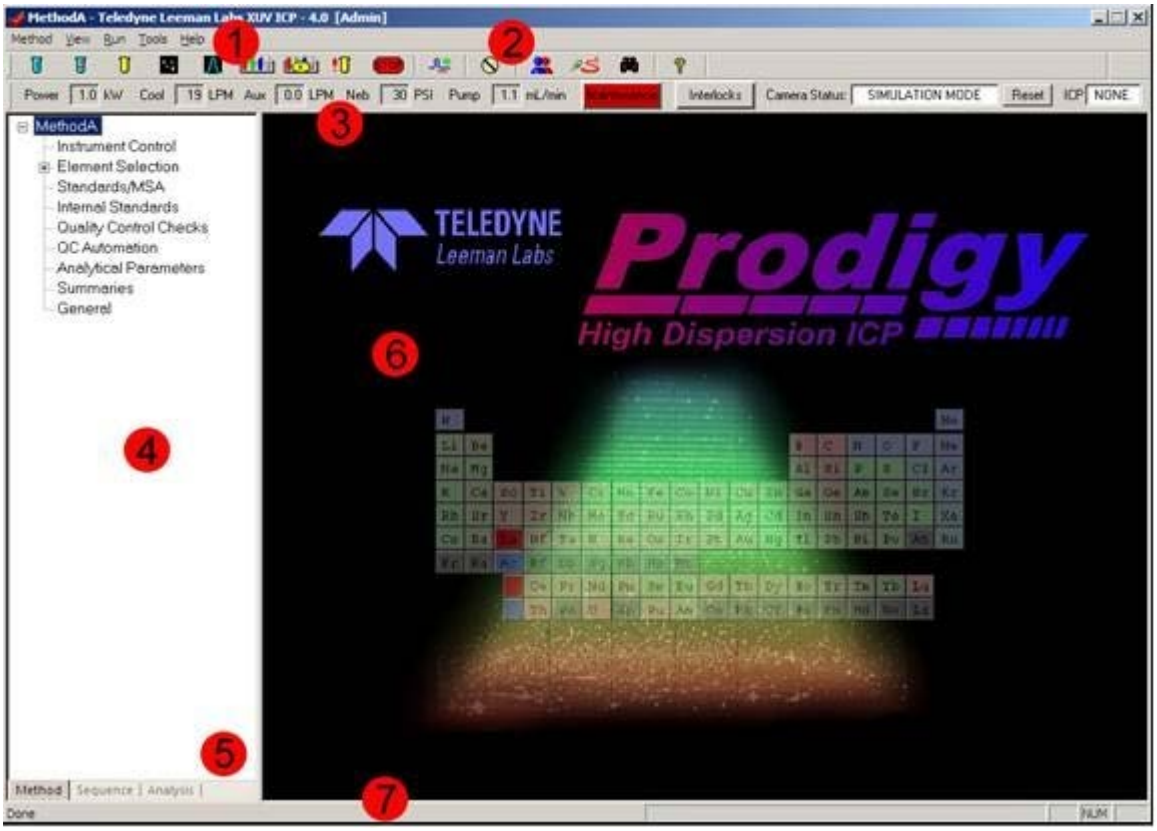
8. Mn Plasma Positioning Standard—Using an Eppendorf 5-ml adjustable pipette, add 1 ml of manganese standard to a 100-ml volumetric flask. Dilute to volume with 2% Nitric Acid, and mix.

9. As Wavelength Peaking Standard— Using an Eppendorf 5-ml adjustable pipette, add 1 ml of arsenic standard to a 100-ml volumetric flask. Dilute to volume with 2% Nitric Acid, and mix.

SALSA Software Overview

The SALSA program consists of three application areas: Method, Sequence and Analysis. Each of these three applications has specific functions and capabilities.

When the software is started, the following main screen is displayed:



On the screen you will find:

1. The Menu Bar with dropdown menus that provide access to all application functions in SALSA.
2. The Tool Bar containing picture ions that provide shortcut access to often-used functions.
3. The Instrument Conditions Bar where the current instrument control parameters (power, coolant flow, nebulizer flow, pump flow, etc) are displayed.
4. The Navigation Panel which provides access to features and applications in the software that get displayed in the Display Panel.
5. Navigation Tabs which allow the user to navigate between application areas (Method, Sequence, and Analysis).
6. The Display Panel which actively displays the feature selected in the Navigation Panel.
7. A Status Bar which displays text in the lower left of the screen to indicate the operation currently being performed.

ICP Test Procedure

1. Go down to the QA storage area below the lab and make sure that argon and nitrogen are flowing to the instrument, and turn on the water recirculator.

2. If the instrument is turned on (green power button is lit), purged with nitrogen, the SALSA software is open, and the camera is chilled (the ‘Camera Status’ in the upper, right corner reads ‘Connected (-40°C)’), then skip to step 18.

3. If the instrument is turned on (green power button is lit), purged with nitrogen, the SALSA software is open, and the camera is not chilled (the ‘Camera Status’ in the upper, right corner does not read ‘Connected (-40°C)’), then skip to step 13.

4. If the instrument is turned on (green power button is lit), purged with nitrogen, and the SALSA software is off, then skip to step 14.

5. If open, close the SALSA software and then power up the ICP by pressing the green power button on the front.

6. Turn on the computer (if necessary), and open the SALSA software (shortcut on desktop with chili pepper icon).

7. After the software loads, a dialog box saying, “opening method lines updating from library” will appear. Click [OK].

8. Another dialog box will appear asking, “has camera purged for recommended period of time before turning chiller on?” Click [NO], and a dialog box will appear saying, “camera cooler not turned on! Exit software and restart after recommended camera purge period is completed.” **NOTE: If the chiller is turned on without the cameras having been purged for the recommended period of time, ice crystals can form on both cameras’ detector chips. This can damage the cameras, so never click [YES] if the instrument has not purged for the recommended period of time.**

9. Click on the Method Navigation Tab, and then the ‘Instrument Control’ feature in the ‘Method’ feature list in the Navigation Panel. The Instrument Control Panel should appear in the Display Panel.

10. Set the purge to high by clicking the [High] button in the ‘Purge’ section of the Spectrometer Control Area on the Instrument Control Panel.

11. Allow the optics to purge for two hours before proceeding further, or four hours if the nitrogen was off for more than one hour.

12. After the specified purge time, set the purge to low by clicking the ‘Low’ button in the ‘Purge’ section of the Spectrometer Control Area on the Instrument Control Panel.

13. Close the SALSA software.

14. Open the SALSA software (shortcut on desktop with chili pepper icon).

15. After the software loads, a dialog box saying, “opening method lines updating from library” will appear. Click [OK].

16. Another dialog box will appear asking, “has camera purged for recommended period of time before turning chiller on?” Click [YES].

17. Allow the camera chiller to reach -40 °C (this will take approximately five minutes).

18. Open the “USP1” method, if not already opened, by clicking [Method] on the Menu Bar, [Open] on the drop down menu, and selecting the “USP1” method.

19. A dialog box saying, “Opening Method Lines Updating from Library” will appear. Click [OK].

20. Look at the Instrument Conditions Bar to see if the [Maintenance] button is red. If the [Maintenance] button is not red then skip to step 26.

21. Click on the red [Maintenance] button, and the Scheduled Maintenance dialog box will appear listing all routine maintenance tasks.

22. Any maintenance task marked with a red X will need to be addressed. Double click on the first task requiring competition.

23. Complete the task by following the instructions provided in the task's Maintenance Wizard, but only if the task does **not** require instrument shutdown. If the task requires instrument shutdown, click [Cancel], and complete the task after all ICP use has finished for the day.

24. When a task is completed, click [Finish] in the task's Maintenance Wizard.

25. Double click on the next task requiring completion, and repeat steps 23 and 24. Do this until all maintenance tasks are completed, or require instrument shutdown.

26. Click on the Method Navigation Tab, and then the 'Instrument Control' feature in the 'Method' feature list. The Instrument Control Panel should appear in the Display Panel.

27. Click on the red [Interlocks] button in the Instrument Conditions Bar, and the Instrument Panel dialog box will appear.

28. Except for the "Air knife" and "Argon high" interlocks, if there are any interlock errors (indicators next to the interlock messages are red instead of green), they will have to be addressed before the plasma can be lit. If there is an "Argon low" interlock error proceed to step 29. If there is no "Argon low" interlock error skip to step 30.

29. Increase the flow of argon to the instrument by opening the pressure building valve on the argon tank until the "Argon low" interlock error goes away (turns green). If this is not sufficient the argon regulator may need to be adjusted, or the argon tank replaced.

30. Once all interlock errors have been addressed, except for "Air knife" and "Argon high", close the Instrument Panel dialog box.

31. Check the autosampler rinse bottle of 2% nitric acid behind the autosampler, to make sure that there is enough rinse acid. Add more, if required. When reattaching the cap to the bottle be sure to leave it a little loose, so that air can enter the bottle.

32. Turn on the ICP exhaust vent by flipping the switch on the wall above the autosampler. This switch should light up when turned on.

33. Clamp the peristaltic pump pressure bars onto the three pump tubes (sample uptake, drain, and autosampler rinse).

34. Click on the [Auto Start] button in the Plasma Control Area of the Instrument Control Panel in the Display Panel. In a few seconds the argon plasma torch should light. In a few more seconds the peristaltic pump should turn on to begin the aspiration of 2% nitric acid rinse. If the torch goes out, make sure there is enough argon pressure, and relight. If it continues to go out, the torch may need to be cleaned or replaced.

35. Allow the instrument to warm up for 30 minutes.

36. Place 9 standard autosampler tubes (50-ml) into the 14 position rack at positions 1-7, 13, and 14.

37. Fill tube one with STD-1 (blank), tube two with STD-2, tube three with STD-3, tube four with STD-4, tube five and six with STD-5, tube seven with deionized water, tube thirteen with As wavelength peaking standard, and tube fourteen with Mn plasma positioning standard.

38. Place the 14 position rack, with the nine filled standard autosampler tubes into the standards rack position (furthest left) of the autosampler.

39. After the plasma has warmed up, select standard tube 14 using the drop-down lists in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

40. Move the autosampler to standard tube 14 (Mn plasma positioning standard) by clicking on the ‘Move Tip To: [Cup]’ button in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

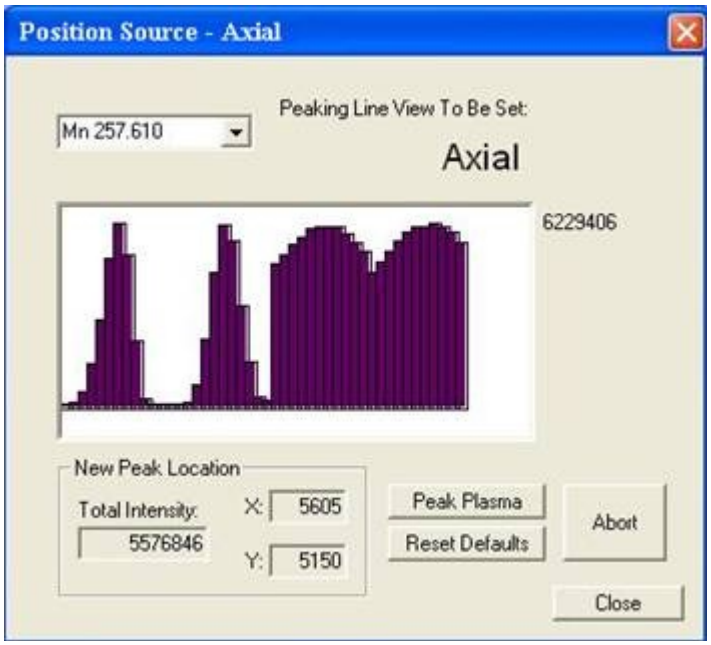
41. Click on the [Position Plasma] button in the Plasma Control Area of the Instrument Control Panel (in the Display Panel). The Position Plasma dialog box will appear.

42. Select “Mn 257.610” from the drop-down list.

43. Wait 35 seconds for sample uptake.

44. Click the [Peak Plasma] button, and the positioning routine begins.

45. Once completed, a purple histogram will be displayed in the Position Plasma dialog box, and an intensity will be displayed at the top right-hand corner of the histogram. The numerical intensity will vary from instrument to instrument (and from wavelength to wavelength). However, the histogram pattern should look similar to the one displayed below:



46. Click on the [Close] button to close the Position Plasma dialog box.

47. Click the 'Move Tip To: [Rinse]' button in the Autosampler Area of the Instrument Control Panel (in the Display Panel). This will send the autosampler probe back to the rinse location.

48. Allow the system to rinse for 40 seconds.

49. Select standard tube 5 using the drop-down lists in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

50. Move the autosampler to standard tube 5 (STD-5) by clicking on the 'Move Tip To: [Cup]' button in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

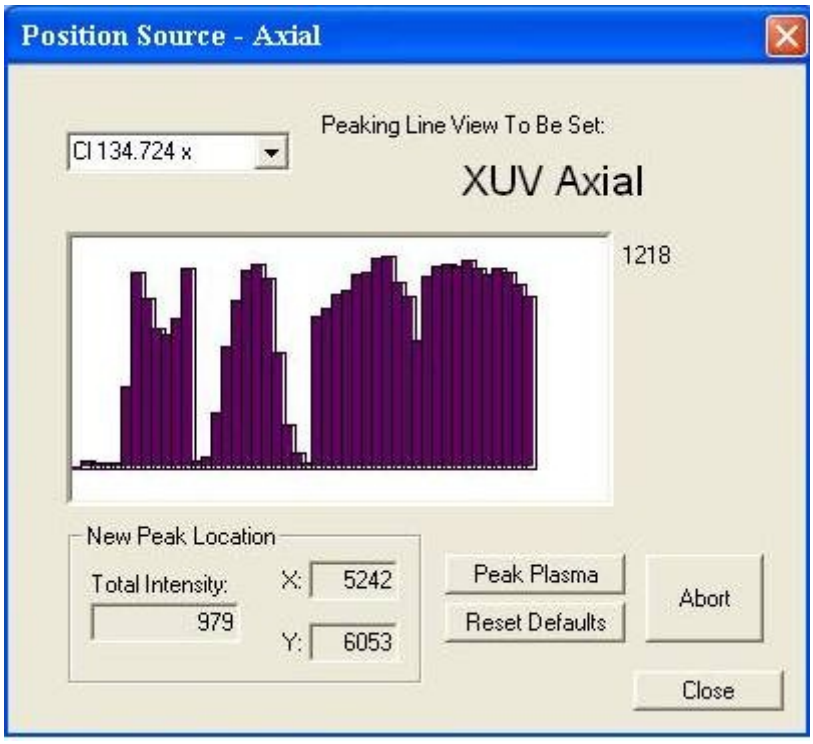
51. Click on the [Position Plasma] button in the Plasma Control Area of the Instrument Control Panel (in the Display Panel). The Position Plasma dialog box will appear.

52. Select “Cl 134.724 x” from the drop-down list.

53. Wait 35 seconds for sample uptake.

54. Click the [Peak Plasma] button, and the positioning routine begins.

55. Once completed, a purple histogram will be displayed in the Position Plasma dialog box, and an intensity will be displayed at the top right-hand corner of the histogram. The numerical intensity will vary from instrument to instrument (and from wavelength to wavelength). However, the histogram pattern should look similar to the one displayed below:



56. Click on the [Close] button to close the Position Plasma dialog box.

57. Click the 'Move Tip To: [Rinse]' button in the Autosampler Area of the Instrument Control Panel (in the Display Panel). This will send the autosampler probe back to the rinse location.

58. Click on the Autoalign Icon  in the Tool Bar. This will open the Auto Wavelength Alignment dialog box.

59. Look at the wavelength list, and make sure that the mercury reference line ("Hg Ref") is checked in the left column.

60. Click the [Auto Align] button. The software will automatically align the Hg 253.653 nm reference line.

61. When the alignment is complete, an [Accept] button will appear under the [Auto Align] button. Click the [Accept] button. This will cause all other wavelengths in the wavelength list to no longer be grayed out.

62. Using the 'Standards' drop-down list, select STD-5.

63. Look at the wavelength list, and make sure that all of the wavelengths except for "Hg Ref" and "Mn 257.610" are checked in the left column.

64. Click the [Auto Align] button, and wait for the software to automatically align the element lines that are checked in the left column. When the alignment is complete the table will fill with dX, dY and time values for each line.

65. Make sure that all wavelengths to be accepted are checked in the left column. If the dX and dY values for As 193.759 are not both zero, then the As 193.759 line should **not** be accepted (checked). If the auto alignment produced any dX and/or dY value of 3 (written in red text) then the subarray movement is too large. These lines should **not** be accepted (checked), and the wavelengths should be manually aligned.

66. Click the [Accept] button.

67. A dialog box saying, "Opening Method Lines Updating from Library" will appear. Click [OK].

68. Click the [Close] button to close the Auto Wavelength Alignment dialog box. If the As 193.759 line was accepted, skip to step 84. If it was not accepted, precede to step 69.

69. Select standard tube 13 using the drop-down lists in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

70. Move the autosampler to standard tube 13 (As wavelength peaking standard) by clicking on the 'Move Tip To: [Cup]' button in the Autosampler Area of the Instrument Control Panel in the Display Panel.

71. Click on the Acquire Full Spectrum Icon  in the Tool Bar. This will open the Run Full Frame Image dialog box.

72. Enter an appropriate name for the echellogram to be acquired.

73. Enter an exposure time of 60 seconds, and leave the XUV time at one second.

74. Uncheck the 'Extended Range' checkbox.

75. Click [OK] to acquire the echellogram.

76. When the Status Bar says that the instrument is done acquiring the echellogram, click the 'Move Tip To: [Rinse]' button in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

77. Highlight the As 193.759 wavelength under the 'Element Selection' feature in the Navigation Panel on the Method Tab.

78. Select the Align Wavelength tab in the Display Panel.

79. Select the echellogram that was just acquired (top image) from the Echelle Images table.

80. To align the subarray, click the [Auto] button on the upper right-hand side of the window. If the wavelength is out of alignment, the subarray should move to position itself.

81. If the subarray did not move to the correct (aligned) position, click the [Cancel] button and use the arrow buttons on the right-hand side of the screen to move the subarray to the desired location. These arrow buttons can be used at any time to move the subarray manually.

82. Check the alignment by moving the subarray one position, and then back in each direction and observing the intensity (bottom right). The intensity should be the highest at the original alignment position, if the subarray is correctly aligned.

83. Once the subarray has been moved to the correctly aligned position, click the [Accept] button to accept the change. NOTE: the [Accept] and [Cancel] buttons only appear if the subarray has moved.

84. All USP samples to be tested should be prepared according to Giles procedure USP ICP-OES Sample Preparation L13-PR-100-058.

85. Place all 15-ml centrifuge tubes (containing the prepared samples to be tested) into a 44 position autosampler rack (with the bottom removed to accommodate the tubes) located next to the standards rack in the autsampler.

86. Highlight the 'QC Automation' feature in the Navigation Panel on the Method Tab.

87. Make sure that all eight QC automation checkboxes are checked (turned on) in the Display Panel.

88. Click on the Analysis Navigation Tab.

89. Click [Analysis] on the Menu Bar.

90. Select 'New…' from the drop-down menu. The New Analysis Chapter dialog box will appear.

91. Enter the date and "USP Testing" in the Analysis Chapter Name field.

92. Click the [OK] button to close the dialog box and create the new analysis chapter.

93. Click on the Sequence Navigation Tab.

94. Enter the date and sample name for each sample next to the appropriate 'Cup' position under the 'Sample ID' column.

95. Enter the original weight of each sample before preparation (usually 3 g) into the 'Wt.' column.

96. Enter the final volume of each sample after preparation (Usually 15 ml) in to the 'Vol.' column.

97. Click the [Update] button to enter the samples into the sequence. The newly entered samples will appear in the Navigation Panel.

98. Click the [Run Sequence] button. The instrument and autosampler will run the calibration standards, samples, and quality control checks in the sequence displayed in the Navigation Panel.

99. Once the calibration standards have finished running, check the calibration for each element by highlighting each wavelength under 'Element Selection' in the Navigation Panel on the Method Tab, and clicking on the 'Calibration' tab. Rerun the calibration if necessary.

100. Click on the Analysis Navigation Tab.

101. Click on the 'Results' tab in the Display Panel. This will list all calibration standards, samples, and quality control checks that have been tested, and will update in real time.

102. After each quality control check has finished running, make sure that they pass for all elements by highlighting them in the Navigation Panel. If a quality control check has failed for any wavelength an (L) for low failure, or an (H) for high failure will appear next the concentration value for the wavelength under the 'Conc.' column in the Display Panel. If a quality control check fails, stop the run by clicking the red STOP button in the Tool Bar, recalibrate the instrument, and rerun the quality control check. If the quality control check passes, rerun all samples associated with the failed quality control check.

103. Once all samples have been tested, and all quality control checks (before and after each set of samples) have passed for all wavelengths, click on the 'Report' tab in the Display Panel.

104. In the Navigation Panel, check the checkboxes for each sample and quality control check that is to be reported.

105. Click the [Load] button, located on the right-hand side in the Report Spec Area. The Select Report Spec dialog box will appear.

106. Highlight the "Simple SDB" report spec.

107. Click the [OK] button.

108. Make sure that all elements required for the report are selected (checked) in the 'Line' drop-down list.

109. Click the [Format 2] button in the Report Area.

110. Review the report at the bottom of the Display Panel to make sure it is correct.

111. Click the [Printer] button in the Output Area. The Report Title dialog box will appear.

112. Type “USP Testing” and the date in the report title field.

113. Click the [OK] button, and the Print dialog box will appear.

114. Make sure that the printer has enough paper, and the paper output tray is open.

115. Click the [Print] button, and the report will print.

116. Determine if all samples pass USP Specifications. In order to pass USP specifications the following four things need to be true:

    a. The chloride concentration needs to be 140 ppm or lower.
    b. The iron concentration needs to be 20 ppm or lower.
    c. The sum of the concentrations of arsenic, lead, mercury, and cadmium needs to be 10 ppm or lower (this represents total heavy metals).
    d. The selenium concentration needs to be 30 ppm or lower.

117. Place the report into the USP Laboratory Notebook, and sign and date it.

118. Make a note in the laboratory notebook about which samples pass USP specifications, and which samples don’t. If a samples does not pass USP specifications, make a note as to why.

119. Allow the sample introduction system to rinse with the 2% nitric acid for 15 minutes.

120. Click on the Method Navigation Tab, and then the ‘Instrument Control’ feature in the ‘Method’ feature list. The Instrument Control Panel should appear in the Display Panel.

121. Select standard tube 7 using the drop-down lists in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

122. Move the autosampler to standard tube 7 (deionized water) by clicking on the ‘Move Tip To: [Cup]’ button in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

123. Allow the sample introduction system to rinse with deionized for 10 minutes.

124. Press the [Air] button in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

125. Allow the sample introduction system to drain.

126. Click the [Extinguish] button in the Plasma Control Area of the Instrument Control Panel (in the Display Panel).

127. Once the plasma is extinguished, Press the [Rinse] button in the Autosampler Area of the Instrument Control Panel (in the Display Panel).

128. Unclamp the peristaltic pump pressure bars.

129. Turn off the ICP exhaust vent by flipping the switch on the wall above the autosampler.

130. Complete any maintenance tasks that were to be completed after all ICP use had finished for the day.

131. Turn the instrument back on (green button of front) when finished.

132. Go down to the QA storage area below the lab and turn off the water recirculator.

133. Check the argon and nitrogen tanks to see if they are getting low, and need to be replaced.

134. Allow the plasma torch to cool for 15 minutes.

135. Open the outer door on the front of the ICP. This allows access to the torch box.

136. Detach the Coolant and Auxiliary tubes from the gas fittings (lower left in the torch box), by squeezing each quick release and disengaging each tube.

137. Disconnect the spray chamber from the torch at the ball joint, by holding the spray chamber and loosening the screw-locking pinch-clamp.

138. Carefully place the spray chamber in a safe position.

139. Open and remove the inner door, allowing access to the torch.

140. Hold the torch near the ball joint, and loosen the white thumbscrew.

141. Pull the white thumbscrew down to release the restraining clamp.

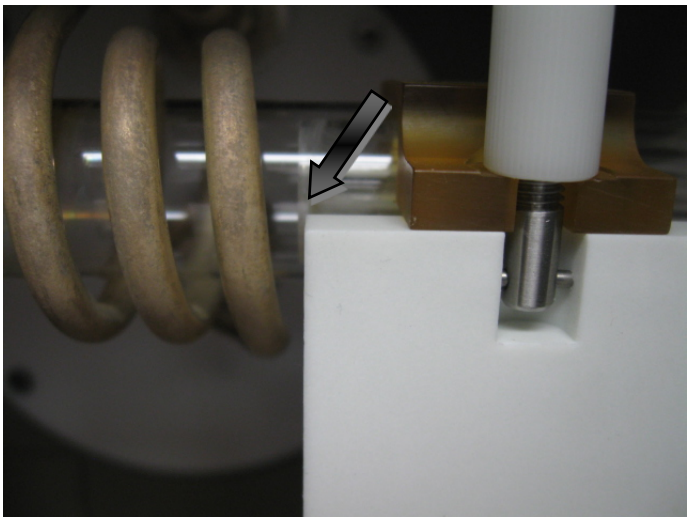
142. Remove the torch by moving it to the right, through the load coil and out of the torch mount.

143. Clean the torch of all salt deposits using deionized water (sonicate if necessary).

144. Dry the torch and gas tubes using compressed air.

145. Reinstall the torch by placing it back into the torch mount and through the load coil.

146. Make sure that the torch is positioned with the auxiliary tube (the middle tube between the outer tube and the injector) sticking out approximately one millimeter past the white mounting block, as shown below:



147. Engage the restraining clamp by flipping it over the torch and pushing the white thumbscrew up to secure the clamp.

148. Hand-tighten the white thumbscrew until the torch is secure.

149. Reattach the Coolant and Auxiliary tubes to the gas fittings. The piece of tubing on the left (closest to the load coil) is the coolant tube, and needs to be plugged into the Coolant gas fitting. The piece of tubing on the right (closest to the ball joint) is the auxiliary tube, and needs to be plugged into the Auxiliary gas fitting. **NOTE: If the tubes are connected to the wrong gas fittings the torch will melt when the plasma ignites, which can damage the load coil.**

150. Reattach the spray chamber to the torch by holding the spray chamber in place, placing the screw-locking pinch-clamp over the ball joint, and tightening it. Make sure that the spray chamber is positioned with the nebulizer tubing pointing up and towards the front at a 45° angle.

151. Replace and close the inner door.

152. Close the outer door.

7.0 Reference Documents

USP ICP-OES Sample Preparation (L13-PR-100-058)

8.0 Change Information

Updated procedure to use a two-part multi-element standard, because the one-part standard originally used was unstable.