

Status: Final

Approved by:

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Technical SOP for Metals Analysis using Perkin-Elmer Optima 7300 ICP-OES

1. SCOPE

This SOP is intended as a technical guide for the operation of the Perkin-Elmer Optima 7300 ICP-OES with attached Elemental Scientific FAST 2DX auto-sampler and describes the technical steps and procedures involved in optimization and sample analysis using the Syngistix software to control the ICP and associated autosampler. Use of this method is restricted to use by analysts appropriately experienced and trained in metals analysis. Each analyst must also demonstrate the ability to generate acceptable results with this method.

2. RESPONSIBILITIES

ECL Staff: Responsible for creating and saving run data used to generate lab data using Syngistix software.

3. PROCEDURE

3.1. Instrument Warm-up and Daily Maintenance

Note: Maintenance required on an as-needed basis (such as the removal and cleaning of the torch and nebulizer) is described in “Metals by ICP AES” DCN:03.6010.00.

3.2. Daily Maintenance Procedures (prior to Warm-Up)

- 3.2.1. Read the most recent entry in the ICP Users Log (the ECL Lab Book in Room 172) for any issues with the previous analytical run. This users log is how issues and maintenance of the ICP can be easily communicated between multiple users.

Note: It is important to keep this log updated with every new analytical run and every piece of maintenance completed on the instrument.

- 3.2.2. Check the liquid Argon supply to ensure there is sufficient Argon for the analysis and that the gas is flowing to the instrument.
- This is done by either examining the float located at the top of the cylinder and/or carefully rocking the cylinder to test the liquid level.

- If there is insufficient Argon for the day's analysis, close any open applications on the computer, power-down the instrument using the breaker switch on the right side of the instrument, close the tank and switch the regulator to the backup tank, then turn the power on. Re-open the ESI SC/FAST application prior to opening Syngistix. Reorder Argon by contacting the Quality Assurance and Technical Support Unit.
- 3.2.3. The chiller should hold the temperature at 15(±1)°C and between 45-60 psi of pressure. The Syngistix software will sound an alert if the chiller fails to maintain these criteria.
- If needed, adjust the temperature using the knob adjacent to the temperature read-out.
 - Adjusting the chiller pressure requires opening the chiller casing and should be performed by a qualified service technician.
- 3.2.4. Check the waste container (under the instrument) to ensure adequate volume remains for the intended sequence. Empty waste containers weekly.
- 3.2.5. Check that there is sufficient internal standard solution and carrier solution for the entire analytical run. If not, prepare fresh carrier solution and internal standard solution as described in "Metals by ICP AES" DCN:03.6010.00.

Note: If you run out of internal standard solution mid-run, you will need to recalibrate the instrument and restart the entire analytical run. As a result, be prepared to add carrier solution in the event the bottle runs low. In addition, if the internal standard runs low, you may top off the bottle if the added internal standard solution is the same batch as the solution used to calibrate.

- 3.2.6. Check the torch and injector for cleanliness. (See "Metals by ICP AES" DCN:03.6010.00 for an example of a dirty torch and injector.)
- Open the torch box by pulling out and down on the release to the left of the viewing window.
 - Inspect the injector and torch for cleanliness; if either shows signs of build-up or staining, remove and clean them as described in "Metals by ICP AES" DCN:03.6010.00. Replace both components with clean components before continuing.
 - If the torch is removed, the IEC model must be reconfirmed or rebuild. See "Metals by ICP AES" DCN:03.6010.00 for the procedure.
 - Examine the RF coil for deposits and ensure that it is clean and dry.
 - Close the torch box carefully and ensure it is sealed. The plasma will not ignite if the torch box does not close correctly.

3.2.7. Inspect that the sample introduction system is correctly connected and all tubes appear to be clean. (See Figure 1 for a diagram of the sample introduction system).

- Examine the nebulizer and spray chamber for any evidence of build-up or other contamination. If either is dirty, remove and clean (see “Metals by ICP AES” DCN:03.6010.00) before igniting the plasma and beginning analysis.
- Check the peristaltic pump tubing on the instrument for discoloration or flat spots between the colored tabs by rolling the tube gently between thumb and forefinger.
 - If there are flat spots or discolorations, replace the pump tubing.
 - In general, sample and IS pump tubing should last for approximately 10-16 hours of analysis time.
 - Waste tubing only needs to be changed as a result of notable flat spots or reduction in waste flow.
 - The waste tubing has two grey tabs, is not transparent, and belongs in the slot on the peristaltic pump closest to the instrument. The IS tubing has one green and one orange tab. The carrier solution tubing has two black tabs. The carrier solution and the internal standard tubing occupy the outermost two slots on the peristaltic pump.
- Check the rest of the tubing associated with the sample introduction system for any evidence of staining, depositions, or other potential contamination or damage. This includes:
 - The probes and attached tubing
 - The tubing to/from the FAST valve
 - The mixing tee and outflow tubing to the nebulizer
 - If any of the tubing appears to be discolored, blocked, or damaged, replace the tubing. The internal standard mixing tee can be back-flushed with the fittings found in the consumables kit.

3.2.8. Properly hook the peristaltic pump tubing around the pump rotor using the hook points adjacent to the rotor.

- The peristaltic pump on the instrument runs **CLOCKWISE** meaning that all samples will flow *in from the right side* and flow *out on the left side* of the rotor.
- Loop and tension the carrier, IS, and waste tubing into place around the peristaltic pump as follows:
 - Carefully hook the tab on the “inflow” end of the peristaltic pump tubing in the slot on the right side of the rotor.
 - Loop the tubing under and around the rotor.
 - Gently stretch the tubing to hook the tab on the “outflow” side into the slot of the left side of the rotor.

- Lift the locking arm under the rotor into place to verify it will cover the tubing; if so, lock it in place. Otherwise, release the tension on the tubing and adjust the positioning.

3.2.9. The instrument is now ready for warm-up.

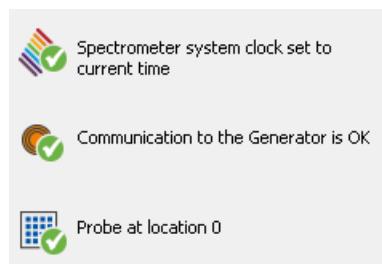
3.3. Instrument Warm-Up Procedures

3.3.1. Initialize the Instrument

- If necessary, log into the computer using the username “Administrator” and the password “Icp7300”.
- Ensure the ESI SC/FAST software is running
- Start the Syngistix software by clicking on the <Syngistix for ICP> icon on the computer desktop.

Note: It is critical that the ESI SC/FAST application is running prior to starting the Syngistix application.

- The Status Panel on the right side of the application will indicate startup progress.
 - If the system is working properly, all three systems will receive a green check mark.

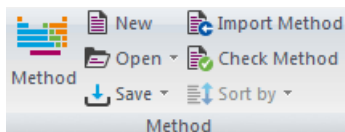


- If there is a problem in one of the subsystems, it will have a red “X” next to it in the Status Panel. Double click on the appropriate icon (Spectrometer, Plasma, or Autosampler) in the Status Panel to open the diagnostics window and see the error message. Correct any indicated problems.
 - This window can be accessed at any time by clicking on <Diagnostics> under the **Instrument** menu.
- Once the diagnostics are successful, the instrument will begin to warm-up.
 - Generally the instrument warm-up takes less than 5 minutes.
 - If the instrument has not had Argon flow to the detector for any length of time, this warm-up can take up to 90 minutes to remove moisture and air from the detector.

3.3.2. Open the instrument workspace and set the system for the correct analytical method

- Click on the <Syngistix Icon> button in the upper left corner of the application.

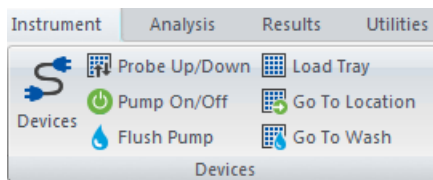
- Select a workspace file (e.g. default) to open that workspace.
- Open the Analysis menu and click the <Open> button in the **Method** ribbon.



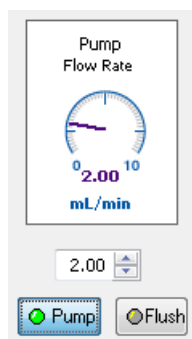
- Select the appropriate method file (e.g. 6010 Low – 22AUG2017) and click <OK>

3.3.3. Activate the peristaltic pump to check solution flow through the sample introduction system.

- Open the **Instrument** menu and turn the peristaltic pump on with the *Pump On/Off* button. This will open the *Plasma Control* window.



- In the *Plasma Control* window, verify the pump is activated and the speed is set to 2.00. A gray indicator in the window will change to green, indicating the pump is active.



- Allow solution to flow until the tubing is full.
- Adjust the tension on the peristaltic pump tubing for optimal solution flow.
 - Introduce an air bubble into the line by removing the sample probe (or weighted inlet) from the solution until the air bubble can be seen in the line.
 - Loosen the control screw in the locking arm until the air bubble STOPS moving through the tube by turning counterclockwise.
 - Slowly retighten the control screw until the air bubble moves smoothly through the tube.
 - Once the solution is moving smoothly, tighten the control screw ¼ turn further for optimal flow.

Note: If the internal standard pump tubing is not full of solution when the flow rate and pump tension are adjusted, the internal standard flow rate may not be set correctly due to the narrowness of the tubing.

- Turn off the peristaltic pump by again clicking the **<Pump>** button in the Plasma Control window. The green indicator should become gray.

3.3.4. Ignite the Plasma

- Click the **<Plasma Off/On>** button in the Plasma Control window to initiate plasma ignition. The button should switch to the “on” position.



- Wait for the plasma to ignite. If the plasma will not ignite or ignites, but will not stay lit:
 - Ensure there is sufficient Argon in the tank and that the pressure is in the correct range (80-120 psi) on the regulator.
 - Check the sample introduction system to ensure all connections are made and air is not leaking into the system.
 - If the torch, injector, or sample chamber/nebulizer were removed prior to ignition, check that all o-rings are in good condition and that all parts have been seated correctly in their holders.
- Once the plasma is lit, allow the plasma to stabilize for 30 minutes prior to continuing. You may use DI during the stabilization period in order to conserve the internal standard and carrier solutions.

3.3.5. Begin filling out the ICP User Log.

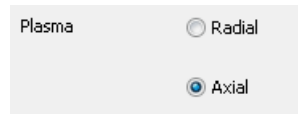
- Begin the log entry with the date, analyst name, time the plasma was ignited, and a brief description of the samples that will be run.
- When the plasma is extinguished, record the time in the same log.
- Write a description of any issues that arose during analysis, such as frequent QC failures, reporting limit changes, plasma flame-outs, problems with tubing, etc.
- Indicate if any parts were replaced prior to or after analysis and what cleaning or maintenance needs to be conducted before the next analytical run.

3.4. Daily Optimization

3.4.1. If the torch was removed or replaced, realign the x-y position of the torch relative to the spectrometer windows.

- Open the Spectrometer Control window (if not already open) by clicking on **<Spectrometer Control>** under the **Instrument** menu.

- Realign the axial view.
 - Select the **<Axial>** setting under **<Manual Settings, Plasma>** in the right side of the Spectrometer Control window.



- Click on the **<Align View>** button on the bottom left corner of the window. This will open the Align View window.
 - Ensure the wavelength is set for **Mn 257.610** and the delay time is set to **0** in the Align View window.
 - Place the sample probe into a solution containing 1 ppm Mn and allow the solution to be drawn up for ~ 1 minute.
 - Click **<Align>** in the Align View window to begin the alignment.
- Realign the radial view.
 - Select the **<Radial>** setting under **<Manual Settings, Plasma>** in the upper right side of the Spectrometer Control window.
 - Click on the **<Align View>** button on the bottom left corner of the window. This will open the Align View window.
 - Ensure the wavelength is set for **Mn 257.610** and the delay time is set to **0** in the Align View window.
 - Place the sample probe into a solution containing 10 ppm Mn and allow the solution to be drawn up for ~ 1 minute.
 - Click **<Align>** in the Align View window to begin the alignment.
- Record the maximum intensity for both the axial and radial alignments in the Daily Optimization worksheet.

3.4.2. Run the Hg realignment to correct any detector wavelength shifts.

- Click the **<Hg Realign>** button in the Spectrometer Control window to start the process. This is an automated process that takes ~2 minutes to complete.

3.4.3. After calibration, record the following in the Daily Optimization worksheet:

- The RSD for the internal standard in the calibration blank

Note: The RSD for the internal standard must be less than 2% in the blank. If it is not, check and adjust the tubing and pump tension and rerun the calibration blank until the RSD meets the criteria.

- The nebulizer back pressure from the calibration blank
- The intensities for two elements (commonly Sb and Pb) in both the mid-level standard and the blank

Note: Changing the torch and/or the x-y position of the detector can cause significant shifts in signal intensity; however, these recorded values can be used to monitor changes in signal between runs using same torch.

4. ANALYTICAL PROCEDURE

4.1. Sample Preparation

- 4.1.1. Begin preparation by starting a new page in the ICP-AES Analytical Run Log Book. Enter all sample dilution and post digestion spiking information in the log.
- 4.1.2. Prepare the samples for analysis, including any necessary dilutions and post digestion spikes. The sample preparation for WET samples is described in detail in “Metals by ICP AES” DCN:03.6010.00, Appendix E.

Note: Any sample whose concentration exceeds the highest calibration standard by more than 10% for any reported element or exceeds 300 ppm Fe must be diluted such that the concentration falls within the calibration range or below 300 ppm Fe.

- 4.1.3. Place the prepared samples in the autosampler rack.

4.2. Sample Information Entry

4.2.1. Enter the sample information.

- Click the <New> button in the Sample Information ribbon found in the **Analysis** menu. Select “ESI-prepFAST.sidx” and click <OK>.



- The <Batch ID> shall be set as the date of the run, using two integers for the day, three letters for the month and four integers for the year (i.e. 08 SEP 2017).
- List the sample preparation batch numbers in the <File description> field. (i.e. “3050-245-1”).
- In the <A/S Loc> column, enter the locations of the samples using “101” for the front left position of the leftmost autosampler rack (Rack 1). If the additional rack is required, those positions begin with the number “301” being assigned to the front left position.
- In the <Sample ID> column, enter the ECL number of the sample, QC label, its dilution factor and other pertinent information (e.g. BB00879 10x PS or BB00888 LCS 10x).

4.2.2. Optional Sample Information:

Note: The following data entry is not currently being used for the Perkin-Elmer Optima 7300. The instructions are included in case future analyses require the use of these features.

- For samples digested from solid material:
 - Enter the mass of the original solid sample in grams (g) in the <Initial Sample Wt.> column.
 - Enter the final volume at the end of the digestion and dilution in milliliters (mL) in the <Sample Prep Vol> column.

The resulting values produced by the analysis will automatically be corrected for the mass of the initial solid sample and the dilution inherent in the digestion, yielding results in $\mu\text{g/kg}$ soil.

- For dilutions of other samples:
 - Enter the volume of original sample used in milliliters (mL) in the <Aliquot Volume> column.
 - Enter the final volume after dilution in milliliters (mL) in the <Diluted to Vol.> column.

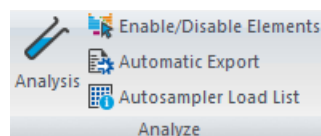
The resulting values produced by the analysis will automatically be corrected for the dilution of the samples.

4.2.3. Save the sample information file.

- Click the <Save> button in the Sample Information ribbon.
- Enter a filename using the pattern Date-Month-Year (i.e. 14Sept2011). If multiple analytical runs occur on the same date, append the next letter to the end of the date (i.e. 14Sept2011b).
- Click <Save> to save the file.
- Write the sample information file name in the ICP-AES Analysis Log Book.

4.3. Calibration

- 4.3.1. For normal instrument operation, prepare the calibration standards and place in the autosampler as described in “Metals by ICP AES” DCN:03.6010.00.
- 4.3.2. Set up the Automated Analysis Control to the correct sample information and results files.
 - Click the <Analysis> icon to open the Analysis window. Select the [Automated] tab at the top of the window if it is not already active.



- Verify the appropriate method is selected in the queue. If not, double-click the first cell under “Method” and select the appropriate method.
 - Look at the file name listed below “Use Sample Info”. If it is not the same as the desired sample information file, click on the <Open> button (it looks like a folder within the field) and select the desired file name. Click <Open> to load the file.
 - Click on the <Open> button next to <Results Data Set Name>. Enter a name identical to the batch ID used in the *Sample Information Editor* window, list the batch numbers in the “Description” field and click <OK>.
 - Make sure the boxes next to “Print log during analysis”, “Use Sample Info” and “Save data to Results Data Set” are checked; if not, check the boxes.
 - Make sure all samples and QC appear in the sample list. If they do not, click the <Rebuild List> button.
 - Write the results file name in the ICP-AES Analysis Log page.
- 4.3.3. Start the calibration by clicking the <Calibrate> button.
- 4.3.4. After calibration is complete, review the calibration results to ensure all QC criteria are met. If not:
- Fix any instrument issues causing calibration problems
 - Refill or remake any problematic calibration solutions
 - Clear the calibration blank (by clicking on <Clear> under the **Results** menu)



- Recalibrate the system

4.4. Sample Analysis

- 4.4.1. Click on <Analyze Samples> to start the sample analysis. It will begin with the first QC sample listed in the method and proceed from there through the end of the run.
- 4.4.2. Monitor the results of the automated quality control cycle every 10 samples. If an error is encountered, stop the analysis. Correct any errors that arise as described in “Metals by ICP AES” DCN:03.6010.00.
- If there is a need to restart the analytical run after a QC failure, click <Analyze Sample>. Select <Continue with Sequence #:> and enter in the number (from the

run list) of the QC sample that needs to be rerun. Click **<OK>** to restart the analysis at that point.

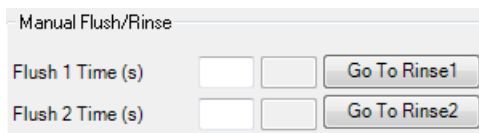
4.4.3. After the sample run is complete, print out the run list by clicking <Print List> in the Analysis window.

*Note: An uninterrupted calibration and sample analysis cycle can be activated by clicking the **<Analyze All>** button in the Analysis window; however, there is no pause between calibration and the start of instrument quality control analysis. This means there is no time to review the calibration prior to the start of sample analysis. While use of this feature is acceptable, it is not recommended unless calibration and instrument quality control are unimportant or expected to pass without issue.*

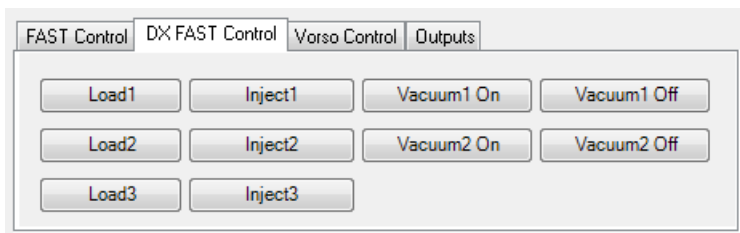
5. INSTRUMENT SHUT DOWN

5.1. Rinsing the Instrument

- 5.1.1. Acid rinse the sample introduction system, injector, and torch for at least 25 minutes to remove any acid soluble deposits that may have been left by analysis. Leave the carrier probe in the holder at the rinse station but transfer the internal standard probe from the internal standard into a bottle containing DI water.
- 5.1.2. Use the ESI SC/FAST application to cycle the switching valve between the load and inject positions.
 - Click the **Manual** menu item, opening the *Manual Movement* window.
 - Position the sample probe in the rinse station by clicking the **<Go To Rinse1>** button.



- Turn on Vacuum 1 by clicking the **<Vacuum 1 On>** button.



- Cycle between “Load1” and “Inject1” three to four times. Finish with the valve in the load position.
- Turn off the vacuum by clicking the **<Vacuum 1 Off>** button.

- 5.1.3. Transfer the carrier probe to the DI water bottle and rinse the entire system with DI water for at least 5 minutes to remove any acidic solutions. This is important because it extends the life of the entire sample introduction system and helps minimize clogs in the nebulizer.

5.2. Shutting Down the Instrument

5.2.1. Extinguish the Plasma

- Click the <Plasma Off/On> button in the *Plasma Control* window to initiate plasma ignition. The button should switch to the “off” position.
- Monitor the *Plasma Control* window until the nebulizer, auxiliary, and plasma gases have all been shut off. The indicators will turn gray and the gas meters will drop to zero.

5.2.2. Exit the Syngistix software.

- Click the Syngistix icon in the upper left corner of the application, select <Exit>

5.2.3. Release the locking arms on the instrument peristaltic pump and unhook the pump tubing from the hook points to release all tension.

5.2.4. Leave both the sample probe and the internal standard tubing in the DI water bottle until next use.

5.2.5. Check the level of all of the Argon tanks and order any replacements if needed.

6. REFERENCES

“Metals by ICP AES” DCN:03.6010.00, California Department of Toxic Substances Control, Environmental Chemistry Laboratory, Analytical Support Section, 2018

Syngistix for ICP. Computer software. Version 1.0.1.127. Perkin-Elmer. Inc., 2014.

ESISC/FAST. Computer Software. Version 2.9.0.202. Elemental Scientific, 2017

7. FIGURES

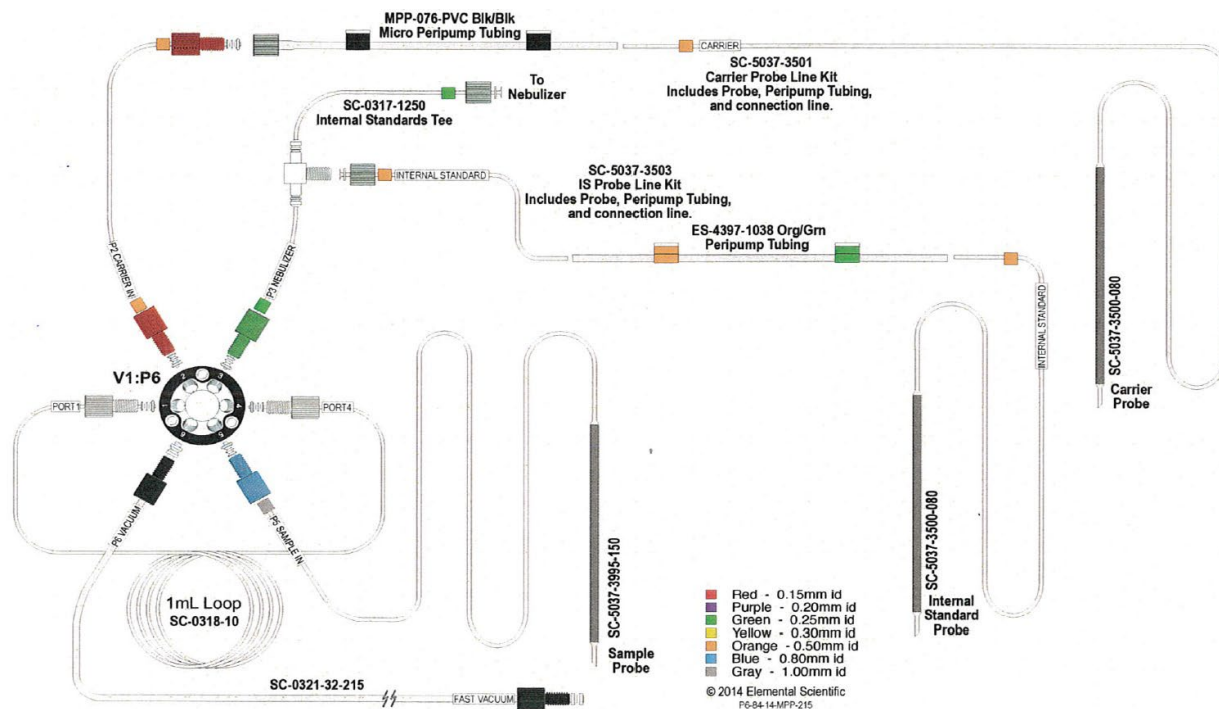


Figure 1. Elemental Scientific FAST 2DX Auto-sampler Schematic
 (actual loop used by ECL is 4 mL)

8. REVIEW

Signatures

Date
