

Status: Final

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Technical SOP for Metals Analysis using Agilent SVDV 5100/5110 ICP-OES

1. SCOPE

This SOP is intended as a technical guide for the operation of the Agilent SVDV 5100/5110 ICP-OES with attached SPS4 auto-sampler and describes the technical steps and procedures involved in optimization and sample analysis using the ICPEXpert 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 ICPEXpert 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 OES" 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 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 the tank and switch the regulator to the backup tank. Reorder Argon by contacting the Quality Assurance and Technical Support Unit.
- 3.2.3.** The chiller should hold the temperature at 20 (± 1)°Celsius and between 45-60 psi of pressure. The ICPExpert 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.

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 on the release to the right 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.
 - 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 blue tabs, is transparent, and belongs in the slot on the peristaltic pump closest to the instrument. The IS tubing has two white tabs. The carrier solution tubing has two white tabs. The carrier solution and the internal standard tubing occupy the next two slots on the peristaltic pump. The outermost slot can be used to bypass the Advanced valve System (AVS) valve in order to directly aspirate wavelength calibration solutions.
- 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 AVS valve
- If any of the tubing appears to be discolored, blocked, or damaged, replace the tubing.

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 **COUNTER-CLOCKWISE** meaning that all samples will flow *in from the left side* and flow *out on the right 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 “outflow” 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 “inflow” 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.
 - Engage the peristaltic pump on the autosampler.
 - Ensure the internal standard, rinse and carrier lines are in their respective reservoirs.

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 correct username and password.
- Start the ICPExpert software by clicking on the **ICPExpert** icon on the computer desktop.
- Click the **Instrument** menu to open the instrument panel.

- If necessary, open the **Connect** menu and establish communication between the computer and the instrument. The connection will be indicated in the lower left corner of the Instrument panel.
- The *Status Panel* on the left side of the application will indicate the condition of the instrument components.
 - If the system is working properly, all systems will receive a green check mark.



- If there is a problem in one of the subsystems, it will have a red mark next to it in the *Status Panel*. In addition, the related component on the Instrument will pulse red. Clicking on the appropriate icon in the *Status Panel* or the associated component on the instrument will open the help file for that component. Correct any indicated problems.
 - This window can be accessed at any time by clicking on the **Instrument** menu.
- If necessary, calibrate the detector by opening the **Calibration** menu on the left side of the panel and clicking the **Calibrate** button under “Detector”.

Instrument

Use axial configuration during calibration ⓘ

Organic wavelength calibration

Calibrate ⓘ

Last results:

Element	Wavelength (nm)	Wavelength Error (nm)	Result

Last successful calibration: N/A

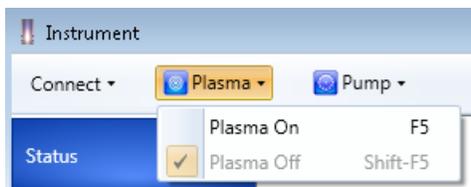
Detector

Calibrate ⓘ

Last successful calibration: N/A

Note: *The detector cannot be calibrated when the plasma is lit.*

- Ignite the plasma by selecting “Plasma On” from the **Plasma** menu.



- ❖ 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.
- Check solution flow through the sample introduction system.
 - ❖ 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.

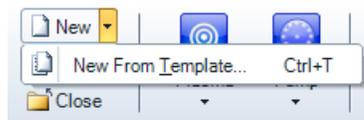
- If necessary, perform a wavelength calibration.
 - ❖ Use the outermost channel on the peristaltic pump to aspirate a 10x dilution of the *Wavelength Calibration* solution.
 - ❖ Open the **Calibration** menu on the left side of the instrument panel and click the **Calibrate** button under “Instrument”.

3.3.2. Create a new workbook from the most recent template for the appropriate analytical method

- From the main page, select “New from” in the center of the screen, and choose the appropriate template.

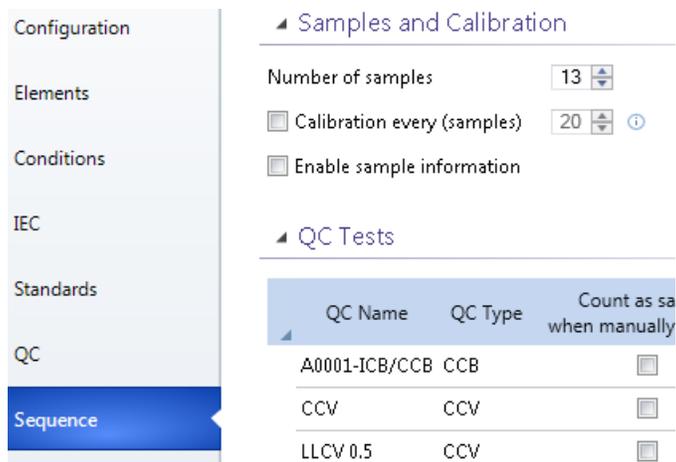


- If a workbook is already open, open the **Open** menu and select the **NEW RUN** template.



Note: Alternatively, one of latest workbooks can be used if previously recorded data is deleted and file saved with a new name.

- Save the workbook with the date and, if desired, a description of the run.
- In the Sequence tab in the left side of the workbook, enter the number of samples.



- Once the plasma is lit, allow the plasma to stabilize for 15 minutes prior to continuing. You may use DI during the stabilization period in order to conserve the internal standard and carrier solutions.

3.3.3. 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.

4. ANALYTICAL PROCEDURE

4.1. Sample Preparation

- 4.1.1. Begin preparation by creating a new “Agilent ICP Analytical Run Log” (DCN:07.0240.00.) Enter the necessary information in the log.
- 4.1.2. Prepare the samples for analysis, including any necessary dilutions and post digestion spikes.

Note: Any sample whose concentration exceeds the highest calibration standard by more than 10% for any reported element or exceeds 500 ppm Fe should be diluted such that the concentration falls within the calibration range or below 500 ppm Fe. Concentrations above the calibration range (50-100ppm for Sb, As, Ba, Cd, Cr, Co, Cu, Pb, Mo, ni, Se, th, V and Zn and 10-20ppm for Ag and Be) may be reported if the linear range is confirmed by analysis of a CalWS 100 (100 ppm) standard.

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

4.2. Sample Information Entry

- 4.2.1. Enter the sample information.
 - In the Rack:Tube column, verify the locations of the samples.

- In the *Solution Label* column, enter the ECL number of the sample, QC label, its dilution factor and other pertinent information such as analysis type (e.g. AB01234 TT 10x, BC04321 WET, BB00879 PS 10x or BB00888 LCS 10x).

4.2.2. Additional Sample Information:

- For samples digested from solid material:
 - Enter the mass of the original solid sample in grams (g) in the *Weight (g)* column.
 - Enter the final volume at the end of the digestion and dilution in milliliters (mL) in the *Volume (mL)* 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 mg/kg soil.

- For dilutions of other samples:
 - Enter the dilution factor of original sample in the *Dilution* column.

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

4.2.3. Save the workbook.

- Click the **Save** button in the application ribbon.

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 OES” DCN:03.6010.00.

4.3.2. Open the analytical panel.

- Click the **Analysis** tab on the left side of the application to open the *Analysis* window.
- Write the workbook file name in the ICP-AES Analysis Log page.

- 4.3.3. Start the calibration by selecting the IEC Blank, Interferent Standard and all calibration standards (i.e. Cal 0.1 through Cal 50). Click the Run button in the application ribbon.
- 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
 - Recalibrate the system

4.4. Daily Optimization

- 4.4.1. 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 IEC 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, found on the Instrument panel dashboard
 - The intensities for two elements (commonly Sb and Pb) in both the mid-level standard and the blank

4.5. Sample Analysis

- 4.5.1. Select the initial QC samples and click Run.
- 4.5.2. Monitor the results of the quality control 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.
- Select and analyze ten samples followed by interim QC samples (CCV, CCB, LLCV(s))
- 4.5.3. After the sample run is complete, save the workbook and save the raw data file report by selecting Report under the File menu, then report to PDF to save raw data file report.
- 4.5.4. Export LIMS data by clicking LIMS under File, then click Export to save csv data file for LIMS.
- 4.5.5. Copy and paste the sequence data, iron concentration and yttrium ratios into the “Agilent ICP Analytical Run Log.” for ICP sequence run log.

5. INSTRUMENT SHUT DOWN

5.1. Rinsing the Instrument

- 5.1.1. Acid rinse the sample introduction system, injector, and torch for at least 2-5 minutes to remove any acid soluble deposits that may have been left by analysis. Leave the carrier probe in the dilute nitric reservoir but transfer the internal standard probe from the internal standard into a bottle containing DI water.
- 5.1.2. Cycle the switching valve between the uptake and inject positions on the Instrument page.



The screenshot shows a control panel for the Switching Valve. It has two columns: 'Uptake' and 'Inject'. The 'Uptake' column has a 'Valve position' dropdown set to 'Uptake', a 'Pump rate (mL/min)' input field set to '0', and a 'Bubble injector' checkbox that is checked. The 'Inject' column has a 'Valve position' dropdown set to 'Inject', a 'Pump rate (mL/min)' input field set to '0', and a 'Bubble injector' checkbox that is unchecked. There is a 'Reset' button at the bottom center and a help icon (question mark in a circle) at the bottom right.

	Uptake	Inject
Valve position	Uptake	Inject
Pump rate (mL/min):	0	0
Bubble injector	<input checked="" type="checkbox"/>	<input type="checkbox"/>

Reset 

- Cycle between “Uptake” and “Inject” three to four times. Finish with the valve in the load position.
- 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
 - Select **Plasma Off** from the **Plasma** button in either the Instrument window or the workbook.
- 5.2.2. Monitor the Instrument window until the nebulizer, auxiliary, and plasma gases have all been shut off.
- 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 rinse/carrier tubing and internal standard tubing being placed in an empty bottle until next use. Turn off the chiller, and wait Peltier temperature return to ambient temperature before close the argon.
- 5.2.5. Check the level of all of the Argon tanks and order any replacements if needed.

6. REFERENCES

DCN:03.6010.00 “Standard Operating Procedure for EPA Method 6010C: Inductively Coupled Plasma-Optical Emission Spectroscopy”.

ICPEXpert. Computer software. Version 7.4.0.10280. Agilent Technologies. Inc., 2017.

DCN: 07.0240.00 “Agilent ICP Analytical Run Log”

7. FIGURES

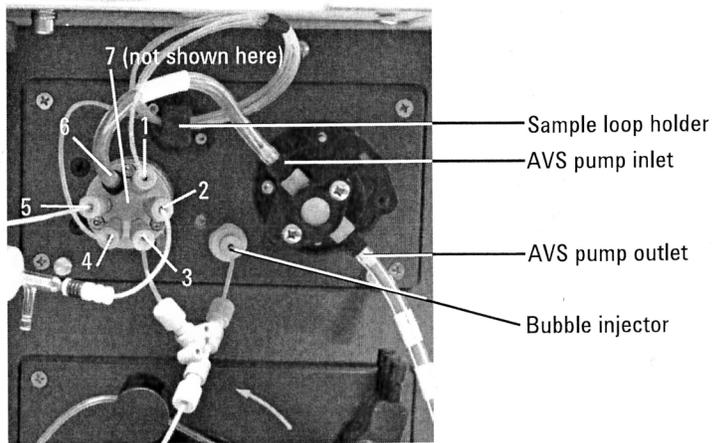


Figure 1. Agilent SVDV 5100/5110 AVS valve plumbing

Ports 1,4 (sample loop), Port 2 (nebulizer), Port 3 (carrier), Port 5 (sample), Port 6 (AVS pump),
Port 7 (IS)

8. REVIEW

Signatures

Date
