ICP-MS Standard Operating Procedures

GGC Chemistry Instrumentation Protocol Series

04/19/2018

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Introduction

Georgia Gwinnett College (GGC) currently maintains a PerkinElmer Elan DRC-e Inductively-Coupled Plasma Mass Spectrometer (ICP-MS, **Figure 1A**). The instrument is located in Building H, room 1133, and is utilized routinely by faculty and students for research and educational purposes. Dr. Kathryn Zimmerman currently supervises maintenance, operation and troubleshooting associated with the instrument.

The ICP-MS can detect very low concentrations (ppt) of metals and some non-metals by ionizing the sample in an electrically conductive plasma and then separating and quantifying sample ions using a mass spectrometer.

The objective of this protocol is to provide an introduction to basic operation of the instrument for faculty and students interested in utilizing ICP-MS for instructional purposes and/or to conduct undergraduate research projects. Faculty advising students on this instrument should undergo training before operating instrument alone.

I. Getting Started

A. Sample Preparation

Samples should be digested in HNO_3, detailed in your lab protocol, and then diluted to 2% HNO_3 (by volume) using DI or MiliQ H_2O. Standards for standard curve can be diluted from the Instrument Calibration Standard 2 (Perking Elmer Pure Plus) in the same HNO_3 matrix as your samples. Labeled samples may be stored in the shared refrigerators in the service hallway. When you have completed your project, please dispose of your samples in an appropriate time frame.

B. Instrument Prep/Setup

1. Check waste container.

Before running samples, check the waste container located on the floor beside the instrument (**Figure 1B**). If the container is full, please contact Dr. Zimmermann or Janet Whelan.

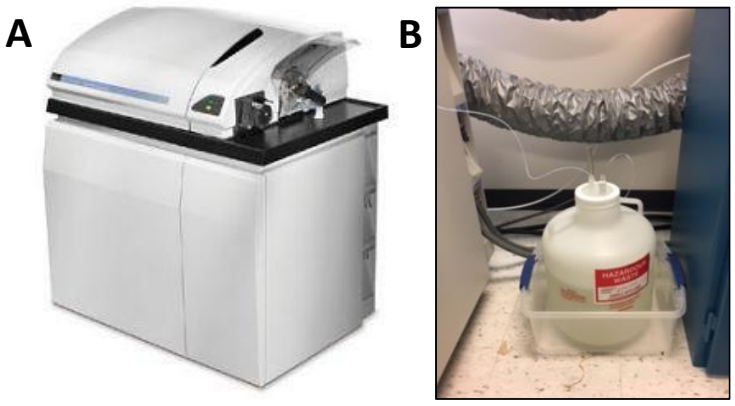

Figure 1: (***A***) PerkinElmer Elan DRC-e ICP-MS. ***(B)*** Waste container for ICP-MS, located on floor on right-hand side of instrument.

2. Open lines for chiller.

The chiller is the cooling system for the instrument electronics. Prior to use of the instrument, the chiller needs to be started. There are two pairs of valves connecting the instrument to the chiller: one pair is located in H1133 above and to the right of the instrument, and the second pair is located in the back prep hall above the chiller unit. Both pairs of valves must be moved from the closed positions (**Figure 2A**) to the open positions (**Figure 2B**).

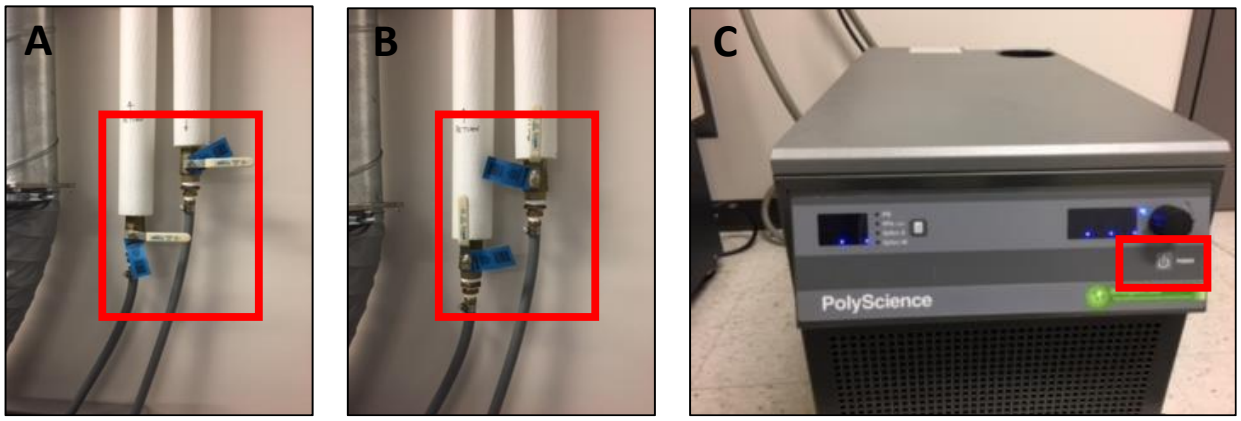

Figure 2: (**A**) Chiller lines in H 1133 in closed positions, and (**B**) open positions. (**C**) Chiller located in prep hallway.

3. Turn on power to the chiller.

The chiller (**Figure 2C**) is located in the back prep hall. Once the power is turned on, confirm that the temperature is maintained at or near 18 °C.

4. Open Argon.

a. Open valve on the compressed Argon tank (**Figure 3A**).
b. Open valve on wall to allow flow of Argon into instrument (**Figure 3B, 3C)**.

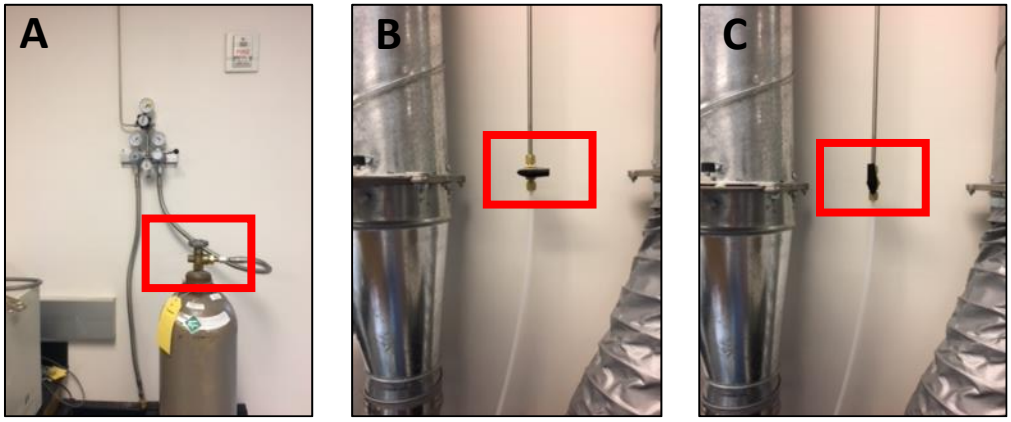

Figure 3: (**A**) Valve on Argon Compressed gas tank. (**B**) Argon line to instrument closed. (**C**) Argon line to instrument open.

5. Set up Sampling Lines.

a. Stretch sampling lines into position over the peristaltic pump and clamp into place.
b. Pump lines are color coded: gray tabs for waste, black tabs for sample wand and blue/orange tabs for internal standard wand.

c. There are two sampling wands. The smaller inner diameter (i.d.) wand (blue/orange tabs) is for the internal standard, while the larger i.d. wand (black tabs) is for the sample or calibration standard.

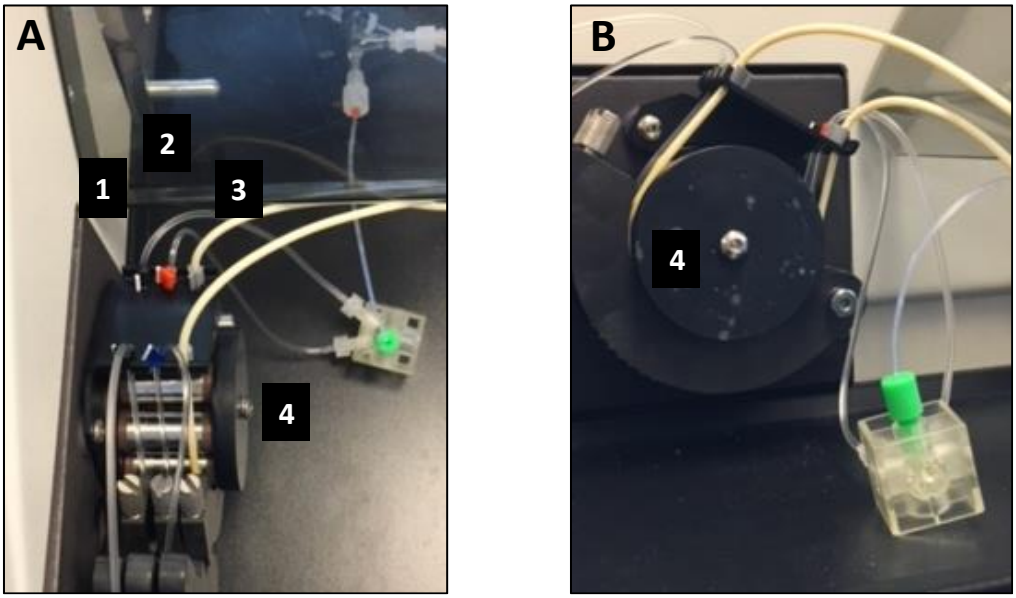

Figure 4: (**A**) Overhead view of sampling lines. (**B**) Side view of lines properly clamped onto peristaltic pump [4]. [1] Sample/calibration line (black), [2] internal standard (blue/orange), [3] waste wand (gray) and [4] peristaltic pump.

6. Software Set up.

a. On the Desktop open the ICP-MS User folder.

b. Open the Instrument Log folder and the Elan 6000 Log Excel Spreadsheet (**Figure 5**).

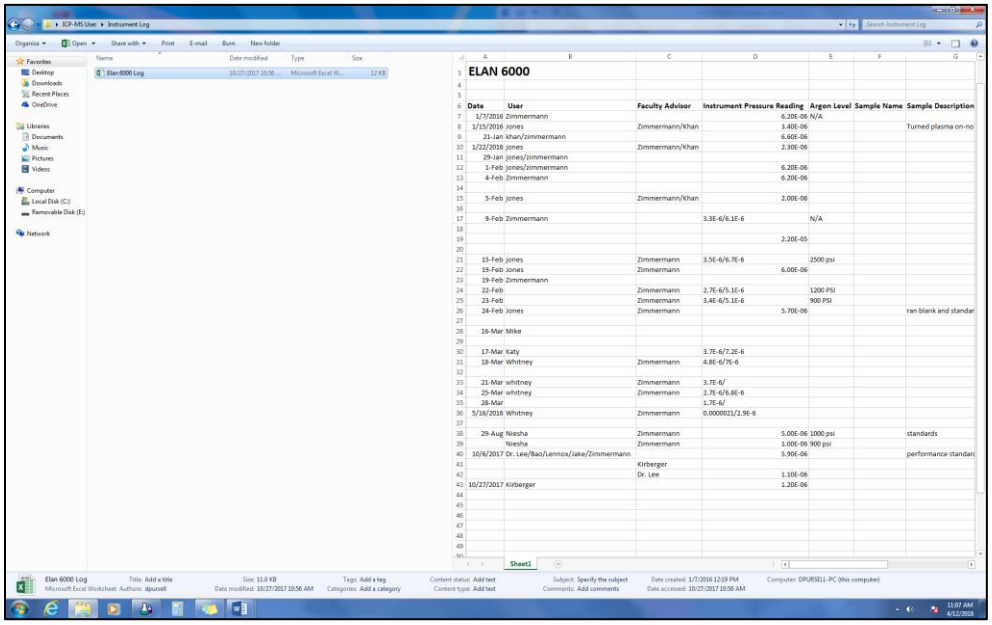

Figure 5: Instrument Log File for ICP-MS

c. Fill in the appropriate information regarding your use.
d. If not already open, click the Elan Icon (**Figure 6A**) to load the ICP/MS software (**Figure 6B**).

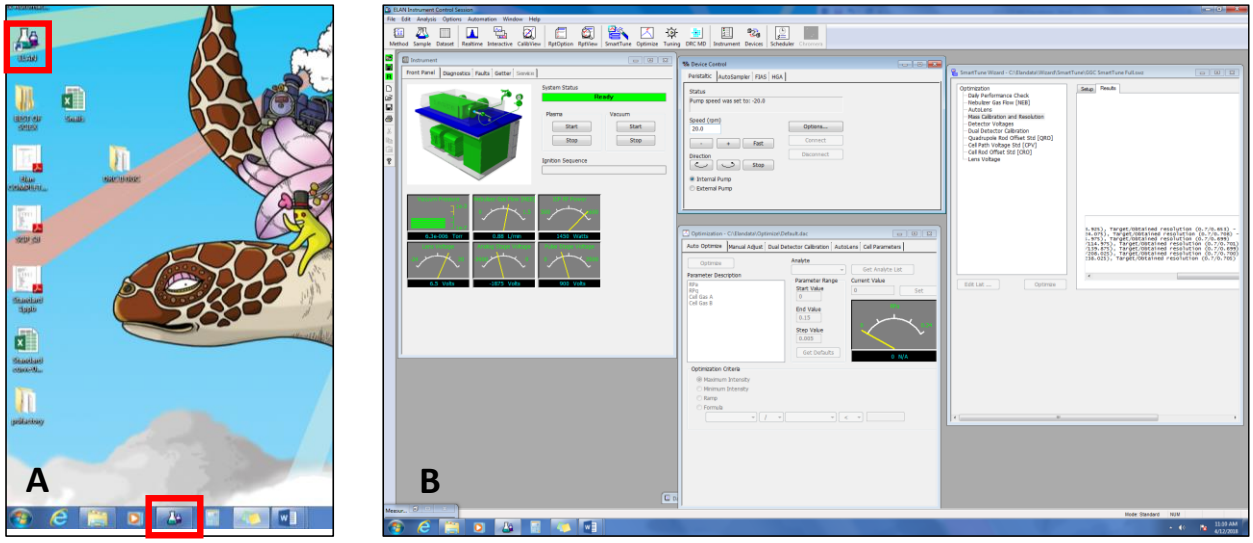

Figure 6: (***A***) Two options on desktop for opening Elan software to run ICP-MS. (B) Elan software.

7. Open a new dataset (Figure 7).

a. Click the R icon on the left hand side of the screen.
b. Click the Dataset icon, select "New" and use the date "MMDDYY" as the dataset name.
c. Select Create.

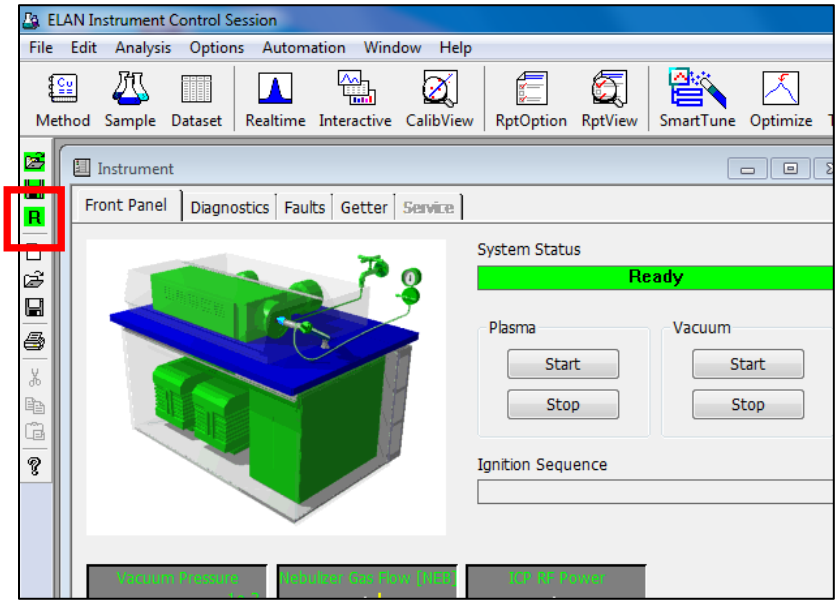

Figure 7: Opening a new dataset.

8. Rinse sampling wands/tubing.

a. Insert both sampling wands in 2% nitric acid (HNO_3) solution (**Figure 8**).

Figure 8: Both sampling wands in 2% HNO_3. The HNO_3 used is trace clean so that there are no metal contaminants.

b. Start the peristaltic pump.
c. Select the Device tab from the Elan software (**Figure 9**, red rectangle).
d. Enter "20.0" in the box for Speed (rpm) (**Figure 9**, blue rectangle). This will start the pump which will pull the 2% HNO_3 into the sampling lines.
e. Let the pump run for ~1 min.

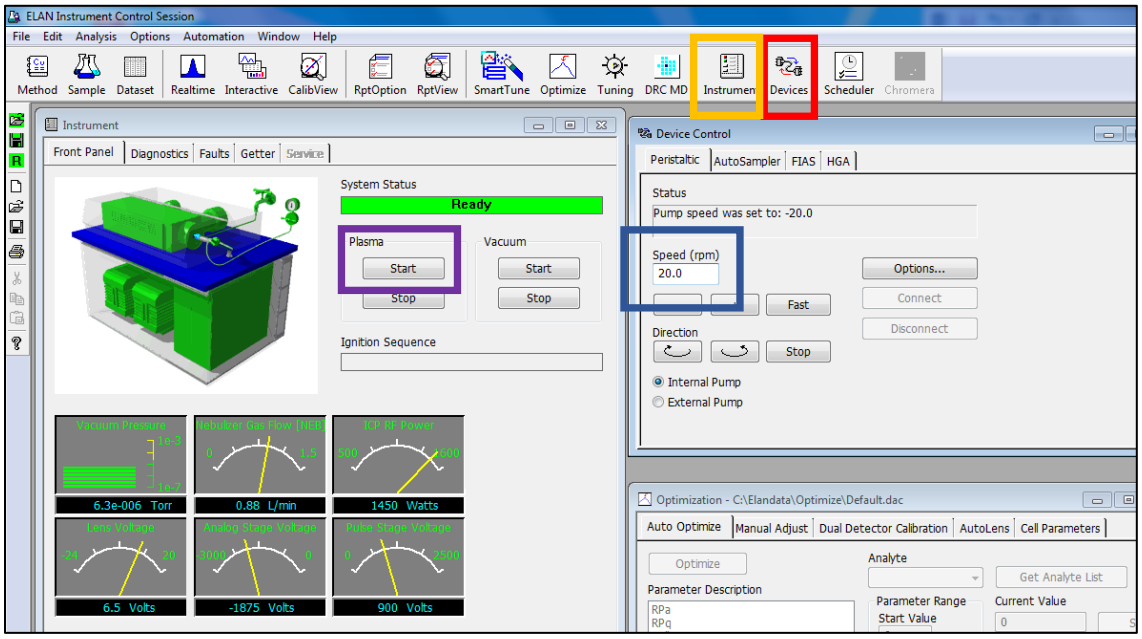

Figure 9: Instrument and Device controls.

9. Light the plasma.

a. Select the Instrument tab (**Figure 9**, orange rectangle). If the System Status displays the green ready bar (**Figure 9**, green bar) the instrument is ready to light the plasma.
b. Click the Start button (**Figure 9**, purple rectangle) to light the plasma.
c. When the plasma lights, you will see a white glow from the instrument interface (**Figure 10**). **CAUTION: ONLY OBSERVE PLASMA THROUGH THE UV SHIELD LOCATED ON THE INSTRUMENT.**
d. You will also hear the sound of the interface gate valve open (swish). There may be a delay between when the start button is selected, and the valve opens, so wait approximately 15 seconds.
    i. If the torch fails to ignite, press the Plasma Stop button.
    ii. After waiting 30 seconds, retry the Plasma Start. This could take up to a minute to light. This may take several tries.
e. Let the plasma stabilize for 10-15 min with the wands in the 2% HNO_3 solution.

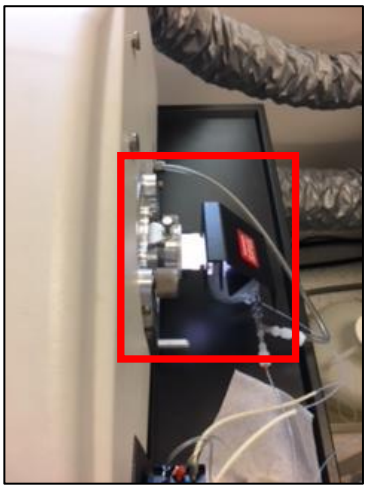

Figure 10: Lit plasma (white glow) at instrument interface.

10. Perform Daily Performance Analysis.

a. Fill the centrifuge tube labeled Daily Performance Solution (**Figure 11A**) with Smart Tune Solution (**Figure 11B**).
b. Place both wands into the Daily Performance Solution (**Figure 11A**). Run solution through both lines for approximately 30 s. Do NOT place wands directly into the Smart Tune Solution bottle.

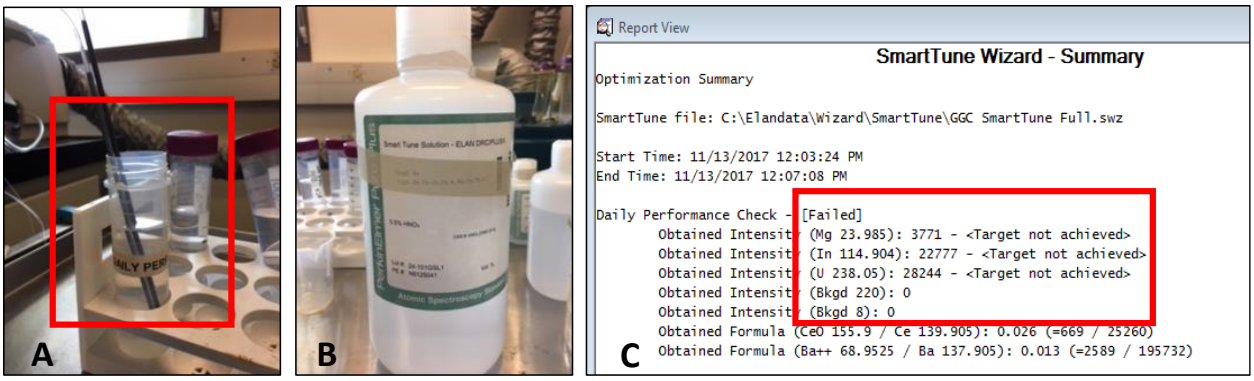

Figure 11: (***A***) Sampling wands inserted into the Daily Performance Solution. (***B***) Smart Tune Solution for daily performance analysis. (***C***) SmartTune Wizard Summary of performance results.

c. Open "SmartTune Wizard Tab" and right-click on Daily Performance Check.
d. Select Quick Optimize. After Optimization has finished, a report will pop up indicating whether the instrument passed or failed (**Figure 11C**).
e. If the instrument passes, return wands to the 2% HNO_3 rinse lines.
f. If the instrument does not pass:
    i. Right-click on Nebulizer Gas-flow and select Quick Optimize.
    ii. Open the "Optimize" tab. If this reads (Modified) click File → Save.
    iii. Return wands to the 2% HNO_3 rinse lines.

11. Set up method for analysis.

a. From main window, click on ***File→Open→Select Method*** to locate the method file for analysis. If you need to set up a new method file, please talk to Dr. Zimmermann.
b. In the Method window, select the Calibration tab (**Figure 12**).
c. In the table, enter (or alter) the concentration for each of your standards (Std 1, Std 2…Std n) depending on dilutions selected for standards (µg/L).
d. As seen in **Figure 12**, do not enter values for elements that have a triangle in the internal standard column.

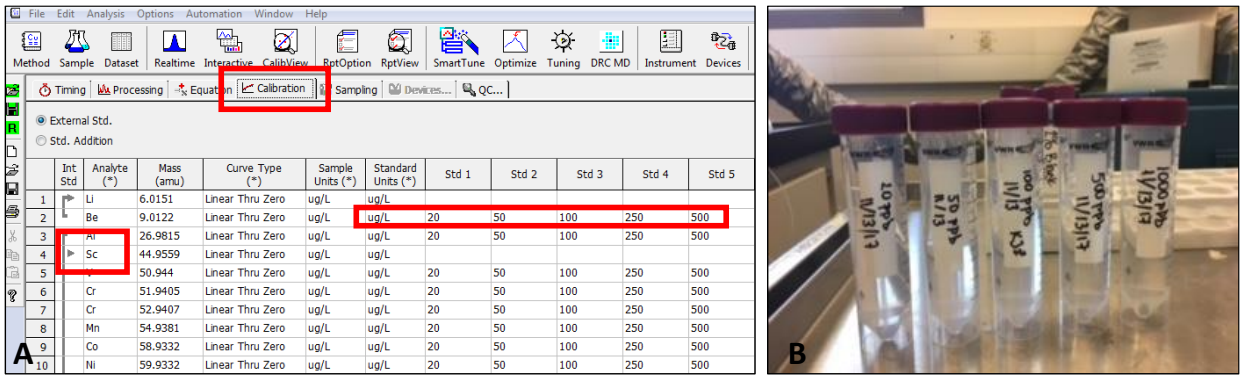

Figure 12: (**A**) Example of calibration screen. Internal standards are indicated with arrows (column 1). (**B**) Calibration standard solutions for ICP-MS analysis.

12. Sample analysis.

a. Insert blue/orange wand (smaller i.d.) into the internal standard solution (**Figure 13A**). This wand will remain in the internal standard for the duration of the analysis.
b. Insert black wand (larger i.d.) initially into blank solution (sample matrix, e.g., 2% HNO_3, with no analytes).
c. Allow solutions to run through wands for ~1 min to clean wands and tubing.
d. Select Sample Window icon (**Figure 13B**).
e. With the sample wand still in the blank solution, select "Analyze Blank."

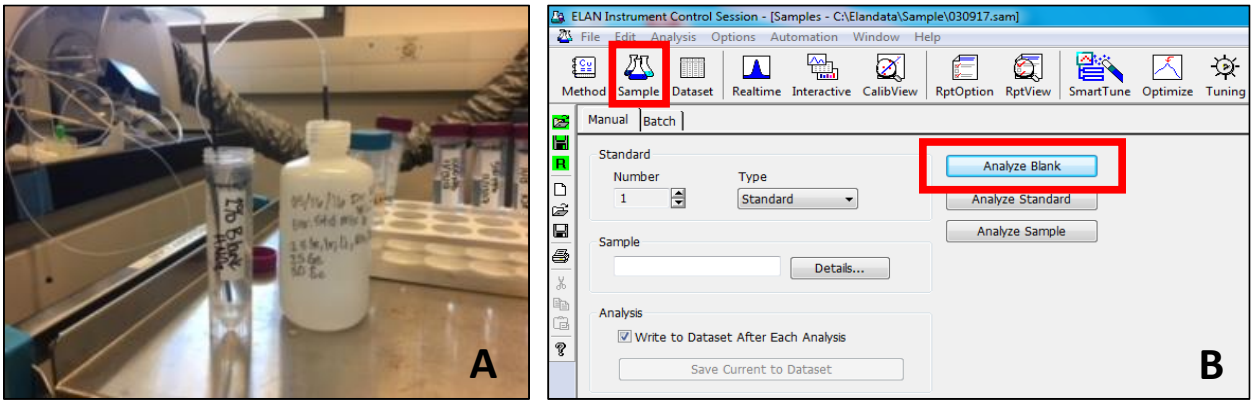

Figure 13: (**A**) Sample wand in 2% HNO_3 blank solution and internal standard wand positioned in internal standard solution. (**B**) Sample window for selection of sample type (Blank, Standard, or Sample).

f. A Measurement Status screen will appear, which will show a progress bar for the duration of the Blank analysis (**Figure 14**), followed by a report screen (**Figure 15**).
    i. Note that there is no concentration data listed under the Mean column, because this is the Blank.

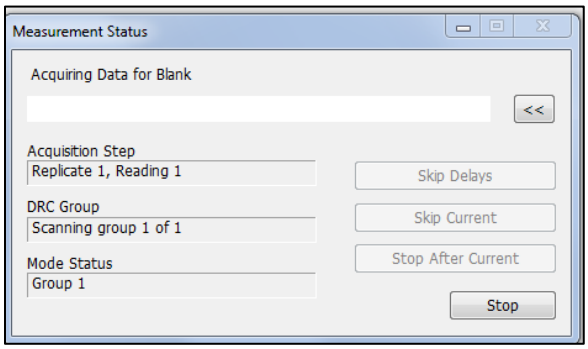

Figure 14: Progress bar for data acquisition.

GGC Chemistry Dept. ELAN DRC ICP-MS Summary Report

Sample ID: Blank
Sample Date/Time: Monday, November 13, 2017 12:18:03
Sample Type: Sample
Sample Description:
Batch ID:
Method File: C:\Elandata\Method\Snail Analysis.mth
Dataset File: C:\Elandata\DataSet\2016-1214\Blank.415
Sample Prep Volume (mL):
Initial Sample Quantity (mg):
Aliquot Volume (mL):
Diluted To Volume (mL):

	Analyte	Mass	Mean	Std.Dev.	Units	Meas. Intens. Mean	Blank Intensity	Intens. RSD
>	Li	6			ug/L	265.7		3.0
	Be	9			ug/L	3.7		56.8
	Al	27			ug/L	2577.7		1.0
>	Sc	45			ug/L	125939.0		0.6
	V	51			ug/L	762.6		2.6
	Cr	52			ug/L	2434.0		0.9
	Cr	53			ug/L	253.0		4.8
	Mn	55			ug/L	387.3		2.8
	Co	59			ug/L	111.0		15.8
	Ni	60			ug/L	124.3		8.2
	Cu	63			ug/L	1596.8		3.6
	Cu	65			ug/L	800.7		2.1
	Zn	66			ug/L	1819.9		10.6
	Zn	67			ug/L	297.3		10.0
	Zn	68			ug/L	1281.1		13.1

Figure *15*: Example dataset for analysis of Blank.

g. Without exiting the Sample window, switch the black sampling wand from the Blank to the Standard 1 solution (i.e., the concentration listed as Standard 1 in the method).
h. Allow the line to flush with this solution for ~1 min.
i. As seen highlighted in **Figure 16**:
    i. Indicate the Standard Number in the Number box.
    ii. Make sure the Type is listed as Standard.
    iii. Make sure the "Write to Data Set" box is checked, then click on Analyze Standard.

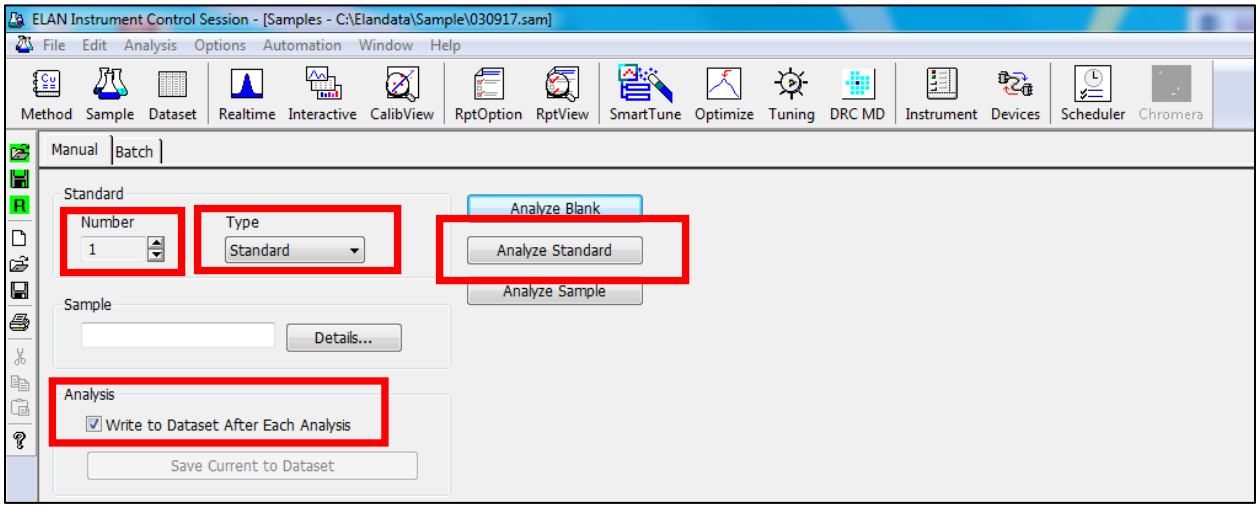

Figure 16: Setting up standard curve analysis.

j. Repeat this process for each standard, changing the Number as you switch the wand between each standard.
k. After each standard is analyzed, a report for that standard is generated (**Figure 17**).

GGC Chemistry Dept. ELAN DRC ICP-MS Summary Report

Sample ID: Standard 4
Sample Date/Time: Friday, April 13, 2018 16:15:00
Sample Type: Sample
Sample Description:
Batch ID:
Method File: C:\Elandata\Method\BIODIESEL2018.mth
Dataset File: C:\Elandata\DataSet\041318\Standard 4.005
Sample Prep Volume (mL):
Initial Sample Quantity (mg):
Aliquot Volume (mL):
Diluted To Volume (mL):

	Analyte	Mass	Mean	Std.Dev.	Units	Meas. Intens. Mean	Blank Intensity	Intens. RSD
>	Li	6			ug/L	525.0	534.3	3.8
	Be	9	**248.96**	10.21603	ug/L	82758.7	2.0	0.8
	Al	27	**251.41**	0.36027	ug/L	1708819.2	6955.3	0.4
>	Sc	45			ug/L	397968.0	416241.8	0.4
	V	51	**247.64**	2.06783	ug/L	2899075.1	1287.3	1.0
	Cr	52	**248.69**	0.60444	ug/L	2614305.0	5970.6	0.6
	Cr	53	**251.72**	0.51868	ug/L	335855.5	463.3	0.6
	Mn	55	**248.18**	2.64397	ug/L	4386236.9	1333.4	1.3
	Co	59	**247.55**	2.84551	ug/L	3155031.0	176.7	1.3
	Ni	60	**251.09**	2.16310	ug/L	702768.7	306.7	0.6
	Cu	63	**250.56**	1.84404	ug/L	1476948.5	791.7	0.7
	Cu	65	**250.74**	1.33540	ug/L	727549.8	432.3	0.3
	Zn	66	**251.48**	2.60948	ug/L	380250.4	892.4	0.6
	Zn	67	**251.48**	1.62509	ug/L	67296.2	166.3	0.6
	Zn	68	**251.37**	1.01652	ug/L	283311.2	707.4	0.4
>	Ge	74			ug/L	54098.1	55134.3	0.7

Figure 17: Example dataset for a calibration standard.

l. The calibration curve can be viewed as it is being made by selecting the CalibView window icon (**Figure 18**).

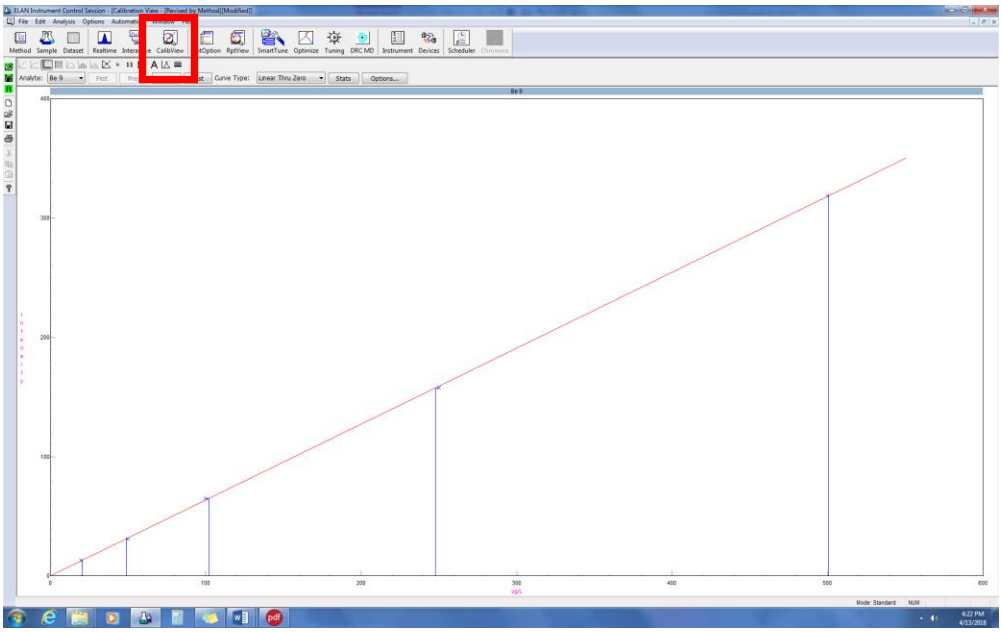

Figure 18: Calibration view. Users may switch the calibration element being viewed to compare calibration curves for all analytes.

m. Once all calibration standards have been analyzed, samples can be analyzed.
n. To analyze samples, return to the Sample window.
o. Place the sampling wand into your digested sample tube, enter a Sample identifying name and select Analyze Sample (**Figure 19**).

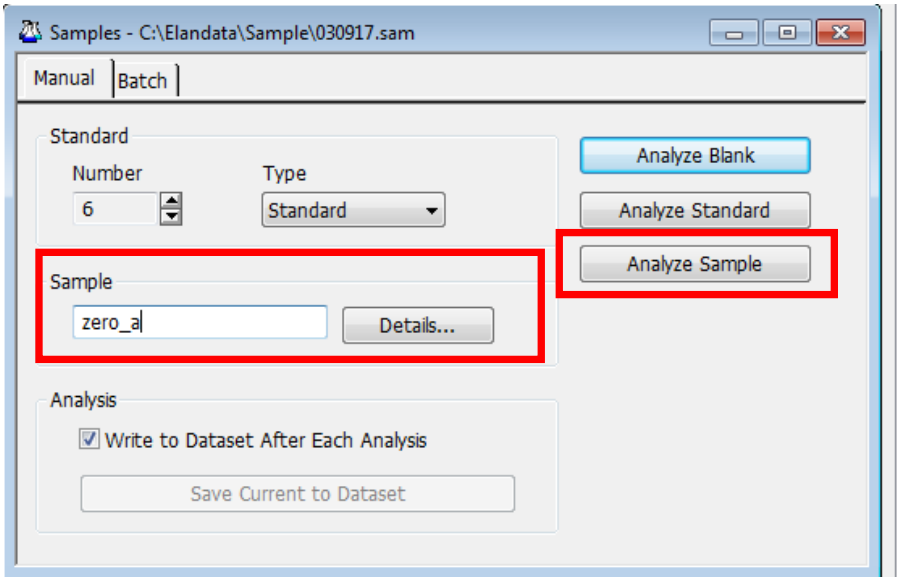

Figure 19: Initiating a sample analysis.

p. At the end of the sample run, a summary report is generated with sample ID at the top of the page and Mean analyte concentrations reported in ppb (**Figure 20**).

GGC Chemistry Dept. ELAN DRC ICP-MS Summary Repor

Sample ID: Biodiesel 1
Sample Date/Time: Friday, April 13, 2018 17:13:13
Sample Type: Sample
Sample Description:
Batch ID:
Method File: C:\Elandata\Method\BIODIESEL2018.mth
Dataset File: C:\Elandata\DataSet\041318\Biodiesel 1.016
Sample Prep Volume (mL):
Initial Sample Quantity (mg):
Aliquot Volume (mL):
Diluted To Volume (mL):

	Analyte	Mass	Mean	Std.Dev.	Units	Meas. Intens. Mean	Blank Intensity	Intens. RSD
⌈>	Li	6			ug/L	738.4	534.3	9.9
⌊	Be	9	**0.00**	0.00136	ug/L	4.0	2.0	25.0
⌈	Al	27	**24.24**	0.91445	ug/L	228408.3	6955.3	1.8
|>	Sc	45			ug/L	571009.2	416241.8	2.8
|	V	51	**3.13**	0.10210	ug/L	52480.9	1287.3	0.4
|	Cr	52	**11.21**	0.39244	ug/L	170366.6	5970.6	0.6
|	Cr	53	**1.01**	0.07825	ug/L	2518.7	463.3	4.3
|	Mn	55	**0.25**	0.01432	ug/L	7866.7	1333.4	3.0
|	Co	59	**0.36**	0.01379	ug/L	6606.4	176.7	1.1
|	Ni	60	**0.45**	0.03500	ug/L	2098.9	306.7	4.6
|	Cu	63	**0.55**	0.04397	ug/L	5218.2	791.7	5.0
⌊	Cu	65	**0.55**	0.02427	ug/L	2623.4	432.3	1.8
⌈	Zn	66	**70.03**	2.76075	ug/L	135064.3	892.4	0.8
|	Zn	67	**63.91**	2.92325	ug/L	21908.0	166.3	1.3
|	Zn	68	**70.92**	2.22585	ug/L	101761.1	707.4	0.6
|>	Ge	74			ug/L	68377.1	55134.3	3.3
|	Kr	83	**1.30**	0.03395	ug/L	-271.5	55.0	1.4
|	Sr	88	**1.22**	0.03271	ug/L	47410.6	344.0	1.0
|	As	75	**0.10**	0.00418	ug/L	428.3	165.0	3.5
|	Se	77	**0.16**	0.07393	ug/L	112.0	69.3	7.6
⌊	Se	82	**-0.05**	0.05474	ug/L	78.3	73.0	13.4
⌈	Mo	98	**0.02**	0.00106	ug/L	198.0	27.7	0.5
|	Ag	107	**0.02**	0.00421	ug/L	450.0	90.3	11.2
|	Cd	111	**0.04**	0.01145	ug/L	302.6	129.2	15.3
|	Cd	114	**0.00**	0.00178	ug/L	15.4	-0.6	83.6
|>	In	115			ug/L	263824.9	207232.4	4.0
|	Sb	121	**3.19**	0.13314	ug/L	32122.3	44.3	0.2
⌊	Sb	123	**3.16**	0.12942	ug/L	24507.6	28.2	0.2
⌈	Ba	135	**100.27**	2.88136	ug/L	362378.8	391.7	0.5
|	Ba	137	**102.69**	3.17660	ug/L	632757.1	701.7	0.1
|>	Tb	159			ug/L	408606.3	289595.0	3.1
|	Pb	208	**0.11**	0.05092	ug/L	6798.1	744.0	37.1
⌊	Bi	209	**-5639.13**	616.83424	ug/L	490.0	555.0	4.6
⌈	Na	23	**465.55**	21.91588	ug/L	4051246.3	66270.4	2.6
|	Mg	24	**3.63**	0.12304	ug/L	22967.0	1645.1	1.0
|	K	39	**142.15**	6.10472	ug/L	2293260.6	184224.6	1.7
|	Ca	44	**80.68**	4.44985	ug/L	90879.7	31342.9	1.5
|	Fe	54	**-34.08**	2.45310	ug/L	112569.8	115496.8	0.4
⌊>	Sc	45			ug/L	571009.2	416241.8	2.8

Sample ID: Biodiesel 1
Report Date/Time: Friday, April 13, 2018 17:15:38

Figure 20: Example sample dataset.

13. Post-analysis.

a. After completion of all sample analyses, rinse wands in 2% HNO_3 (~1 min), then DI water (~1 min), then allow some air to flow through them (~1 min).
b. Select Instrument (**Figure 21**, orange rectangle), then Plasma Stop (**Figure 21**, purple rectangle).
c. Select Device (**Figure 21**, red rectangle). Set Peristaltic Pump Speed to zero (**Figure 21**, blue rectangle).

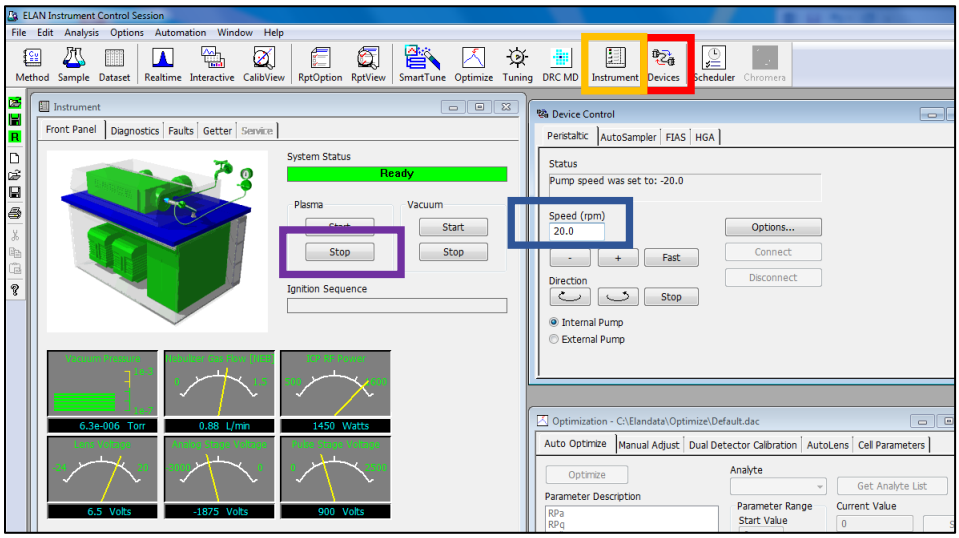

Figure 21: Termination steps.

d. Turn off chiller, then close all four chiller lines in both H1133 and the back prep hall.
e. Close both argon gas valves.
f. Unclamp sampling lines from the peristaltic pump. This increases their lifetime of use.
g. All report data from the completed analysis can be found, based on date and time, under:

Documents→PDF Files→Autosave

Analyze your data! Good Luck!

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