Agilent 5110 ICP-OES: Quick Start Guide

Balance_UG_371T_draft

v1.1 Prepared by V.Percuoco

Based off Teledyne-Leeman ICP-AES Quick Start Guide

Introduction

The ICP-OES is used to run major and minor elements on squeezed IW samples and major element oxides and minor elements on digested solid samples. Elemental analysis, especially in sediments and hard rocks, is complex and it is highly recommended that the user read both the **ICP-OES User Guide** and **pXRF User Guide** before proceeding.

ICP Operation

Starting the instrument and igniting the plasma

1.	Turn on the wall-mounted argon valve. The pressure should be between 80-90 psi.
2.	Turn on the instrument.
3.	Turn on the water chiller. Allow the peltier cooling to reach -40°C.
4.	Start ***ICP Expert*** and allow 45 mins for the polychromator to reach 35°C with ***Boost*** purge enabled
5.	Turn on the plasma, then turn on ***Snout*** purge. Allow the plasma to stabilize for at least 20 minutes.
6.	Engage the peristaltic pump tubing. Prime the autosampler rinse station.
7.	Open an ***ICP Expert*** template.
8.	Ensure values for the calibration standards are in the standards table located on the ***Standards*** tab.
9.	Enter the number of samples, sample names, and dilution factor information on the ***Sequence*** tab. Ensure check standards are analyzed every 8-10 samples.
10.	Place standards and samples in appropriate locations within the autosampler rack

Save and Run a Sequence

1.	Click on the ***Sequence*** tab to create a sequence.
2.	Enter in the sample name (TEXTID) and, if the sample was diluted differently than the regular scheme, enter in the dilution factor. Dilution factors may be added after a run has completed.
3.	Organize the sample sequence as follows: Blank Standards Samples (with a check standard as the first sample, and every 8-10 samples afterwards.)
4.	Verify the blank, standards and samples are placed in the appropriate locations within the autosampler racks.

5.	Press ***Run*** on the top menu bar to begin the analysis

Verify the Results

1.	On the ***Analysis*** tab, observe the scan profile, calibration curve, and measurement statistics for sample replicate integrations.
2.	Adjust the peak integration area and background fit as needed. The adjustments are propagated through all samples of the edited wavelength.
3.	Reject erroneous measurements by deselecting the ***Replicates*** checkboxes.
4.	Adjust the calibration curve on the ***Standards*** tab as needed.

Shut Down the ICP

1.	Let the instrument pump: • Rinse solution for 10 min • DI water for 10 min • Air for 10 min (until spray chamber is dry).
2.	In instrument control panel, select ***Plasma off***.
3.	Disengage the peristaltic pump tubing.
4.	Turn off the water chiller.
5.	If the ICP will be used within the subsequent days leave the argon flowing and the instrument on, otherwise shut off the argon and instrument.

Export, analyze and upload data to LIMS

1.	After the analysis has finished, the results are displayed in tabular format on the ***Analysis*** tab. Right-click the first cell in the header row, then select ***Export Selected Solutions***. Save the file to an Expedition\Site folder.	
2.	Open the Agilent Data Reduction Program.xlsm. On the raw data tab open the menu (CTRL+SHIFT+W) and select import data. Select the ICP analysis results file to import.	
3.	Copy the Condition Sets and Element Lists tables from ICP Expert to the appropriate tables located on the ***Condition Set and Element List*** tab in the Agilent Data Reduction Program.	
4.	Download a curatorial report from LORE and paste it to the table on the Curatorial Report tab.	
5	On the ***Reduced Data*** tab, open the menu (CTRL+SHIFT+W) and click ***Refresh PivotTable.***	
6.	Use the slicers to filter out undesired elemental wavelengths. Porewater graphs may be generated by opening the menu and selecting “Show Concentration Plots”	
7.	After the data has been vetted by the scientists, open the menu on the ***Reduced Data*** tab and select ***Spreadsheet Uploader Format***. A new worksheet with the transposed data will be created.	
8.	Upload the data with Spreadsheet Uploader.	

Sample Preparation

Solids

1.	Choose representative standards to bracket the elemental concentrations expected in sediment or hard rock samples. Prepare beads according to the ICP User Guide.
2.	Add 50 mL of 10% nitric acid solution to a 125 mL Nalgene bottle, drop in the sample bead, close the lid, and shake on the wrist-action shaker for 1 hour.
3.	Extract 20 mL solution at a time from the Nalgene bottle and filter through a 0.45 µm Acrodisc into a 60 mL Nalgene bottle.
4.	Pipette 500 µL of acidified sample or standard into a sample tube, add 100 µL internal standard and 4.4 mL of 2% nitric acid solution
5.	Analyze on the ICP.

Blank: Flux blank prepared as a sample

Check Standards: Choose a representative standard solution. Pipette 5 mL standard solution + 1 mL internal standard into a 50 mL vial, make up with HR matrix solution. Shake thoroughly. Pour into separate aliquots and analyze every 8-10 samples.

Interstitial Water Method

1.	Pipette 500 µL of acidified sample into a sample tube, add 100 µL internal standard and 4.4 mL of 2% nitric acid solution
2.	Cap and vortex mix the vial
3.	Analyze on the ICP

Blank: 18 M water prepared as a sample

Check Standard:s Prepare a 100 mL aliquot of the 100% level in-house standard (and the 100% level IAPSO, if desired). Shake thoroughly. Pour into separate aliquots and analyze every 8-10 samples.

Reagent solutions

Solutions

Nitric Acid Solutions: Add 14.3 mL of concentrated HNO_3 per 1 L of MQ water for each percentage point increase in concentration of acid solution (v /v), e.g. 1% HNO_3: 14.3 mL, 2%: 28.6 mL, 3%: 43 mL HNO_3, 10%: 143 mL HNO_3, made up to 1 L in a volumetric flask with MQ water. **Warning: Always add acid to water.**

-IW Matrix: 2% Nitric, **IW Rinse:** 3% Nitric

-HR Matrix: 10% Nitric, **HR Rinse:** 10% Nitric

IW Internal Standard: 100 ppm Be, In, Sc, 200 ppm Sb: Add 10 mL of each Be, In, Sc and 20 mL Sb elemental reference standards (1000 ppm) to a single 100 mL volumetric flask, make up with 2% trace metal clean nitric acid. 100 mL is enough for 1000 samples.
Sediment/Hardrock Internal Standard: The same as the IW internal standard except no Scandium is added.

–Acidified Synthetic seawater (ASSW): 1 L = 35 g NaCl/L in 2% Nitric Acid

IW Majors and Minors Standards

Step 1: Prepare Salt solutions

Consult the ICP User Guide for information of making major cation salt solutions. The solutions are stable indefinitely and may be used across multiple expeditions. However, the accuracy of the solutions may diminish due to evaporation, so they should be prepared fresh periodically.

Step 2: Prepare Primary In-House Cocktail

Prepare the following standard cocktail by combining the specified volume of each minor element standard and major cations from the in-house salt solutions in a 100 mL volumetric flask.

Minor element	Primary (ppm)	Volume (mL)
B	1000	3
Ba	1000	5
Fe	1000	0.5
Li	1000	0.5
Mn	1000	0.5
P	1000	0.5
Si	1000	2.5
Sr	1000	5
Na	~13000	62
Mg	~10000	12
Ca	~10000	4.5

K	~10000	4

Step 3: Prepare Calibration Standard Sets

Prepare both of the following calibration standard sets. Make up each standard to 100 mL with 2% nitric acid. Both sets are used in order to obtain values for barium and sulfur (as sulfate) which precipitate if in the same solution (even when acidified). These recipes detail solutions ready to be analyzed, there are no subsequent dilutions of these solutions. The standards may be prepared once at the beginning of an expedition and used for all subsequent interstitial water analyses.

	Standard	**In-House Cocktail (mL)**	**3.5% Acidified Seawater Solution (mL)**	**100 ppm Internal Standard (mL)**
In-house Standard Set	200%	20	0	2
	100%	10	0	2
	75%	7.5	2.5	2
	50%	5	5	2
	25%	2.5	7.5	2
	10%	1	9	2
	5%	0.5	9.5	2
	1%	0.1	9.9	2
	0%	0	10	2
	Standard	**IAPSO (mL)**		
IAPSO Standard Set	100%	10		2
	75%	7.5		2
	50%	5		2
	25%	2.5		2
	10%	1		2
	5%	0.5		2
	1%	1		2
	0%	0		2