Leeman ICP-AES: Quick Start Guide

V1.1 Approved: 8/21/2013 (DJH)
V362T Approved: July 2016 (DJH)

Introduction

The ICP-AES is used to run major and minor elements on squeezed IW samples and major element oxides and minor elements on digested solid samples.

ICP Operation

Ignite and position the plasma

1. Turn on argon and purge the optical chamber for at least 4 hours.
2. Turn on water chiller and ICP.
3. Start SALSA**;** answer **Yes** to camera purge status (assuming the optics have been purged > 4 hours with argon).
4. If *Maintenance* button or *Interlocks* button is red, click to see the issue.
5. Light the torch in **Instrument Control** > **Auto-Start**, and let the plasma stabilize for 45 min.
6. Load a method: **Method** > **Open**; add additional elements if needed using **Element Selection**.
7. Calibrate Mn:
    - Aspirate a *100 ppm Mn standard* for ~60 s.
    - Go to **Instrument Control** > **Position Plasma.**
    - Select the first Mn wavelength and wait ~5 min for the operation to complete.
    - The total intensity of the Mn solution should be within an order of magnitude of 1M counts.

Run Alignment Solutions (Set 1 and Set 2 or Individual Element)

1. Prepare tubes with alignment solution sets or individual element alignment solutions.
2. Name the chapter (run): click **Analysis** tab then **Analysis** > **New Chapter**.
3. Under the **Instrument Control > Method** tab, direct the needle (command "Cup") to the first set or element to be aligned. Allow the solution to aspirate for 60 seconds.
4. Change to the **Analysis** tab and click on **Echelle Spectrogram.**
    a) In the first text box, name the run (e.g., Ca_0.1 seconds)
    b) In the second text box, enter the time (e.g., 0.1)
    c) Uncheck both boxes
    d) Click **OK**
    e) Repeat Step 4 for 1 and 10 seconds (in total, each alignment will contain a 0.1 second, 1 second, and 10 second exposure)
5. When the spectrogram is done, click the **Image** tab (change the view in **Instrument Control** to *Axial* if only snow is visible).
6. Click the **Analysis** tab and expand the element menu. For each element, perform *"Align Element Wavelengths,"* as below.
7. Repeat Steps 3–6 for each set or individual element alignment. Be sure to allow the needle to aspirate the acid rise solution for 60 seconds between alignment solutions.

Align Element Wavelengths

For each peak, select the Echelle image from the **Echelle Images** selection box and adjust the following:

- **Viewport offset (XY)**: center the window around the signal of interest.
- **Viewport height**: increase discrimination between elements using 1, 3, or 5.
- **Background offset and width**: set at 2 squares immediately to the left and right of the peak.
- **Peak offset and width**: set *position* at left and right shoulders of the peak and *beginning* and *end* of peak at first and last squares that are higher than the baseline.

Save and Run a Sequence

1. Click on the **Sequence** tab to create a sequence.
2. Enter sample rack information and save sequence using **File** > **Save Rack**. A typical sequence is as follows:
    - Drift-1 [e,g., BHVO-2]
    - Blank
    - BIR-1 [calibration]
    - PWDR293984 [unknown sample]
    - PWDR848484
    - Drift-2
    - JGB-1 [calibration]
    - PWDR93984
    - PWDR938394
    - Drift-3
3. Click **Update** and verify that the sequence diagram matches samples to be run.
4. Click **Run Sequence**.

Analyze data and Upload to LIMS

1. Select **Analysis** tab > **Report** tab, and select the red folder that contains the data.
2. Click **All** > **Load,** select the LIMS format definition, and then click **Format1** > **CSV File.** Name the file the same as the chapter name given above in *"Run Alignment Solutions, Step 2."*
3. Open the exported CSV file in *ICP ANALYZER* and perform the following:

    Classify Samples: verify software-determined sample classifications.

    Check Drift: use the menu to control which lines are displayed.

    Check Calibration: the following options are available for each line:

    - Use drift correction.
    - Reject an entire line.
    - Choose first- or second-order curve fit.
    - View calibration, regression, and correlation.
4. Click **Generate Spreadsheet** to generate the *All Measurements* worksheet, which contains detailed data analyses steps.

    Note: Let the Scientists' view this file before any data is uploaded to the LIMS.
5. Click **Upload Data to LIMS**.

Shut Down the ICP

1. Let the instrument pump:
    - Rinse solution for 10 min
    - DI water for 10 min
    - air for 1 min (until spray chamber is dry).

2. In instrument control panel, select **Extinguish**.
3. Disengage pump tubing and shut down **SALSA**.
4. Shut off the ICP and water bath but leave the argon flowing for 20–30 min to allow the camera to equilibrate to room temperature. After the warm-up period, turn off the argon flow.

Preparing Samples

Solids

1. 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.
2. 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.
3. Pipette 1.25 mL filtered solution into a 20 mL scintillation vial; dilute with 8.75 mL bead dissolution solution.
4. Analyze on the ICP.

Interstitial Water Majors (Na, Ca, Mg, K)

1. Pipette 100 µL of acidified sample or standard into a sample tube and add 9.9 mL of IW matrix solution.
2. Analyze on the ICP.

Interstitial Water Minors (B, Ba, Li, Mn, Fe, Sr)

1. Pipette 500 µL of acidified sample or standard into a sample tube and add 9.5 mL of IW matrix solution.
2. Analyze on the ICP.

Reagent solutions

Solutions

–IW matrix solution (2% HNO_3/10 ppm Y): 20 L = 400 mL HNO_3 + 19.4 L reagent water + 200 mL 1000 ppm Y

–Synthetic seawater (SSW): 1 L = 35 g NaCl/L in 100%

–Rinse solution, 3% HNO_3: 1 L = 43 mL HNO_3/L reagent water

–Drift solution: 100% IAPSO

IW Majors

–Blank for IW majors: 10 mL = 100 µL reagent water + 9.9 mL IW matrix solution

–IW majors curve: Serial dilutions of IAPSO in DI water and 4% HNO_3 (unless otherwise noted):

Conc.	Components
120%	36.0 mL IAPSO + 24.0 mL 4% HNO_3
100%	30 mL IAPSO + 20.0 mL 4% HNO_3
75%	22.5 mL IAPSO + 7.5 mL reagent water + 30.0 mL 4% HNO_3
50%	15 mL IAPSO + 15 mL reagent water + 30.0 mL 4% HNO_3
25%	7.5 mL IAPSO + 22.5 mL reagent water + 30.0 mL 4% HNO_3
10%	3 mL IAPSO + 27 mL reagent water + 30.0 mL 4% HNO_3
5%	1.5 mL IAPSO + 28.5 mL reagent water + 30.0 4% HNO_3
2.5%	0.75 mL IAPSO + 29.25 mL reagent water + 30.0 4% HNO_3

IW Minors

Blank for IW minors: 10 mL = 500 µL SSW + 9.5 mL IW matrix solution

IW minors stock solution (100%): use 1000 ppm standards in 500 mL in acidified synthetic seawater (ASSW):

Minor element	Volume of 1000 ppm standard/500 mL ASSW
B	7.50 mL
Ba	2.50 mL
Fe	0.500 mL
Li	1.00 mL
Mn	1.50 mL
Si	15.0 mL
Sr	10.0 mL

IW minors standard curve: serial dilutions of IW minors stock solution in 30 mL of ASSW:

Conc.	Components
100%	30 mL stock
75%	22.5 mL + 7.5 mL ASSW
50%	15 mL + 15 mL ASSW
25%	7.5 mL + 22.5 mL ASSW
10%	3 mL + 27 mL ASSW
5%	1.5 mL + 28.5 mL ASSW
2.5%	0.75 mL + 29.25 mL ASSW

Sediments/Hard Rocks

–**Bead dissolution solution**, 10% HNO_3 + 10 ppm Y: 1 L = 143 mL HNO_3 + 857 mL DI water + 10 mL 1000 ppm Y

–**Rinse solution**, 10% HNO_3: 1 L = 143 mL HNO_3/L reagent water

–**Drift solution**: use one of the SRMs prepared as a sample

–**Blank for sediments/hard rocks**: 100% SSW

ICP Alignment Solutions

–**Mn solution**, 100 ppm: 100 mL = 10 mL 1000 ppm Mn + 90 mL reagent water

–**Set 1**, 10 ppm Al, Ca, Mg, Na, P, Ba, Sc, Y, Sr: 1 mL of each 1000 ppm stock to 100 mL 1% HNO_3

–**Set 2**, 10 ppm Fe, K, Mn, Si, Ti, Cr, Ni, V, Zr, Zn, Cu, Co, Nb, Ge: 1 mL of each 1000 ppm stock to 100 mL 1% HNO_3