#### WEBINAR

# Custom Analysis by Radial/Axial ICP-OES:

How to Reduce Interferences for 69 Elements

THURSDAY, JULY 1 | 9:00-10:00AM EST

SPEAKERS:

Thomas Kozikowski Manager Quality Control



Michael Booth
Director
Quality Control



### **Key Topics**

- Background Info
- Identify Wanted Improvements
- Creating the Calibration Standards
- Managing Spectral Interferences
- Validating the Method
- Benefits



# **Audience Participation**

- Today we are going to poll the audience throughout the presentation to better understand our collective challenges in the lab.
- Most of these questions will be multiple choice, and we will get to see the results after each question.
- Don't fall asleep!



#### Question 1

What type of ICP-OES instrument do you use in your laboratory?

- A. Axial (End-On Plasma)
- B. Radial (Side-On Plasma)
- C. Axial AND Radial (Dual-View Capability)
- D. Axial OR Radial (Must Choose before Plasma On)

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# What's the Purpose of this Method?

 Inorganic Ventures manufactures over 10,000 custom blends per year. Daily production can range from 30-60 custom blends per day.

 A testing method had to be created that could handle the high sample throughput and also be capable of guaranteeing the quality of widely different samples.

# Regular Blend Examples

| ppm  | IV-STOCK-3 (2% v/v HNO <sub>3</sub> )   | Prep 1 (1000X)  |
|------|---|---|
| 1000 | Ca, Mg, K, Na   | 1.0μg/g Ca, Mg, K,<br>Na  |
| ppm  | IV-ICPMS-71A (3% v/v HNO <sub>3</sub> )   | Prep 1 (10X)  |
| 10   | Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr3, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ho, K, La, Lu, Mg, Mn, Na, Nd, Ni, P, Pb, Pr, Rb, S, Se, Sm, Sr, Th, Tl, Tm, U, V, Yb, Zn | 1.0µg/g Ag, Al, As,<br>B, Ba, Be, Ca, Cd,<br>Ce, Co, Cr3, Cs, Cu,<br>Dy, Er, Eu, Fe, Ga,<br>Gd, Ho, K, La, Lu,<br>Mg, Mn, Na, Nd, Ni,<br>P, Pb, Pr, Rb, S, Se,<br>Sm, Sr, Th, Tl, Tm, U,<br>V, Yb, Zn |



# Complicated Blend Example

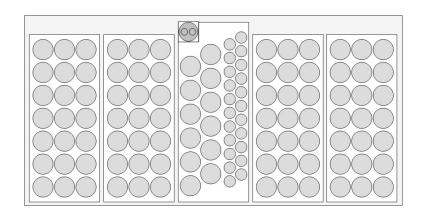
| ppm  | WW-LFS-1 (5% v/v HNO <sub>3</sub> ) | Prep 1 (1000X)             | Prep 2 (100X)                          |
|------|-------------------------------------|----------------------------|--|
| 1000 | K                                   | 1.0µg/g K                  | n/a                                    |
| 600  | P                                   | 0.6μg/g P                  | n/a                                    |
| 300  | Fe, Na                              | 0.3μg/g Fe, Na             | n/a                                    |
| 200  | Al, Ce, Mg, Se, Tl                  | 0.2μg/g Al, Ce, Mg, Se, Tl | n/a                                    |
| 100  | Ca, Pb                              | 0.1μg/g Ca, Pb             | 1.0μg/g Ca, Pb                         |
| 80   | As                                  |                            | 0.8μg/g As                             |
| 70   | Hg                                  |                            | 0.7μg/g Hg                             |
| 50   | Ni                                  | Too low for reliable       | 0.5μg/g Ni                             |
| 40   | Cr                                  | results; these will        | 0.4μg/g Cr                             |
| 30   | B, Cu, V                            | need another prep.         | 0.3μg/g B, Cu, V                       |
| 20   | Ba, Be, Cd, Co, Li, Mn Sr, Zn       |                            | 0.2μg/g Ba, Be, Cd, Co, Li, Mn, Sr, Zn |
| 7.5  | Ag                                  |                            | 0.075μg/g Ag (Below Curve, but ok)     |



#### **Production Workflow**

|                                | 30 Blends         | 45 Blends            | 60 Blends       |
|--------------------------------|-------------------|----------------------|-----------------|
| Average Preps Required         | 1.5/blend         | 1.5/blend            | 1.5/blend       |
| Prep Time at Bench             | 2.25hrs           | 3.4hrs               | 4.5hrs          |
| <b>Tube Positions Required</b> | 45                | 68                   | 90*             |
| Instrument time                | N/A: Run starts a | t end of workday wit | h auto shutdown |

<sup>\*</sup>Autosampler can only hold 84 tubes.



#### Old Custom Blend Method

- 3 Calibration Standards in 10% HCl (making Ag Photosensitive)
  - Blank (0μg/g) STD, Low (0.1μg/g) STD, High (1.0μg/g) STD
  - Includes 500μg/g of Cs ionization buffer for Li, Na, and K
    - Ionization Buffer needed to be added to all samples also
- 67 Elements as part of the method
  - Axial mode only
- With all elements present in the STD, some lines have major interferences making it difficult to utilize ideal lines

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#### Most Wanted Method Improvements

1. Decrease the number of preps required per sample Reminder - 1.5 preps/sample with old method

#### How?

- A. Expand the Calibration Curve Range
  - Increase the number of calibration solutions and increase the concentration of the "High" standard
  - Take advantage of Radial view to avoid the signal going overrange at higher concentrations
- 2. Add Rb and Os to the method

#### How?

A. Identify the correct calibration solution that will allow for the addition of Rb and Os and remain stable

#### Most Wanted Method Improvements

Decrease the amount of time required for each sample prep

Reminder - 1.5 preps/sample with old method

#### How?

- A. Remove Cs Ionization Buffer
  - Eliminates the step of spiking Cs buffer to each prep
- B. Change default prep volume from 20-50mL to 10mL
  - Requires an expanded calibration curve to ensure large enough sample aliquots
- 4. Decrease the amount of corrosion on the instrument Reminder Matrix for old method was 10% HCl

#### How?

A. Eliminate or lower the amount of HCl introduced to the instrument



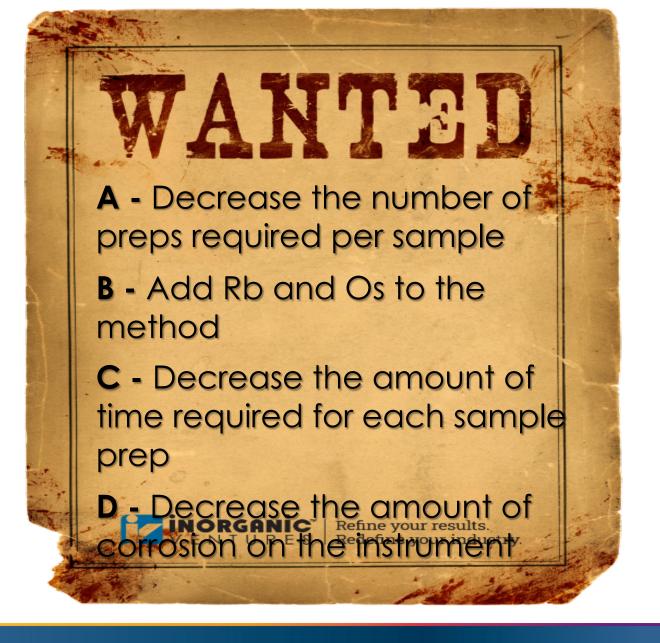
#### Corrosion Caused by HCl Over Time



- Load Coil
- Optical Plasma Interface (OPI)
- Water Lines
- RF Shielding
- Walls of the Torch Box

#### Question 2

What would be at the top of your most wanted list?





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#### Why split the calibration standards?

- Chemical compatibility
  - Certain elements don't behave well with other elements
  - Usually due to chemical form (Ba + S forms insoluble BaSO<sub>4</sub>)
- Matrix compatibility
  - Certain elements precipitate in the presence of HCl or HF
  - Certain elements require HCl or HF for stability
- Spectral interferences
  - Can happen with any element regardless of matrix

| Н  |    |    | All | ele | mer      | its a | t 1p | pm   | are | ok |    |    |    |    |    |    |    | Не |
|----|----|----|-----|-----|----------|-------|------|------|-----|----|----|----|----|----|----|----|----|----|
| Li | Be |    |     | 10  | 0% I     | HCI , | 0.2  | 2% F | łF  |    |    |    | В  | C  | Z  | 0  | F  | Ne |
| Na | Mg |    |     |     |          |       |      |      |     |    |    |    | Al | Si | Р  | S  | Cl | Ar |
| K  | Ca |    | Sc  | Ï   | <b>V</b> | Cr    | Mn   | Fe   | Со  | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr |    | Υ   | Zr  | Nb       | Мо    | Тс   | Ru   | Rh  | Pd | Ag | Cd | In | Sn | Sb | Te | -  | Xe |
| Cs | Ba | *  | Lu  | Hf  | Ta       | W     | Re   | Os   | Ir  | Pt | Au | Hg | TI | Pb | Bi | Ро | At | Rn |
| Fr | Ra | ** | Lr  | Rf  | Db       | Sg    | Bh   | Hs   | Mt  | Ds | Rg | Cn | Nh | Fl | Mc | Lv | Ts | Og |

\* La Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb

\*\* Ac Th Pa U Np Pu Am Cm Bk Cf Es Fm Md No

| Н  |    |    | All | elen | nent | ts at | 50p | pm,  | , not | ok |    |    |    |    |    |    |    | Не |
|----|----|----|-----|------|------|-------|-----|------|-------|----|----|----|----|----|----|----|----|----|
| Li | Be |    |     | 10   | 0% I | HCI , | 0.2 | 2% F | łF    |    |    |    | В  | C  | N  | 0  | F  | Ne |
| Na | Mg |    |     |      |      |       |     |      |       |    |    |    | Al | Si | Р  | S  | Cl | Ar |
| K  | Ca |    | Sc  | Ti   | V    | Cr    | Mn  | Fe   | Со    | Ni | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr |    | Υ   | Zr   | Nb   | Мо    | Тс  | Ru   | Rh    | Pd | Ag | Cd | In | Sn | Sb | Te | 1  | Xe |
| Cs | Ba | *  | Lu  | Hf   | Ta   | W     | Re  | Os   | Ir    | Pt | Au | Hg | TI | Pb | Bi | Ро | At | Rn |
| Fr | Ra | ** | Lr  | Rf   | Db   | Sg    | Bh  | Hs   | Mt    | Ds | Rg | Cn | Nh | Fl | Mc | Lv | Ts | Og |

| *  | La | Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Но | Er | Tm | Yb |
|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| ** | Ac | Th | Pa | U  | Np | Pu | Am | Cm | Bk | Cf | Es | Fm | Md | No |

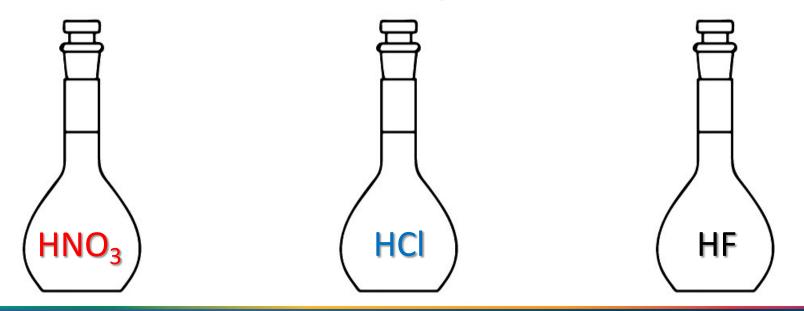
# Observations with increasing to 50ppm

- All of the Rare Earth Elements (REEs) + Th precipitate
  - Reason: HF
- W and Sn precipitate
  - Reason: Fluoride ligands stolen by B, P, Al, & others
- Ag and Pb Precipitate
  - Reason: HCl
- Pt precipitates
  - Reason: Formation of an insoluble Cs<sub>2</sub>PtCl<sub>6</sub> complex



#### Initial desired breakdown of standards

- Solution 1: Elements that are okay in only HNO<sub>3</sub>
- Solution 2: Elements that require HCl for some reason
- Solution 3: Elements that require HF for some reason



#### Solution Matrix - HF

| Н  |    |    | Not | Checl | ked by | y ICP | HF | Ele  | mer | nts |    |    |    |    |    |    |    | Не |
|----|----|----|-----|-------|--------|-------|----|------|-----|-----|----|----|----|----|----|----|----|----|
| Li | Be |    | HF  | "th   | ieve   | es"   | Å  | \voi | d H |     |    |    | В  | С  | Ν  | 0  | F  | Ne |
| Na | Mg |    |     |       |        |       |    |      |     |     |    |    | Al | Si | Р  | S  | Cl | Ar |
| K  | Ca |    | Sc  | Ti    | V      | Cr    | Mn | Fe   | Со  | Ni  | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr |    | Υ   | Zr    | Nb     | Мо    | Тс | Ru   | Rh  | Pd  | Ag | Cd | In | Sn | Sb | Te | -  | Xe |
| Cs | Ba | *  | Lu  | Hf    | Ta     | W     | Re | Os   | lr  | Pt  | Au | Hg | Τl | Pb | Bi | Ро | At | Rn |
| Fr | Ra | ** | Lr  | Rf    | Db     | Sg    | Bh | Hs   | Mt  | Ds  | Rg | Cn | Nh | Fİ | Мс | Lv | Ts | Og |

| *  | La | Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Но | Er | Tm | Yb |
|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| ** | Ac | Th | Pa | U  | Np | Pu | Am | Cm | Bk | Cf | Es | Fm | Md | No |

#### Solution Matrix - HCl

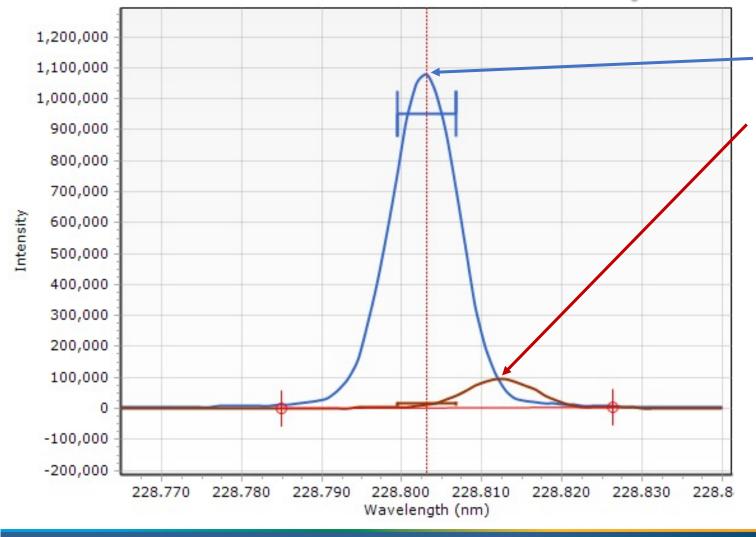
| Н  |    |    | Not | Check   | ked by | y ICP | НС | l Ele | eme  | nts |    |    |    |    |    |    |    | Не |
|----|----|----|-----|---------|--------|-------|----|-------|------|-----|----|----|----|----|----|----|----|----|
| Li | Be |    | Can | wor     | k w/o  | o HF  | 4  | voi   | d HC |     |    |    | В  | C  | Z  | 0  | F  | Ne |
| Na | Mg |    |     | Al Si P |        |       |    |       |      |     |    |    |    |    |    |    | Cl | Ar |
| K  | Ca |    | Sc  | Ë       | ٧      | Cr    | Mn | Fe    | Со   | Ni  | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr |    | Υ   | Zr      | Nb     | Мо    | Тс | Ru    | Rh   | Pd  | Ag | Cd | In | Sn | Sb | Te | =  | Xe |
| Cs | Ba | *  | Lu  | f       | Ta     | V     | Re | Os    | Ir   | Pt  | Au | Hg | T  | Pb | Bi | Ро | At | Rn |
| Fr | Ra | ** | Lr  | Rf      | Db     | Sg    | Bh | Hs    | Mt   | Ds  | Rg | Cn | Nh | Fl | Мс | Lv | Ts | Og |

| *  | La | Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Но | Er | Tm | Yb |
|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| ** | Ac | Th | Pa | U  | Np | Pu | Am | Cm | Bk | Cf | Es | Fm | Md | No |

### Spectral Interference Examples

- Cd 228.802 / As 228.812
  - Shoulder Interference
- Pd 340.458 / Zr / Th
  - Example where the background point can skew results
- Ir 212.681 / Y
  - Almost complete spectral overlay
  - Would not be able to use Y as an internal STD for Ir

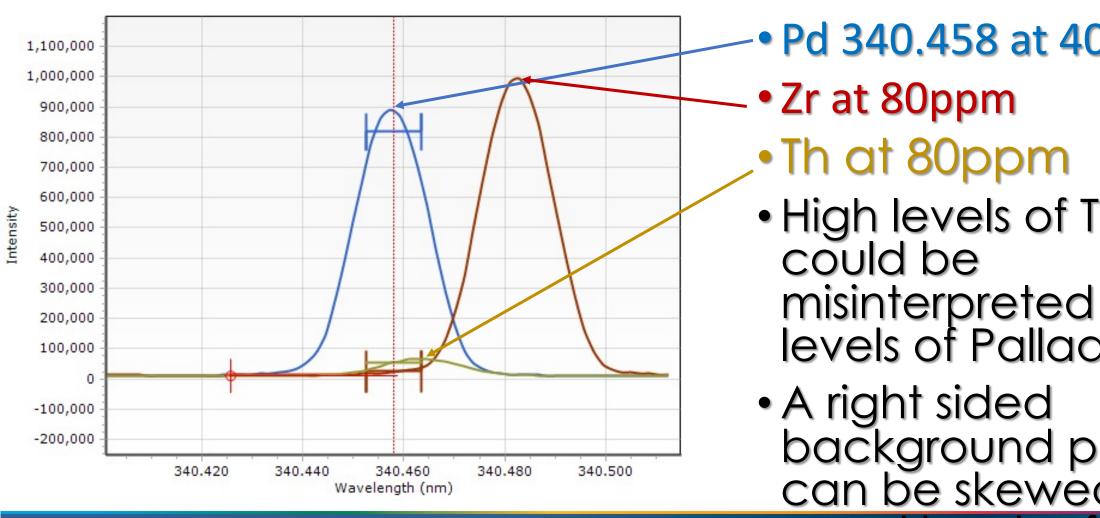
#### Cd 228.802 / As 228.812



- Cd 228.802 at 40ppm
- As 228.812 at 80ppm
- It's very easy to calibrate for Cadmium and then misinterpret a value for Arsenic
- Peak centering is critical for distinguishing between these two elements



#### Pd 340.458 / Zr / Th

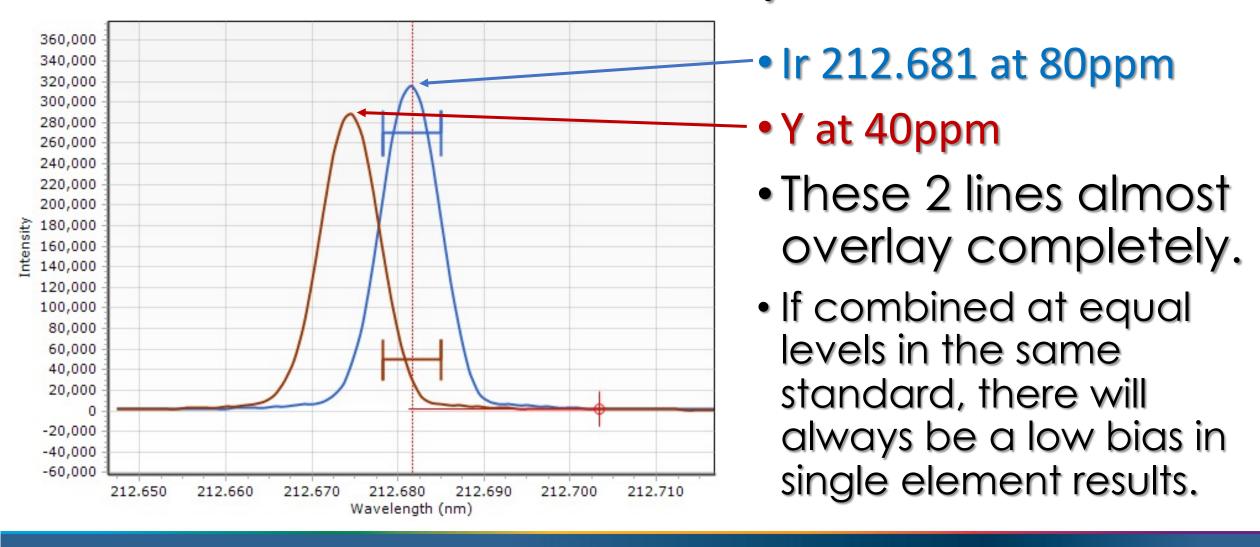


Pd 340.458 at 40ppm

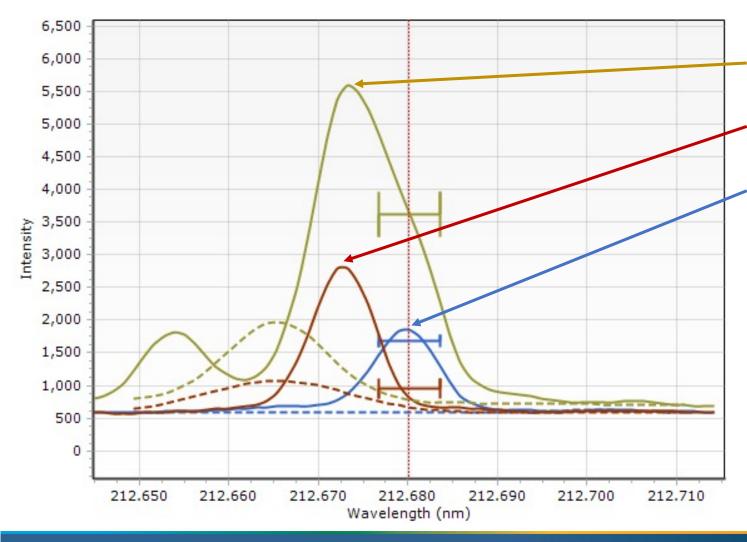
- High levels of Thorium misinterpreted as low levels of Palladium
- A right sided background point can be skewed by



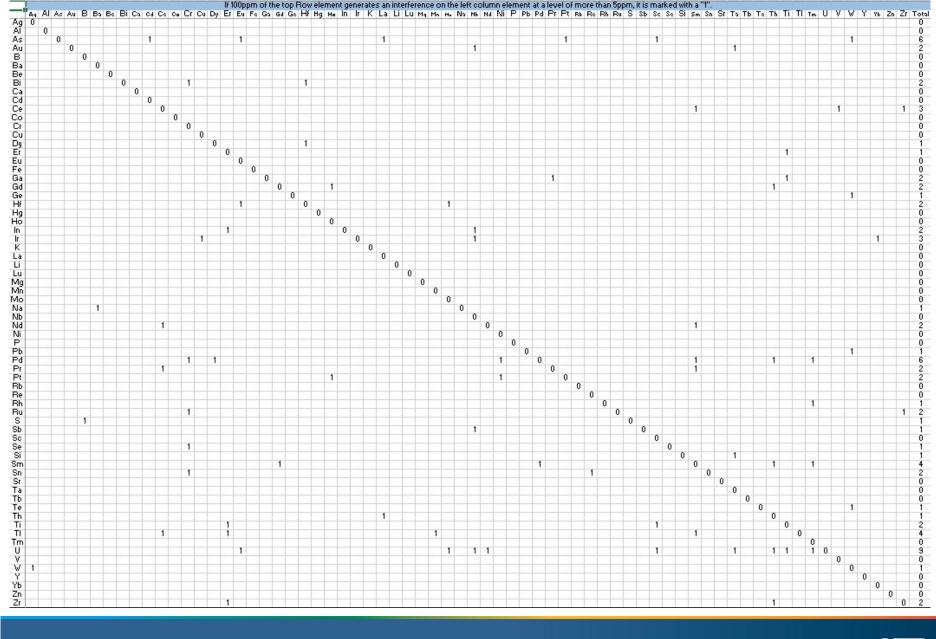
#### Ir 212.681 / Y



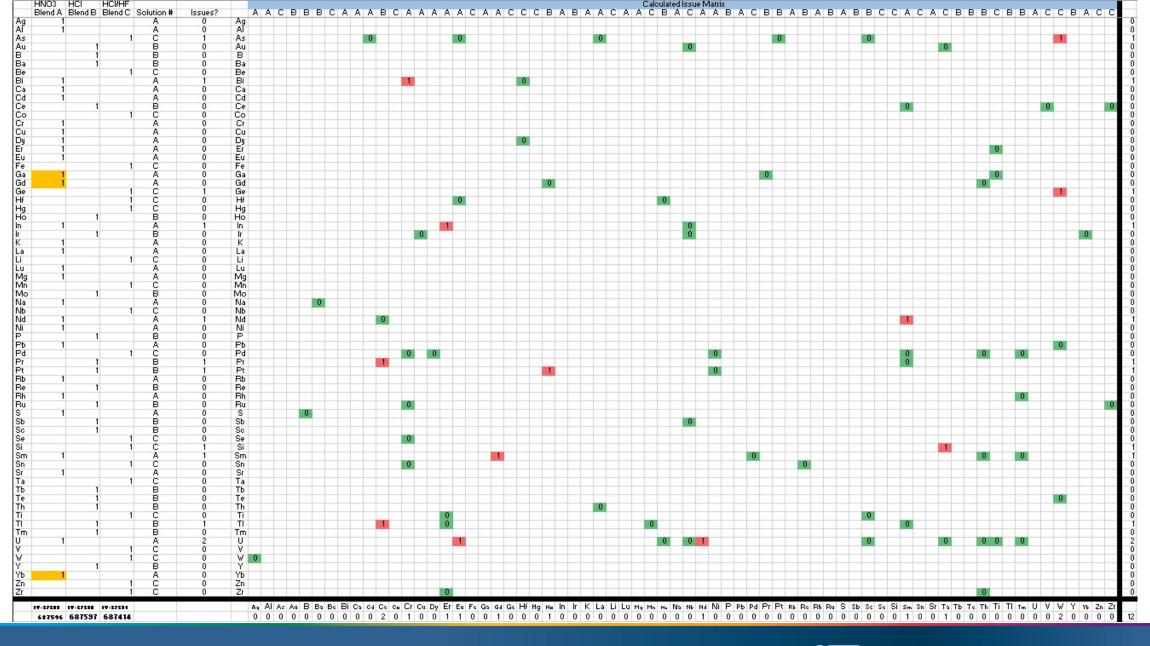
#### Ir 212.681 / Y



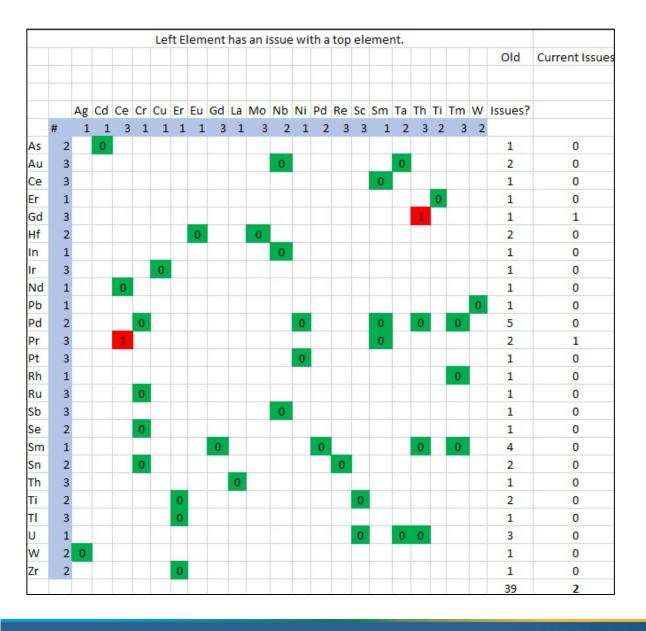
- Ir and Y both at 1ppm
- Y at 0.5ppm
- Ir 212.681 at 0.5ppm
- The Ir + Y at 1ppm looks like a misshapen peak.
- At first glance this looks like 1 peak



- Some offline math was used to determine a solution scheme that minimized the number of major interferences.
- Major was defined as > 5ppm.







- Further optimization of elements got the number of major interferences from 39 to only 2
- Great care was taken to ensure that elements were placed in compatible matrices

|    | 1  |    |      | MEB      | I-A  | ME   | B4-B    |      | EB4-C               | ME   | 34-Os  |    |    |    |    |    |    |    |    |
|----|----|----|------|----------|------|------|---------|------|---------------------|------|--------|----|----|----|----|----|----|----|----|
| Н  |    |    | Zero | 5% v/v H | INO3 | 5% ۷ | v/v HCl |      | v/v HCl<br>6 v/v HF | 5% v | /v HCl |    |    |    |    |    |    |    | Не |
| Li | Ве |    | Low  | 0.5      | opm  | 0.   | .5ppn   | n C  | ).5ppm              | n 0. | 5ppm   |    |    | В  | С  | N  | 0  | F  | Ne |
|    |    |    | Mid  | 5.0ppm   |      | 5.   | .0ppn   | n 5  | .0ppm               | า 5. | 0ppm   |    |    |    |    |    |    |    |    |
| Na | Mg |    | High | 50.0ppm  |      | 50   | .0ppn   | n 50 | ).0ppm              | 50.  | 0ppm   |    |    | Al | Si | Р  | S  | C  | Ar |
| K  | Ca |    | So   | c Ti     | V    | ,    | Cr      | Mn   | Fe                  | Со   | Ni     | Cu | Zn | Ga | Ge | As | Se | Br | Kr |
| Rb | Sr |    | Y    | Zr       | N    | b    | Мо      | Тс   | Ru                  | Rh   | Pd     | Ag | Cd | In | Sn | Sb | Te | _  | Xe |
| Cs | Ba | *  | Lu   | ı Hi     | T    | a    | W       | Re   | Os                  | lr   | Pt     | Au | Hg | F  | Pb | Bi | Ро | At | Rn |
| Fr | Ra | ** | Lı   | r R1     | D    | b    | Sg      | Bh   | Hs                  | Mt   | Ds     | Rg | Cn | Nh | Fl | Mc | Lv | Ts | Og |

\* La Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb

\*\* Ac Th Pa U Np Pu Am Cm Bk Cf Es Fm Md No



### Other changes / limitations

- Some wavelengths go overrange at 50ppm in Axial
  - With Radial they do not
  - Ca 393, Ba 455, Li 670, etc.
- Cs is no longer used as an ionization buffer
  - This limits linearity for K, Na, and Li, but Radial mitigates
  - Reduces the number of reagents required for analysis
  - Less chance of contamination (from pipetting)

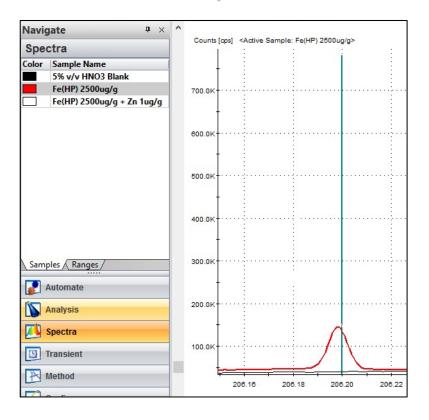
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# What is a Spectral Interference

A spectral interference occurs when multiple elements share the same or very close emission wavelengths.



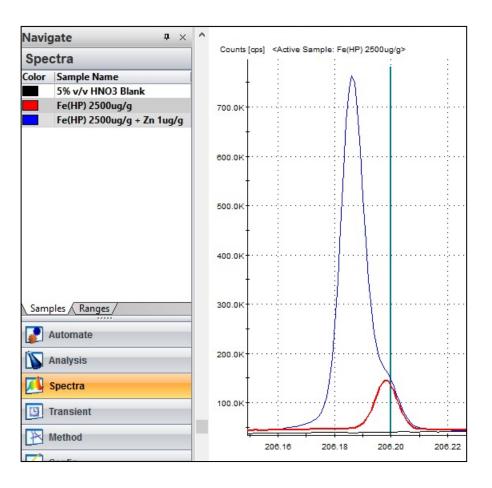
### Is this Zn, or is it Fe?

Sample: High Purity Fe at 2,500 μg/g

Software Emission Line: Zn 206.200nm

Does this sample contain a Zn impurity or is it a spectral interference from Fe?

### What is a Spectral Interference



#### Is this Zn, or is it Fe?

#### Sample:

High Purity Fe at 2,500  $\mu$ g/g (Red) High Purity Fe at 2,500  $\mu$ g/g + Zn at 1  $\mu$ g/g (Blue)

Software Emission Line: Zn 206.200nm

We can see that the red peak is actually Fe!

This example could have been less of a question with proper peak centering and wavelength calibration.

# What is a Spectral Interference

The intensity of light being emitted on shared wavelengths can be different based on element.

#### So how can you manage these interferences?

- Instrument Software is usually a good source, but it may not account for individual instrument performance.
- Running Single Element Standards is the gold standard to help determine what spectral interferences will cause problems for your method.

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### Question 3

Do you run any ICP-OES methods that need to account for spectral interferences?

A. Yes

B. No

C. Not Sure



# Building an Interference Table

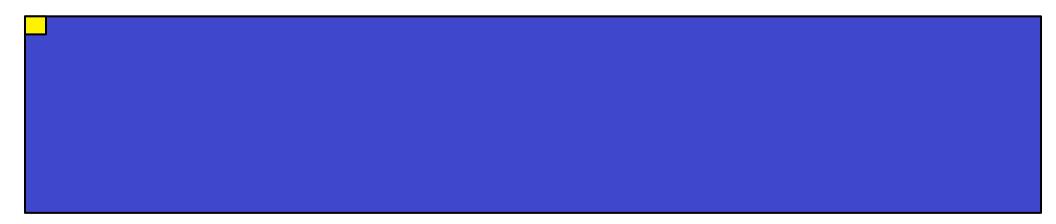
Running **High Purity Single Element Standards** for your method will allow you to construct an Interference Table that you can use to help interpret your data.

|   | $\sim$ |
|---|--------|
| X | a      |
| 9 |        |

| <u>a</u> | Single<br>Element<br>Standard<br>(80 µg/g) | Ga<br>417.204 nm<br>Radial | Gd<br>335.863 nm<br>Axial | Mn<br>259.372 nm<br>Axial | Pd<br>360.955 nm<br>Axial | Pr<br>422.532 nm<br>Axial | W<br>220.449 nm<br>Axial |  |
|----------|--|----------------------------|---------------------------|---------------------------|---------------------------|---------------------------|--------------------------|--|
|          | Al   | -                          | -                         | -                         | -                         | -                         | 0.9 μg/g                 |  |
|          | Ce   | 4.5 μg/g                   | -                         | -                         | 4.2 μg/g                  | -                         | -                        |  |
|          | Fe   | -                          | -                         | 0.1 μg/g                  | -                         | -                         | -                        |  |
|          | Sm   | -                          | -                         | -                         | 121 μg/g                  | 30.1 μg/g                 | -                        |  |
|          | Th   | -                          | 9.4 μg/g                  | -                         | 20.4 μg/g                 | -                         | -                        |  |

# Building an Interference Table

Our Previous Example showed an interference table for 5 single element standards with data from 6 monitored wavelengths.



Our new method uses an interference table for 75 single element standards with data from 396 monitored wavelengths.

Note: To successfully utilize this method you must keep your wavelength calibration up to date and use a validated method to keep your peaks centered.



### Interference Tables – Does it Work?

|              |              |              |            | Not a | Accounting fo | r Interferences |            | Accounting for Interferences |             |                |
|--------------|--------------|--------------|------------|-------|---------------|-----------------|------------|------------------------------|-------------|----------------|
| LIMS Analyte | Analyte Conc | Inst Element | Conc (ppm) | P/F?  | best % diff   | % passed lines  | # of lines | P/F?                         | best % diff | % passed lines |
| Ruthenium    | 1            | Ru           | 1.000      | Pass  | 0.18          | 67              | 6          | Pass                         | 0.18        | 83             |
| Samarium     | 1            | Sm           | 1.000      | Pass  | 3.29          | 50              | 6          | Pass                         | 0.44        | 67             |
| Scandium     | 1            | Sc           | 1.000      | Pass  | 0.10          | 100             | 6          | Pass                         | 0.10        | 100            |
| Selenium     | 1            | Se           | 1.000      | Pass  | 2.24          | 50              | 4          | Pass                         | 0.67        | 100            |
| Silicon      | 1            | Si           | 1.000      | Fail  | 13.64         | 0               | 8          | Pass                         | 6.39        | 13             |
| Silver       | 1            | Ag           | 1.000      | Pass  | 0.21          | 100             | 4          | Pass                         | 0.18        | 100            |
| Sodium       | 1            | Na           | 1.000      | Pass  | 0.77          | 50              | 4          | Pass                         | 0.77        | 50             |
| Strontium    | 1            | Sr           | 1.000      | Pass  | 0.00          | 100             | 8          | Pass                         | 0.00        | 100            |
| Sulfur       | 10           | S            | 10.000     | Pass  | 6.74          | 50              | 6          | Pass                         | 6.71        | 50             |
| Tantalum     | 1            | Та           | 1.000      | Pass  | 0.15          | 100             | 6          | Pass                         | 0.47        | 100            |
| Tellurium    | 1            | Te           | 1.000      | Pass  | 2.58          | 83              | 6          | Pass                         | 5.47        | 33             |
| Terbium      | 1            | Tb           | 1.000      | Pass  | 0.05          | 100             | 4          | Pass                         | 2.10        | 100            |

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### Validation Concerns

1. Stability of Calibration Standards

2. Calibration Verification

3. Matrix Matching Concerns

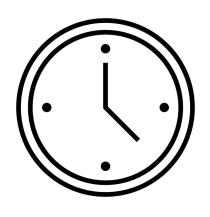
4. Instrument Setup

# Stability of Calibration Standards

The highest concentration versions of MEB4-A, MEB4-B, and MEB4-C have been entered into IV's Stability Testing Program.

 We have collected 18 months of stability data and will continue to collect data for 5 years even though we do not expect to identify instability in these blends.

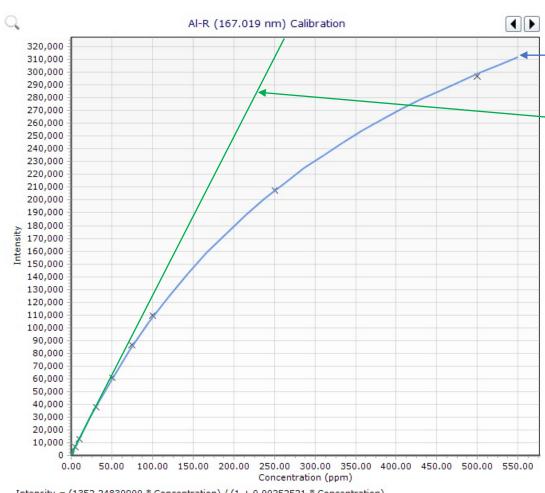
A dedicated technician runs our stability program and schedules retesting of lots based on date of manufacture and/or results of previous stability tests.



### Calibration Verification

- On the Agilent 5110 VDV, Axial view not always linear
- Parabolic Rational Function (Rational Method)
  - Agilent White Paper (Originally published in August 1977)
  - This scheme works for Echelle systems; not necessarily so on other types of optical systems
- Rational Method gave a much better calibration equation for all elements, so it was chosen for final validation verification

### Calibration Verification



Intensity = (1352.24830900 \* Concentration) / (1 + 0.00252521 \* Concentration)
Correlation coefficient: 0.99995

Rational Method

- Linear Regression
- Linear calibration would not calibrate past 50ppm
- Across all elements/wavelengths, rational fits were better models
- Axial benefitted most, radial wavelengths were mostly linear



# Peak Centering and Backgrounds

- Peak positions are locked down in a template
- Peak drift is corrected by "peak track lines" built into the Agilent Software (Carbon 192.027 & 5 Argon Lines)
- If the background is not "fitted", it is set once and never changed (positions preserved in the template file)
- Changing any of these parameters will invalidate the interference table and would require revalidation

### DLs and LOQs

- Detection Limits (DLs)
  - Depend on the element & wavelength
  - 3 x StDev of the blanks
- Limits of Quantification (LOQs)
  - A better limit for reliable measurements
  - 10 x StDev of the blanks
- These limits help us determine what dilution factor to use in samples with varying concentration levels

|  |                                    | IdealMin | Ideal | IdealMax | MaxTest |    | IdealMin                         | Ideal      | IdealMax | MaxTest |    | IdealMin | Ideal | IdealMax | MaxTest |
|--|------------------------------------|----------|-------|----------|---------|----|----------------------------------|------------|----------|---------|----|----------|-------|----------|---------|
| 1  | ٩g                                 | 0.05     | 5     | 50       | 80      | Но | 0.05                             | 5          | 50       | 80      | Ru | 0.05     | 5     | 50       | 80      |
|  | ΔI                                 | 0.05     | 5     | 50       | 80      | In | 0.20                             | 5          | 50       | 80      | S  | 0.5      | 5     | 50       | 80      |
| A  | ٩s                                 | 0.20     | 5     | 50       | 80      | Ir | 0.20                             | 5          | 50       | 80      | Sb | 0.5      | 5     | 50       | 80      |
| P  | ۱                                  | 0.05     | 5     | 50       | 80      | Κ  | 0.50                             | 5          | 50       | 80      | Sc | 0.01     | 5     | 50       | 80      |
| L  | В                                  | 0.05     | 5     | 50       | 80      | La | 0.05                             | 5          | 50       | 80      | Se | 0.2      | 5     | 50       | 80      |
| E  | 3a                                 | 0.01     | 5     | 50       | 80      | Li | 0.50                             | 3          | 50       | 80      | Si | 0.2      | 5     | 50       | 80      |
| E  | 3e                                 | 0.05     | 5     | 50       | 80      | Lu | 0.05                             | 5          | 50       | 80      | Sm | 0.1      | 5     | 50       | 80      |
| L  | Bi                                 | 0.20     | 5     | 50       | 80      | Mg | 0.05                             | 5          | 50       | 80      | Sn | 0.5      | 5     | 50       | 80      |
|  | Са                                 | 0.01     | 5     | 30       | 80      | Mn | 0.05                             | 5          | 50       | 80      | Sr | 0.05     | 5     | 40       | 80      |
|  | Cd                                 | 0.01     | 5     | 50       | 80      | Мо | 0.05                             | 5          | 50       | 80      | Та | 0.05     | 5     | 50       | 80      |
|  | Се                                 | 0.10     | 5     | 50       | 80      | Na | 0.20                             | 5          | 50       | 80      | Tb | 0.05     | 5     | 50       | 80      |
|  | Со                                 | 0.05     | 5     | 50       | 80      | Nb | 0.05                             | 5          | 50       | 80      | Те | 0.5      | 5     | 50       | 80      |
|  | Cr                                 | 0.05     | 5     | 50       | 80      | Nd | 0.1                              | 5          | 50       | 80      | Th | 0.5      | 5     | 50       | 80      |
|  | Cu                                 | 0.05     | 5     | 50       | 80      | Ni | 0.05                             | 5          | 50       | 80      | Ti | 0.05     | 5     | 50       | 80      |
| L  | Эу                                 | 0.05     | 5     | 50       | 80      | Os | 0.5                              | 5          | 50       | 80      | TI | 0.5      | 5     | 50       | 80      |
| L  | Er                                 | 0.05     | 5     | 50       | 80      | Р  | 0.5                              | 5          | 50       | 80      | Tm | 0.1      | 5     | 50       | 80      |
| LE   | Eu                                 | 0.05     | 5     | 50       | 80      | Pb | 0.2                              | 5          | 50       | 80      | U  | 0.5      | 5     | 50       | 80      |
| F  | -e                                 | 0.05     | 5     | 50       | 80      | Pd | 0.2                              | 5          | 50       | 80      | ٧  | 0.05     | 5     | 50       | 80      |
|  | Эa                                 | 0.20     | 5     | 50       | 80      | Pr | 0.1                              | 5          | 50       | 80      | W  | 0.2      | 5     | 50       | 80      |
|  | Gd                                 | 0.20     | 5     | 50       | 80      | Pt | 0.2                              | 5          | 50       | 80      | Υ  | 0.02     | 5     | 50       | 80      |
|  | эe                                 | 0.20     | 5     | 50       | 80      | Rb | 0.5                              | 50         | 50       | 80      | Yb | 0.02     | 5     | 50       | 80      |
| L  | Hf                                 | 0.05     | 5     | 50       | 80      | Re | 0.1                              | 5          | 50       | 80      | Zn | 0.05     | 5     | 50       | 80      |
| ŀ  | Нg                                 | 0.10     | 5     | 50       | 80      | Rh | 0.2                              | 5          | 50       | 80      | Zr | 0.05     | 5     | 50       | 80      |
| Ag Might need 10% HCl & 1ppm max Li & Rb Ideal Value |                                    |          |       |          |         |    | alue i                           | s not 5ppm | 1        |         |    |          |       |          |         |
|  | Must dilute Au, Hg, Os in ≥ 5% HCl |          |       |          |         |    | LOQ is same as LOW STD (0.5ppm)  |            |          |         |    |          |       |          |         |
|  | Typically diluted in 5% HCl        |          |       |          |         |    | Most elements diluted in 5% HNO3 |            |          |         |    |          |       |          |         |
|  |                                    |          |       |          |         |    |                                  |            |          |         |    |          |       |          |         |

- IV's Sample Prep Logic
- Prep elements to match the Mid STD
- In blends with differing concentration levels:
  - Prep high elements to match IdealMax
  - If low levels don't fall within IdealMin:
    - 2<sup>nd</sup> prep
- IdealMin is basically LOQ
- Max Test is highest validated level allowed

### Question 4

Does your method have specified minimum and maximum level prepping requirements to fall within your validated calibration range?

A. Yes

B. No

C. Not Sure



## Matrix Matching Concerns

- Switching samples from HNO<sub>3</sub> to HCl does not seem to have a significant effect on signal intensity
- Prepping samples in 10% HCl instead of 5% HCl does not cause major changes in signal intensity
  - It does enhance stability of low-level Ag (1ppm or less)
- Radial mode less affected by changes in matrix
- High Total Dissolved Solids (TDS) causes the most signal quenching – Radial View helps

### Instrument Setup

#### Sample Introduction **System**

We utilize a HF resistant system to help mitigate Si and B contamination.

- PTFE Cyclonic Spray Chamber
- PEEK Concentric Nebulizer
- Demountable Torch
- Alumina Injector

Radial modes

#### Rinse Regimen

#### **Global Rinses**

- Start of Run HNO<sub>3</sub> and HCl
- End of Run HNO<sub>3</sub>, HCI, 2.5% RBS-25, DI  $H_2O$

#### Method Rinse

 Usually 1% HNO<sub>3</sub>, (swap for 1% HCl if running Os)

#### **Dummy Samples**

 Technicians will insert blanks as "dummy samples" after sample solutions that have

• Ceramic OuterisTrulues was designed for the Agilent 5110 VpV utilizing Axial and

Radial modes.

• Chian total dissolved solids



# **Key Topics**

- Background Info
- Identify Wanted Improvements
- Creating the Calibration Standards
- Managing Spectral Interferences
- Validating the Method
- Benefits



### Benefits of the New Method

#### **Method Robustness**

 The expanded calibration curve and method validation allows for some samples to be run without prepping

#### <u>Time Savings</u>

- Reduced the preps/sample
  - We have reduced the preps/sample from 1.5 to 1.1 (~26% Reduction)
- Reduced the time/prep
  - Our QC Staff have reported a time savings of approximately 30%.

#### Reduction of "2nd" checks

Our QC Staff refers to blends that have one or multiple elements fail the initial check as "2nd" check briganicventures.com

# Are there any downsides?

#### Yes, but they are limited

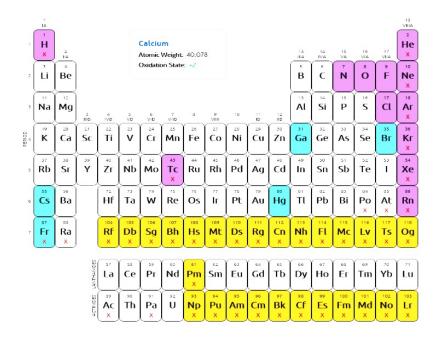
 Since the different calibration solutions have different matrices our technicians must switch diluent based on blend.

#### But you don't have to take my word for it...

- Most concentrated versions for Calibration Curve Standards MEB4-A, MEB4-B, & MEB4-C
- IV-57589, IV-59761, & IV-57591
- If you are looking for Os
  - CGOS1



# Technical Support – Available to Everyone Online Resources at inorganicventures.com









Customers can visit our website's Tech Center, which includes:

- Interactive Periodic Table
- Sample Preparation Guide
- Trace Analysis Guide
- ICP Operations Guide
- Expert Advice
- And much, much more.









### Thank You!

Please feel free to ask questions!