



SAFETY

Implications for Analytical Laboratory Testing

Elemental Impurities: Lessons Learned

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QUALITY



RESPONSIBILITY

The views and opinions expressed in the following presentation and discussion are those of the individual presenter and should not be attributed to any organization with which the presenter is employed or affiliated.



Best Practices

- Digestion Considerations
 - Total Digestion
 - Exhaustive Digestion
- Analytical Considerations
 - Interference Mitigation
 - Internal Standardization
 - LOQs for multiuse materials

One size may fit many but not all



Digestion Considerations

■ Total Digestion

- HNO_3 😎
- HCl 😊
- H_3PO_4 😞

P^{31} $\text{PP}^{62}=\text{Ni}$ $\text{PN}^{45}=\text{Sc}$ $\text{PN}_2^{59}=\text{Co}$

- HBF_4 😞

Purify H_3BO_4 to start

HF in significant quantities

Exothermic Reaction

- Microwave 180°C _IPV
- Microwave 250°C _SRC

■ Exhaustive Extraction

- HNO_3 😎
- HCl 😊

- Microwave 175°C _R10H10



Digestion Considerations

■ Total Digestions

- Target Visual Clarity
- Majority of Requests
- HF vs HBF_4
 - Insoluble Fluorides CaF_2 MgF_2
 - Titanium Oxide; Silicates; Talc
- Development Work
- Special Standards

■ Exhaustive Extraction

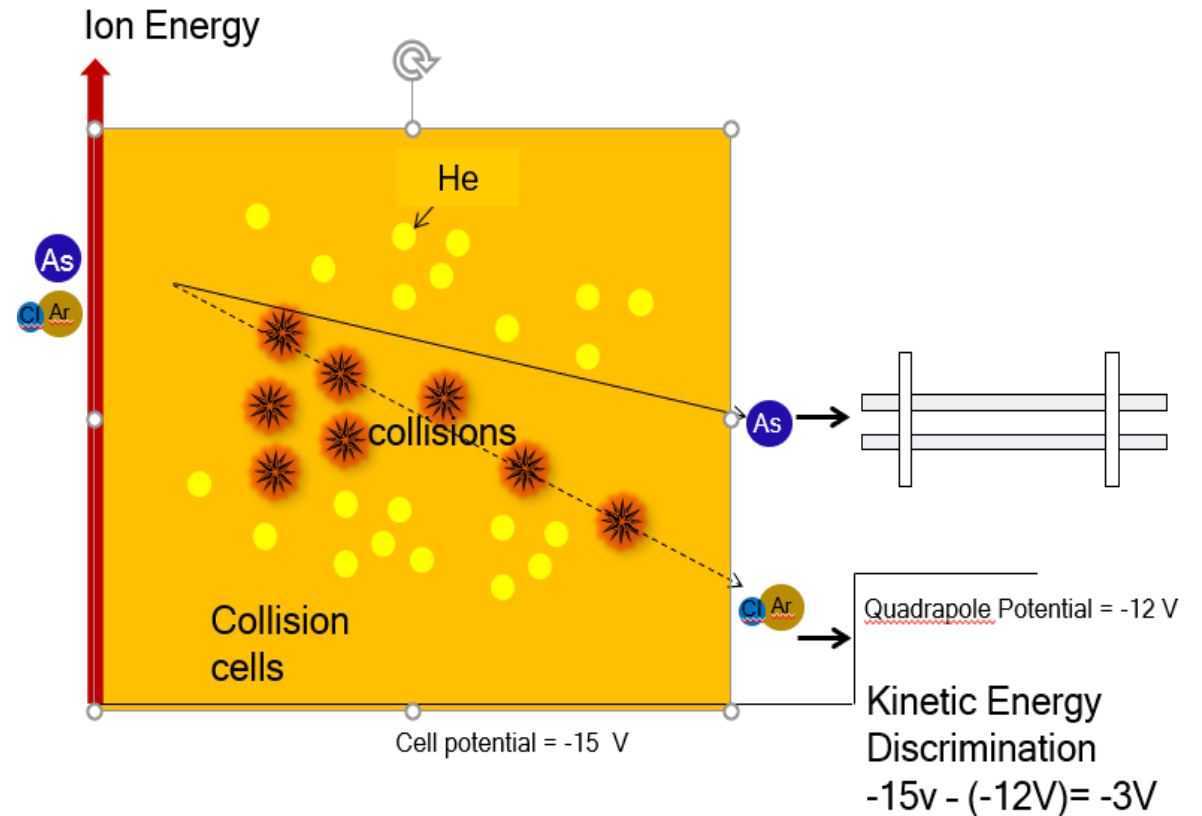
- Justification Fears
- Increasing % of Requests
 - Raw Materials
 - Cosmetics Formulations
- Fixed and Reproducible
- Standard Matching Easier
- Study Results Comparable

Analytical Considerations

Interference Removal

Collision Cells

- Inert Gas - Helium
- Minimal Optimization
- Preferred for Ease 😊
- Selective Problems (V)



Analytical Considerations

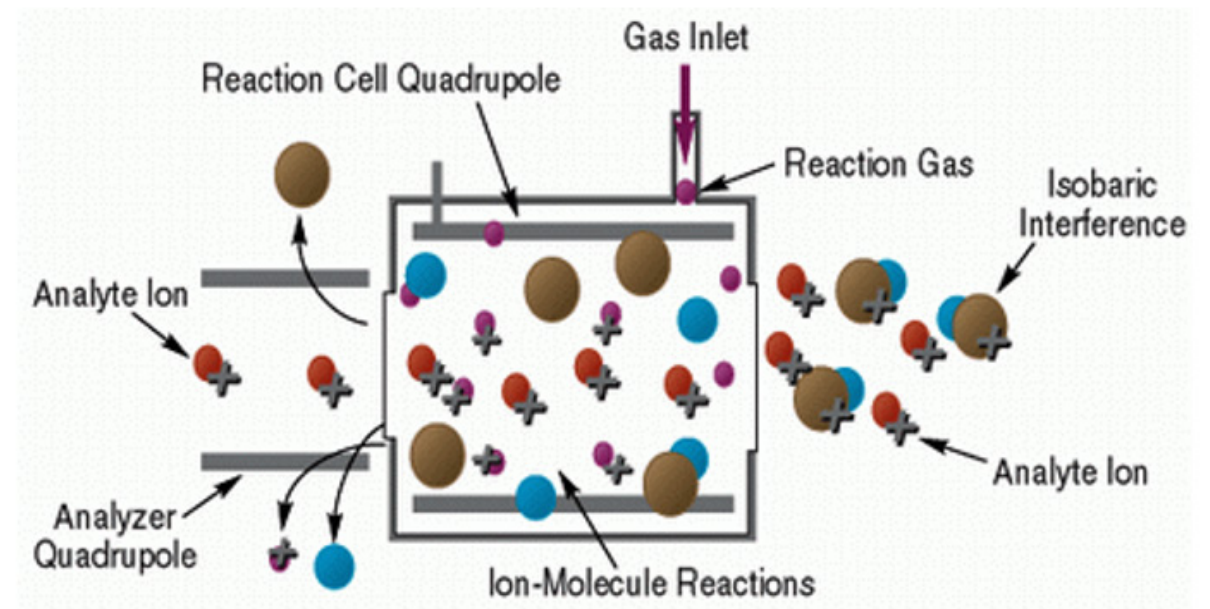
■ Interference Removal

• Reaction Gases

- Reactive Gas

NH_3 , O_2 , CH_3

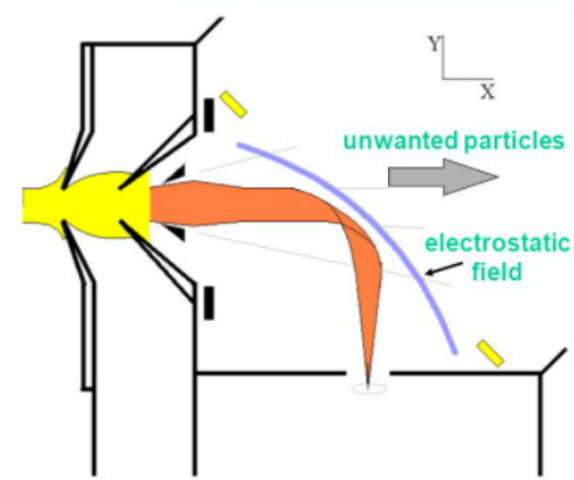
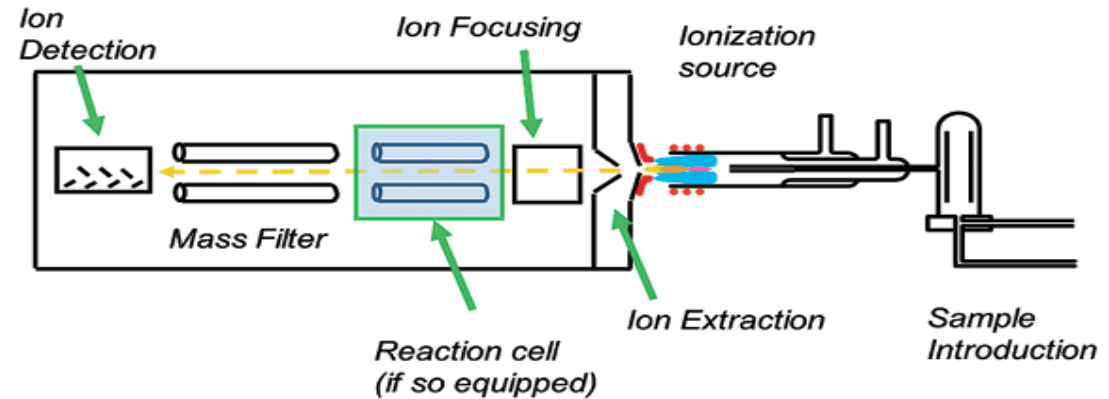
- Optimization Required ☹️
- Better Sensitivity
- Selectivity



Analytical Considerations

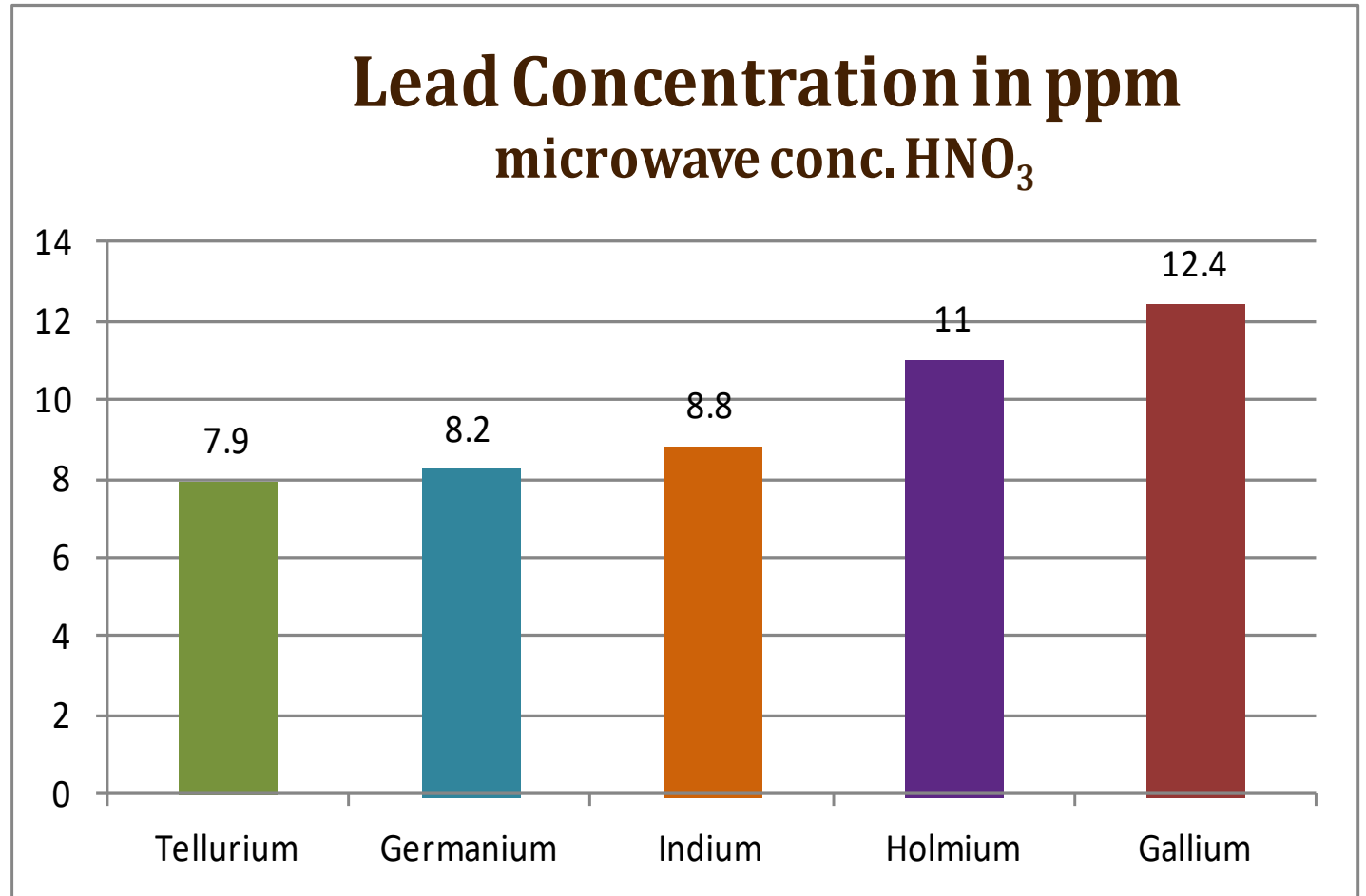
- Internal Standards

- Association Considerations
- Ionization Potential
- Mass Dependency
- Element Specific
- Instrument Specific

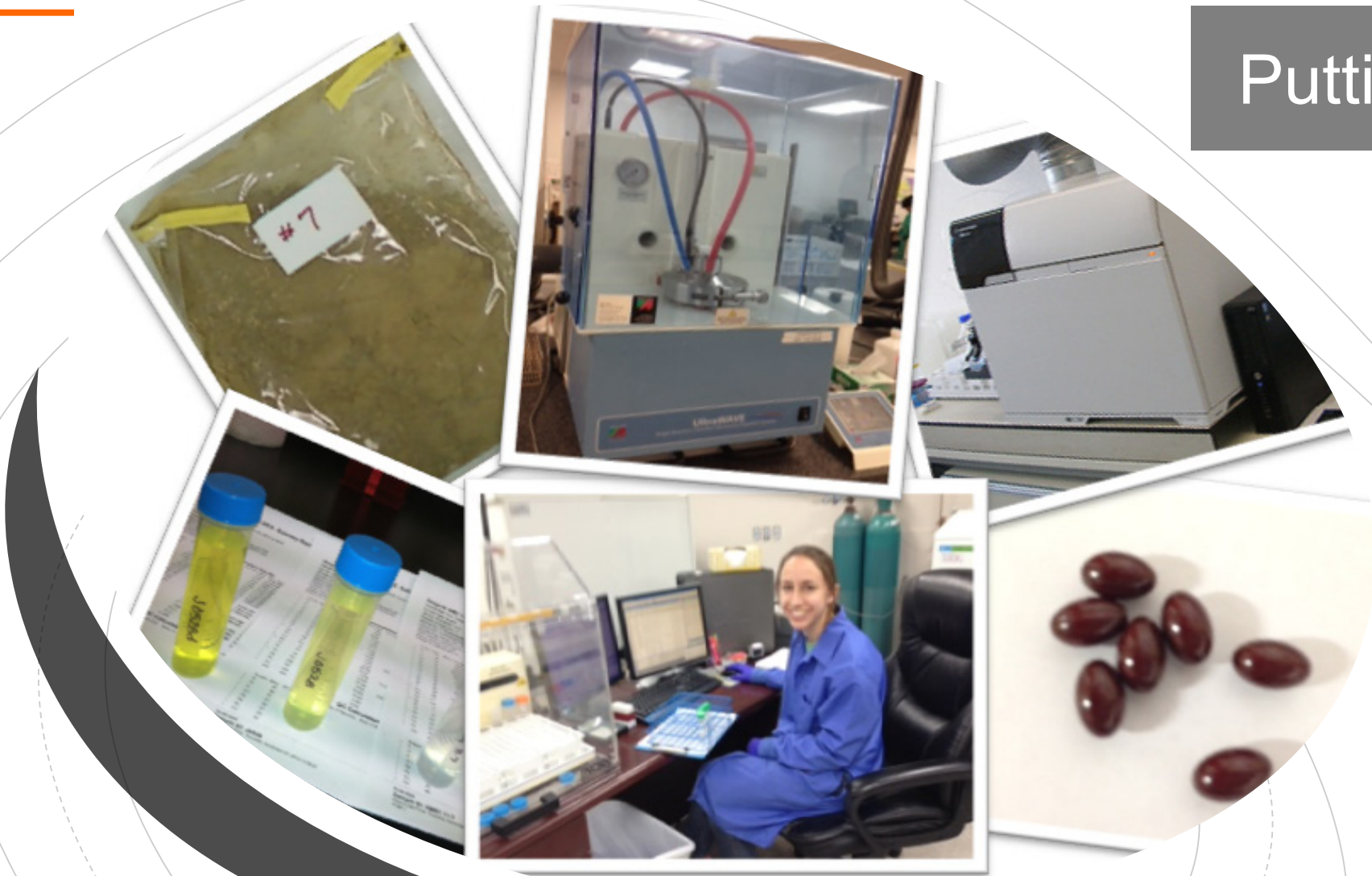


Analytical Considerations

- Internal Standards
 - Ionization Potential
 - Mass Dependency
 - Element Specific
 - Instrument Specific



Putting it all together



One size may fit many but not all

- Standardization is desired but materials and instrumentation differences require some flexibility
- Method Development / Applicability / Transfer
- Validation Consideration
 - USP <233> Alternative Technique Criteria
 - Spiking of Samples
- LOQ considerations



LOQ Considerations

Parameter	Result	Serving Size	Matrix Spike Recovery	Matrix Spike Level	Specification
Antimony	<45 ug/serving	4.76 g	118%	75.6 ug/g	≤ 1200 ug/serving
Arsenic	<0.57 ug/serving	4.76 g	94%	0.94 ug/g	≤ 15 ug/serving
Cadmium	<0.38 ug/serving	4.76 g	103%	0.32 ug/g	≤ 5 ug/serving
Chromium	<410 ug/serving	4.76 g	98%	693 ug/g	≤ 11000 ug/serving
Cobalt	<1.8 ug/serving	4.76 g	99%	3.15 ug/g	≤ 50 ug/serving
Copper	<110 ug/serving	4.76 g	111%	189 ug/g	≤ 3000 ug/serving
Lead	<0.19 ug/serving	4.76 g	103%	0.32 ug/g	≤ 5 ug/serving
Mercury	<1.1 ug/serving	4.76 g	96%	1.89 ug/g	≤ 30 ug/serving
Molybdenum	<110 ug/serving	4.76 g	116%	189 ug/g	≤ 3000 ug/serving
Nickel	<7.4 ug/serving	4.76 g	97%	12.6 ug/g	≤ 200 ug/serving
Selenium	<5.6 ug/serving	4.76 g	101%	9.45 ug/g	≤ 150 ug/serving
Silver	<5.6 ug/serving	4.76 g	96%	9.45 ug/g	≤ 150 ug/serving
Tin	<220 ug/serving	4.76 g	104%	378 ug/g	≤ 6000 ug/serving
Vanadium*	<3.7 ug/serving	4.76 g	99%	6.30 ug/g	≤ 100 ug/serving

Thank you!

Do you have any questions?



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