BE BOLD

2022 INORGANIC VENTURES WEBINAR SERIES

CONSIDERATIONS FOR

Sample Preparation and Working Solutions

THURSDAY, MAY 19 | 9:00–9:30AM EST

PRESENTED BY:

James King, M.Sc.
Director, Production

Overview

Matrix/analyte compatibility considerations

Special cases/solutions

Matrix Considerations

Analytes involved – what matrices do your analytes of interest require for stability?

Method requirements/limitations?

Instrumentation – interferences, system cleanliness, etc.

Safety concerns/regulations – does your facility prevent any common matrix components?

Nitric Acid

Majority of elements are stable in dilute nitric acid matrices; some will require minimum amounts of other acids.

ICP-MS users tend to prefer HNO_3 due to spectral cleanliness.

HNO_3 can oxidize Os in certain conditions making it volatile. This presents a health hazard and makes reliable measurements extremely difficult.

HNO_3 Compatibility

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca		Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr		Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	*	Lu	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	**	Lr	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn	Nh	Fl	Mc	Lv	Ts	Og

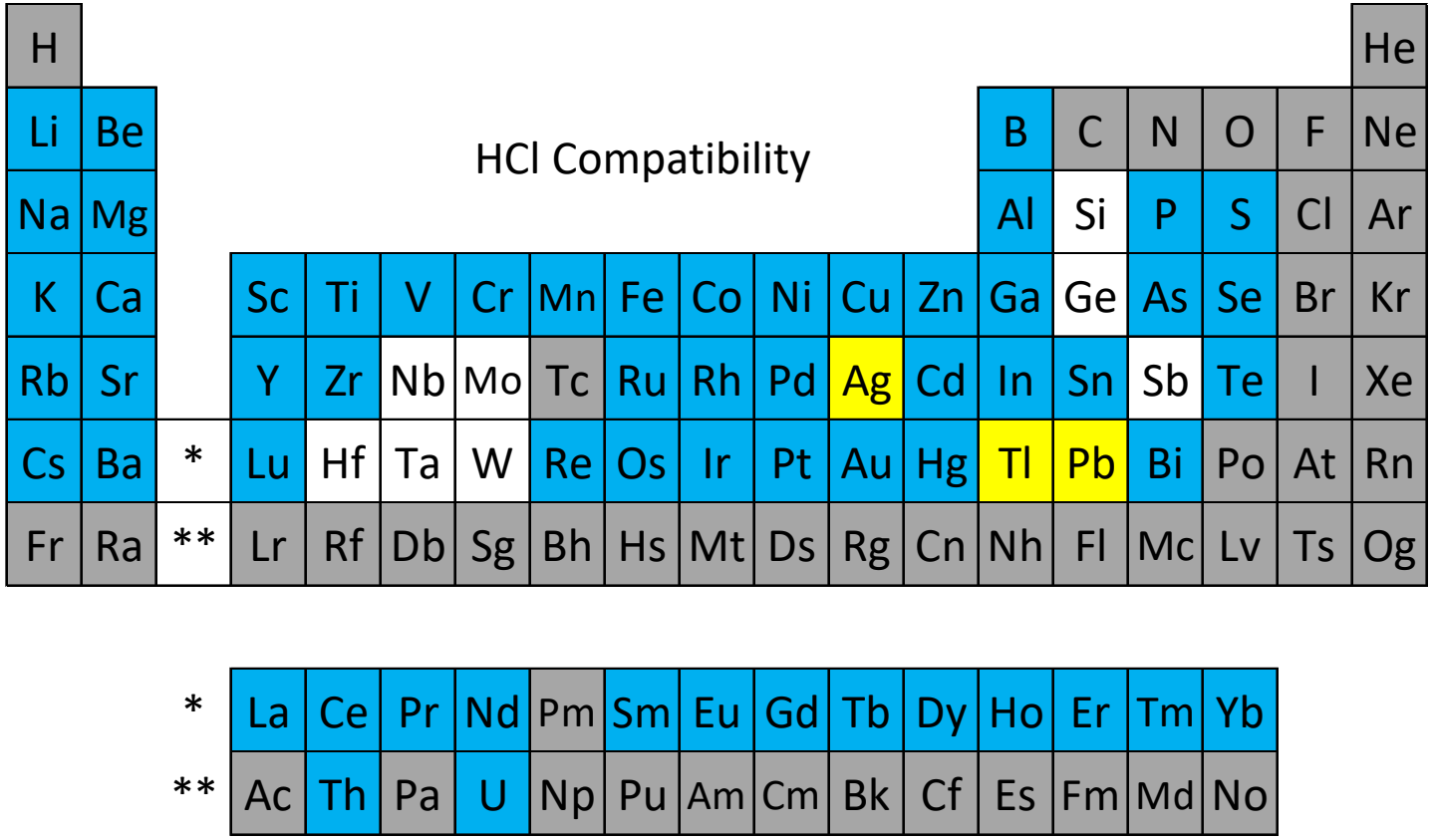
*	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb
**	Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No

Hydrochloric Acid

Nearly all elements are stable in dilute HCl.

Cl^- can cause interferences on the ICP-MS, especially Arsenic and Vanadium.

Memory effects from HCl-containing stock standards can be a stability concern for elements like Silver and Thallium.



Hydrofluoric Acid

Certain elements require an amount of HF for long-term stability.

HF can be extremely useful in dissolutions and digestions.

Introduction system compatibility?

Rare earth elements, Group 2A elements and Th tend to precipitate out of solution as fluorides.

Elements such as Al, As, Bi, P can mitigate stability issues by tying up excess F^- ions in solution

HF Compatibility

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca		Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr		Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	*	Lu	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	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

Matrix Considerations

Be mindful of matrix-matching standards and samples.

Keep it simple when buying/making stock solutions (same acids, same concentrations, etc.).

What impact will your matrices have on your instrumentation?

Be aware of the alternatives that exist for various compositions and any associated consequences.

Stock/Working Solutions

The stock solution(s) being used must be designed with long-term stability in mind or the resulting working solutions could be compromised.

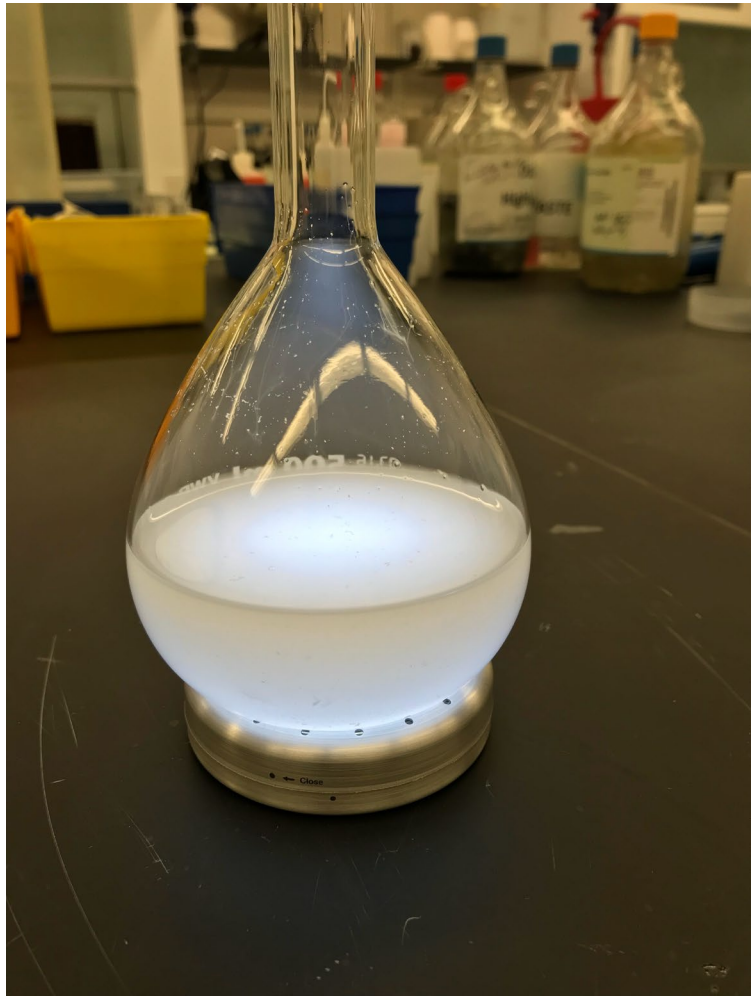
2 stable stock solutions >> 1 stock solution with questionable stability.

Reach out to your CRM provider for info on starting materials and matrix availabilities.

Special Cases

What are some of the common incompatibilities we come across?

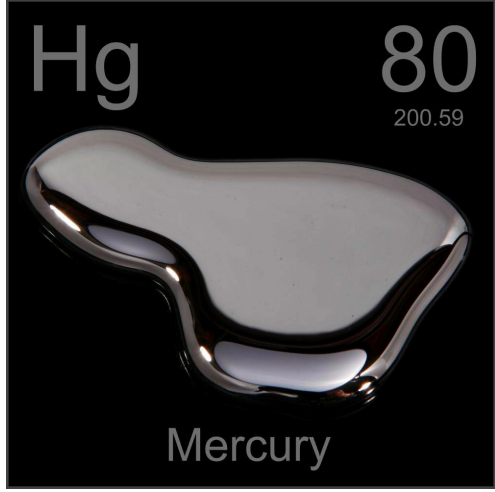
How do we overcome these?



Special Cases

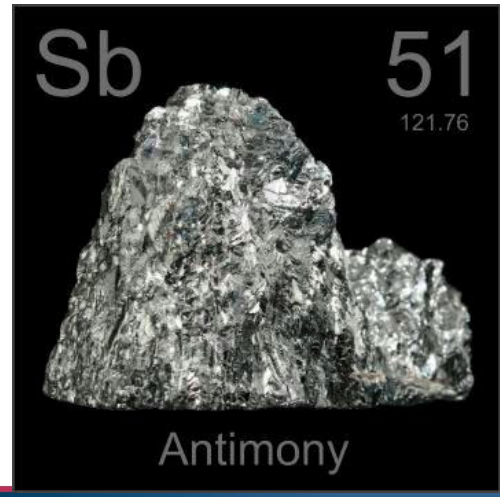
Problem	Solution
Alkaline earth/rare earth fluoride instability	Add alkaline earths/rare earths last after partial dilution; split solutions
AgCl insolubility	Increase HCl content, protect from light
TlCl insolubility	Use Tl_2O_3 starting material (contact CRM vendor for availability)
As/Bi/Pb instability at high concentrations (≥ 1000ppm)	Use 15% v/v HNO_3
Os volatility due to HNO_3-bearing analytes	Add Os last after diluting nearly to final weight/volume

Special Cases – Antimony + Mercury



- Sb: stable in HF or tartaric acid in conjunction with HNO_3/HCl
- Hg: stable in HNO_3 (in borosilicate glass), HCl (any container)
- Tartaric acid reduces Hg to metallic form
- HNO_3 above 3% v/v will degrade tartaric acid, destabilizing Sb
- HF attacks borosilicate glass (Na, B, Si contamination)
- What's the solution??

Dilute HCl/HF is the best available option for long-term stability.



Special Cases – Elemental Impurities

Element	Class
Cadmium	1
Lead	1
Arsenic	1
Mercury	1
Cobalt	2A
Vanadium	2A
Nickel	2A
Thallium	2B

Element	Class
Gold	2B
Palladium	2B
Iridium	2B
Osmium	2B
Rhodium	2B
Ruthenium	2B
Selenium	2B
Silver	**2B**

Element	Class
Platinum	2B
Lithium	3
Antimony	3
Barium	3
Molybdenum	3
Copper	3
Tin	3
Chromium	3

Special Cases – Elemental Impurities

Element	Class
Cadmium	1
Lead	1
Arsenic	1
Mercury	1
Cobalt	2A
Vanadium	2A
Nickel	2A
Thallium	2B

Stock A: Dilute HCl

Element	Class
Gold	2B
Palladium	2B
Iridium	2B
Osmium	2B
Rhodium	2B
Ruthenium	2B
Selenium	2B
Silver	**2B**

Stock B: High HCl/Light Sensitive

Element	Class
Platinum	2B
Lithium	3
Antimony	3
Barium	3
Molybdenum	3
Copper	3
Tin	3
Chromium	3

Stock C: Dilute HNO_3/HF

Special Cases – Elemental Impurities

Element	Class
Cadmium	1
Lead	1
Arsenic	1
Mercury	1
Cobalt	2A
Vanadium	2A
Nickel	2A
Thallium	2B

Stock A: Dilute HCl

Element	Class
Gold	2B
Palladium	2B
Iridium	2B
Osmium	2B
Rhodium	2B
Ruthenium	2B
Selenium	2B
Silver	**2B**

Stock B: Dilute HCl

Element	Class
Platinum	2B
Lithium	3
Antimony	3
Barium	3
Molybdenum	3
Copper	3
Tin	3
Chromium	3

Stock C: Dilute HNO_3/HF

Summary

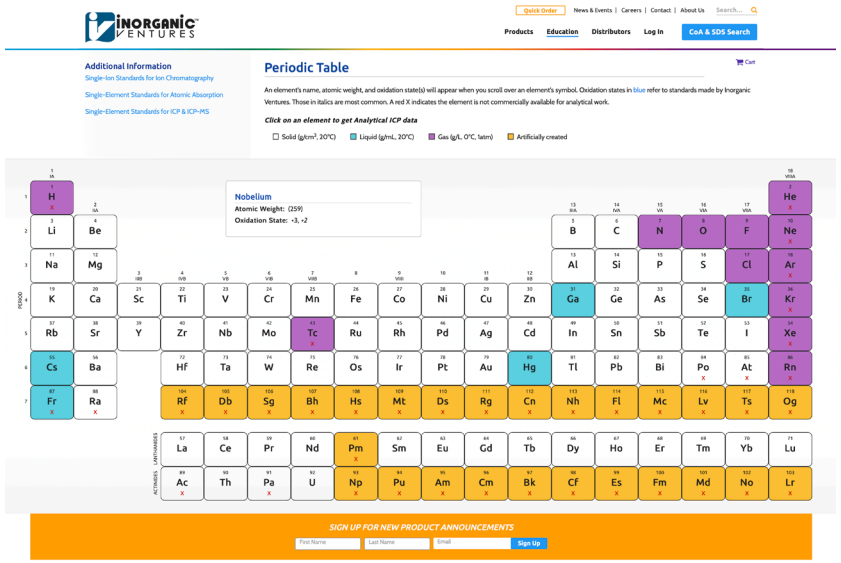
Be familiar with the matrix options for analytes of interest.

Start with method for reliable measurements and strive for better efficiency, not the other way around.

Always be aware of what is in your stock standards; CRM providers will be able to provide this information.

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.**