Element Stability and Compatibility

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Stability and Compatibility of …. ?

Discussion Topics

- Limited to solutions
- Two basic types of solutions
  - ICP calibration standards
  - Samples
- Types of stability and compatibility
  - **chemical**
  - physical
  - ooops….
  - *other*

Stability and Compatibility of …. ?

Discussion Topics

- Limited to solutions
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- Types of stability and compatibility
  - **chemical**
  - physical
  - ooops….
  - *other limits*

Stability – chemical and physical *values* do not change with time

Compatibility – elements don't form insoluble compounds

Limits

Limits of chemistry

- Solubility!
  - Must be electrically neutral (i.e., counter ions)
  - Common anions NO_3, Cl, SO_4, PO_4, F
  - *Salt solubility important*
- Compatibility (Ag + Cl, Th + F)

Limits of ICPs

- How many elements? *….about 75 for both OES and MS*
- Is it feasible to measure all 75? *….depends on concentration*

Any limits due to both ICP and chemistry?

ICP Limits

High concentration limit

- Total dissolved solids (TDS)
  - <0.2% TDS (2000 ppm) for ICP-MS
  - <2% TDS (20,000 ppm) for ICP-OES
- Newer instruments allow higher TDS (sample intro systems)
- 75 elements combined @ 100 ppm each = 0.75% TDS

Low concentration limit = Detection/Quantification limits

TDS vs. DL/QL

Can you measure 1 ppb in 10% TDS?

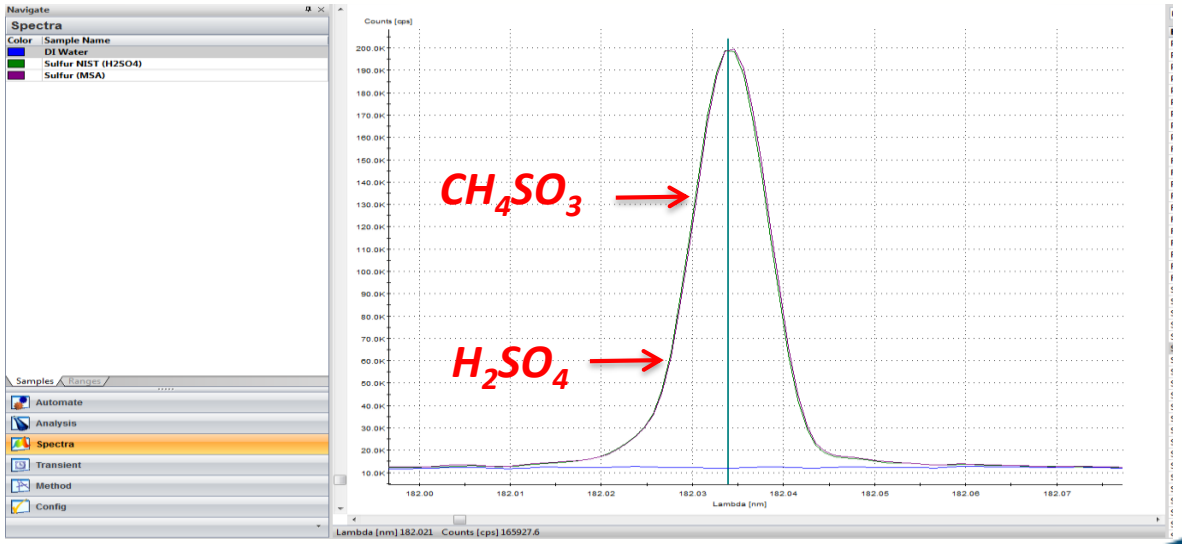
ICP + Chemistry Limits

Does starting material matter for ICP?

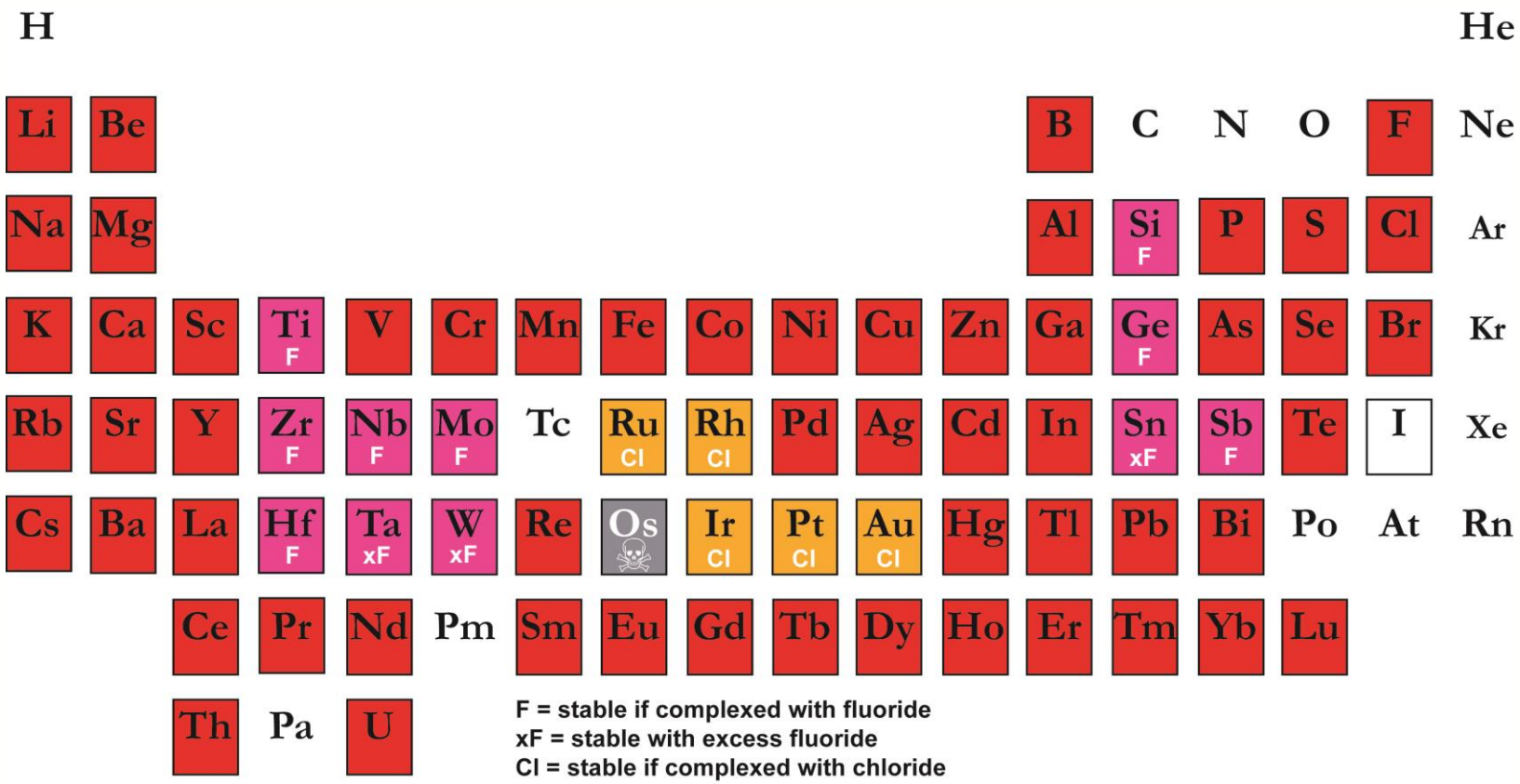
- Not for elements

  e.g., *methanesulfonic acid sulfur (CH_4SO_3) is indistinguishable from H_2SO_4*

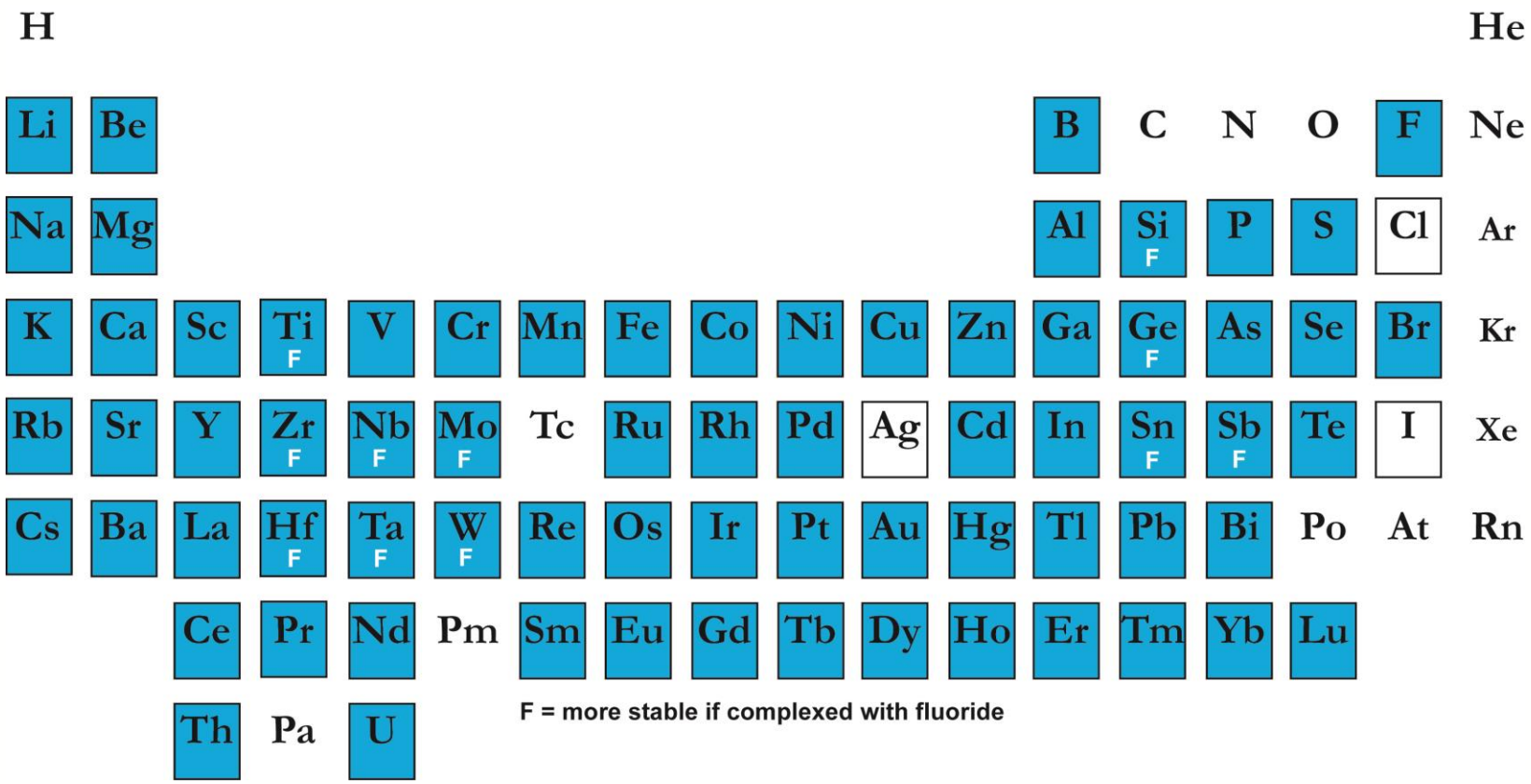
- Only a concern for matrix contributions



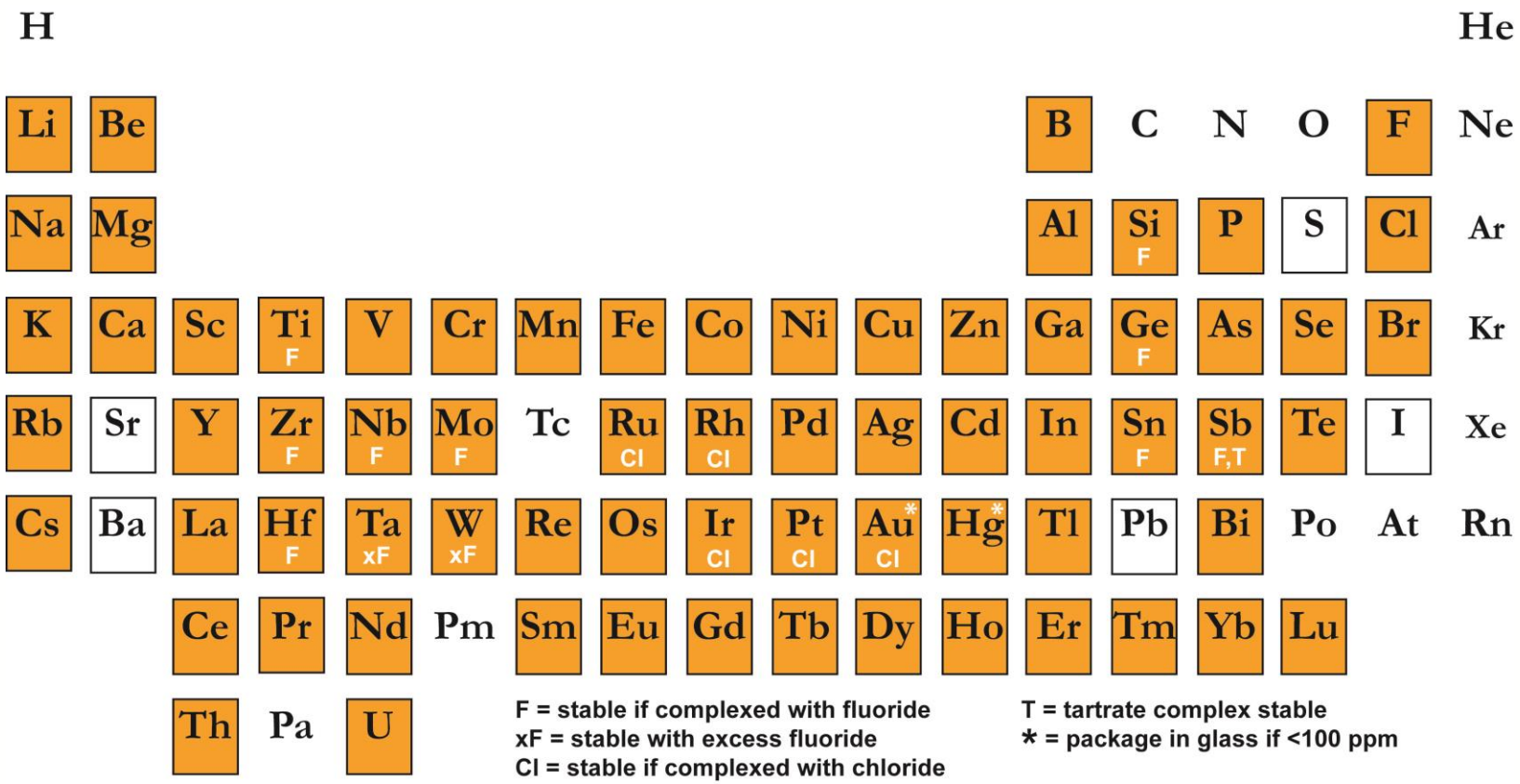
Chemistry Limits – HNO_3 solubility



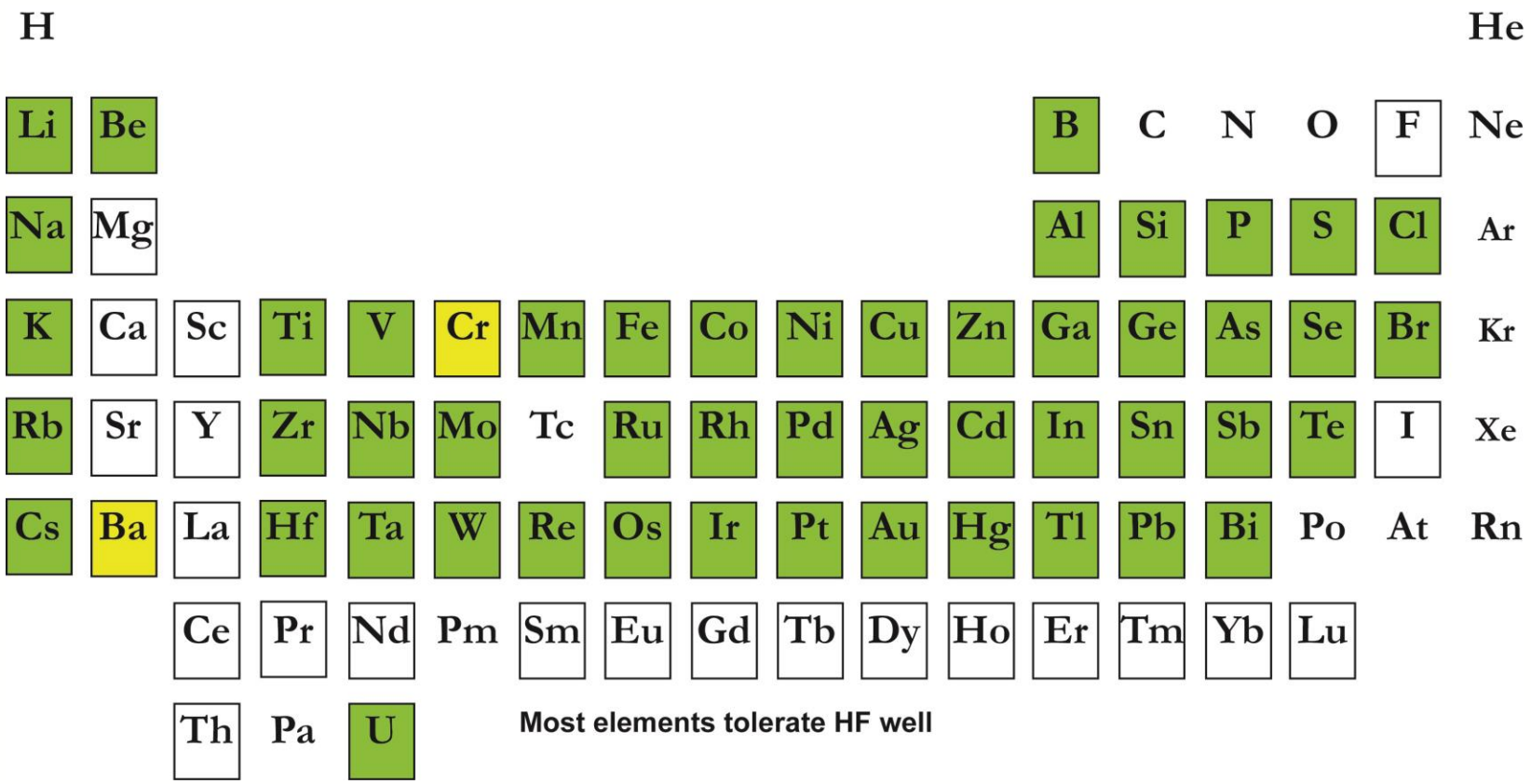
Chemistry Limits – HCl solubility



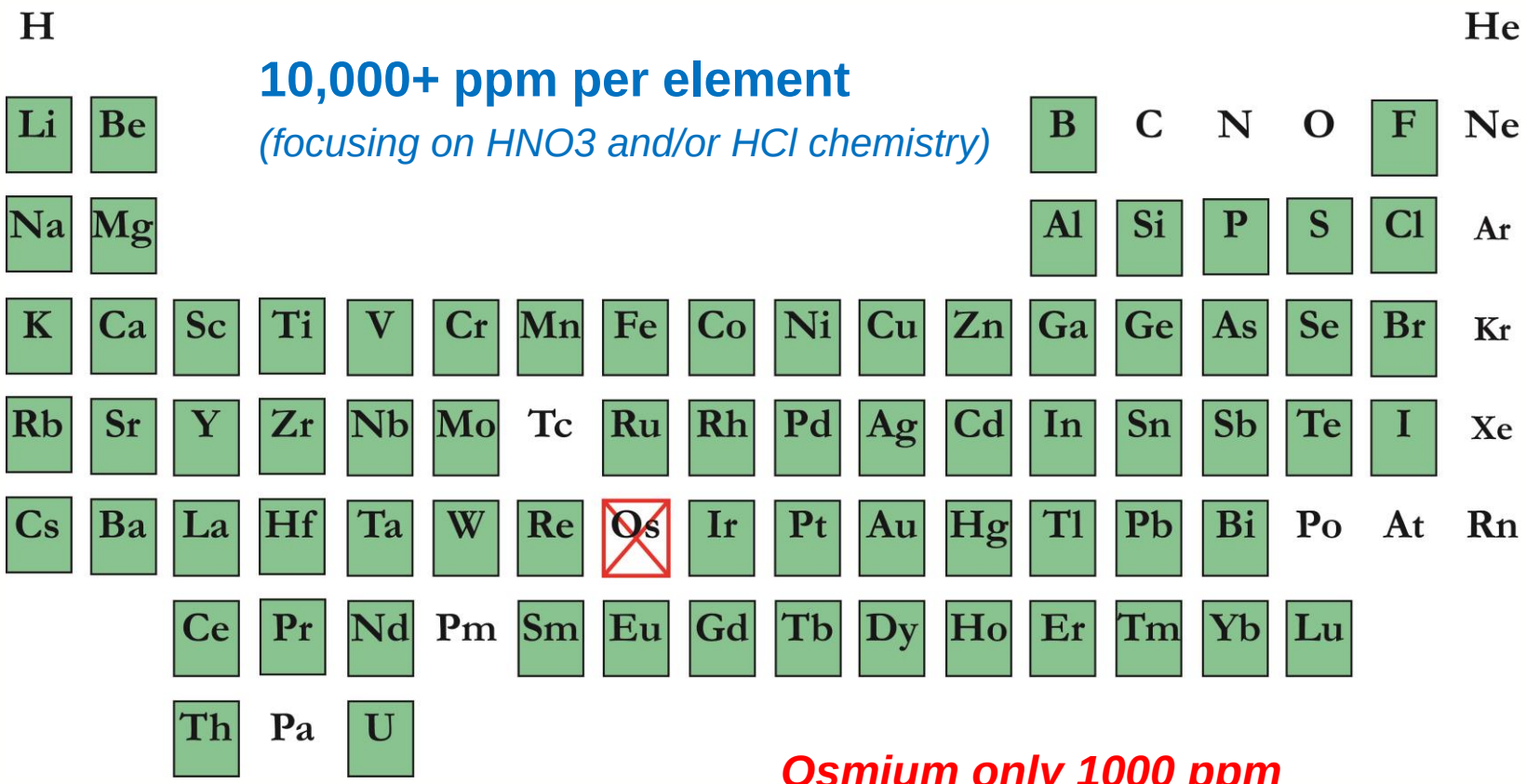
Chemistry Limits – H_2SO_4 solubility



Chemistry Limits – HF solubility



Chemistry Limits – Single Elements



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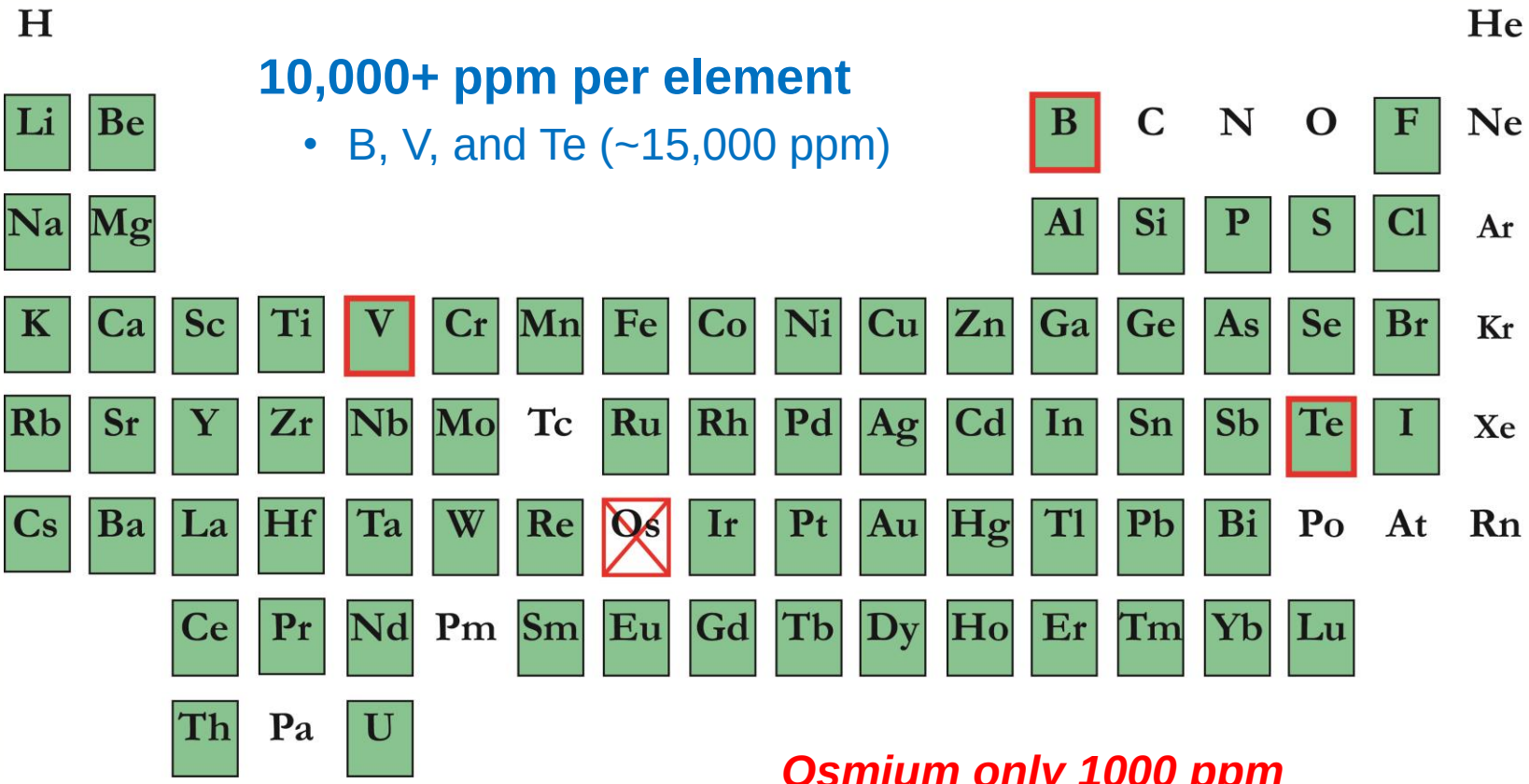
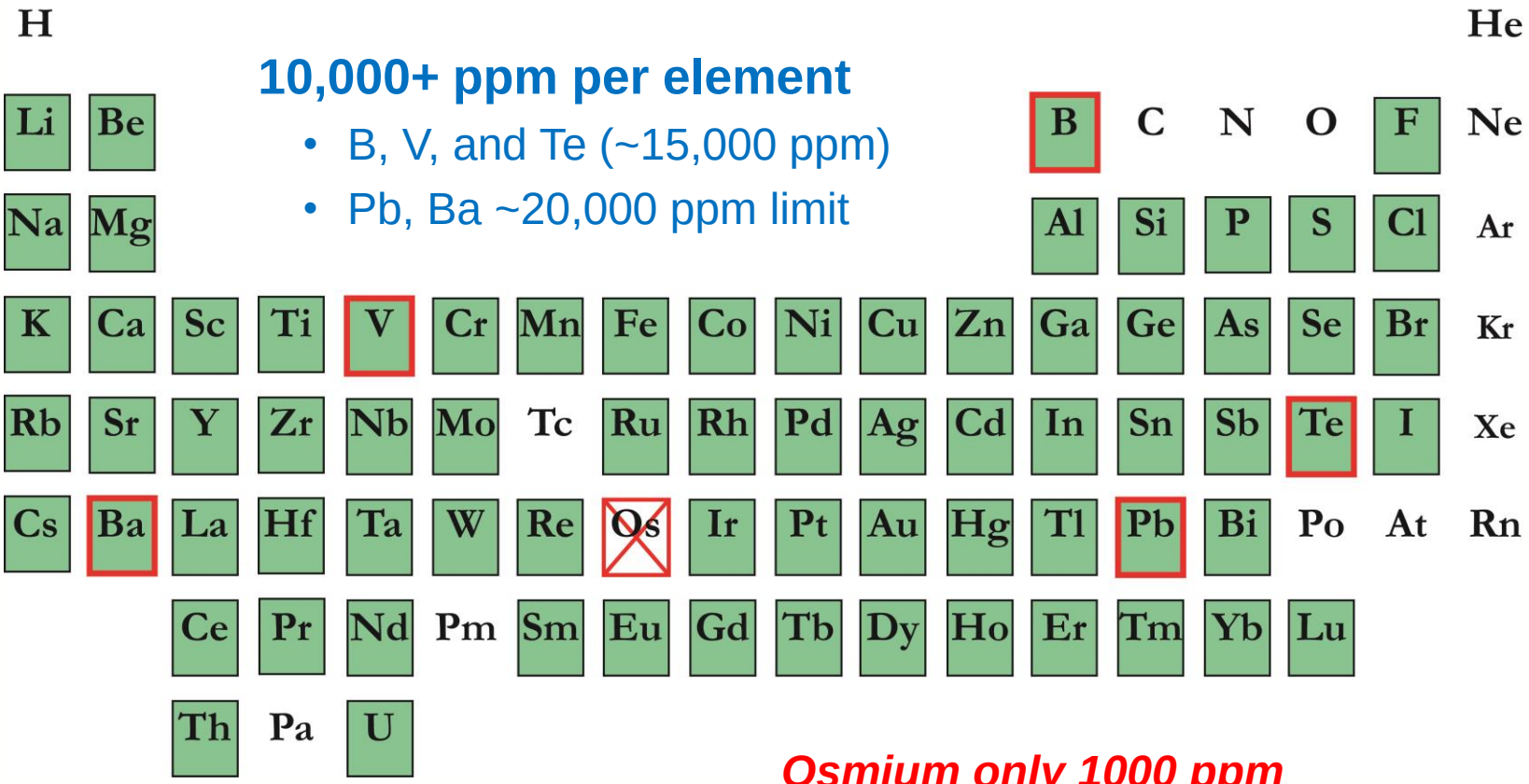


10,000+ ppm per element

- B, V, and Te (~15,000 ppm)

Osmium only 1000 ppm

Chemistry Limits – Single Elements

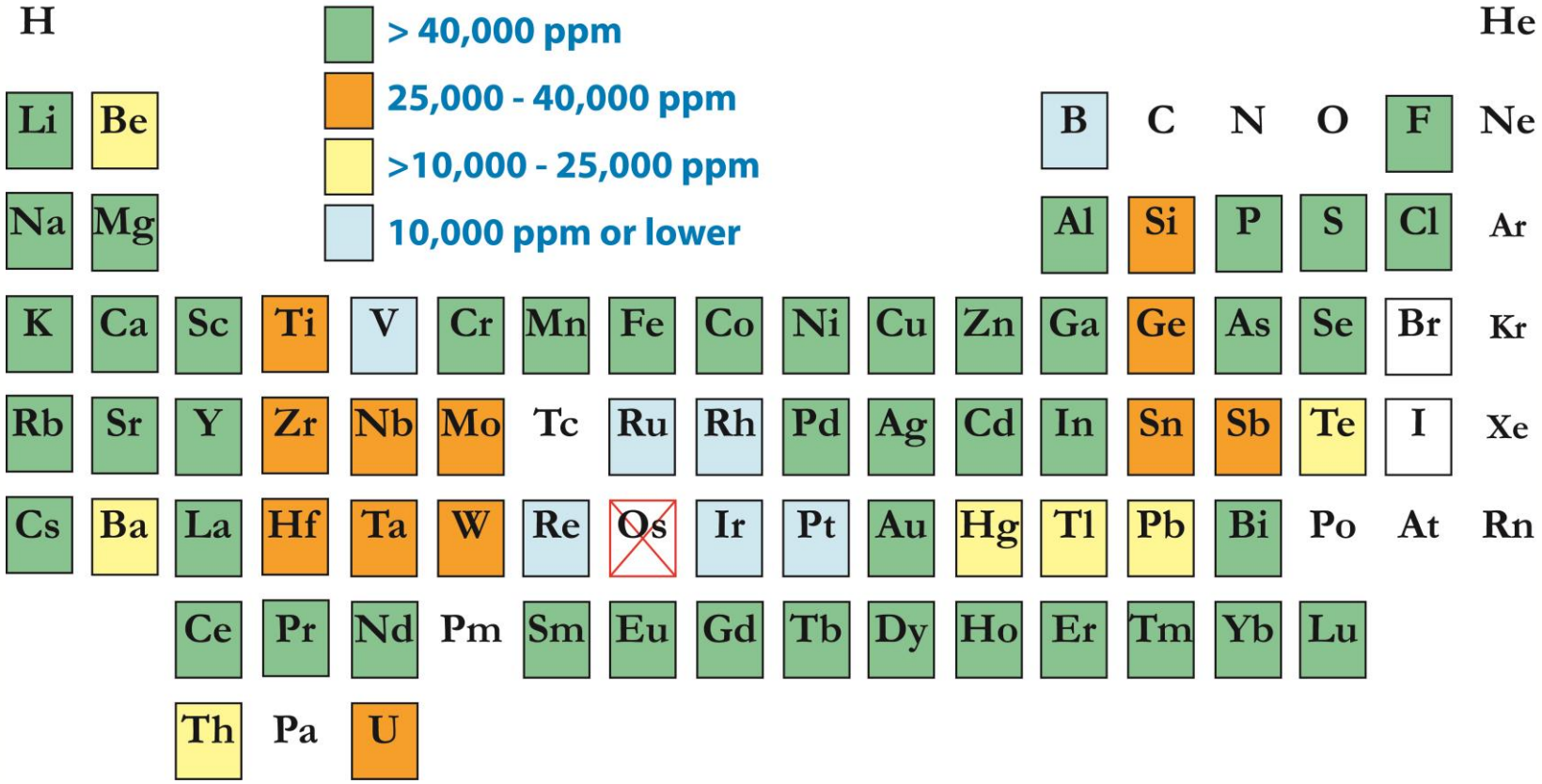


10,000+ ppm per element

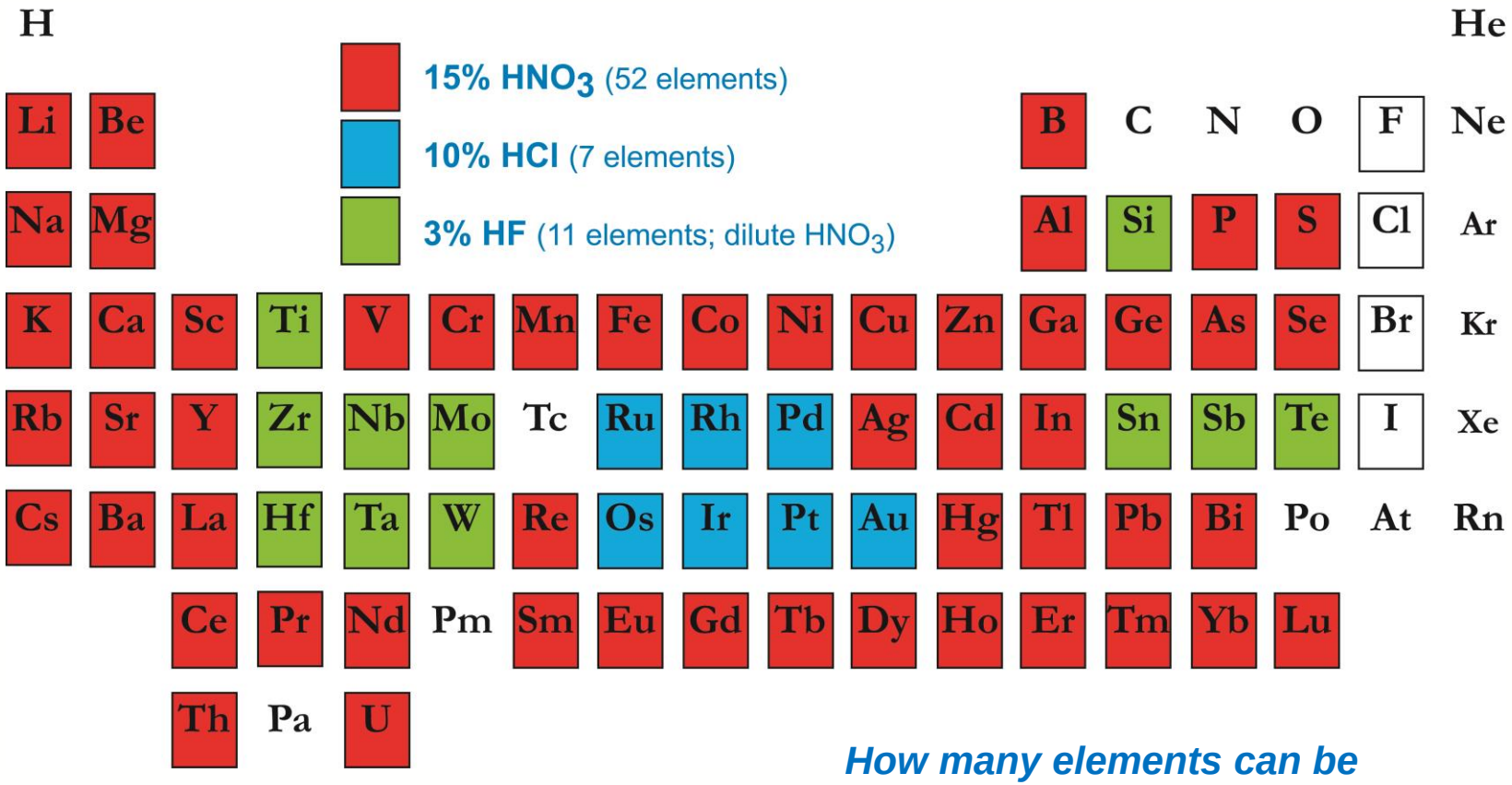
- B, V, and Te (~15,000 ppm)
- Pb, Ba ~20,000 ppm limit

Osmium only 1000 ppm

Chemistry Limits – IV Concentrates

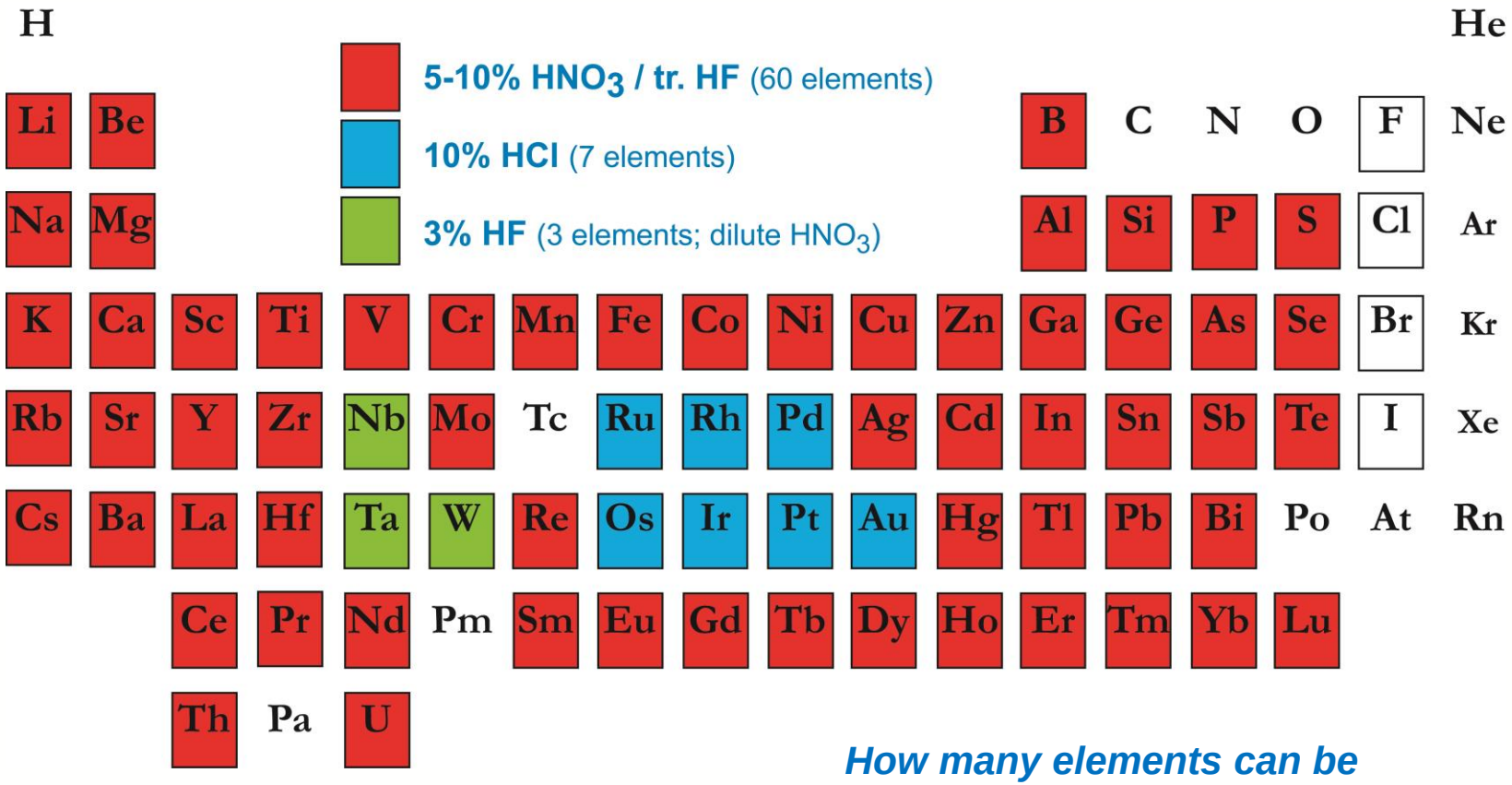


Chemistry Limits – Multi-Element Compatibility



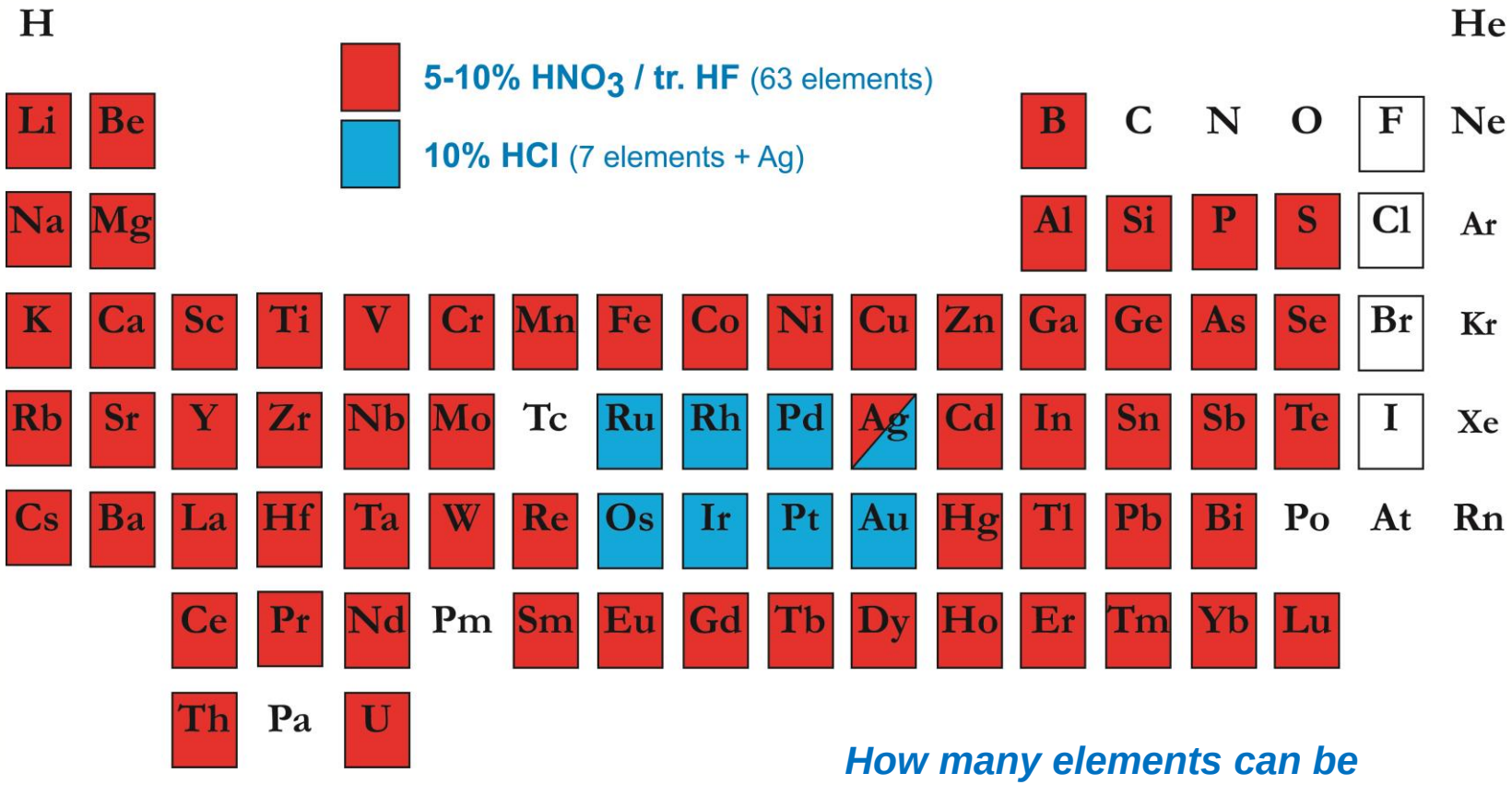
How many elements can be combined at 1000 ppm?

Chemistry Limits – Multi-Element Compatibility



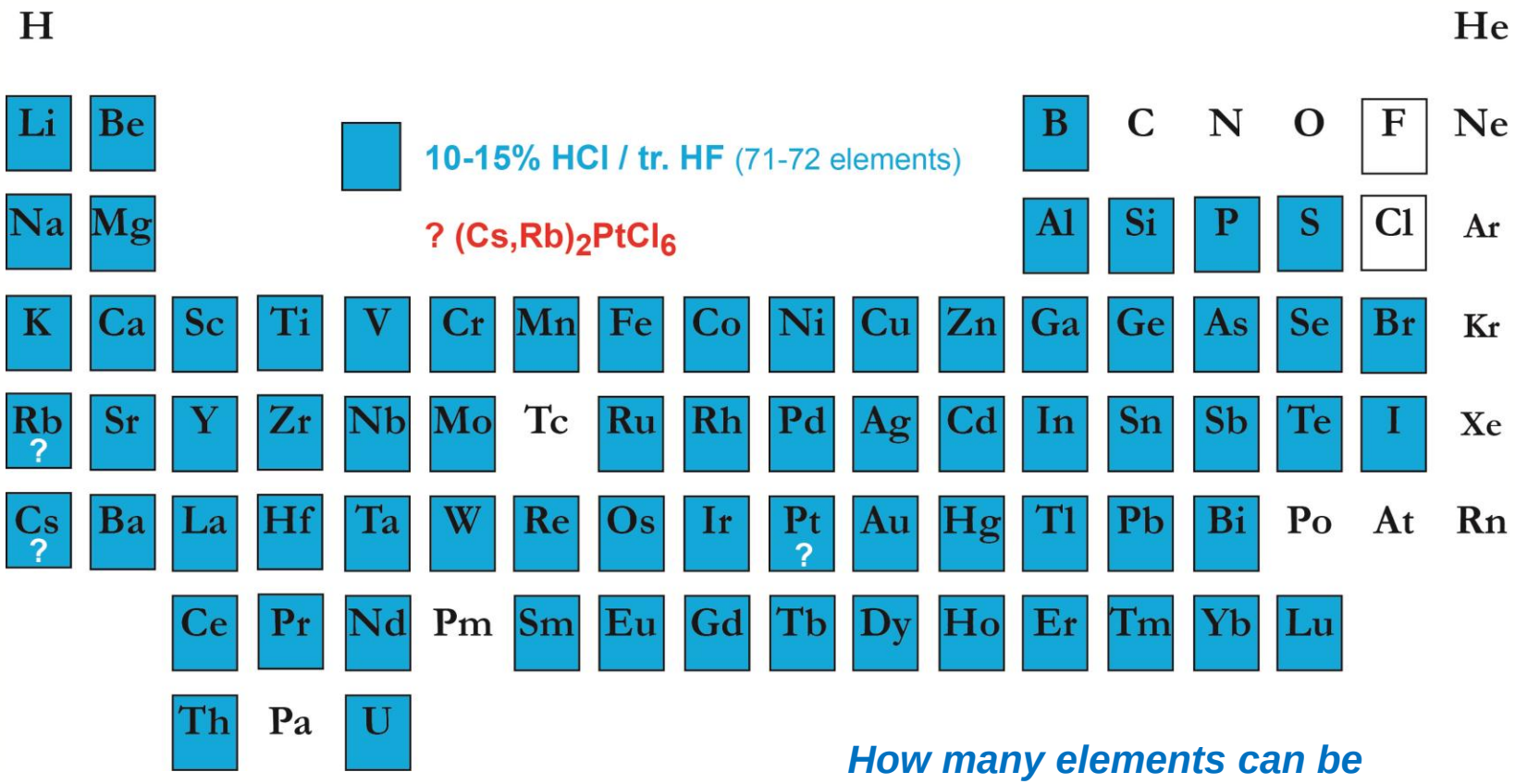
How many elements can be combined at 100 ppm?

Chemistry Limits – Multi-Element Compatibility



How many elements can be combined at 10 ppm?

Chemistry Limits – Multi-Element Compatibility



How many elements can be combined at 10 ppm?

Stability of Trace Element Standards

Parts-per-billion Study

Design

- 65 elements: 1% (v/v) HNO_3, tr. HCl, tr. HF (LDPE)
- 2, 10, 100 ppb mixtures
- Tested at 1, 3, 25, 75, 137, 300, and 375 days

Results

- **Hg** was not stable long enough to measure
- **Au** unstable at all concentrations after 3 days
- **Ag** unstable at 10 and 100 ppb after 137 days.
- **Mo**, **Sn** unstable only at the 2 ppb level at 375 days.
- All other elements stable at 2-100 ppb for 375 days

The Troublemakers…..

Hg, Sb, Au

Mercury

- Adsorbs to plastic if matrix is HNO3, so <100 ppm package in glass
- Can disproportionate around organics (e.g., tartrate)
- HCl is ideal matrix, particularly if mixing with other elements

Antimony

- Classic tartrate chemistry incompatible with Hg and Pd
- HF chemistry prevents packaging in glassware ∴ (Hg + Sb) < 100 ppm ≡ HCl

Gold

- Stable as $AuCl_3$, can only be mixed with other elements in HCl
- ★ Can be used to stabilize ppb Hg in HNO_3 + plastic containers

Factors Affecting Stability

Chemical stability

Well designed multi-element standards stable indefinitely

Physical stability

Loss of water vapor (transpiration) = systematic error

Oooops!

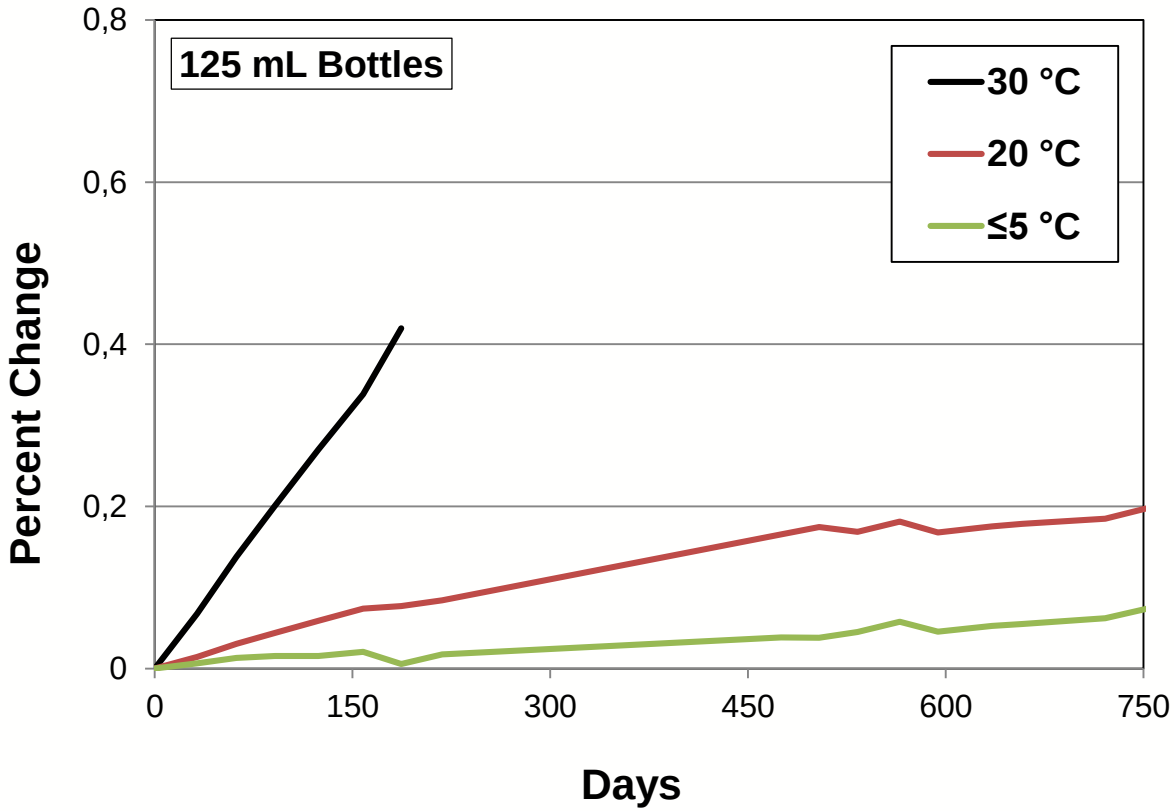
Cross contamination, mistakes

Other…..

Do you refrigerate your standards?

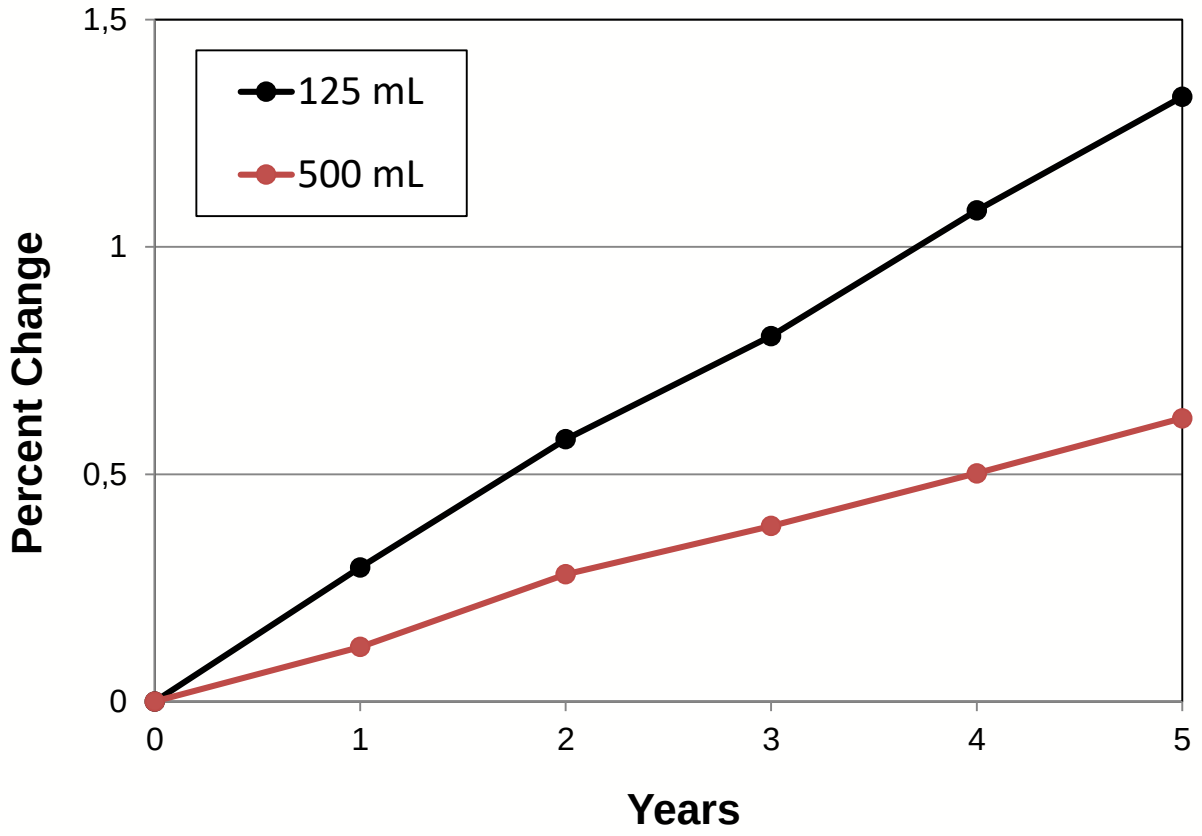
Transpiration: Effect of Temperature

Higher temperatures
= faster transpiration



Transpiration: Effect of Bottle Size

Smaller bottles transpire faster

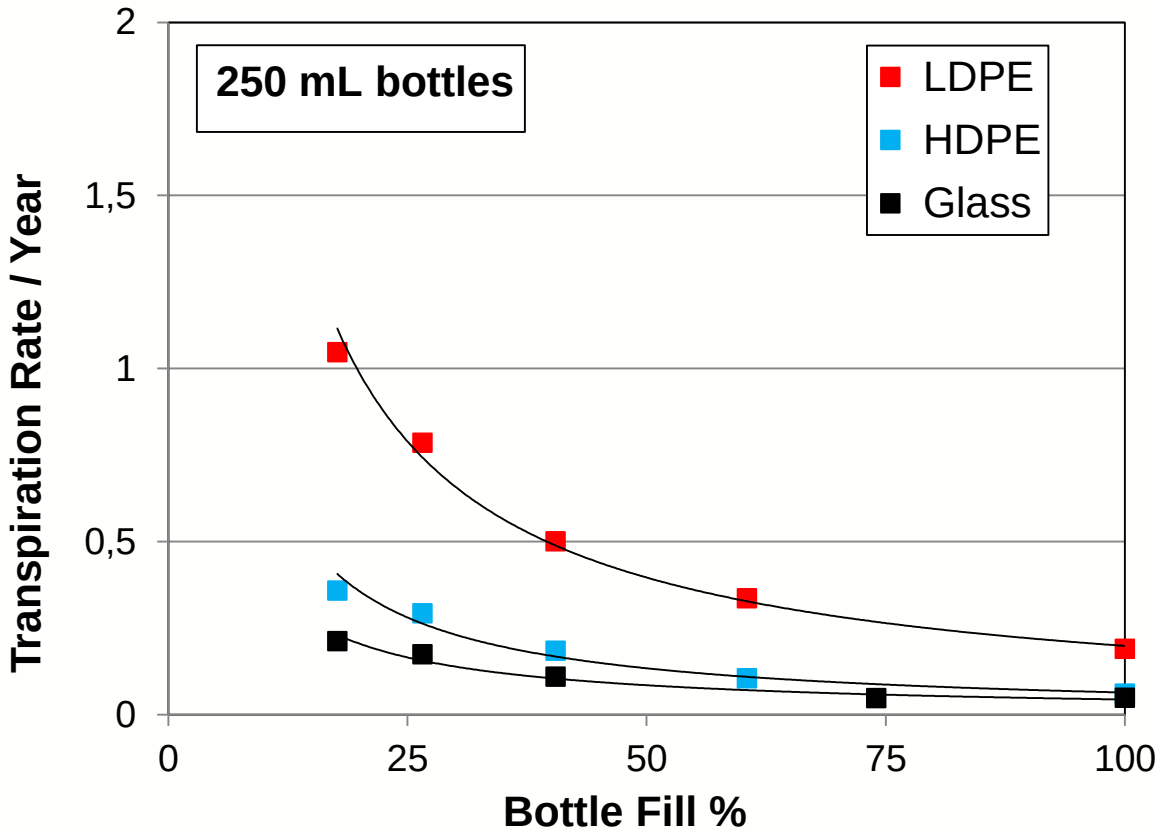


Transpiration: Container Material and Fill Level

Rate of transpiration:

LDPE > HDPE > glass

Partially full > full



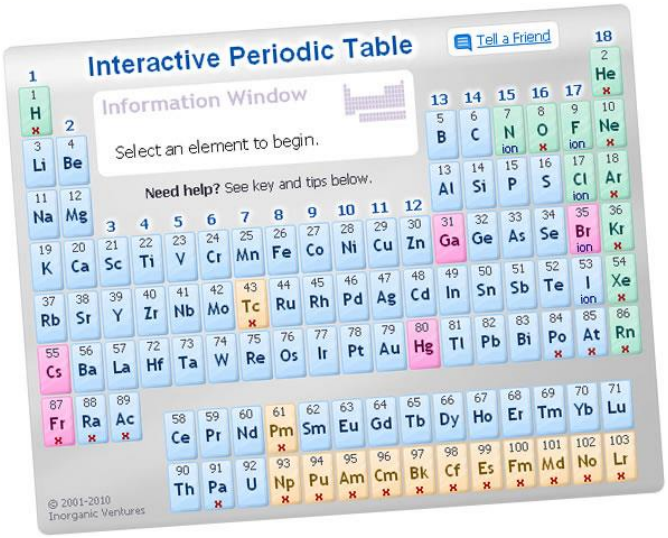
Summary – Element Compatibility and Stability

Can we mix everything together? Yes, but…

- lower is better
- acid matrix – what can you use in your lab?
- element matrix chemistry
- transpiration (systematic error) can be significant
- *if in doubt, use HCl….*

Technical Support – Available to Everyone

Online Resources at inorganicventures.com



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- **Sample Preparation Guide**
- **Trace Analysis Guide**
- **ICP Operations Guide**
- **Expert Advice**
- **And much, much more.**

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