How to Avoid Contamination When Using ICP-MS

Inorganic Product Specialist

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Definition of Contamination

• An unwanted, detectable enhancement of the background signal caused by an agent which may interfere with the measurement of an analyte of interest
Do You Have a Contamination Problem?

- Reporting negative answers for blanks
- Run blanks and compare results
  - Different sources of water
  - Different sources of acids
  - Different samples and sampling vessels
  - Different analysts
- Run blanks exposed to laboratory environment over time
  - Checks cleanliness of laboratory environment and the instrument
Sources of Contamination  (Sampling the Raw Sample)

- Sampling Equipment
- Sampling Environment
- Sample Bottles/Sample Storage
- Sample Transport
- Sampling Personnel
- Preservation Reagents
Sources of Contamination (Post-sampling)

- Analytical Containers (flasks, pipettes,..)
- Storage Containers
- Lab Reagents (including lab pure water)
- Lab Environment
- Analyst
- Instrumentation (carry-over)
Sources of Contamination - Glass Vessels and Volumetric Flasks

NEVER USE THEM!!
DO NOT USE for storage!
DO NOT USE for dilutions!
DO NOT USE for Preparing Standards
NEVER USE FOR DISSOLUTION OR DIGESTION

USE A BALANCE TO MAKE STANDARDS AND PERFORM DILUTIONS
Sources of Contamination - Glass Pipettes

NEVER USE GLASS PIPETTES!!

- Use Pipettors with disposable tips whenever possible
- Use Separate Pipette Tips for Each Solution
- Take Care Not to Contaminate the Tips During Use
- Pour Samples, Standards and Reagents Into Secondary Containers Before Pipetting
How clean are your pipettes?

2% Nitric acid run through 5ml pipets and scanned on ICPMS

<table>
<thead>
<tr>
<th>Element</th>
<th>Conc. PPB</th>
<th>Detection limit</th>
<th>Element</th>
<th>Conc. PPB</th>
<th>Detection limit</th>
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<tbody>
<tr>
<td>AG</td>
<td>2.33</td>
<td>0.0088</td>
<td>MN</td>
<td>1.72</td>
<td>0.012</td>
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<tr>
<td>AL</td>
<td>6.43</td>
<td>0.13</td>
<td>NA</td>
<td>19.1</td>
<td>0.6</td>
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<tr>
<td>BE</td>
<td>2.62</td>
<td>0.007</td>
<td>NI</td>
<td>0.96</td>
<td>0.18</td>
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<td>BI</td>
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<td>0.0006</td>
<td>PB</td>
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<td>CA</td>
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<td>2.9</td>
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<td>0.0033</td>
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<td>CO</td>
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<td>0.004</td>
<td>TH</td>
<td>0.24</td>
<td>0.0003</td>
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<tr>
<td>CR</td>
<td>0.91</td>
<td>0.28</td>
<td>TI</td>
<td>0.56</td>
<td>0.003</td>
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<tr>
<td>FE</td>
<td>1.62</td>
<td>0.75</td>
<td>TL</td>
<td>1.53</td>
<td>0.0075</td>
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<tr>
<td>MG</td>
<td>2.56</td>
<td>0.016</td>
<td>ZN</td>
<td>9</td>
<td>0.4</td>
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</table>
Acceptable Levels of Contamination

What do you want to get from your ICP-MS?

- The answer to this question determines the level of acceptable contamination
- What level of contamination reduction is necessary to achieve your analytical goals

Detection limits are limited by contamination

- Routine analyses (i.e. Environmental)
- Clean - low level analyses (i.e. Semiconductor)
Laboratory Pure Water

Water is the major component of aqueous standards and samples. Its contribution to the overall quality of the sample and standard is considerable.

**Contaminants in water**
- Dissolved, ionic solids eg. sodium chloride and calcium carbonate present as minerals in water
- Dissolved non-ionic solids or pyrogens from organic matter formed as a result of exposure to biologicals
- Particulate matter : Dirt, rust, sand, dust etc.
- Microbials : Bacteria, viruses, etc.
Laboratory Pure Water

RO SYSTEM

- Meets ASTM Type III water standards
- Carbon filtration
- Reverse osmosis

POLISHING SYSTEM

- Meets ASTM Type I or Type II water standards
- Carbon filtration
- Deionization
- Organic adsorption
- Ultrafiltration
- Ultraviolet Exposure (ASTM Type I)
# ASTM Water Specifications

<table>
<thead>
<tr>
<th>ASTM Type</th>
<th>I</th>
<th>II</th>
<th>III</th>
<th>IV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total matter (mg/L max.)</td>
<td>&lt;0.1</td>
<td>0.1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Specific Resist. (megohm, min)</td>
<td>15-18</td>
<td>1</td>
<td>&gt;1.0</td>
<td>0.2</td>
</tr>
<tr>
<td>pH</td>
<td>NA</td>
<td>NA</td>
<td>6.2-7.5</td>
<td>5-8</td>
</tr>
<tr>
<td>Min. color retention time of KMnO₄ mins</td>
<td>60</td>
<td>60</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Soluble Silica</td>
<td>ND</td>
<td>ND</td>
<td>10ug/l</td>
<td>high</td>
</tr>
<tr>
<td>Bacteria Count</td>
<td>0/ml</td>
<td>0/ml</td>
<td>10/ml</td>
<td>100/ml</td>
</tr>
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</table>
This Purifier Trace Metals Work Station is designed specifically for the demands of trace metals analysis of environmental samples included EPA Method 1631 Mercury in Water.

- Total exhaust vertical clean bench provides a Class 100 particulate-free and metal-free work environment.
- All components in the HEPA-filtered air stream are non-metallic including the work area, which is PVC and tempered safety glass.
- Applications involving corrosive chemicals such as acid digestions are well-suited to this enclosure.
- Provides personnel and product protection from background contamination.
Routine Analysis – Nebulizers and Spray Chambers

Common Materials

- Glass
- Quartz
- Ryton

Sample Introduction

- Nebulizers
- Spray Chambers
Routine Analysis – Nebulizers

- Cross-flow
- Meinhard
Routine Analysis - Meinhard Nebulizer

- Cleaner solutions (Usually <1% TDS)
- Best Precision and Detection Limits
  - 0.5% Precision
  - D.L. 40 % lower than cross-flow
- Can be run without pump (self-aspiration)
- Susceptible to clogging
Routine Analysis - Spray Chambers

- Scott - Type Double Pass Chamber
- Cyclonic Spray Chamber
- Baffled Cyclonic Spray Chamber
Routine Analysis – Ryton Scott Style Spray Chamber

- Separates the small droplets (<10 microns) from the larger droplets

- Small droplets travel through center tube, large droplet drop out of center tube and into the drain

- 2 - 4% efficient
- Durable and inert

- Cleaned with 10-20% warm not boiling HNO3

- Can run most any solvent
Routine Analysis - Cyclonic Spray Chamber

- Very efficient
  - 40% higher efficiency than a Scott-Type
- Excellent Rinse-out
- Detection limits improve by a factor of 2 over a Scott-Type
- Clean with hot mineral acids
- Precision worse than a Scott-Type (1%)

Sample Introduction

Cyclonic Spray Chamber
Cyclonic spray chamber has fast rinse out will little memory affects

Baffle reduces efficiency, but increases precision and reduces oxides

Detection limit comparable with Scott; maybe 10% lower

Allows for the running of volatile solvents
AAS grade acids and standard solutions contain impurities.

High purity acids and standard solutions must be used for ICP-MS.

- Fisher Scientific International Inc.
  http://www.fisherscientific.com/

- Cole Parmer
  http://www.coleparmer.com/

- SPEX
  http://www.spexcsp.com/crmmain/crm/toc_cl.htm

- High Purity Standard
  http://www.hps.net/toc.html
Routine Analysis – Some Suggestions to Reduce/Avoid Contamination

- Very difficult to determine 20 or more elements at ICP-MS detection limits without contamination
- Do as much preparation in plastic or Teflon as possible
  - Rinse with 1% nitric acid and keep nitric acid in them until used
  - Use once and discard or rinse with nitric acid (1%) or high purity water and store until used
  - Use Eppendorf®-type pipettes or automatic diluter
- Do dissolution in metal-free clean hood if contamination is a problem
- Use high purity acids when possible
- Best results when dilutions are done on a balance (weight/weight)
Routine Analysis – Some Suggestions to Reduce/Avoid Contamination

- Water – ASTM Type II (18 megohm) or better**
  ** ASTM Type I for Clean – Ultra low level analyses

- Acids, other reagents
  - Cleanest are NH₄OH and HNO₃
  - Dirtiest are HCl and NaOH

- Containers
  - Glassware: B, Si, Na, and half the periodic table
  - Polyethylene: Sn, Ba
  - Teflon: Fe
  - Polypropylene: best
Clean - Class 100 Work Station

- 99.99% efficient HEPA filter
- Class 100 air (ISO Class 5 conditions)
- Corrosion-resistant PVC interior, air foil and work surface
- Exterior of epoxy-coated steel and aluminum
- Designed for ducting to the outside
- Tempered safety glass sides and angled pivoting sash
- Filter condition indicator
- Fluorescent lighting

Particle-free Class 100 air (fewer than 100 particles 0.5 µm or larger per cubic foot of air)
Clean Laboratory

- Environment of class 100 (less than 100 particles of 0.5 microns per m³)
- Walls, ceilings and floors sealed and dust free
- HEPA filters mounted in the ceiling
- No exposed metal parts
- All work performed under clean hood
- Exposure of Samples and Standards to air is limited
PFA Volumetric Flasks and Pipette Tips

- PFA Volumetric Flask
  - Contamination comes from the cap and mouth.
  - The mouth of volumetric flask is too small.
  - Solution touches the cap while mixing, this is unavoidable.

A wide mouth PFA bottle is recommended.

- Pipette Tips
  - Most pipette tips have coating to allow better delivery of solution. **Can also contaminate solution!**
  - Colorless polypropylene tips can be used for sampling of standard solution, but not good for pure chemicals.
  - PFA tip is available from ESI.
  - Pipettors with a stainless steel plunger might be corroded by acid. **DON’T USE THEM!**
## Impurities in Container Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Total No. of Elements</th>
<th>Total PPM</th>
<th>Major Impurities</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polystyrene-PS</td>
<td>8</td>
<td>4</td>
<td>Na, Ti, Al</td>
</tr>
<tr>
<td>Teflon-TFE*</td>
<td>24</td>
<td>19</td>
<td>Ca, Pb, Fe, Cu</td>
</tr>
<tr>
<td>Teflon-FEP*</td>
<td>25</td>
<td>241</td>
<td>K, Ca, Mg</td>
</tr>
<tr>
<td>Polycarbonate-PC</td>
<td>10</td>
<td>85</td>
<td>Cl, Br, Al</td>
</tr>
<tr>
<td>Low Density PE-LDPE</td>
<td>18</td>
<td>23</td>
<td>Ca, Cl, K</td>
</tr>
<tr>
<td>Polypropylene-PP</td>
<td>21</td>
<td>519</td>
<td>Cl, Mg, Ca</td>
</tr>
<tr>
<td>Polymethyl Pentene-PMP</td>
<td>14</td>
<td>178</td>
<td>Ca, Mg, Zn</td>
</tr>
<tr>
<td>High Density PE-HDPE</td>
<td>22</td>
<td>654</td>
<td>Ca, Zn, Si</td>
</tr>
<tr>
<td>Borosilicate Glass</td>
<td>14</td>
<td>497</td>
<td>Si, B, Na</td>
</tr>
</tbody>
</table>

*TFE-Tetrafluoroethylene  
*FEP=FluorinatedEthylenePropylene
Cleaning Procedure

1. Soak in (1+1) HCl for one week.
2. Soak in (1+1) HNO3 for one week.
3. Heat with the chemical to be used for three hours (under the boiling point of chemical)
4. Heat with UPW for three hours (under the boiling point).

This PFA bottle was cleaned by the above procedure.
It was used for pre-concentration of HF.
It still took 14 runs to eliminate the contamination from the bottle.
Clean Acids and Standard Solutions

Two companies guaranty impurity level better than 10 ppt:

Tama Chemical
   http://www.tama-chem.co.jp/english/reag-line-up.html

Kanto Chemical

AAS grade single standard solution contain impurities.
High purity standard solutions must be used for ICP-MS.

SPEX
   http://www.spexcsp.com/crmmain/crm/toc_cl.htm

High Purity Standard
   http://www.hps.net/toc.html
• No jewelry, cosmetics or lotions
• Wear gloves, but No Powder
• Cover hair and mouth
• Protect samples and standards from dust, airborne particles and fibers
Conclusion

How do you determine how clean your lab is?

RUN A SERIES OF BLANKS!!!