Tech Note

PlasmaQuant MS Series: Lab Preparation

Preparing Your Laboratory for a PlasmaQuant MS ICP-MS Mass Spectrometer

Introduction

One of the reasons for purchasing an Inductively Coupled Plasma Mass Spectrometer (ICP-MS) is the superior sensitivity that it affords over other instrumentation, such as Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) or Graphite Furnace Atomic Absorption (GFA A). The best detection limits and lowest background levels can only be achieved if contamination associated with sample handling and the laboratory environment is minimized. Contaminants usually present in our surroundings and on our bodies include, but are not limited to, compounds of Na, Mg, Al, Fe, Ca and K, which are also common constituents of building and construction materials. Sample preparation materials can also be a source of contamination. As contamination levels increase, background count rates in ICP-MS also increase and detection limits will suffer.

Materials and reagents

The focus of this document is to discuss how to minimize environmental influences during sample handling and preparation, and to give examples of some reagents and equipment that are suitable for ICP-MS analysis. The items listed have proven themselves to be extremely clean and reliable over a number of years. This information will help to eliminate the guesswork associated with choosing products for ICP-MS analysis, particularly in the early stages of preparing your laboratory or transitioning from ICP-OES or GFA A analysis.

NOTE: Analytik Jena receives no endorsement or any other compensation from any of the listed vendors. Mention of vendors, trade names or commercial products does not constitute endorsement or recommendation for use of specific vendors or items. Supplier information is provided only as a guide for users.

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Clean environment and handling for ICP-MS

Laminar flow hood: An enclosed preparation area with a hood is very important for clean sample preparation. This will help to minimize contamination from the laboratory surroundings. It is best to have a hood with laminar flow that has both a primary air filter (changed regularly, every 2-3 months), as well as a High Efficiency Particulate Air (HEPA) filter. This minimizes the particulates that are introduced into the sample vials during preparation. If possible, reagents (such as acids) and calibration solutions should also be kept in this area, as well as pipetting equipment (pipettes and tips) and sample vessels (test tubes, measuring cups, and so on). This hood should be located in an area that is isolated from airborne particulates generated by other types of sample preparation.

It is important that no other equipment such as hot plates, microwave digestion equipment, ultrasonic baths and similar apparatus reside within the laminar flow hood, as they will contaminate the area.

Clean water: It is essential to have a water purification system that delivers 18.2 M deionized (DI) water (the purest possible). Without clean water for dilutions and sample preparation, background levels will be elevated and detection limits will be compromised.

Clean acids: It is necessary to purchase the cleanest possible grades of acid, such as “optima” or double-distilled grades, for making calibration solutions and diluting samples. Nitric acid is preferable.

Clean solutions: Purchasing high quality stock calibration solutions will ensure purity and consistency. A good quality stock solution comes with a certificate that specifies the levels of trace metals present. It is recommended that stock calibration solutions be used before the expiration date.

Clean gases: Clean gases are important for the PlasmaQuant MS ICP-MS.

The following limits apply to the argon gas supply:

- Minimum argon purity 99.996%
- Maximum oxygen content 5 ppm
- Maximum nitrogen content 20 ppm
- Maximum water content 4 ppm
- Maximum hydrogen content 1 ppm

The following limits apply to the nitrogen and oxygen used for the Nitrox option:

- Minimum nitrogen purity 99.996%
- Minimum oxygen purity 99.996%
- Maximum water content 4 ppm

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The following limits apply to the integrated Collision Reaction Cell (iCRC) gases:

- Minimum hydrogen purity 99.996%
- Minimum helium purity 99.996%

Hydrogen can be sourced either as a compressed gas stored in cylinders or alternatively, using from hydrogen generator. While less cost-effective, the hydrogen generator is considered a safer option to compressed hydrogen gas.

Clean tools: It is advisable to dedicate a set of pipettes for ICP-MS; a 10 mL, 1 mL and 100 µL set is a good combination to have. This will help to minimize contamination. Clean pipette tips are also necessary, along with small, disposable plastic (e.g., polystyrene) cups into which solutions are dispensed. In this way, pipettes are not introduced directly into the bottles and cross-contamination is avoided. The pipette tips should also be dedicated to ICP-MS and be placed in the laminar flow hood with the pipettes. Whenever possible, avoid colored pipette tips and storage container lids.

Clean hands: All sample preparation for ICP-MS must be performed using powder-free vinyl or latex gloves. The instrument sample introduction components (that is, torch, sheath gas tube, spray chamber and nebulizer) and the sampler and skimmer cones should also be handled using gloves.

Clean ceiling tiles: It is possible to buy non-porous ceiling tiles that are designed specifically for use in clean laboratory areas. Ordinary ceiling tiles will generate significant levels of contamination that can fall into solutions and onto sample preparation areas.

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Sample preparation supplies

In addition to the items listed above that will help to provide a clean environment, there are several consumable items that are needed for sample preparation for ICP-MS.

Peristaltic pump tubing: Two sizes of peristaltic pump tubing are needed; one for the nebulizer/sample uptake and the other for the spray chamber drain. The drain tubing has a larger diameter than the sample uptake tubing to ensure that no sample accumulation occurs in the spray chamber during operation. It should be noted that in some ultra-trace applications, it is preferable to use a self-aspirating nebulizer system to avoid potential contamination from pump tubing.

Spare set of sample introduction glassware: A spare spray chamber, nebulizer and sheath gas tube will allow the ICP-MS operator to clean one set of glassware while the other is in use. Having a spare handy is also helpful should any part be broken. If an inert sample introduction system is used, it is advisable to have a second system that can be cleaned while the other is in use.

Sample cups: Stock reagents should be dispensed into sample cups to avoid cross contamination of samples; 5 mL, 15 mL and 50 mL cups are very useful.

Pipette tips: As mentioned previously, it is necessary to purchase clean/colorless pipette tips that are dedicated for ICP-MS use. Trace metal free tips are available in 100 µL and 1000 µL sizes. It is also advisable to keep a small bottle of dilute high-purity nitric acid in the sample preparation area to rinse each tip prior to use.

Carboys for acids: Nalgene, for example, produces a 5 L carboy with a plastic stop-cock located at the bottom of the vessel for storing and dispensing acids. Two carboys are recommended. One should be dedicated to storing 10% nitric acid (i.e., 10% concentrated nitric acid, 90% 18.2 MΩ DI water by volume), which is used for soaking volumetric flasks. The second must be dedicated to storing ultra-pure 1% nitric acid (1% concentrated nitric acid, 99% 18.2 MΩ DI water by volume), which is used for dilutions and for preparing calibration solutions. The diluent solution must be made fresh daily to avoid leaching and contamination over time. These containers must only contain these acids throughout their service, to maintain cleanliness. Each should be soaked with their respective reagents for 1 week prior to first using them, to eliminate any contamination.

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Volumetric flasks: It is better to use plastic flasks because they are much cleaner than glass. More importantly, plastic will not leach elements over time as glass flasks do. Nalgene makes PMP (polymethylpentene) volumetric flasks. For semiconductor applications or other applications requiring ultra trace levels, PFA ("perfluoroalkoxy", that is, tetrafluoroethylene-perfluoropropylvinylether copolymer) flasks should be used. A range of volumes should be obtained; 50 mL, 100 mL, 250 mL, 500 mL, and 1000 mL flasks are suitable ranges for either PMP or PFA flasks. Either type of flask must be soaked and rinsed prior to use. Flask surfaces should be kept wet and acidified at all times, so that impurities are mobilized and can subsequently be rinsed out. These flasks are used to make up calibration solutions for all elements, with the exception of mercury. Mercury has the tendency to adhere to plastic, so mercury calibration solutions should be made in glass volumetric flasks reserved for that purpose.

Sample tubes: It is necessary to have test tubes and/or autosampler tubes on hand for sample preparation. These tubes should be rinsed and soaked in dilute nitric acid until ready to use. Avoid tubes with colored lids if possible.

Vendor information and part numbers

Laminar flow hoods:

Pure Aire Corporation, www.pureaire.com

Labconco Corporation, www.labconco.com

Bigneat Ltd, www.bigneat.com

Hydrogen Generators:

Parker Hannifin Corporation, www.parker.com

F-DGSi, www.f-dgs.com

Deionized water system:

Merck Millipore Corporation, www.merckmillipore.com

Elga LabWater, www.elgalabwater.com

Barnstead International, www.barnstead.com

Acids:

Sigma Aldrich, www.sigmaaldrich.com

GFS Chemicals, www.gfschemicals.com

VWR International, www.vwr.com

Single element stock solutions:

Sigma Aldrich, www.sigmaaldrich.com

VHG Labs, www.vhglabs.com

Spex Certiprep, www.spexcsp.com

Inorganic Ventures, www.ivstandards.com

Ricca Chemical Company, www.riccachemical.com

Stock solutions should be purchased in small volumes to reduce possible contamination of the bottle during the stock solution's lifetime. 100 mL volumes should be sufficient for most laboratories.

Certified reference materials:

It is very important to obtain a Certified Reference Material (CRM) for calibration verification.

The National Institute of Standards and Technology (NIST) has a number of different types of CRMs for conventional and laser ablation ICP-MS analysis.

www.NIST.gov

For many ICP-MS applications, certified reference waters such as NIST 1643 or NIST 1640 are useful CRMs because they contain a variety of elements in a range of concentrations. They typically contain Ca, Na and Mg in the mg/L to sub-mg/L level, with trace elements in the lower micrograms per liter range. The United States Geological Survey (USGS) is also a source of Laser Ablation reference materials for many applications.

www.usgs.gov

Other suppliers of reference materials include:

LGC Standards, www.lgcstandards.com

Institute for Reference Materials and Measurements, www.ec.europa.eu/jrc/institutes/irmm

Pipettes and pipette supplies:

Eppendorf and Rainin are excellent sources for digital pipettes.

www.eppendorf.com

www.mt.com

Digital pipettes are highly recommended for ICP-MS because of their accuracy and low maintenance. 10 mL, 1000 µL, and 100 µL are suggested.

The tips for the pipettes can be purchased from Eppendorf, Sigma Aldrich, Rainin or VWR International.

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Peristaltic pump tubing:

Spare peristaltic pump tubing can be purchased through Analytik Jena or your local Analytik Jena distributor.

- Peristaltic tubing grey/grey 0.051 in ID,
- Peristaltic tubing white/white 0.04 in ID
- Peristaltic tubing blue/blue 0.065 in ID standard drain,
- Peristaltic tubing, black/black 0.03 in ID

Carboys for dispensing acids:

Nalgene makes HDPE (high density polyethylene) carboys with a spigot (plastic stop-cock). These carboys are excellent for dispensing acids into volumetric flasks. It is best to purchase two of these, and dedicate one for the 10 % nitric acid for soaking flasks and sample tubes, and one for the 1% nitric acid for sample/calibration dilutions.

Volumetric flasks:

Various suppliers also carry PMP or PFA volumetric flasks in a range of volumes.

Gloves:

Always use talc-free gloves. The powder inside some gloves will contaminate anything that is touched by them.

Spare sample introduction glassware:

Spare sample introduction glassware can be purchased through Analytik Jena or your local Analytik Jena distributor.

Sample cups:

Small volumes of calibration solutions or samples are poured into cups so that they can be dispensed elsewhere, to help avoid contamination. It is best to purchase a variety of sizes depending on the sample/standard volumes needed. 5 mL, 15 mL, and 50 mL volumes are useful.

Test tubes:

Colorless polypropylene tubes for test tube racks are recommended. It is advisable to avoid colored caps and colored plastics in general, as they can generate significant contamination.

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Conclusion

To obtain best performance from the PlasmaQuant MS ICP-MS, it is important to follow the guidelines in this document. The highest signal and lowest background counts, and therefore the best detection limits, can only be achieved if contamination is minimized.

Further information

For further updates, applications and other literature, please visit the Analytik Jena website at www.analytik-jena.com.

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