NANOCOMPOSIX'S GUIDE TO ICP-MS MEASUREMENT AND ANALYSIS

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Note to the Reader: *We at nanoComposix have published this document for public use in order to educate and encourage best practices within the nanomaterials community. The content is based on our experience with the topics addressed herein, and is accurate to the best of our knowledge. We eagerly welcome any feedback the reader may have so that we can improve the content in future versions.*

Please contact us at info@nanocomposix.com or 858-565-4227 with any questions or suggestions.

Introduction

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is a type of mass spectrometry that is used to detect metals in a sample at concentrations as low as 1 part per trillion. The ICP-MS can be utilized as a quantitative tool to determine the concentration of a specific analyte, or as a qualitative tool to determine the metal speciation in a sample.

NanoComposix has a Thermo Fisher Xseries2 ICP-MS instrument that consists of two primary components: The inductively coupled plasma (ICP) and the mass analyzer, or mass spectrometer (MS). Liquid samples are introduced into the ICP using a nebulizer. The ICP is an electrically conductive Argon gas at very high temperatures (~10,000K) which evaporates the solvent and atomizes and ionizes the sample before it is analyzed in the MS. The MS is a quadrupole mass spectrometer
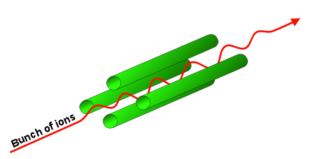

Figure 1: Quadrupole mass spectrometer schematic.

that consists of two pairs of electrically conductive rods that are connected in series (Figure 1). The charged samples travel through the quadrupole at a rate that is determined by the mass to charge (m/z) ratio of the ionized atoms. Only the selected m/z ratio will reach the ion detector with all other ions colliding with the quadrupole rods allowing for both sensitive and selective detection.

Careful sample preparation is very important for ICP-MS analysis. Typically, the samples are digested in an acid before analysis. Due to the high sensitivity of the instrument, it is often necessary to perform multiple dilutions of the sample in order to reach a concentration that is optimal for analysis.

Data from the ICP-MS is straightforward and easy to interpret as the primary output is a mass concentration.   ICP-MS analysis is one of the cornerstone analytical instruments at nanoComposix. However, as with any analytical technique, it is important to understand the limitation of the instrument and to be aware of situations where alternative sample preparations can increase the quality of your ICP-MS data.

Sample Preparation

Our ICP-MS nanoparticle analysis service is typically used by our customers looking to:

1) Accurately determine the mass concentration of nanoparticles in solution

2) Measure the dissolution rate of ions from the surface of nanoparticles
3) Measure the concentration of nanoparticles within into a biological or environmental sample

The main difference is that in the first and second case, the nanoparticles are at a relatively high concentration and there is not a lot of background mass associated with other elements. With tissue or environmental samples, there is a much higher degree of degree of solid loading and the acid digestion of the sample is more challenging. The following is a list of general criteria that defines how difficult it is to make an accurate ICP-MS measurement for a given sample:

Ideal samples for ICP-MS analysis are:

- Dispersed in water or in a sample matrix that is well known and does induce matrix effects.
- Nanoparticles made from the elements Al, Fe, Pt, Ag, Au, Cu

More challenging samples:

- Nanoparticles mixed with or embedded in a high mass loading of non-analyte material (e.g. other nanoparticles, surfactants, tissue, etc.)
- Nanoparticles that are coated with a shell (e.g. silica)
- Nanoparticles made from the elements As, Cr, Sn, Hg, S

Sample Preparation Advice

To maximize the accuracy and reproducibility of ICP-MS samples submitted to nanoComposix, follow these guidelines:

1. Sample Isolation:

   Higher ratios of analyte to the other solids present in your sample will improve the accuracy and sensitivity of the ICP-MS data. If possible, all extraneous material should be removed from your sample. For example, careful washing of tissue samples and filtration of particulate contaminants from a nanoparticle solution can improve data quality.

2. Acid Digestion:

   Acid digestion is the cornerstone sample preparation technique that is necessary for all samples containing solid analytes, i.e. colloidal dispersions of gold, silver or other metal analytes. As previously mentioned, samples need to be in ionic form prior to entering the mass analyzer in order to be detected. A strong, often oxidizing acid can accomplish this; common examples are concentrated Nitric Acid, Aqua Regia or HCl, depending on

the metal analyte. It is strongly recommended that this step be performed at nanoComposix to ensure reproducibility and accuracy or your data. Samples that are already in ionic form do not need acid digestion and can be immediately diluted for ICP-MS analysis.

3. Appropriate Dilution

   The ICP-MS concentration is determined by running a calibration curve with a known concentration of the target analyte. In order to obtain an accurate reading, the concentration of the sample analyte must be diluted to be within this calibration curve. For some samples this requires a dilution by a factor of 10,000 or more.

4. Ion Dissolution/Release Study:

   Often times a customer may wish to know the time dependence of release of ions into solution from metal colloidal particles. This knowledge is critical for nanotoxicological researchers or anyone interested in the ion release properties of their colloidal dispersions. Since the ion dissolution time course can be affected by atmospheric exposure, matrix effects, heat and light, it is critical that all potential variables are tightly controlled. In an ion release study, the particles will be incubated with a solution and then the nanoparticles will be separated from the matrix. Typically, the separation is accomplished using a very fast spin in an ultracentrifuge or a centrifuge based filter with a low molecular weight cut-off. Providing the isolated matrix in a sealed container kept away from the light is recommended.

We encourage you to work with one of our analytical specialists to determine what sample preparation techniques can be used to maximize your data quality. Ideally, the necessary steps can be taken before we receive the sample but, if requested, we can provide any required processing when the samples arrive.

Interpreting Your Results

Data interpretation from ICP-MS analysis is straight-forward. Your result will be reported as concentration in mg/L, which can easily be converted to the mass concentration metric of your choice. The raw data from the instrument will be reported as a ppb concentration. Below is an example of raw data of a diluted analyte sample from our ICP-MS software:

KJW1380 7/10/2012 5:45:45 PM
User Pre-dilution: 1.000

Run	Time	107Ag ppb	115In ppb
1	17:45:52	17.220	0.000
2	17:45:58	17.400	0.000
3	17:46:04	16.880	0.000
x		17.170	0.000
σ		0.264	0.000
%RSD		1.538	0.000

KJW1382 7/10/2012 5:47:47 PM
User Pre-dilution: 1.000

Run	Time	107Ag ppb	115In ppb
1	17:47:53	65.780	0.000
2	17:48:00	66.210	0.000
3	17:48:06	66.640	0.000
x		66.210	0.000
σ		0.429	0.000
%RSD		0.648	0.000

KJW1382 repeat 7/10/2012 5:49:24 PI
User Pre-dilution: 1.000

Run	Time	107Ag ppb	115In ppb
1	17:49:30	67.720	0.000
2	17:49:37	67.140	0.000
3	17:49:43	67.300	0.000
x		67.390	0.000
σ		0.297	0.000
%RSD		0.441	0.000

The ppb data from the ICP-MS run is entered into an excel spreadsheet containing the dilution information for your sample. Depending on the approximate initial concentration, your sample may have 1 or 2 dilutions performed. The resulting concentration (mg/L) will be reported as calculated in this spreadsheet. Here is an example of a dilution spreadsheet with results for the three samples listed above:

	Dilution 1		Dilution 2			
Sample Name	Mass Product (g)	Total Mass (g)	Mass from Dil 1 (g)	Total Mass (g)	ICP Measured Conc. (ppb)	Dilution Corrected Conc. (mg/L)
KJW1380	0.10011	40.0379	0.10333	41.1113	17.17	2732.1
KJW1382	0.099	40.0031	0.20059	10.09292	66.21	1346.1
KJW1382 Duplicate	0.09933	40.1563	0.20038	10.00146	67.39	1359.8

Improving Data Quality

After examining your ICP-MS data, a nanoComposix analytical specialist will make recommendation on further processing that could be done to improve the quality of your data. Additional samples can be sent in for analysis or sample optimization of the already provided sample can be conducted at nanoComposix.