

Sample Form for Solution-based HR-ICP-MS

Element XR

Radiogenic Isotope Geochemistry Lab

Sample type (e.g., carbonate, silicate, water, soil, etc.) and estimated number:

Sample digestion acid:

Most common used acids used are 1-2% HNO₃, HCl, HF,

Elements to be analyzed:

Amount (ml) of final sample solution:

Sample concentration range (ppm, ppb, ppt):

***Total dissolved solids should be kept below 2000ppm.**

Calibration Method (isotope dilution or standard working curve method):

Concentrations of working standards, internal standards, and/or isotopic spikes:

Protocol for solution-based HR-ICP-MS for internal users

- Hourly rate includes time for the analysis of standard, blanks, spikes, etc. It is necessary that concentrations of the sample solutions match those of the standards. All solutions entering mass spectrometer need to be in 1-2 % HNO_3 (prepared from Milli-Q water (18 Ω) and 14N distilled HNO_3 .) An HNO_3 matrix is preferred due to its oxidizing ability and the relative freedom from chemical and spectral interferences as compared to acids containing Cl, S, F, or P.
- The preparation cost includes acid to dissolve samples and to prepare standards, centrifuge tubes and pipette tips. IMPORTANT: It does not include Teflon bottles to prepare standards, and the standards and spikes themselves. These are the responsibility of the user.
- Training will be provided to users by the lab manager according to their needs and anticipated period of time on the ICP. The lab manager will help set up initial runs, but as the user become fully acquainted with proper machine protocol, he/she will be responsible for the analysis of his/her samples. The lab manager will assist when needed.
- Samples should be free of particulates that could clog the tubing and nebulizer (<2000ppm TDS).
- Before scheduling time on the ICP on the lab calendar (<http://my.calendars.net/radiogeniclab>) please fill out the necessary form information and discuss it with the lab manager (email: Iromero@tamu.edu)

Below is a summary of the types of calibration methods used in solution ICP-MS.

Methods of Calibration

The accuracy and precision of ICP-MS measurements is highly dependent on the calibration method. There are several ways to analyze trace elements that allows detection from sub-ppt (parts per trillion) to ppm (parts per million) levels. The selection of calibration method is highly dependent on the sample matrix because it influences the nebulization efficiency, which is proportional to the signal intensity. Importantly, these calibration methods assume that the instrument response is described by the equation of a straight line. For the ICP-MS analyst, the most common matrix component that alters the physical characteristics of a solution (viscosity, density and surface tension) is the acid content. The following is a brief description of the ICP-MS calibration methods

External standardization

This form of quantitative analysis is used when there is very little difference between the sample and standard matrix components. The instrument is calibrated by measuring the intensities of the element of interest and comparing them with a known calibration standards which will likely cover the concentration range of interest.

Standard addition

This method of calibration is used when it is difficult to match the matrix of the standard with the samples. It provides an effective way to minimize sample-specific matrix effects through the use of sample solutions that have been "spiked" with a known concentration of analytes. The difference in analytical response between the spiked and unspiked samples is due to the amount of analyte in the spike. This provides a calibration point to determine the analyte concentration in the original sample.

Isotope dilution

This method is free of matrix effects and provides measurements that are extremely accurate because the concentrations of the isotopes are being measured in the same solution as the unknown sample. However, this method does not apply to monoisotopic elements. Another limitation is that it requires certified enriched isotopic standards, which can be very expensive. This technique involves the addition of a known amount of an enriched isotope of the element of interest to the sample. This addition is made prior to sample preparation during which the spiked addition of the enhanced isotope is 'equilibrated' with the sample. By measuring the isotope ratio of the sample and sample + spike isotope addition and knowing the isotopic ratio of the enhanced addition, the sample concentration can be calculated. The entire measurement is based upon ratio measurements of one isotope of the element to another

Internal standardization

Although modern ICP-MS instruments generally provide relatively stable signals for extended periods of time, instrument drift (i.e. temporal variations, most commonly a decrease, of signal intensity with time) does occur and has to be corrected for in order to ensure good quality quantitative analysis. This drift-correction may be achieved by internal standardisation, i.e. the normalisation of all data to a non-analyte isotope present in the same or known concentrations in all samples and standards. Ideally, internal standards should be uninterfered, mono-isotopic species. Isotopes commonly used as internal standards in solution ICP-MS include ^9Be , ^{45}Sc , ^{89}Y , ^{103}Rh , ^{115}In and ^{209}Bi .