Sample Preparation

Purpose and Background

For solution ICP-MS, analytes must be completely dissolved for accurate analysis of solutions by ICP-OES and ICP-MS. The goal of digestion is to dissolve the analytes and to decompose solids while avoiding loss or contamination of the sample. For example, digestion with nitric acid oxidizes organic matter to CO_2 and NO while forming soluble nitrates with most elements which can then be analyzed. Undissolved analytes will not be accurately measured if the samples are not digested.

The methods provided in the next sections are commonly used digestion methods. However, we always encourage you to read the literature in your field and determine the best digestion method for your research questions.

Acid-Leaching Plastic Sample Tubes

Sample tubes are filled with 2% nitric acid and heated for 24 h at 60°C. The tubes are rinsed 3 times with DI water and stored with DI water. Before use, the water is discarded and sample tubes are dried.

Water

Samples should be collected in acid-cleaned metal-free containers. Keep in mind that the containers may leach analytes into your sample. Choose accordingly. Samples with high salinity (seawater or biological buffers) cannot be analyzed undiluted because of the high total dissolved solids. Please let us know if your sample contains high salinity. Process a sample of DI water following the same procedure as the samples and submit as a method blank.

For the analysis of dissolved analytes, the sample should be filtered through a 0.45 µm membrane filter. Acidify the filtrate with trace-metal grade nitric acid to pH<2 (but no greater than 10% acid by volume).. For the analysis of total recoverable elements in aqueous samples, do not filter. Acidify with trace-metal grade nitric acid to pH <2 (but no greater than 10% acid by volume).

Cell Pellets

Sample tubes are pre-weighed. Washed cells in sample tubes are digested with 0.1 mL of concentrated, trace-metal grade nitric acid. The samples are heated for 2 hours at 90°C. The tubes are allowed to cool and 0.05 mL of trace-metal grade hydrogen peroxide (30%) is added. The samples are heated for an additional hour at 90°C. The sample is diluted to a final acid concentration of 2-5%. Process 2 samples of DI water (with same volume as cell pellet) following the same procedure as the samples and submit as a method blank.

Nanoparticle Biological Samples

For most samples, nitric acid digestion is sufficient. For platinum group metals (e.g. gold and platinum), digest sample with aqua regia.

Weigh sample (0.2 – 0.5 g) into a pre-cleaned sample tube. Add 0.5 mL of concentrated trace-metal grade nitric acid (or aqua regia). Heat the sample at 90°C for 2 hours. If particulates remain, add 0.25 mL trace-metal grade hydrogen peroxide and heat for an additional hour. Repeat until no precipitate remains. Dilute with deionized water to final acid strength of 2-5%. Process 2 samples of DI water (with same volume as sample) following the same procedure as the samples and submit as a method blank.

Trace elements in plant material

Weigh 0.2 g of sample into a pre-cleaned 50 mL plastic tube. Add 1 mL of trace-metal grade nitric acid and **loosely** cap (to prevent the tube from exploding). Heat the sample at 90°C for 2 hours. Remove the sample from heat and allow the sample to cool. Add 0.5 mL of hydrogen peroxide and loosely cap. Heat the sample for an additional hour. If particulates remain, add 0.5 mL of hydrogen peroxide and heat for an additional hour; repeat this step until no particulates are visible. Dilute to a final acid concentration of 2-5%. Process 2 blank samples following the same procedure as the samples and submit as a method blank. Process 2 certified reference materials (of similar composition to your sample) following the same procedure as the samples and submit.

Trace elements in soils and sediment

NOTE: We do not perform total digestion or analyze total digestates of these matrices (i.e. we do not digest with HF and do not accept samples that have been digested with hydrofluoric acid).

Sample should be dried and homogenized. Weigh 0.25 g of sample into a pre-cleaned 50 mL plastic tube. Add 5 mL of nitric acid. **Loosely** cap the tube to allow gasses to escape without exploding the tube. Let the sample react at room temperature for a minimum of 1 hour. Heat samples at 90°C for 30 minutes. Allow the sample to cool, add an additional 5 mL of nitric acid and heat for 30 minutes; repeat until no brown fumes are emitted from the sample. Heat the sample (still loosely capped) until all but 5 mL have evaporated. Add 2 mL of DI water and 3 mL of trace-metal grade hydrogen peroxide. Heat until effervescence subsides. With the tube still capped, heat the sample until 5 mL remains. Dilute to a final acid concentration of 2-5%. Process 2 blank samples following the same procedure as the samples and submit as a method blank. Process

2 certified reference materials (of similar composition to your sample) following the same procedure as the samples and submit.