ICP Prep suggestion
CEST updated 08/2018

ICP analysis requires a certain set of specific conditions. Ensuring that your samples meet these conditions minimizes any potential error or problems during analysis. Typically, samples are brought for measurement in 15 ml sample/centrifuge tubes (BD falcon, Fisher, or VWR brand centrifuge tubes work well). In addition, you will want to have standards for any element of interest in a range from just below your expected lowest sample, to above your highest expected sample. I would suggest 5 points for your calibration curve(s) plus a blank (just the solvent). Additionally, make your standards in a combined solution using the same acidic solvent you will use for the samples. If you do not have standard stocks, I have some that may get you started.

As for sample and standard prep. You will want everything in a solution of 2-5% HNO_3* (by volume, all solvent concentration listed here are percent by volume). You may want to just take a 1 or 2 ml aliquot of your unknown sample and dilute it with the acid solution. Regardless of how much material you use to prepare your samples, you will need to filter the samples to remove any undissolved particulates.

The range of concentration measurable is dependent on the element you are looking at. We have had the best experiences with concentrations that approach zero (50-100 ppb) to 10 ppm. This does vary from analyst to analyst based on the conditions of their samples.

A very potent limitation is the **total dissolved solid** (TDS) content of the sample. With the current system configuration, we need to ensure that all samples (standards, etc.) have no more than 15% (**12.5% when using the Prep3 system**) of their mass from dissolved solids in solution. This is not just the analytes you are interested in, but all of the dissolved material in solution. Excitation in the plasma is an indiscriminate process, and all constituents of your solution will become excited then return to a ground state. Keeping the TDS below our operating limit ensures a reliable measurement of your sample.

A preferred way to create the standards is to make a "mother solution" of around 100 ppm of all target elements in the appropriate concentration of HNO_3. This can be diluted with your acid solvent to appropriate concentrations to construct a calibration curve. The Syngistix software will do a bulk of the work for you, but you may export the raw data and construct your own calibration curve if you wish.

Any materials that are to be analyzed must be clear, contain no particulates, and possess a low pH. Samples may possess color, but they shouldn't be cloudy and you should be able to see through them. Sample volumes should

be a minimum of 7ml (in 15 ml tubes), with 10 ml the optimal amount. For samples in 50 ml tubes a minimum volume of 15 ml is needed. These minimum volumes are so that the sample probe remains below the liquid line. Your sample consumption will only be 1.8 ml/min on the Optima 8000 and 0.37 ml/min on the Prep3 Optima 8000. In both cases your samples will run for 2.6-3.5 minutes depending on the total elements you are analyzing.

Lastly, certain analyses will require bringing an acid rinse for your samples. If this is required, you will bring this along with your other material. 1 L should last you for about 10-12 hours of analysis time. The composition and concentration of the sample rinse will vary. If you are running higher TDS sample 12% and higher, 2-5% HNO_3 (whatever matches your sample solvent); Li, As, Cs a solution of 1-2% HNO_3 is recommended; Hg, Au, Ag, Hf 2-5% Aqua regia.

In no circumstances is HF allowed in the Center. Only HNO_3 and Aqua regia are appropriate solvents for these samples.

*Acids are a vital part of the process. Please make sure you are using ACS or better grades of acid. The only allowable acids for ICP analysis are Nitric Acid (HNO3) and aqua regia (the 1:3 molar ratio solution of Nitric Acid to hydrochloric acid). Sulfuric Acid and Phosphoric acid cannot be used due to interference and contamination issues, as well as a potential to damage the seals on the Injector Assembly. Hydrofluoric acid cannot be used in the Center. It is too volatile and dangerous for a general purpose facility. It will also destroy the optics and torch assembly of the ICP unit. If HF is necessary in your digestion process, you will need to distil or otherwise remove it from the solution.