ICP-OES Starter Kit

The purpose of this document is to provide a good starting point for ICP-OES by giving a list of recommended supplies, reagents, and standards. The point is not just to provide information but to spark some questions / reminders whether you are new or experienced with ICP-OES. If you are uncertain about what to purchase to prepare for your training visit please reach out prior to the training so we can discuss this and make sure you have the right stuff.

Parts

Internal Standards

The use of internal stands (IS) is not required but highly recommended. The purpose of the internal standard is to compensate for matrix effects in the sample and the differences between sample prep and calibration standards. One can either spike the IS manually or introduce it on-line. The latter option provides better consistency and ease of use. One usually uses a smaller diameter pump tubing so as not to dilute the sample much. I recommend orange / green (flared end) which has an internal diameter of 0.33 mm. With the standard configuration, you will need a "Y barb" connector and appropriate pump tubing. If you are using the AVS-7 or ADS2 the Y barb is not required but it is recommended that you have this as a backup, should you have any need to bypass the AVS7 or ADS2.

Recommended parts from Agilent:

- Y barb P/N 1610132400
- Orange / green flared tubing P/N 3710068300

Sample Loops for the AVS7 and ADS2

The AVS7 and ADS2 add levels of automation designed to increase throughput for laboratories testing a large number of samples. Critical to using this added technology is the proper sample loops. This controls the amount of volume used in analysis. The amount of volume needed is based on a number of factors including number of modes, read time, and number of replicates. Loop sizes vary from 0.25 mL to 3.5 mL depending on the application.

ADS2:

If you have the ADS2 this also includes the AVS7 and thus you will need 2 identical loops of the same size. So, if you want to change loops then you need to buy 2 of the same size.

The ADS2 system includes 2 each of the 1.0 mL, 1.5 mL, and 2.5 mL sizes in order to cover a large range of applications and is usually sufficient.

Note: if you do not plan to use an internal standard then you will need a plug for port 7 on the AVS-7 normally used to introduce the internal standard.

- Plug P/N UCP316

AVS7 Only

If you have the AVS7 only then it comes standard with the 1.0 mL loop. This is often times not big enough. I would recommend getting a 2.0 mL loop to be safe. You can get a 1.5 mL loop as well but if you just want to get one then get the 2.0 mL loop.

- 2 mL loop P/N 5005-0426
- 1.5 mL loop P/N 5005-0425

Note: if you do not plan to use an internal standard then you will need a plug for port 7 on the AVS-7 normally used to introduce the internal standard.

- Plug P/N UCP316

Spares

I often get asked what spares to get ahead of time. This is a tough question. Sometimes it is best to just wait and see what you will really need.

The most important spare item is a spare nebulizer. Types of nebulizers vary by application need and preference. The standard nebulizer is the SeaSpray; however, we sometimes recommend other nebulizers like the ONE NEB or the MiraMist. Sometimes we try 2 types at training to see what works best. Sales will sometimes include multiple nebulizers on the order. For the most part the SeaSpray works well. If the plan is to try out different nebulizers at training it makes sense to wait to order a spare. If you are certain of the nebulizer type then I would order a spare of that nebulizer prior to training. Just use the part number on the box.

It is nice to have a spare torch, especially in a high throughput lab. In almost all cases the one piece 1.8 mm standard torch is perfect. There is usually no need for a demountable torch for most samples. If this has any benefit, I will alert you prior to the training. The value is only in very special circumstances. The most common is the analysis of highly basic solutions. If you are working with acid solutions then the one piece should be fine. Just use the part number on the box.

NOTE: in some cases, you may need specialized nebulizer / torch / spray chamber combinations. In that case you would want spares of those types.

Examples of specialized kits include: Inert Kit for using HF acid, semi-volatiles kit for semi-volatile solvents, volatile kit plus Isomist for volatile solvents, and high TDS kit for samples with very high total dissolved solids.

<u>Reagents</u>

For aqueous applications:

Water is the most critical reagent. I recommended ASTM Type 1 water. This is characterized by a resistivity greater than 18 megaohms. This can be purchased if your lab DI system is not up to the job. You would purchase this from wherever you buy chemicals (Fisher, VWR, etc.) If you don't have a meter to test resistivity on your DI water system unit I would be concerned. If there is some concern, I would purchase some ASTM Type 1 water and compare your DI to that water at the training.

Acids are the next most critical reagent. You will likely need either or both nitric acid and hydrochloric acid. Fisherbrand Trace Metals Grade (or equivalent to Fisher trace metals grade from another vendor) is required and also usually sufficient for most applications. You usually will not need a higher grade. There is significant risk to using a grade lower than trace metals.

Sample preparation may require additional acids or reagents. Just be aware of the purity of these other acids and reagents. Look for a trace metals grade designation or some way to see if they are tested for metals. If your sample prep includes hydrofluoric acid then you will need an inert sample introduction system as HF will attack the glass in the standard system.

- Inert Sample Introduction Kit G8010-68007

RBS-25

RBS-25 is a cleaning solution for the ICP-OES. This is made by Sigma and can be purchased from anywhere you get chemicals (Fisher, VWR, etc.) I would get it in a 1-liter size.

For non-aqueous applications:

We can test a wide range of solvents and the exact configuration for your solvent should be discussed with your sales rep and ordered with the system. If you are doing organic solvents, it is important that we discuss the particular application(s) prior to our visit. There are also installation considerations so this all has to be worked out prior to installation as well. For semi-volatile oil analysis in "kerosene" we have special clean versions of kerosene with no odor. Common brands are A-Solv (made by Agilent) and V-Solv (made by VHG). Once you pick a brand stay with it.

<u>Standards</u>

For aqueous applications:

I would purchase standards in single element stocks in 125 mL sizes. Ask for a quote from the vendor as you can probably get a discount buying multiple standards. Getting these in single stocks serves 2 purposes: 1) you can make what concentrations you need and 2) you need single element stocks to evaluate, isolate, and correct for spectral interferences. Once you have a method settled you can buy a multi-element standard. It is better to figure out what range you want for each metal and then get a custom mix later rather than to guess or work around a random off-the-shelf mix.

As far as preferred vendors I would go with:

1. Inorganic Ventures – good quality and excellent customer service
2. Spex Certiprep – excellent quality and solid customer service
3. Agilent – good quality but delivery can be a bit slow for standards compared to above vendors

Standards come in various concentrations but the most popular is either 1,000 PPM or 10,000 PPM. In order to blend standards together you want the stock to be fairly concentrated, especially if you have a high number of elements. For elements that you expect to be in higher concentrations I would recommend the 10,000 PPM concentration. For elements in more the trace category go with the 1,000 PPM. We can discuss this prior to training as well. It may make sense to get some pre-made blends if you really know the application and what you are expected to see.

For internal standards the main criterion is that it is not going to be in your sample. Otherwise, there is little to consider. Below are some ideas but people use a variety. Yttrium is by far the most popular internal standard but not everyone uses it.

- Yttrium (Y) at 10,000 PPM – internal standard – this is all you need but we have 2 additional options below
- Tellurium (Te) at 10,000 PPM – this is an optional internal standard for harder to ionize elements (As, Se, Sb, etc) used in addition to but not in replace of Yttrium.
- Rubidium (Rb) at 10,000 PPM – this is another optional internal standard that can work well with Li, Na, K in addition to but not in replace of Yttrium.

For non-aqueous (organic) applications:

In this case we tend to purchase standards in multi-element blends and only purchase single element stocks when evaluating / correcting for interferences or when getting an element that is not in a regular blend. Standards are organometallic and tend to come in two weight sizes (25 cST and 75 cST) which means either a lighter or heavier mineral oil. This does not always approximate sample matrices and so we can use an internal standard here as well. You will want to purchase a bottle of base oil in the same weight as the stock standard to use as a blank and to help matrix match the standards.

All samples and standards are diluted with solvent so the internal standard is added to the diluent rather than mixed with a Y connector.

Stock standards range in concentration from 100 PPM to 900 PPM and everything is done by weight. Because of this you will need an accurate and reliable 4 place (at a minimum) balance in order to make up standards.

The most popular blends of organometallic stock standards are a 21 element or a 21 element plus potassium. You can also get single stocks for elements not in the blend or also if you have a smaller list. Do not mix a sulfur standard with the blend and be careful when mixing other single stocks.

As far as preferred vendors I would go with:

1. Agilent
2. VHG

<u>Labware</u>

Pipettes – for aqueous standards

It is critical to able to accurately make standards and spike samples. I suggest getting at least 2 adjustable micropipettes. Make sure you get the correct tips for each and a large supply. I suggest the following size pipettes. Using glass volumetric or disposable glass pipettes is not recommended.

- 100 ul to 1,000 uL – this will be used most often
- 10 uL to 100 uL – this is important to spike QC samples and is just handy in general.
- 1 mL to 5 mL – this is optional and used for larger volumes and is particularly useful when making dilutions.

There are a variety of vendors of pipettes. Eppendorf is worth the investment if you want the best quality and reliability over time.

Syringe

A plastic syringe is recommended to clean the nebulizer. You may purchase a professional tool called an ELUO from Agilent or Glass Expansion. Or, you can simply use a disposable syringe normally used for syringe filters.

Plasticware

It is highly recommended that you only use only plasticware and not glassware. This is critical when testing for silicon and boron, as these will leach out of glassware. Options include purchase of class A plasticware or verification of class B plasticware or verification of disposable plasticware like digestion vessels. I prefer the latter. I would recommend using plastic digestion vessels.

I recommend Environmental Express for all plasticware. Either 50 or 100 mL digestion vessels are perfect for making standards. I recommend to make smaller amounts of standards more frequently than large amounts less frequently.

Test tubes for the autosampler come in a variety of sizes and we have a variety of racks available. The company Mold Pro is a good quality economical source of autosampler tubes. I would get the caps (that look more like stoppers) for the test tubes. This way you can invert them for dilutions.

<u>Sample preparation</u>

This is somewhat out of the scope of our part but critical to your success. Please have an idea of how you plan to prepare the samples and prepare some, if possible, prior to training. If sample prep does not fully dissolve the sample look into ways to filter out particulates as they may clog the nebulizer. A 2-micron size filter is all that is needed to filter the samples. You don't want to use too small a pore size unless it is part of your method to do so.

There are so many ways to prepare samples that we cannot really get into it. Inorganic Ventures has some good information on its website. Sample preparation includes many ways to dissolve the samples and aids in doing so that bring chemicals, heat, and pressure.

One thing to consider is the initial weight or volume and the final weight or volume. This represents how much of a dilution the sample is going through in the prep step and will determine your sensitivity. Also, how much you start and finish with affects the ability to dissolve the sample.