

## **Procedures for Submitting Air Sensitive Samples for Elemental Analysis**

swv 10-25-07

There are two submission procedures we recommend for air sensitive samples; the choice depends upon your assessment of the stability of the sample.

### **Type A. Samples that are Stable in Air for at Least Five Minutes**

These samples should be submitted in multiple small vials. For two runs per sample, split your sample between two small vials. In your glove box, load plenty of the sample (at least 10 mg) into each vial. Cap the vial. Although we will only use about 2 mg per run, sample transfer and weighing can be done much more quickly if there is excess sample in the vial. Be sure that your sample is a fine powder. This will avoid us having to spend extra time crushing the sample before we weigh it. Once you have loaded your sample into vials, place each vial inside a larger bottle, preferable with a drying agent. Cap the bottle. This should also be done inside your glove box.

When you submit your samples, clearly label them as “Air Sensitive – Run Manually” on the submission form. As long as both the vials and the larger bottle are filled with inert gas (typically nitrogen, helium or argon) the samples should be stable, however you could, at your discretion, notify me that you would like to submit Type A air sensitive samples so that we can coordinate our analysis schedule with your sample submission.

### **Type B. Samples that are NOT Stable in Air for at Least Five Minutes**

These samples need to be loaded into small pre-weighed tin vessels. These should be requested from me several days in advance. NOTE: Because of the weighting procedure used, please do not use pre-weighed vials left over from previous runs. Request new ones. You will receive small numbered vials with one tin vessel in each vial. The tare weight of the tin vessel may or may not be marked on the small vial. Don't worry if it is not. You will also be given one larger glass vial for each small vial. These are not numbered and can be used interchangeably. Inside your glove box, you will load approximately 2 mg of sample into each tin vessel, then crimp and fold the tin vessel in the manner you have been taught and have practiced. It is important that you have a balance inside your glove box in order to roughly weigh the amount you are putting into each tin capsule. Weights may range from 1.5 mg to 2.5 mg.

Place each folded and crimped tin vessel into its numbered small vial. Cap the vial. Don't get them mixed up, or the weights will not be correct. Then place each small vial inside one of the interchangeable larger glass vials. Cap the vial. Use a permanent marker to label each of the vials or vial caps. All of this should be done inside your glove box.

When you submit your samples, clearly label them as “Air Sensitive – Run Manually” on the submission form. As long as both the small vials and the larger vials are filled with

inert gas (typically nitrogen, helium or argon) the samples should be stable, however for Type B air sensitive samples you MUST notify me that you would like to submit air sensitive samples so that we can coordinate our analysis schedule with your sample submission.

The sealed tin vessel will be exposed to air for about sixty seconds during the final weighing process. As long as the tin vessel has been properly crimped and sealed (without any cracks or tears in the tin) we will have a fighting chance of getting good results for you.

### **Training and Tools**

You will need to practice loading samples into the small tin vessels. Using any powdered sample, practice loading, sealing, and crimping the tin vessels on your lab bench. Obviously you do not need pre-weighed vessels for practicing your technique. Next, practice your technique working inside your glove box. Only when you can consistently load, seal and crimp tin vessels while working inside your glove box should you begin loading your air sensitive samples into pre-weighed vials for analysis.

A number of small tools including tweezers and spatulas are necessary. You may borrow mine if you ask for permission.