Overview of XAS sample preparation by filtration of fine powders

I – Equipment:

A. A filtering flask, a.k.a. Erlenmeyer with vacuum connection, such as the one depicted below.

B. A glass funnel kit for filtering from Millipore, available from their website www.millipore.com (Catalogue Number: XX1002514). Looks like this:

C. A vacuum pump (doesn't have to be very powerful, bench-top pumps will do) and, if available, a vacuum vessel (empty metallic container) to save work on the pump and give you a vacuum reading. For example:

D. Filtering papers from Millipore, available with different pore sizes from their website www.millipore.com. The best filters to support the final samples are the ones with pore size of 0.22 μm. Some product codes:
   - Pore size 0.22 μm - Catalogue Number: GVWP02500
   - Pore size 5.0 μm - Catalogue Number: SVLP02500
   - Pore size 10.0 μm - Catalogue Number: AN1H04700

   Here is a picture of the blue box (comes with 100 filters packed), and a filter on top:

E. Some kind of solvent solution. For my samples ethanol worked very well, while water didn't work at all. Acetone is also an option. It is important to use something that doesn't react with your powders.

F. Several 50 ml and/or 100 ml beakers (bigger ones will cause more loss of material stuck at the walls).

G. An ultrasonic bath (any brand will do), such as this one:

II – <u>Procedure</u>:

1. Crush your powders as fine as you can. You can use a milling system if you have one (will-o-bug, for example) or a mortar and pestle. It takes a while to get really fine powders by grinding with a mortar and pestle. Make sure you use the proper mass of powder to get a good transmission sample (use the Xafs mass program to calculate the amount of material for the best absorption and edge step). If you want really fine grains (< 5 μm), put twice the mass on the mortar because not many grains will come out as small as you want them. The area of your final sample is given by the diameter of the filtering tube aperture. For the system depicted in Section I-B above, the sample area will be 2.01 cm^2. (OBS: keep in mind that after the filtration process is complete and the filter is covered by Kapton tape, you can cut it in half and stack the two pieces, effectively having the same amount of material but in half of the initial area).

2. Mix your fine powder with the solvent solution in a beaker. If the mass is 20 mg or less, use at least 20 ml of solvent. If the mass is up to 200 mg, use 80-90 ml of solvent. These numbers are empirical, so you have to optimize them for your system.

3. Put the beaker with solvent and powder in an ultrasonic bath and let it shake for a couple minutes (make sure no powder is deposited at the bottom when you take it out). While the solution is mixing, assemble the filtering system as described below.

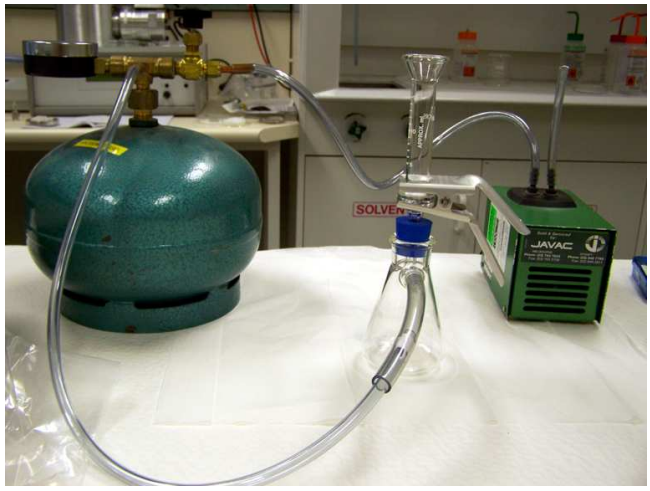
4. Your filtering system should have the components listed in Section I above and depicted here:

   - Firmly attach the connector to the Erlenmeyer and place the metal grid on top of the connector as shown below:

- Place the filter paper on top of the metal grid. Make sure it is well centred:

- Now place the funnel on top of the filter paper, making sure it is well centred, and use the metal clamp to keep them together:

- Connect the pump (or vacuum vessel if you have one) to the Erlenmeyer, making sure there are no leaks in the system:



5. Take the solution out of the ultrasonic bath and pour it on the funnel until it is almost full. Then turn on the vacuum pump to suck the liquid through the filter. Particles

smaller than the filter pore will be in the solution inside the Erlenmeyer while bigger particles will be stuck in the filter paper. If you want the smaller particles, transfer the solution from the Erlenmeyer to a new beaker and continue from step 6 below. But if you are sure your particles are fine enough or if you just don't care about it, all you need is using one 0.22 μm filter paper and collecting all powder on it. When all liquid is sucked into the Erlenmeyer, turn off the pump, break the vacuum by removing the hose attached to the Erlenmeyer, carefully remove the metal clamp and funnel, get the filter containing the powder using tweezers and lay it on a glass slide to dry. After it is well dry, cover both sides of the filter with Kapton tape. Be very careful not to disturb the powder (don't press it with your fingers, for example). It is good practice to weight the filter paper before and after the process, so that you know how much powdered material you really have after the filtration process.

6. In most cases particle size IS important, though. Which means it is safer to first pass the initial solution through 10 μm filters, and then get the solution that made it through the first filtering and pass it through 5 μm filters, and so on, until collecting the final powder in a 0.22 μm filter. This means repeating steps 3-5 many times. Keep in mind that all particles that go through the 10 μm filter into the Erlenmeyer will have sizes smaller than the filter pores (10 μm), but not all particles retained in the filter are bigger than the pores, because the filter may get clogged with bigger particles impeding further passage of small particles. About 30-40 ml of solution is enough to clog a filter, which means you will have to use several filters. You should collect the used filters and place each of them in a small beaker with ~ 20 ml of solvent, then shake them in the ultrasonic bath. You will see that most of the powder gets loose. This powder may still contain small particles, so you should pass these solutions through a new filter. And so on. Keep in mind this requires a considerable amount of solvent, and at the end of the process you may have to pass 100-500 ml of solution through the final 0.22 μm filter to be able to collect enough powder. The complete process takes some time and requires constant work.

7. Needles to say, each researcher is responsible for identifying the hazards associated with such a procedure (spills, inhalation of powders, toxicity of powders and/or solvent solution, etc) and assuring that all cautionary measures are taken (reading MSDS, assessing risks, using personal protection equipment, working in a fume hood, etc).

This document was elaborated as an overview of the process and not every step was explained in extreme detail. But the concept is very simple and users should be able to optimize the procedure for their particular system.

Prepared for the XAS beamline users on 24 May 2010 by Leandro L. Araujo.