

How Much Sample Preparation is Necessary?



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The two most important considerations for sample preparation are the concentration of the element of interest and the thickness of the sample.

XAS can be done on solid samples of a variety of sizes and concentrations, as well as on fluid samples. How much sample preparation you need to do in order to get good quality XAS data will obviously depend on the starting conditions of your sample. Fortunately there are several modes of signal detection possible in XAS and, as a result, you have some flexibility on the degree of sample preparation you need to do. In XAS the two most common modes for signal detection are transmission and the fluorescence modes. There are two main sample issues you must consider for XAS: 1) the concentration of the element you wish to

SAMPLE	Concentrated (>10 mol %)	Dilute (<10 mol %)
Thin ($\mu x < 0.1$)	Transmission/Fluorescence	Fluorescence
Intermediate ($0.1 < \mu x < 3$)	Transmission/Fluorescence (some self-absorption expected)	Fluorescence/Transmission (Tr. may be possible)
Thick ($\mu x > 3$)	Transmission (needs sample thinning / Fluorescence (self-absorption expected)	Fluorescence



Preparing Free-Standing Samples



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Free-standing samples can either be thinned to an appropriate thickness, or measured in fluorescence.

Your sample may be a free-standing piece (for example a metal alloy). In such a case:

If the sample falls in the thick and concentrated regime of table in Slide 2, you may decide to thin the sample by polishing it until it has a usable thickness for transmission experiments. In doing so you can avoid self-absorption effects of fluorescence-mode measurements.

If you want to look at multiple absorption edges from the sample, you may do a hierarchical thinning of the sample to achieve the proper μ for transmission at each absorption edge. The dimple polisher, an instrument often available in

Thicker rim for
lower μ component

Thinner area for
higher μ component



Preparing a Powder Sample on Tape

One way to prepare a powdered sample is to spread thin layers on tape.

Initial preparation of the powder: If powder sample is to be used for XAS, an initial step of sieving the powder should be used to place an upper bound on the thickness of the powder particles. The average particle size should not be more than one attenuation length. If they are larger than that, the powder should be crushed using a mortar and pestle. The video below describes the procedure for preparing and dispersing a powder sample onto an adhesive tape such as the polyamide-based Kapton™. Make sure that the tape must not contain the element of interest (whose absorption you are to measure):



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