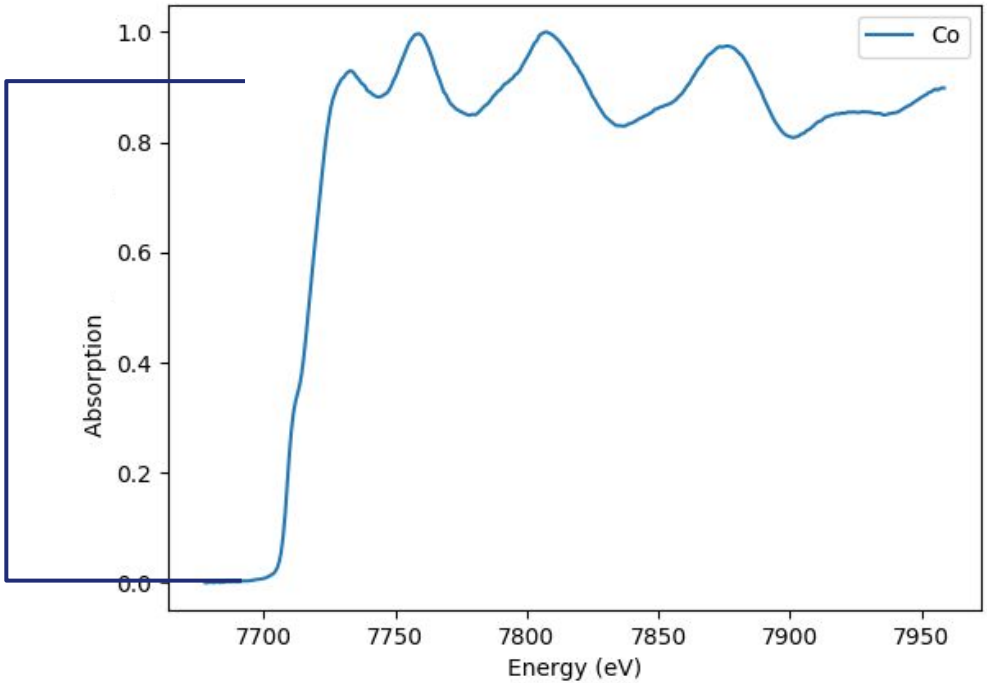
XAS Sample Preparation

Ari-Pekka Honkanen

What we want to measure?

$\Delta \mu x$



Calculating the amount of sample (theory)

- Maximise the quantity of interest: $\Delta\mu x$
- However, we need to have some transmission above the absorption edge, thus the rule of thumb:

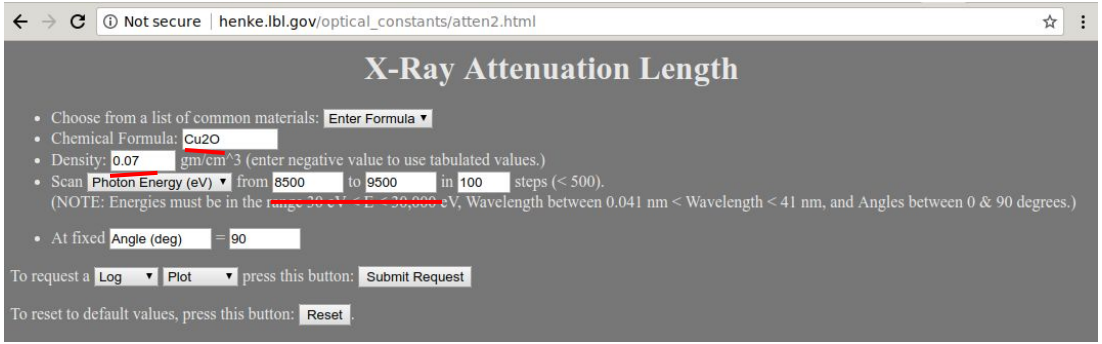
$$\Delta\mu x = 1 - 3$$

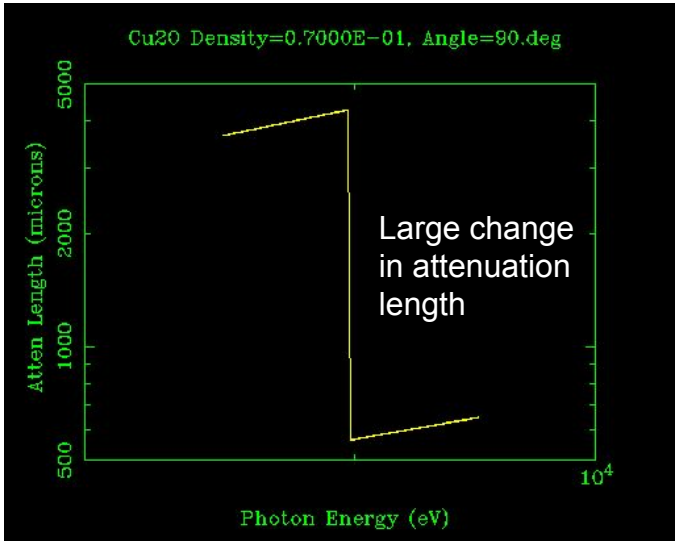
- Exception: If the sample contains other heavily absorbing elements, may have to use smaller $\Delta\mu x$
- For low edge energies (~ 5 keV), check the absorption of the filler and the tape

Calculating the amount of sample (practice)

1. Measure the thickness x of the sample container
2. Open a program or web site to calculate X-ray absorption lengths
   e.g. Hephaestus in Demeter package or
   http://henke.lbl.gov/optical_constants/atten2.html
3. Type in the chemical formula and the energy of the absorption edge
4. Vary the density ρ of the material until the absorption length $1/\mu$ is 0.5 - 1.0 times the sample container thickness (or more, if the sample heavily absorbing)
5. Obtain the needed mass of the sample by $m = \rho V$, where V is the volume of the sample container

Example: Cu_2O at Cu K edge





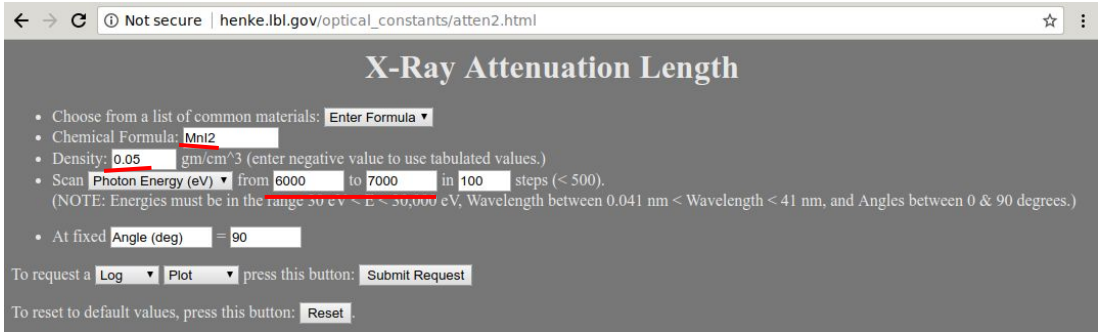
If sample holder thickness = 1 mm

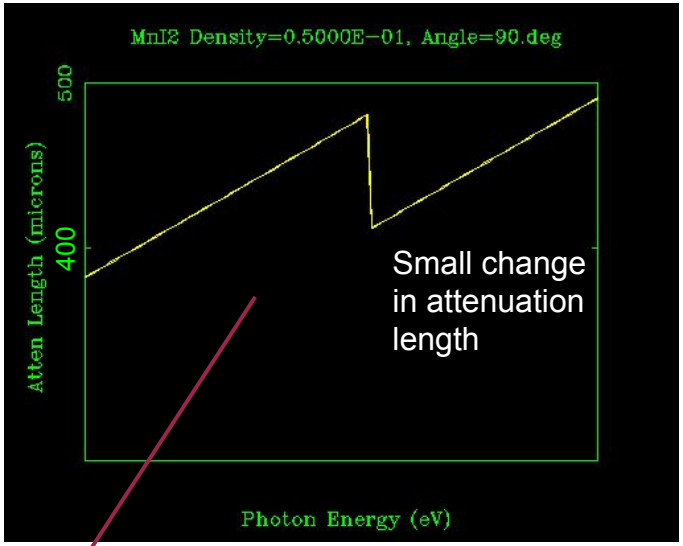
and area = 1 cm^2

-> Good sample density = 0.07 g/cm^3

-> Mass = 7 mg

Example: MnI_2 at Mn K edge

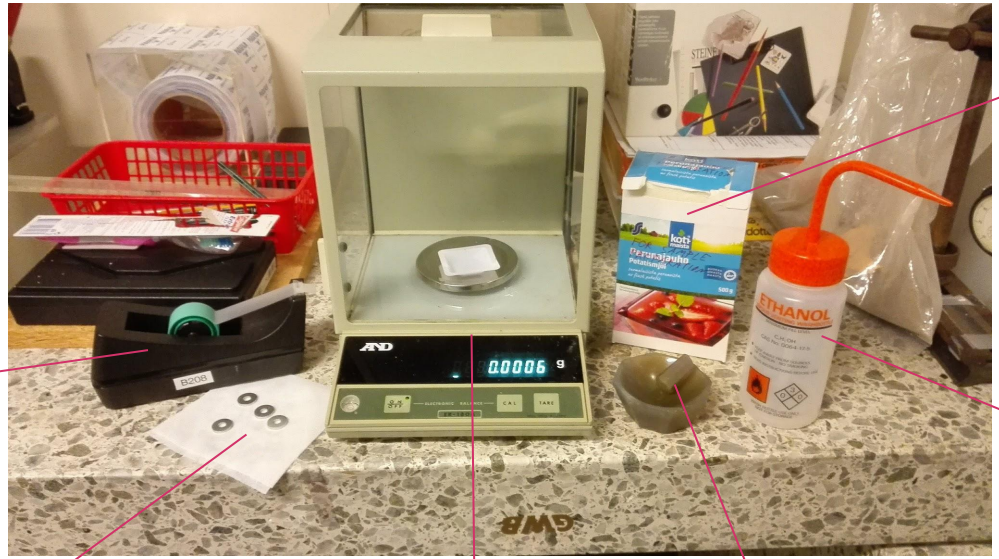




If sample holder thickness = 1 mm
and area = 1 cm^2
-> “Good” sample density = 0.05 g/cm^3
-> Mass = 5 mg

SOME SAMPLES MORE DIFFICULT THAN OTHERS!

Preparation: needed equipment



Scotch tape or Kapton tape

Empty sample containers e.g. washers

Scale

Mortar and pestle

Filler e.g. starch or boron nitride (check the compatibility with the sample)

Ethanol (check the compatibility with the sample)

Preparation steps

1. Check the material safety data sheet (MSDS) and use appropriate protection!
2. Weigh the calculated amount of sample
3. Weigh the filler (test the needed amount with your container)
4. Grind the sample and filler into a homogeneous mix using the mortar and pestle. If possible, use ethanol to get all the sample out of the weighing plate and to help mixing
5. Tape the other side of the sample container
6. Pour in the mix and fill the container tightly (a press is preferable, if available)
7. Clean the edge of the container from powder and seal with tape (note that static charge may attract powder).
8. LABEL YOUR SAMPLE!

DONE!

More difficult samples..

- Low concentrations/extremely high absorption: try using fluorescence mode
- Liquids problematic due to X-ray induced dissociation: gelification, continuous flow
- Sensitive samples: increase the beam footprint, use filters