

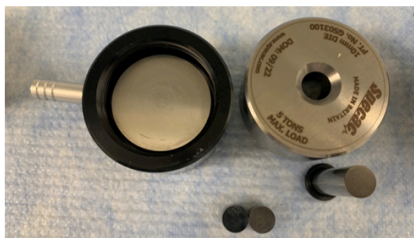
Pellet die set choice and optimal load

There are three available die sets for BioXAS Sector in which samples can be prepared from to make pellets (5mm, 10 mm and 13 mm). Each die set has a maximum load capacity (see below). The difference in choice can be decided on by (available sample mass, density of sample, compressibility of sample, how well it mixes with binder, the required amount obtained from edge step calculations).

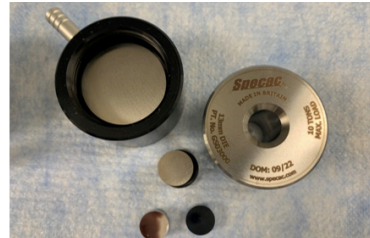
**5 mm die
2 TONS**



**10 mm die
5 TONS**



**13 mm die
10 TONS**



Binder Choice

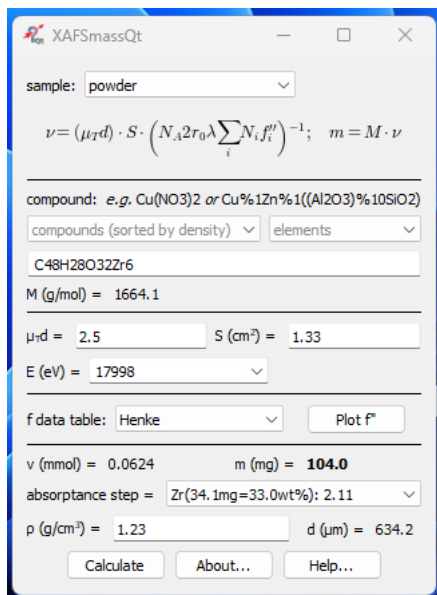
| Binder | density (g/cc) | notes |
|-----------|----------------|--|
| C | 2.26 | doesn't make good pellets |
| BN | 2.1 | Ok, but sticky |
| PEG | ~1.1 | may burn if pellet is going to be heated |
| Cellulose | 1.5 | may burn if pellet is going to be heated |

Edge Step Calculation for Optimal Sample Preparation

Example using ZrMOF with chemical formula of C48H28O32Zr6 (density~ 1.237g/cc) for measurements at the Zr K edge

Using XAFSmass

an edge step of 2.11 will be obtained if using a pure pellet made of the sample (104mg) ; can dilute to make a total mass (diluent and sample) of 100-150 mg (10 mm die)



Using Catmass

an edge step of 2.1 will be obtained if using a pure pellet made of the sample (66.43 mg) ; can dilute to make a total mass (diluent and sample) of 100-150 mg (10 mm die)

CatMass - XAS Sample Mass Calculator

File Edit Resize Help

Sample and Dilution Definition

Sample Builder

Stoichiometry: Sample Diluent

Mass Dilution Ratio:

Reset

File Path

Edge Scan and Absorption Properties Definition

Diluted Sample Chemical Formula:

Element to be Scanned:

Edge to be Scanned:

XAS Cells:

Capillary Diameter [mm]

Pellet Diameter [mm]

Other

Sample Area \perp to Beam [cm²]:

Catalyst Sample Absorption at E0 + 50 eV:

Sample at 45°

Show Plot

Reset

Results

Calculate Sample Mass

Diluted Sample Mass [mg]:



Estimated Edge Step:

Sample Mass [mg]:

Diluent Mass [mg]:

Edge Energy [eV]:

X-ray transmission through media

Using Hephaestus

an edge step of 1.2 will be obtained if using a pure pellet made of the sample (31 mg) ; can dilute to make a total mass (diluent and sample) of 100-150 mg (10 mm die)

Hephaestus

Hephaestus Plot Help

Formulas: compute total cross sections of materials

Absorption

Formulas

Ion chambers

Data

Transitions

Edge finder

Line finder

Standards

F' and F''

Configure

Materials

- Acetone
- Air
- Alcohol (Ethyl)
- Alcohol (Methyl)
- Alcohol (Propyl)
- Aluminum
- Argon
- Beryllium
- Boron Nitride
- Carbon (Diamond)
- Carbon (Graphite)
- Copper
- Fluorite
- Gold
- Helium
- Iron
- Kapton
- Kimol
- Krypton
- Lead
- Lead Titanate
- Mica
- Molybdenum
- Mylar
- Neon
- Nitrogen
- Parylene-C
- Parylene-N
- Platinum
- PMMA
- Polycarbonate
- Polymide
- Polypropylene
- Rutile
- Salt
- Sapphire
- Silicon
- Silver
- SiO2 (Quartz)
- SiO2 (Silica)
- Tantalum
- Teflon
- Toluene

Formula Element

Density g/cm³

Energy

Results

| element | number | barns/atom | cm ² /gm |
|---------|--------|------------|---------------------|
| C | 48.000 | 10.773 | 0.540 |
| H | 28.000 | 0.623 | 0.372 |
| O | 32.000 | 29.925 | 1.126 |
| Zr | 6.000 | 14345.890 | 94.692 |

This weighs 1664.024 amu.

Absorption length = 255.1 micron at 17999 eV.

A sample of 1 absorption length with area of 1 square cm requires 31.555 milligrams of sample at 17999.00 eV.

Unit edge step length at Zr K edge (17998.0 eV) is 309.5 microns

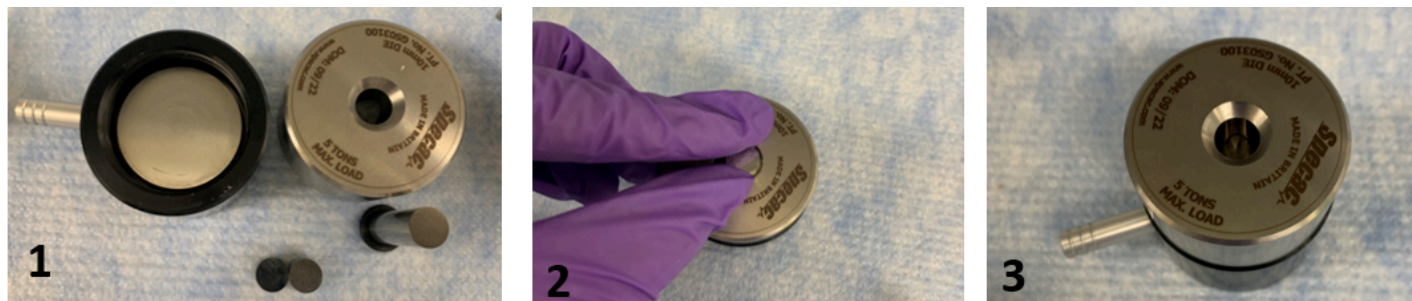
The Elam database and the full cross-sections were used in the calculation (step width = 18.00 eV)

Transmitted fraction

%

This calculation uses the Elam data resource and full cross sections.

Assembly of die set



Preparing a Pellet

After a specific powder sample (make sure finely ground using mortar and pestle) is weighed, it can be transferred into the die seen, as seen below. After waiting at least 5 minutes at the designated pressure, release the black knob and retract die. Open die set carefully and gently push pellet onto a weighing paper. Use Kapton to sandwich the pellet onto it to create a free-standing pellet.

