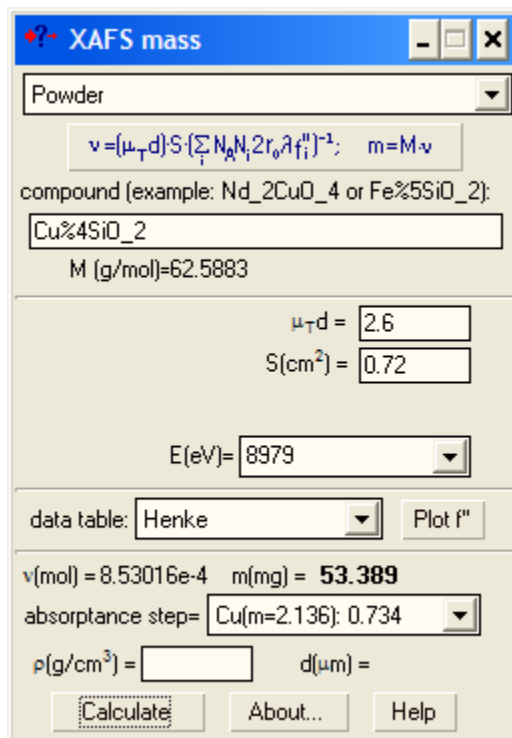


## XAFS Mass

### 1. Calculation of mass and absorption step for powder samples



**XAFS mass**

Powder

$$v = (\mu_T d) S \left( \sum_i N_A N_i 2r_0 \lambda_i^4 \right)^{-1}; \quad m = M \cdot v$$

compound (example: Nd<sub>2</sub>CuO<sub>4</sub> or Fe<sub>5</sub>SiO<sub>2</sub>):

M (g/mol)=62.5883

$\mu_T d =$    
 $S(\text{cm}^2) =$

E(eV)=

data table:  Plot f''

v(mol) = 8.53016e-4    m(mg) = **53.389**

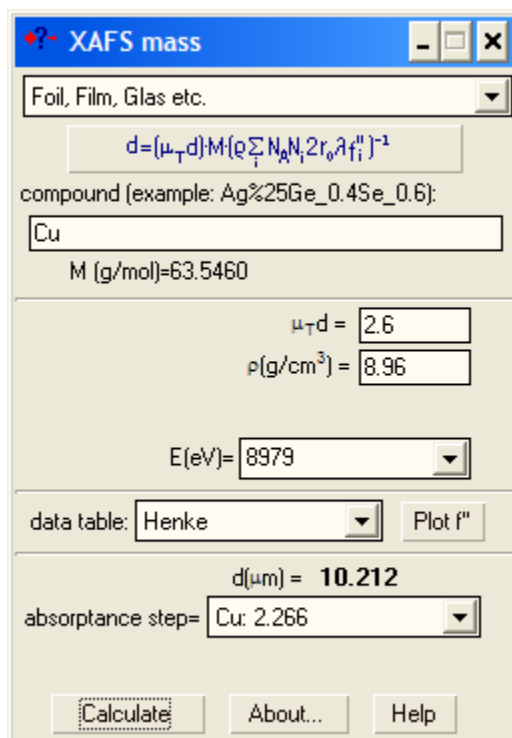
absorptance step=

$\rho(\text{g}/\text{cm}^3) =$       $d(\mu\text{m}) =$

A typical application is the calculation of the mass for a powder sample. The optimal optical sample thickness  $\mu d$  depends on the absorption levels selected for the ionization chambers (see below). Typically,  $\mu d$  is between 2 and 3 (e.g. for a 17.5% absorption level for the 1st chamber and a 50% level for the 2nd chamber, the optimal thickness is 2.41). However, if you get the absorption step more than 1.5, it is recommended to reduce the sample mass to avoid potential thickness effect due to possible inhomogeneity in the wafer. If your sample is diluted and you get a very low absorption step, do not try to make the wafer thicker hoping that you will get better spectra - you will not: The optimal thickness gets the best signal-to-noise ratio (it is in this sense it is optimal). You can only try to measure your absorption spectra with another registration technique: in fluorescence or electron yield modes.

### 2. Calculation of thickness and absorption step for samples with known density



**XAFS mass**

Foil, Film, Glas etc.

$$d = (\mu_T d) M \left( \rho \sum_i N_A N_i 2r_0 \lambda_i^4 \right)^{-1}$$

compound (example: Ag<sub>25</sub>Ge<sub>0.4</sub>Se<sub>0.6</sub>):

M (g/mol)=63.5460

$\mu_T d =$    
 $\rho(\text{g}/\text{cm}^3) =$

E(eV)=

data table:  Plot f''

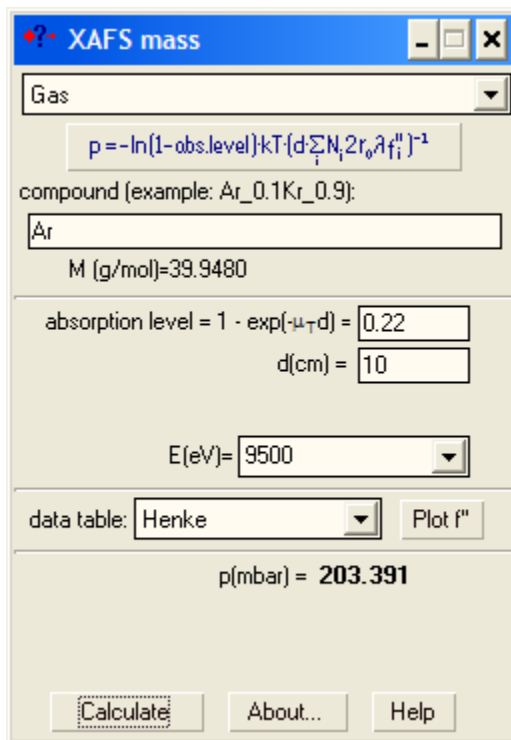
$d(\mu\text{m}) =$  **10.212**

absorptance step=

Here you can calculate the thickness of the sample with known density (usually, a foil). Commercial foils are highly homogeneous in thickness, so that you may ignore large step jumps and pay attention to the total  $\mu d$  only.

### 3. Calculation of gas pressure for ionization chambers



**XAFS mass**

Gas

$$p = -\ln(1 - \text{obs.level}) \cdot kT \cdot (d \cdot \sum_i N_i \cdot 2r_0 \lambda_i^3)^{-1}$$

compound (example: Ar\_0.1Kr\_0.9):  
Ar  
M (g/mol)=39.9480

absorption level =  $1 - \exp(-\mu_T d) = 0.22$   
d(cm) = 10

E(eV) = 9500

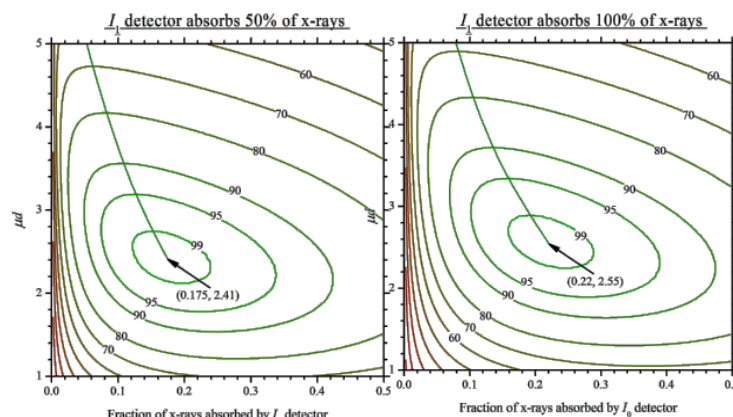
data table: Henke Plot f''

p(mbar) = 203.391

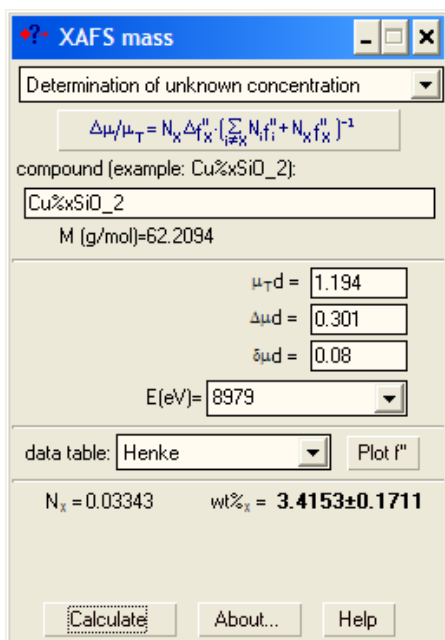
Calculate About... Help

For nitrogen, do not forget the 2: N<sub>2</sub>, not just N.

Start with the 2nd ionization chamber (IC). If a reference foil is placed between the 2nd and the 3rd IC, the fraction of x-rays absorbed by the 2nd IC is usually set to 50%. If the reference foil is not needed, one can select total absorption (100%). For these two cases the optimal absorption of the 1st IC at a certain  $\mu d$  is found from the following figures showing the levels of signal-to-noise ratio:



### 4. Calculation of an unknown elemental concentration



**XAFS mass**

Determination of unknown concentration

$$\Delta\mu/\mu_T = N_x \Delta f_x'' / (\sum_{i \neq x} N_i f_i'' + N_x f_x'')$$

compound (example: Cu%SiO<sub>2</sub>):  
Cu%SiO<sub>2</sub>  
M (g/mol)=62.2094

$\mu_T d = 1.194$   
 $\Delta\mu d = 0.301$   
 $\delta\mu d = 0.08$

E(eV) = 8979

data table: Henke Plot f''

$N_x = 0.03343$  wt%<sub>x</sub> = 3.4153±0.1711

Calculate About... Help

Case 1: You know the composition of the matrix. You need an absorption spectrum taken without the sample (empty spectrum) but with the same state of the ionization chambers. You then subtract it from the spectrum of the sample (e.g. in VIPER) and get a real (i.e. not shifted vertically) absorption coefficient. Determine the value of  $\mu d$  above the edge ( $\mu_T d$ ), the edge jump ( $\Delta\mu d$ ) and its uncertainty ( $\delta\mu d$ ). Specify the chemical formula with x.

**XAFS mass**

Powder

$$v = (\mu_T d) S \left( \sum_i N_i N_i 2r_i \rho_i f_i'' \right)^{-1}; \quad m = M \cdot v$$

compound (example: Nd\_2CuO\_4 or Fe%5SiO\_2):  
  
 M (g/mol)=63.5460

$\mu_T d =$    
 $S(\text{cm}^2) =$

E(eV)=

data table:

v(mol) = 1.37562e-5    m(mg) = **0.874**  
 absorbance step=

$\rho(\text{g}/\text{cm}^3) =$       $d(\mu\text{m}) =$

Case 2: You know the sample mass and area. Determine the edge jump ( $\Delta\mu d$ ). For the pure element find such a value for  $\mu_T d$  that the absorption step in the pull-down list was equal to your experimental  $\Delta\mu d$ . This will give you the mass of the element of interest. Just divide it by the total mass to get the weight percentage.

## 5. Calculation of two unknown elemental concentrations

**XAFS mass**

Determination of two unknown concentrations

$$[\Delta\mu_T/\mu_T]_X = N_X \Delta f_X'' \left( \sum_{i \neq X, Y} N_i f_i'' + N_X f_X'' + N_Y f_Y'' \right)^{-1}$$

compound (example: Cu%zZn%ySiO\_2):  
  
 M (g/mol)=65.1980

for x:	$\mu_T d =$ <input type="text" value="2.499"/>	for y:	$\mu_T d =$ <input type="text" value="2.290"/>
	$\Delta\mu d =$ <input type="text" value="0.915"/>		$\Delta\mu d =$ <input type="text" value="0.256"/>
	$\delta\mu d =$ <input type="text" value="0.1"/>		$\delta\mu d =$ <input type="text" value="0.04"/>

E(eV)=     E(eV)=

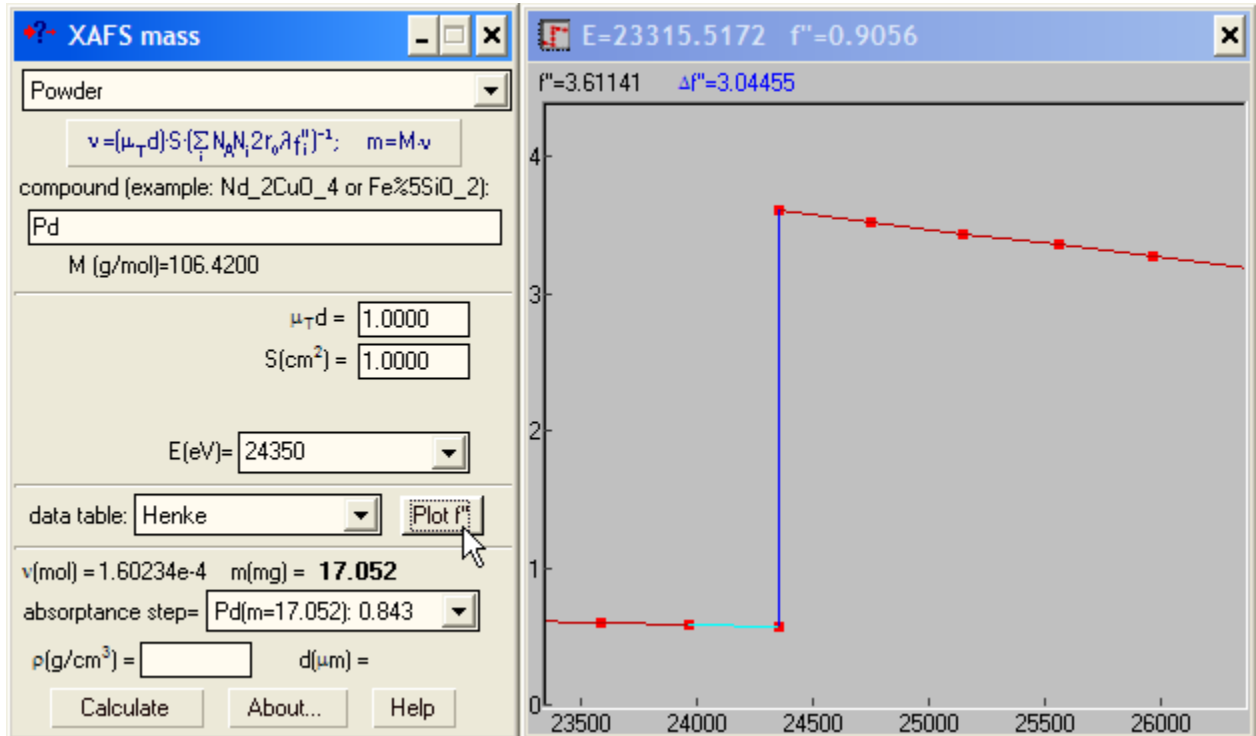
data table:

$N_x = 0.06152$      $\text{wt}\%_x = \mathbf{5.9959 \pm 0.1783}$      $N_y = 0.01842$   
 $\text{wt}\%_y = \mathbf{1.8466 \pm 0.0549}$

Here you also need empty spectra (for each of the two edges) to find the unshifted values for absorption coefficient above both edges.

## 6. Finding the scattering factors $f''$



If you need to know the scattering factor  $f''$  at different energies and/or its jump at an edge ( $\Delta f''$ ), XAFSmass provides a graphical tool for this.

For example, you may need these values to determine the composition of a binary compound if you have the experimental edge heights at two edges. The absorption step  $\Delta\mu d$  at the absorption edge of energy  $E$  is proportional to  $\Delta f''v/E$ , where  $v$  is the amount of (resonantly) absorbing atoms in mole. Hence, the atomic ratio of two elements in the same sample is  $v_A/v_B = (\Delta\mu d)_A/(\Delta\mu d)_B \cdot [\Delta f''_A/\Delta f''_B \cdot E_A/E_B]$ . For binary compounds  $A_xB_{1-x}$  the concentration  $x$  is calculated then as  $x = (v_A/v_B)/[1+(v_A/v_B)]$ .