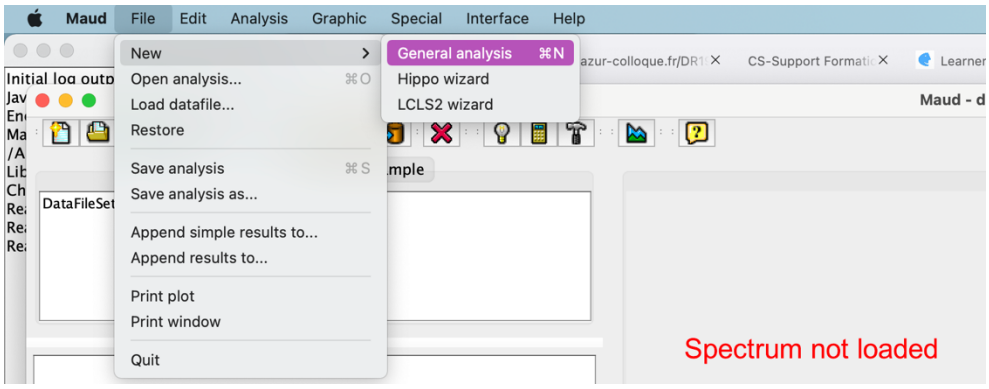
MAUD Software – Basis

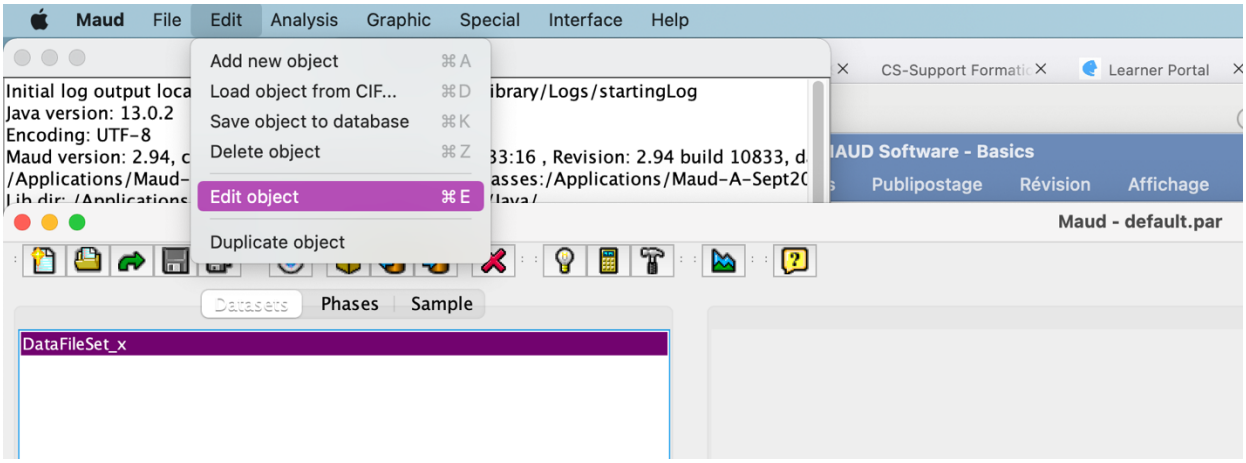
To start a new analysis

1- Open Maud software

2- File -> new -> general analysis

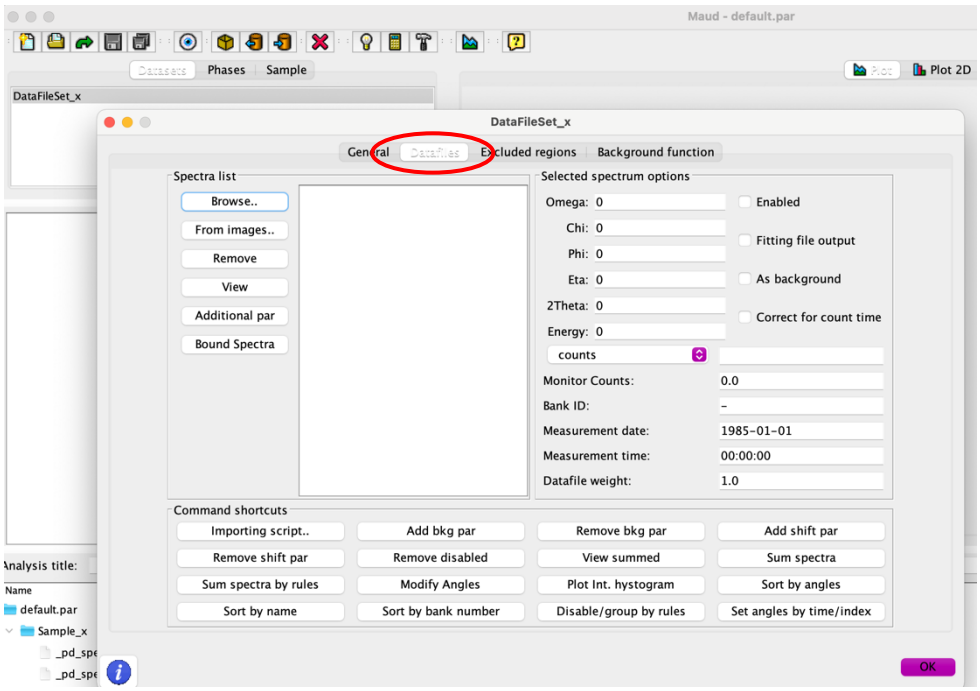


3- Main page - Datasets: select DataFileSet_x -> MAUD menu click on “edit” -> “edit object”

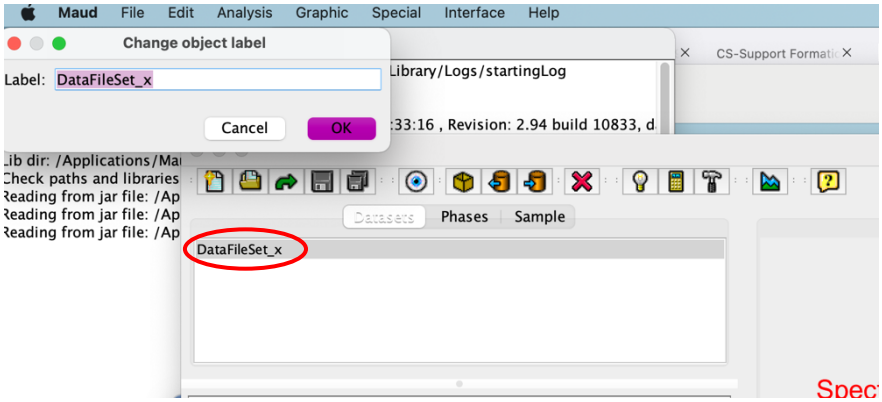


Or click directly on 

4- Go to item “datafiles” -> **browse** -> your **xrd file** -> ok

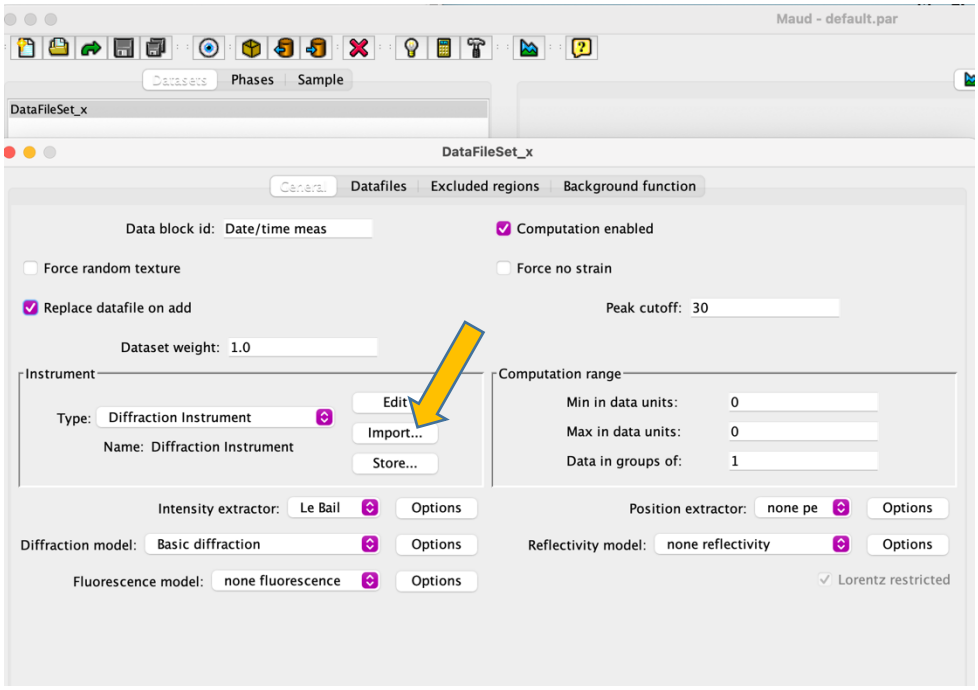


5- To put the sample name: Datasets -> double click on “DataFileSet_x” and change the name

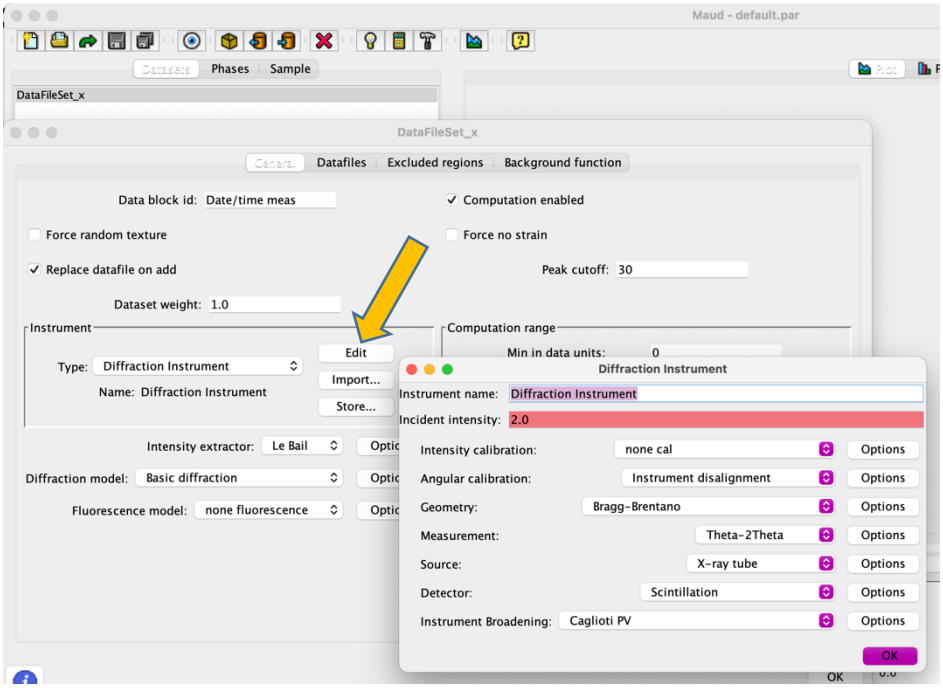


6- **instrument parameters**

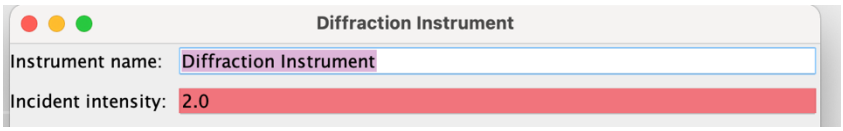
(i) If you have the instrument parameters .mdb files load it “Datafiles” -> select DataFileSet_x ->  -> instrument part -> import your instrument parameters file



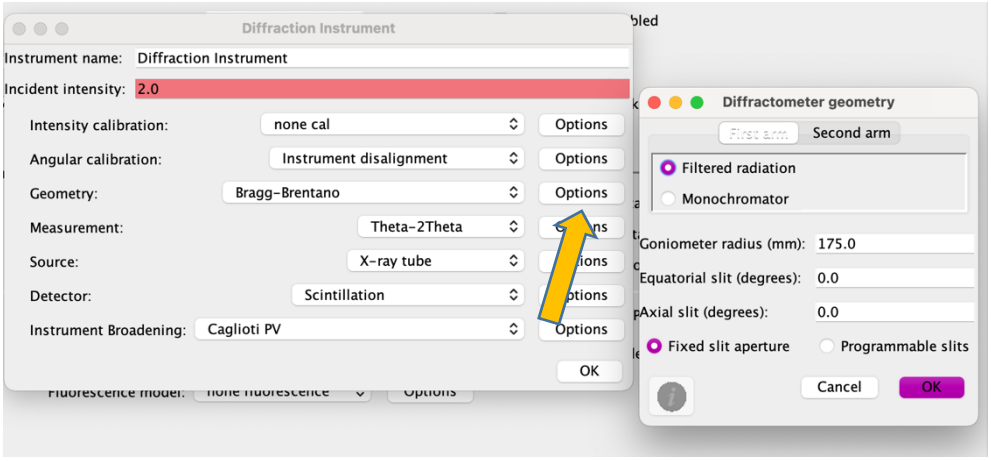
(ii) or complete manually the instrument part clicking on “Edit”



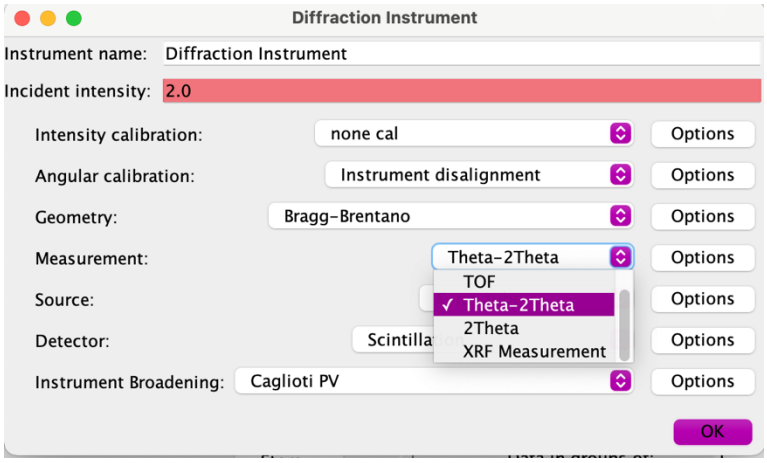
a) put the instrument name



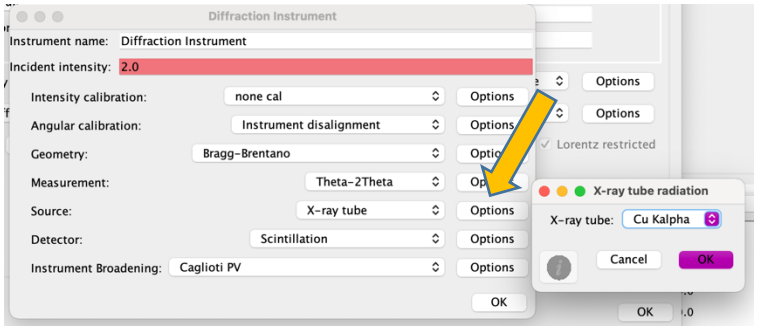
b) Geometry: choose the adapted geometry and click on options to put the type of mirror, slit opening size, goniometer size for both first and second arms.



c) Measurement: select the appropriate configuration



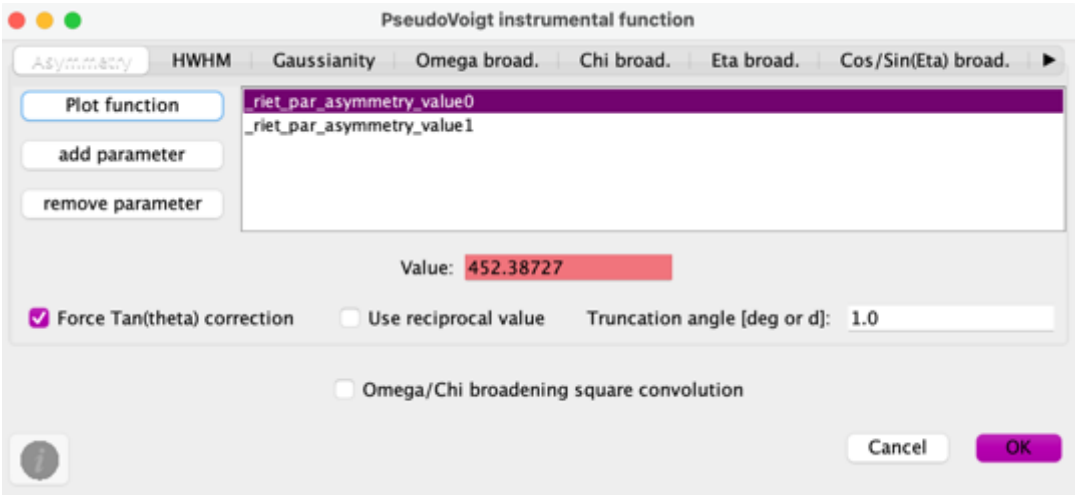
d) Source: select the appropriate source and click on options to put correct values



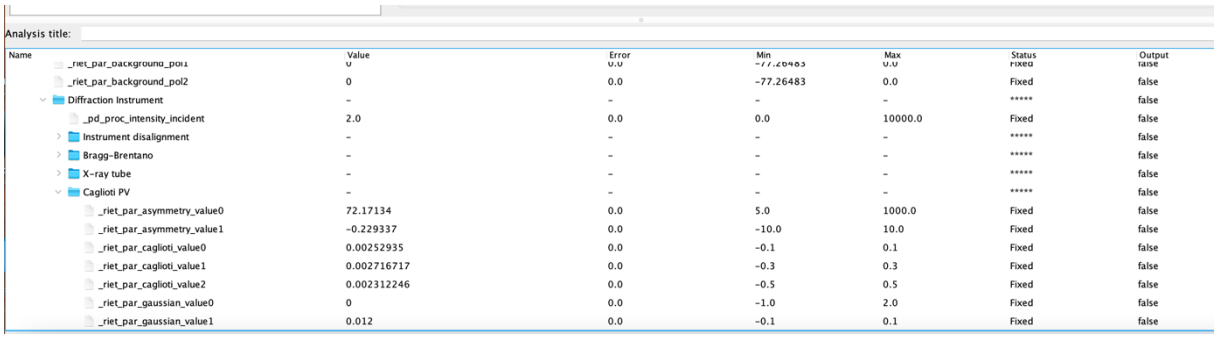
e) Detector: select the appropriate detector

f) Instrument broadening : select Caglioti PV and click on “Options”. See next part to see the strategy.

7- Instrument broadening



NB: These parameters are also available in the Table Tree bottom window of the main MAUD page



a) *Asymmetry*

_riet_part_assimetry_value0

_riet_part_assimetry_value1

Put the 2 parameters at 0

b) *HWHM*

_riet_part_caglioti_value0

_riet_part_caglioti_value1

_riet_part_caglioti_value3

Keep the default values

c) *Gaussianity*

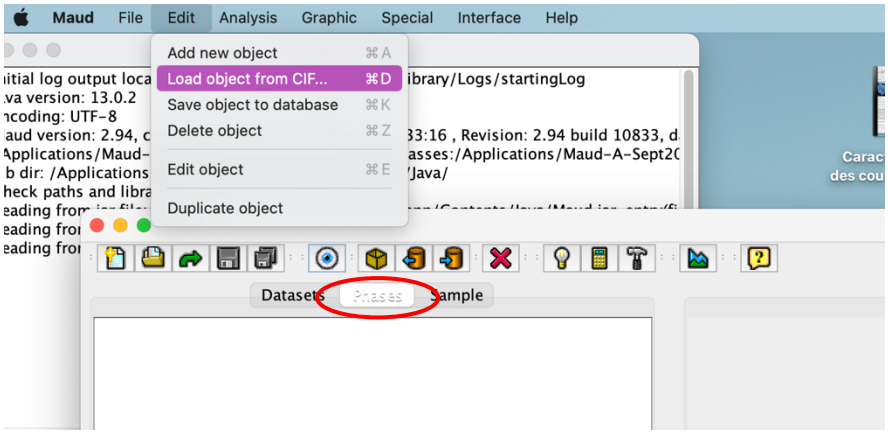
_riet_part_gaussian_value0

_riet_part_gaussian_value0

Put the 2 parameters at 0

8- Cif file

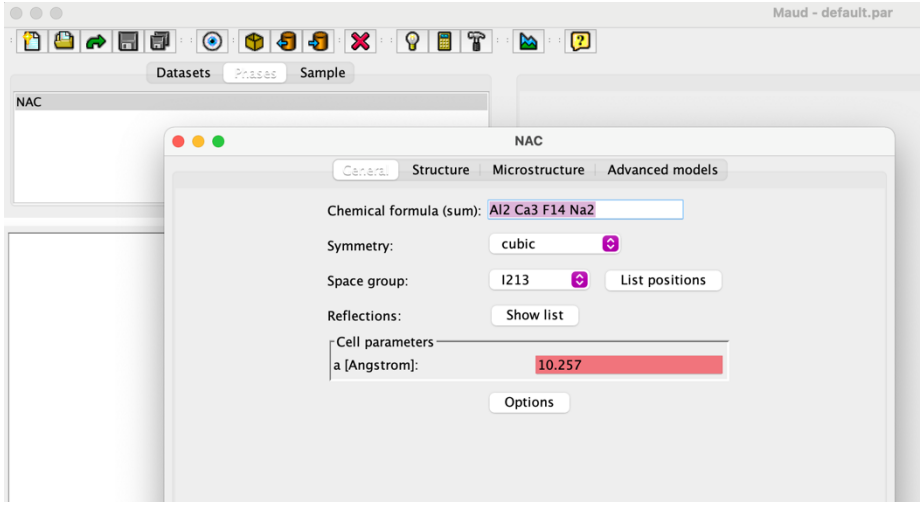
a- Principal page – Phases: edit -> load object from cif -> select your cif file(s)



Or click directly on 

b- Access to structure parameters, crystal size and texture model:

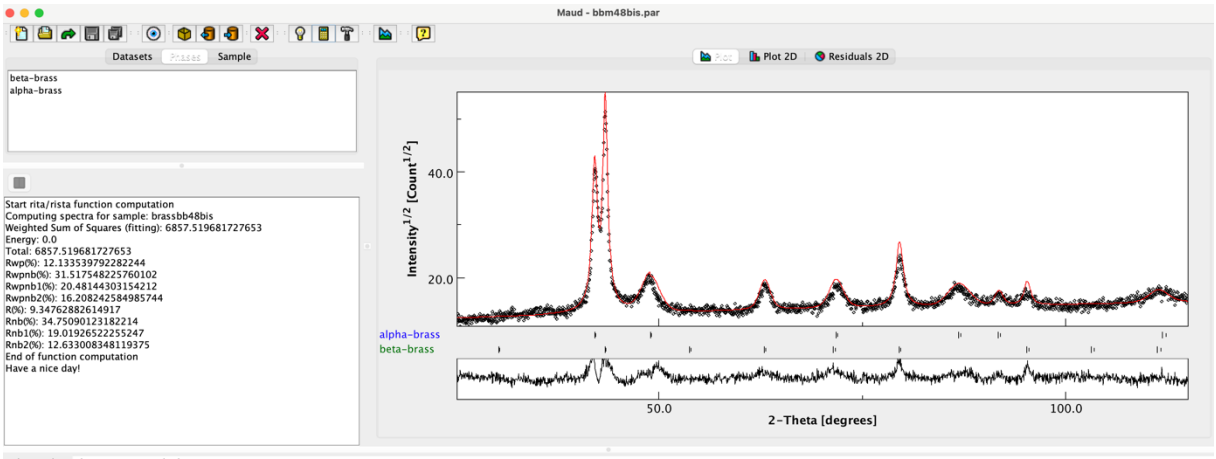
Select “phase” -> edit 



9- Compute spectra

Click on “analysis” -> compute spectra or directly on 

Now on the XRD pattern you see both measured (black) and calculated spectra (red)

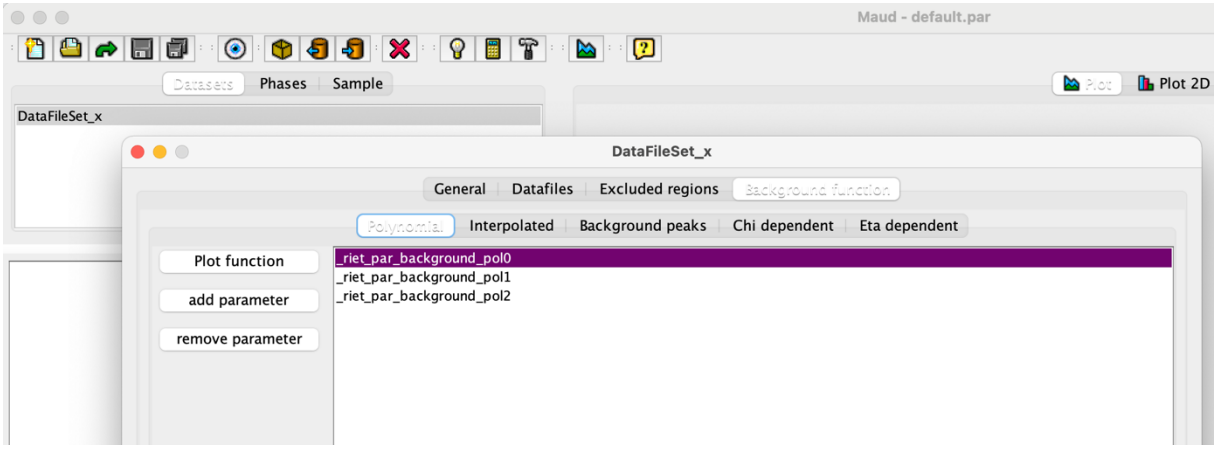


10- Background parameters

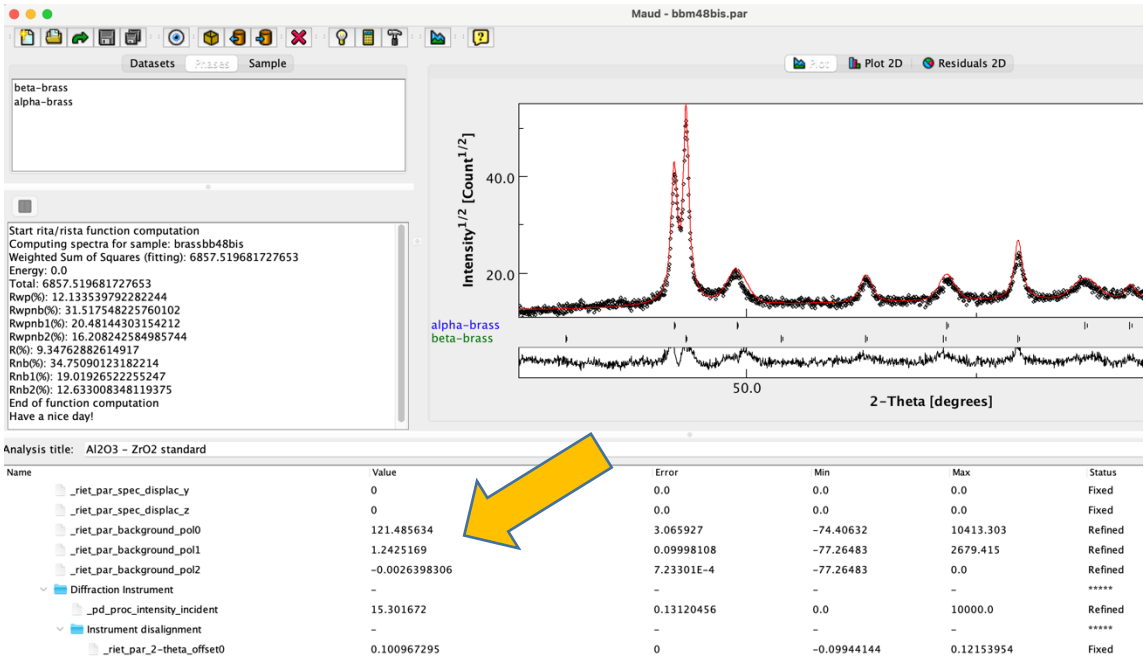
a- Background parameters access: 3 ways

(i) Analysis -> parameters list -> open successively sample_x, then Layer_1 and DataFileSet_x folders

(ii) Or Select DataFileSet_x ->  -> background function -> polynomia



(iii) Or Reach the “background parameters” in the Table Tree MAUD window



b- Adjust manually amap the 3 Background parameters to superpose measured and calculated XRD spectra

c- You can add more Background parameters using way (ii)

11- Incident intensity

Adjust manually the pd_proc_intensity_incident / Access via way (iii)

Then put “pd_proc_intensity_incident” in Refined mode

12- 2 theta offset

Put “_riet_par_2-theta_offset0” in Refined mode

13- Refinement

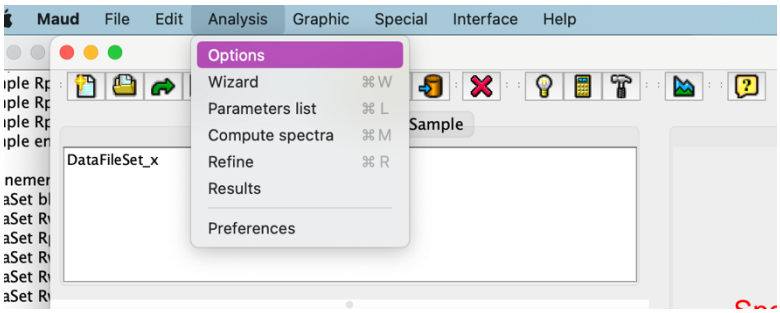
a- Verify that Background parameters, Incident intensity & the 2 theta offset are in “Refined” mode

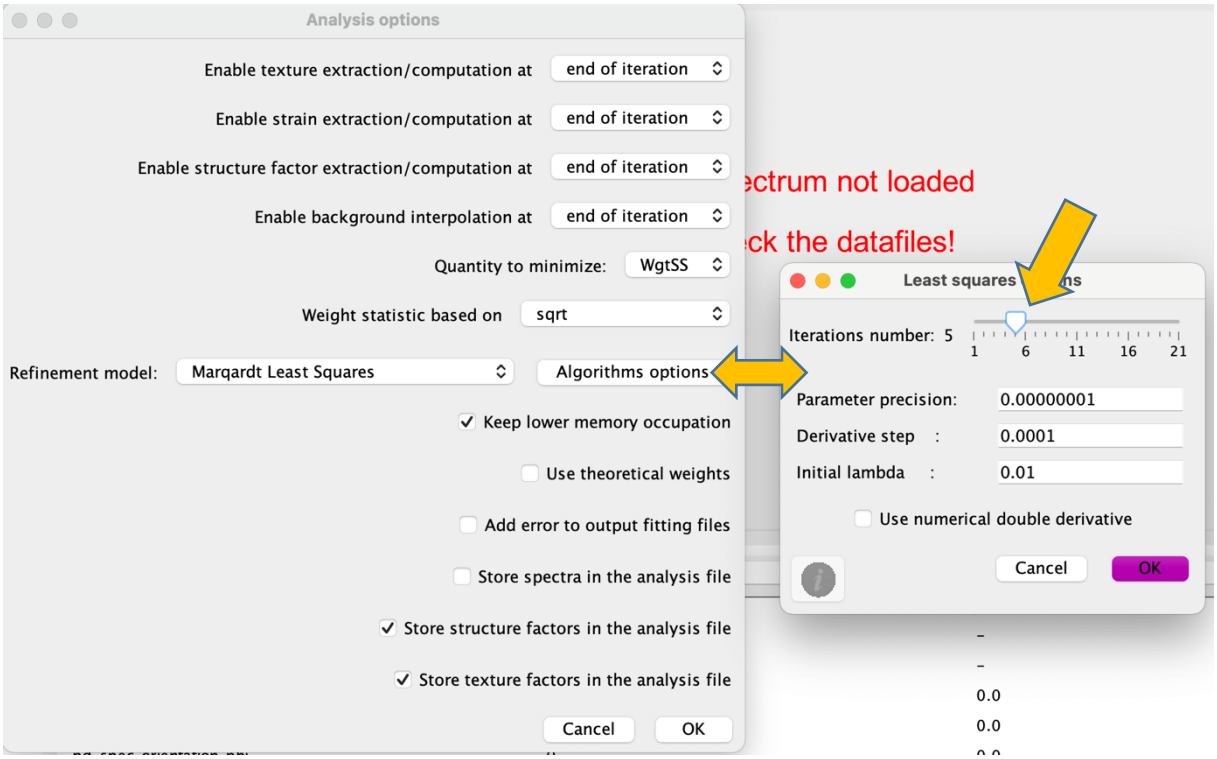
b- Then start a refinement process by Analysis -> Refine

or by clickling on  or hammer button (depends on the configuration)

c- To adjust the number of iterations :

Analysis -> Options -> Algorithms options -> select the value with the iteration item cursor





d- Refinement quality :

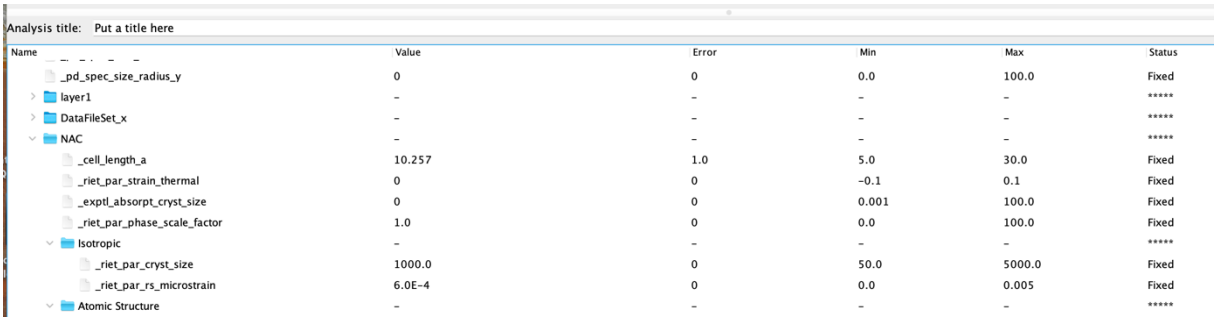
- observation of calculated and measured spectra superposition (zoom on background, ….)
- See the convergence of the reliability factors: sigma and R_{wp} mainly

e- adapt more Background parameters if necessary and restart a refinement

14- Structural part

"phase" -> edit 

or via the Table Tree MAUD window



a- Put *cell parameters* in Refined mode

b- Isotropic Parameters:

=> Adjust the crystallite size manually

=> riet_par_rs_microstrain = 1.0E-4

c- start a refinement

d- verify the cell parameters evolution

e- put *crystallite size* on Refined mode

f- start a refinement

g- verify the crystallite size value

h- put *microstrain* on Refined mode

i- start a refinement

j- verify the microstrain value

15- Caglioti parameters

If you don't have determined your instrumental parameters, you can refine the Caglioti values to calibrate your instrument using a measurement on a standard sample.

a) fix all refined parameters

(Background, Incident intensity, 2 theta offset, cell parameters, ……)

b) Refine only the caglioti values in this order:

_riet_par_caglioti_value1 -> 

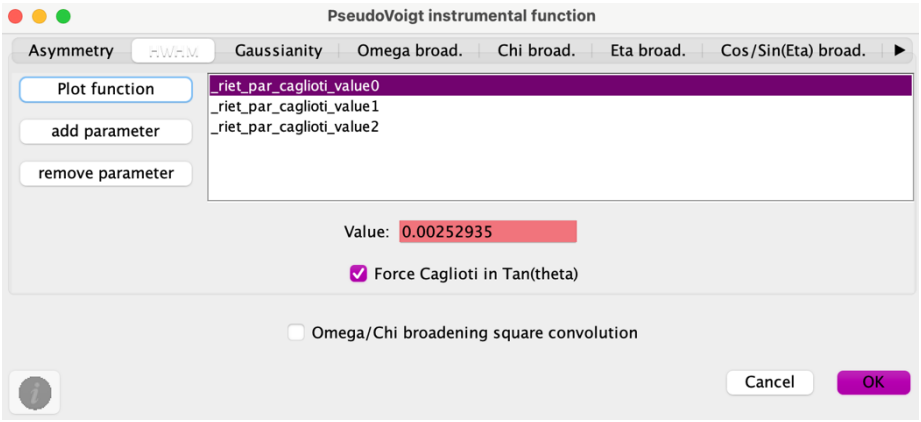
_riet_par_caglioti_value0 -> 

_riet_par_caglioti_value2 -> 

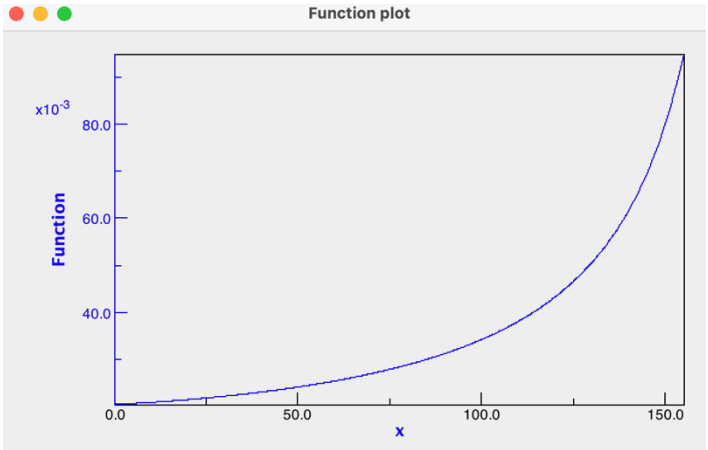
Caglioti PV	–	–	–	–	*****
_riet_par_asymmetry_value0	72.17134	0.0	5.0	1000.0	Fixed
_riet_par_asymmetry_value1	-0.229337	0.0	-10.0	10.0	Fixed
_riet_par_caglioti_value0	0.00252935	0.0	-0.1	0.1	Fixed
_riet_par_caglioti_value1	0.002716717	0.0	-0.3	0.3	Fixed
_riet_par_caglioti_value2	0.002312246	0.0	-0.5	0.5	Fixed
_riet_par_gaussian_value0	0	0.0	-1.0	2.0	Fixed
_riet_par_gaussian_value1	0.012	0.0	-0.1	0.1	Fixed
NAC	–	–	–	–	*****

To see if the parameter evolution is good plot the HWHM function:

Instrument -> edit -> HWHM -> plot function



The curve must have a 2nd order polynomia evolution with no singular points like this:



c) you can now release the background parameters, incident intensity, 2 theta offset, cell parameters, crystallite size and microstrains.

It is important to follow between each refinement the evolution of the reliability factors and the value of each refined parameters

16- Asymmetry

To improve your refinement, you can also refine the asymmetry parameters

17- Gaussianity

To improve your refinement, you can also refine the gaussianity parameters

Recommendation: *remember to save your work often*