Manual - Melt inclusion analysis

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Data import

Project mode

1. Below “Select mode”, click on “Melt inclusions”.

If you use an Agilent 7900s, Agilent 8900, a Finnigan MAT ELEMENT or a PerkinElmer Syngistix, select your instrument in the ICP-MS file setup. If you do not use one of these ICP-MS instruments, please create an ICP-MS file. For this purpose, click on “Setup” below “Define ICP-MS” and define the necessary information. Click then on “Save ICP-MS setup” and “Confirm settings”.

Now, we are able to import our measurement files that we got from the ICP-MS instrument.

Data import (standard files)

2. Below “Standard files”, click on “Add” and select all csv files that belong to your measured standard reference material (for example NIST 610 or Scapolite 17).

Data import (sample files)

3. Below “Sample files”, click on “Add” and select all csv files that belong to your measured sample files.

4. Click on “Settings” below “Melt inclusions”.

Main settings

Project information

5. (Optional: Add your name and the source ID below “Project information” to give your project some structure and context.)

The next steps are a recommendation and must not be done in the presented order. It is actually also not mandatory to do them at all but they can help to increase the user's efficiency and to reduce the number of clicks that would be necessary otherwise.

Standard reference material (SRM)

6. Below "Standard Reference Material (SRM)", select your standard reference material (for example NIST 610) after "Standard files" and "Isotopes". If we have measured more than one calibration material (for example also Scapolite 17 as a second standard reference material), we have to assign which standard files belong to NIST 610 and Scapolite 17. We have to do the same also for the isotopes. In our example, it would be useful to assign the halogen isotopes to Scapolite 17.

Mineral matrix (host)

7. Below "Matrix settings", select the quantification method that is used exclusively for the quantification of the mineral matrix.
   a. Internal standard:
      i. Click on "Setup" next to "Internal standard".
      ii. In the next sub window, the user can select between different methods how the concentration of the matrix-related internal standard can be estimated. If the user has measured values that come from an experiment, it is of course highly recommended to use them. For this purpose, click on "Custom data", then "Setup" and add your experimental values.
      If the concentration value has to be estimated, the user has to decide between "Oxide stoichiometry" or "Mineral stoichiometry". It is necessary here to click on "Setup" after selecting the desired method.
      iii. Example - "Oxide stoichiometry": the concentration value will be estimated based on the cation amount. For this purpose, select first a suitable oxide on the right side below "Default settings". In the next step, define the oxide amount (for example 100 for 100 % SiO2 if the inclusions are embedded into a quartz matrix or 67.39 if the inclusions are embedded into an albite matrix). Select now a suitable isotope in the option menu below. It will act as a matrix-related internal standard. Note: values in the entry fields will be updated after pressing Enter.
      iv. Example - "Mineral stoichiometry": the workflow here is actually very similar. The only difference here is that the user has to select a mineral instead of an oxide and that it is not possible to define the mineral amount. It

will be assumed that the mineral matrix around the inclusions is always composed of 100 % of the selected mineral.

b. 100 wt.% oxides:
   i. Click on “Setup” next to “100 wt.% "oxides".
   ii. This estimation method is based on the assumption that the mineral matrix is composed of x wt.% oxides (the default value for x is 100). This value should be changed if the user knows that the matrix contains volatiles like water or CO2. The first step is to activate the checkbox “Matrix/Sample” below “Run 100 wt.% oxides calculation for”.
   iii. The next step is very important and directly controls the accuracy of the calculated values. Now, the user has to select all (significant) oxides that describe the mineral matrix. It is possible to select or deselect all oxides with one click but it is also possible to select manually every single oxide or to select only oxides that are related to the rock-forming elements.
   iv. If Fe or Mn are part of the chemical composition, it is necessary to define the ratio between FeO and FeO+Fe2O3 and MnO and MnO+Mn2O3.
   v. The last two steps are the selection of a reference element (or isotope) that is used for the calculation of needed intensity ratios and to change eventually the amount of the solid mass. Note: it is not necessary here to confirm the values by pressing Enter.

Quantification method

8. Below “Quantification method”, select the method that is used for the quantification of the inclusion.
   a. Matrix-only tracer (SILLS)
      i. Click on “Setup” next to the option menu.
      ii. If the mineral matrix was already defined before, it is not necessary to change any entry field values or option menu selections. The only two decisions that should be made here are about the choice of the equation that is used for the calculation of the background- and matrix-corrected inclusion intensity as well as of the inclusion concentration. You can find further information about the equations on the ReadTheDocs theory page of PySILLS.
   b. Second internal standard (SILLS)
      i. Click on “Setup” next to the option menu.
      ii. This method uses a second internal standard for the calculation of the inclusion concentration. For this

purpose, it is necessary to select which isotope will act as this second internal standard and to define its concentration value.

iii. As for the matrix-only tracer, it is also possible here to decide about the choice of the equation that is used for the calculation of the background- and matrix-corrected inclusion intensity as well as of the inclusion concentration. You can find further information about the equations on the ReadTheDocs theory page of PySILLS.

c. Geometric approach (Halter et al., 2002)

i. Click on "Setup" next to the option menu.

ii. Define the geometric and physical properties that are necessary for this quantification method.

iii. Select an isotope that can be called a matrix-only tracer. It is necessary for the calculation of the background- and matrix-corrected inclusion intensity.

iv. Note: the method of Halter et al. uses an iterative approach for the calculation of the inclusion concentration. You can find further information about the equations on the ReadTheDocs theory page of PySILLS.

d. Geometric approach (Borisova et al., 2021)

i. Click on "Setup" next to the option menu.

ii. Define the geometric and physical properties that are necessary for this quantification method.

iii. Select an isotope that can be called a matrix-only tracer. It is necessary for the calculation of the background- and matrix-corrected inclusion intensity.

iv. Note: the used equation for the calculation of the inclusion concentration was modified to keep consistency with the other quantification methods. You can find further information about the equations on the ReadTheDocs theory page of PySILLS.

Assemblage setup

9. (Optional: Below "Assemblage settings", select a letter that will change the sample ID of all your sample files based on your choice.)

Dwell time settings

10. The default value is 0.002 s. It can be changed for every isotope in a specific sub window that will appear after clicking on "Setup" below "Dwell time settings".

Default calculation interval (background)

11. Below "Default time interval (background), it is possible to set a default calculation interval manually or automatically. The first option

is useful if the user knows that the laser started always after 40 s for example and if the transition zone between background and signal is not always perfect. In this case, the user should define a start value (for example 5) and an end value (for example 35). The input values are only numbers without units. Now, PySILLS will search for every file which time value is the closest neighbor to the user-defined start and end value.
As an alternative to this method, the user can click on “Run” after “Auto-detection” and PySILLS will define a background calculation interval for every file.

Default calculation interval (signal - only standard files)

12. Below “Default time interval (matrix), it is possible to set a default calculation interval manually or automatically. The first option is useful if the user knows that the laser started always after 40 s and ended always after 90 s for example. In this case, the user can define a start value (for example 45) and an end value (for example 85). The input values are only numbers without units. Now, PySILLS will search for every file which time value is the closest neighbor to the user-defined start and end value.
As an alternative to this method, the user can click on “Run” after “Auto-detection” and PySILLS will define a signal calculation interval for every standard file.

Outlier detection and spike elimination

13. (Optional: Below “Spike elimination”, select the detection algorithm that will be used for the outlier detection. The significance level defines which error probability will be accepted. The default value is 5 % which results in an input value of 0.05. The threshold defines the minimum value that will be considered for the outlier detection algorithm. If the checkbox “Exclude inclusion” is activated, outliers that lie within the inclusion calculation interval, can be detected but keep its unsmoothed value. If the checkbox “Check only inclusion” is activated, the user can check only the outliers that lie within the inclusion calculation window. This check-up is possible after a click on “Check” behind “Standard/Sample files”.)

File setup (standard files)

Calculation interval (background)

14. A missing or additional calculation window can be added by clicking the limits of the interval on the diagram. For this purpose, it is necessary that the radio button “Background” was activated before. As an alternative, it is also possible to type the start and end value in the

entry fields. Each value has to be confirmed by pressing Enter. If an interval has to be removed, it is necessary to activate the related radio button, here “Background”, and to click then on the specific entry in the list. Now, a click on “Remove” should delete the selected interval.

Calculation interval (signal)

15. A missing or additional calculation window can be added by clicking the limits of the interval on the diagram. For this purpose, it is necessary that the radio button “Matrix” was activated before. As an alternative, it is also possible to type the start and end value in the entry fields. Each value has to be confirmed by pressing Enter. If an interval has to be removed, it is necessary to activate the related radio button, here “Matrix”, and to click then on the specific entry in the list. Now, a click on “Remove” should delete the selected interval.

File setup (sample files)

Calculation interval (background)

16. A missing or additional calculation window can be added by clicking the limits of the interval on the diagram. For this purpose, it is necessary that the radio button “Background” was activated before. As an alternative, it is also possible to type the start and end value in the entry fields. Each value has to be confirmed by pressing Enter. If an interval has to be removed, it is necessary to activate the related radio button, here “Background”, and to click then on the specific entry in the list. Now, a click on “Remove” should delete the selected interval.

Calculation interval (inclusion)

17. A missing or additional calculation window can be added by clicking the limits of the interval on the diagram. For this purpose, it is necessary that the radio button “Inclusion” was activated before. As an alternative, it is also possible to type the start and end value in the entry fields. Each value has to be confirmed by pressing Enter. If an interval has to be removed, it is necessary to activate the related radio button, here “Inclusion”, and to click then on the specific entry in the list. Now, a click on “Remove” should delete the selected interval.

Calculation interval (matrix)

18. A missing or additional calculation window can be added by clicking the limits of the interval on the diagram. For this purpose, it is necessary that the radio button “Matrix” was activated before. As an alternative, it is also possible to type the start and end value in

the entry fields. Each value has to be confirmed by pressing Enter. If an interval has to be removed, it is necessary to activate the related radio button, here “Matrix”, and to click then on the specific entry in the list. Now, a click on “Remove” should delete the selected interval.

Advanced setup

When all calculation intervals (background, matrix, inclusion) were defined as well as matrix-related internal standards and its concentration values, it is possible to do the next steps concerning the inclusion setup.

Inclusion settings

19. Below “Inclusion settings”, select the option that has to be used for the definition of the internal standard concentration and click then on “Setup”.
    a. 100 wt.% oxides:
        i. Click on “Setup” next to “100 wt.% "oxides".
        ii. This estimation method is based on the assumption that the melt inclusion is composed of x wt.% oxides (the default value for x is 100). This value should be changed if the user knows that the inclusion contains volatiles like water or CO2. The first step is to activate the checkbox “Inclusion” below “Run 100 wt.% oxides calculation for”.
        iii. The next step is very important and directly controls the accuracy of the calculated values. Now, the user has to select all (significant) oxides that describe the melt inclusion. It is possible to select or deselect all oxides with one click but it is also possible to select manually every single oxide or to select only oxides that are related to the rock-forming elements.
        iv. If Fe or Mn are part of the chemical composition, it is necessary to define the ratio between FeO and FeO+Fe2O3 and MnO and MnO+Mn2O3.
        v. The last two steps are the selection of a reference element (or isotope) that is used for the calculation of needed intensity ratios and to change eventually the amount of the solid mass. Note: it is not necessary here to confirm the values by pressing Enter.
        vi. Note: it is currently not possible to define two different sets of oxides for the matrix and the inclusion if this quantification method will be used for both applications. We can imagine that this feature will be

available in a next update but it should not make a difference. It is actually better to select one oxide more than one less.

b. Custom data
   i. Custom data can be defined manually or imported from a csv file. A demo file that can be used for this purpose is in the demo_files folder of the PySILLS directory.
   ii. As for the quantification of the mineral matrix, it is also necessary to define the internal standard and its concentration value. This can be done by using the default variables or manually for every file.

c. External calculation
   i. Select all parameters that have to be included to the exported file. Click then on “Export data”.
   ii. Use the exported data for your external calculation.
   iii. Import your results from a csv file or set the concentration values manually for every file.

Results and data export

Final results

20. When all settings are defined, the user can run a final calculation and display its results by clicking on “Results” in the PySILLS main window.

Sensitivity drift

21. (Optional: blablabla)

Data export

22. In order to be able to export the calculated results, click on “Export results”, select the report style and click on “Export results”.

Troubleshooting and frequently asked questions (FAQ)

Coming soon

Further information

- PySILLS manual - mineral analysis
- PySILLS manual - fluid inclusion analysis
- PySILLS manual - melt inclusion analysis
- PySILLS frequently asked questions (FAQ)
- PySILLS on GitHub

- PySILLS on ReadTheDocs
- PySILLS on YouTube