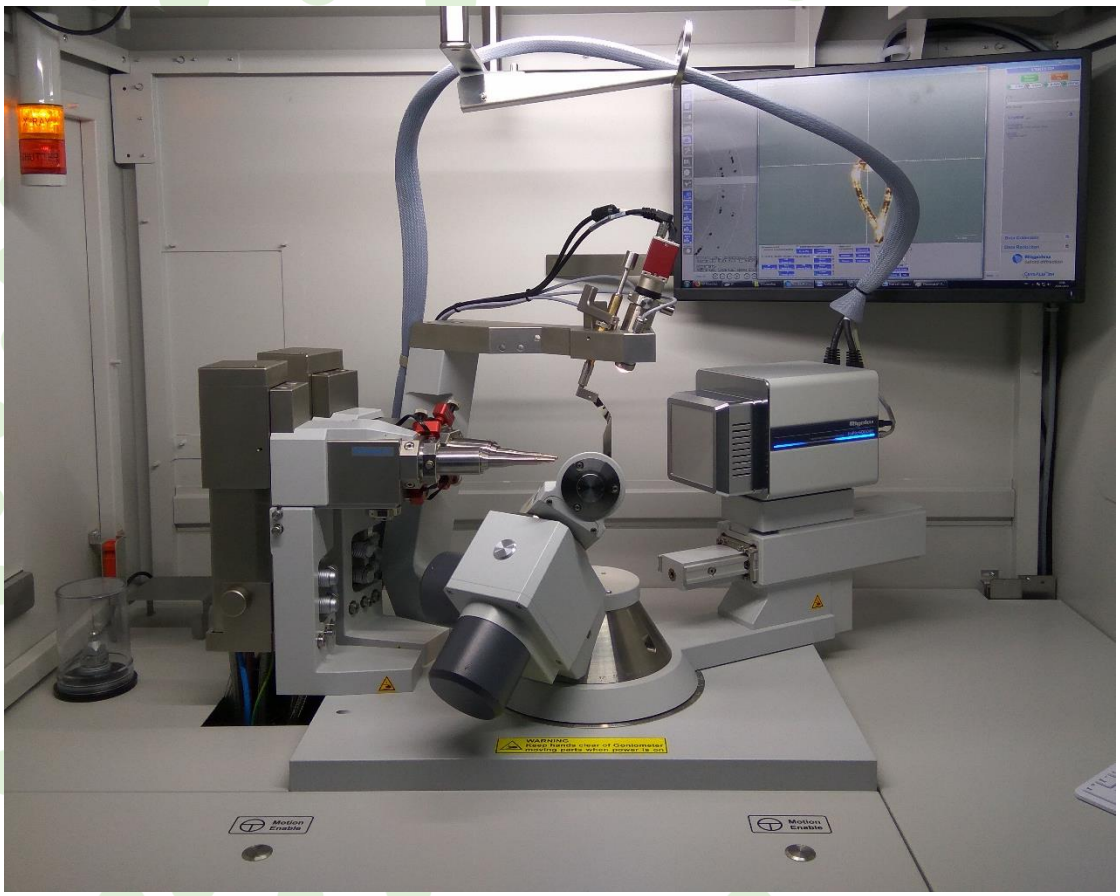
UiO : Naturhistorisk museum

Rigaku XtaLAB Synergy-S single-crystal X-ray diffractometer

User instructions



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Geo Lab

Department of technical and scientific conservation |
Seksjon for konservering og forskningsteknikk

U562 – Analyselaboratorie SEM, XRD, CT

- **Scanning electron microscopy**
  Hitachi S-3600N scanning electron microscope (SEM)

- **X-ray diffraction**
  Siemens D5005 powder X-ray diffractometer (PXRD)
  Rigaku Dual Beam Synergy-S single-crystal X-ray diffractometer (SXRD)

- **Computed tomography**
  Nikon XT H225 ST micro computed tomograph (micro-CT)

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** If you call from the laboratory telephone, digit the last 5 digits of the number only.*
Exp: Nélia Castro - 51641

Contents

1. Introduction

The Geo Lab at the Natural History Museum, University of Oslo, is equipped with a Rigaku XtaLAB Synergy-S X-ray diffractometer, that is used mainly for single-crystal X-ray diffraction and subsequent crystal structure refinements.

The instrument is equipped with a Hybrid Photon Counting Detector (HyPix-6000HE) and two PhotonJet-S microfocus sealed tube X-ray sources (Cu and Mo radiation).

In addition to the single-crystal mode, the instrument can also run in Gandolfi mode for phase identification of small grains or powders.

The Mo radiation is mainly used in for experiment on single-crystals in order to collect data for crystal structure refinements. However, if the investigated material contains only light elements or very small grains are available the Cu radiation can also provide high quality data for a structure refinement.

Operation of this instrument is restricted to trained personnel familiar with the analyze system as well as the product documentation, general safety precautions, and laboratory rules. New users need to undergo a training period before they are allowed to work with this equipment independently. Contact the laboratory manager or deputy if you want to be trained to operate this instrument independently.

2. Hygiene and safety in the laboratory

2.1. General safety rules

All equipment in this laboratory uses **high-voltage**.

Laboratory users are **NOT allowed to**:

- Walk in the back of the equipment
- Touch equipment or computer cables
- Remove protection covers of the equipment
- Touch the interior of the equipment

Not attending these rules can lead to:

- Electrocution
- Burns

2.2. Emergency contacts

Fire: 110

Ambulance: 113

Police: 112

In case of unexpected incidents **during the use of the equipment (for example if an alarm goes off),** contact immediately **one of the persons listed below.**

Lab manager, Nélia Castro

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tlf: 228 51622 e-mail: henrik.friis@nhm.uio.no

Scientist, Fabrice Dal Bo

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2.3. Other rules

- The **computers** in this lab are **not for personal use** (e.g. e-mail or internet search); their use is limited to data collection and data processing.
- Users are **only allowed to use the computer(s) of the instrument** they have booked.
- Users are **not allowed** to **connect any device (including USB devices) to the computers** in this lab.
- The use of **laptops** in the laboratory **shall be avoided**. Users are welcome to use the common area outside the laboratory to work in between analysis (computers available for UiO users).
- If the users need to connect a personal device to the electricity (e.g. laptop, mobile, etc.), **the electricity plugs available in the column in the middle of the room** must be used.
- **Food or drinks** are **not allowed** in the lab.

2.4. Safety summary for Rigaku XtaLAB Synergy SXRD.

Personal injuries can result from the inappropriate use of the instrument. The following warnings apply to the use of the Rigaku XtaLAB Synergy SXRD:

WARNING	Consequences	Safety measures
Beware of high voltage!	Electric shock can cause fatal or serious injury.	DO NOT remove the protective covers from the instrument. Do not disconnect the high-voltage cable.
Beware of high temperature!	Can cause burns.	DO NOT remove the protective covers from the instrument.
Beware of radiation!	Exposure to radiation.	SXRD is serviced and safety checked regularly. The sample chamber is shield to stop the scape of any radiation and interlocked, meaning that cannot be opened when in use. The operator will not be exposed to X-rays or any radiation when the machine is in working under normal working mode. DO NOT deviate from normal working mode. NEVER try to override the interlock.

The following cautions should be taken when using the Rigaku XtaLAB Synergy SXRD:

Action	Consequences	Safety measures
Prolonged use.	Eye strain, tiredness, and musculoskeletal strain or injury.	Take regular breaks (about 10 to 15 minutes break per hour during operation). Adjust height of the seat, so that the back and neck and upright.
Opening and closing the specimen chamber to exchange sample.	Personal injuries caused by jamming your hand.	Handle the specimen chamber with care.

3. Configuration

3.1. Appearance of Rigaku XtaLAB Synergy diffractometer

Figure 1 shows a general view of the cabinet of the Rigaku XtaLAB Synergy S. The instrument is connected to a computer that contains all the software necessary for acquisition and data treatment.

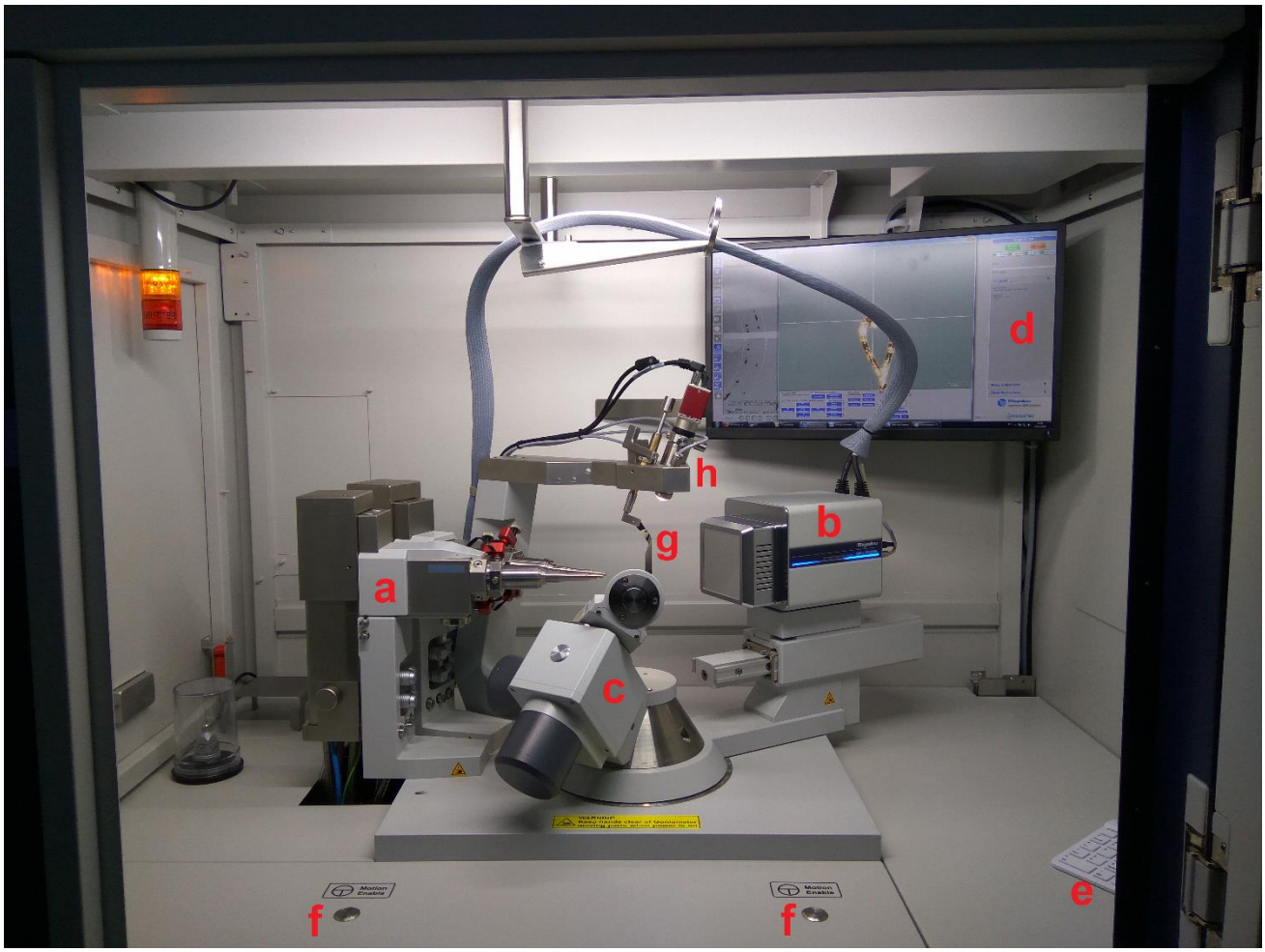


Fig. 1. General view of the inside of the cabinet of the Rigaku XtaLAB Synergy diffractometer with: (a) X-ray sources; (b) Hybrid Photon Counting Detector; (c) goniometer arm and head; (d) centering screen; (e) numeric pad to run the goniometer movements; (f) Motion Enable buttons to allow the goniometer and detector movements; (g) beam stops; (h) camera.

The diffractometer consists of two microfocus X-ray sources (Fig. 2), a goniometer arm and head, and a photon counting detector (Fig. 3) mounted on a two independent moving arms. The screen inside the cabinet is used to center the sample.

The cabinet's door has to be close when the arms are moving, otherwise the user has to press **continuously** and **simultaneously** the two *Motion Enable* buttons (Fig. 1f).

The door has to be close during data collection, otherwise the system will enter in security mode.

Fig. 2. Detailed view of the Cu (first plan) and Mo (second plan) PhotonJet-S microfocus sealed tube X-ray sources.

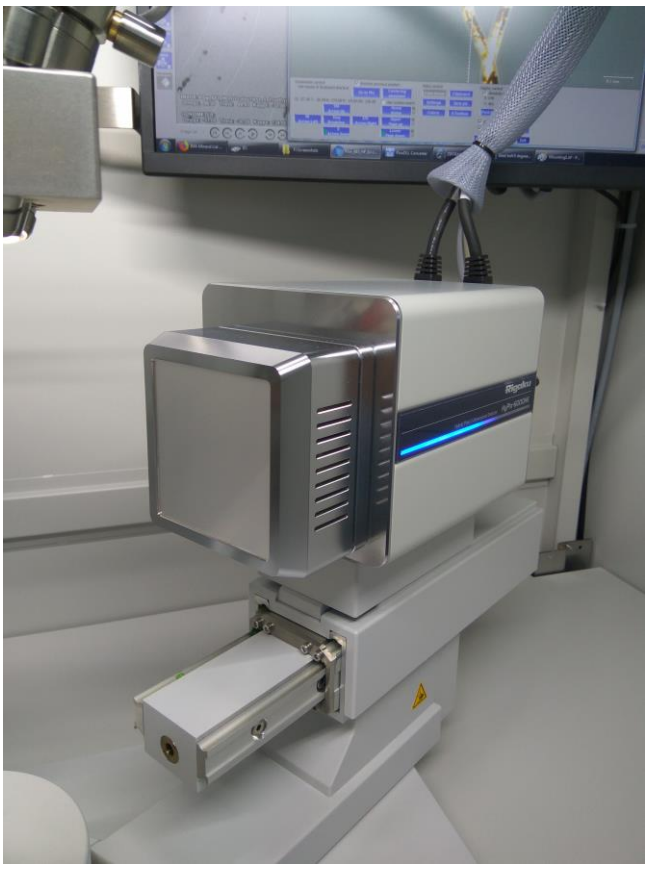
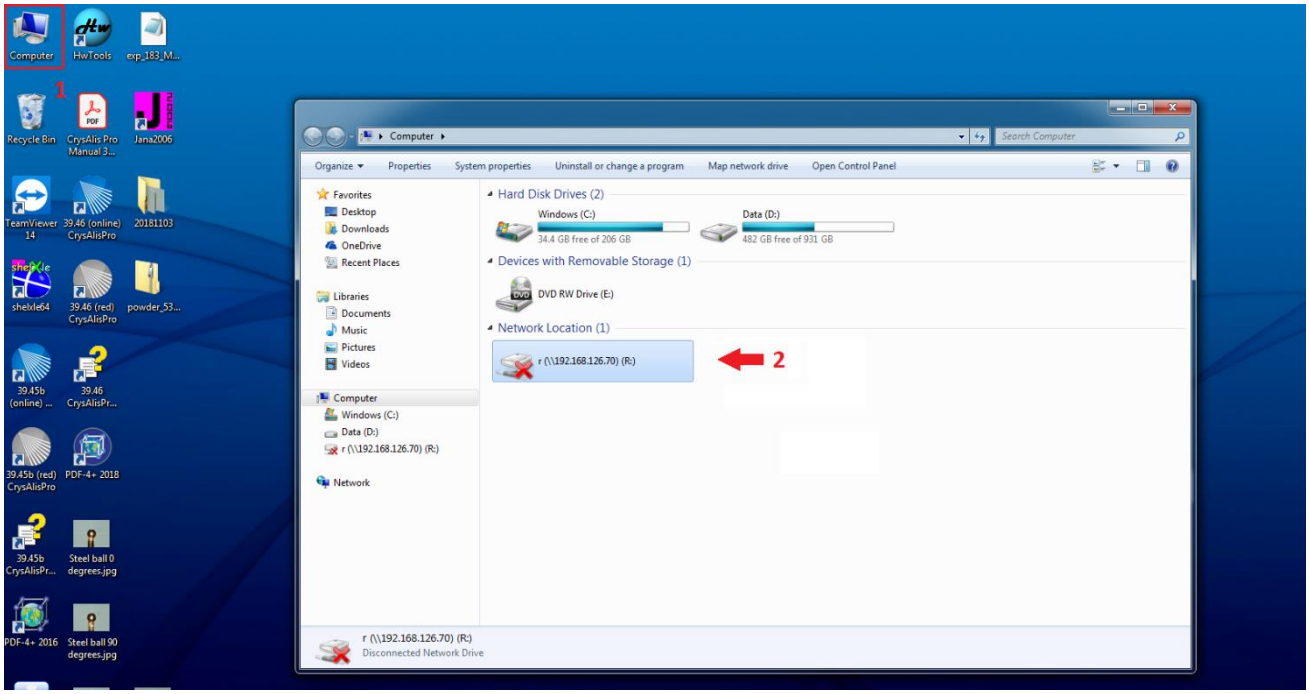


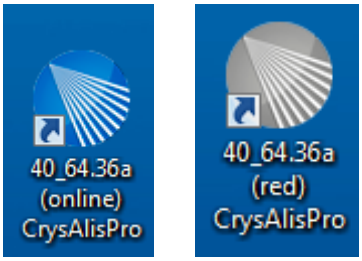
Fig. 3. Detailed view of the Hybrid Photon Counting Detector.

3.2. Software

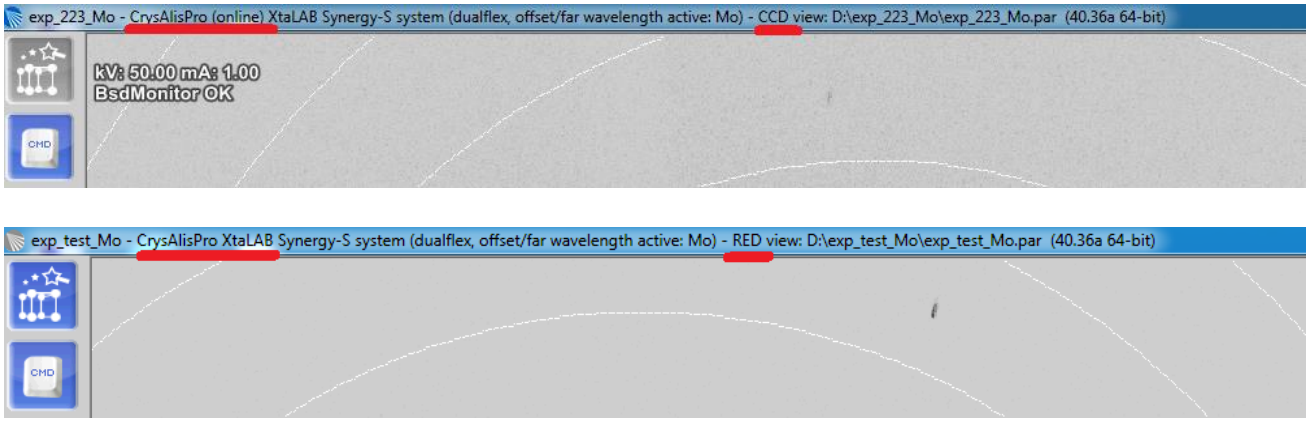
If the computer has been turned off you need to reconnect it to the network. To do so, hit the *Computer* shortcut on the desktop (1), and then hit the button below *Network Location* (\\192.168.126.70 (:R))(2).
A window will pop up whereby the *User Name* is **Rigaku**, and the *Password* field has to stay blank, in order to connect to the network to operate the diffractometer.



The CrysAlisPro software package is used (i) to control the operation of the diffractometer and for data collection (online (CCD) view, left shortcut), and (ii) for the data reduction and processing (red view, right shortcut):



NOTE: New versions of CrysAlis Pro are installed regularly, but the shortcuts from the desktop are always the currently used version. Users have to be careful as both parts of the CrysAlisPro software package have exactly the same layout. The easiest way to distinguish them is to verify the name in the upper part of the window, as highlighted below:



4. Collecting data in single-crystal mode

4.1. Switching from Cu to Mo radiation and *vice versa*.

The orange button in Fig. 4A displays the currently engaged X-ray source. When the diffractometer is not collecting any data it's possible to switch from one radiation wavelength to another by clicking on the orange button in the upper right corner in the CrysAlisPro (online) software (*X-ray Cu* or *X-ray Mo*) (Fig. 4A). If the tube is under high voltage the generator will automatically ramp down the current and voltage to 0, and then increase progressively the current and voltage of the other X-ray tube (Fig. 4B). NOTE that is not possible to mount or center the crystal during this operation and the cabinet door has to be closed.

Fig. 4. Control panel to switch between Cu and Mo X-ray radiation (A), and indication of the X-ray tube's voltage (B).

4.2. Mounting and centering the crystal.

Several steps are needed for mounting and centering the sample. These are described below:

1) By pressing *F12* on the keyboard the operator will get access the mounting menu.

Before opening the cabinet's door, the goniometer movements have to be completed. Otherwise, the goniometer will stop to avoid any collisions. To restart the movements you will have to manually press the **two** *Motion Enable* buttons located inside the cabinet (Fig. 1f).

2) Check that the goniometer is on the *0/Arrow Down* (Fig. 5A) and *Lower*/*Page Down* positions (Fig. 5B). If that's not the case, just press the corresponding blue buttons to put the goniometer in the correct position. If the buttons A-E in Fig. 5 are grey and not blue, it means the goniometer and detector are not yet in the mounting position.

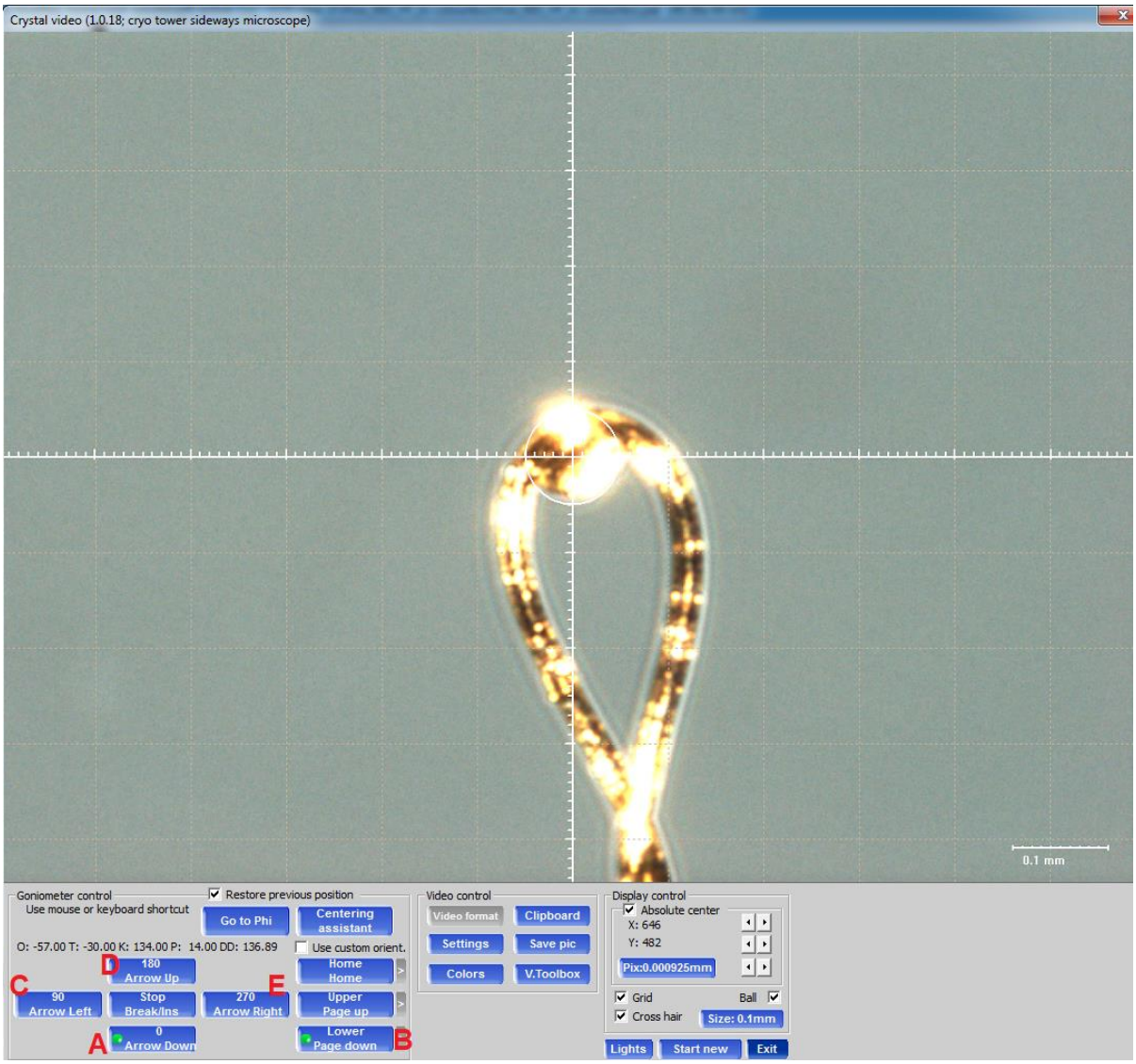


Fig. 5. Mounting and centering menu, with the *Arrow down* (A), *Lower Page down* (B) buttons, 90° rotation (C), 180° rotation (D), and 270° rotation (E) buttons.

3) Remove the goniometer head by unscrewing the ring (counter clockwise), while holding onto the goniometer head. Place your loop on the magnet and return the goniometer head back to the moving arm and tighten the ring by tuning it clockwise until fasten. The loop is secured on the goniometer head by magnetic force. There is only one correct way to put back the goniometer back, look for the **two** stops to find the good orientation (Fig. 6).

NOTE: care must be taken to not touch the front of the detector or the beam stop when removing and placing the goniometer head on the arm.

Fig. 6. View of the back of the goniometer head.

4) The centering required that you turn with the five different knobs placed on the goniometer head. For centering the crystal, guided by the centering screen, you may have to first adjust the height of the loop by using to top knob (Fig. 7A), and then use the lower knob (Fig. 7B) to move towards the right or left.

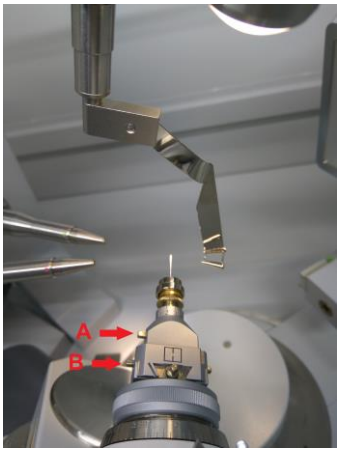


Fig. 7. Goniometer head in position for the centering of the sample.

5) When your sample is centered, hit the button *90/Arrow Left* (Fig. 5C or press the number *4* on the numeric keyboard located inside the cabinet) (NOTE: the *NumLock* button of the computer's keyboard has to be deactivated). Center your crystal by using the knob on the **left** of the goniometer head. Repeat this operation in the positions *180/Arrow Up* (Fig. 5D, or press number *8* on the numeric keyboard inside the cabinet) and *270/Arrow Right* (Fig. 5E, or press the number *6* on the numeric keyboard).

6) Hit the button *0/Arrow Down* (Fig. 5A or press the number *2* on the numeric keyboard) to put back the goniometer head in the starting position. Follow your sample on the centering screen (Fig. 1d) to confirm that the crystal is centered. When satisfied with the centering, remove the Pad and any other items in the cabinet out of the path of the goniometer and detector to avoid collision during the data collection.

7) Close the cabinet's door and hit the button *Doors OK* located on the right of the diffractometer (Fig. 8), and then click on *Exit* to leave the mounting menu (Fig. 5).

Fig. 8. Safety System control panel with the highlighted *Doors OK* button.

8) To remove your sample you have to repeat the same operations as for the mounting procedure. Pressing *F12* on the keyboard to get access the mounting menu and check that the goniometer is going to the *0/Arrow Down* (Fig. 5A) and *Lower*/*Page Down* positions (Fig. 5B)

4.3. Measuring a pre-experiment (single crystal).

A pre-experiment is first performed in order to check the quality of the investigated single-crystal, *i.e*. does it diffract, is it a single crystal or multiple grains, etc. The data obtained from the pre-experiment (symmetry of the crystal, unit-cell parameters, intensity of the diffracted peaks) will be used by the software to establish a strategy to measure a complete data set.

1) When you have chosen the X-ray radiation and centered your sample, press the *START/STOP* button in the upper right corner of the CrysAlis Pro software (Fig. 9A) and then press *Start New* on the window that just popped up (Fig. 9B).

2) Give a name to your sample (Fig. 9C), try to follow the numbering (ExpXXX_Mo or ExpXXX_Cu) by checking the instrument notebook, and then select the exposure time you want (Fig. 9D) (NOTE: a total time of 4 minutes is usually enough if the crystals is >100 µm in size. Very tiny crystal can require a total time of 15 min, especially with the Mo radiation).
Press the button *Pre-Exp.* to start data collection (Fig. 9E).

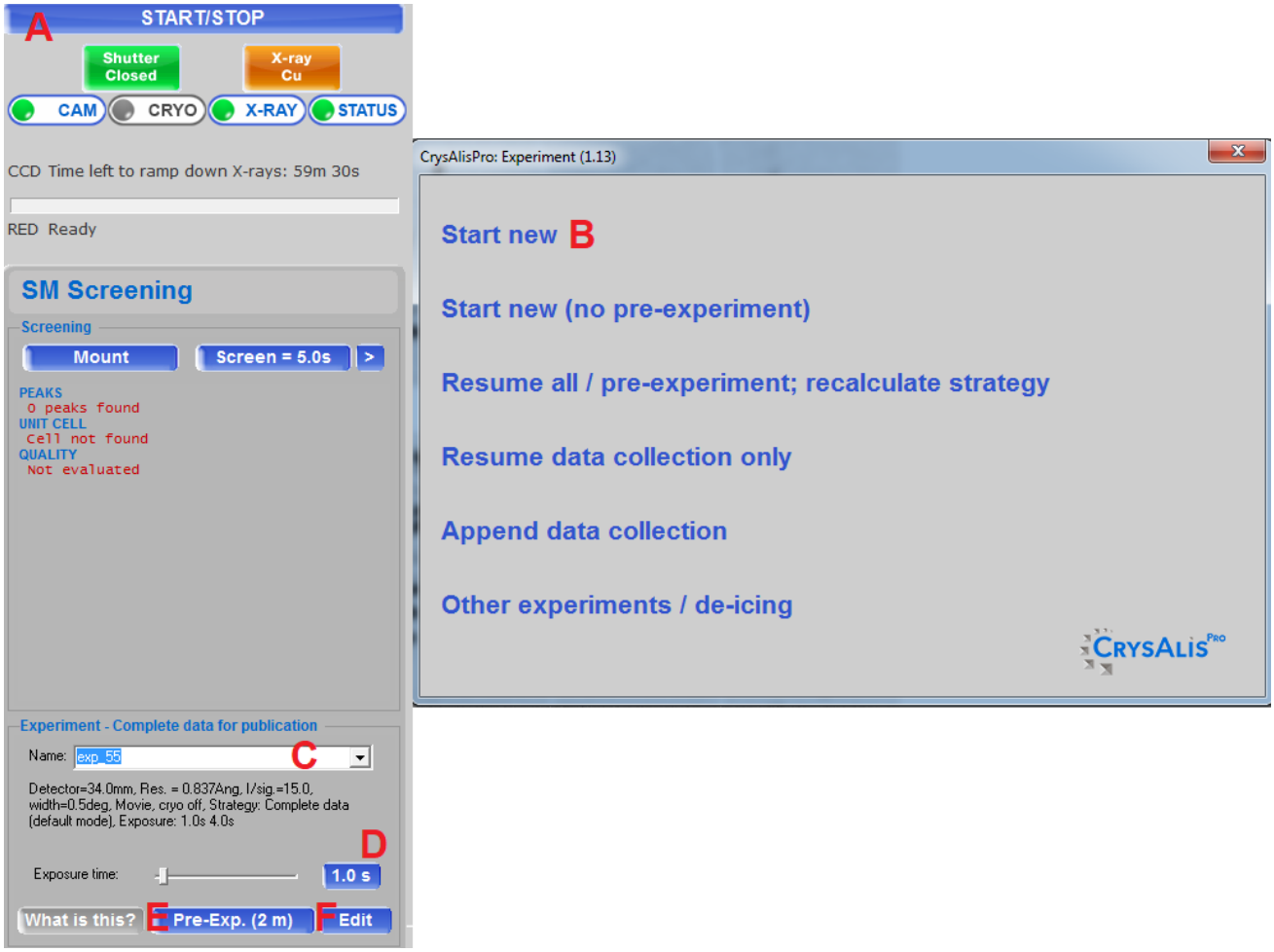


Fig. 9. Starting menu to run a pre-experiment with minimal options.

3) You can also press the *Edit* button (Fig. 9F) if you want to add more information as for example the expected chemical formula of your compound, some comments and sample description (colors and shape). Press the button *Exit & Start pre-experiment* to start the data collection (Fig. 10A).
You can start a pre-experiment with the same name as the previous one by hitting the button *Clear path* (Fig. 10B). This is used if the pre-experiment reveals that your crystal is not suitable for full structure solution.

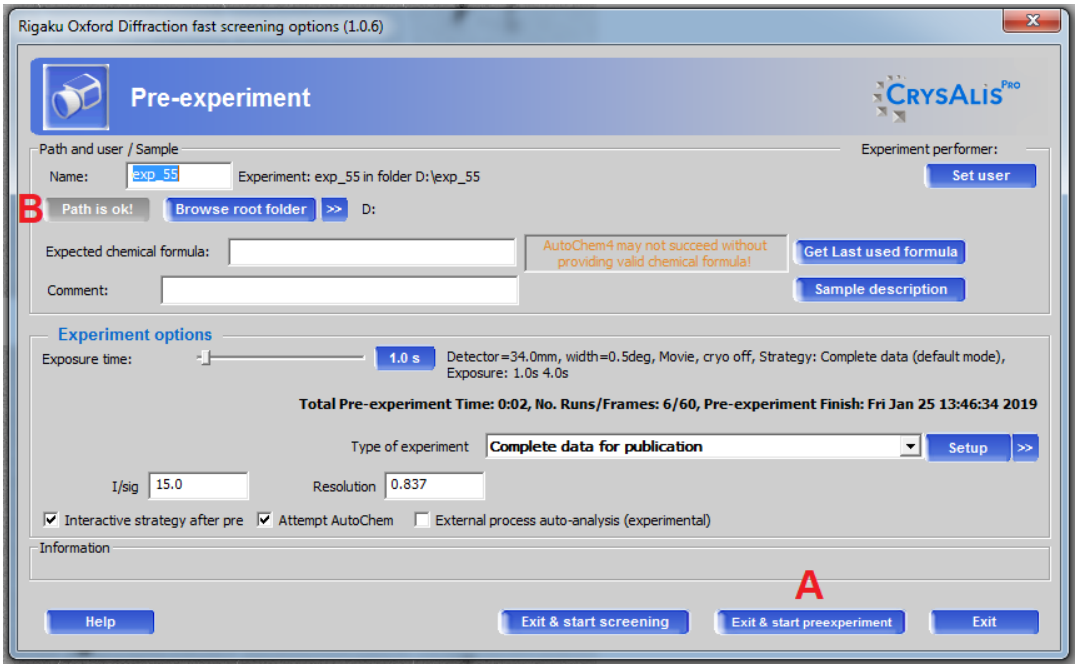


Fig. 10. Starting menu to run a pre-experiment with detailed options.

4.4. Running a complete experiment.

When the pre-experiment is over a window (Fig. 11) will pop up in order to run a complete experiment. On this window, you can see the unit-cell parameters (Fig. 11A), as well as the number of collected reflections and the indexation ratio (Fig. 11B). If you want to check more carefully the data or to change the unit-cell setting you have to use the *Lattice Wizard* (Fig. 11C). In the *Lattice Wizard* menu you can also see the unit-cell parameters (Fig.12A) and the indexation ratio (Fig.12B). In addition several options are available:
Peak Hunting: to extract once again the reflections from the frames collected during the pre-experiment (Fig.12C),
Unit cell finding: to calculate the unit-cell according to the peak table (Fig.12D),
Reindexation with current cell: to re-index the diffraction peaks according to the selected unit cell (Fig.12E),
Lattice transformation: to change the unit-cell setting (*eg*: orthorhombic to monoclinic setting) (Fig.12F),
Ewald explorer: to check your unit-cell and the position of the diffraction peaks into the reciprocal space (Fig.12G),
Save information: save the change you have applied (Fig.12H).

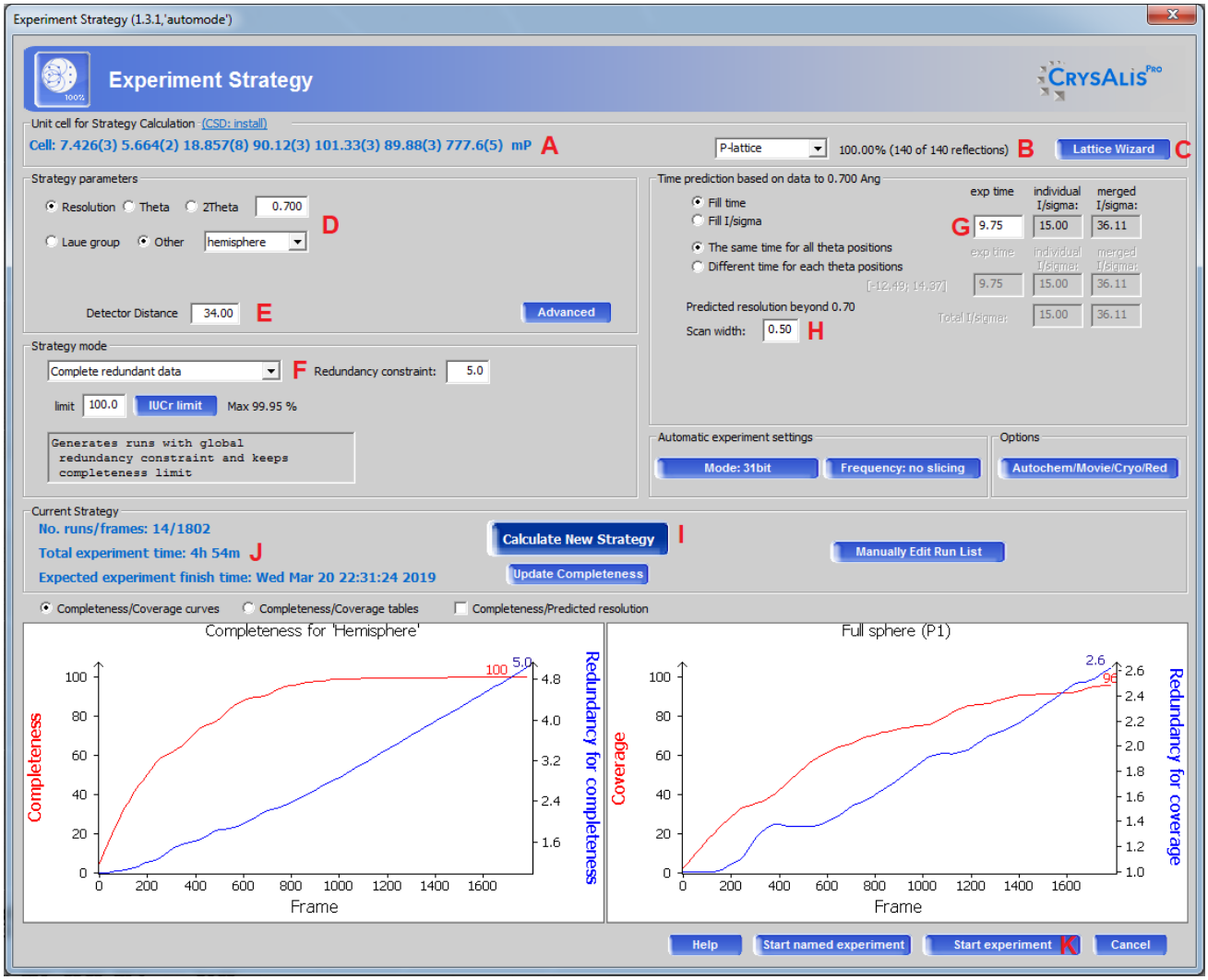


Fig. 11. Experiment strategy window.

Different options are available for the data collection:

Strategy parameters: fix the resolution you want to achieve in your data set. You can also choose to collect data according to Laue group of your sample or to collect an hemisphere or sphere of data regardless of the true symmetry of the crystal (Fig. 11D).

Detector distance: can be increase if you suspect the presence of twinning (Fig. 11E) (the minimum detector distance is 32 mm).

Strategy mode: you can increase the redundancy of your data by choosing *Complete redundant data* and a *Redundancy constrain* (usually 5 or 6 is used) (Fig. 11F).

Exposure time: the optimal exposure time is proposed by the software but you can increase or decrease this value depending on your/diffractometer availability (Fig. 11G). Note that for Cu-radiation experiments the default is to have two different exposure times for low and high 2θ, and as a rule of thumb the time for high angle data should be 4 times longer than for low angle positions.

Scan width: can be decrease to improve the resolution (0.3° is the common value) (Fig. 11H).

Calculate New Strategy: click on that button to recalculate the total experiment time if you have modified the experiment strategy (Fig. 11I).

Total experiment time: display the total time for the data collection (Fig. 11J).

Start experiment: This button will start the data collection (Fig. 11K).

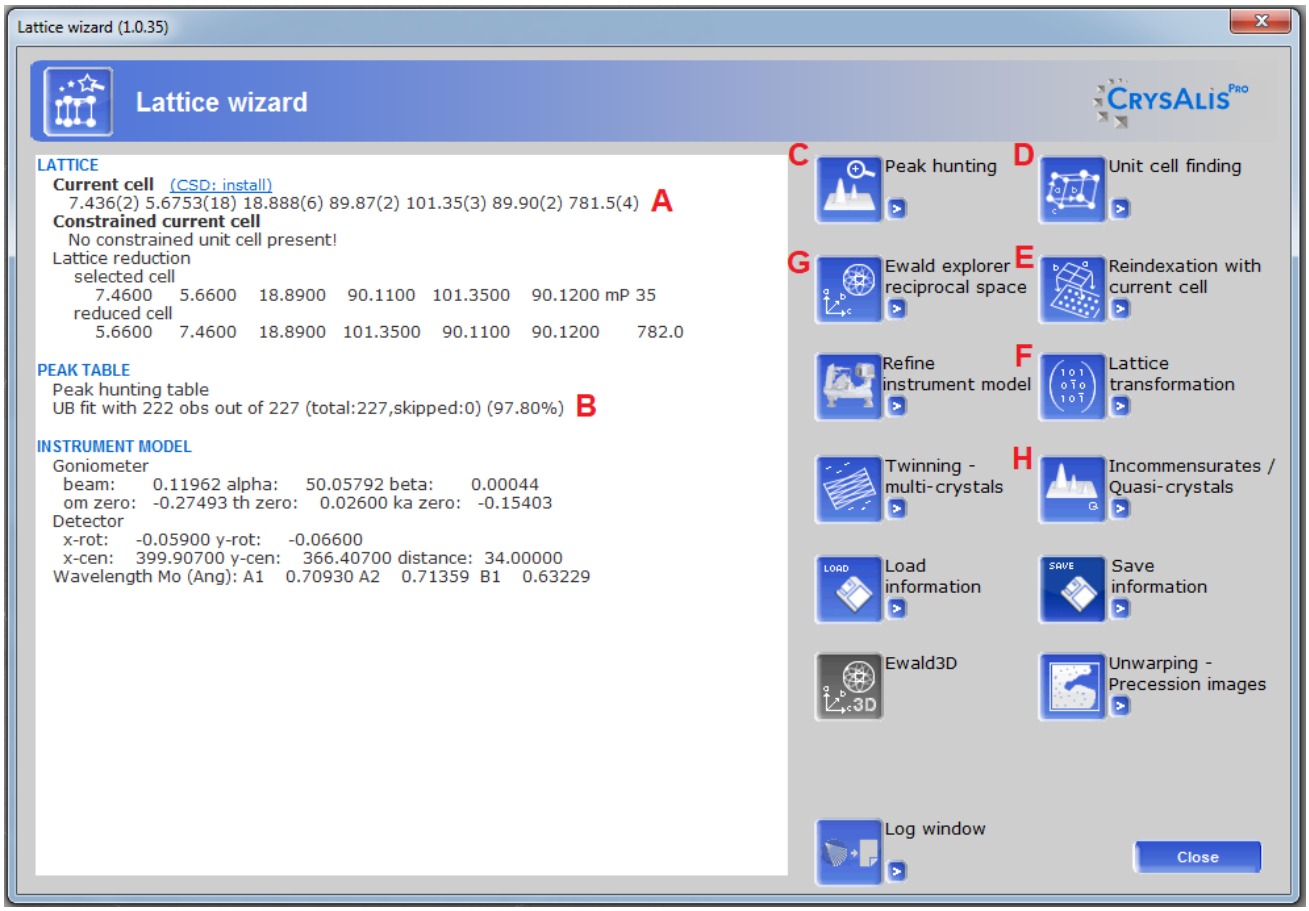


Fig. 12. Lattice wizard menu with the unit-cell information and options.

4.5. Integration of the reflections.

Data processing requires many steps and is highly dependent upon many parameters such as the quality of your data, the presence of twin, and the uncertainties of the true symmetry or space group of the crystal.
Therefore, in this part, we only take as example a simple case.

1) Open your data set by going into the experiment folder (*Computer/Data(D:)/Experiment-name/*) and by opening the file *Experiment-name.run*. The file will open through the CrysAlisPro (red) software.

2) To control your unit-cell parameters as well as your reflection indexation ratio you have to open the *Lattice wizard* (Fig. 13A). On this window you can see the unit-cell parameters (Fig. 13B) and the indexation ratio (Fig. 13C). You can open the Ewald Explorer (Fig. 13D) to investigate in detail your data set in the reciprocal or direct lattice.

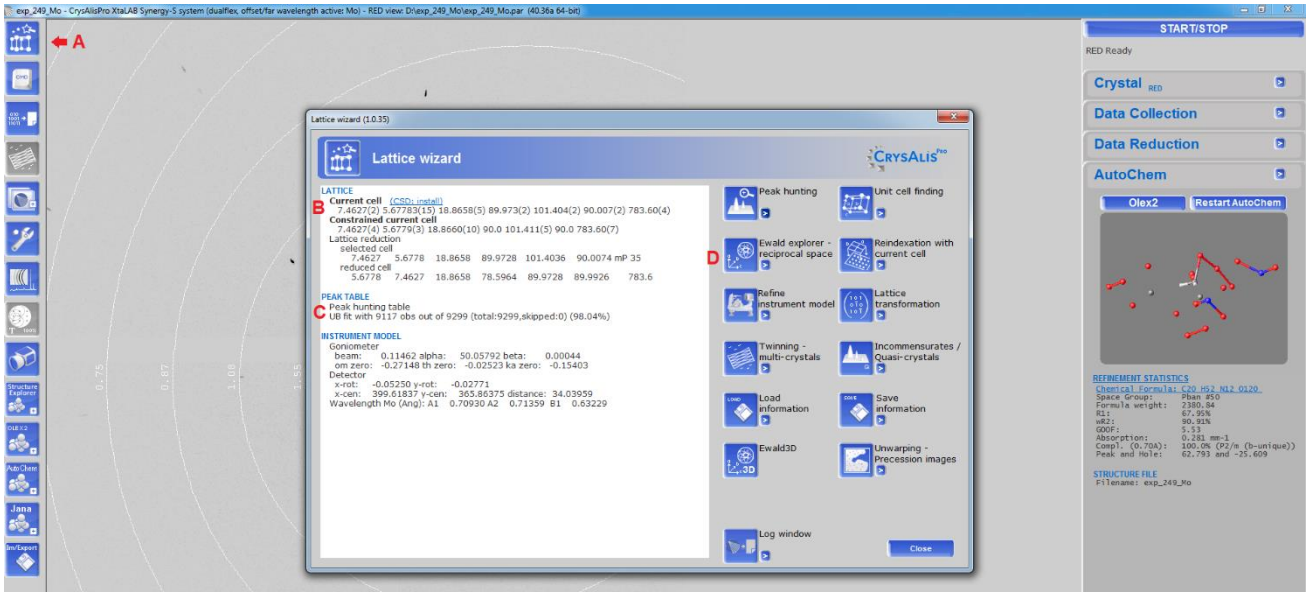


Fig. 13. The lattice wizard window in the CrysAlisPro (red) software.

3) Close the lattice wizard windows and click on the arrow close by the *Crystal* menu (Fig. 14A), and then *Edit chemical formula* (Fig. 14B) to update the chemical formula of your crystal (NOTE: at this point it is mandatory to insert a chemical formula because it will be used later on to calculate the absorption corrections).

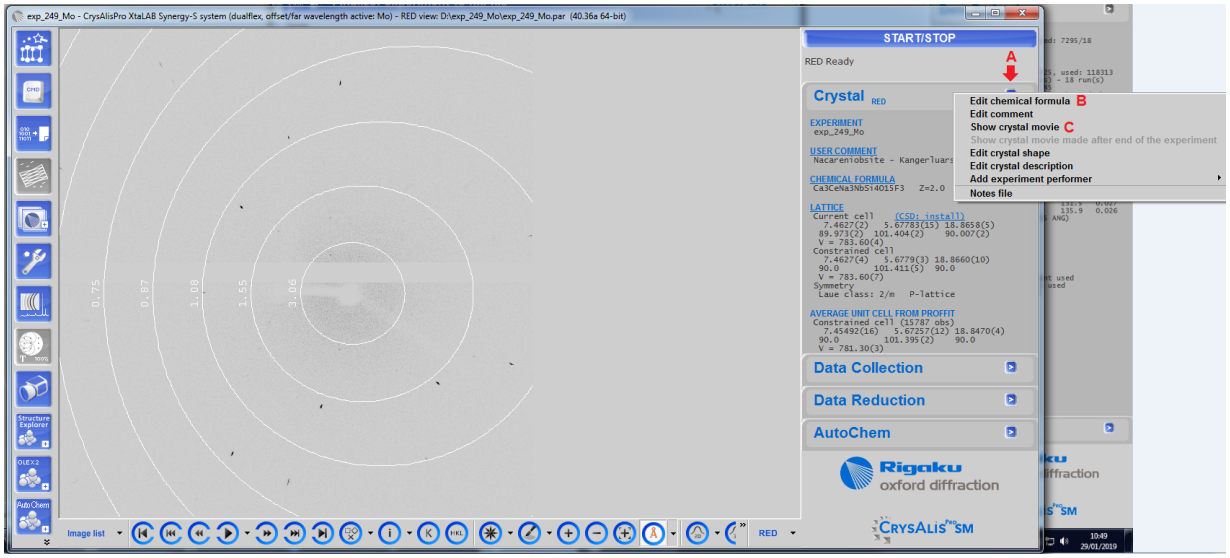


Fig. 14. Editing of chemical formula and of the crystal shape.

4) Then go to *Show crystal movie* (Fig. 14C) to open a new window in which you can draw the shape of the crystal (for the calculation of the absorption corrections).
A) Chose *Drag* in the face marking menu (Fig. 15A).
B) Use the *Page Up* and *Page Down* buttons on the PC keyboard to rotate your crystal.
C) Draw the crystal faces by clicking continuously on the mouse's left button, and by dragging the mouse cursor. Then click on the mouse's right button and press *Add face* (Fig. 15B) (NOTE: if the shape of the crystal is regular you can press *Add mirror faces*).
D) On the new window, chose *Integer hkl small* (Fig. 15C), and press add face (Fig. 15D) (NOTE: you can see the list of the drawn faces on the right (Fig. 135), and you may delete them by clicking on them and pressing *delete* on the keyboard).
E) The menu on the right also displays the crystal dimension (Fig. 15F).
F) Press *Exit* when you have finished the crystal drawing (Fig. 15G).

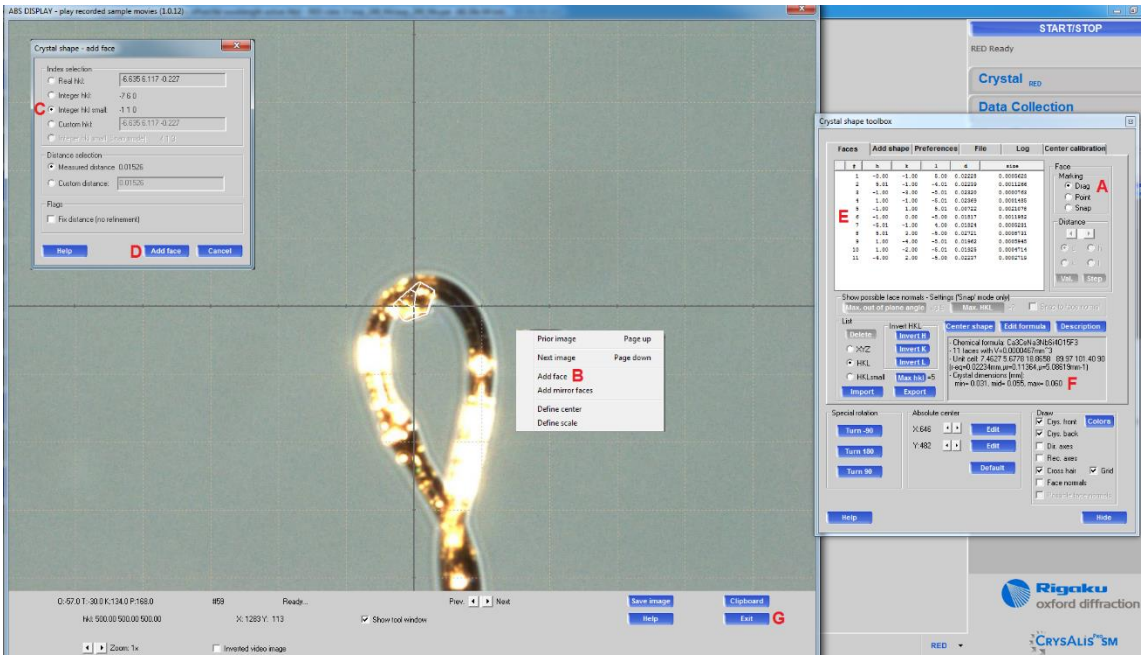


Fig. 15. Drawing of the crystal shape.

5) Press the arrow close by the *Data Reduction* menu (Fig. 16A) and then *Data reduction with option* (Fig. 14B).

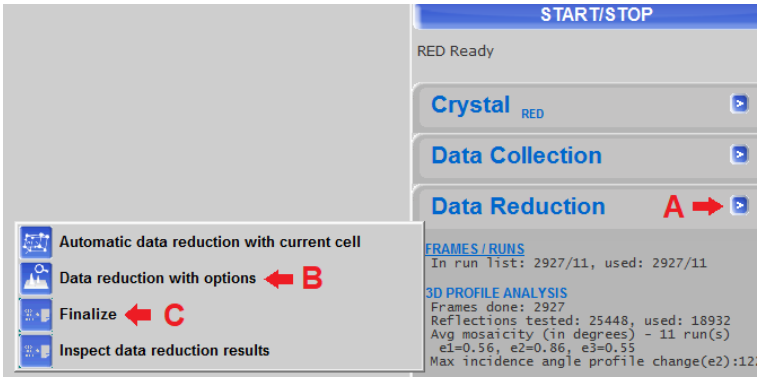


Fig. 16. Data reduction options.

6) The window of the *Data reduction assistant* will open, this assistant is divided into six steps (Fig. 17). Press *Next* to access to the following steps, and *Finish* to run the reduction procedure. Many options are available in the assistant and some of them are only useful in specific cases. However, in all cases, you have to (Fig. 17):

A) Press *Clear all data from tmp* to clear the temporary data;

B) Check that the Laue group and the unit-cell parameters are correct;

C) Choose *manual* for the space group determination;

D) Verify the chemical formula and *Z* value.

7) Press *Finish* and the software will start to reprocess the data, this step usually takes a couple of minutes to be completed.

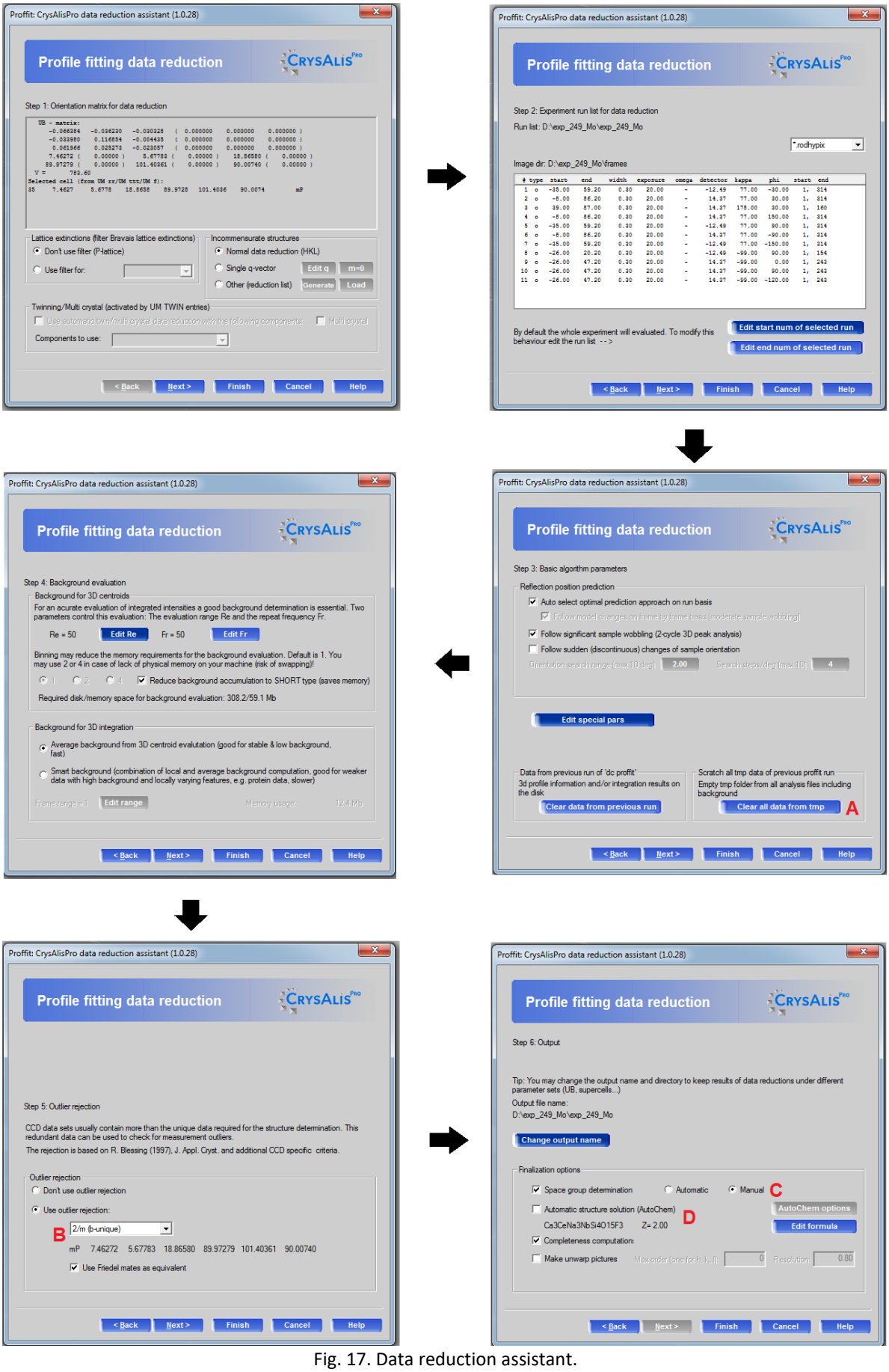


Fig. 17. Data reduction assistant.

8) Once the reprocessing completed, a new window for the space group determination will appear. The space group determination is divided into eight steps (Fig. 18) (NOTE: during these steps the software will always propose the most likely solution, but not necessary the correct one). Press *Next* to access to the following steps. The important parameters to check are the following:
A) Check the unit-cell parameters;
B) Check again the unit-cell parameters (it may change to another cell setting);
C) Check carefully if the presence or absence of a center of symmetry;
D) Check the *Z* value as well as the chemical formula.
Press *Finish* to complete the last step.

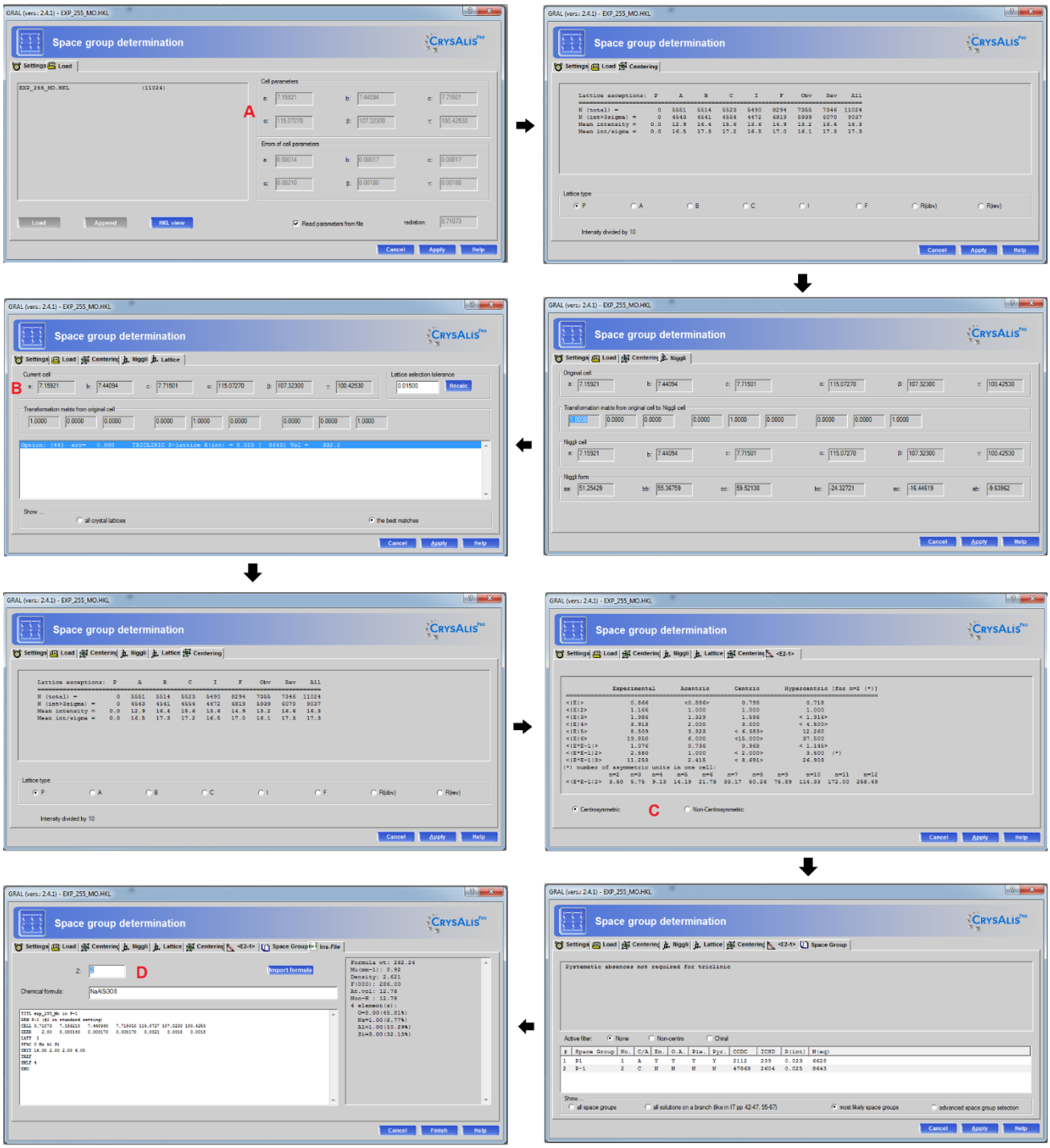


Fig. 18. Space group determination assistant.

9) Press the arrow close by the *Data Reduction* menu (Fig. 16A) and then *Finalize* (Fig. 16C), to open the *Finalize: experiment to hkl file* window (Fig. 19).

10) In this window check again the unit-cell parameters (A), the chemical formula and *Z* value (B), and press *Interactive* for the space group determination (NOTE: if you are sure of the space group choice you can use *Auto*).

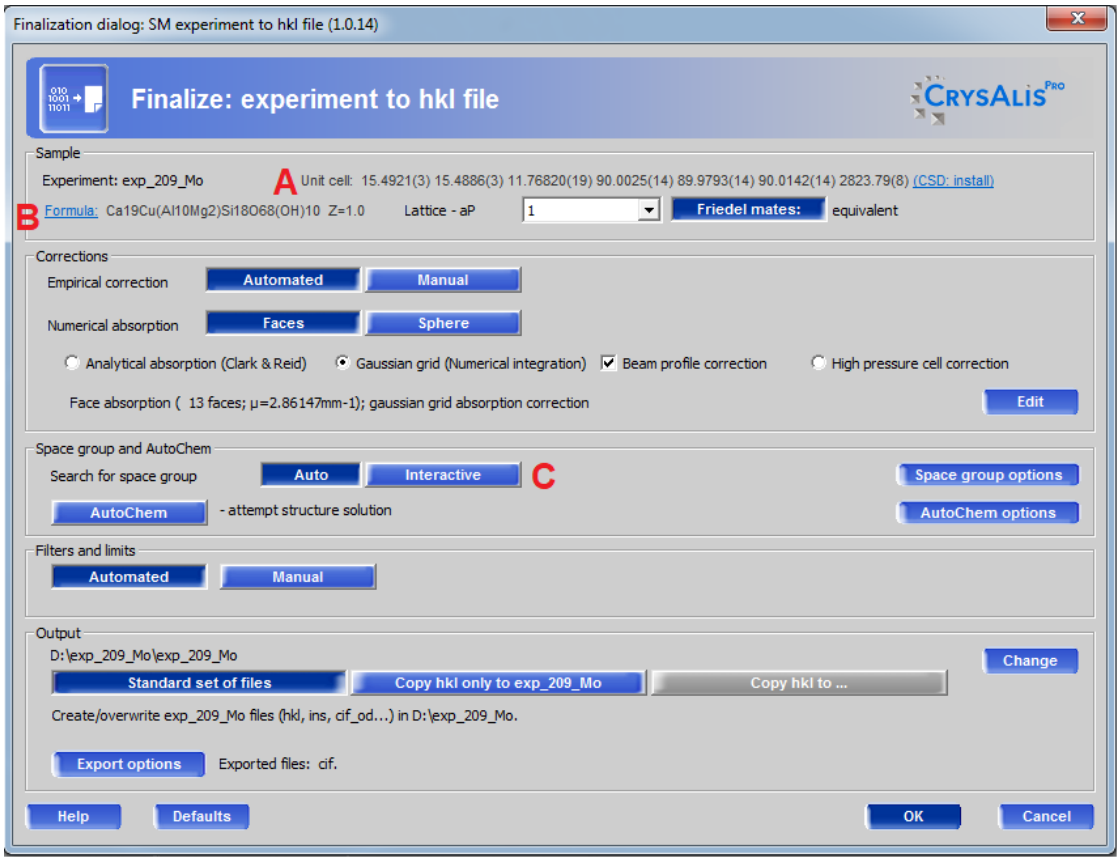


Fig. 19. Finalize assistant window.

11) If you have chosen *Interactive*, the same window as the one on the Fig. 18 will be displayed. Follow the same steps than those explained in *point 8)*.

12) Your data has been integrated and corrected. You can find the new *hkl* and *ins* files in the experiment folder (*Computer/Data(D:)/Experiment-name/*).

5. Collecting data in Gandolfi mode

Follow the sections **4.1.** and **4.2.** to select the radiation source, to mount and center the sample.

5.1. Measuring a sample

1) Press the button representing a powder diffractogram in the left menu on the CrysAlisPro (online) software to open the Powder experiment dialog box (Fig. 20a).

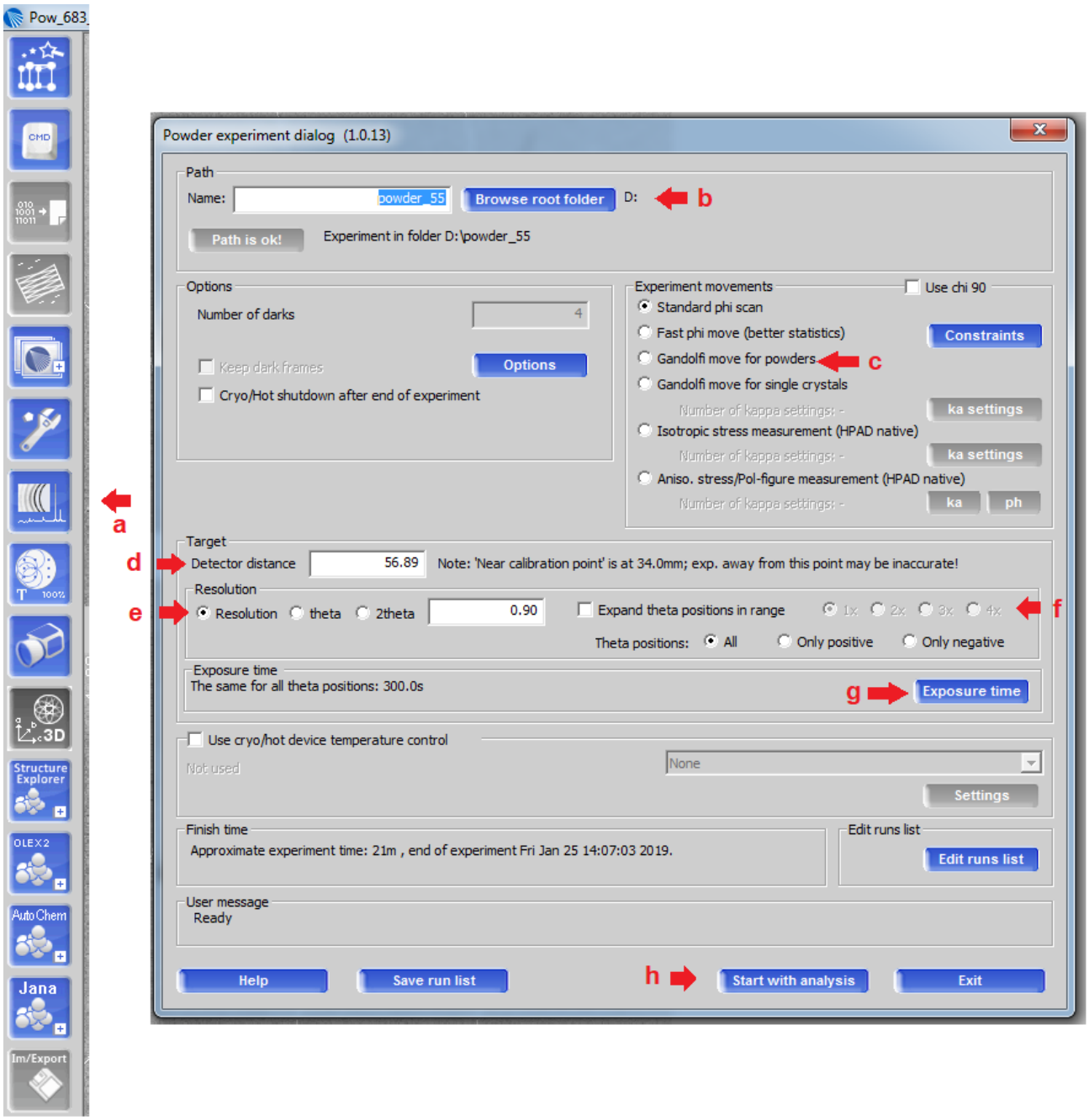


Fig 20. Starting menu to collect a powder diffractogram.

2) Insert a name for your experiment (Fig. 20b), use the prefix Pow, the experiment number, and your initial as follow: Pow_XXX_Initial (check the previous data on the *D*: drive for number);
3) Select *Gandolfi move for powders* (Fig. 20c);
4) Change the *Detector distance* to 65.0 mm (Fig. 20d);
5) Change the resolution to *2theta* = 80 ° (Fig. 20e);
6) Press the box *Expand theta positions in range*, and select 1x (Fig. 20f);
7) Select the exposure time you want (Fig. 20g). (NOTE: 30sec is usually enough to get a good powder diffractogram (4min in total), but you can increase the time if it is required;
8) Click on *Start with analysis* (Fig. 20h).

5.2. Data integration

When the data collection is completed, the spectra has to be reprocessed and transformed. In the Geolab, the EVA software is used to analyze the powder pattern. However, EVA cannot read the file generated from CrysAlis software, and therefore the file has to be transformed using the software *PowDLL Converter*.

1) Click on the *Options* button below the spectra (Fig. 21a) and tick the boxes *PDXL/Jade format* (Fig. 21b) and *Baseline correction* (Fig. 21c). Then press *OK*.
2) On the main window, press *Reprocess* (Fig. 21d) to recalculate the powder pattern.

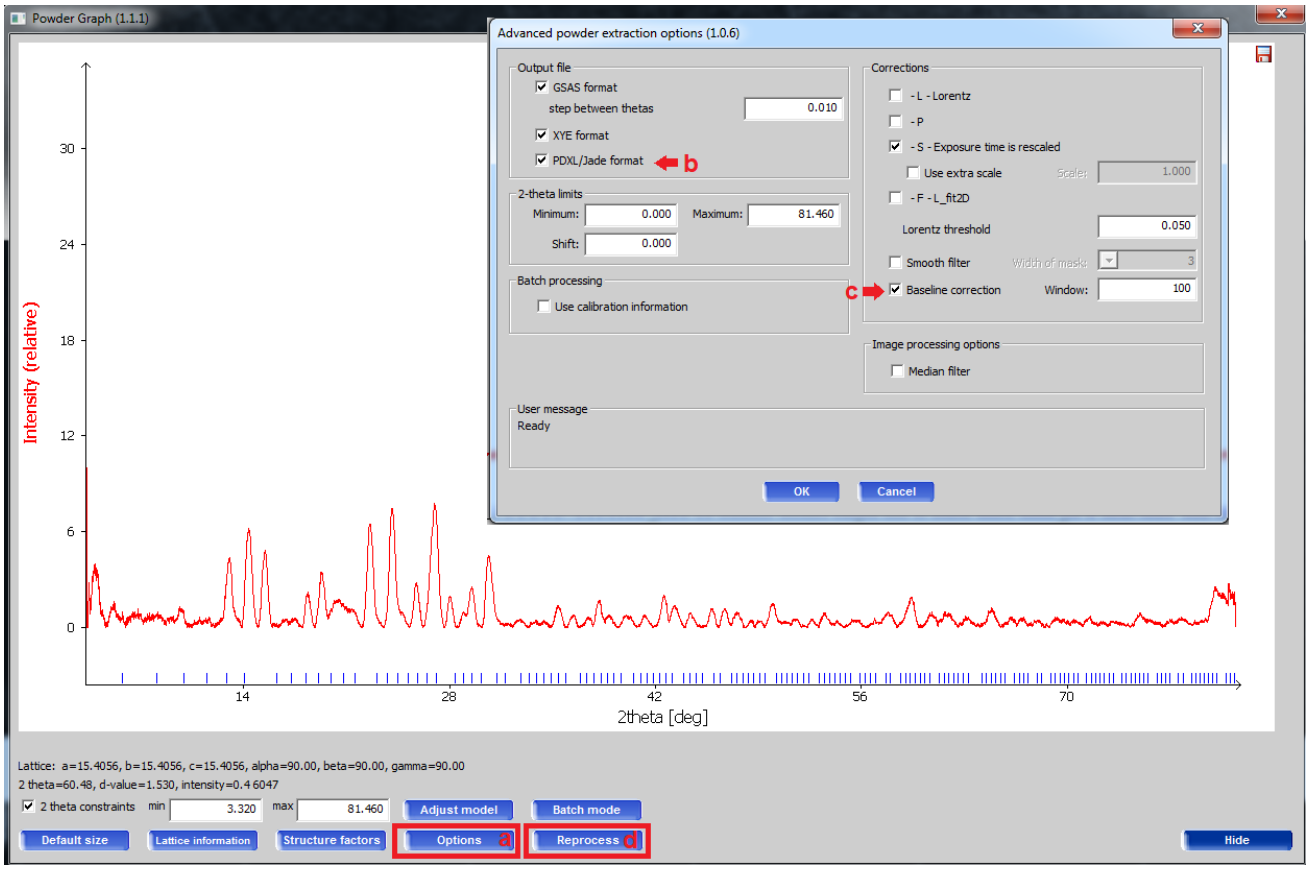


Fig. 21. Reprocessing menu for the powder patterns.

3) Go to the Windows menu to open the *PowDLL Converter* software (Fig. 21).
4) Press *Open* button to define the source. Follow the path *Computer/Data(D:)/Your-experiment-name/plots_red* to find your spectra to convert.
5) The destination folder will be automatically updated and the name of the output file will be the same. To avoid overwriting the original pattern, simply add a number at the end of the spectra name. Press the *Save as* button and change your sample name.
6) Press *Convert* and say *OK*.

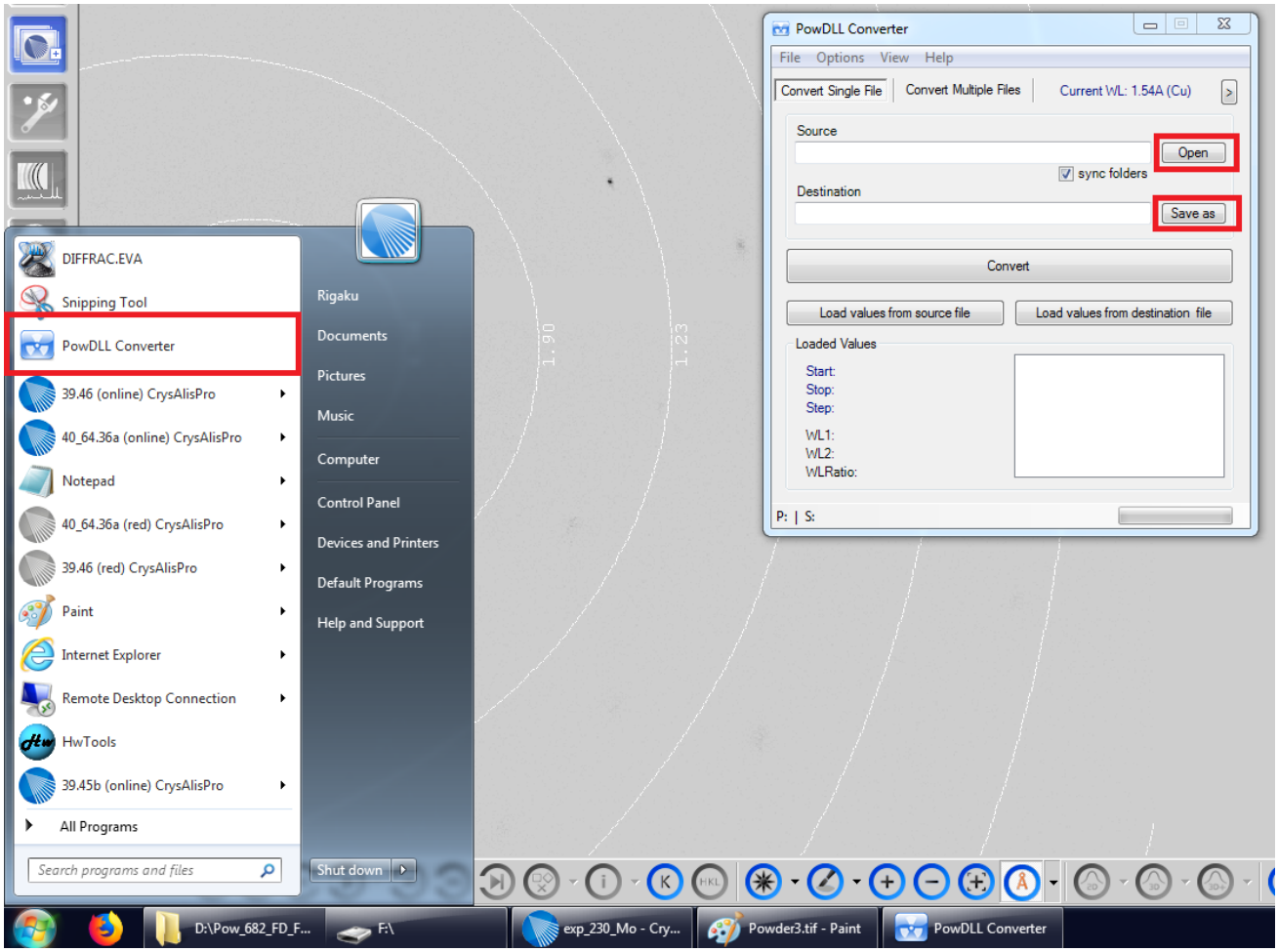
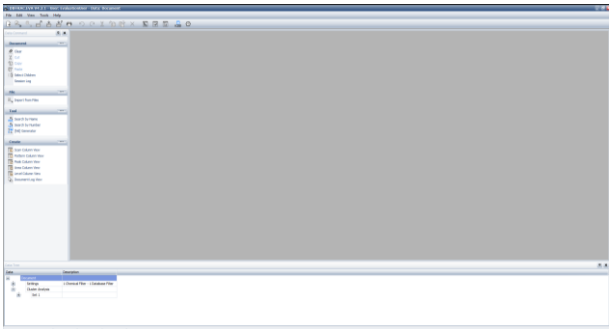
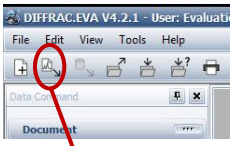
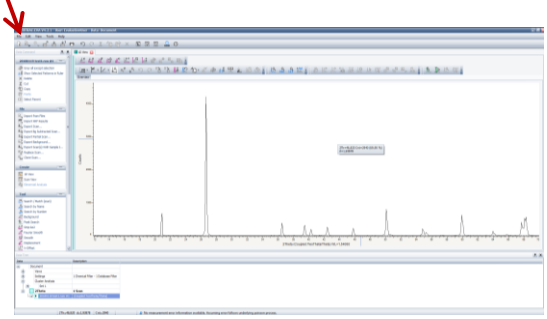
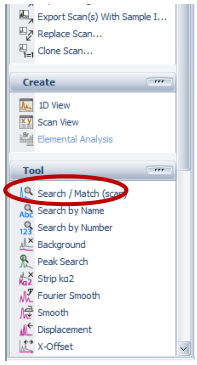
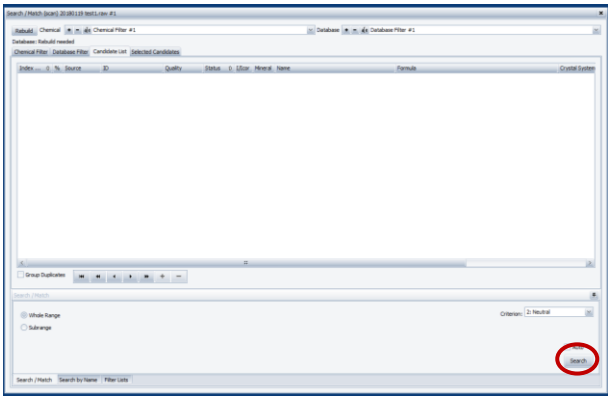


Fig. 21. PowDLL converter software.

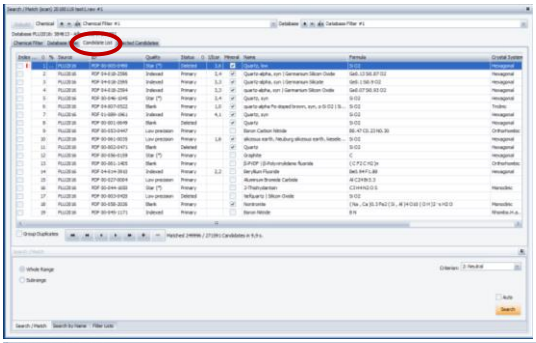
5.3. Interpretation of the spectra with EVA.

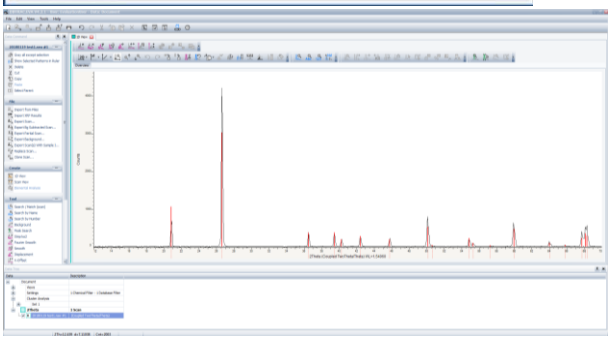
Step	Example/hints
1 Open the software ***DIFFRAC EVA*** main window.	
2 ***Open*** the ***scan (RAW file)*** of your interest by clicking in the indicated button.	  *A scan is a diffractogram resulting from the collection of scattered X-ray radiation when analyzing a sample with a powder X-ray diffractometer.* *The scan (RAW file) can also be open in the menu File.*
3 Click ***Search and Match (Scan)*** in the Section Tool.	

4 Click ***Search***.



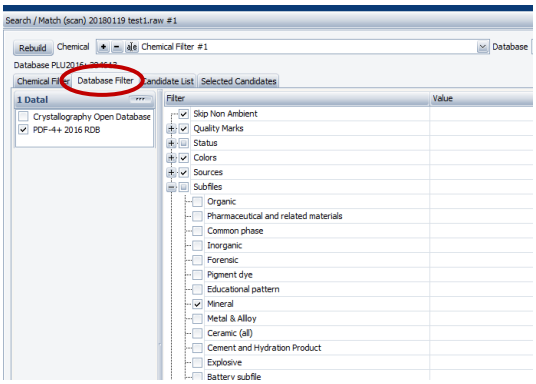
5 A list of suggested patterns will be generated. Try to ***find the pattern that fits better with your scan*** (best ***match***) in order to identify the mineral(s) in your sample.





If you have more than one mineral in your sample you may have to repeat the process of matching several times as just one mineral can be matched at the time.

6 Use the Section ***Database Filter*** to select the database that you want to use.



Unfortunately, you have to run a search before being able to filter the database you want to use.

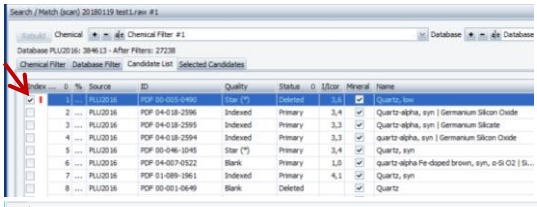
This is necessary when the results displayed in the first search are not satisfactory enough to lead to a good match.

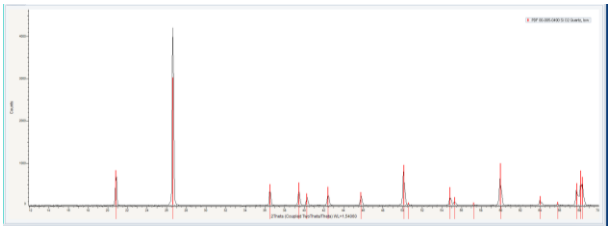
7 Go back to the Section ***Candidate List*** and click ***Search***. A new list will be generated.

Try to ***find the pattern that fits better with your scan*** (best ***match***) in order to identify the mineral(s) in your sample.

Repetition of steps 4 and 5.

8 When you find the pattern that best matches with your scan ***click in the square*** in the left hand side ***to select*** that pattern as the correct choice.

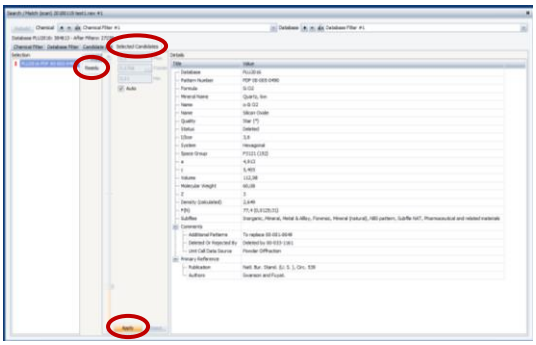


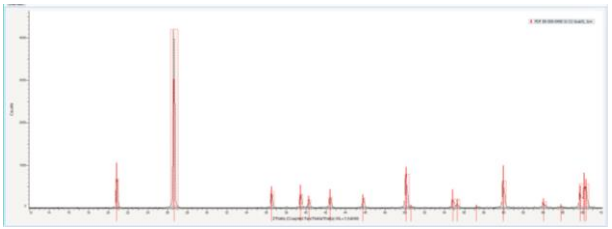


9 If ***other mineral(s)*** are present in your sample you can identify them by going to the ***Section Selected Candidates***.

Click in ***Residual***. Red shadow areas will be visible over the peaks of the already identified mineral(s) in your scan.

- If the ***automatic selection*** is correct (usually is), click ***Apply***.
- If the automatic selection leaves some peaks unidentified you can do so ***manually*** adjusting the parameters in the boxes before clicking ***Apply***.





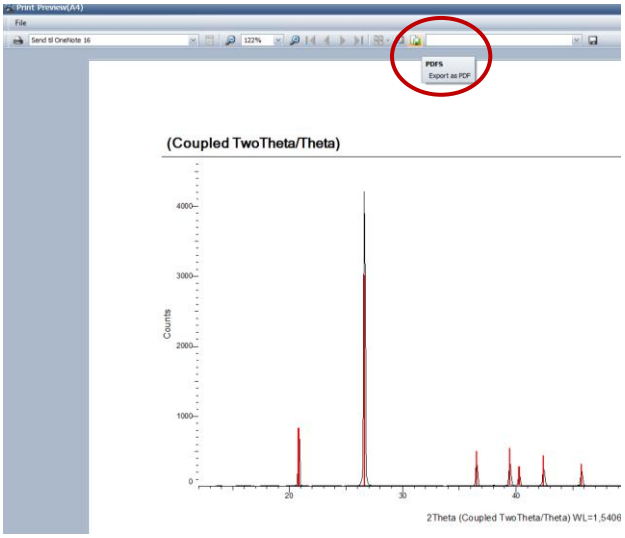
10 ***Repeat the steps 4 to 9*** until you are satisfied that you found all minerals in your sample.

11 Click the icon save or use the menu File to ***save the results in an EVA file*** (extension .eva).

EVA files contain a copy of the raw file and every processing that you may have added to it. Thus, the original data (background subtraction, smoothing, angular shift, …) can be adjusted without modifying the original raw file itself.

EVA files shall be saved in the folder C:\DIFFDAT1 with the same name as the correspondent RAW file.

12 You can save a ***pdf file*** of your scan by selecting ***Print Preview*** in the EVA main window and then ***Export as PDF***.



Pdf files shall be saved in the folder C:\Users under a folder with your name (e.g. C:\ Users\ Waldemar Brøgger).

13 Fill the ***sample ID*** in the ***Logbook*** and save the changes made to the file.

Excel file available in the Desktop.

6. Sample mounting

The samples for both single-crystal and Gandolfi measurements are mounted on cryoloop (Fig. 23A), and maintained on place by using oil (Fig. 23B). The usual procedure to mount the sample consists of putting a small drop of oil on a glass slide (Fig. 23C), and place a sample in the oil droplet. The oil ensures that the crystal fragment does not jump if it needs to be reduced in size.

To extract and separate the sample many tools of different size are available (Fig. 23D) (NOTE: for single-crystal investigations, crystal of 100 µm in dimension is usually sufficient).

Attach your sample to the loop by dropping the loop into the oil. Try to mount the sample on the top of the loop. For large crystal it's better to mount the sample inside the loop (close to the top part), in order to avoid any movement of the crystal during the data collection. Try to remove as much of the excess of oil as you can. Crystals mounted in a lot of oil or with little contact to the loop have a tendency to move during the data collection and it can in a worst case scenario result in a useless data collection.

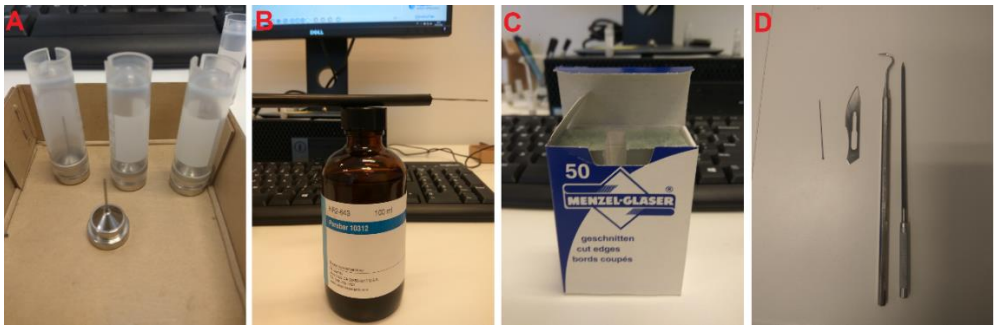


Fig. 23. View of the cryoloop (A), oil (B), glass slides (C) and tools (D) used to mount the samples.

The optical microscope used to mount the samples is equipped with a top light (Fig. 24A) and a back light (Fig. 24B). There is also a Nicol prism (Fig. 24C), that can be used by rotating the ring located close by the objective lens. This enables you to check the crystal quality and to see if it is a single grain, *i.e*. the entire grain becomes extinct at the same time. If this is not the case check other crystal fragments before deciding on the best to mount.

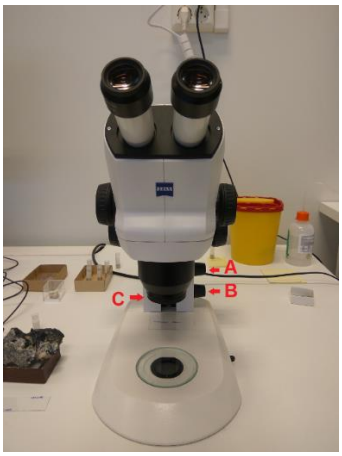


Fig. 24. View of the optical microscope located in the diffraction room.

7. Booking the instrument

You are required to book time for your analysis using the ***instrument calendar on Outlook or Webmail***. The name of this instrument calendar is: ***Økern: Geologisk museum, SXRD***. Instructions are available at the Geo Lab webpage:
https://www.nhm.uio.no/english/research/infrastructure/geo-lab/xrd/

If you don't have access to the instrument calendar (e.g. if you are an external user), please contact the scientist in charge of the instrument if they can assist you with your analysis.

8. Instrument notebook

You are required to fill the instrument notebook by adding at least the name of your experiment and the date.

You are also required to log your usage of the instrument using the excel file named ***Logbook*** available at the SXRD computer (check the Desktop). At the beginning and end of each analyze please fill in the relevant information.

9. Saving data

USB devices are NOT allowed in this computer.

A copy of your data will be transferred to the following folder *\Økern-GeoLab\GeoLab_Users data_Temporary storage\"yourname"*
The data will be available from Monday the week after your booking and for a period of one month. Contact the lab manager or deputy if you do not have access to this folder or if you need your data earlier.