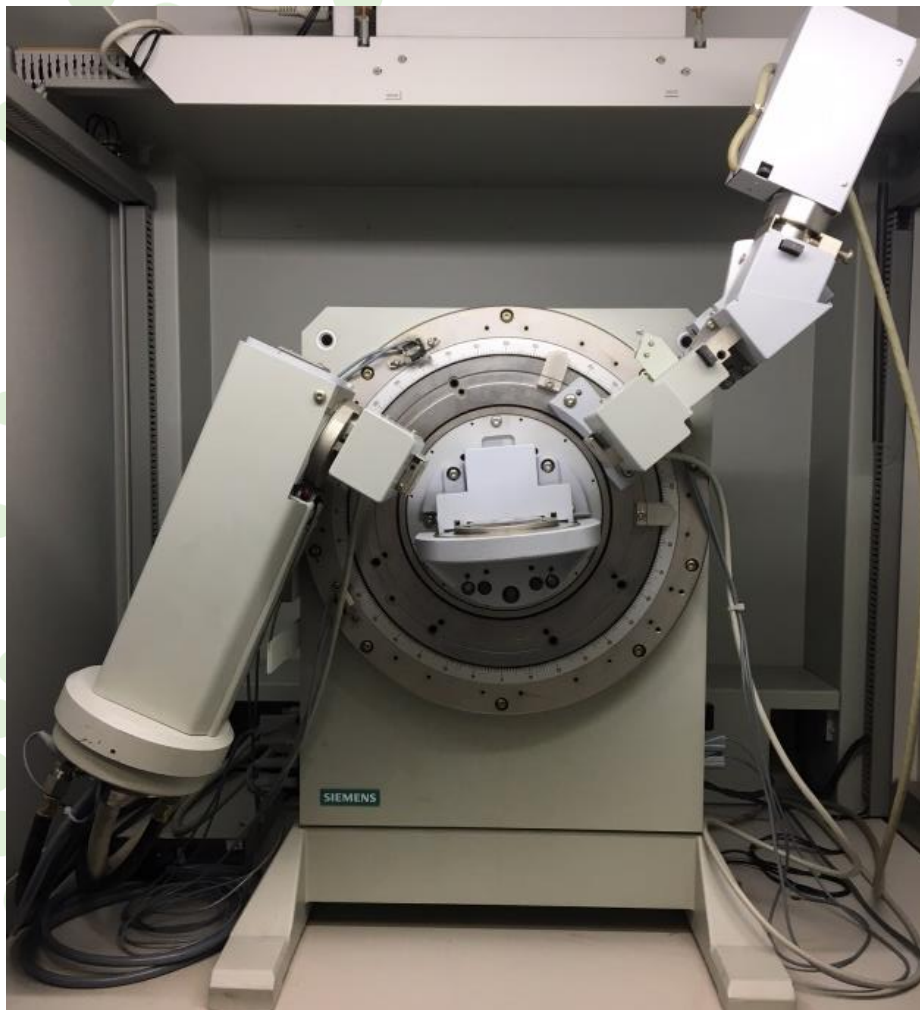


Siemens D5005 X-ray Diffractometer

User instructions



Geo Lab

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

U562 – Analyzelaboratorie 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)

Contact | Kontakt oss

Lab manager | Laboratorieleder

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

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1. Introduction

The Geo Lab at the Natural History Museum, University of Oslo, is equipped with a Siemens 5005 X-ray diffractometer (XRD) used primarily for phase identification of minerals in rocks.

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

tlf: 228 51641 e-mail: nelia.castro@nhm.uio.no

Associate Professor, Henrik Friis

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

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 Siemens 5005

Personal injuries can result from the inappropriate use of the instrument. The following warnings apply to the use of the Siemens 5005 XRD:

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.	The equipment 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 Siemens 5005 XRD:

Action	Consequences	Safety measures
Prolonged use.	Eye strain, tiredness, and musculoskeletal strain or injury.	Take regular breaks (about 10-15 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 Siemens 5005

Figure 1 shows the appearance of the Siemens 5005. The instrument is connected to a computer that contains all the software necessary for acquisition and treatment of the data.

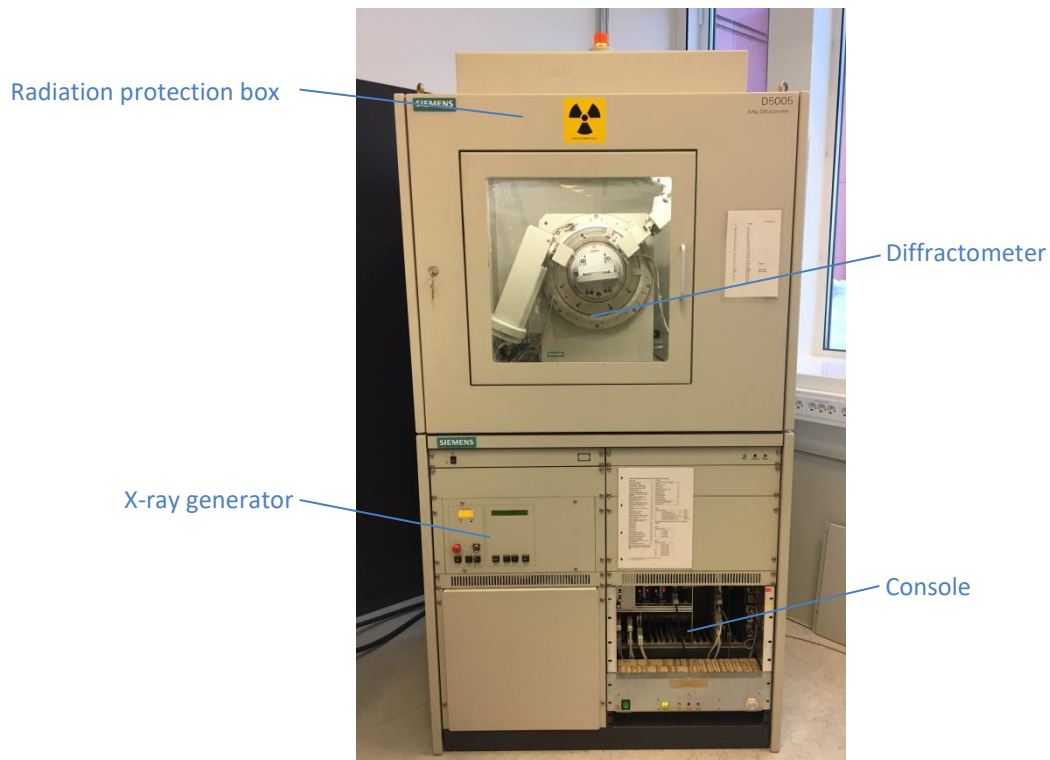


Fig. 1: Appearance of the Siemens 5005.

The diffractometer (Fig. 2) consists of the goniometer, the tube, the tube stand, the diaphragm system required for the measurement and the sample changer. The unit is installed vertically in a radiation protection housing, meeting the requirements for fully protected instrument. A lead glass window at the front of the radiation protection housing enables the samples to be changed. The window shutter of the tub stand closes automatically when the window is opened.

CAUTION: NEVER try to override the interlock. Danger of exposure to radiation.

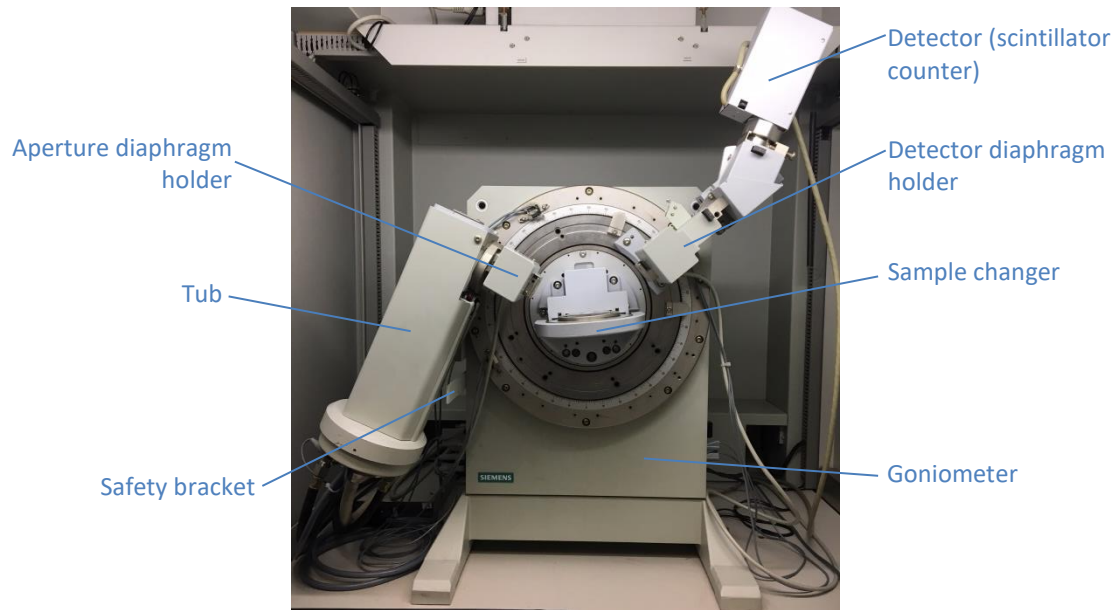


Fig. 2: Diffractometer, vertical installation.

3.2. Software

The software DIFFRAC XRD Commander is used to control the operation of the XRD. Described next (Fig. 3) is the main window used to control the Siemens 5005.

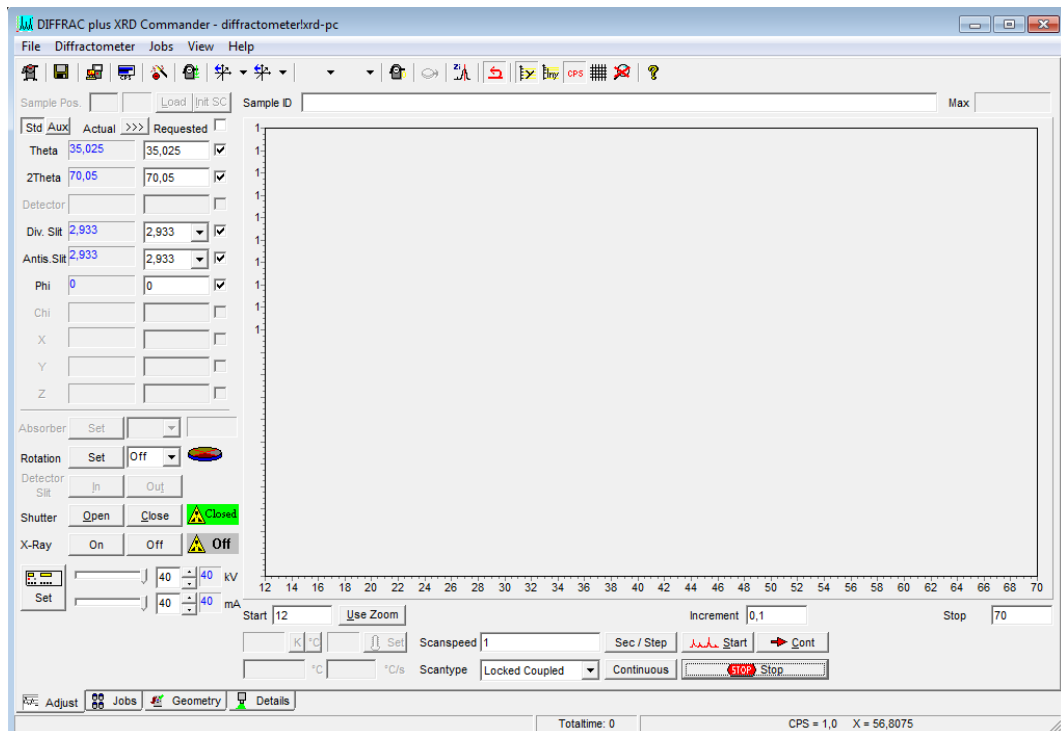


Fig. 3: Overview of the main window of the software used to control the Siemens 5005 XRD.

The software DIFFRAC EVA is used to evaluate X-ray diffraction data. Figure 4 shows the main window of DIFFRAC EVA software.

Two distinct pattern databases are available at the Geology lab: the ICDD PDF 4+ 2019 and the Crystallography Open Database.

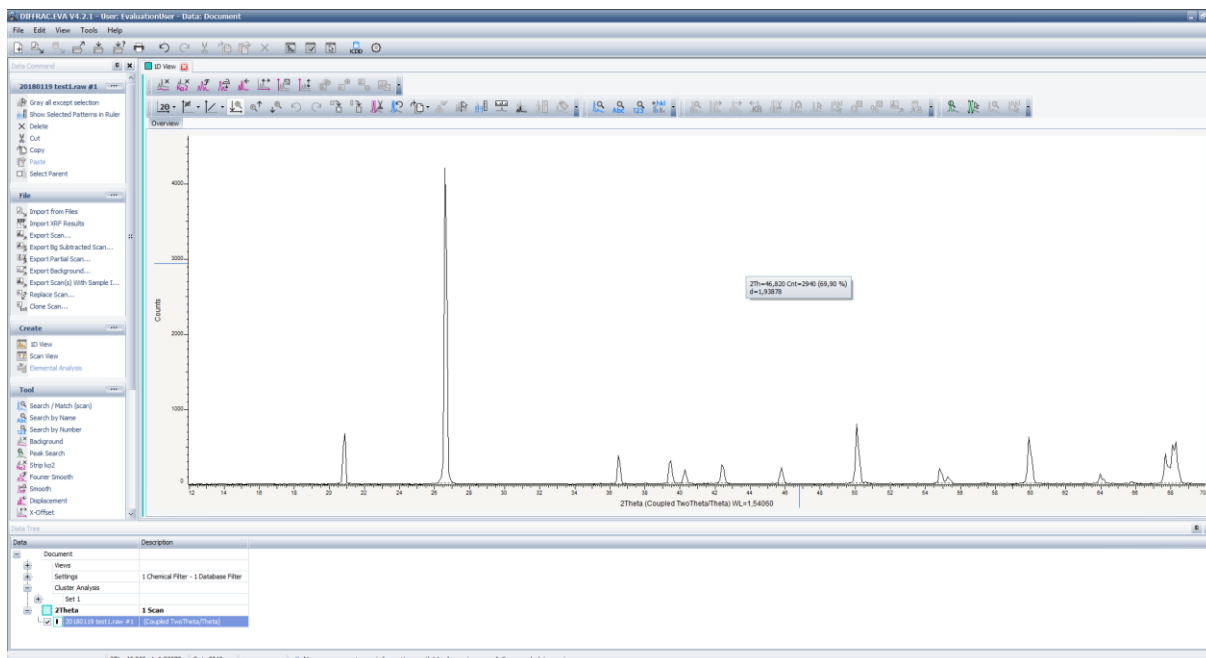


Fig. 4: Overview of the main window of the software DIFFRAC EVA with a scan opened.

4. 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, PXR**D. 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 laboratory manager or deputy and ask if they can book time for your analysis.

5. Logbook

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

6. Sample preparation

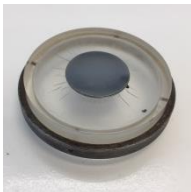


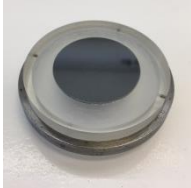
Remember that the success of a powder diffraction analysis is dependent on the nature of the sample, the quality of its preparation, and the experimental conditions. The main objective of powder sample preparation is to minimize source errors.

Sample preparation for XRD powder diffraction at the Geo Lab is done in the Room U560 – Prep. 4 (Sample Preparation SEM, XRD, CT). All material and sample holders necessary for preparation of powder samples are available at this room.

6.1. Specimen holders

There are a number of different sample holders available for the Siemens 5005 XRD. Choose the sample holder according to the characteristics of your sample and the sample preparation method that you will be using. The sample holders available at the Geo Lab and respective type of sample to which they are indicated can be seen in Table 1.

Table 1: Overview of the sample holders available for the Siemens 5005 XRD and respective type of sample and sample preparation method to which they are indicated.

Sample holder	Type of sample / Sample preparation method
	Indicated for when a very small amount of sample is available and the sample preparation Method 1 is used (Section 6.2).
	Indicated for when a very small amount of sample is available and the sample preparation Method 1 is used (Section 6.2).
	Indicated for when a large small amount of sample is available and the sample preparation Method 2 is used (Section 6.2).
	Indicated for when a very small amount of sample is available and the sample preparation Method 1 is used (Section 6.2). However, this sample holder has an especially delicate surface polishment and shall only be used for special samples.

6.2. Sample preparation methods

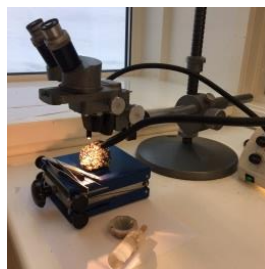
6.2.1. Method 1

The most common sample preparation method used at the NHM for powder XRD was developed to situations when a **very small amount of sample** is available for powdering and analyze:

Step

1 Obtain a few tenths of a gram (or more) of the material, as pure as possible.

Example/hints



2 Place the material in a mortar, add ethanol (a few drops), and with the help of a pestle grind the sample to a fine powder.



Grinding your sample in a fluid like ethanol will minimize induced strain and randomize orientations, contributing for the quality of your analyze because strain can offset peak positions and preferential orientation can influence the peak intensity.

3 Place the slurry into a sample holder using the pestle. Care must be taken to create a flat surface and to achieve a random distribution of lattice orientations.



There are two main options of sample holders for this type of samples.

If the slurry is too thin, i.e. there is too much ethanol in the slurry, the chances of preferred orientation will increase. Let the slurry dry a bit before placing it into the sample holder.

6.2.2. Method 2

If a relatively **large amount of sample** is available the following method can be used:

Step

- 1 Obtain a few grams (or more) of the material, as pure as possible.
- 2 Place the material in a mortar, add ethanol (a few drops), and with the help of a pestle grind the sample to a fine powder. Alternatively, you can use another type of mortar (not available in our lab) to grind your sample.

Example/hints



Grinding your sample in a fluid like ethanol will minimize induced strain and randomize orientations, contributing for the quality of your analyze because strain can offset peak positions and preferential orientation can influence the peak intensity.

- 3 Let the powder dry (or dry it in an oven) and place enough powder in the sample holder to fill the central part.



There is only one type of sample holder indicated to this type of sample.

- 4 Use a glass slide to even the surface of the powder. Make sure that the powder surface is at the same height as the sample holder border. Care must be taken to create a flat surface and to achieve a random distribution of lattice orientations.

If the surface is not flat and/or there is preferential orientation of lattice orientations the quality of your analyze will be compromised.

Contact the laboratory manager or deputy if you need help preparing your samples or if you want to use a different sample preparation method.

7. Operating instructions

The procedure for startup, specimen exchange, start a measurement, and pattern search and match are explained in this section.

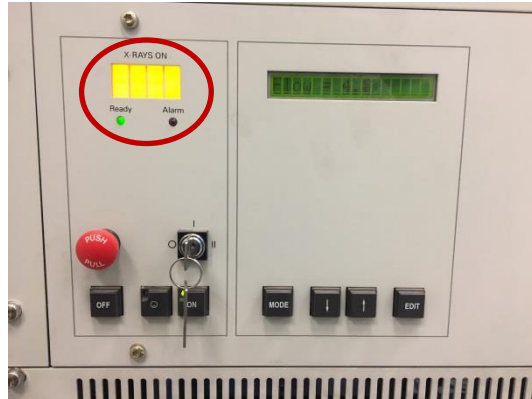
7.1. Startup

Step

1 When arriving at the laboratory make sure that the **X-rays** and **Ready light** are **ON**.

2 Now you are ready to insert your first sample in the instrument.

Example/hints



Contact the laboratory manager or deputy if at your arrival to the laboratory the XRD conditions differ from the description above.

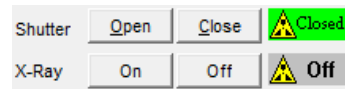
Follow the instruction for specimen exchange (Section 7.2).

7.2. Specimen exchange

Step

1 If a measurement is ongoing (X-rays are on) wait until the measurement is finished or, if the ongoing measurement is not of interest, stop the measurement and **make sure the shutter is closed**.

Example/hints



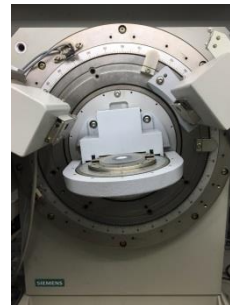
This information is displayed in the XRD Commander main window.

Even when indicates that the x-ray is ON, as long as the shutter is closed it is safe to open the glass window.

2 Open the glass window and remove the sample holder from the sample changer (if there is any).



3 Place your sample in the sample changer and close the glass window.



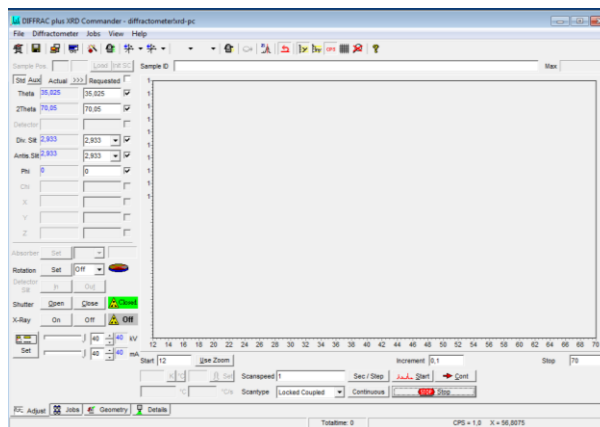
7.3. Start a measurement with Commander

Step

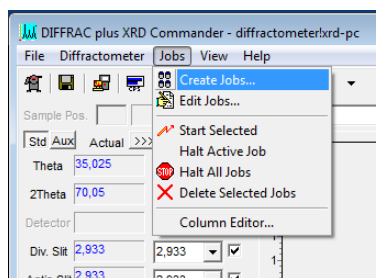
- 1 Fill in the **logbook**.
- 2 Open the **XRD Commander** main window.

Example/hints

Excel file in the desktop.



- 3 Click **Jobs** and select the option **Create Jobs**.

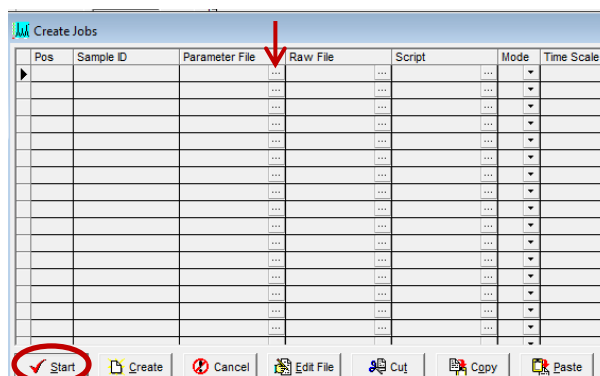


- 4 Fill in the required **information** and click **Start**.

Sample ID corresponds to the name of your sample.

Click in the button on the right hand side to choose the right **parameter file***

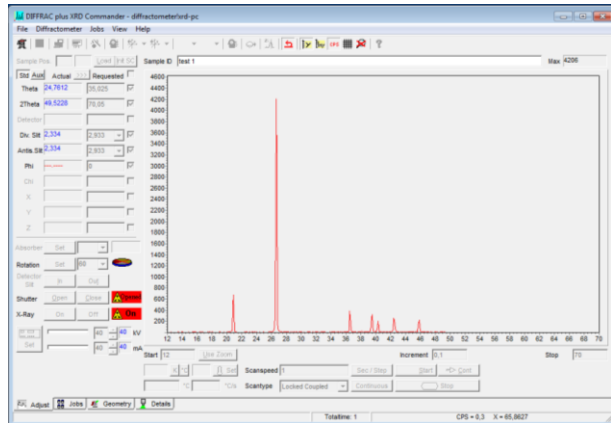
The **RAW File name** needs to be input. Start with the **date** and add the **name** of your sample (e.g. 20180119 test1).



*A number of parameter files are available. If you need to perform an acquisition at different definitions, contact the laboratory manager or deputy.

Remember that the RAW File name used here and registered in the logbook must be the same.

5 The measurement starts.



Different components of the diffractometer move to analyze position, which leads to some noise.

The diffractometer will slowly start to form in the XRD Commander main window.

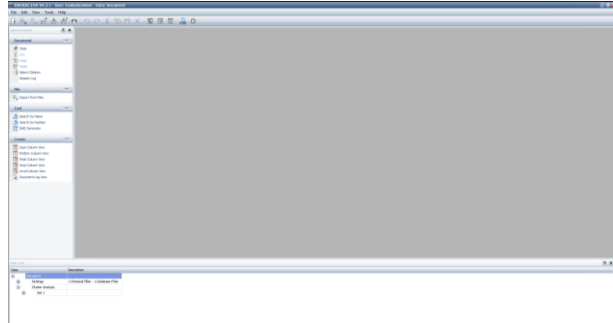
6 The RAW file is automatically saved in the folder C:\DIFFDAT1.

7.4. Pattern search and match with EVA

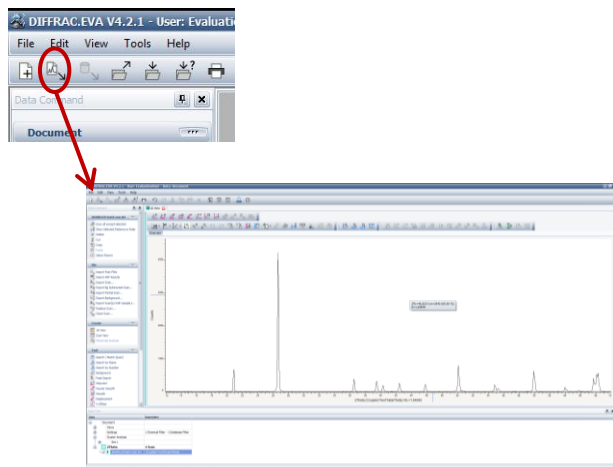
Step

1 Open the software **DIFFRAC EVA** main window.

Example/hints



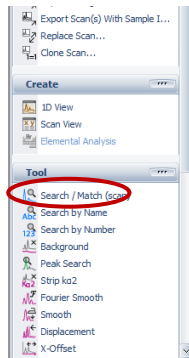
2 Open the scan (RAW file) of your interest by clicking in the indicated button.



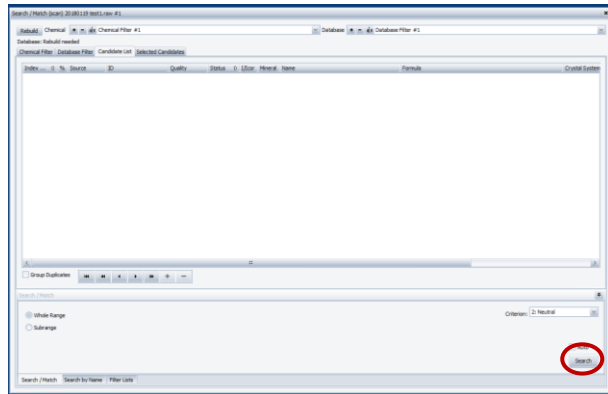
A scan is a diffractogram resulting from the collection of scattered X-ray radiation when analyzing a sample with a powder X-ray diffractometer.

The scan (RAW file) can also be open in the menu File.

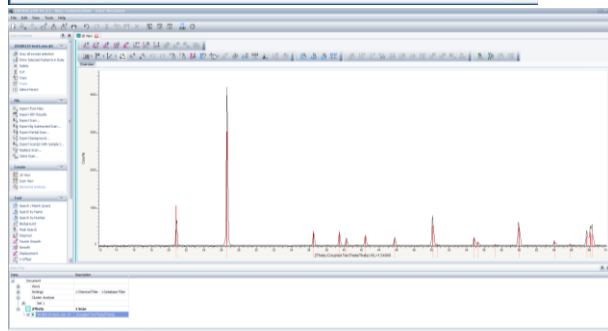
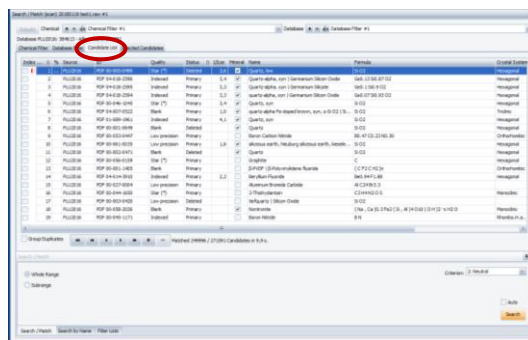
3 Click **Search and Match (Scan)** in the Section Tool.



4 Click Search.

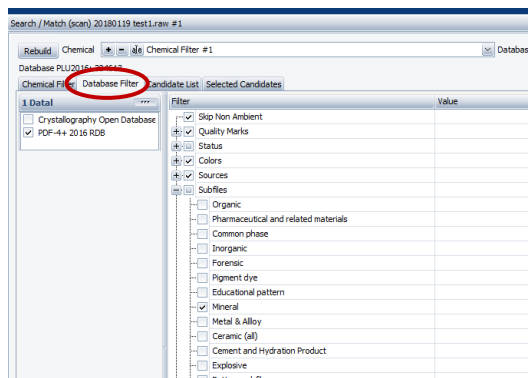


5 A list of suggested patterns will be generated. Try to find the pattern that fits better with your scan (best match) in order to identify the mineral(s) in your sample.



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.

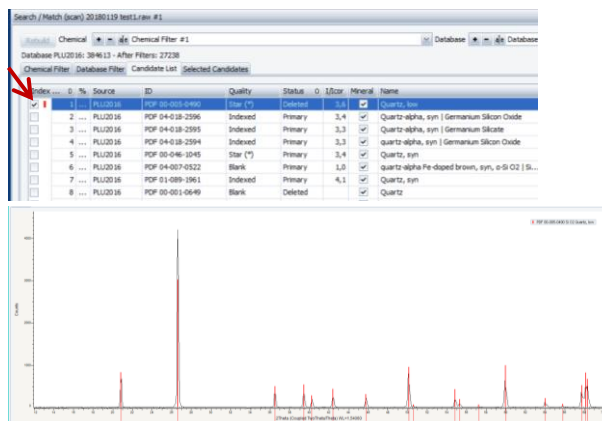
This is necessary when the results displayed in the first search are not satisfactory enough to lead to a good match.

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.

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.

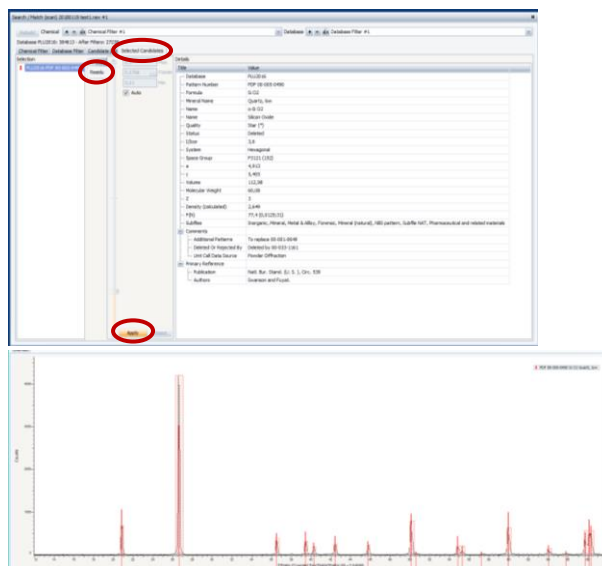
Repetition of steps 4 and 5.



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**.



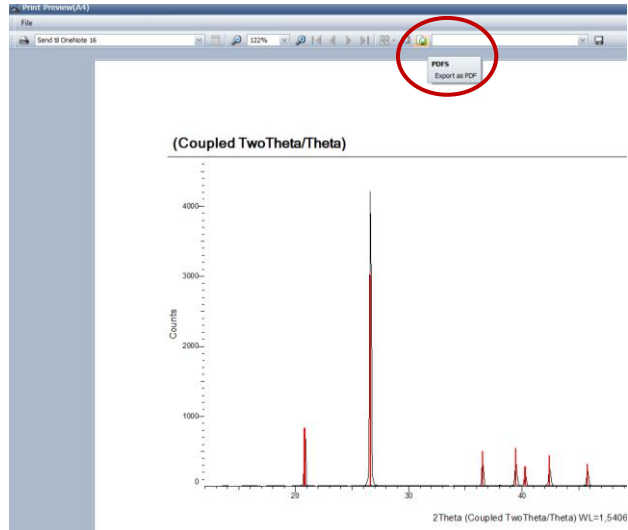
10 Repeat the steps 4 to 9 until you are satisfied that you found all minerals in your sample.

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.

7.5. Saving data

RAW files and .EVA files are saved in the folder *C:\DIFFDAT1*.

PDF files of the scans shall be saved in the folder *C:\Users* under a folder with your name (e.g. *C:\Users\ Waldemar Brøgger*). There is a shortcut to this folder in the computer desktop.

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.

7.6. Shutdown

Step	Examples/hints
1	When your last measurement is finished, open the glass window and remove the sample holder from the sample changer. Close the glass window .
2	Close the XRD Commander and EVA software. DO NOT turn off the computer.
3	Clean the sample holder(s) and any other material that you have used for sample preparation with ethanol and put all the material away in their respective drawers (room U 560 – Prep.4 (Sample preparation SEM, XRD, CT)).