Sample preparation and analysis

Introduction

In many cases powder samples are analyzed, but there is also the possibility of examining alteration layers or small pieces of stone, brick etc. without prior grinding. For more detailed identification of clay minerals, oriented aggregates (OA) are prepared, and for very small sample amounts there are zero-background sample holders (silicon crystal), which do not produce reflections by themself. As the samples do not suffer any alteration during X-ray analysis, they can be used afterwards for other types of analysis such as thermogravimetry, nitrogen sorption, electron microscopy, etc.

Powder Samples
- Earth
- Brick
- Mortar/Plaster
- Stone/Rock
- Alteration products

Oriented aggregates
- Clay fraction (<2 µm)

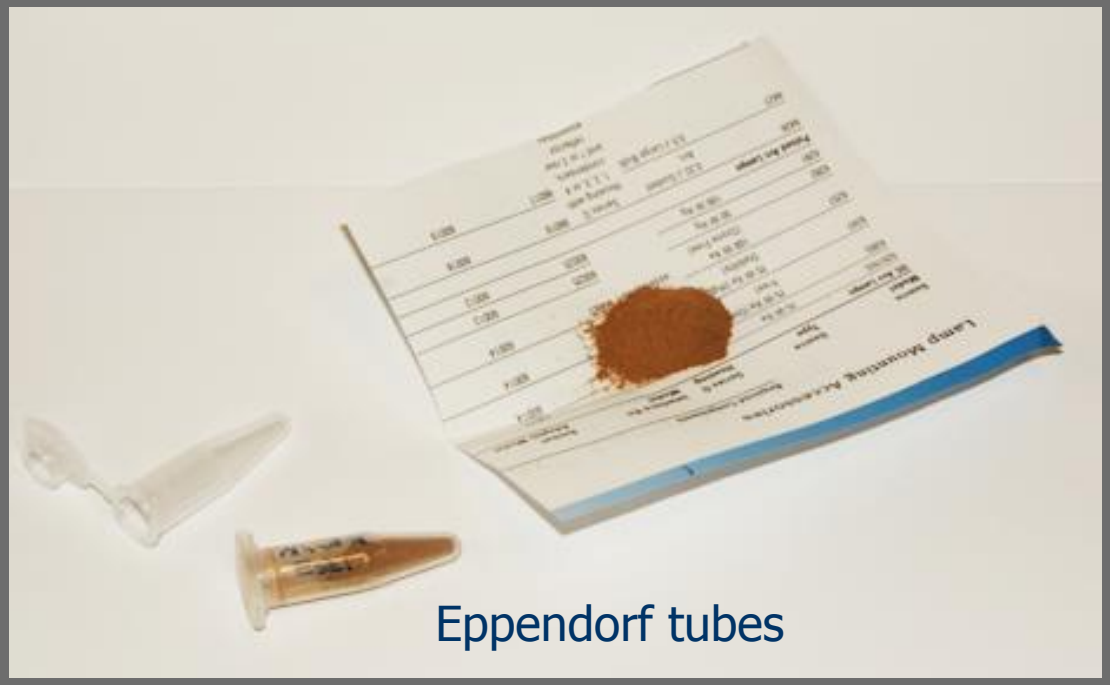
Solid samples
- Stone/Rock etc. (alteration layers)
- Paint

Preparation of powder samples

For the preparation of powder samples we need about 0.5-1 g of material (depending on the density of the sample). We have to grind the sample in an agate mortar until we obtain a fine and homogeneous powder.

Preparation of powder samples

We can store the samples in Eppendorf tubes with the help of a folded paper. The Eppendorf tubes will facilitate the loading of the powder sample holder (see below). Important: Do not forget to write down the name of the sample on the tube.



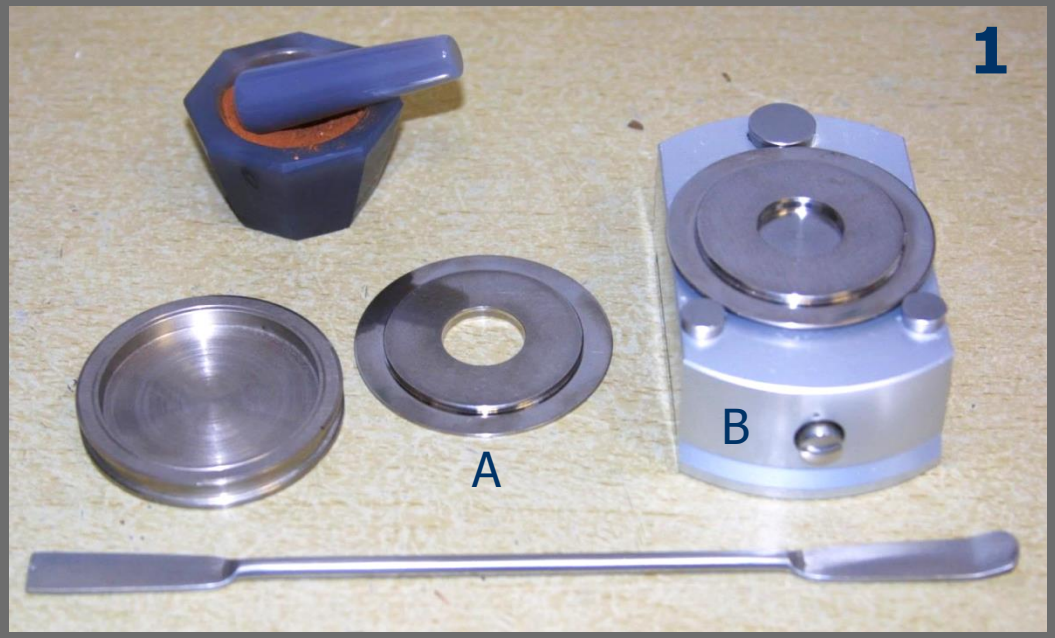
Eppendorf tubes

Preparation of powder samples

In the case of minerals with considerable hardness, we use a hammer to crush the sample prior to grinding in the agate mortar. The sample is placed in a plastic bag and wrapped in newspaper or the like so as not to damage the bag during crushing.

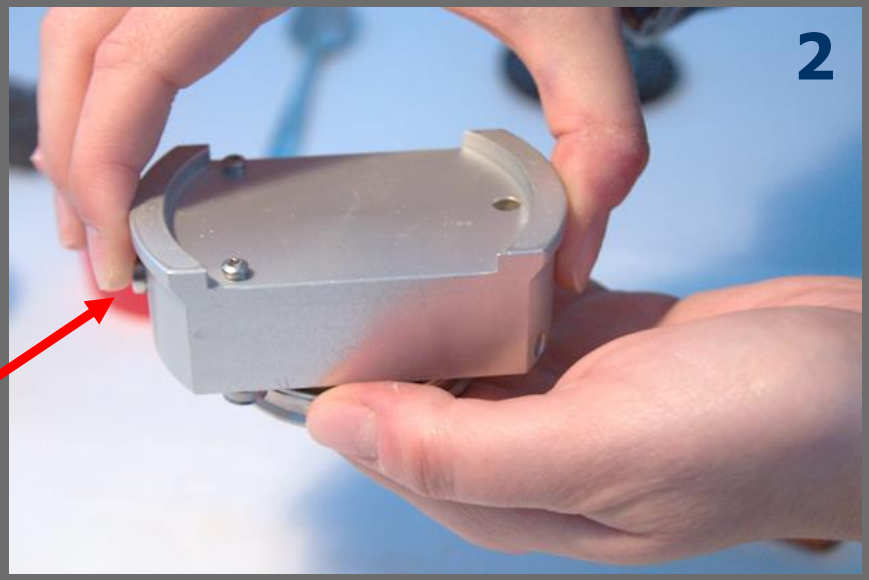
Preparation of powder samples

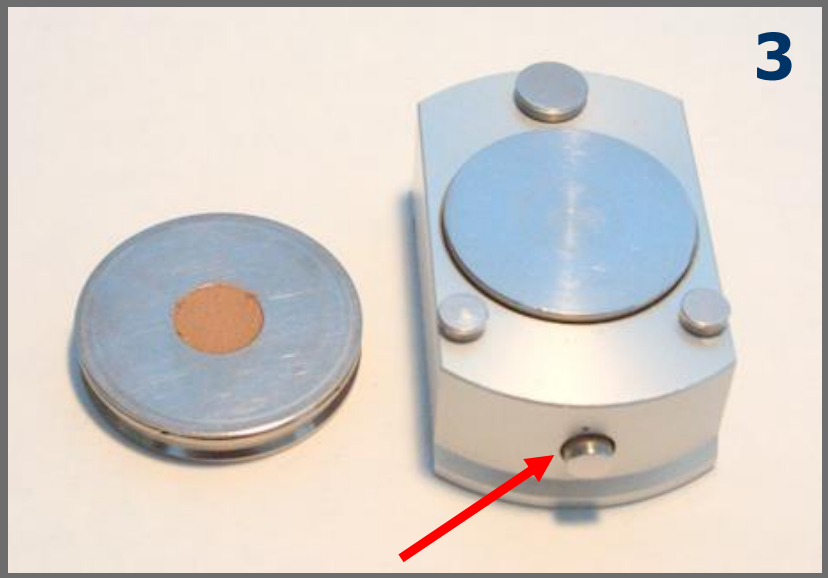
Image 1 shows all parts required for the preparation of powder samples via rear loading. Rear loading is used to avoid preferred orientation of the crystals in the sample. First we put the ring (A) in the support (B). Fill the ring with the powder sample (2), with the help of a spatula if necessary. Important: All parts must be cleaned between samples using alcohol to avoid contamination.



Preparation of powder samples

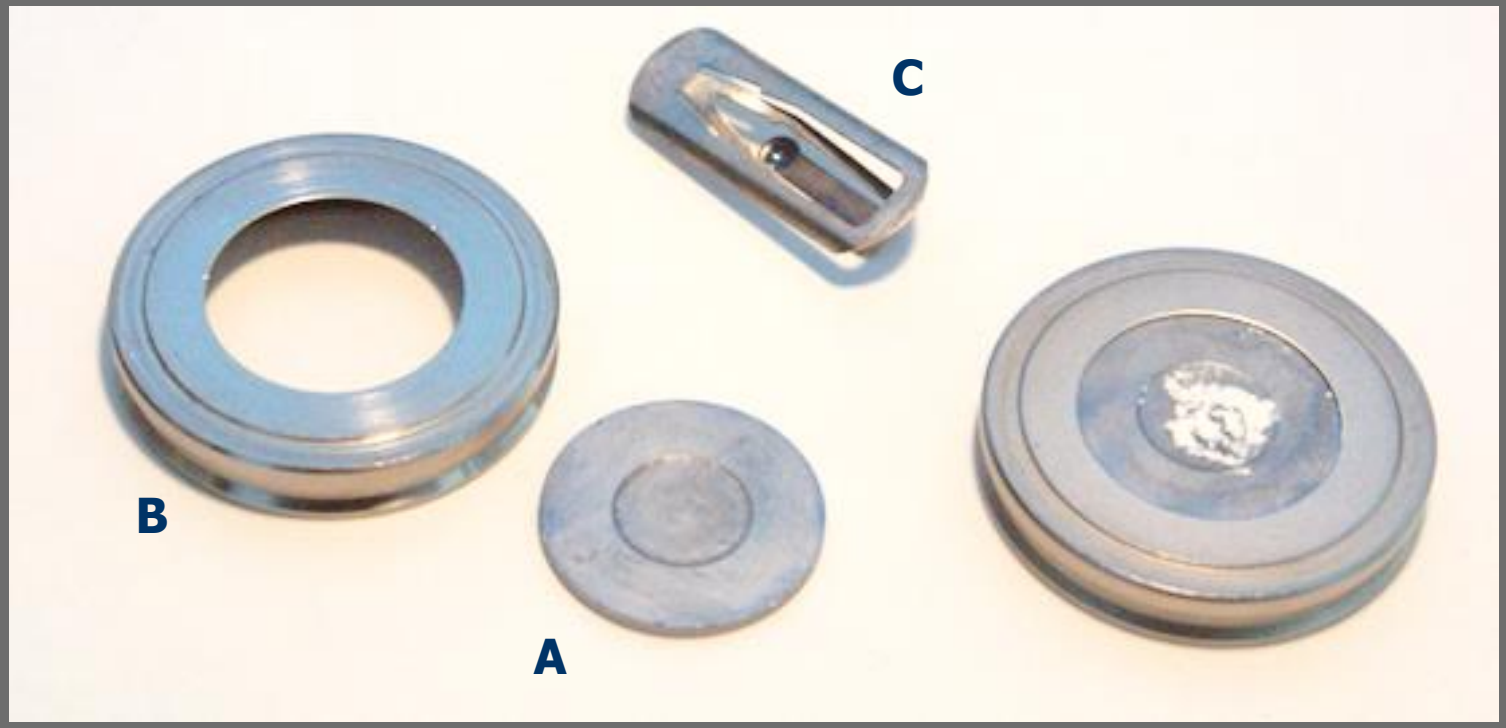
The sample holder is closed (1). The assembly is carefully turned over and the button (arrow) is pressed to release the sample holder (2). Image (3) shows the sample ready for analysis.





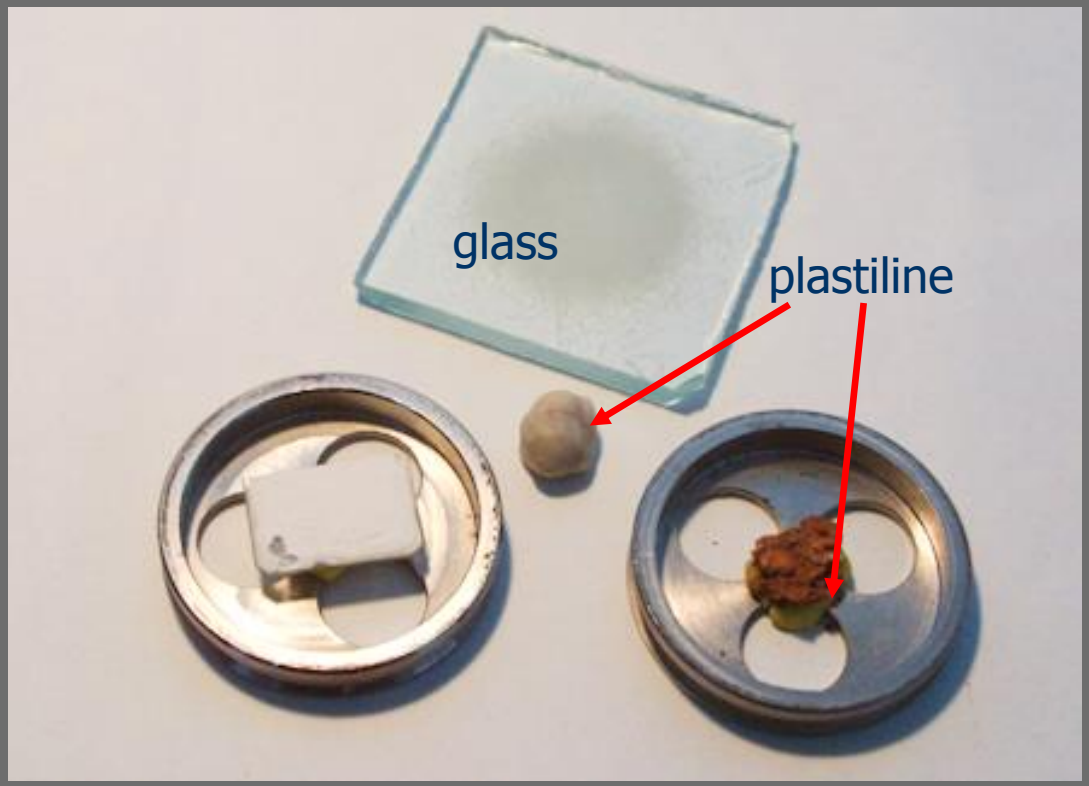
Preparation of powder samples

For very small quantities of powder samples zero-background sample holders (silicon crystal) can be used, which do not produce any reflections by themselves. The zero-background (A) is held in the sample holder (B) by a clip (C). A spatula is used to homogeneously apply the sample on the zero-background sample holder.



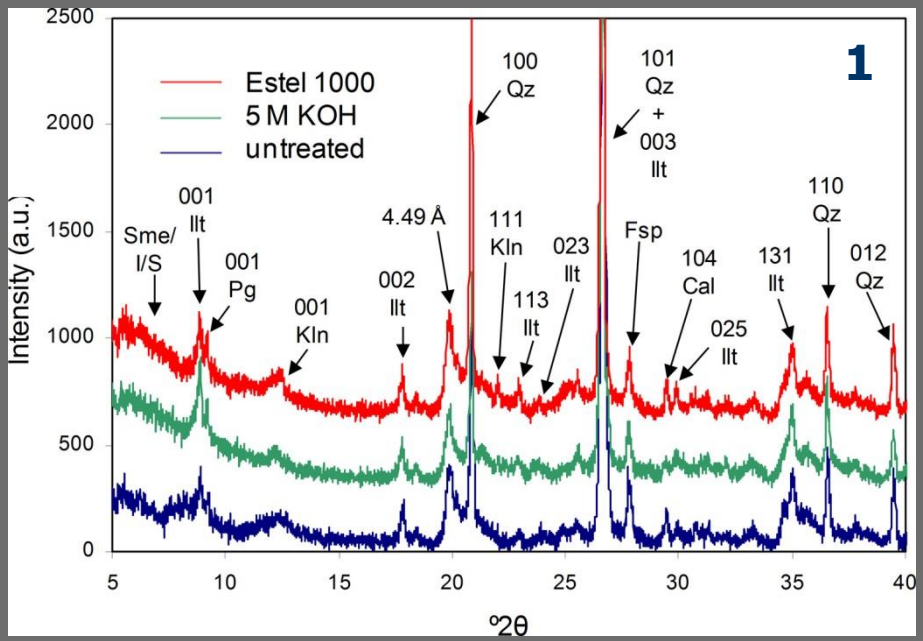
Preparation of solid samples

In some cases we cannot prepare powder samples, for example when the intact sample is required for further analysis or because the samples are covered with thin alteration layers, which can not be separated easily. In this image we see sample holders for solid samples. Samples are hold in place with plasticine during analysis. A glass slide is used to level the sample and, thus, ensure that it is at the correct height. The incorrect height of the sample can cause a shift of its peaks of the diffractogram.

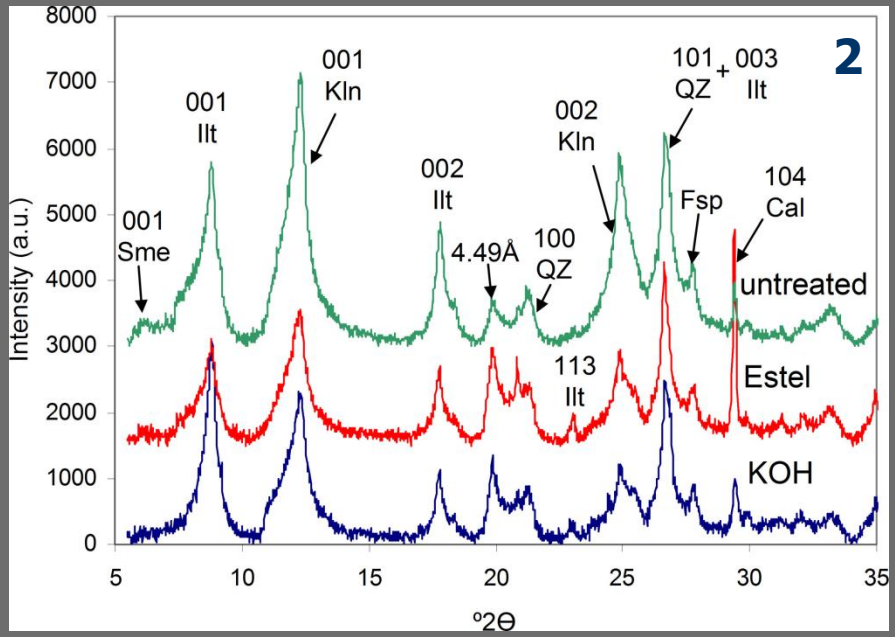


Preparation of oriented aggregates (OA)
¿What are they used for?

We generally use OAs for the study of clay minerals by analyzing the <2 μm fraction of the sample (adobe or rammed earth, low-temperature bricks) or to determine possible causes of deterioration of stone materials with high clay content. In image (1) we see diffractograms of three powder samples (soil from the Alhambra Formation treated with different consolidants) and (2) diffractograms of the clay fraction of the corresponding samples. Due to the preferred orientation of the clay minerals along their basal planes, the intensity of 001 peaks increases in image (2).



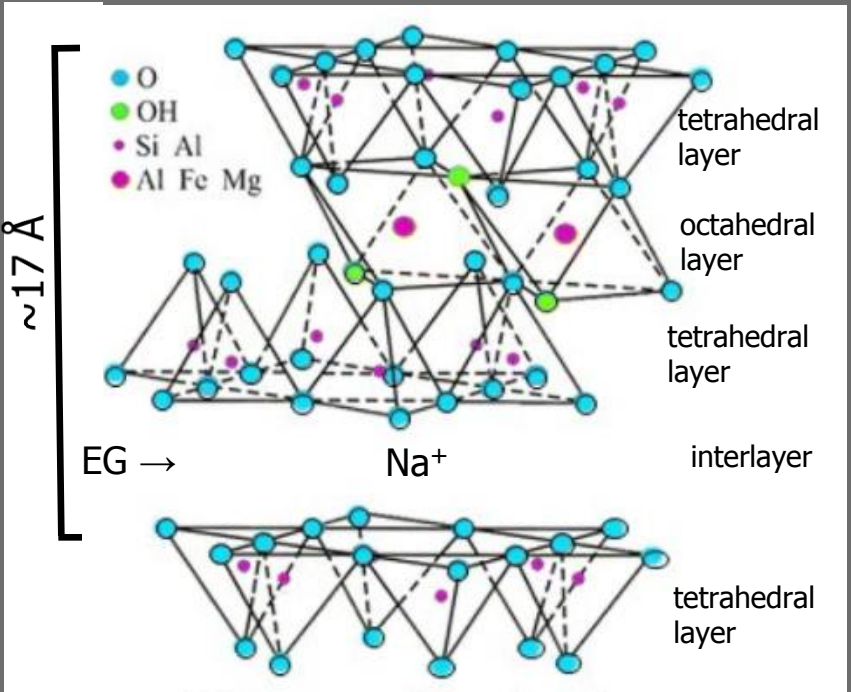
1) XRD of powder samples (Alhambra Formation)

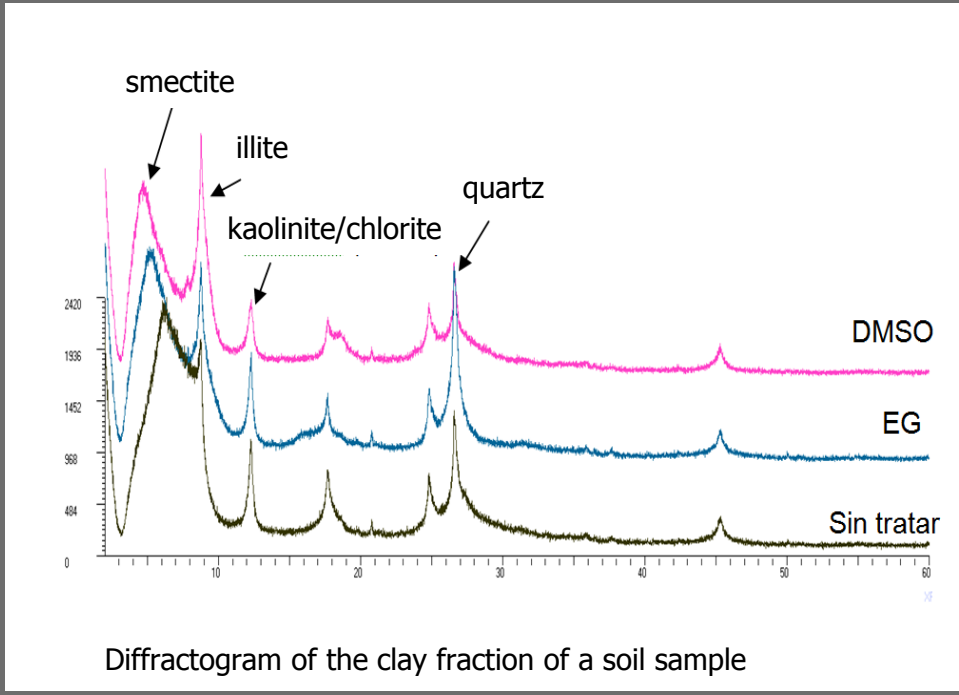


2) XRD of oriented aggregates (<2 μm) (Alhambra Formation)

Oriented aggregates (OA) – Treatments

To study the characteristics of the clay minerals in more detail, you can treat OAs with ethylene glycol (EG) or dimethylsulfoxide (DMSO) to provoke swelling of smectites (causing an increase in the d_{001} spacing from 13-15 Å to ~17 Å (EG) and ~19 Å (DMSO)), or perform a heat treatment (550 ° C), causing the collapse or degradation of some clay minerals (shift or disappearance of peaks).



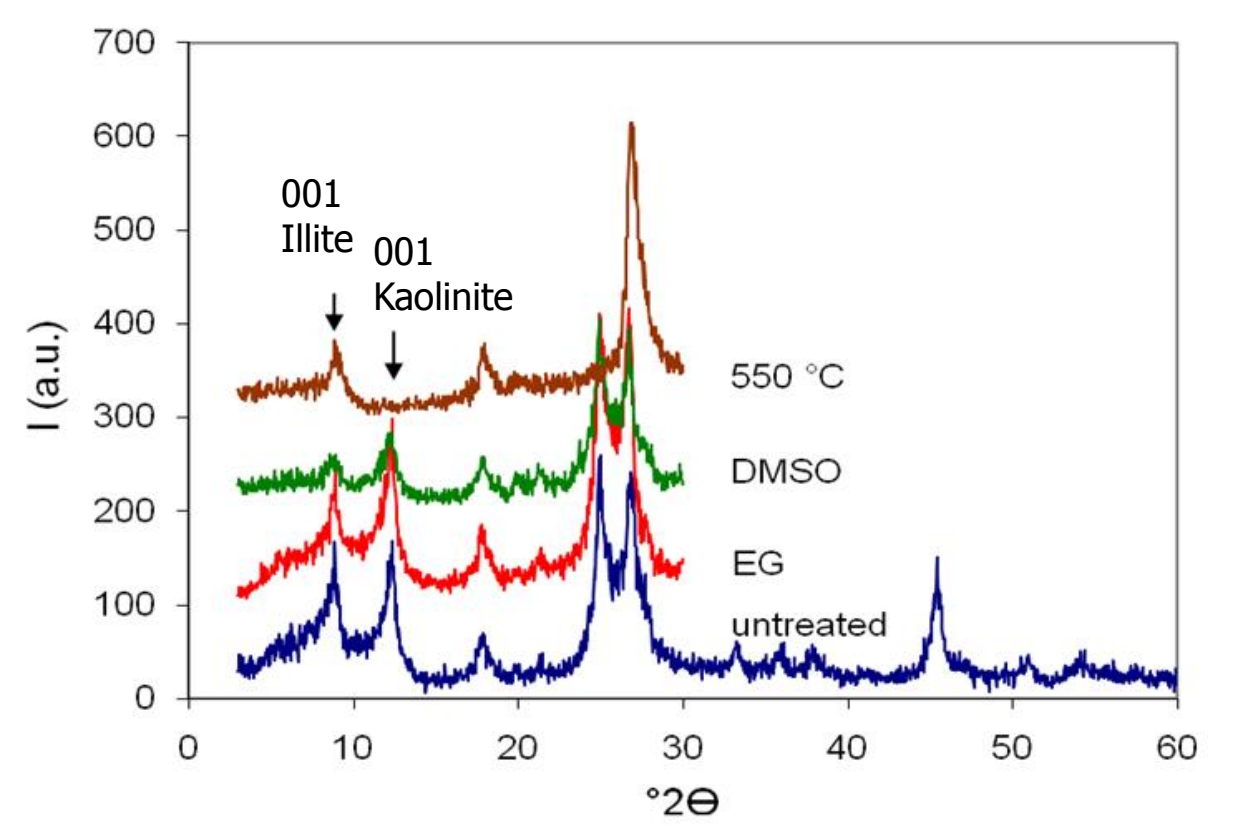


Diffractogram of the clay fraction of a soil sample

More information: D.M. Moore y R.C. Reynolds, X-ray diffraction and the identification and analysis of clay minerals, Oxford University Press, New York, 1989.

Oriented aggregates (OA) - Treatments

Heat treatment (550 ° C) causes the disappearance of kaolinite peaks (brown line) because kaolinite undergoes dehydrolyzation and becomes amorphous (transformation to metakaolin).



More information: D.M. Moore y R.C. Reynolds, X-ray diffraction and the identification and analysis of clay minerals, Oxford University Press, New York, 1989.

Preparation of oriented aggregates (OA)

Laboratory No. 28, Dept. of Mineralogy and Petrology

To separate the clay fraction (<2μm), the ground sample is dispersed in water. Separation is done by sedimentation (the heavier and larger particles settle faster). To increase the rate of sedimentation, centrifugation is used with a predetermined program (the time required for sedimentation can be calculated by applying Stokes' law). The clay fraction is separated by decantation. The dispersion of the particles can be facilitated by eliminating carbonates (using dilute acetic acid) and organic matter (using H_2O_2), by applying ultrasound and by adding calgon (sodium hexametaphosphate).

Preparation of oriented aggregates (OA)

The dispersion of the clay fraction is applied on glass sample holders with the help of a disposable pipette. OA should be dried slowly under ambient conditions. Important: Do not forget to write the name of the sample on the back of the glass sample holder.

Preparation of oriented aggregates (OA)

Once dry, the glass sample holders are fastened to the sample holder ring using a clip (1). Image (2) shows the OA ready for analysis.

XRD analysis of samples

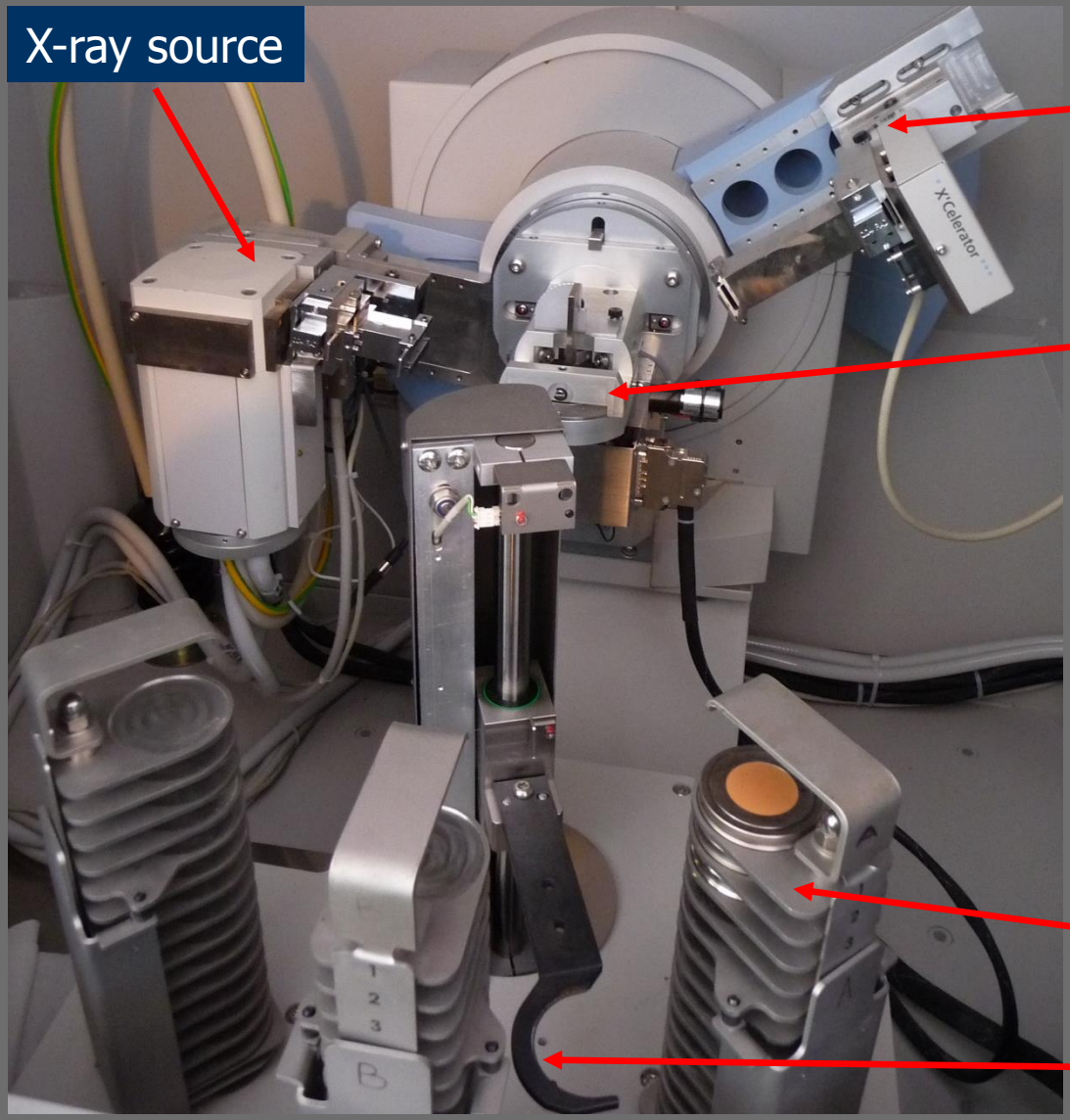
Diffractometer PANANALYTICAL X'PERT PRO at the Dept. of Mineralogy and Petrology

Equipment configuration:
Radiation: Cu-Kα
Wavelength(λ):
1.5405 Å
Filter: Ni
Voltage: 45 kV
Intensity: 40 mA

(this information has to be included in the «Materials and method» section of a TFM or scientific articles, together with the exploration range and the goniometer speed)

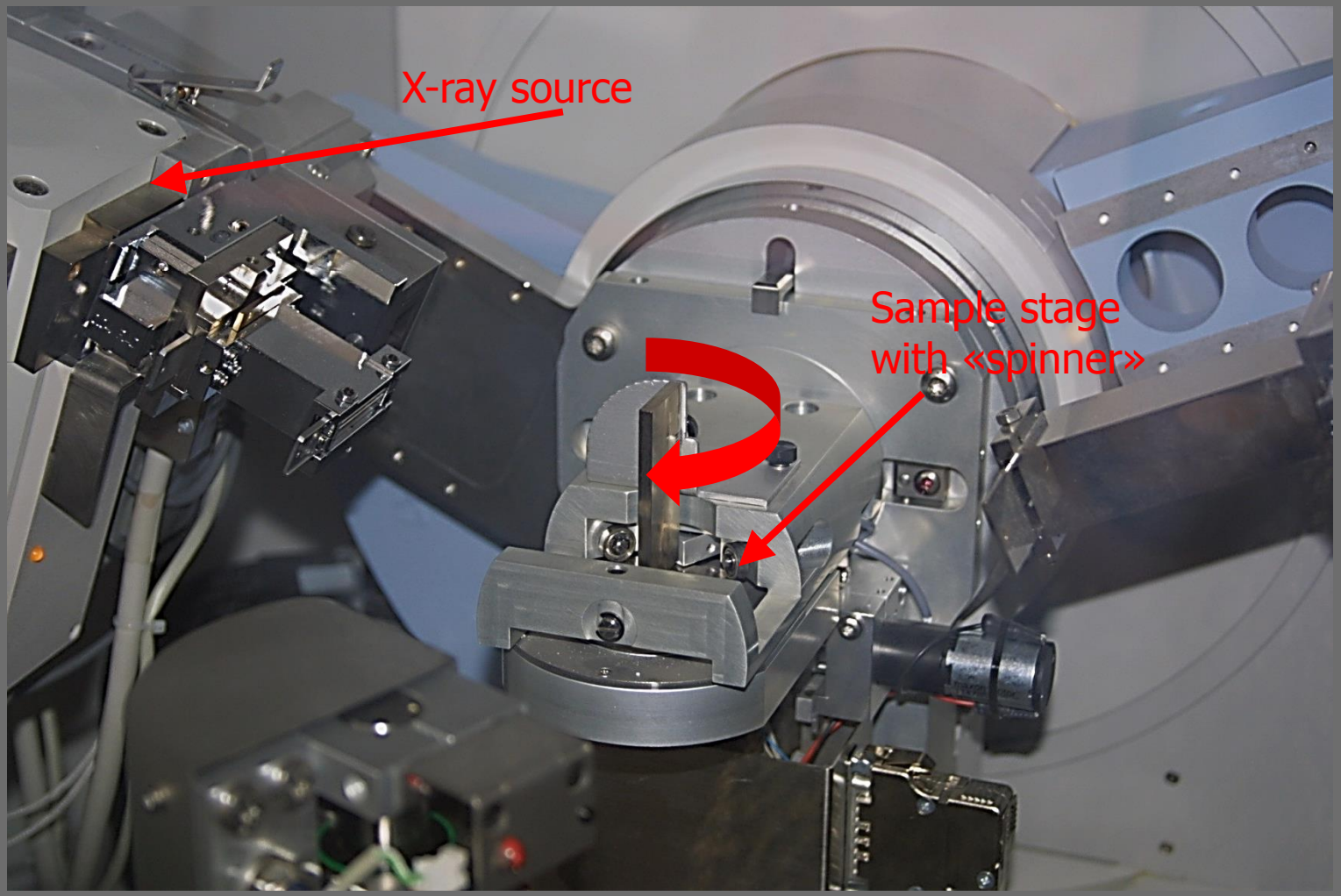
XRD analysis of samples

The equipment has an automatic charger system for up to 45 samples.
The sample changer arm takes the sample to the sample stage.



XRD analysis of samples

The equipment has a spinner, which rotates the sample in order to increase the possibilities that crystals are oriented adequately to enable diffraction.



XRD analysis of samples

The control of the equipment and the programming of the analysis is done via computer. The "HighScore" software program for the analysis of the XRD data is also installed on this same computer.

XRD analysis of samples

To facilitate the interpretation of the diffractograms, they must have a good quality, which depends to a great extent on the analysis parameters (recording speed and angular resolution). There are numerous predetermined programs, which can be chosen depending on the required quality of the analysis.