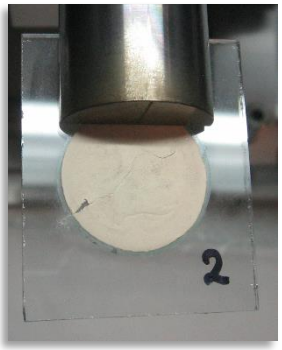
Explorer

Clays and clay minerals

Introduction

Clays consist mainly of clay minerals, sandy-silty, oxide, sulfate and amorphous fractions.

Clay minerals are an important group of minerals (phyllosilicate), because they are among the most common products of chemical weathering, and thus are the main constituents of the fine-grained sedimentary rocks called mudrocks. Clay minerals are the main constituent of soils: their understanding is important from an engineering point of view, as some minerals expand significantly when exposed to water. Clay minerals are also used extensively in the ceramics industry and are thus important economic minerals[1].



Identification of clay minerals requires acquisition of diffraction pattern starting at low angles ($2\theta < 5$ deg.) in order to collect basal reflections at very large d-spacings (e.g. Smectite, Chlorite groups)[2].

Quantitative methods by XRD are not straightforward and usually "clay fraction" separation is required to identify correctly the type of clay minerals in a sample[3]. Nonetheless, in last years several methods have been proposed to quantify the content of mineralogical samples, among which standard calibration curves and Rietveld refinement are the most important. It must be pointed out that complimentary analytical techniques (e.g. XRF, TGA) are highly beneficial to support phase identification and quantification, therefore they should be used when available.

In this note we show how Explorer and Rietveld refinement can quantify a mixture containing clay minerals with large d-spacing reflections (i.e. low angle peaks), provided a good starting structural model is available.

Summary

Clays consist mainly of clay minerals, sandy-silty, oxide, sulfate and amorphous fractions.

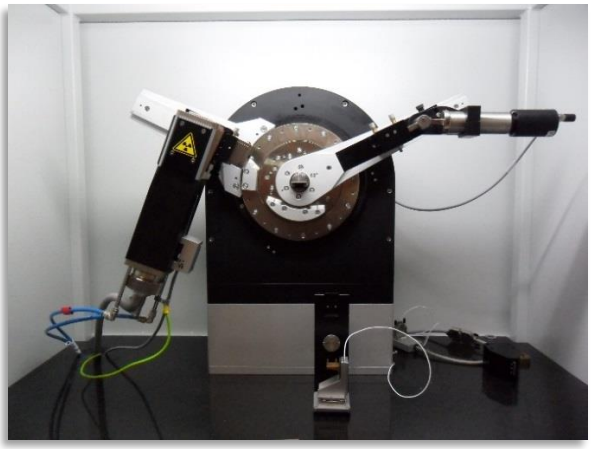
In this note we show how the Explorer diffractometer, coupled with Match! software and Rietveld refinement, can succeed in quantifying a mixture containing clay minerals with large d-spacing reflections (i.e. low angle peaks), provided a good starting structural model is available.

[1] http://www.tulane.edu/~sanelson/eens211

[2] A laboratory Manual for X-Ray Powder Diffraction, USGS, http://pubs.usgs.gov/of/2001/of01-041/

[3] F. Bergaya, B.K.G. Theng and G. Lagaly, Handbook of Clay Science, Developments in Clay Science, Vol. 1 2006 Elsevier.

Product Specifications



Explorer is a Multi-Purpose - Theta/Theta - high resolution diffractometer which, thanks to its direct drive torque motors, offers top performances in many analytical areas, ranging from phase analysis to determination of microstructural properties on bulk or thin film materials.

Thanks to its modularity and the wide range of accessories and attachments available, EXPLORER allows to perform measurements in different configurations: traditional X-Ray Powder Diffraction (XRPD), Reflectometry (XRR), Grazing Incidence Diffraction (GID), High Resolution X-Ray Diffraction (HRXRD), Total Reflection X-Ray Fluorescence (TXRF), Residual Stress and Texture X-Ray Diffraction.

Explorer high resolution diffraction system incorporates the high efficiency of the direct drive torque motors controlled by optical encoders, allowing to reach an angular accuracy of 0.00001°.

Sample Preparation and Measurement Parameters

Powder sample was ground in a mortar and then gently pressed in a flat Al sample holder with a 1 mm cavity. The holder was then mounted on a spinner set at a speed of 60 rpm.

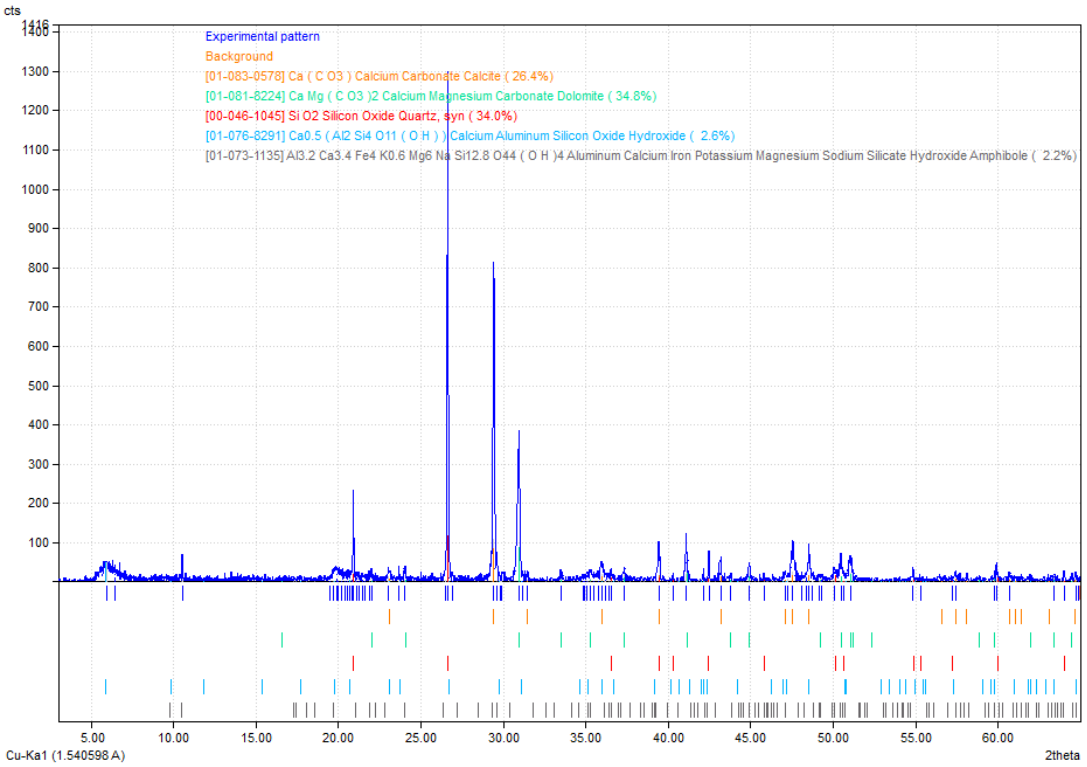
Goniometer radius [mm]:	200
X-Ray Source:	Cu LFF
Power [kV; mA]:	40;30
Divergent slit[°]:	0.5
Anti-scatter slit[°]:	0.5
Soller slit (x 2) [°]:	2.3
Receiving slit [mm]:	0.1
Secondary monochromator:	Graphite
Detector:	Point scintillation detector
Step[°]:	0.02
Time/step [s]:	2

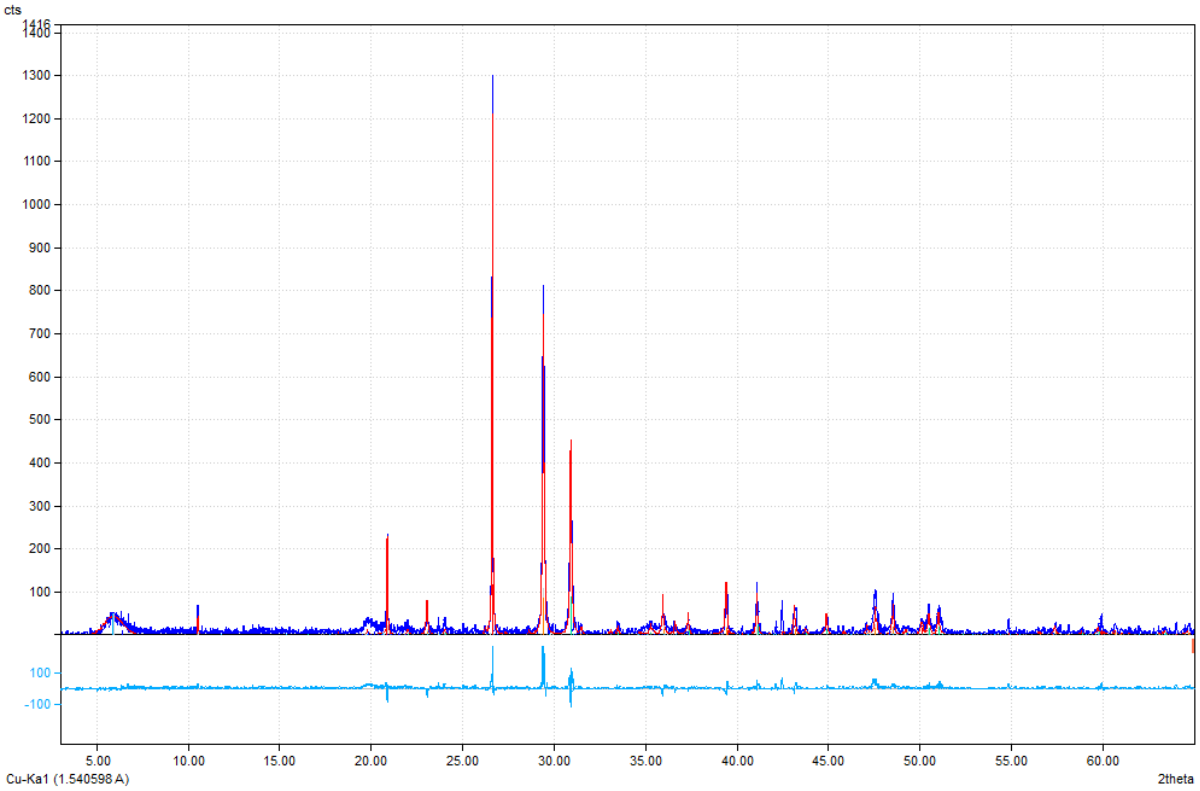
Results

Search and Match procedure was performed by Match! software and ICDD PDF-4 Minerals database; quantitative analysis was done by Rietveld refinement through FullProf software.

Resulting Reduced Chi-square is ~3.

Phase	Formula	Estimated Content [%]
Quartz	SiO_2	34±1
Calcite	$CaCO_3$	26±1
Dolomite	$CaMg(CO_3)_2$	35±1
Montmorillonite	$Ca_{0.5}(Al_2Si_4O_{11}(OH))$	2.6±1
Amphiboles	$Al_{3.2}Ca_{3.4}Fe_4K_{0.6}Mg_6NaSi_{12.8}O_{44}(OH)_4$	2.4±1





Conclusions

Phase identification and quantification of a clay sample have been carried out thanks to the Explorer diffractometer and properly conditioned-Rietveld software. The possibility of starting the acquisition at low scattering angle (2θ< 5 deg.) assures that large d-spacing clay minerals can be identified.

About GNR SRL

With 30 years of technological experience, GNR is a worldwide market manufacturer of advanced analytical instruments in Optical Emission Spectrometer and XRD / XRF domain, developing procedures of analysis for various applications, supplying the corresponding laboratory equipment and providing consulting and customer support worldwide.

GNR can rely on a well-established team of highly qualified researchers and technicians, supported by the cooperation with leading University departments, which ensures a constantly updated technological growth.

GNR is present on the main international markets through an efficient and motivated technical and commercial network, able to provide outstanding support for any customer requirements.