

XRD

The most important mineral characterization tool

Gilles Mertens



What we will cover

Introduction of Qmineral

Applications of mineralogical characterization

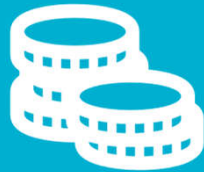
Tools for mineralogical characterization, focus XRD

Case studies

Practical information



Europe (Belgium) based



Commercial lab



Global client base



7 colleagues



Material Test Laboratory



Analytical Experts

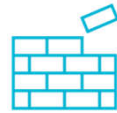
Mineralogical characterization is used in various situations

Examples



Deleterious components

Reactive minerals in raw materials for Concrete



Construction issues

Efflorescence on bricks



Occupational hygiene

Air samples in workplaces



Secondary raw materials

Slag characterization



Primary raw materials

Ballast free of crystalline silica



Exploration

Correlation between boreholes



Process optimization

Scalings in pipelines



Quality control

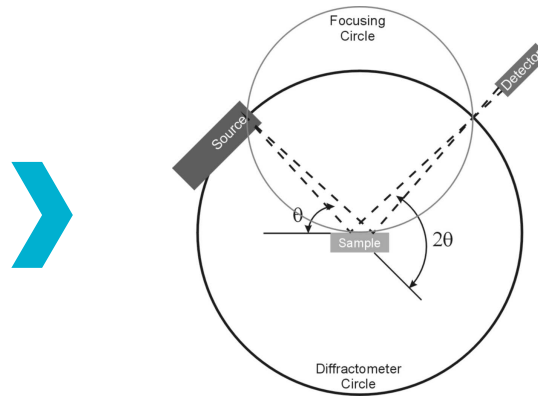
Purity of extracted minerals

XRD probes at the crystal structure of minerals

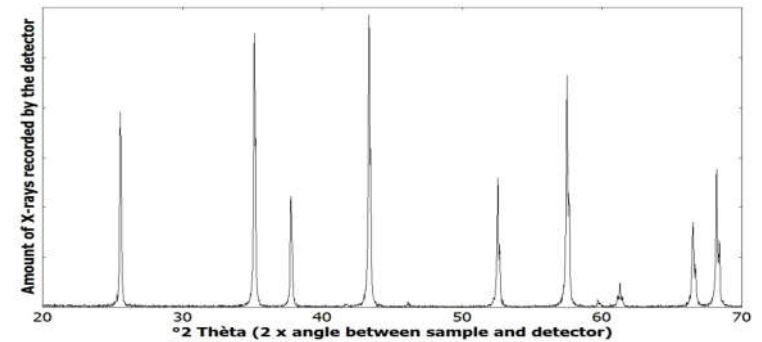
Flow of an XRD measurement



X-rays are produced by an X-ray tube



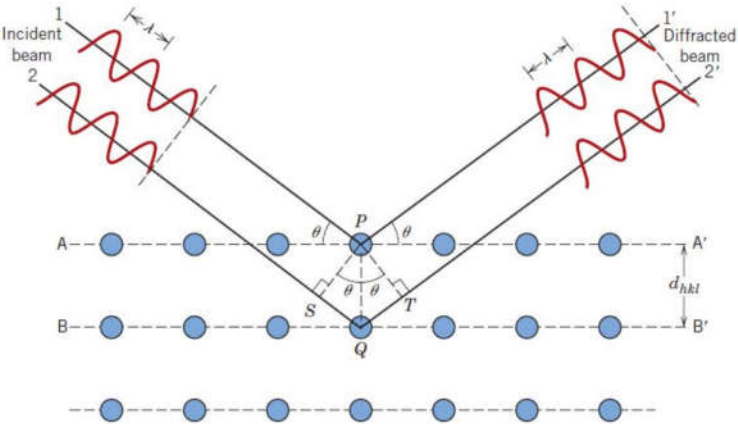
These X-rays are scattered by the samples



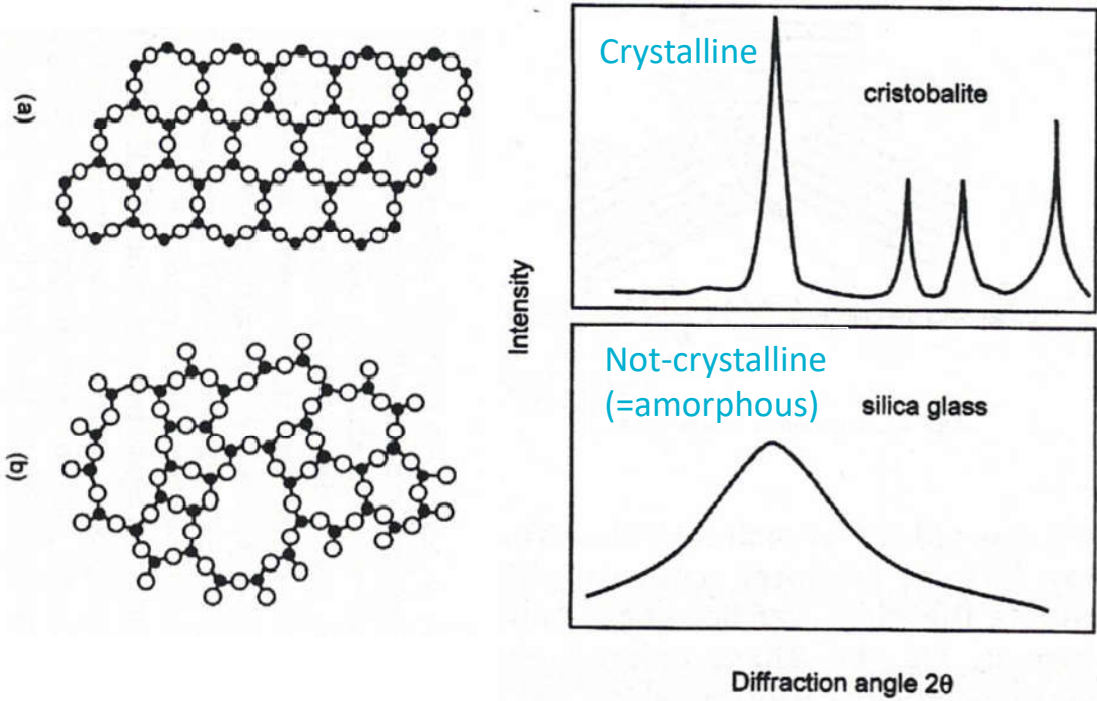
Reflected X-rays are collected by a detector

The reflection of X-rays at the level of the atoms/crystals in the sample return a diffraction pattern

X-rays reflect at the level of atoms/crystals...

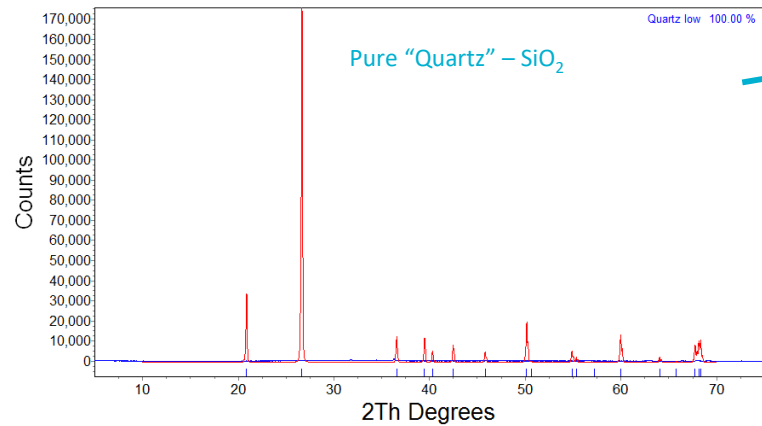
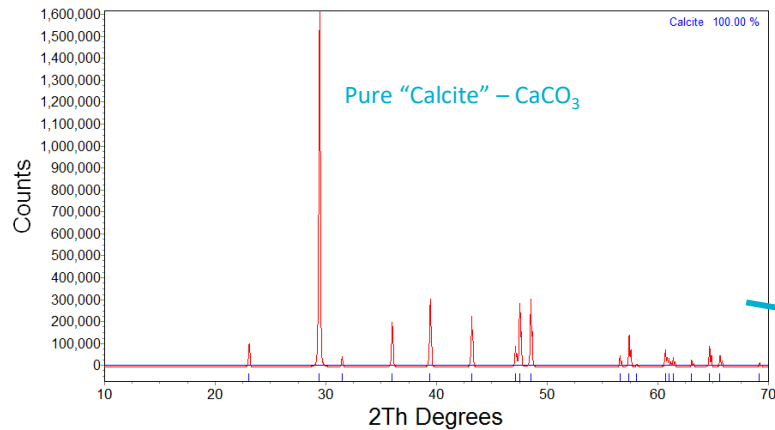


...returning a peak-pattern for crystalline materials

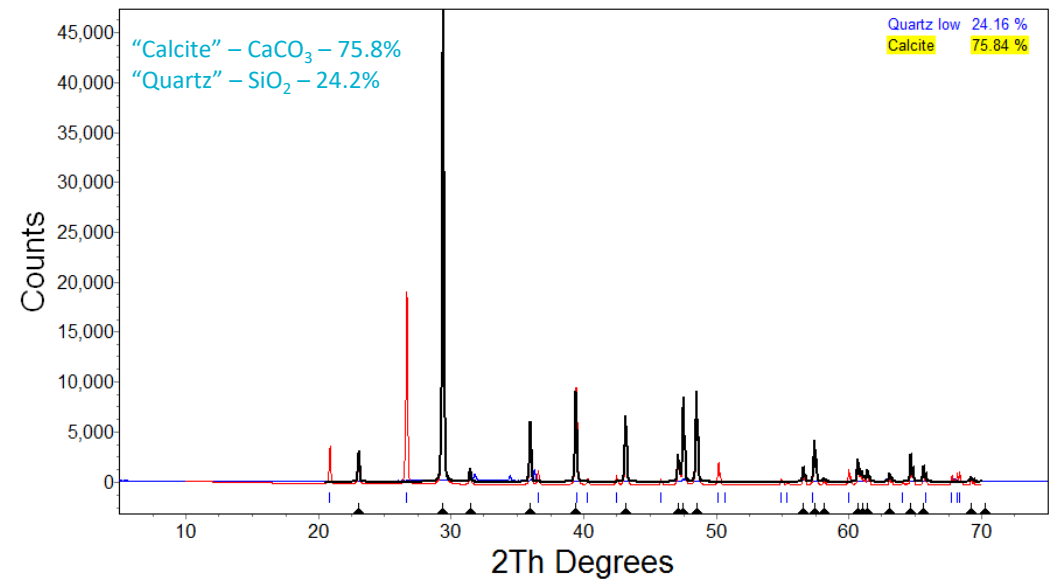


In a sample with several phase, the diffraction peaks from both phases are visible and can/will overlap

XRD patterns of pure compounds



XRD pattern of unknown sample



XRD measurements give you a wide range of information

Identification

Which minerals or phases occur in the sample?

Quantification

What is the concentration of each mineral in the sample?
Most common technique for quantification is Rietveld

Amorphous material

Is there an amorphous phase in the sample?

Crystal size

What is the crystal size of the mineral/phase?

Strain

What is the concentration of defects in crystal lattice?

Crystal lattice

What is the size of the crystal lattice?
What is the composition of the minerals?

With XRD, you can measure solid, inorganic materials

Most common types of samples

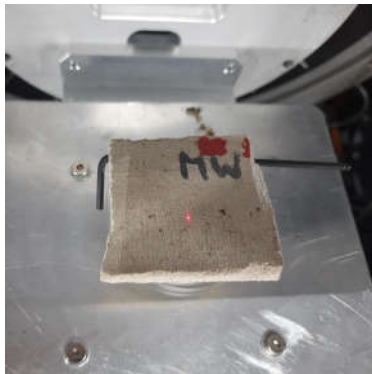
Powders
(mg-size)



Rocks,
ground into
powder
(sample
prep.!)



Large
samples, as
such



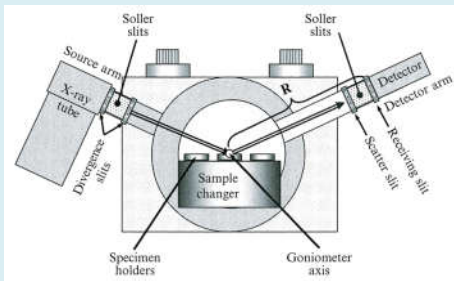
Filters





Type that is being discussed in this presentation

There are different types of X-ray diffraction



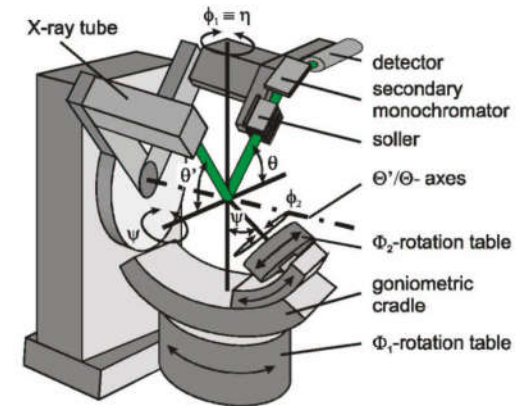
X-ray powder diffraction

characterization of powders



Single crystal diffraction

Determination of crystal structure (of new minerals: e.g. pharmaceuticals)



Texture analyses

Looking at orientation of crystals in solid samples (e.g. in plates of steel)

XRD is the main tool for mineral characterization

Results and techniques used in the Reynolds cup 2020



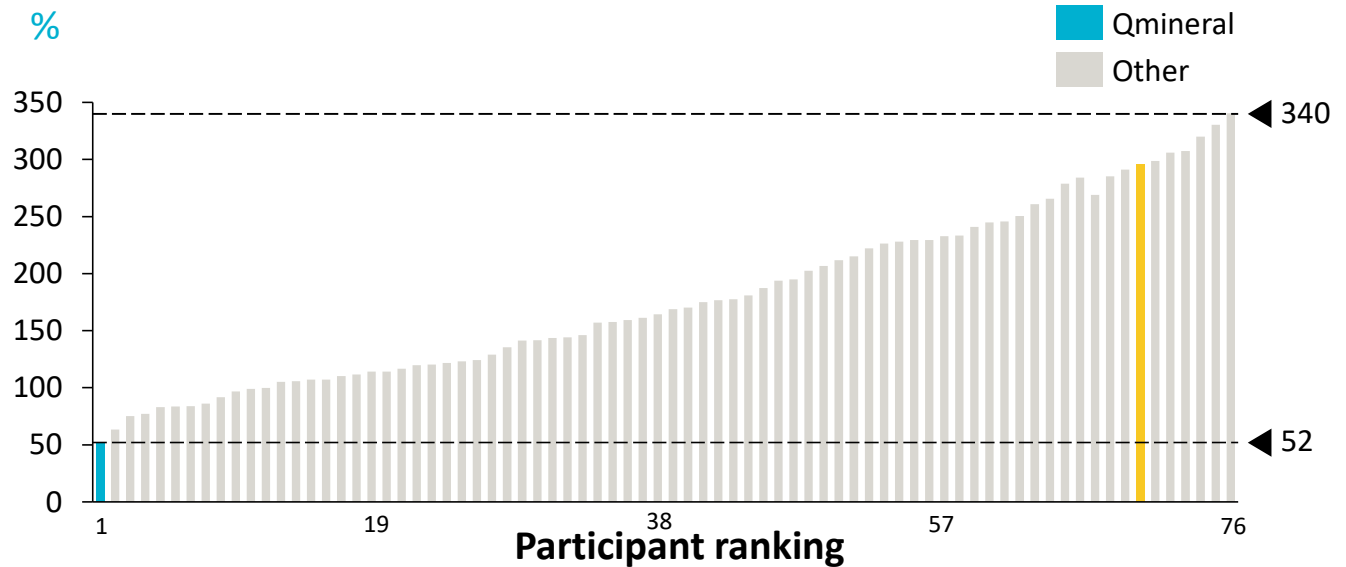
Reynold Cup

World Championship for Mineralogical Quantification

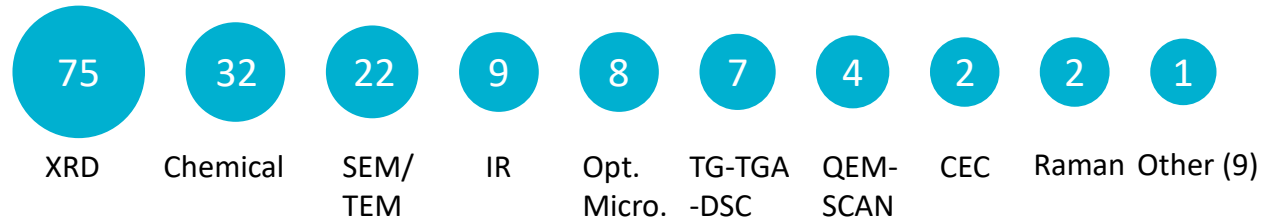
3 samples – What are the minerals present and what is their concentration in each mixture?

- all techniques are allowed
- 75-100 labs over the whole world participate
- After the results are returned: the errors are summed
- Summing up the bias for each mineral gives the total bias.
- The one with the lowest score wins the Cup

Reynolds Cup results | Total bias by participant



























Techniques by number of labs (some use more than 1 technique)



XRD is the leading tool for mineral characterization

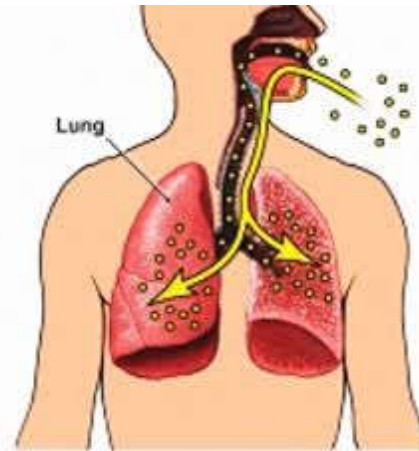
Alternative techniques can be interesting in certain applications

 partially useful
 useful

Application	XRD	SEM/TEM	FTIR	XRF	QEM Scan/MLA	RAMAN
Mineral Identification						
Mineral Quantification						
Composition of individual minerals/amorphous phases						
Crystallite size						
Strain in crystallites						
Mineral/Grain shape						
Spatial association						
Characterizing one particle in a rock sample						

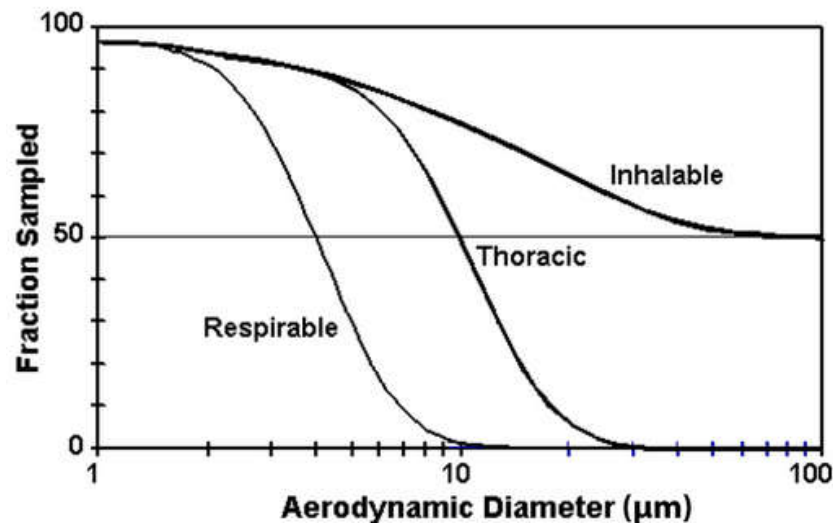
Case Study – Occupational Hygiene

Very fine dust can be generated at the workplace, and may enter the lungs of workers



Gravimetric measurements indicate the grain size of the fine dust

Fine Dust

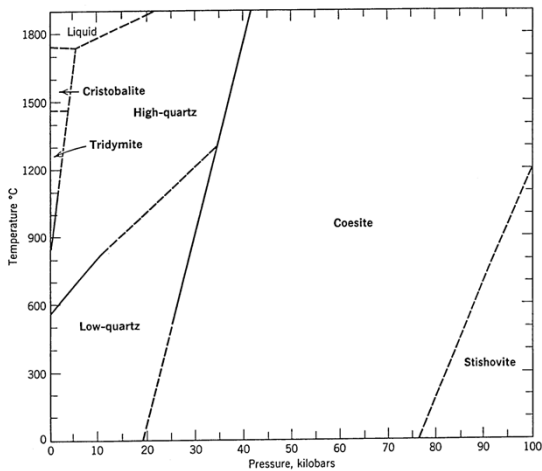


Type of fine dust

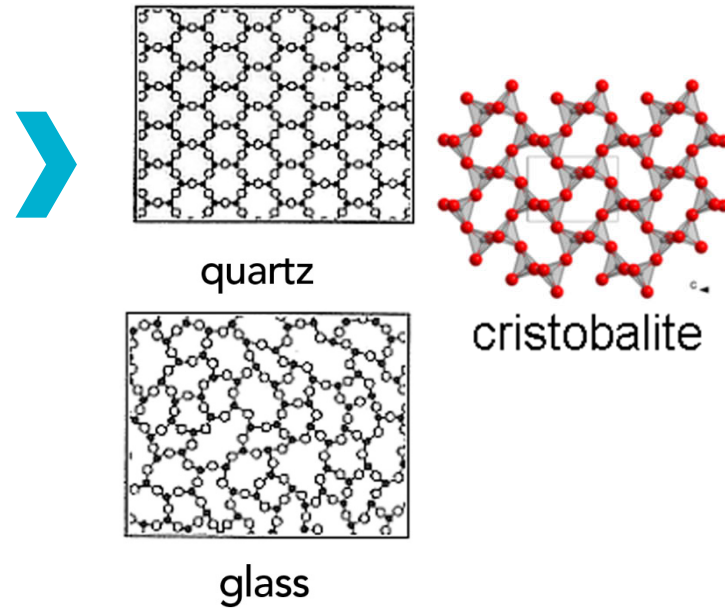
- Inhalable
- Thoracic
- Respirable

XRD measurements can bring additional insights in the harmfulness of fine dust

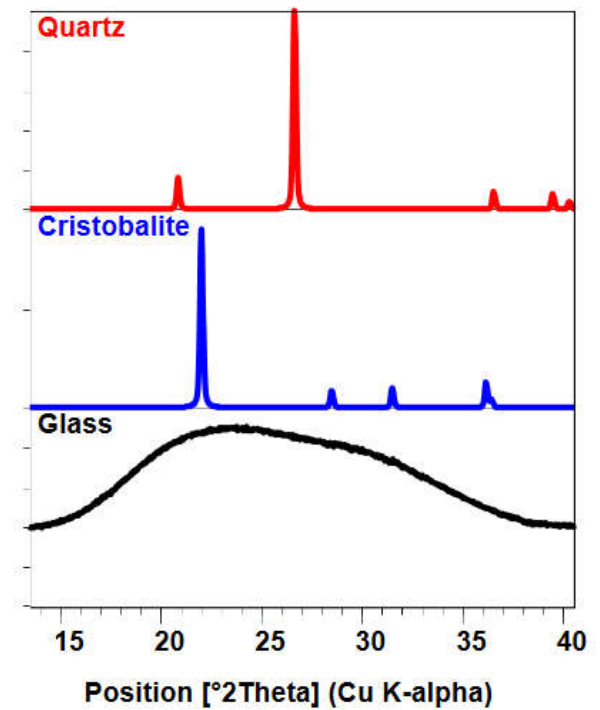
Crystalline forms of silica are particularly harmful



Different polymorphs of SiO₂ have different crystal structures...



... and hence different diffraction patterns

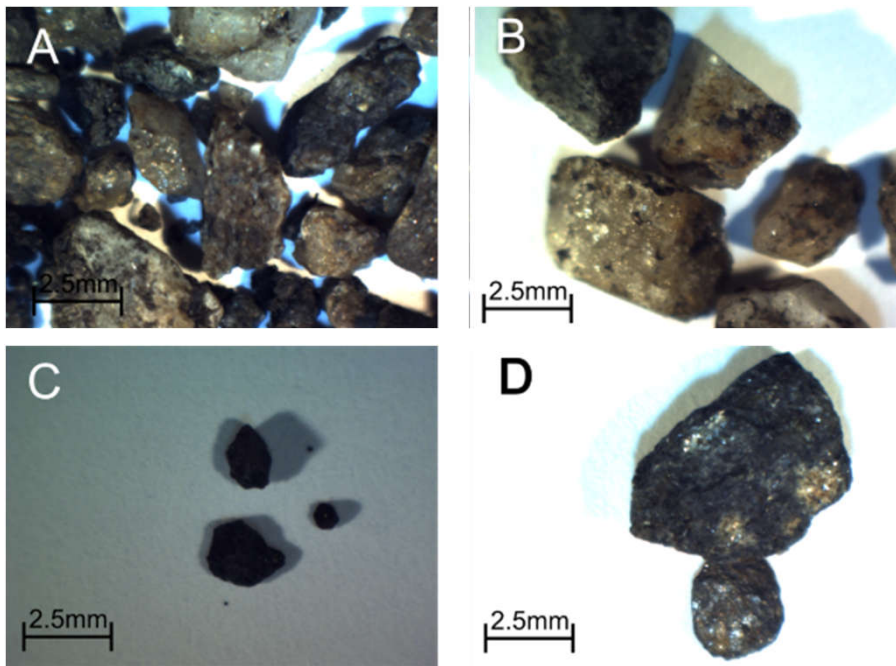


Case Study – Origin of contamination in jam

Contaminant particles were found in jam...



Optical microscopy shows the hard pieces are natural stones... pointing in the direction of to the fruit growers



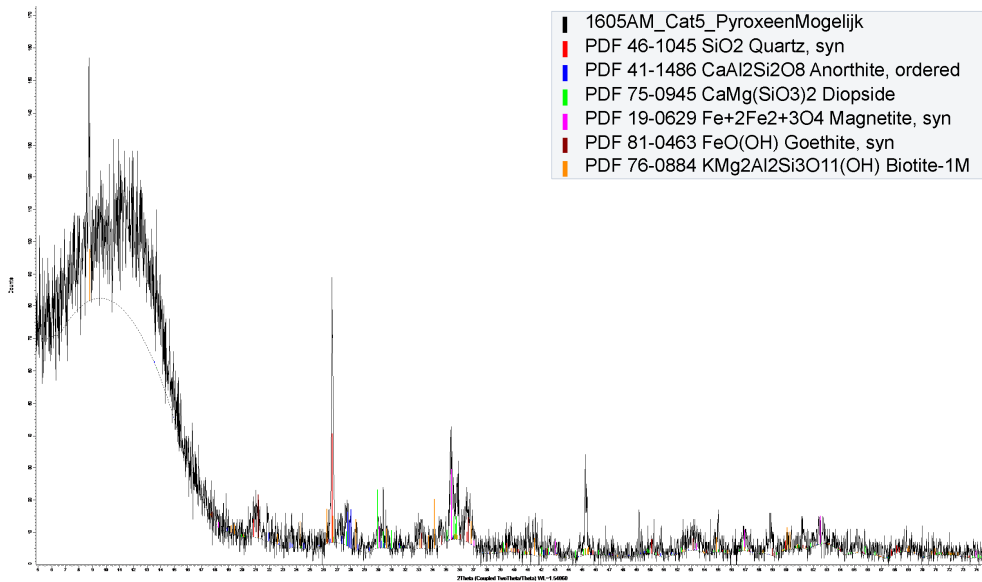
3 potential fruit growers were looked upon

- Lublin area in Poland
- Malemort-sur-Corrèze in France
- Leskovac area in Serbia

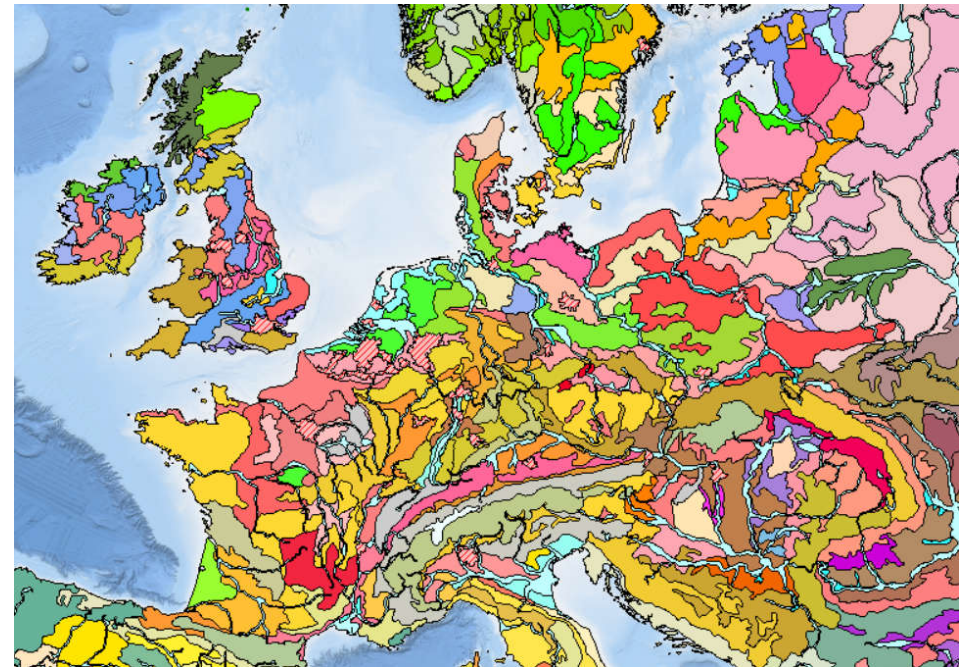
Images under the binocular of the some grains. A/ General view. B/ Quartz-particles. C/ dark particles. D/ Mica-rich grains.

Mineral identification through XRD and geological mapping give more insights

XRD pattern, indicating the mineral composition



Geological map, indicating mineral occurrence



Practical information

Flexible sample analysis conditions

SAMPLE PREPARATION

- samples **prepared and measured by Qmineral personnel**
- samples **prepared by PhD student and measured at Qmineral**

DATA TREATMENT

- Data treatment (e.g. mineral quantification) **by PhD student**
- Data treatment **by Qmineral personnel**



Qmineral
Gaston Geenslaan 1
3001 Heverlee



info@qmineral.com



016 75 13 77