



Natural Materials Analytical Research Laboratory



We serve the EERC by analyzing solid materials:

- *Coal, rock, metal, combustion and corrosion products, particulate, catalysts, fuel cell components and other engineered materials,*

My name is Blaise Mibeck and I work at the Energy and Environmental Research Center at the University of North Dakota.

EERC started in 1951 as a U.S. Bureau of Mines Lignite (coal) research lab. From 1977 to 1980 we were a DOE National Lab.

We are currently a part of UND but we function as a non-profit contract research lab. We compete for funding. While more than half our dollars come from Federal research grants, more than 80% of our clients are commercial.

These wonderful people are my friends and colleagues in the NMARL – all have geology backgrounds but different areas of expertise.

I am a lab guy! I love measuring things. I develop new methods; routines for image and data analysis; collect microscope images; process x-ray CT images; maintain and modify equipment; work with vendors and field technicians; manage data and reporting and analyze any sample that comes to our lab that is not a rock.

We have weekly meetings we use to share methods and research ideas.



NMARL Petrographic Workflow

- **Problem:** need **Fast** and **Quantitative** Mineral Composition of Large (100s) sample sets to support massive subsurface projects
- **Complimentary Analyses:**
 - X-ray Fluorescence (element composition)
 - X-ray Diffraction (mineral composition)
 - Optical and Electron Microscopy
- Pulling everything together into the **Petrographic Workflow**

Every year projects in the EERC subsurface group are becoming larger -- more complex -- and time sensitive. Most of these projects aim to characterize the sub-surface for long term storage of CO₂.

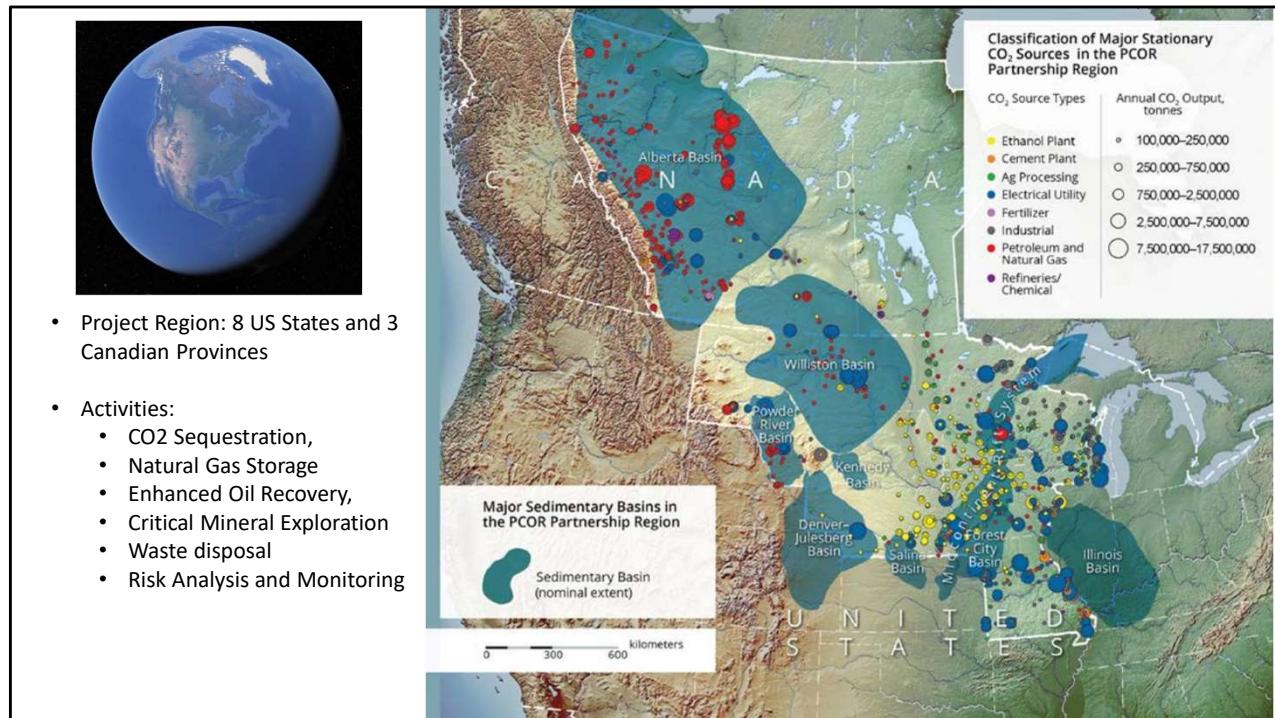
About ten years ago, I brought Rietveld analysis to the NMARL and developed methods for Quantitative Phase Analysis of ash, cement and rock samples.

These methods take a significant amount of time and expertise -- making them difficult to apply to the current workload.

We are using X-ray Diffraction and X-ray Fluorescence and are focused on combining them:

XRD for search/match of mineral patterns as before,

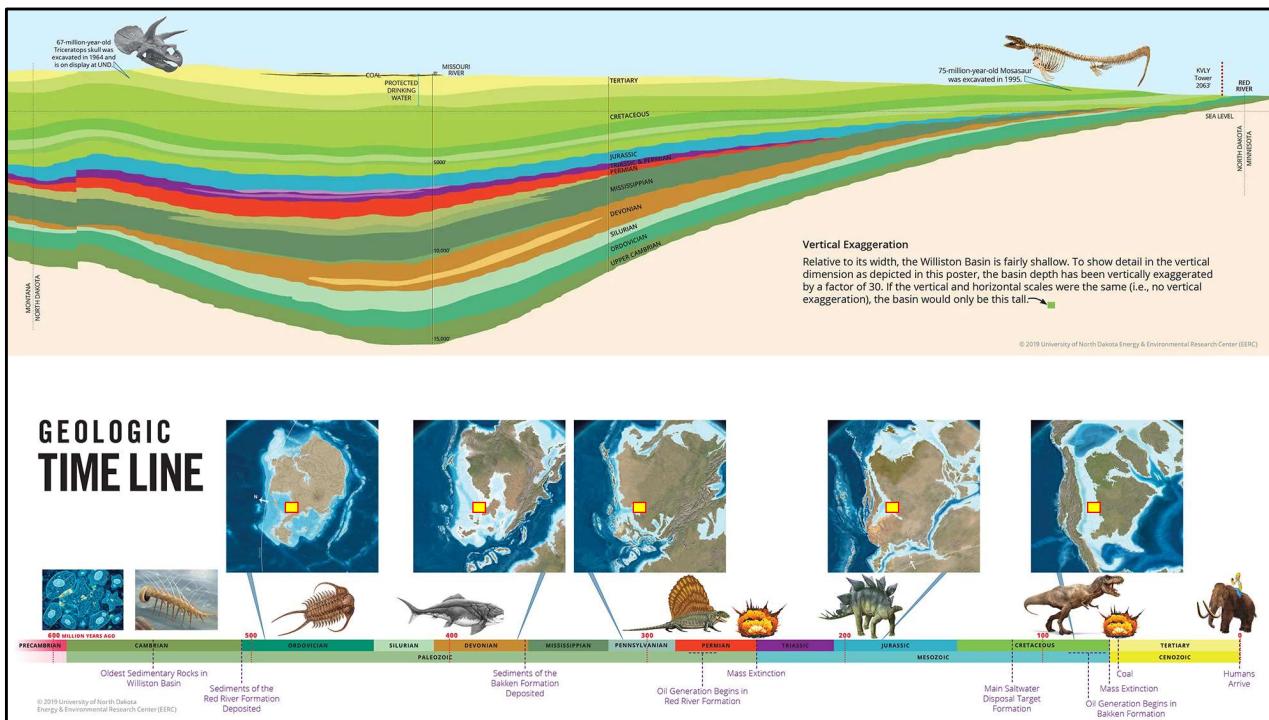
While relying more heavily on XRF element composition to improve the Quantitative Phase Analysis.



Here is the big picture. We have projects or completed projects that involve a large part of North America.

The EERC manages projects by organizing industry and government entities into partnerships, like PCOR, BPOP, etc...

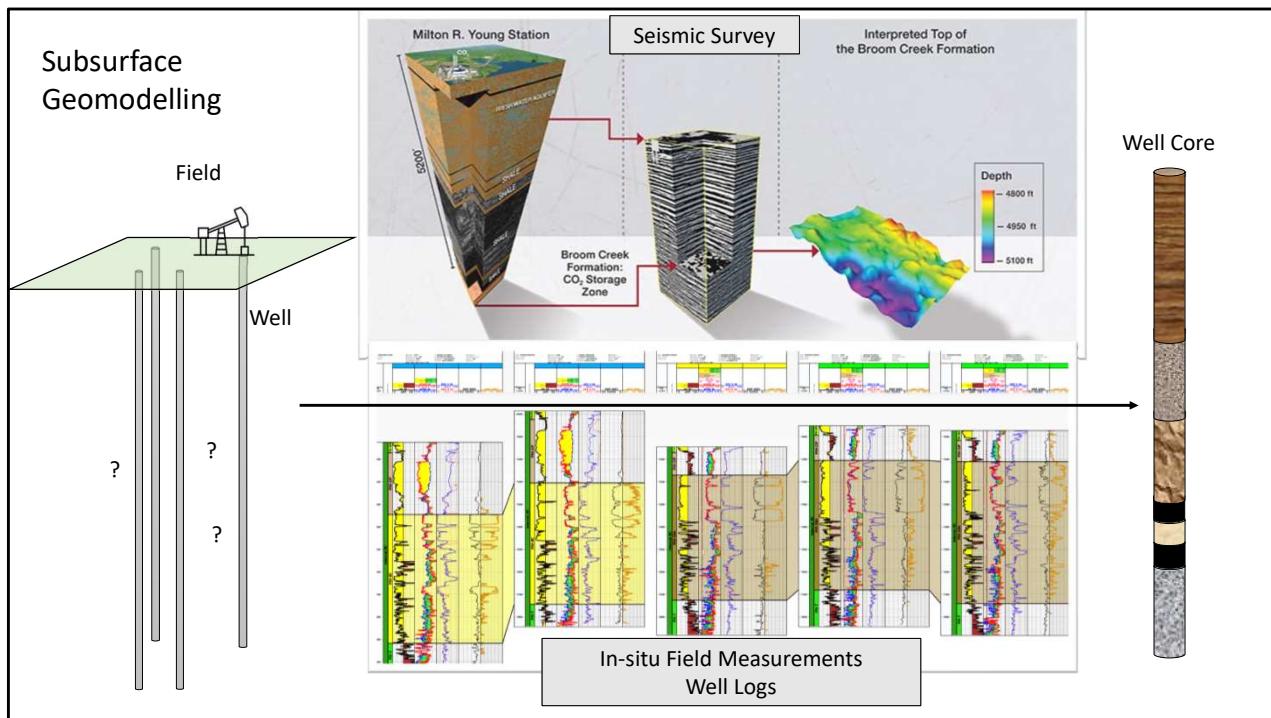
This map shows the location of CO₂ sources in relation to candidate basins.



Basins are regions of sub surface structure that can be broken down into formations each with varying degrees of homogeneity, permeability, porosity, fluid and solid chemistry.

This is a West-East cross section of North Dakota on top.

Below is a time line and illustration of where North Dakota is in relation to the formation of North America



Fields are county sized regions populated with wells.

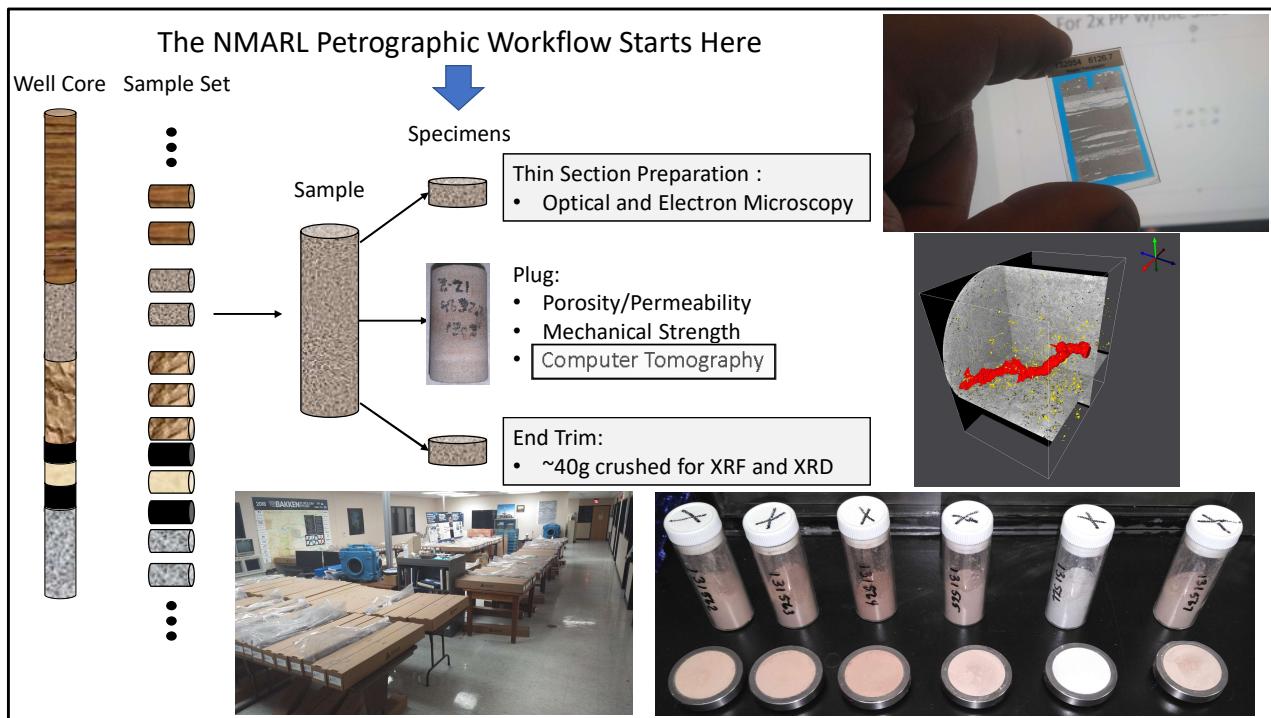
The subsurface is invisible to us, so we use:

- Seismic techniques to image large subsurface structures, in 3D (sometimes 4D)
- Well logging equipment (down hole techniques) to measure properties like gamma ray spectroscopy,

The **only** time we get to physically handle this rock is when 4 inch diameter Core is retrieved from a well or core library.

Core analysis is necessary to verify down hole measurements, calibrate seismic data and collect information that can not be had any other way.

- Petrophysical: speed of sound, mechanical properties
- Petrochemical: Fluid properties, contact angle, Flow Through Experiments
- Petrographic (what the NMARL does):
 - Macro/microscopic/tomographic imaging,
 - X-ray Fluorescence: Element Composition
 - X-ray Diffraction: Mineral composition



Often thousands of feet of core are collected from each well.

A few hundred feet of core will be chosen for a particular study.

Sample locations are chosen based on the study goals. A sample set can be a few samples or 50 to 150 samples per well. The trend is to increase the number of samples collected for each project.

The sample is split into a core plug and two end pieces.

- The plug is for petrophysical analyses, (we will often process CT data collected by and outside lab)

The next two pieces are used for petrographic analysis:

- A piece is sent out for thin section preparation
- Finally, a 40g piece is used for XRF and XRD analyses.

Differences between XRF and XRD

X-Ray Fluorescence

- Interaction **inside** atoms
- LOD: 100ppm (F) to 0.5ppm (U)
- Characteristic X-ray **wavelength** related to **electron energy levels** inside the atom
- Energy Levels Depend on the **Element**

Quantitative Element Conc.
Fast and Easy

X-Ray Diffraction

- Interaction in **outside** the atoms
- LOD: 0.1% (pyrite) to 20% (smectites)
- The **Peaks Positions** are related to **space between** atomic planes
- Space between planes depend on the **Crystal Structure**

Semi-Quantitative Mineral Conc.
Slower and Less Easy

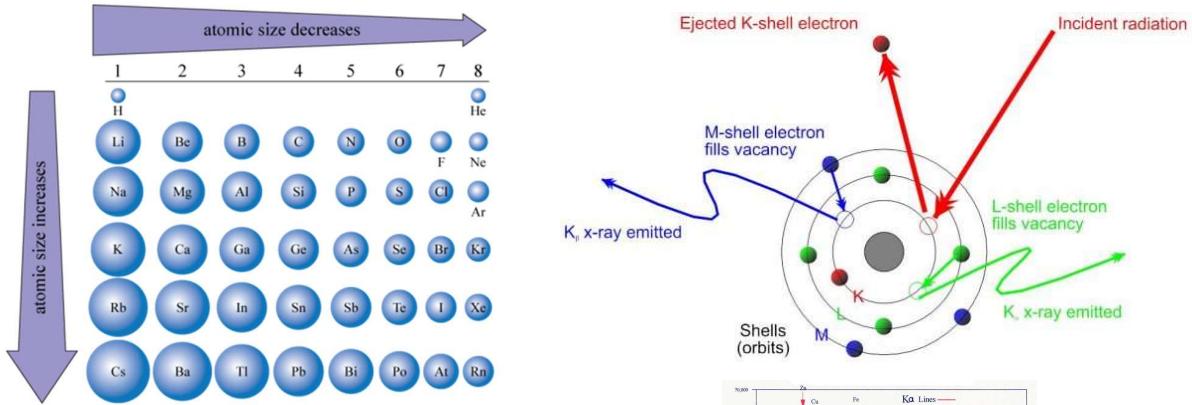
It is common for people using our results to be confused about XRF and XRD -- which technique does what?

Main thing to remember is that XRF is looking **inside** the atoms – and XRD is looking outside the atoms, but inside the minerals. In other words, XRD is looking at how the atoms are arranged.

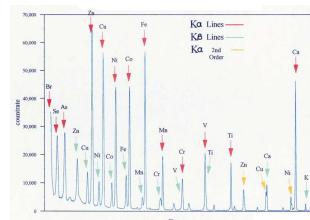
XRF “blind” to lighter elements (lighter than fluorine)

While XRD has trouble with amorphous material, or unknown structures.

Emitted X-ray wavelength **dependent on** Electron Energy Transition **dependent on** Atomic Number Z **dependent on** the Element



XRF Principle: Energy Spectrum gives Element Concentrations



The next few slides are examples of some of the training I do for people in our lab.

X-ray Fluorescence occurs when an incident beam of photons hits the sample.

Any time a photon is absorbed an electron is accelerated. Knocking out an inner shell electron makes the atom unstable.

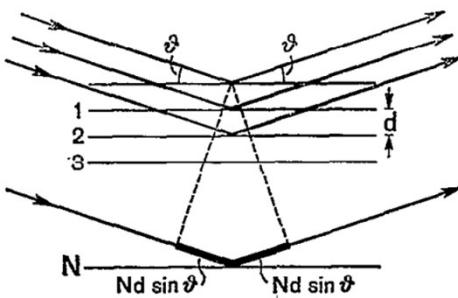
An upper shell electron will fall into the hole left by the ejected electron. Any time an electron deaccelerates it emits a photon. (this is fluorescence)

The energy of this x-ray **depends** on the energy difference between the energy levels involved. These energies levels **depend** on the **size** of the atom and so, measuring the characteristic x-ray energies can be used to **identify** and **quantify** the elements present in the sample.

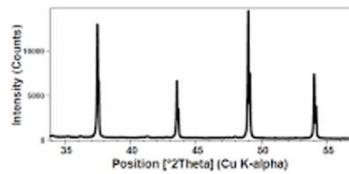
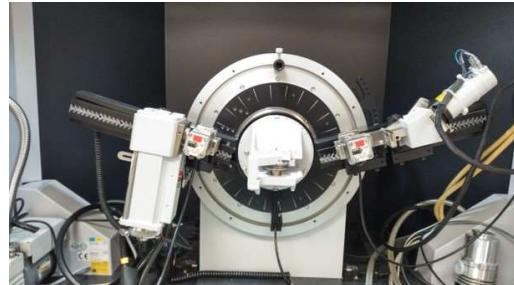
The characteristic x-ray energies are separated by wavelength and measured using a detector. The rest is electronics used for counting and binning the x-rays.

XRD Principle: Reflection Angle related to space between atomic planes (structure)

$$n\lambda = 2d \sin \theta$$



From: *50 Years of X-ray Diffraction*,
edited by P. P. Ewald



(diffraction patterns are images of frequency space)

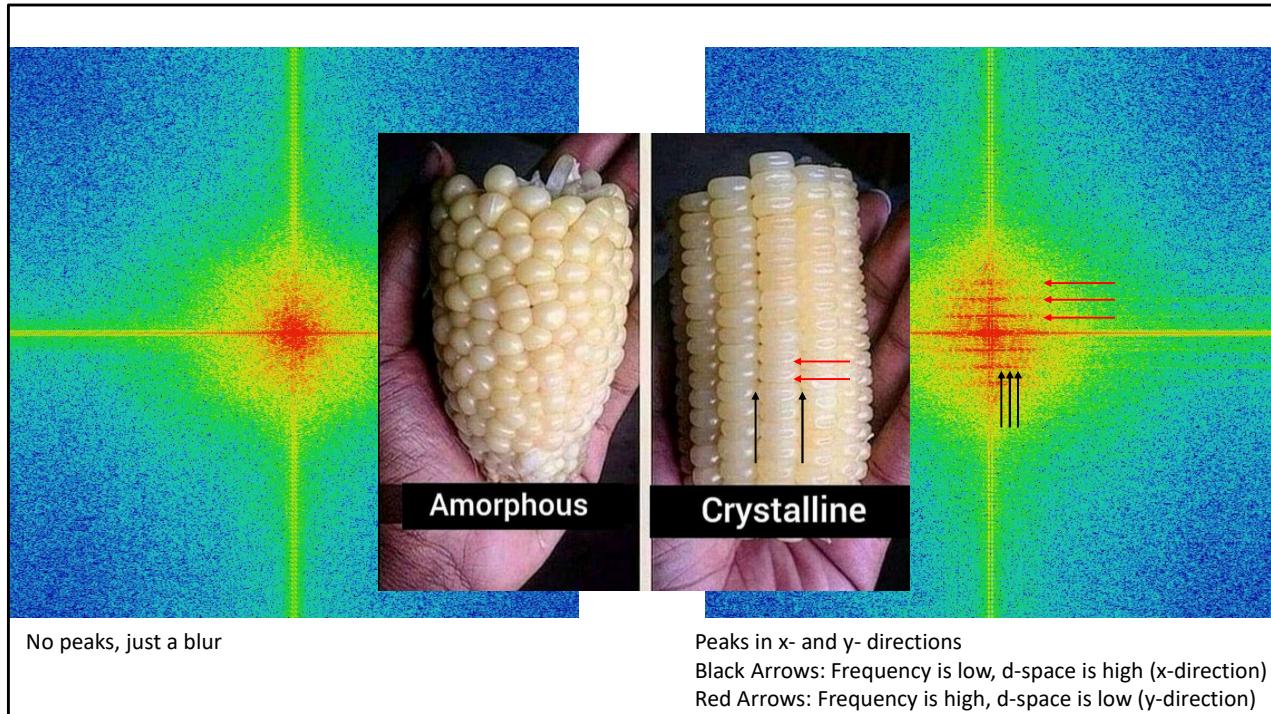
In XRD the x-rays reflect off the sample, and in the presence of repeating atomic planes these reflections can cancel or form sharp diffraction peaks.

High Angle peaks correspond to small d-spacings

These d-spacings depend on how the atoms are arranged.

This makes XRD sensitive to crystal structure.

Of course, the structure depends on which elements are involved and the larger atoms do reflect x-rays more easily.



Now for something completely different

This meme is going around in material science circles using Corn on the Cob to explain the difference between a crystalline amorphous materials.

The meme does a good job, but to improve it I added FFT images of the corn.

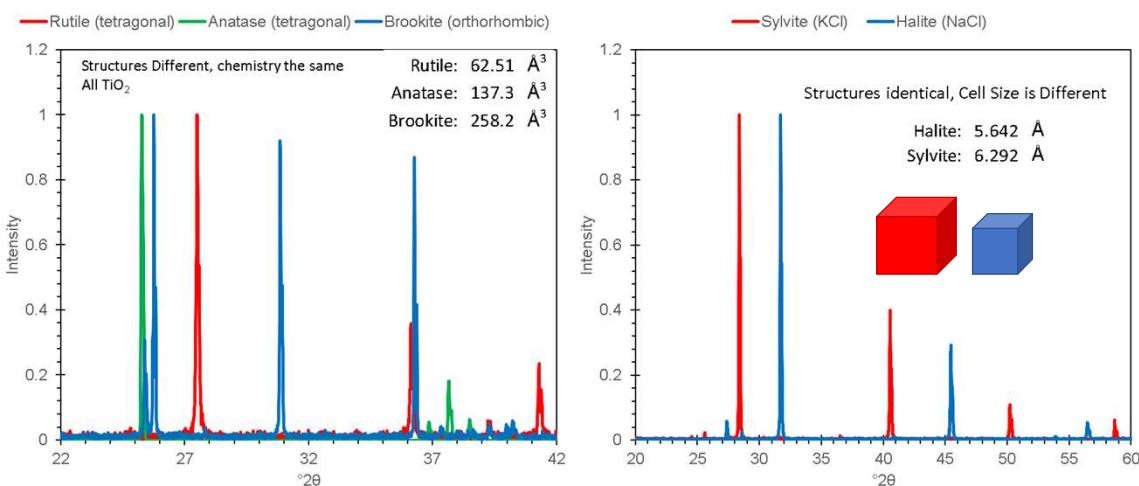
The center of each FFT corresponds to 0 frequency. The farther away from the center, the higher the frequency. The redder the color, the stronger the signal at that frequency.

An XRD is basically a camera that images the frequency space similar to how a Fast Fourier Transform can change an image from real space to frequency space.

The amorphous structure shows up as a blur while the crystalline structure has distinct “peaks” in the FFT.

You can also see that as d-spacing increases (black arrows), the frequency decreases (closer to the center).

XRD Structure Examples



Here are two classic examples of the diffraction patterns from XRD.

On the left are patterns for Rutile, Anatase and Brookite – all three are chemically identical. (TiO₂).

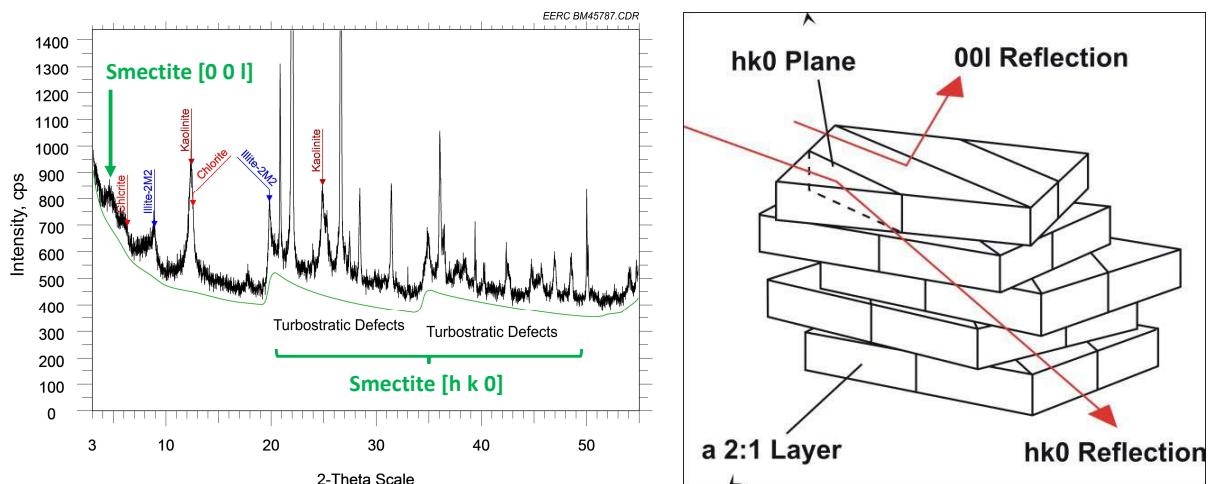
They produce completely different patterns because each crystal structure is unique.

On the right we have Halite (NaCl) and Sylvite (KCl) . As you can see the patterns are similar – in fact each of these minerals has the same face-centered-cubic structure.

The patterns are the same, just shifted.

Sylvite peaks are at lower angles (remember higher d-spacing). This is a consequence of the size of potassium atoms being slightly larger than sodium atoms.

Defects in Swelling Clays



Clays are notorious for being difficult to identify in XRD because these structures can be complicated. Also, they often have small crystallite sizes leading to peak broadening.

In general clays are plate shaped particles that lay flat causing the peak intensities to be different from an ideal sample

The more complicated an assembly of atoms (or anything else), the more likely there are defects.

Swelling clays have a part of the structure that can expand or contract depending on the ions in that layer. This can cause defects that appear as broad asymmetric peaks with a sharp low angle side from about 20 degrees and up.

These correspond to diffracting rays from the edges in the clay structure and are common in smectites (aka swelling clays). So, while the main $[0\ 0\ l]$ peak is swamped in the low angle noise, these defects let us know it's worth a closer look.

Swelling clays are a sign of local volcanism, but more importantly can cause damage to expensive engineering projects so finding it can be a big deal.

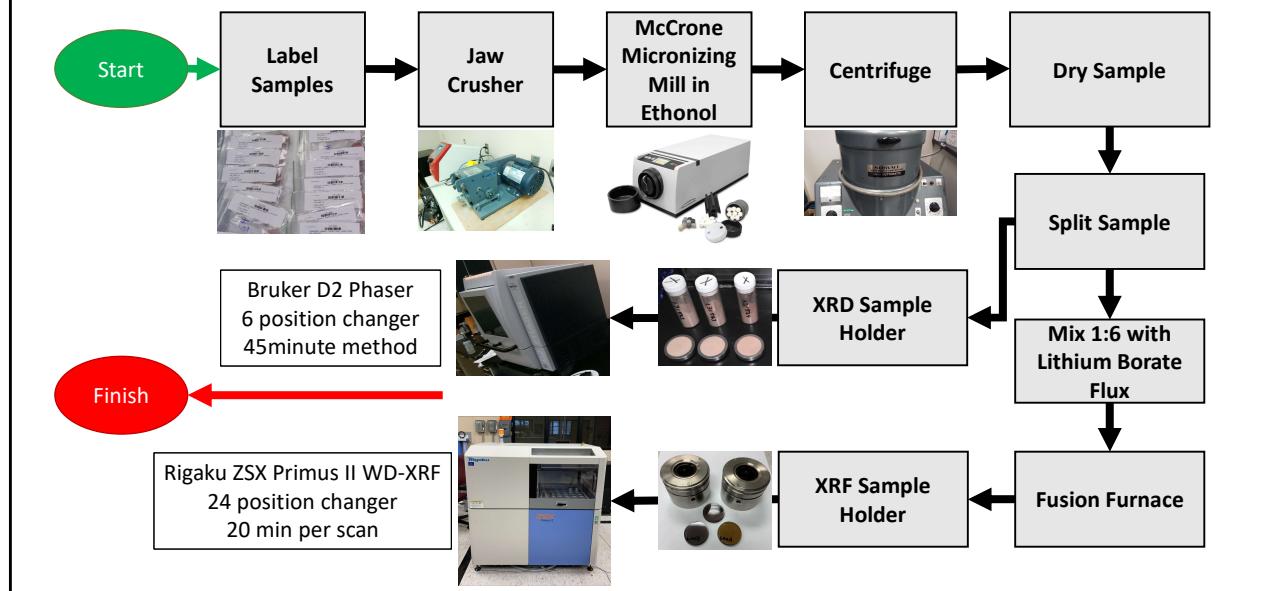
XRF Great for Quantitative Elements XRD is Great for Mineral ID

End of the Day, all the end user wants is a **quantitative** list of **minerals**



We know how XRF and XRD work and are compatible,

Sample Preparation Procedure



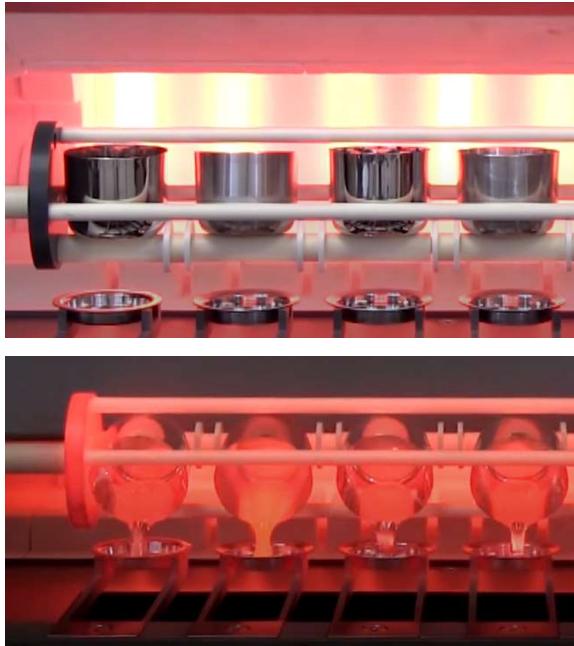
The workflow begins with sample preparation. We crush, micronize , centrifuge and dry the sample... we occasionally test the grind to ensure the majority of our sample is below 100um.

Then we split the sample for XRF and XRD.

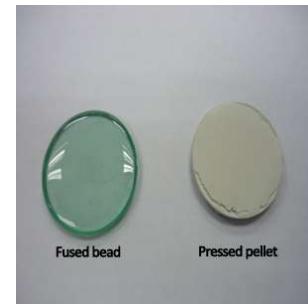
XRF samples are turned into glass bead using a furnace.

XRD samples use the micronized powder.

We ask for 40g as this is about the limit to what we can micronize in one go. We want a homogenized representative sample and the same sample for XRF and XRD. This gives us plenty for duplicate scans in needed.



XRF Fusion Bead



Important considerations:

- Some minerals partly gas off:
 - $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2(\text{g})$
 - $\text{CaMg}(\text{CO}_3)_2 \rightarrow \text{CaO} + \text{MgO} + 2 \text{CO}_2(\text{g})$

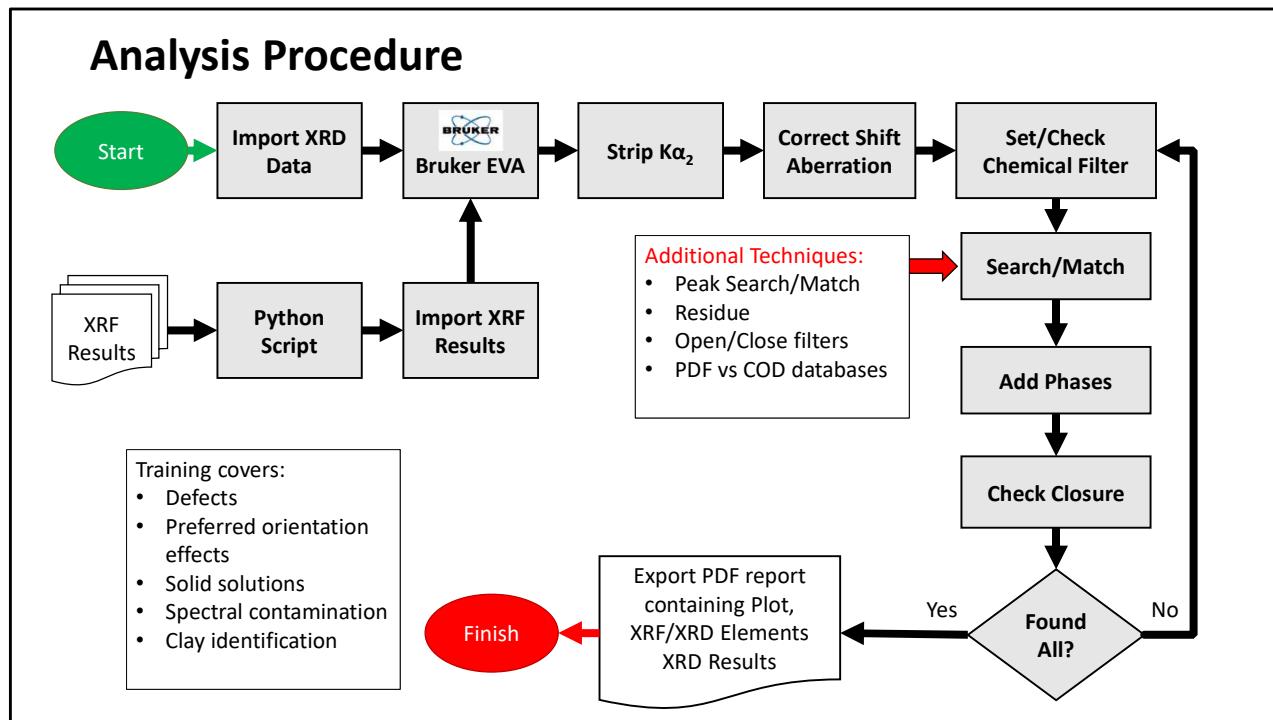
Sample homogeneity is important to XRF.

As mentioned earlier, XRF is blind to light elements. We mix 1:6 sample to flux. The flux Lithium Borate Flux (which contains elements with $Z \leq 5$) The sample and flux is heated to 1050C for about 20 minutes.

The result is glass matrix with sample oxidized and evenly distributed through out.

This is better than pressing the powder in into a pellet with cellulose binder (which we do occasionally), as it doesn't expand and crack due to humidity. Also the reabsorption of primary x-rays in the sample is lower for glass beads.

Some minerals decompose at these temperatures (carbonates like calcite and dolomite, hydrated minerals like gypsum, etc...)



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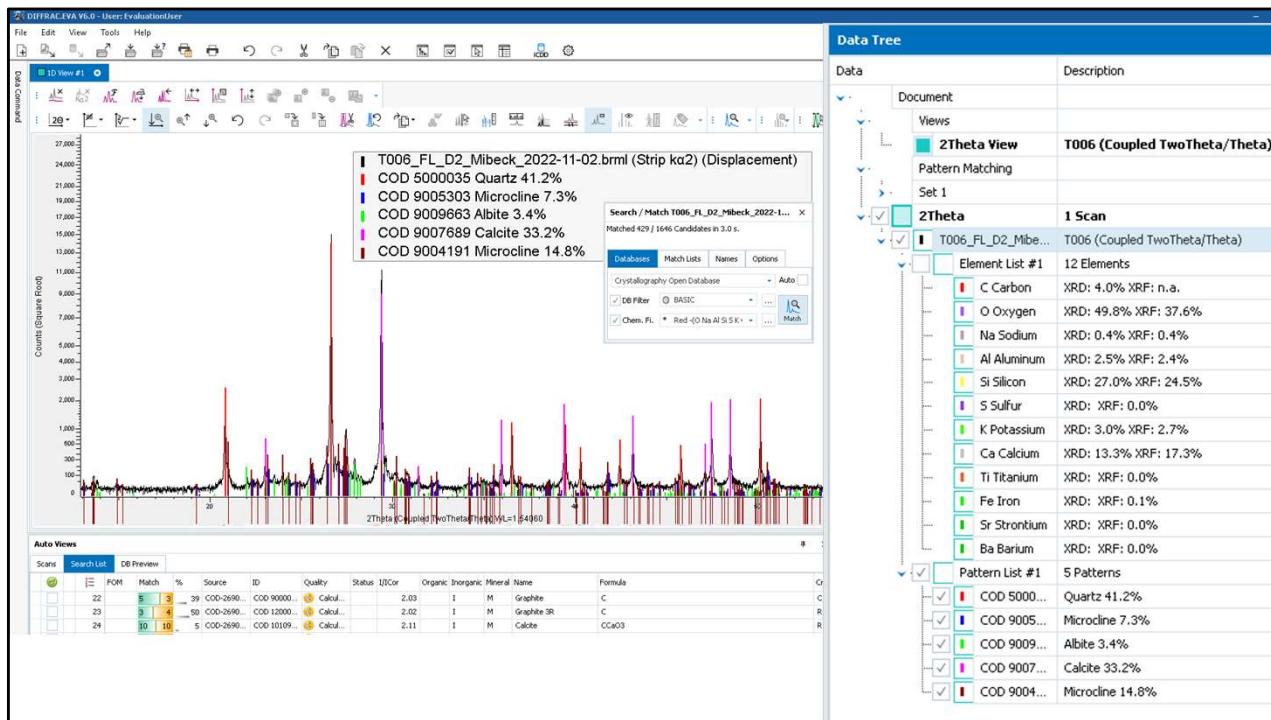
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Analyses are done by Rajesh, Chioma and myself, and we access the raw data in these working directories.

We meet weekly to review, correct or trouble-shoot each others work.

At the end of a sprint (a 50-sample set) we meet one more time before sending the draft results manager Shane for comment. It's like a game and getting the report past him is a win. ☺

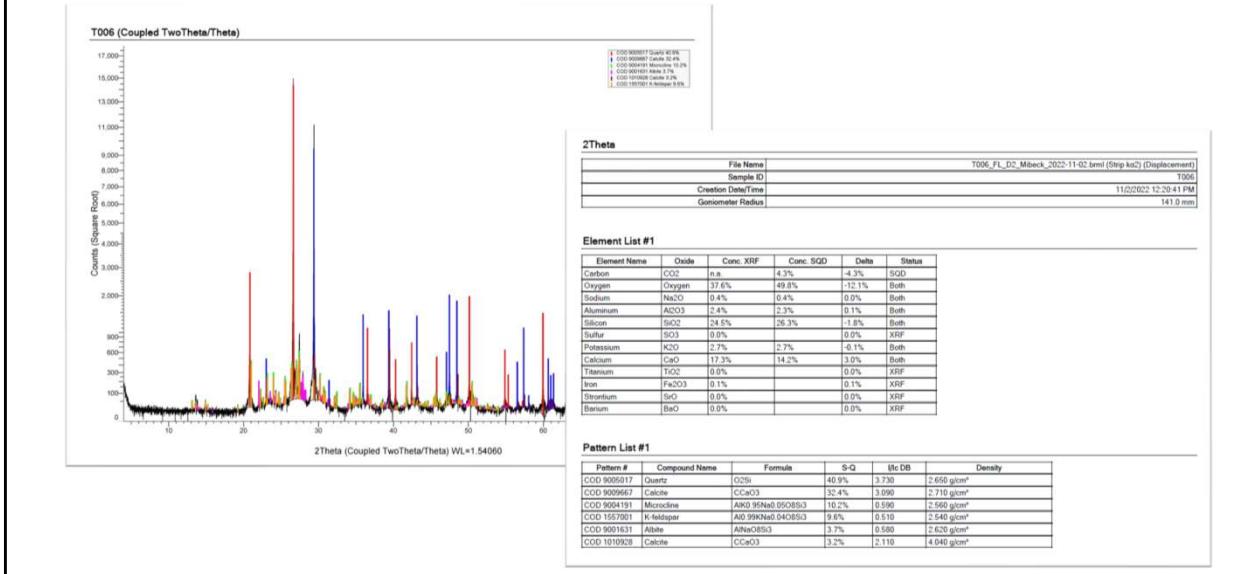


After accounting for major peaks, using the XRF data as a guide, we get to the point where the XRD phases are identified.

We can adjust peak heights manually to attempt closure between XRF and XRD Element concentrations.

We rely more on the closure between XRF and XRD elements – this is partly due to the use of air-scatter knife. This air scatter knife is needed to see low angle peaks, but also diminishes the high angle peaks.

Export PDF report

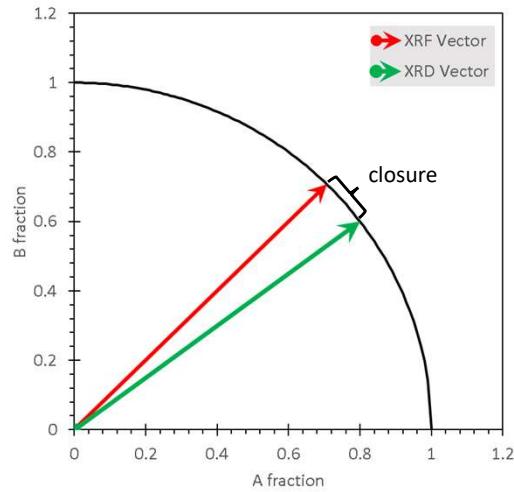


Each sample is reported and this is exported in PDF format.

How to compare XRF and XRD?

- Think of each element as a component in a Vector Space
- Closure refers to how different two vectors are; can be evaluated in many ways $\vec{A} - \vec{B}$ or $\frac{\vec{A} \times \vec{B}}{|\vec{A}| |\vec{B}|}$ for example.
- We defined Closure as the Euclidian Distance distance between the XRF and XRD element vectors

$$d(p, q) = \sqrt{(p_1 - q_1)^2 + (p_2 - q_2)^2 + (p_3 - q_3)^2} \\ = \text{SQRT}(\text{SUMXMY2}(A1:A5, B1:B5))$$



We think of the difference between XRD and XRF element concentrations as the difference between two vectors.

Squaring the difference between each component, adding these together and taking the square root is called the Euclidian distance. The goal is to minimize this distance.

This is also called a cost function.

Example of Closure with and without correcting for Calcite gas out....



	C:Ca	O:Ca
Calcite	0.2997	0.7984

Element	XRF Conc.	XRD Conc.	Delta	Status
Carbon	n.a.	4.30%	-4.30%	SQD
Oxygen	37.60%	49.80%	-12.10%	Both
Sodium	0.40%	0.40%	0.00%	Both
Aluminum	2.40%	2.30%	0.10%	Both
Silicon	24.50%	26.30%	-1.80%	Both
Sulfur	0.00%	NaN	0.00%	XRF
Potassium	2.70%	2.70%	-0.10%	Both
Calcium	17.30%	14.20%	3.00%	Both
Titanium	0.00%	NaN	0.00%	XRF
Iron	0.10%	NaN	0.10%	XRF
Strontium	0.00%	NaN	0.00%	XRF
Barium	0.00%	NaN	0.00%	XRF

Closure: 13.4%

Element	XRF Conc.	XRD Conc.	Delta	Status
Carbon	4.99%	4.30%	0.88%	SQD
Oxygen	49.44%	49.80%	1.61%	Both
Sodium	0.38%	0.40%	0.00%	Both
Aluminum	2.31%	2.30%	0.10%	Both
Silicon	23.56%	26.30%	-1.80%	Both
Sulfur	0.00%	0	0.00%	XRF
Potassium	2.60%	2.70%	0.00%	Both
Calcium	16.64%	14.20%	3.10%	Both
Titanium	0.00%	0	0.00%	XRF
Iron	0.10%	0	0.10%	XRF
Strontium	0.00%	0	0.00%	XRF
Barium	0.00%	0	0.00%	XRF

Closure: 3.8%

$$d(p, q) = \sqrt{(p_1 - q_1)^2 + (p_2 - q_2)^2 + (p_3 - q_3)^2}$$

By checking for the presence of calcite in the mineral list we can make corrections to account for the CO₂ lost during the fusion bead process.

Right now this is being tested so the closure is more of an indicator of how well the XRD analysis has gone. If much higher than observed with Test Samples (mixes of known minerals) than we investigate the cause.

I am working on how to solve for the mineral concentrations – there are a few papers out there (mainly by Frank Chung of Sherwin-Williams Research Center Chicago but an internal standard is necessary)

Element Count Space Vector: $\begin{bmatrix} C \\ O \\ Na \\ Al \\ Si \\ K \\ Ca \end{bmatrix}$	Examples: Calcite = $(CaCO_3)$ $\begin{bmatrix} 1 \\ 3 \\ 0 \\ 0 \\ 0 \\ 0 \\ 1 \end{bmatrix}$	Microcline = $(K_{0.95}Na_{0.05}AlSi_3O_8)$ $\begin{bmatrix} 0 \\ 8 \\ 0.05 \\ 1 \\ 3 \\ 0.95 \\ 0 \end{bmatrix}$
Atomic Mass Space Vector: $\begin{bmatrix} 12.011 \\ 15.100 \\ 22.990 \\ 26.981 \\ 28.086 \\ 39.098 \\ 40.078 \end{bmatrix}$	$A_{\text{calcite}} = [12.011 \quad \dots \quad 40.078] \begin{bmatrix} 1 \\ 3 \\ 0 \\ 0 \\ 0 \\ 0 \\ 1 \end{bmatrix} = \begin{bmatrix} 12.011 \\ 47.998 \\ 0 \\ 0 \\ 0 \\ 0 \\ 40.078 \end{bmatrix}$	Three possibilities: 1. One Solution 2. Many Solutions 3. No Solution One big Unknown: Possible Amorphous Phase (XRF will "see" the amorphous elements; the XRD will not)

The concept continues the idea of elements as vectors

The number of dimensions equal the number of elements.

Each mineral is also a vector – I have calcite and microcline here as examples...

To go from a count basis to a mass basis we can use a look up table of atomic mass

It all boils down to a matrix equation – that may be missing information. Chung use an internal standard to make it solvable. I am considering possible modeling the background of XRD patterns.

I have experience of using internal standard in the past in Rietveld Analysis, but it doesn't always work. Multiple additions are often needed and that costs time and introduces errors.

XRF XRD Review Tool
Low closure example

Note:
C and O dropped from Element Table

Keep Elements in both XRD and XRF

Sample Info:
Scan: 132326_FL_D2_Singh_2022-06-17.brm1 (Strip ko2) (Displacement)
Analysis: 132326_SQA_Onwumelu_2022-06-27.pdf
Sample Info: 132326 Summit_Slash Lazy H #5_NDIC# 38701_Newcastle Fm_4030.6 ft
Grain Density: 2.84g/cm3
XRF_XRD Closure: 0.41 (using 100x E.D.)

Open PDF

Results

Element	Element	Conc_XR	Conc_SQ	Delta	Status
Na	Sodium	0.1%	0.2%	-0.2%	Both
Al	Aluminum	1.6%	1.7%	-0.1%	Both
Si	Silicon	38.5%	38.3%	0.3%	Both
S	Sulfur	0.2%	0.1%	0.0%	Both
K	Potassium	0.1%	0.4%	-0.3%	Both
Ca	Calcium	5.0%	5.0%	0.0%	Both
Fe	Iron	0.6%	0.7%	-0.1%	Both

7 rows x 6 columns

Pattern #	Compound	S-Q	Wt DB	Density	
COD 101	Quartz_low	77.1%	3.42	2.660 g/c	
2	Kaolinite	2.6%	0.97	2.560 g/c	
3	COD 101	Calcite	9.8%	2.11	4.040 g/c
4	COD 901	Calcite	2.7%	2.86	2.710 g/c
5	COD 110	Muscovite	4.3%	0.33	2.890 g/c
6	COD 155	albite	24%	0.56	2.590 g/c
7	COD 900	Magnetite	0.9%	5.09	5.680 g/c
8	COD 500	Pyrite	0.3%	2.91	5.020 g/c

8 rows x 5 columns

Review

Send Back Ignore Mineral Check Mineral Accept Save & Exit Exit

I wrote a python program for reviewing this data. It scrapes the element and mineral tables from these PDF reports.

The sample info is displayed and a button can be used to open the PDF in a web browser.

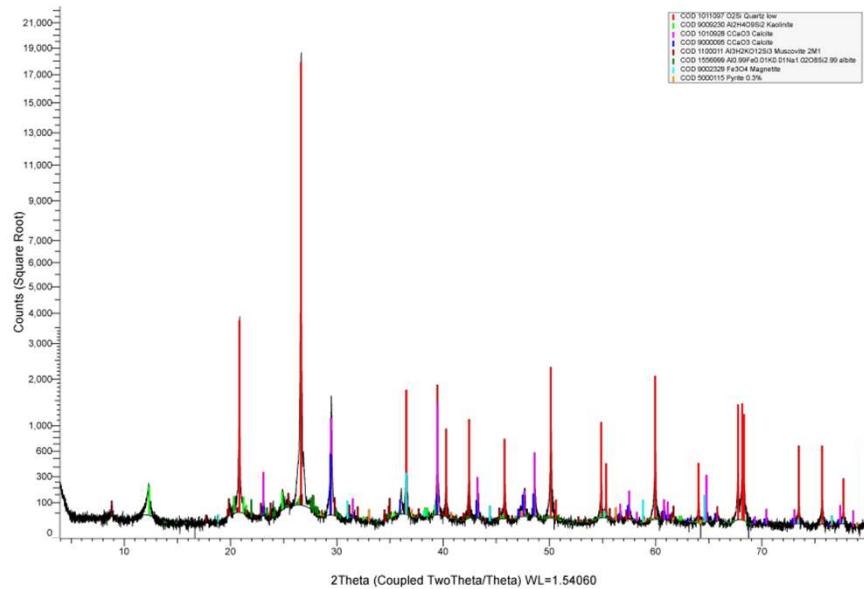
Additionally, the Sum Product of mineral density and mineral concentration is computed to estimate grain density (also called skeletal density)

A different lab at the center measures the same property for the bulk core plug using a gas pycnometer.

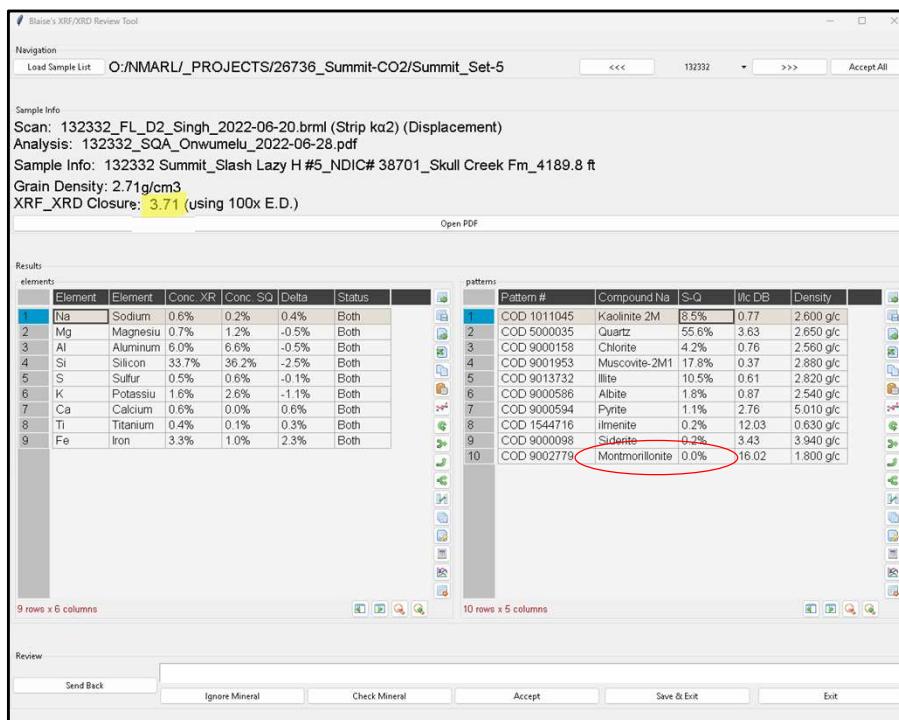
Finally, any comment can be entered in the text box and saved to an excel file at the end.

We use that comment list to break the work up among the analysts.

Low closure: Flat background, clean, few phases...



No surprise that low closure samples general have fewer, simpler phases, sharp peaks and well behaved background.

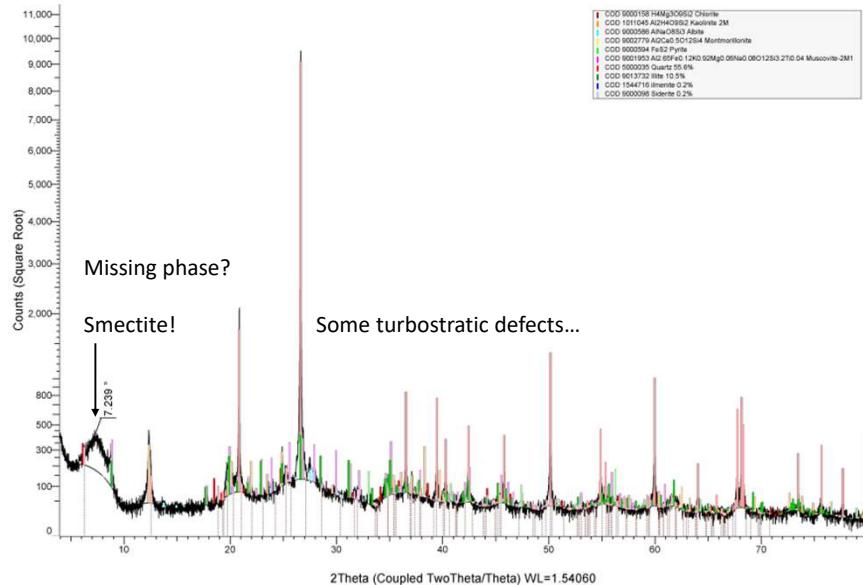


High Closure Example

This is the review tool for a sample with high closure.

The program is a work in progress...

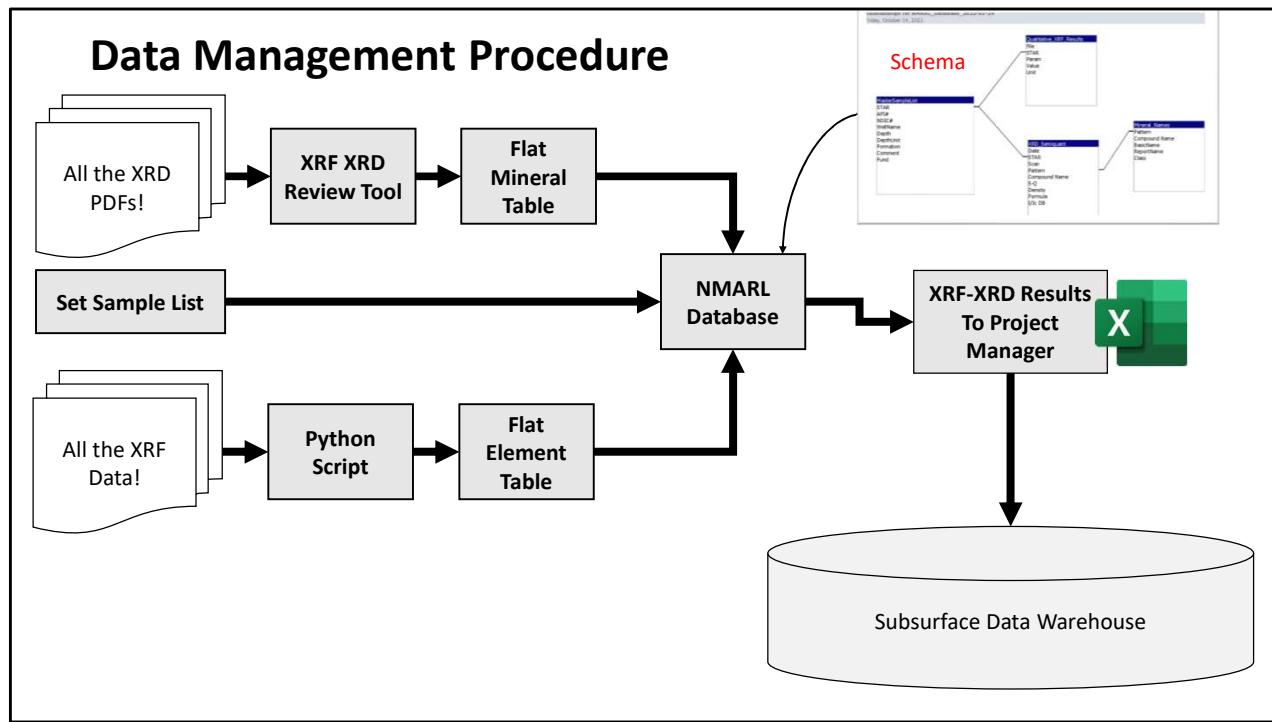
High closure: Complicated Background, many phases...



Montmorillonite was found by XRD but at trace concentrations
Montmorillonite is a smectite

We quickly brought this to the attention of the project manager

It turns out in this case the drill company used a mix of bentonite (another smectite) and diesel to lubricate the well bore. As this was the only sample with smectite it was noted but not included as it was not natural.



To keep our sanity we have rules regarding the naming of files.

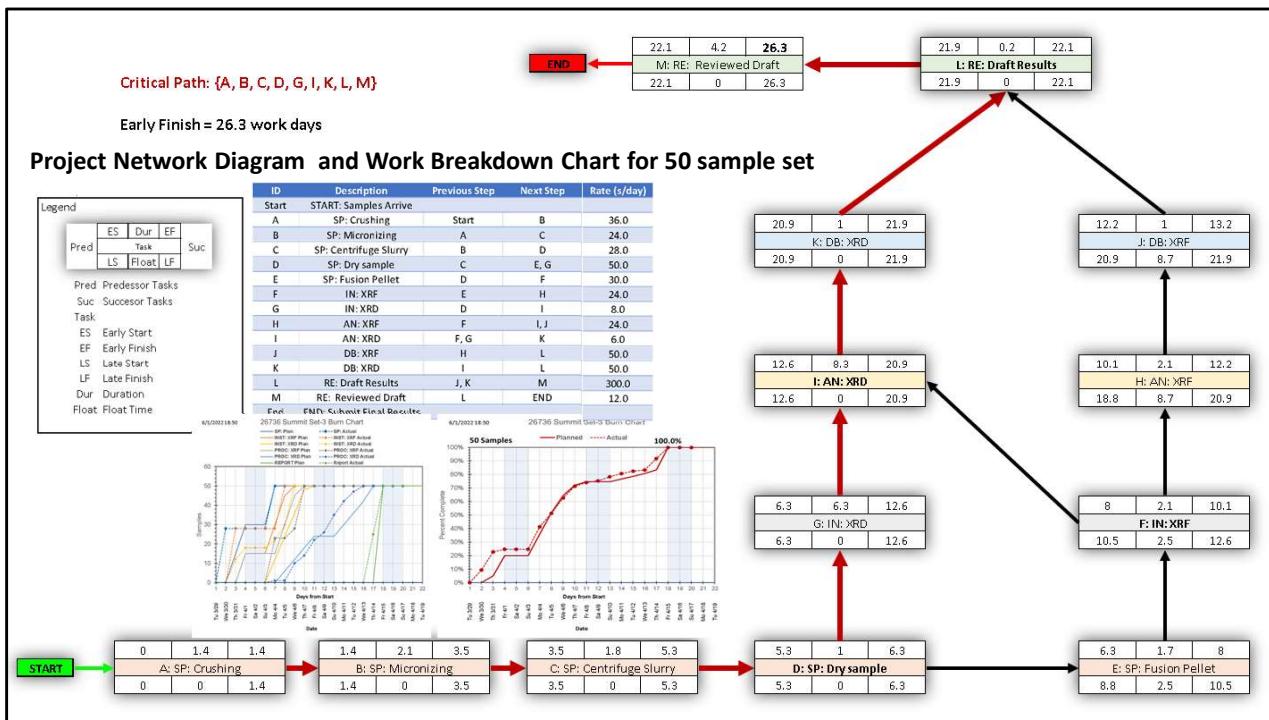
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Finally, I developed tools to aid us in planning sand execution

– we built a work-break-down-chart and I used it to create a Project Network diagram.

The bottle neck in the workflow is the XRD Analysis, (not a surprise) but the network diagram shows us the best case scenario for how long 50-samples will take and a clear understanding of the steps that are most critical each day.

A natural consequence of designing the work flow is the creation of burn charts. One of these is used to keep track of individual tasks in the lab and an overall chart is used to update project managers.

We use these to keep track of how well the actual work is going compared to the plan on a day by day basis. If we fall behind we can make adjustments, or record the reasons for any delays. They burn charts have been collected and used to re-evaluate the work break-down chart so we can make better predictions.

Imaging Techniques

Optical Microscopy

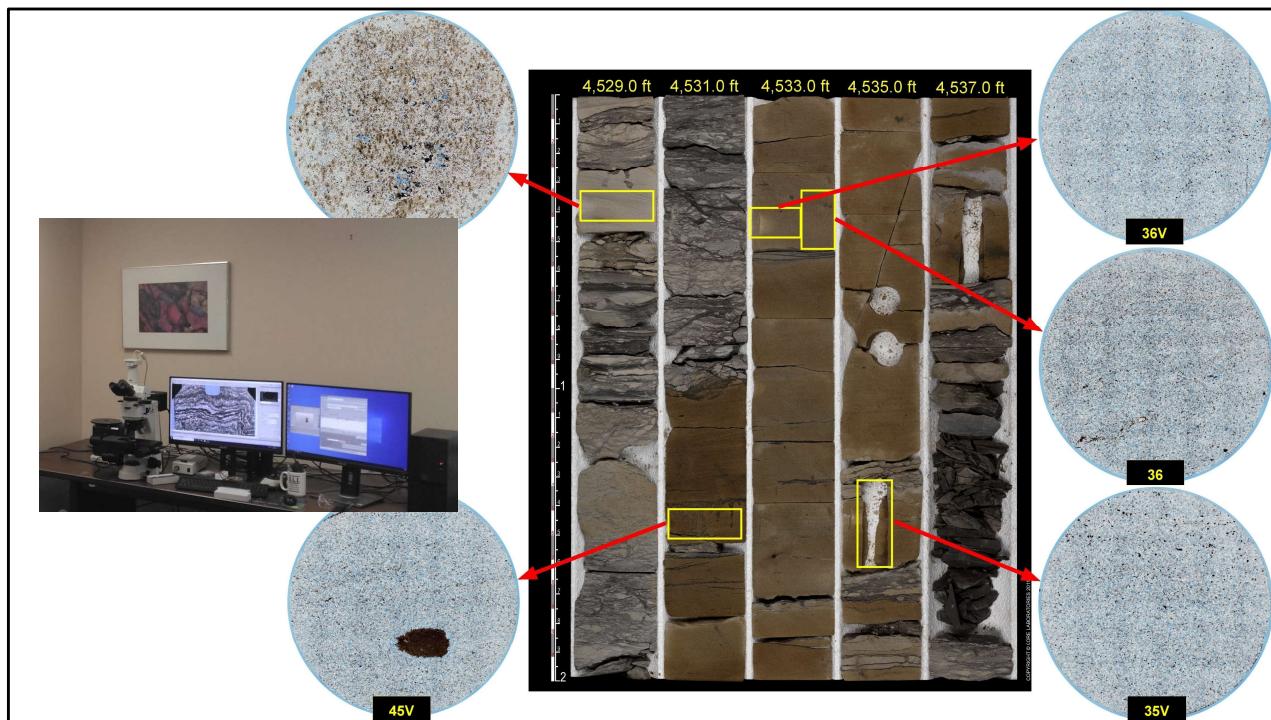
- Transmission, Reflected Light, Cross Polar modes
- From cm to um
- Color, size, shape, and some crystallographic properties.

Scanning Electron Microscopy

- Back Scatter and Secondary Electron and UDF modes
- From cm to nm
- BSE Brightness (Z_{ave}), Energy Dispersive Spectroscopy (element composition), size and shape. Limited cathode luminescence capability with UDF mode

I won't go into much detail here – just a quick overview of how we use Optical microscopy and SEM in these projects.

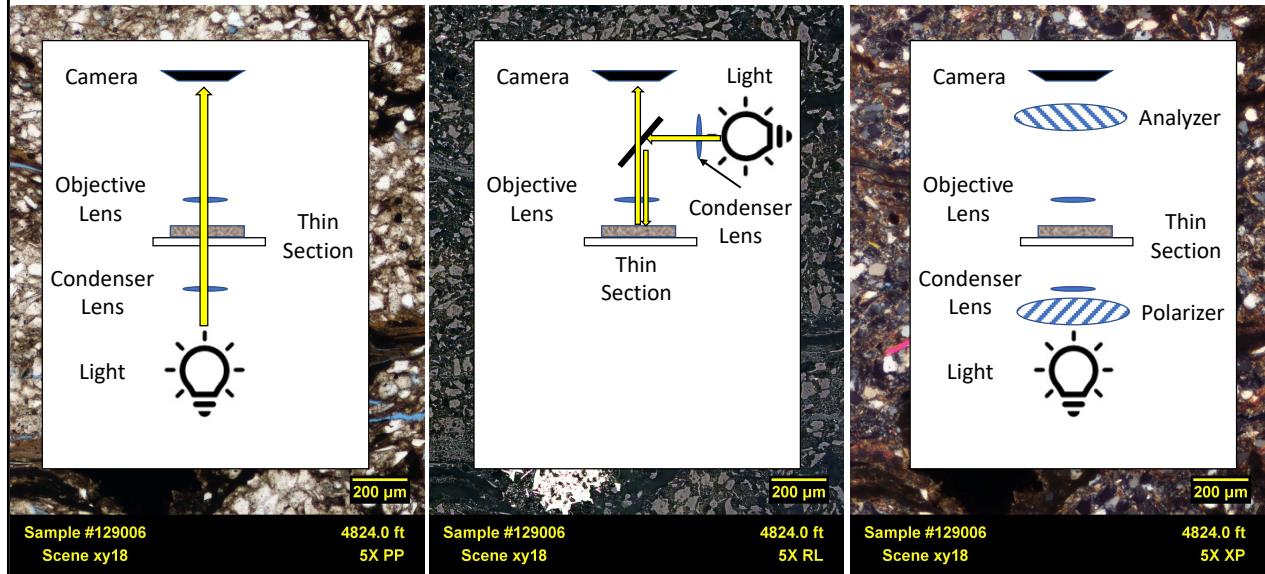
Each imaging system has various modes



Here is a box of core, 2ft high. Not all of the rock makes it out of the ground in one piece. Some core looks like Swiss cheese with how many core plugs are removed.

The circular call outs are microscope images of the thin sections made from samples at each location – the pore space is filled with blue die to provide contrast.

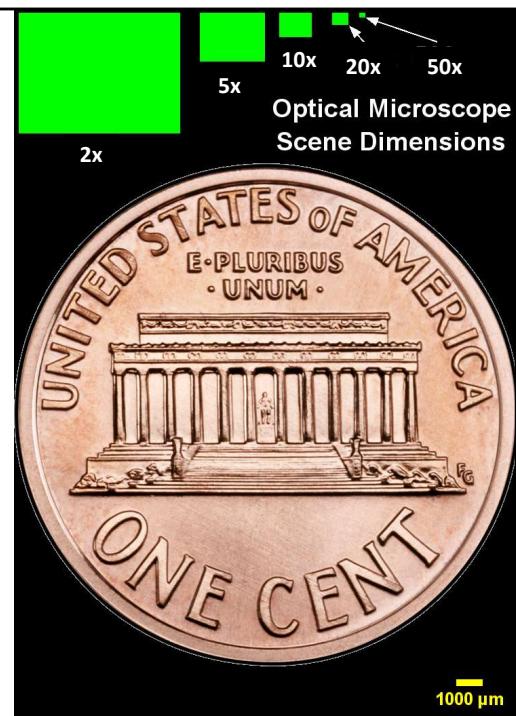
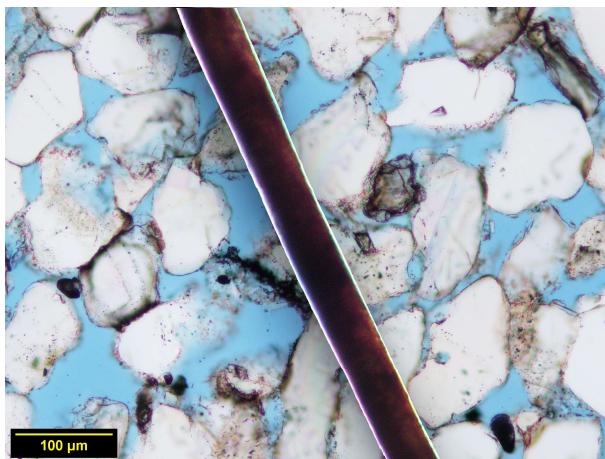
Plane Polar (PP) Reflected Light (RL) Cross Polar (XP)



These are the three modes of the optical microscope.

Note this is the sample slide, same location (or scene) but each mode excels at a different observation.

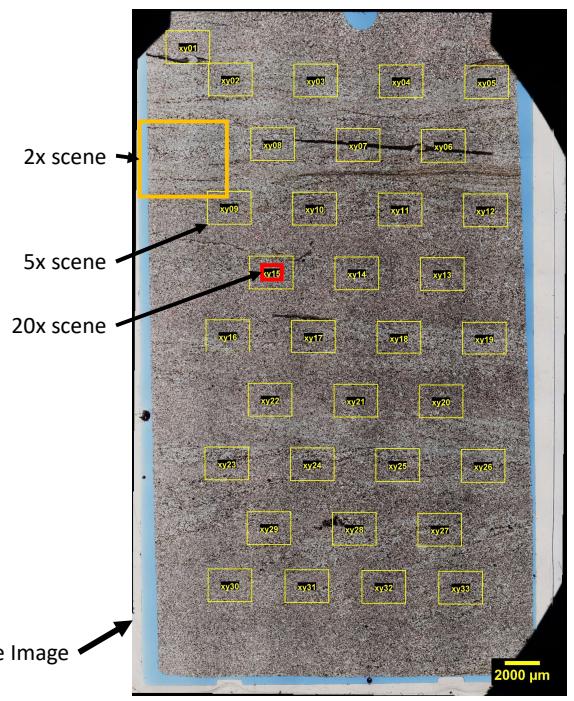
OM: Field-of-View



The field of view is be part of what we are able to see. That image is a 20x objective (200x magnification if looking in the eyepiece).

Standard Image Collection

- Whole Slide Image:
 - 2x PP grid of about 50 images with 25% overlap of images that are stitched to make Whole Slide Image
- Multipoint: 25 to 36 images spaced throughout the sample:
 - 5x PP, 20x PP, 20x RL, 5x RL
- 20x scenes coincide with 5x
- 2 magnifications, 2 modes, up to 36 scenes = 144 images



Our thin section work flow is depicted here.

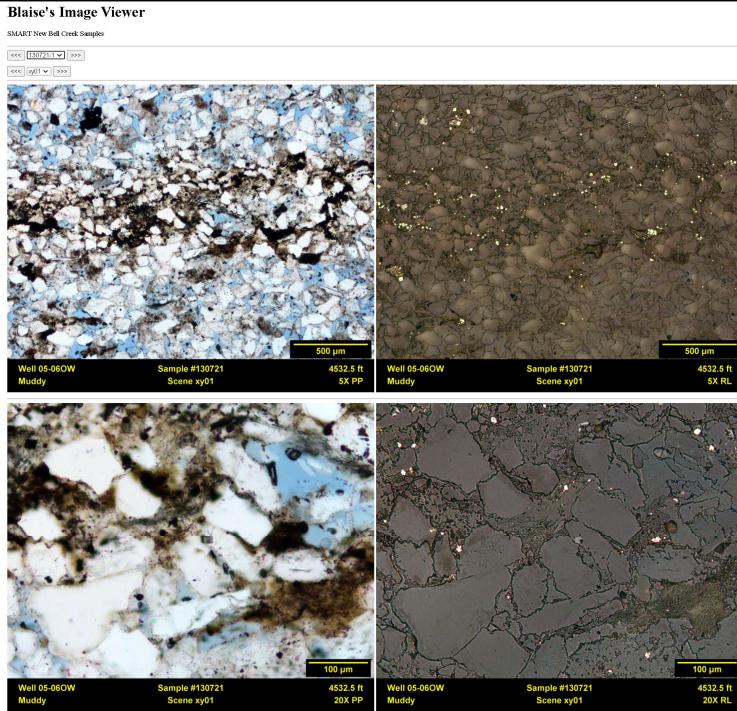
Image Viewer

Collections are large, so an html file is used to move through samples/scenes

(I very much want to improve on this)

Notice:

- PP shows volume of the thin section
- RL show upper surface
- Some opaque in PP are seen as pyrite in RL
- Each mode has pros and cons



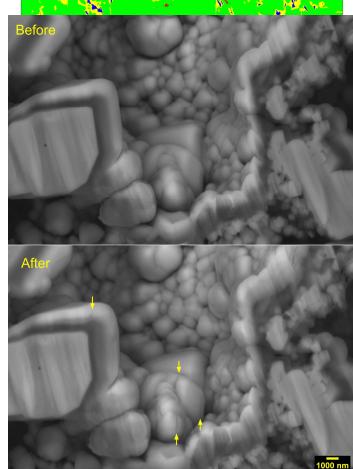
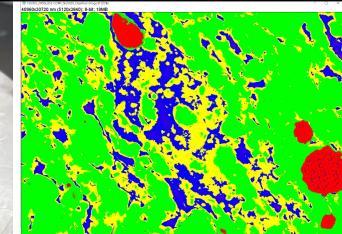
I created this tool in html – all the files are arranged on a special network drive that these HTML files like to. We store the original uncompress file with scale information and metadata encoded in the tif header. These tifs are used for analysis.

We also label the images and store as jpeg for publications and easy use by the end user.

Imaging: Electron Microscopy

Used to Measure:

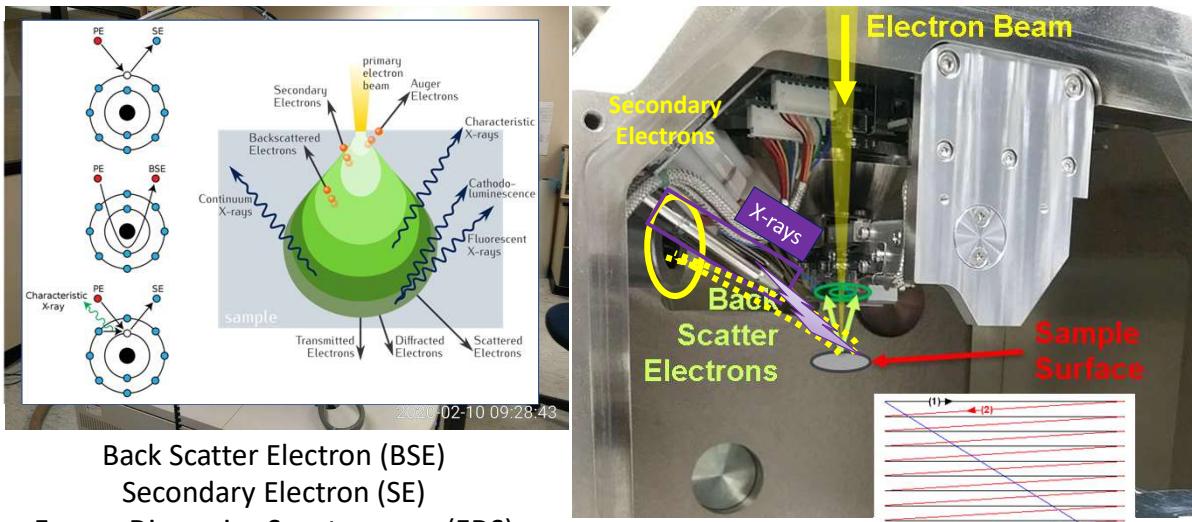
- Chemical composition (BSE and EDS)
- Extremely fine detail,
- Sample Prep and Advanced Methods:
 - Feature, AMICS, EERC-QIA



We have a SU5000 Field Emission Scanning Electron microscope and the ion ill to prepare samples.

I developed methods for identifying porosity in organic shale samples by using a BSE calibration target.

Scanning Electron Microscopy



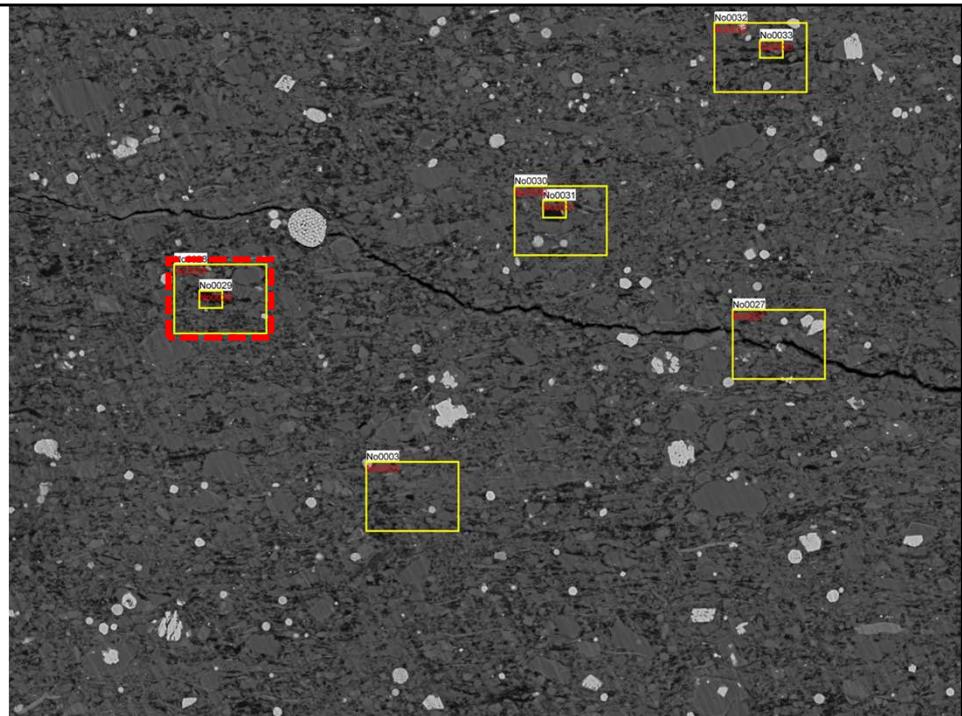
The FESEM methods are shown here.

Field of View and Resolving Power

300x
pixel = 82.7 nm

W: 425 μ m
H: 319 μ m

Fine Mechanical Pencil Lead



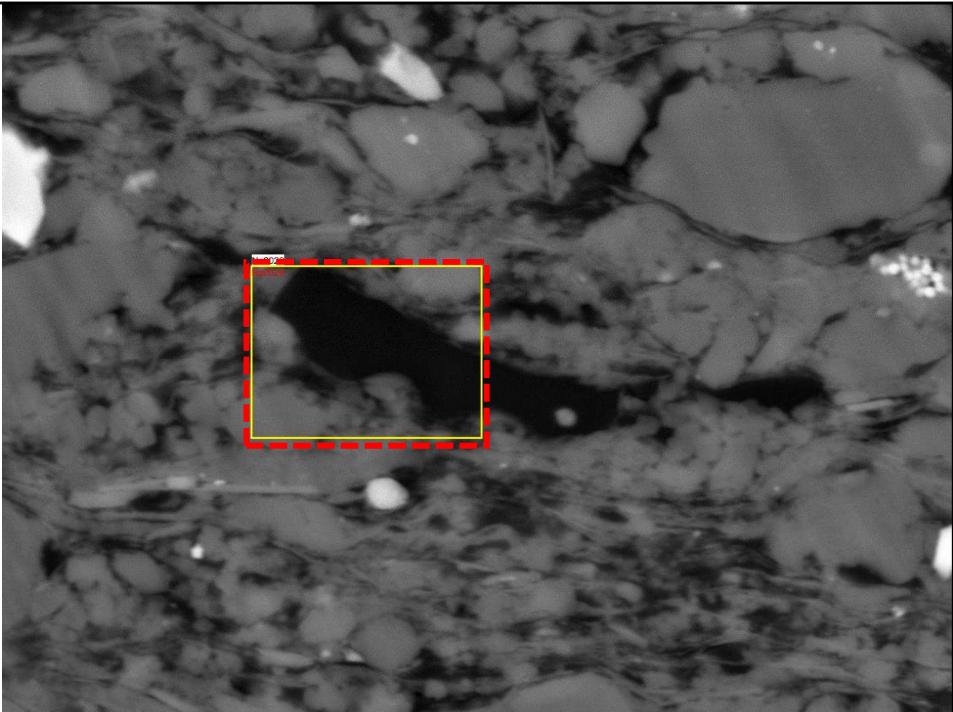
Mechanical Pencil Lead is between 500 and 700um in size

Field of View and Resolving Power

3000x
pixel = 8.3 nm

W: 41 μ m
H: 31 μ m

Human Hair

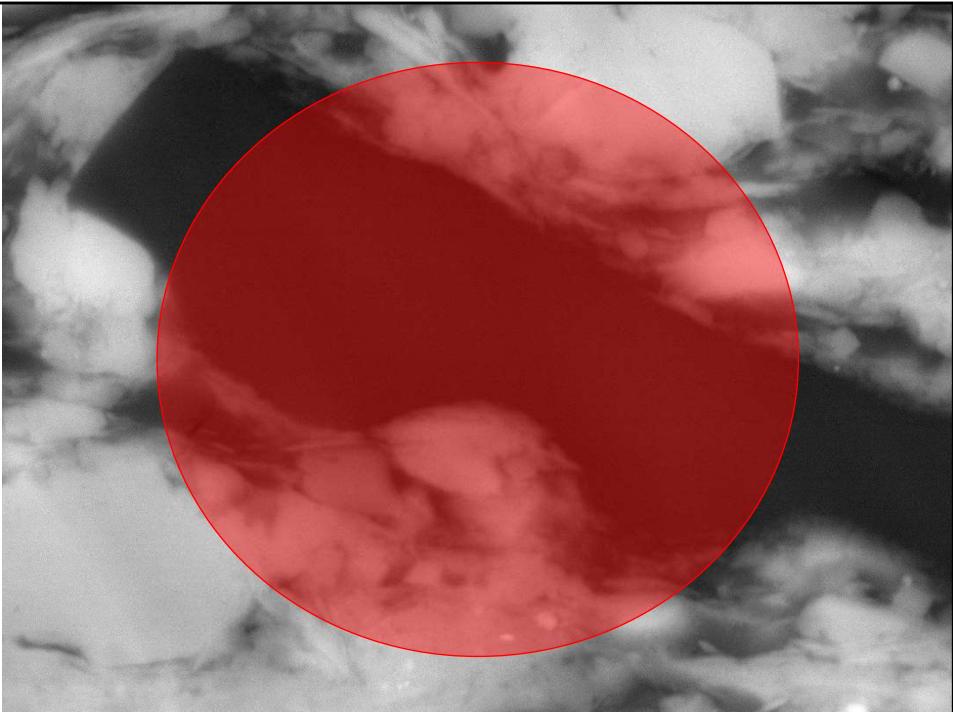


Field of View and Resolving Power

10,000x
pixel = 2.5 nm

W: 10.2 μ m
H: 7.7 μ m

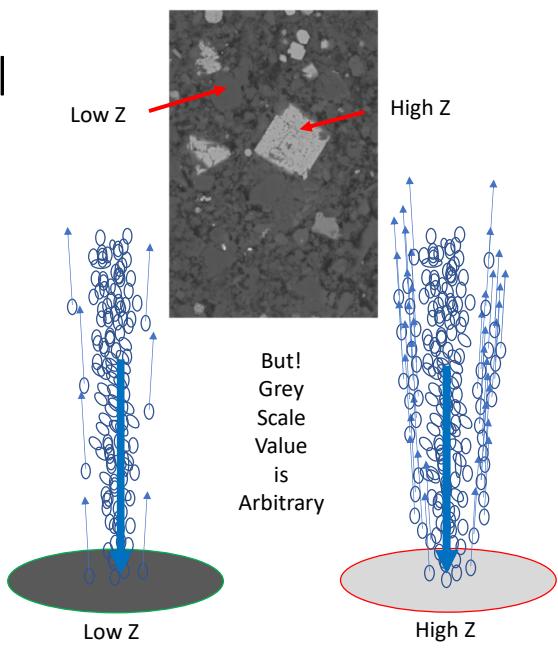
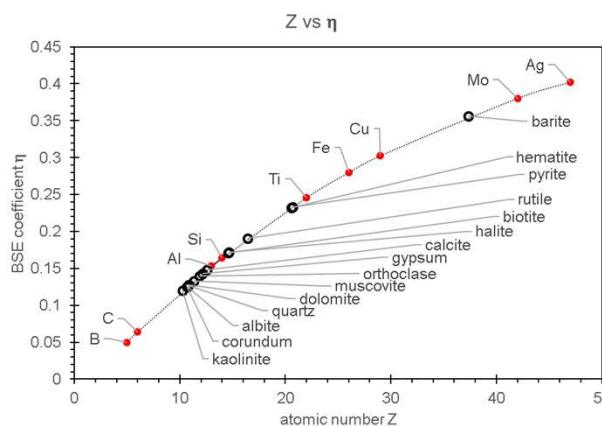
Red Blood Cell



Guess the object that

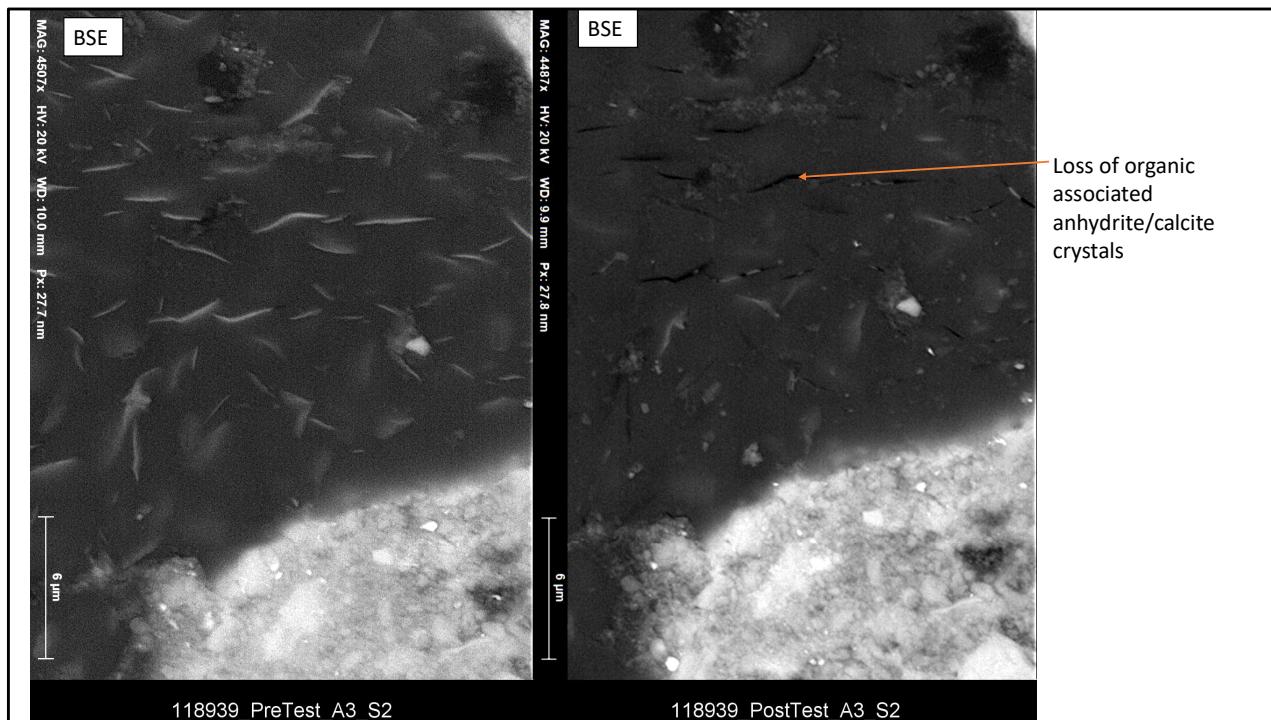
Back Scatter Electron Signal

- Black and White Image Contrast from Z (atomic number)
- Atomic Number i.e., Number of Protons



Brightness in BSE land depends on Atomic Number

The ratio of reflected to incident electrons is Higher for High Z
Segmenting solid classes first and treating Pores separately.



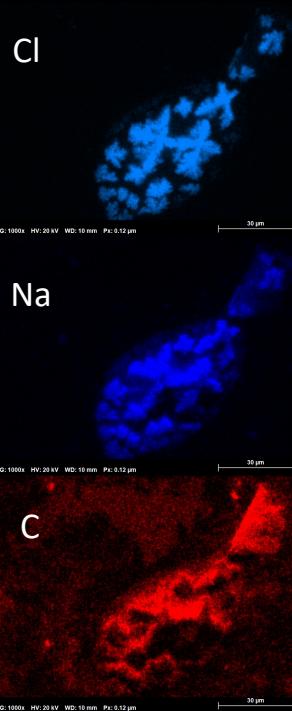
Probably the trickiest thing to do is before/after images. Before some exposure experiment you have know idea what locations are going to be interesting.

It's amazing when you relocate a specific are of the sample a month after imaging it the first time.

The organic mater has a changing porosity making it difficult for oil recovery and CO₂ sequestration. The flow regimes are complicated too referring to how fluids at high pressure and temperature permeate.

X-ray Signal (EDS)

Strong evidence of diffusion in organic minerals
Salt crystals appear as dendrites, evidence of
crystal formation in an environment where
transport is limited by diffusion.

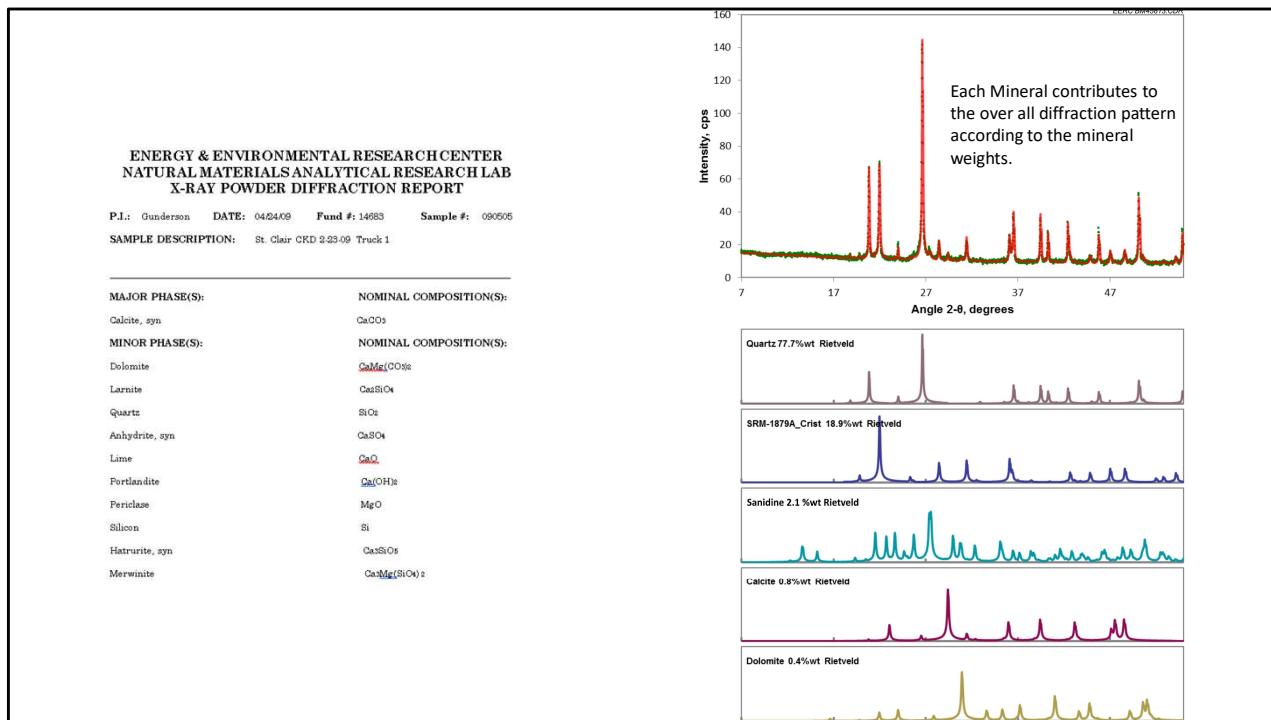


Element
Mapping
from X-rays
and EDS
detector

In this case the experiment a methane/ethane/propane mixture was able to mobilize brine and these previously clean organic materials “grew” beautiful salt dendrites. Eds is able to identify the elements here and this observation helped us show that diffusion need to be including in the models.

THANK YOU for your Attention! And you Patience on Tuesday!

Thank you for your attention!



Before I came to the NMARL, XRD reports indicated the phases found in the sample, but not their concentrations. At most would be an indication of minor or major phase. On top of this power surges caused problems with the electronics.

After purchasing the Bruker D-8 Advanced, I immediately started using Rietveld analysis to perform quantitative phase ID on Sub-surface samples.

Problems arose:

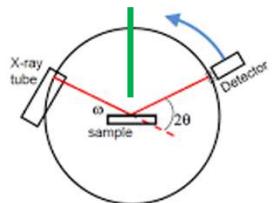
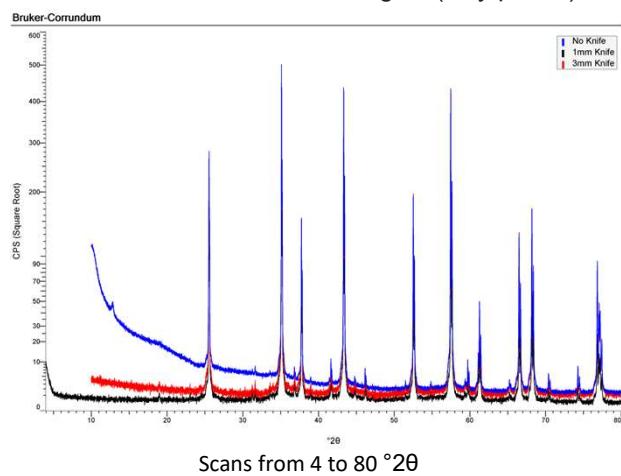
- I lacked the background to know what mineral phases were realistic
- Including an internal standard allowed be to report amorphous content, but this would require several different additions of internal standard to get right.
- I would also report too much information of now importance to the end user (crystallite size for example)

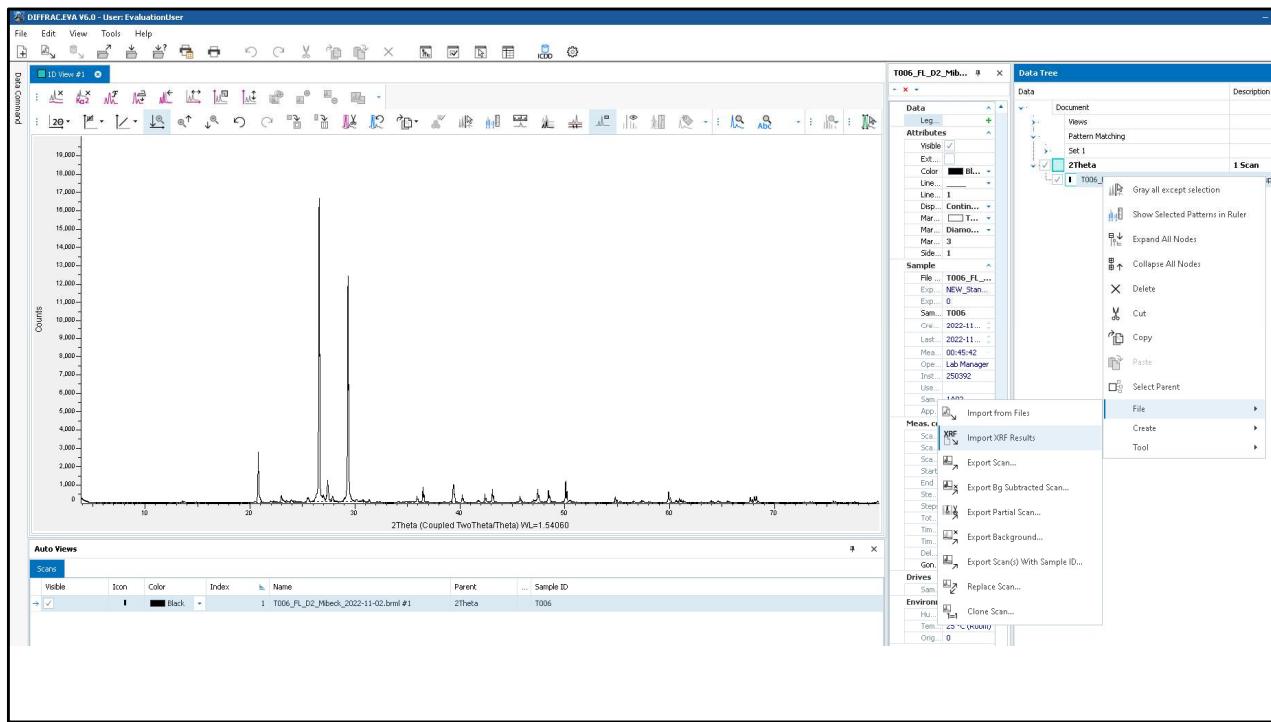
I tried to find standard methods in the petroleum industry and found nothing more than “use of the RIR method” and “vendor software” like EVA.

XRD Instrument Parameters

Air-Scatter Knife above sample:

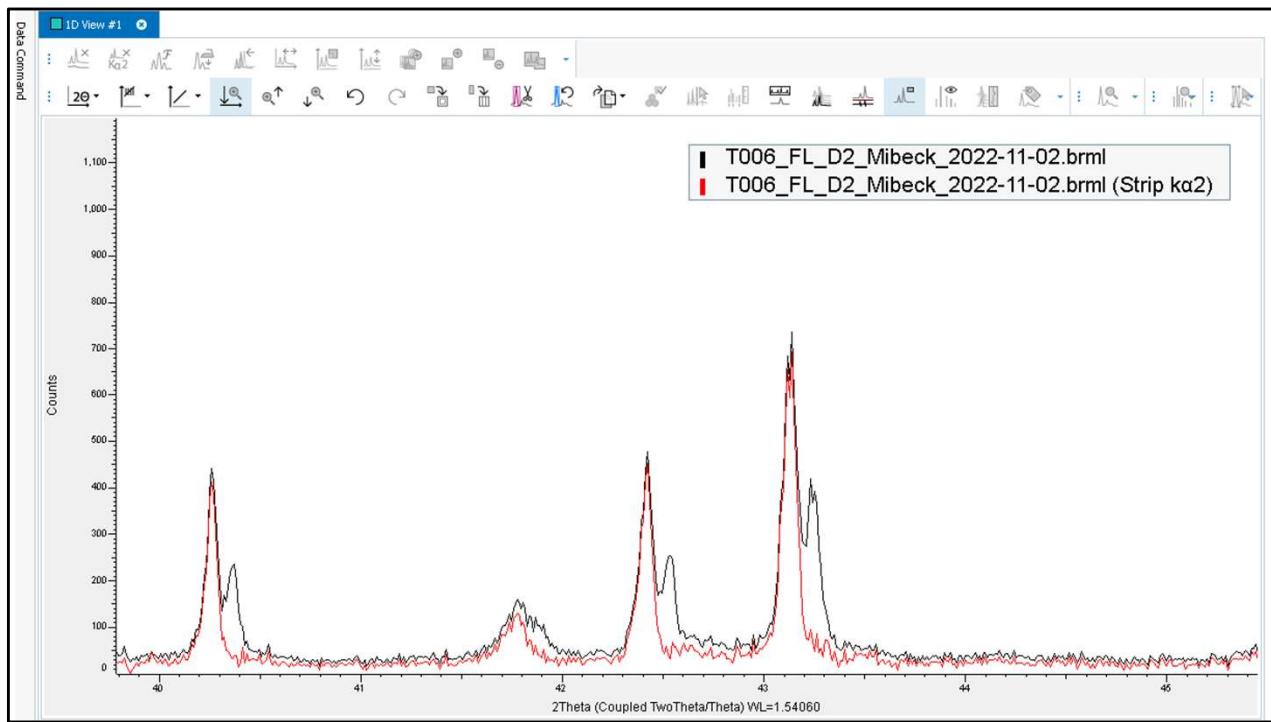
- 1mm, 3mm or ∞ mm (no knife)
- Decided on 1mm for low angles (clay peaks)





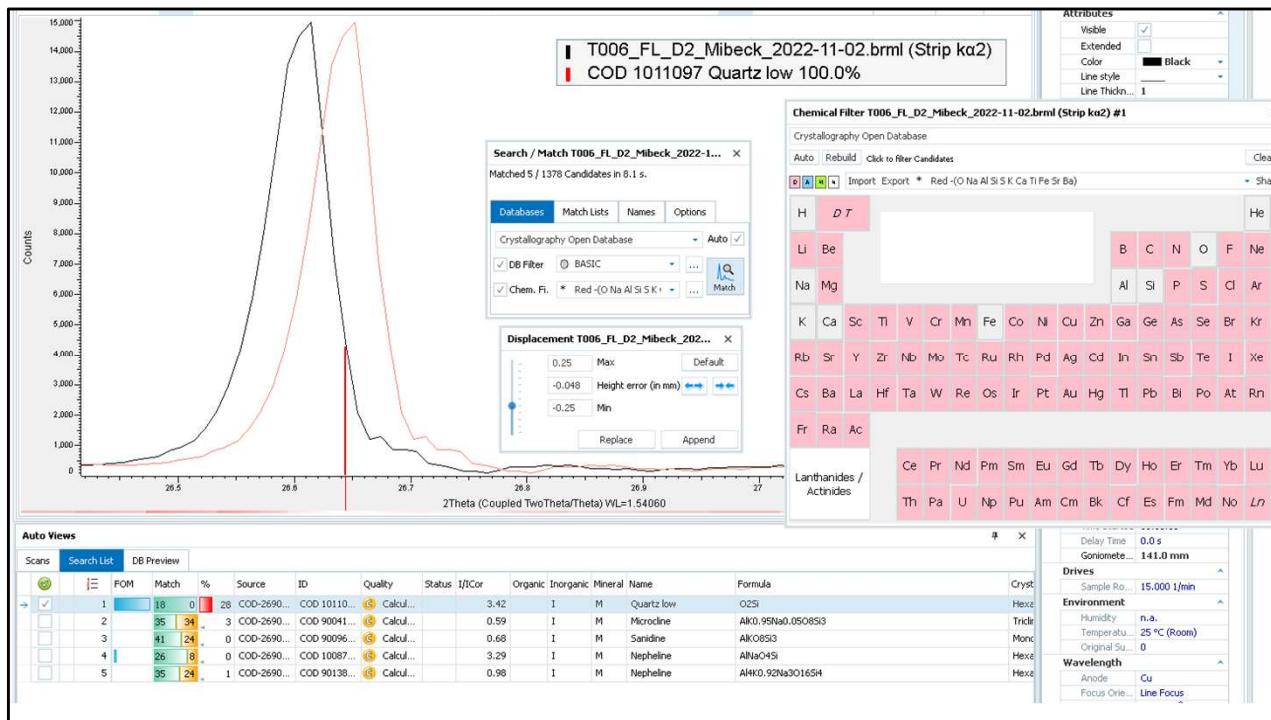
We use a python script to save the XRF results in a format compatible with EVA.

The sample script moves the raw data and XRF results in a folder that acts as working director – this is on a network drive so we all have access to the same files and folders...



After importing data we use EVA to strip the copper K alpha 2 line from the data.

Other wavelengths to be aware of are copper K beta and tungsten L



Quartz is everywhere and of all the minerals, it is the most well behaved.

So we usually start by using quartz to correct for sample displacement (shift aberration).

The XRF results are used to create an element filter for the Search/Match Step