Wellsite Mineralogical Data Acquisition; Understanding Results From Multiple Analytical Sources*

Brian J. Davis[1], Amber Koch[2], Gerald Braun[2], Cassie O'Connor[4], Elton Frost[5], Terry Quinn[5], Chi Vinh Ly[2], Guy Oliver[3], Graham Spence[3], Bill Whatley[6], Michael Wilson[2], and Neil Cardy[7]

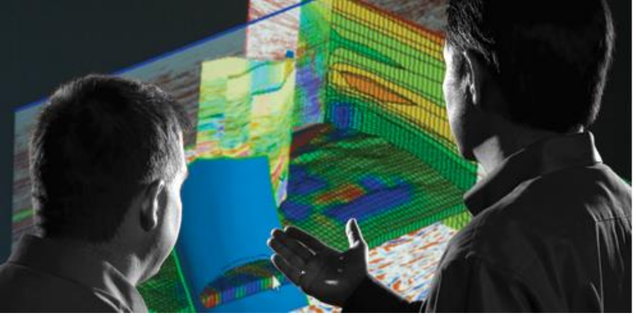
Search and Discovery Article #80478 (2015)**
Posted October 13, 2015

*Adapted from oral presentation given at AAPG Annual Convention & Exhibition, Denver, Colorado, May 31-June 3, 2015

[1]Geological Services, Baker Hughes Inc., Tomball, Texas, United States (bj.davis@bakerhughes.com)
[2]Geological Services, Baker Hughes Inc., Tomball, Texas, United States
[3]Robertson, CGG, Houston, Texas, United States
[4]Surface Logging Services, Baker Hughes Inc., Houston, Texas, United States
[5]Wireline Services, Baker Hughes Inc., Houston, Texas, United States
[6]Business Development, Baker Hughes Inc., Houston, Texas, United States
[7]Consultant

Abstract

Technological advances in both equipment and computer software have enabled the implementation of new approaches in generation of mineralogical datasets at petroleum wellsites. These datasets are currently utilized by hydraulic fracturing engineers to assist in designing optimized fracture stage intervals in horizontal wellbores, rather than using evenly spaced intervals between treatment stages. Mineralogical data is generated by downhole wireline logging tools, and on drill cuttings, conventional wholecores, and rotary sidewall coreplugs utilizing a variety of analytical instrumentation techniques. This paper documents a study undertaken to assess mineralogical datasets generated on comparable samples, focused on evaluating analytical limitations and variances, toward obtaining consistent mineralogical results. Instrumentation typically used to generate these datasets include x-ray diffraction (XRD), x-ray fluorescence (XRF), scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), fourier transform infrared spectroscopy (FTIR), and inductively coupled plasma techniques (ICP mass spectroscopy or ICP optical emission spectroscopy). Variables introduced into the analysis in addition to the different analytical techniques include sample types and sizes, sampling methods, sample preparation, drilling mud contaminants, lithological heterogeneity, and depth correlations between cuttings, cores, and wireline measurements. Equipment destined for wellsite analysis was evaluated in a controlled laboratory environment using reference mineral standards and standard mixtures to understand testing limitations and refine mineral phase calculations. Mineral terminologies, classifications, compositions, and the resulting databases were reviewed for consistency. Multiple cuttings and core sample sets from conventional sandstones, carbonates, and current mudrock plays such as the Eagle Ford and Marcellus shales were sub-divided and analyzed to allow direct comparisons of generated datasets. This study yielded increased confidence in wellsite and laboratory analyses, including caveats where necessary, procedural guidelines for each analytical technique, and verification of deliverables appropriate to unconventional mudstone reservoirs. Example datasets, graphical comparisons, and report formats are included. The resultant wellsite datasets, in tandem with additional wellsite analytics, enhance confidence in optimized fracture stage interval decisions.



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[1] Baker Hughes Incorporated [2] CGG [3] Consultants

Acknowledgements

- Baker Hughes
- CGG
- Co-authors
- Dr. Gerald Braun

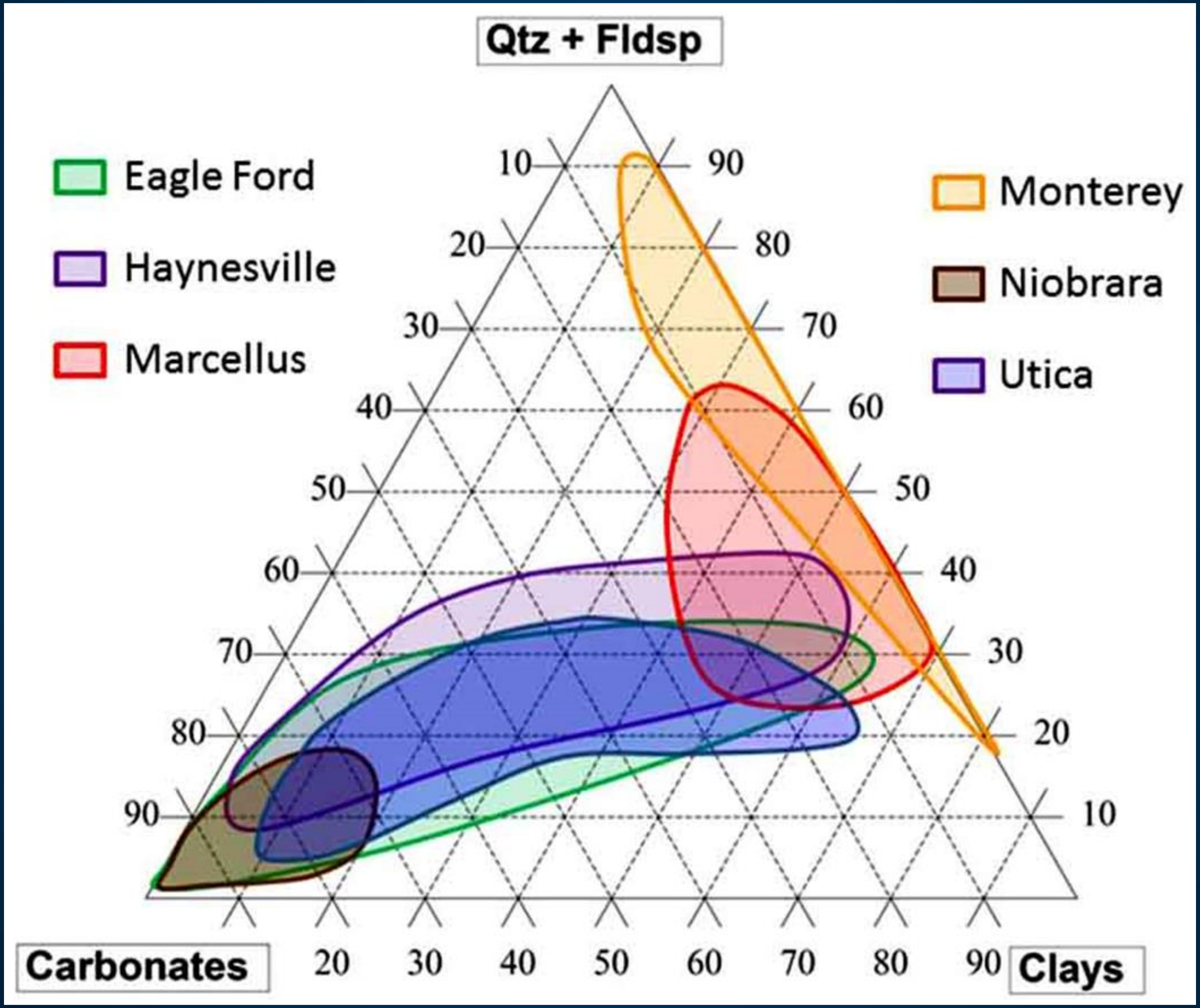
Overview

- Evaluation and vetting two new wellsite mineralogical services
- Comparative mineralogical studies: Laboratory vs Wellsite
    - Drill cuttings
    - Coreplugs
- Study yielded increased confidence in the quality of wellsite mineralogical analysis
    - improvements in mineralogical standards
    - sample acquisition

Outline

- Background of mineralogical analysis
- Description of mineralogical analytical techniques used in study
- Data sets from study
- What did we learn?

Background → Techniques → Data sets → Knowledge

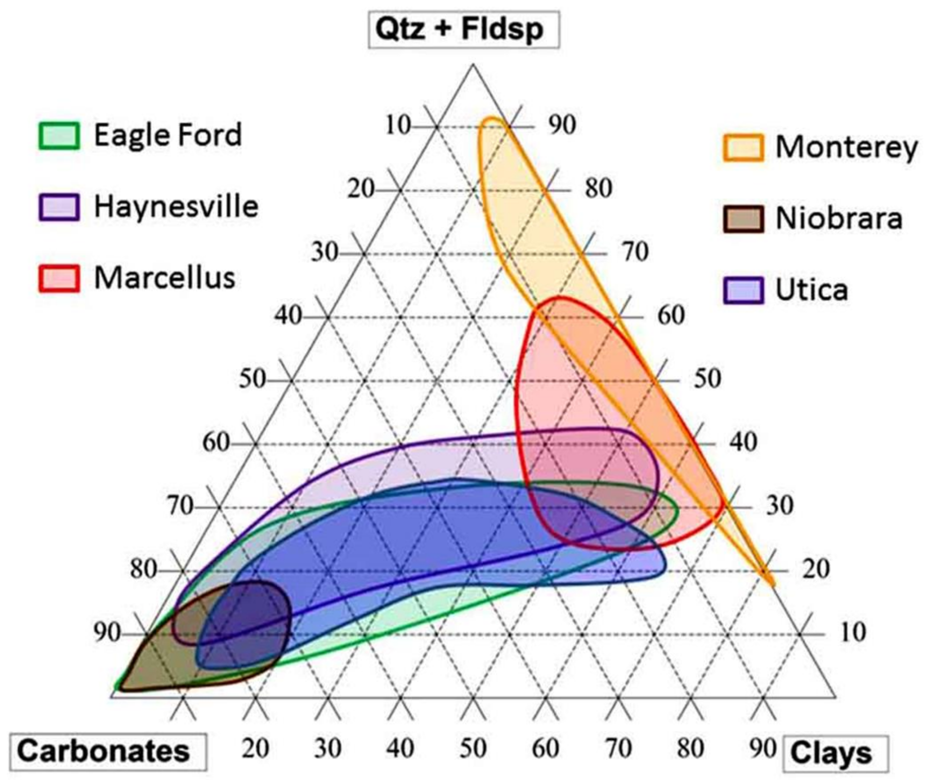


Background → Techniques → Data sets → Knowledge

- Silicates, iron sulfides, calcium phosphate
  - quartz, feldspars, pyrite, marcasite, apatite

- Carbonates
  - calcite, dolomite, fe-dolomite, siderite

- Clays
  - illite, chlorite, kaolinite, mixed-layer illite/smectite, mixed-layer chlorite/smectite, smectite, mica



Mineralogical Analysis Goals at the Wellsite

- Understanding reservoir
  - Formation boundaries used to indicate landing points for horizontal laterals
  - Locate ideal placement for perforation clusters using Brittleness Index
- Wellbore geosteering
  - Requires rapid data turnaround
- Insights on fluid compatibility issues
  - Mineral compositions that may create precipitates or formation damage
- Optimize hydraulic fracture treatment designs
  - >70% of USA wells are horizontal laterals are hydraulically fractured with very limited data on reservoir variability

Mineralogical Data Sources

- Logging While Drilling (LWD)
- Wireline Logging

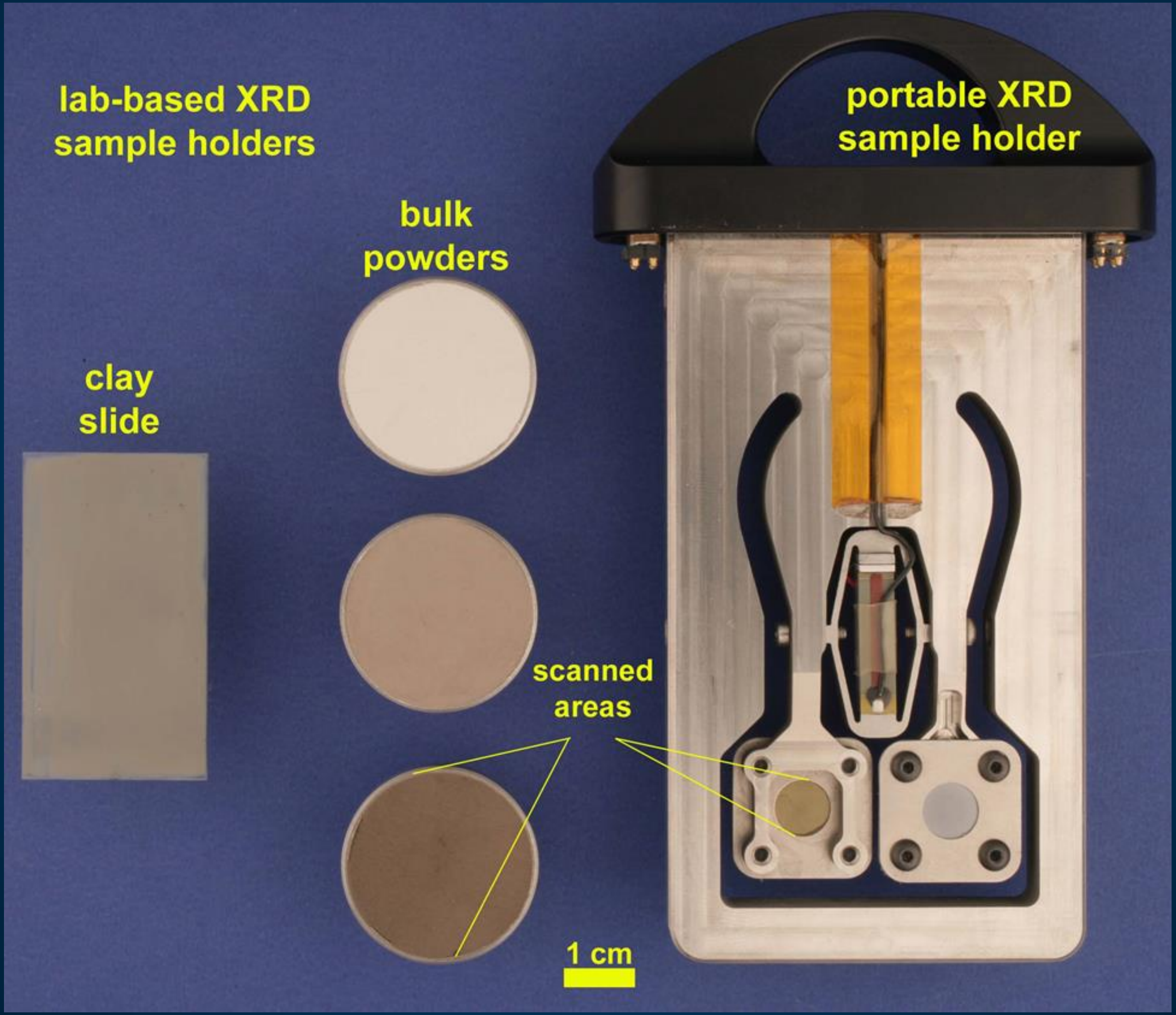
- X-ray Diffraction (XRD)
- X-ray Fluorescence Elemental Analysis (XRF)
- Inductivity Coupled Plasma Spectroscopy (ICP)
- Fourier Transform Infrared Spectroscopy (FTIR)
- Near Infrared Spectroscopy (NIR)
- Automated Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS)

Portable vs Lab X-ray diffraction techniques

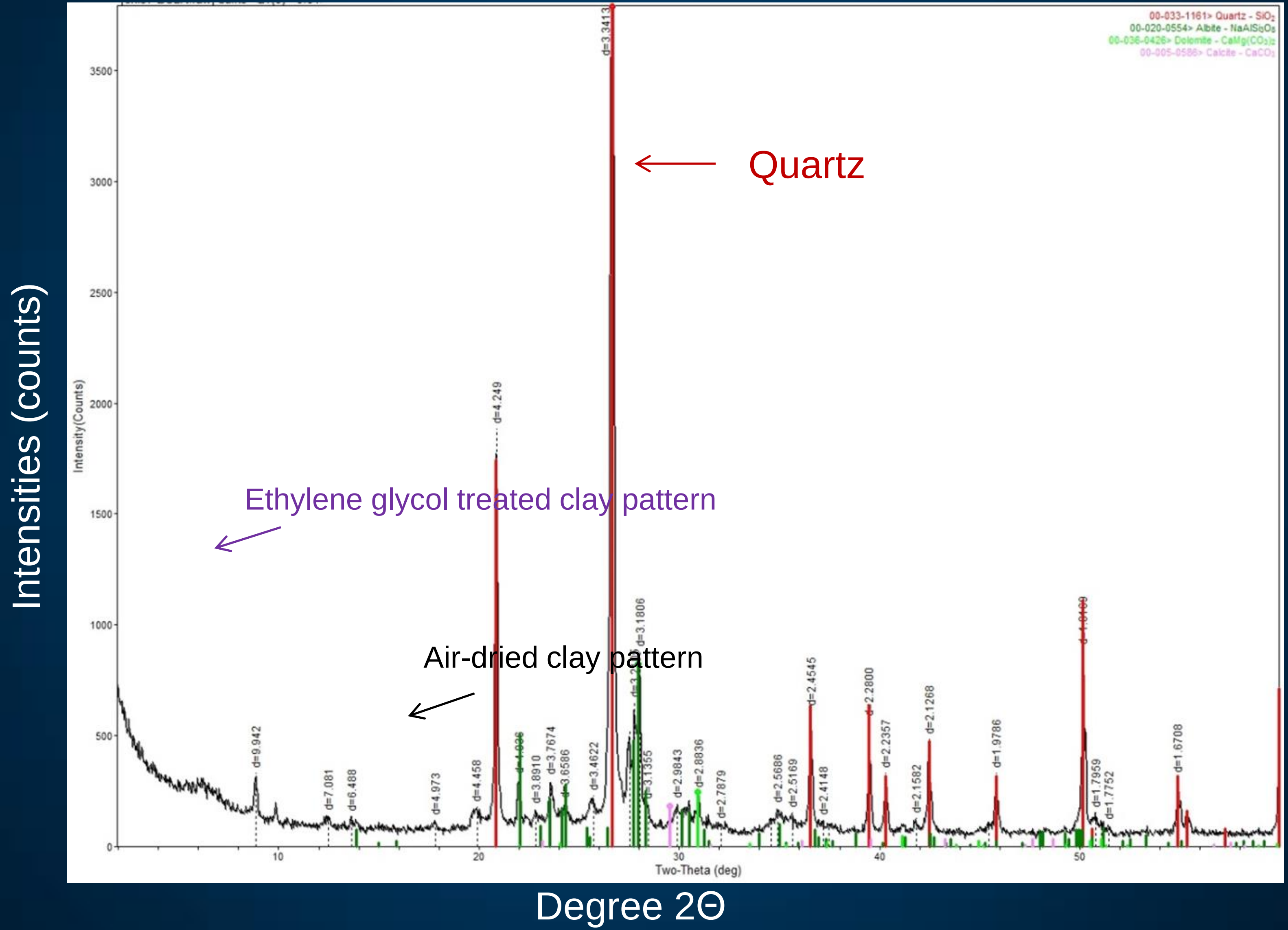
- Proprietary “tuning fork” sample holder
- Cuttings ground to ~150 microns with mortar and pestle
- Cobalt anode allowing for improved observation of clay minerals and greater peak separation
- Expedited results due to short sample analysis time

- Disc sample holder with larger area of investigation
- Sample ground to ~5 microns
- Variety of milling techniques available
- Copper anode allowing for general use in Lab setting
- High resolution results due to longer analysis time
- Determination of clay expandablity

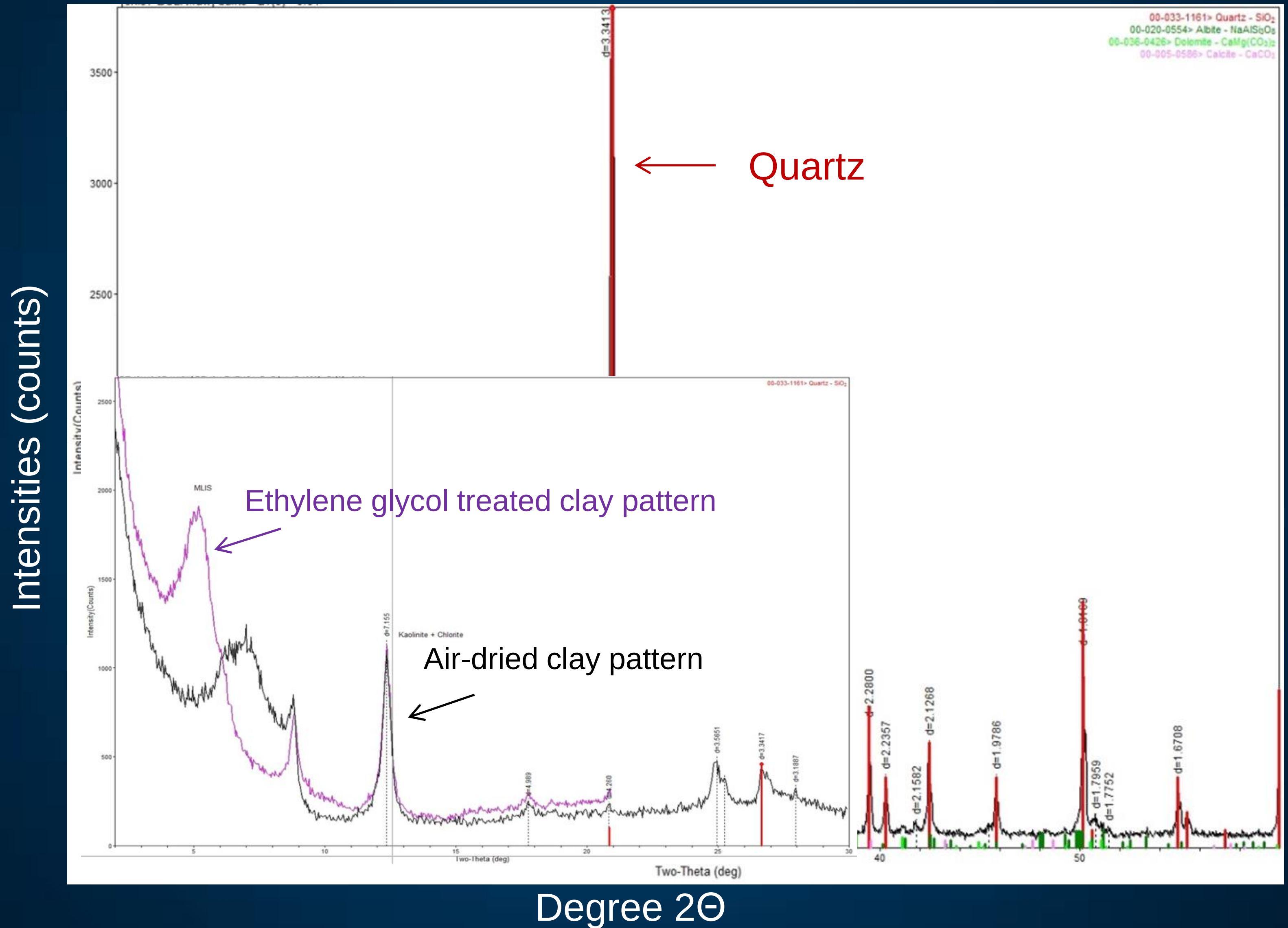
Background → Techniques → Data sets → Knowledge



Background → Techniques → Data sets → Knowledge



Background → Techniques → Data sets → Knowledge



Background → Techniques → Data sets → Knowledge

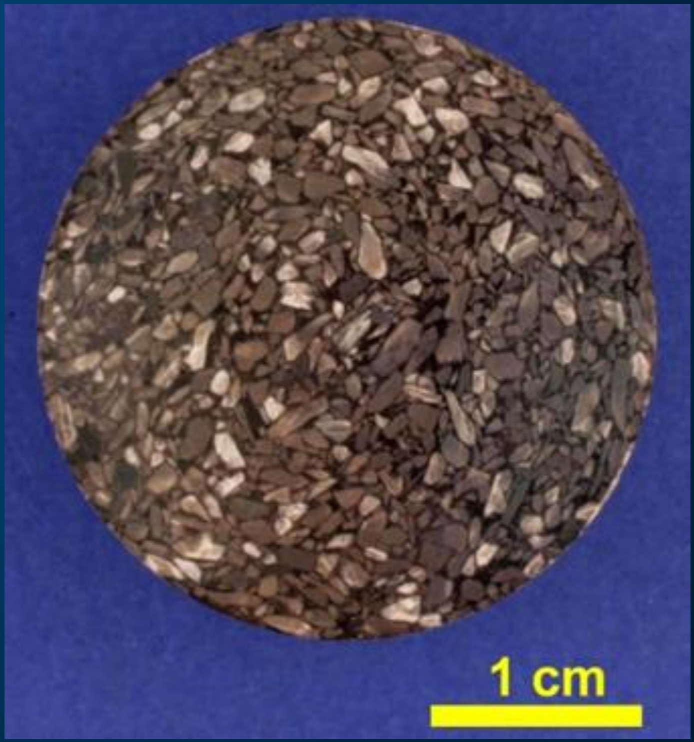
Mineral Phases	14429'	14551'	14802'	14905'	14940'
Quartz (SiO_2)	75	72	65	74	40
Plagioclase Feldspar	8	8	12	9	22
Potassium Feldspar	6	8	5	3	14
Dolomite ($CaMg[CO_3]_2$)	*trace*	*trace*	*trace*	*trace*	*trace*
Pyrite (FeS_2)	nd	nd	nd	nd	*trace*
Mica and/or Illite	1	2	3	1	3
Kaolinite	5	3	5	4	7
Chlorite	*trace*	*trace*	*trace*	*trace*	1
Mixed-Layer $Illite_{60}/Smectite_{40}$	3	5	8	7	11
TOTALS	100%	100%	100%	100%	100%

Review of the air-dried and glycol-solvated clay slides indicates that these mixed-layer illite/smectite clays are composed of **40% expandable smectite layers**.

nd - if present, the amount was below the detectable capabilities of x-ray diffraction analysis.

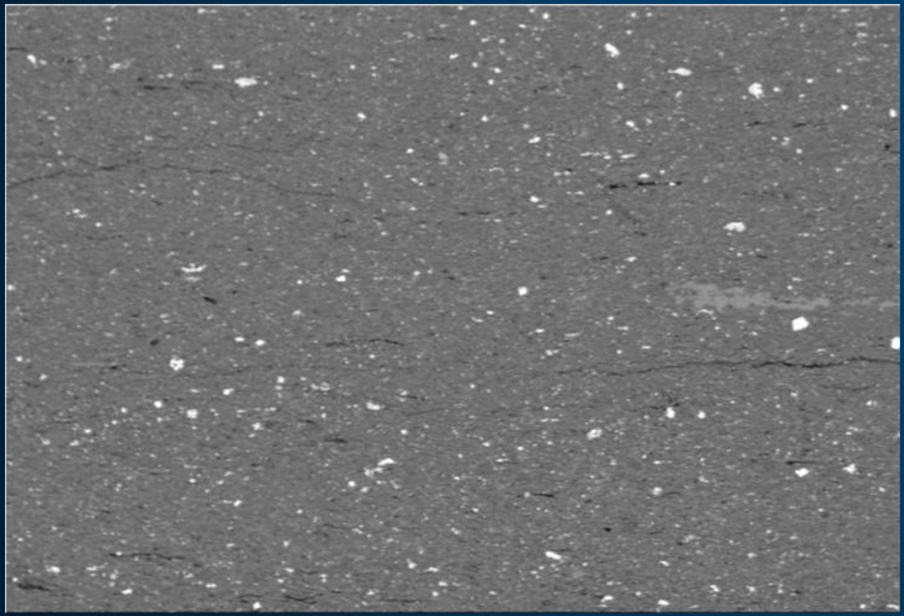
SEM-EDS Mineralogical and Textural Analysis

- Ruggedized SEM with 1-4 EDS detectors
- Computer-controlled motor driven stage
- Samples mounted in acrylic sample holder
- Ground with 1200 grit powder
- Carbon sputter-coated
- Pixel-by-pixel image analysis
- Pixel-by-pixel EDS chemical signatures
- Dictionary listing of mineral phases with associated elemental suites
- Yields spatially-resolved mineral species

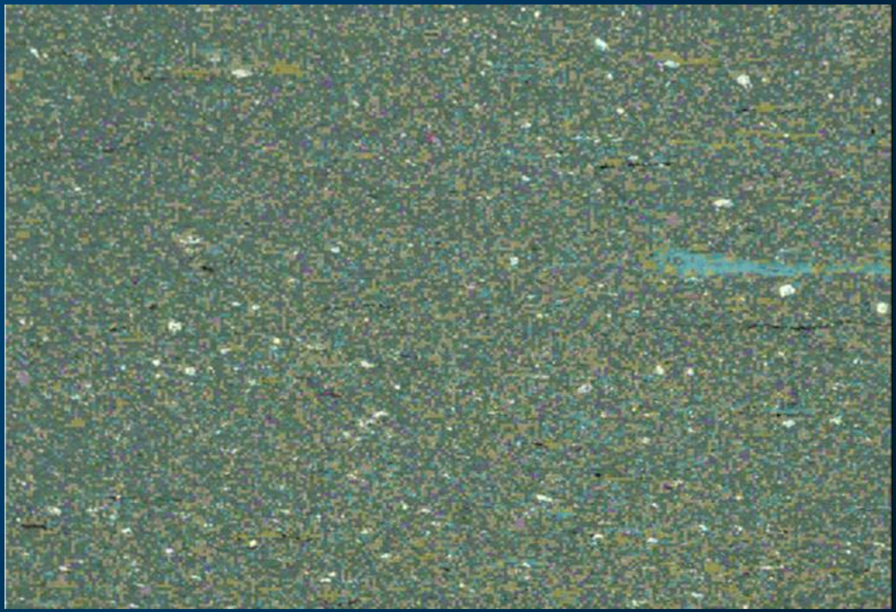


SEM-EDS Automated Mineralogy example:

Marcellus Shale



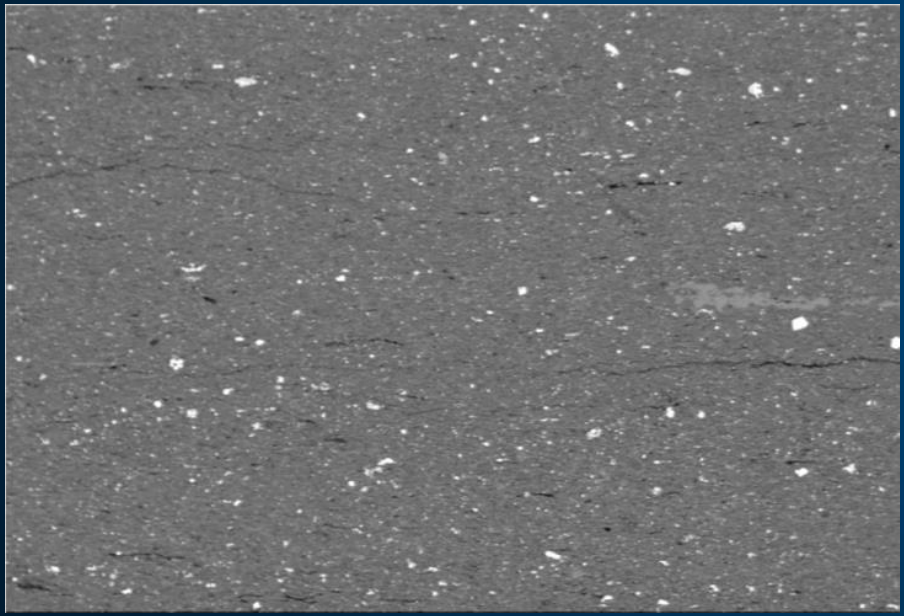
BSE Elemental Image



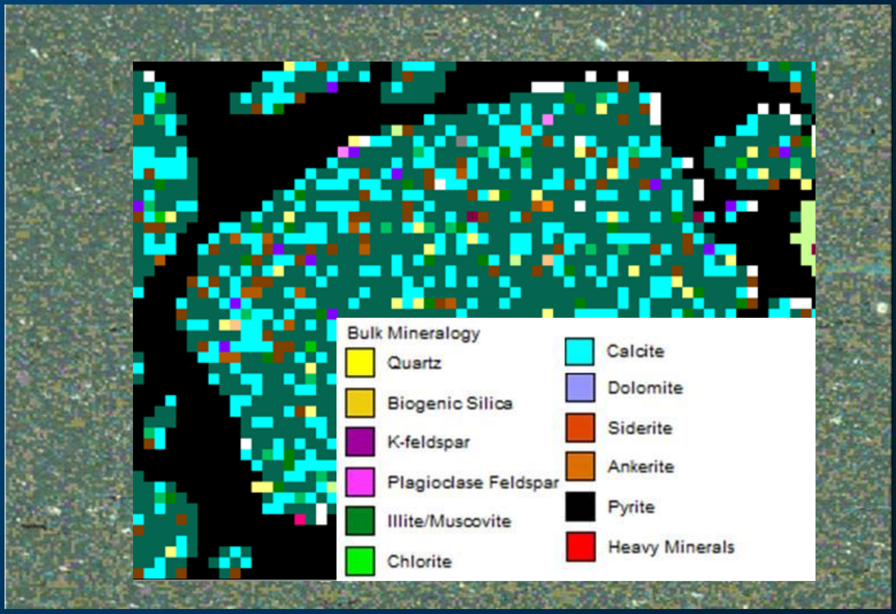
Interpreted Mineral Map

SEM-EDS Automated Mineralogy example:

Marcellus Shale

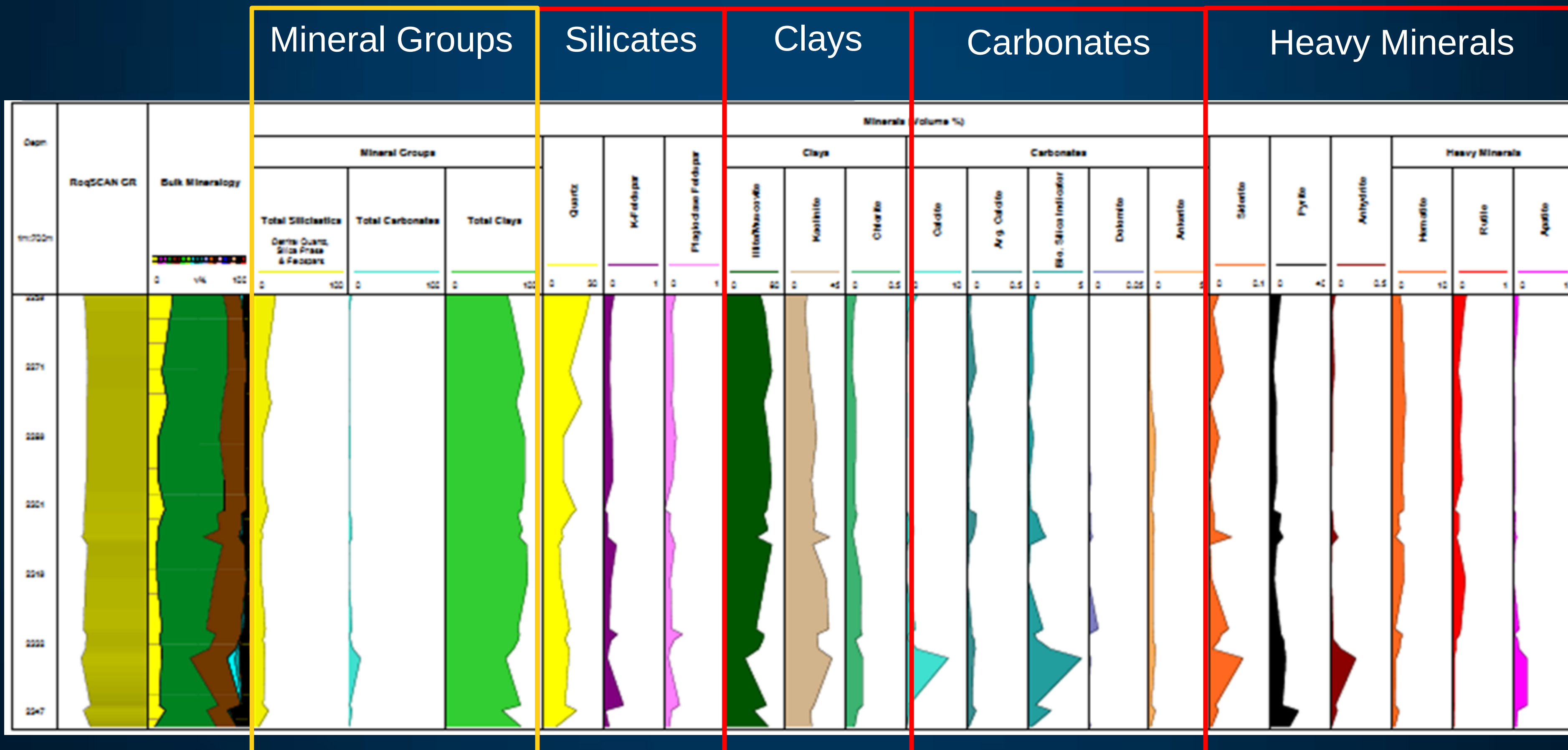


BSE Elemental Image



Interpreted Mineral Map

SEM-EDS Mineralogical Graphic



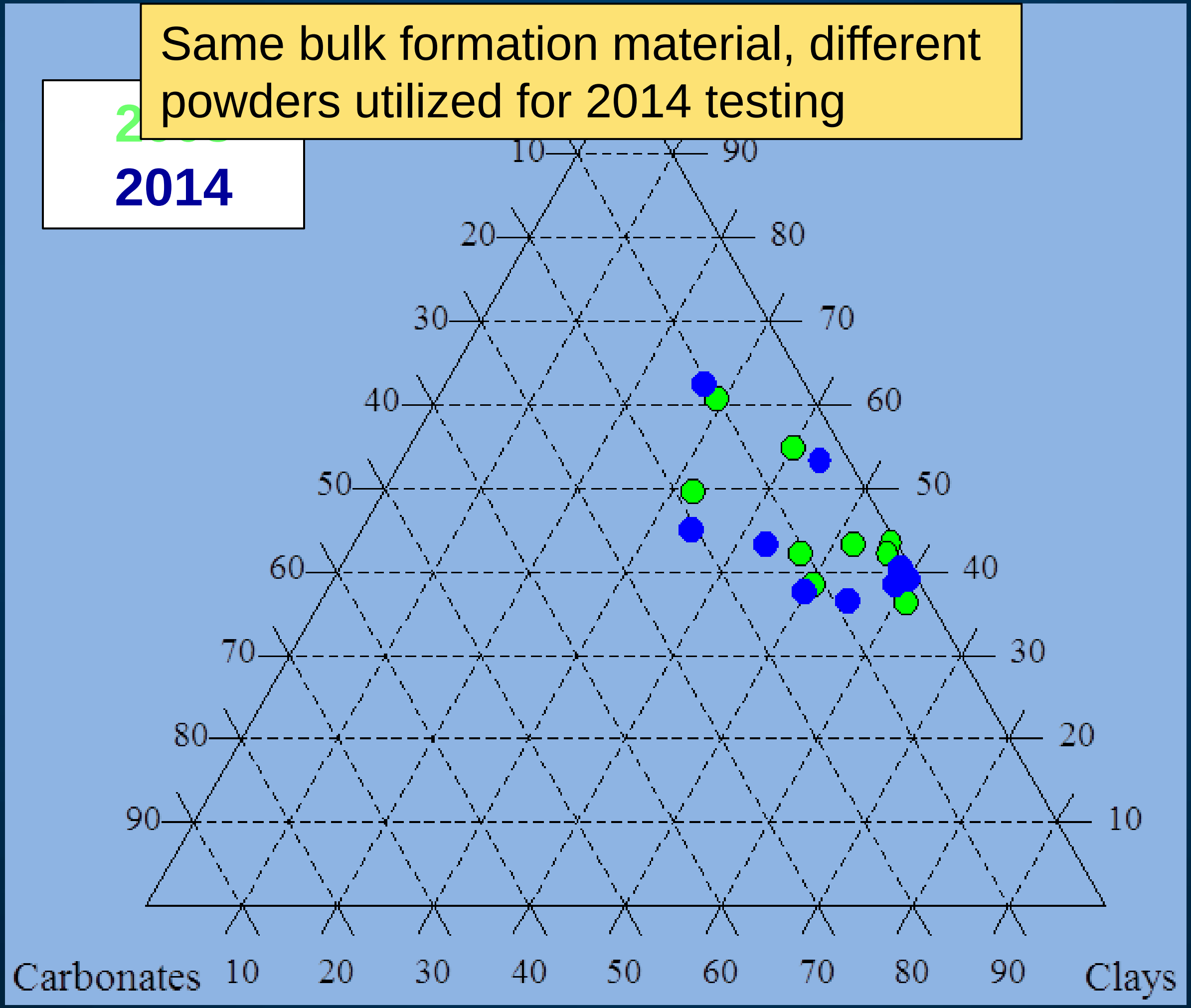
Background → Techniques → Data sets → Knowledge

- **Comparison of X-ray Diffraction (XRD)**
  - **Lab-based XRD**
  - **Portable XRD**
- **Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS)**

- **Drill Cuttings (4 sample sets)**
  - **Eagle Ford Shale**
  - **Woodbine Sandstone**
  - **Wolfcamp Shale**
  - **Marcellus Shale**

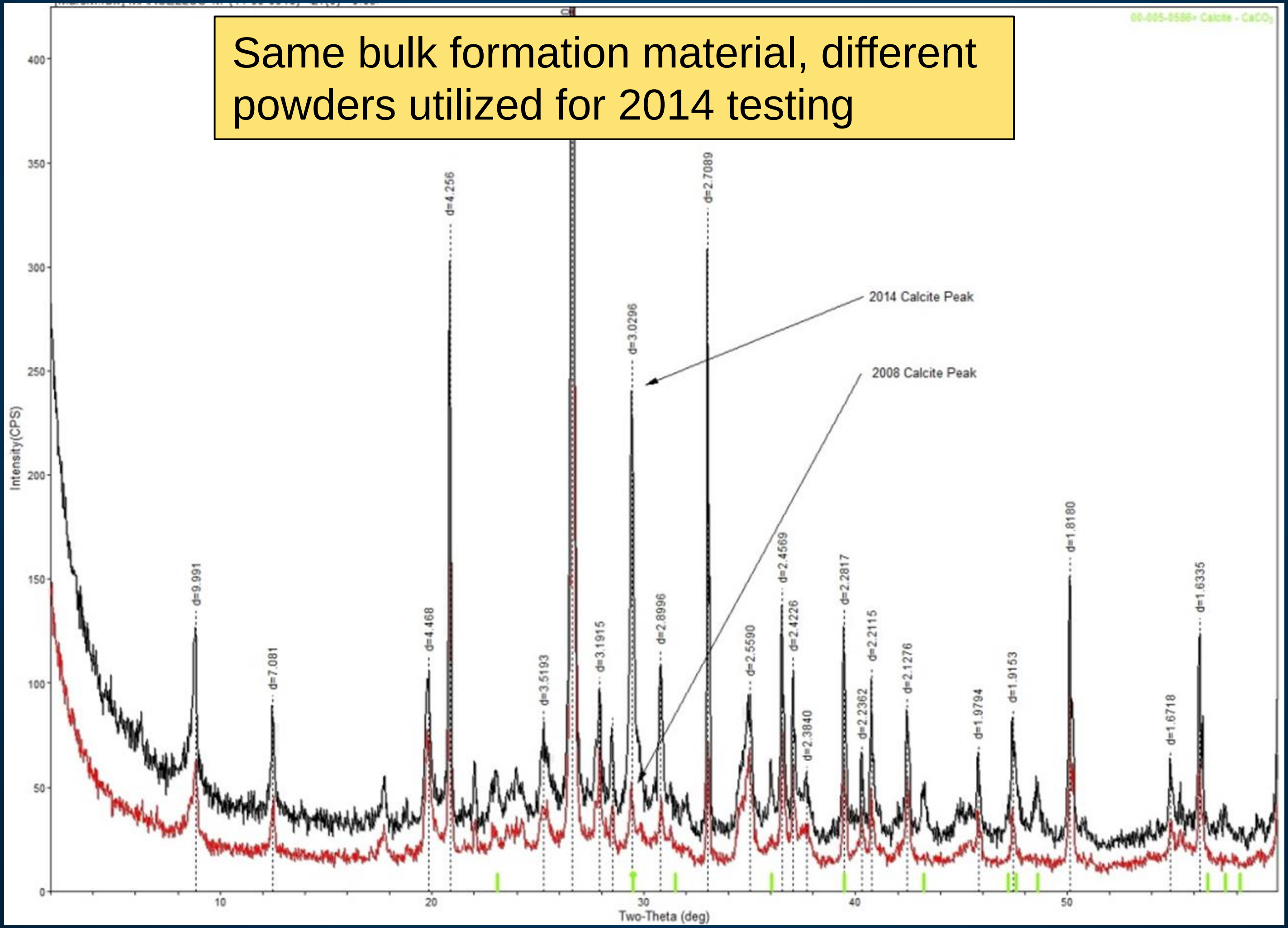
- **Coreplugs (4 sample sets)**
  - **Marcellus Shale**
  - **Pennsylvanian Sandstones & Shales from Oklahoma**
  - **Diverse Lithologies" set: Australian siltstone, sandstones from Chile, Alberta, and West Texas, onshore carbonates from Mississippi and Congo, California diatomite**
  - **Clay-rich international shale**

Comparison of 2008 and 2014 Lab XRD mineralogical analysis

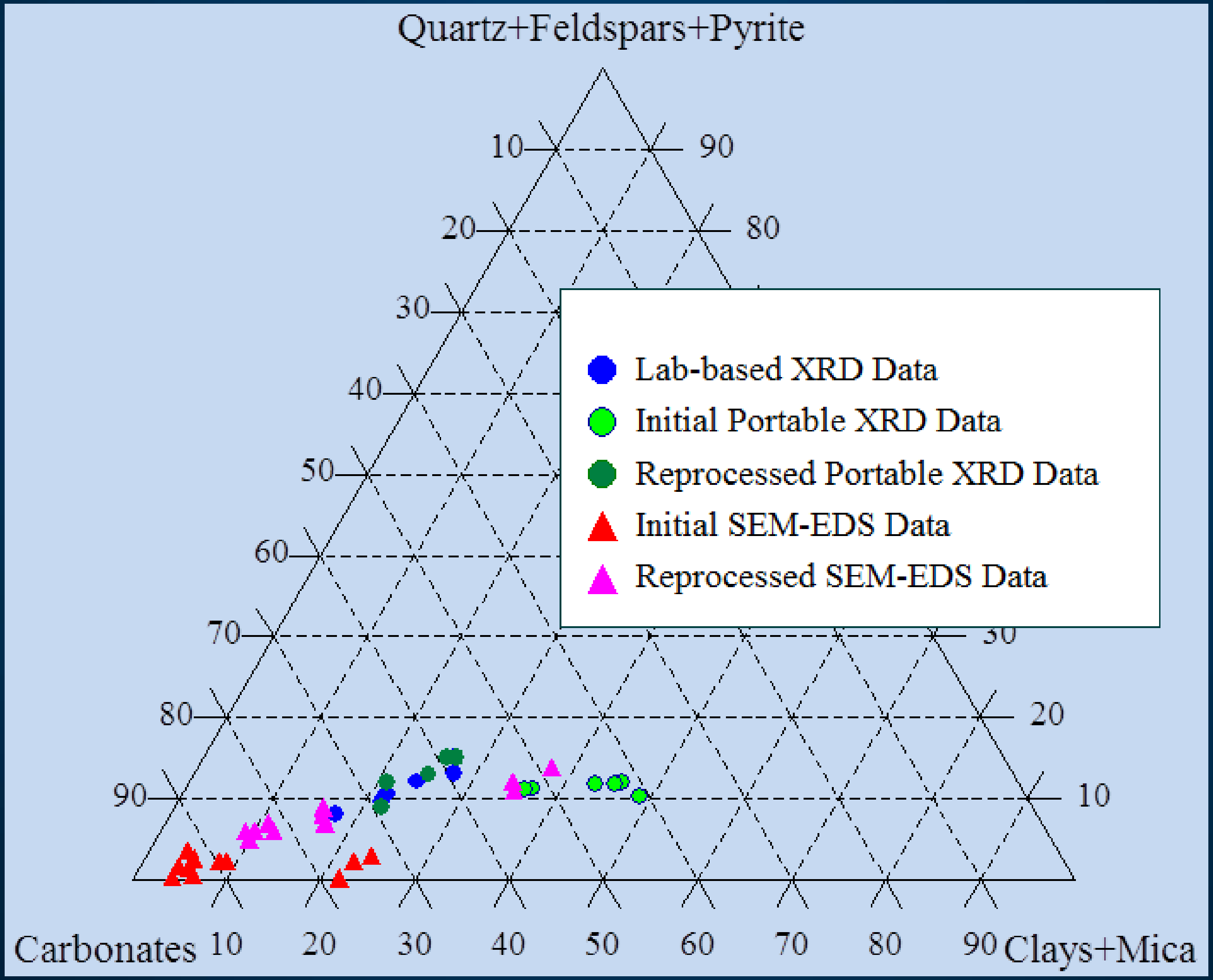


Comparison of 2008 and 2014 Lab XRD mineralogical analysis

Same bulk formation material, different powders utilized for 2014 testing



Cuttings dataset 1:Eagle Ford Shale (carbonate mudstone)

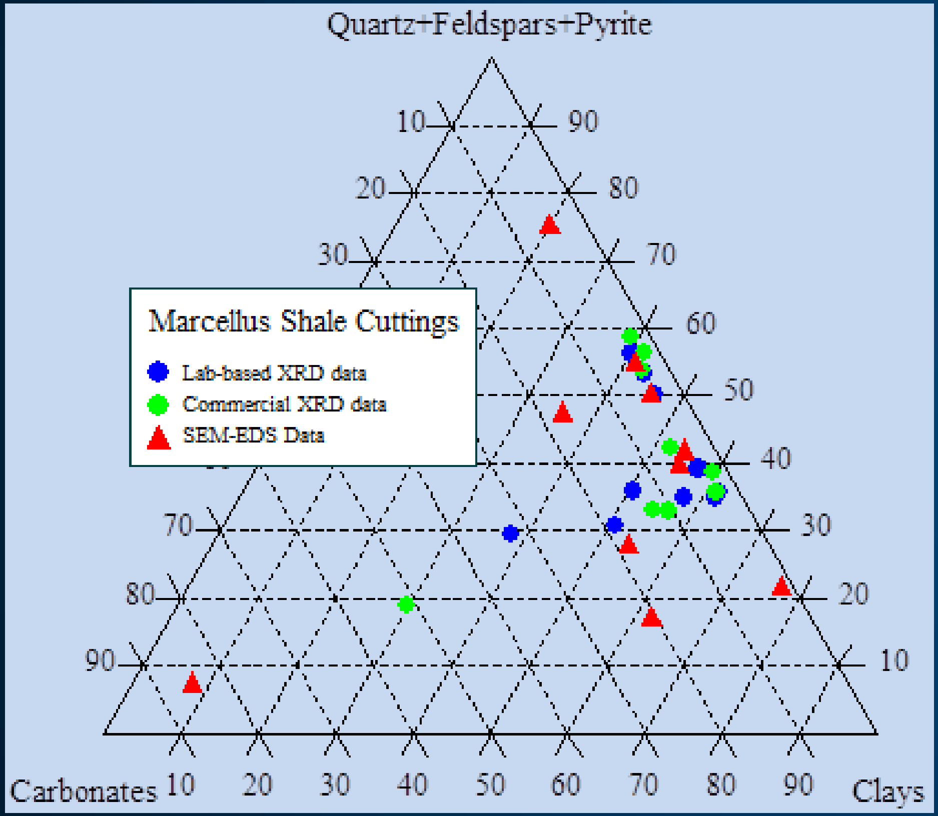


Background → Techniques → Data sets → Knowledge

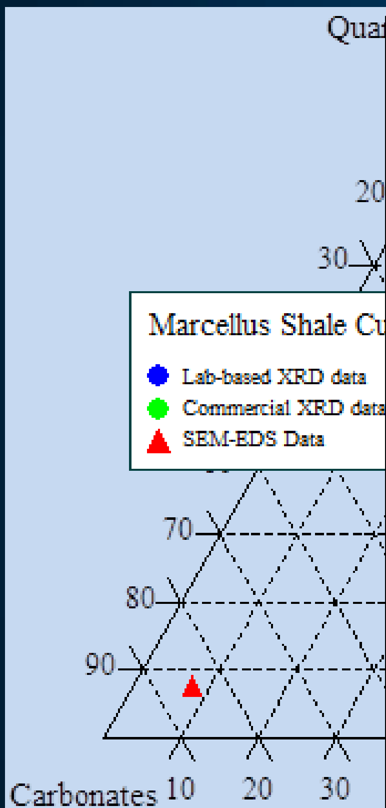
Data set 2:
Marcellus Frm.

Data set 3:
Wolfcamp Frm.

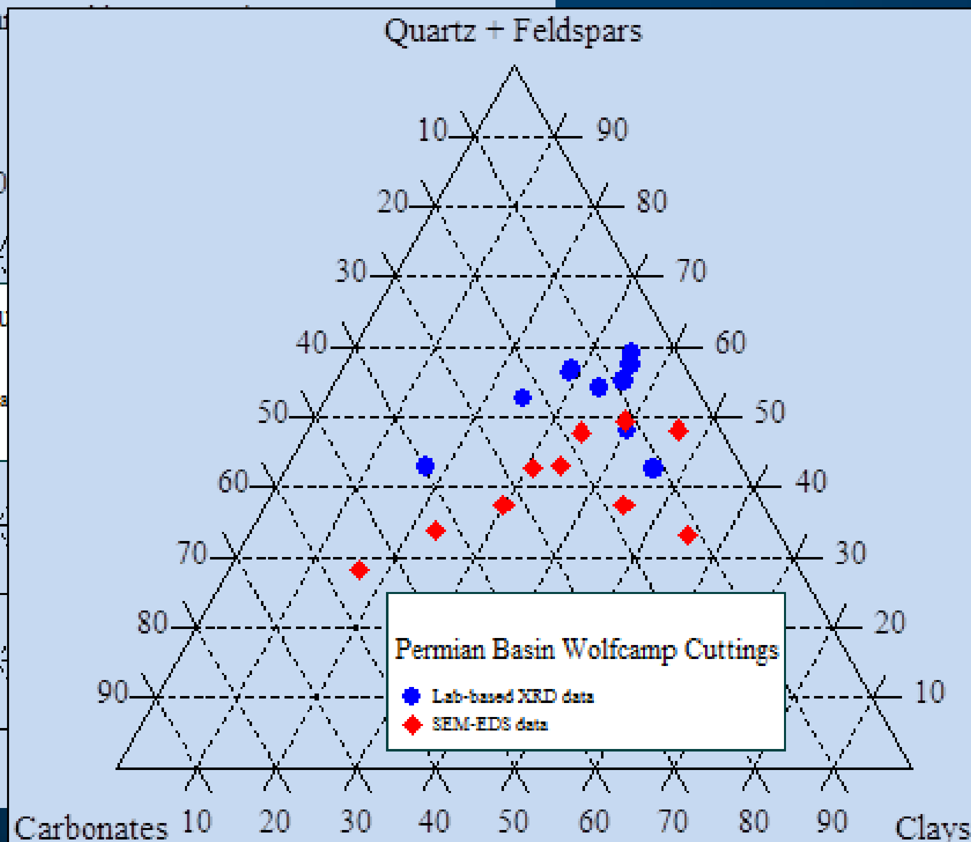
Data set 4:
Woodbine Frm.



Data set 2: Marcellus Frm.



Data set 3: Wolfcamp Frm.

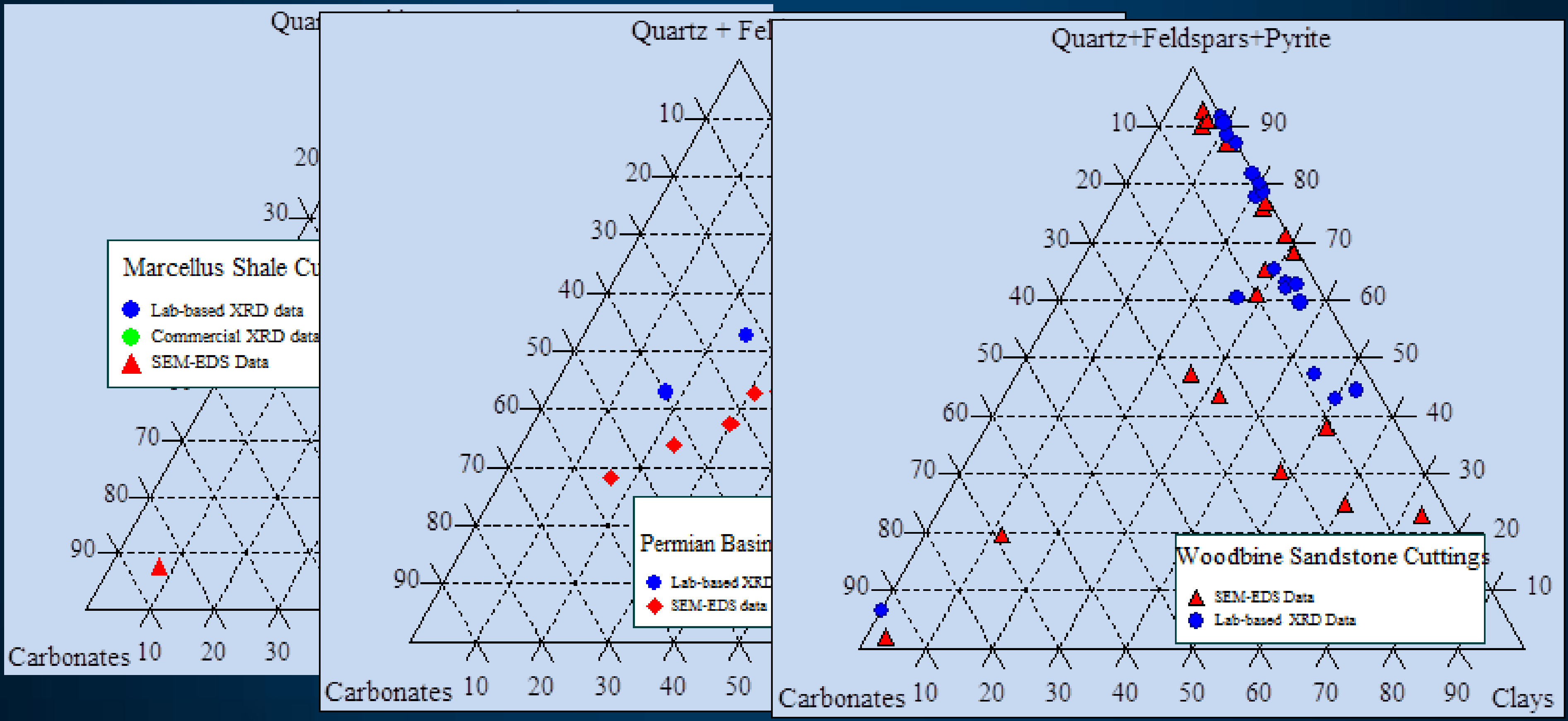


Data set 4: Woodbine Frm.

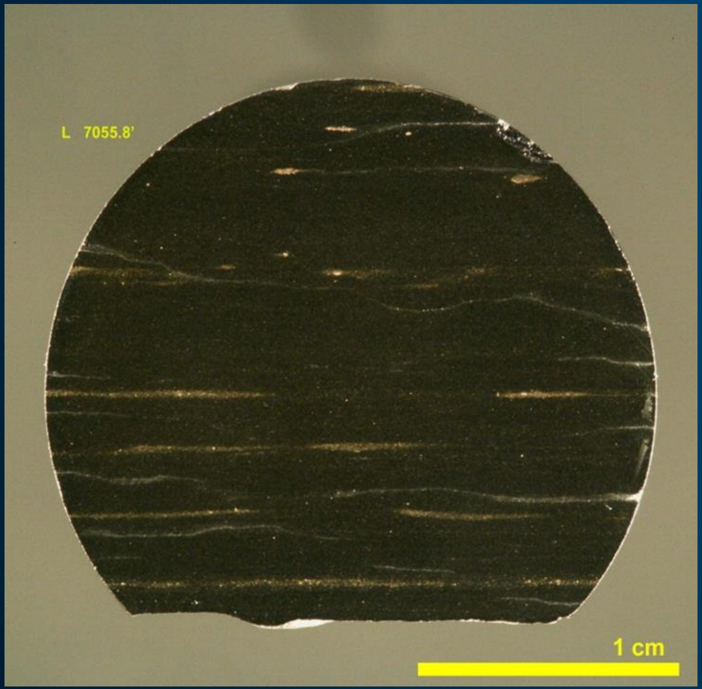
Data set 2: Marcellus Frm.

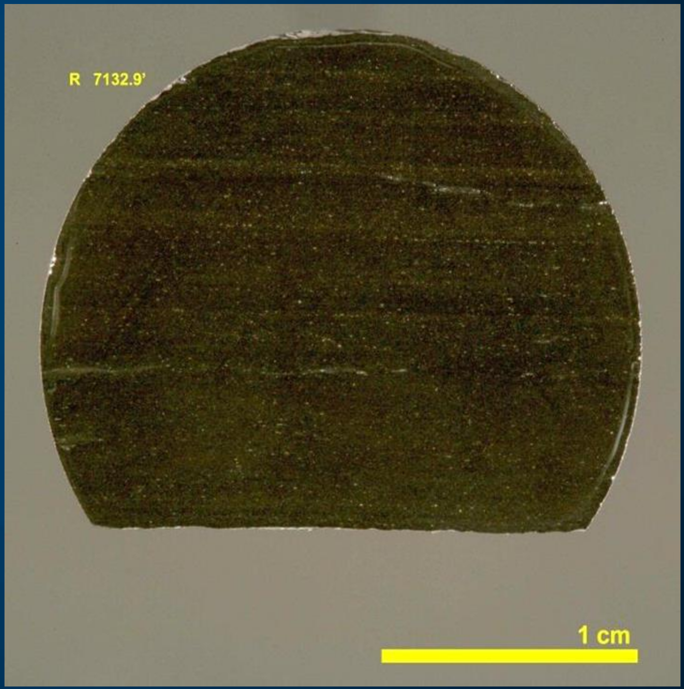
Data set 3: Wolfcamp Frm.

Data set 4: Woodbine Frm.



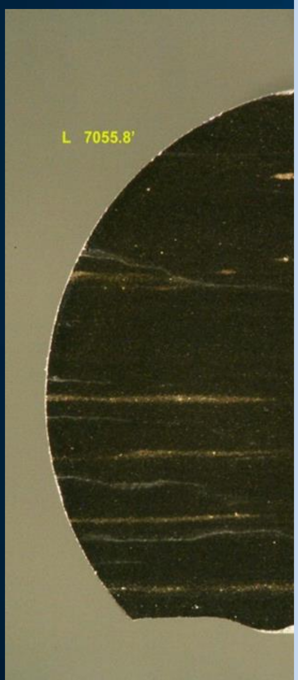
Coreplugs, Data set 1: Marcellus Shale (Drilled 1” Coreplugs)

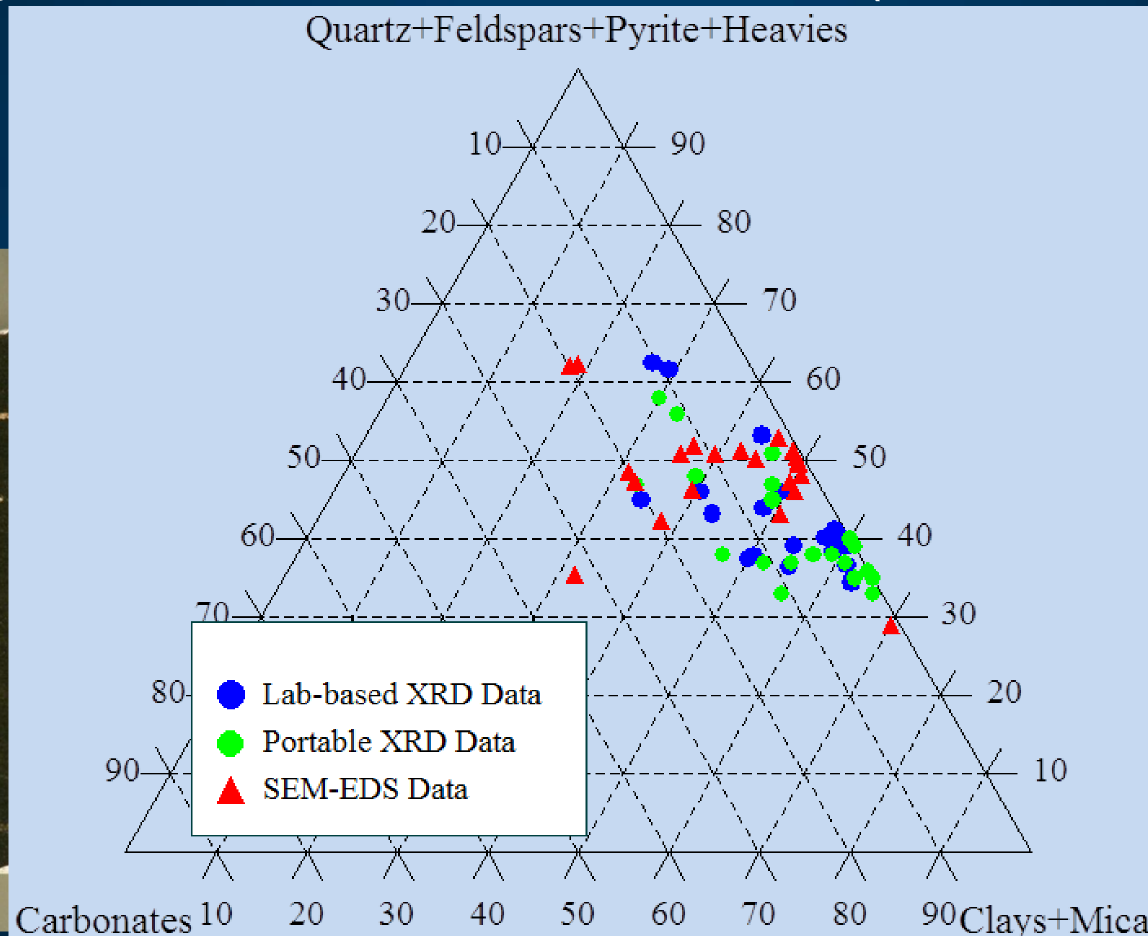


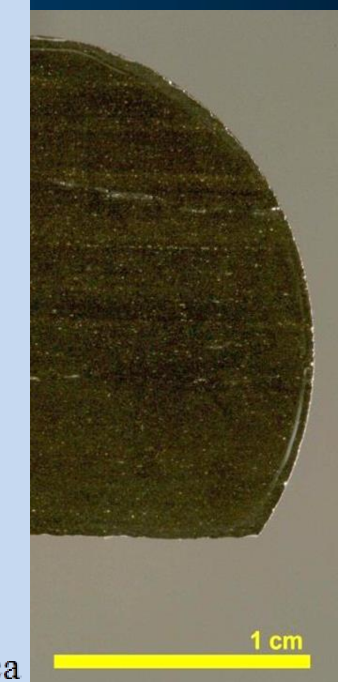


Enhanced image contrast illustrates variability and concentrations of mineral phases

Coreplugs, Data set 1: Marcellus Shale (Drilled 1" Coreplugs)

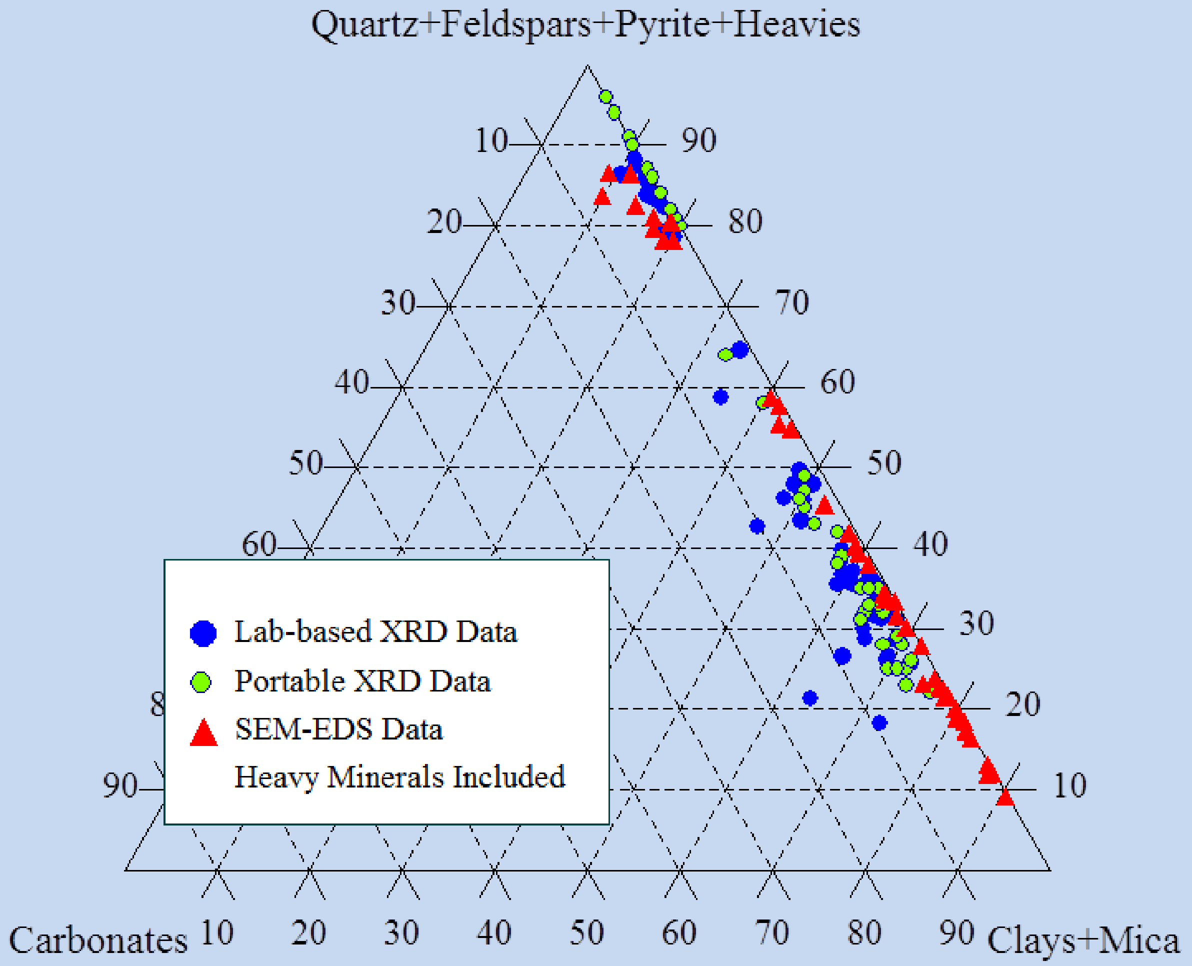




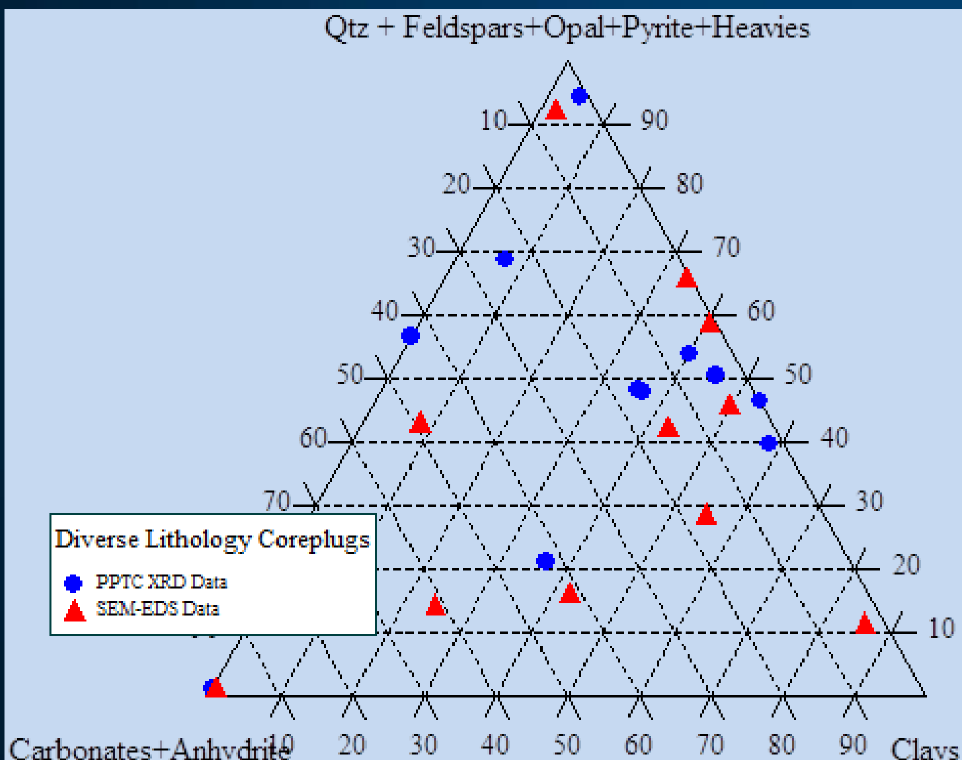


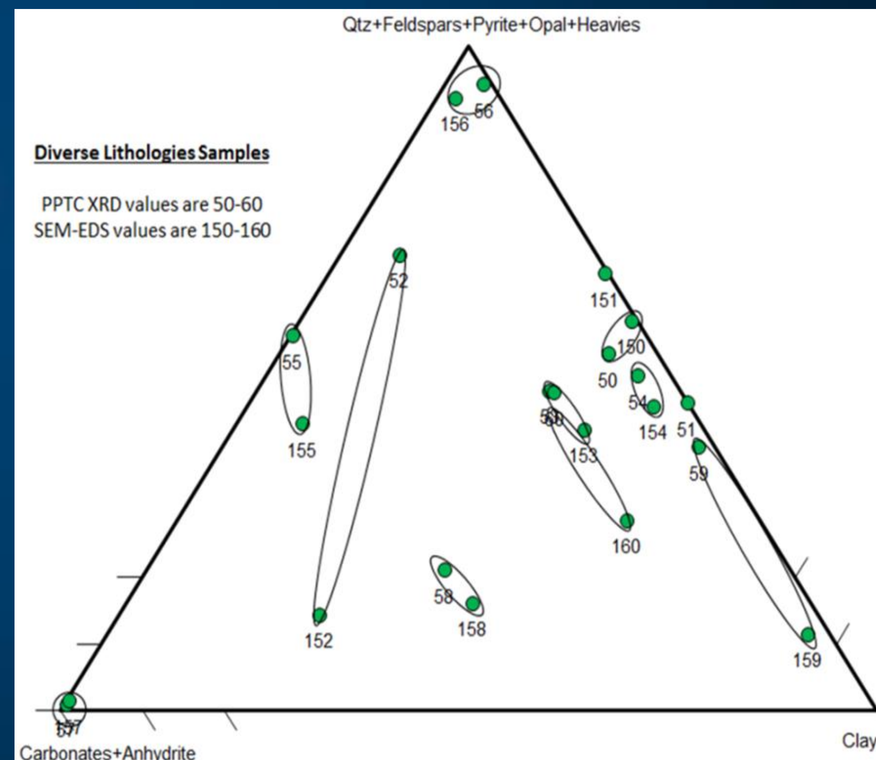
Enhanced image contrast illustrates variability and concentrations of mineral phases

Coreplugs, Data set 2: Pennsylvanian Sandstones and Shales (Rotary Sidewall Coreplugs)



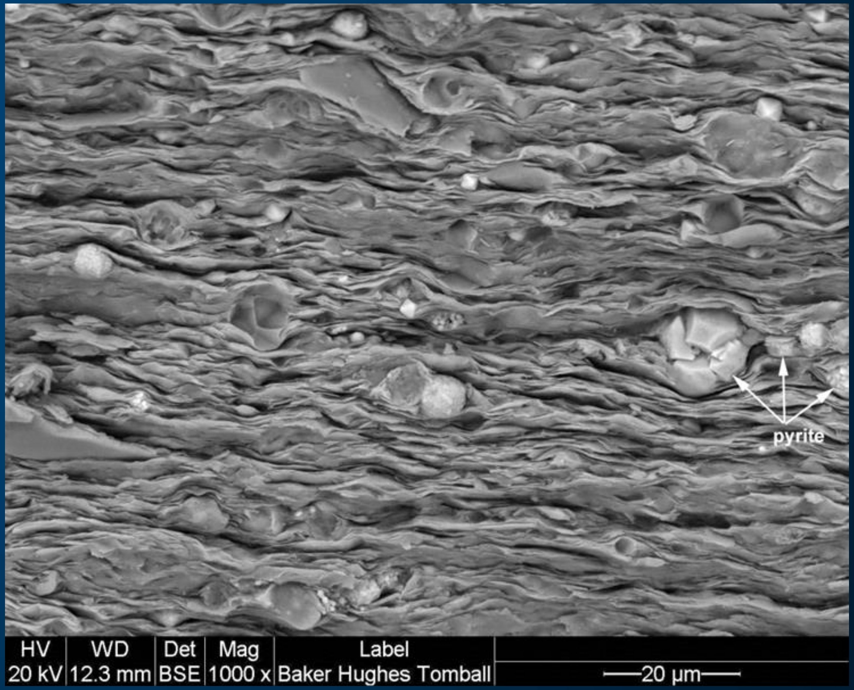
Data set 3: Diverse Lithologies: Sandstones, Siltstones, Shales, Carbonates, Diatomite Mudstone





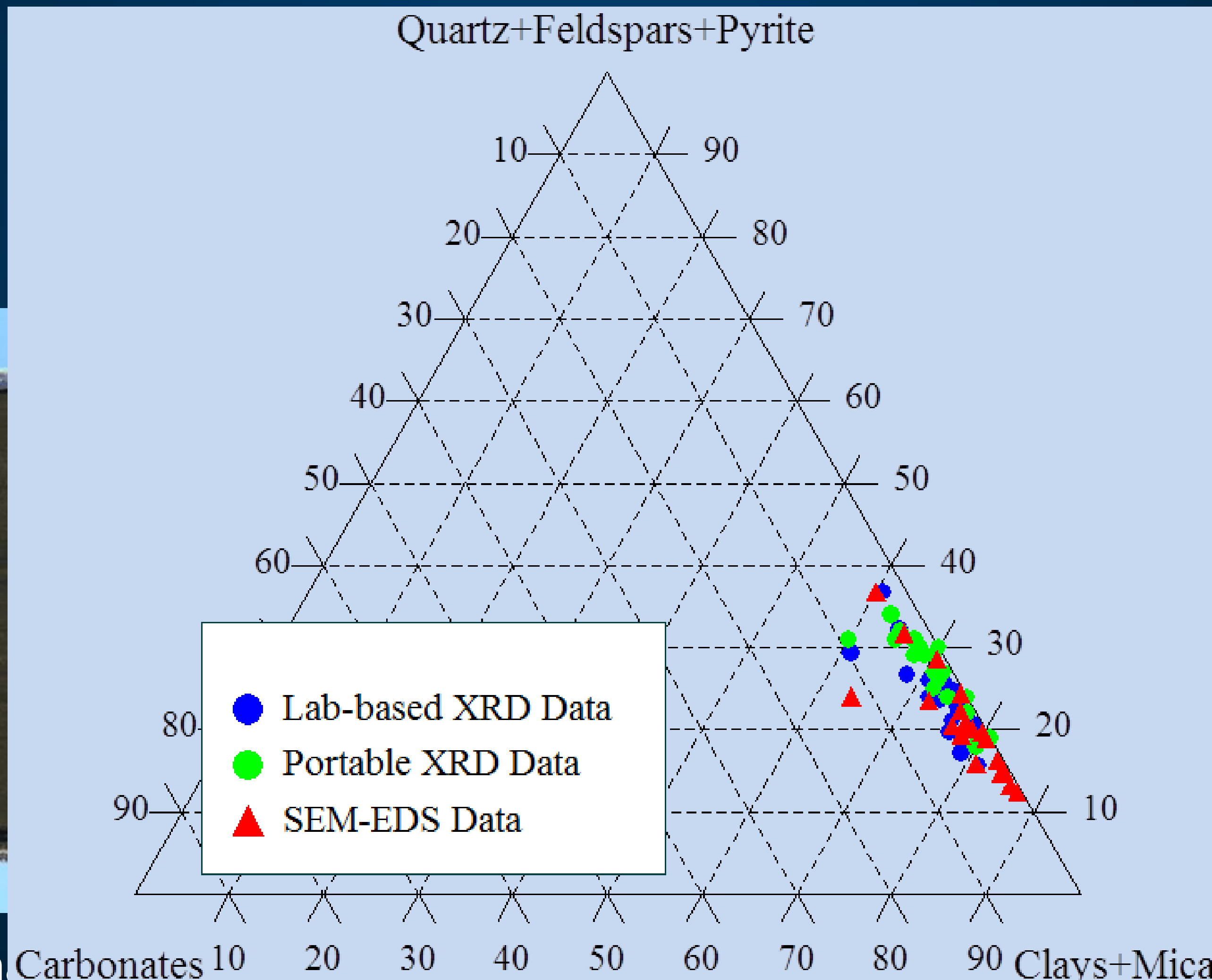
Tri-plot Ternary Diagram Software © 2003

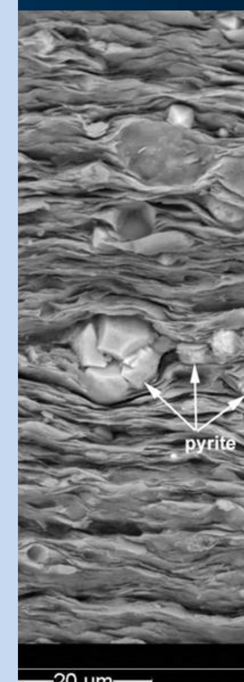
Coreplug data set 4: International Mudstone



Enhanced image contrast illustrates variability and concentrations of mineral phases

Coreplug data set 4: International Mudstone





Enhanced im… …al phases

Cuttings Analysis Challenges: What did we learn?

- Wellsite sampling & missing depths
- Insufficient material leading to composite samples
- Questionable sample splits
- Contaminants
  - Barite
  - lost circulation materials (sized calcium carbonate, carbonized graphite), oilfield cement, iron filings, plastic lubricant beads, walnut shells, quartz proppant
- Portable XRD manufacturer calibration factors did not meet accuracy needs. Reference mineral standards were used to fine-tune Relative Intensity Ratio (RIR) calibration factors

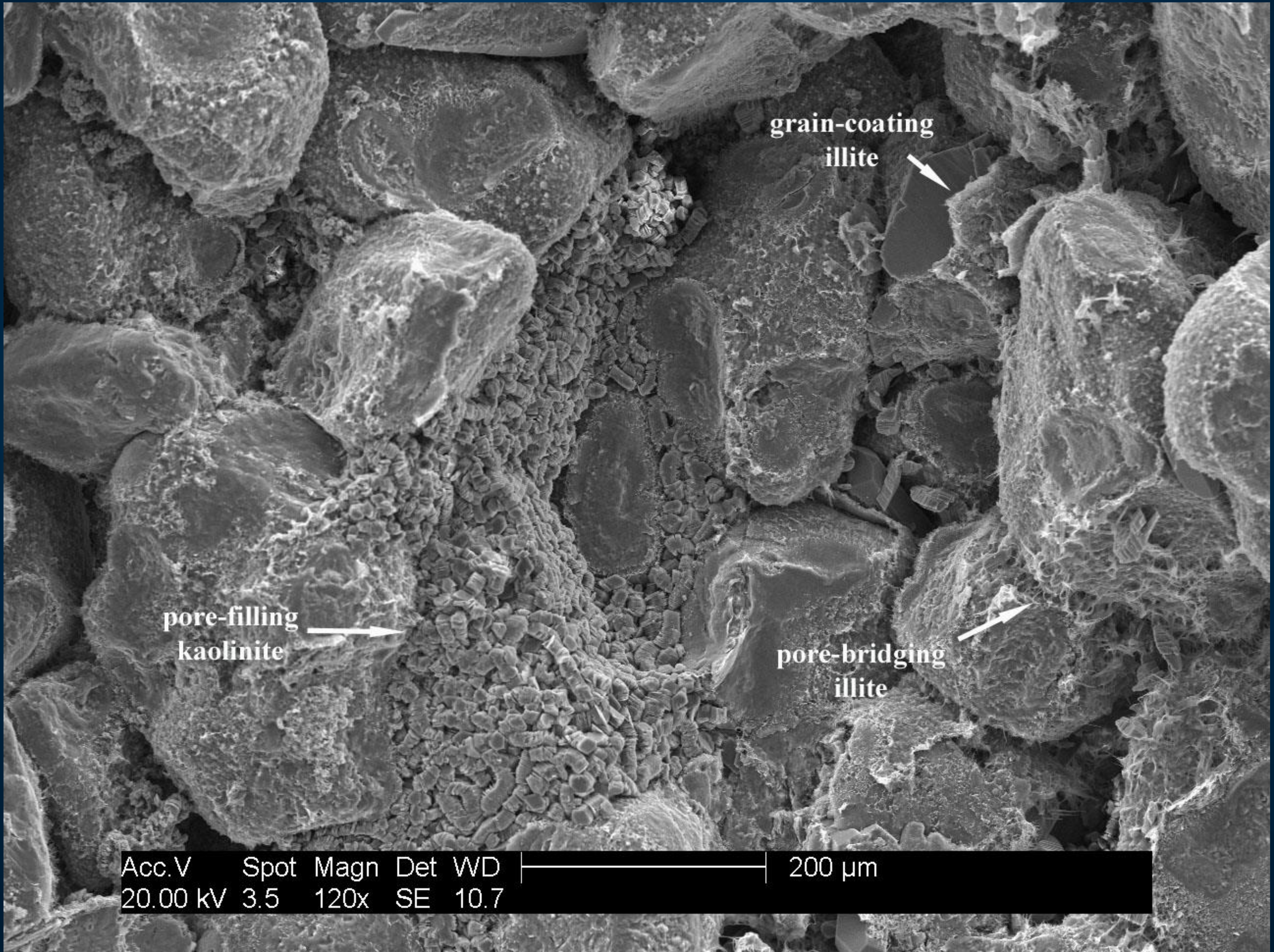
Coreplug Analysis Challenges: What did we learn?

- Best data matches in conventional sandstones
- SEM-EDS area of investigation on cores increased from 3x9mm (27mm^2) to 10x13mm (130mm^2). Minimizes impact of micro-bedding.
- SEM-EDS mineral phase nomenclatures streamlined to avoid confusion
- Mudstone pores are beyond the resolution of SEM-EDS analysis.
  - Porosity data was removed from SEM-EDS reports on mudstones
  - Aspect ratio values reflect drilling-induced breaks, rather than mudstone porosity

Conclusions

- Study yielded increased confidence in the quality of wellsite mineralogical analysis
  - improvements & standardization of sample preparation procedures
- Data Scatter Common: Over-riding problem is sampling
  - Knowledge of contaminants
  - Sample splitting
- Type of sample analysis performed should be fit-for-purpose
  - Rapid-turnaround datasets valuable for geosteering and establishing a “Brittleness Index”
  - Slightly longer turnarounds suitable for fracturing stage selections, identifying clay species and expandability potential

Questions???


grain-coating illite
pore-filling kaolinite
pore-bridging illite
Acc.V Spot Magn Det WD
20.00 kV 3.5 120x SE 10.7
200 µm