

# **A High-Definition Mineralogical Examination of Potential Gas Shales\***

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## **Abstract**

In 2008 shale gas production totaled over 5 Bcf/d and accounted for 8% of the total daily natural gas production in North America. While 70% of this was from the Mississippian Barnett Shale of Texas, production in other fields has started to increase. Numerous thermally mature, organic rich shales have been identified and targeted as potential analogues to the Barnett. The evaluation of these shales has proven challenging as many conventional techniques such as well logging, petrophysics and core analysis are inconclusive in the very fine-grained sediments that make up these reservoirs.

It has been recognized that mineralogy and texture can be a critical component in the resource potential of shales. Rocks with high silica (quartz) and low clay content typically have high Young's modulus and low Poisson's ratio making them more brittle and more prone to natural fractures and good candidates for fracture stimulation. Many shales exhibit covariance between silica and TOC (total organic carbon) suggesting a biogenic origin for the quartz. This biogenic quartz can also contain trace or rare earth elements typically found in organic materials. Aluminosilicates such as illite can have microporosity suitable for the adsorption of gas while other clay minerals can increase moisture content which reduces the adsorption capacity of shale.

In this study, we utilize QEMSCAN, a high definition, automated mineralogical analysis tool, to evaluate the mineralogy and micro textures of potential gas shales. QEMSCAN allows a detailed evaluation far beyond the resolution of conventional thin section petrography and at a speed much faster than conventional SEM analysis.

We present a brief summary of data from a diverse selection of North American shales over a broad geological time frame: Cambrian Conasauga; Ordovician Utica; Devonian Muskwa and Duvernay; Mississippian Barnett and Floyd; Triassic Montney; and Cretaceous Colorado. The emphasis has been placed on correlating QEMSCAN with other analytical data including XRD, geochemistry, TOC and Rock-Eval.

We illustrate how QEMSCAN can rapidly identify different depositional facies within a shale section by classifying drill cuttings on the basis of mineralogy and texture. This combined with the bulk mineralogy from the analysis can rapidly identify optimal completion intervals, zones for horizontal development and intervals with a high propensity for fracturing.

### **Reference**

Davies, D.K., R.K. Vessell, and J.B. Auman, 1997, Improved prediction of reservoir behavior through integration of quantitative geological and petrophysical data: SPE Annual Technical Conference and Exhibition, 5-8 October 1997, San Antonio, Texas, Web accessed 26 July 2010,

<http://www.onepetro.org/mslib/servlet/onepetroreview?id=00038914&soc=SPE>

# A High-Definition Mineralogical Examination of Potential Gas Shales

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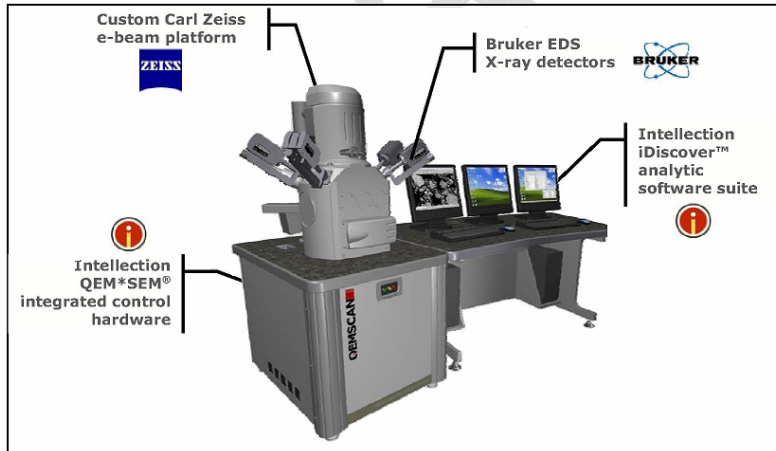
WHEN YOU NEED TO BE SURE



**Presenter's Notes:**

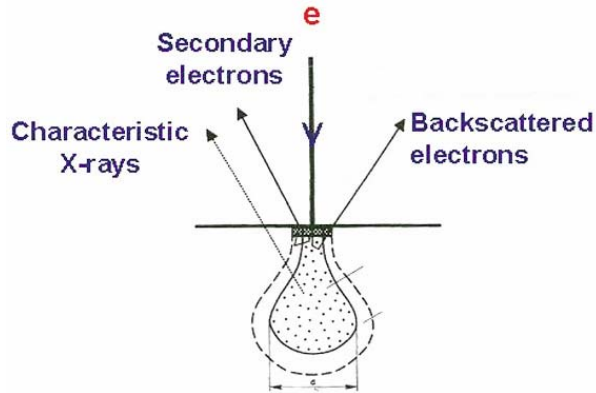
Good-morning. This morning I am going to present some of the mineralogical work we are doing on shale gas. Most of the work utilizes our QEMSCAN automated mineralogy instruments but I hope to show you by the end of the presentation that by integrating the QEMSCAN results with other analytical results and data we have achieved something new

- How to measure mineralogy
  - QEMSCAN
  - XRD
  - QEMSCAN vs. XRD
- Silica, Carbonates and Organics
- Lithotyping
  - Rock Typing using quantitative mineralogy
  - Shale gas lithotyping
- Mineralogical Heterogeneity
- Integration of SEM data



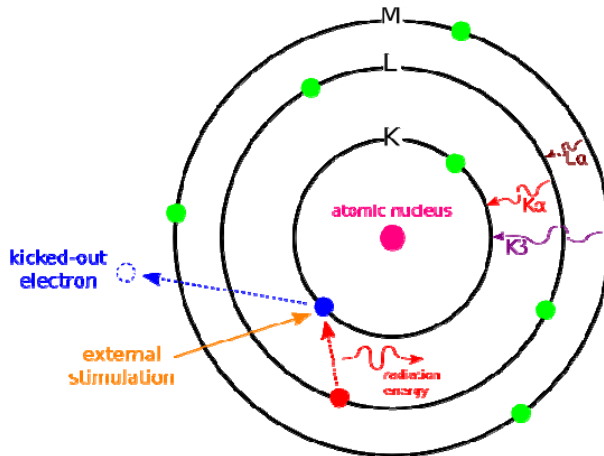
**Presenter's Notes:**

I will start off by giving a quick overview of the two primary instruments or mineralogical analysis the QEMSCAN and The QEMSCAN system consists of 4 components a Carl Zeiss now FEI SEM; four Bruker EDS detectors, a little more on those later; the hardware, and finally the analytic software package that allows these components to work together to provide a fully automated analysis.

**Presenter's Notes:**

I'll briefly touch on the theory of electron imaging by explaining this diagram. When the electron beam strikes the sample, secondary and backscattered electrons are excited near the surface. In addition characteristic x-rays are generated from a depth of approximately 2 $\mu$ m depending on density of the sample, this is effectively the limiting factor on overall resolution.

Secondary electron (SE) imaging is used to produce topographic images that most of you are probably familiar with, since we are using polished samples there is no information from the Secondary electrons. Instead we use backscattered electron (BSE) imaging which reveals relative density because BSE intensity is proportional to the atomic weight of the phase under the beam.

**Presenter's Notes:**

Characteristic x-rays are generated, when a beam of high energy electrons is focused on the sample, the incident beam may excite an electron in the inner shell of an atom causing it to be ejected. This creates a hole, an electron from the outer, higher energy, shell fills the hole and the difference in the energy level is emitted as an x-ray. These x-rays are characteristic of the energy difference between the shells and the atomic structure of the element from which they were emitted. Thus these x-rays can be measured by the four EDS detectors and the elemental composition of the specimen can be determined.

## How does QEMSCAN® work?

- The QEMSCAN System is a Scanning Electron Microscope (SEM) combined with 4 Energy Dispersive X-ray (EDX) Spectrometers.
- The SEM images the field of view using Back-Scattered Electrons (BSE) to determine particle boundaries.
- The EDX captures a spectrum at each “pixel” of the particles.
- The EDX spectrum are compared against a look-up table of known minerals (Species Identification Protocol or SIP) to generate a mineralogy map.
- Each analysis takes less than 1 ms allowing over 1 million pixels to be mapped per hour.

**Presenter's Notes:**

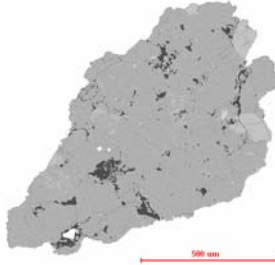
These interactions of the electron beam and the sample form the basis for the measurements of the QEMSCAN. A backscattered electron (BSE) image is first captured, the computer separates the particles from the resin on the basis of BSE intensity. The beam is then steered back to the particles and mapping begins. One “pixel” is mapped for each analysis point with the resolution specified by the operator. The process is very quick with each pixel being analyzed in less than 1ms allowing over 1 million pixels to be mapped an hour.



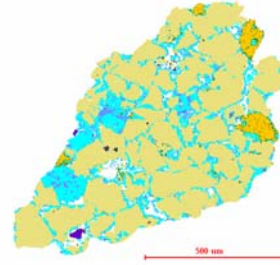
Optical Microscope Image



BSE Image



QEMSCAN Mineral Map

**Presenter's Notes:**

Here we have an example of a particle, in this case from a siltstone, on the left is an optical microscope image and on the right the corresponding false color Mineral Map of a cuttings particle acquired by the QEMSCAN

**Note**

- Individual quartz grains (yellow) in a calcite cement (the blue), and the white is the pores.
- Bright spots
  - Barite - drilling mud in a pore (barium because of its high atomic number, 56, shows up as a bright spot on the BSE)
  - 2 pyrite grains (red)
- the kaolinite weathering (in green) coating the (orange) potassium feldspar grains
- the calcite cement is pore filling

- Analysis is performed on a pulverised bulk sample.
- Uses the diffraction of x-rays at characteristic angles to identify the crystal structure.
- The peak intensities are used to calculate abundance.
- Results are typically presented as a table of abundances of the crystalline materials in the sample.
- Results need to be corrected for non-crystalline phases (i.e. kerogen, amorphous silica and porosity)



**Presenter's Notes:**

Most are probably more familiar with XRD which measures the diffraction of x-rays by a samples. The peaks diffraction peaks are measure and the different angles is a measure of the distance between discrete crystallographic diffracting planes within minerals and therefore characteristic of the mineral, the intensity of the peak is indicative of the quantity of the mineral. The diffraction peaks can be strongly influenced by the geometry of the sample so grain size, oriantaion, etc. can have a strong influence on the measurement, therefore the samples are ground to powder before the analysis.

**XRD**

- Identification based on crystal structure
- Analysis of powdered bulk sample
- Results include:
  - Quantitative mineral abundances
  - Detailed clay speciation
- Limitations
  - Cannot identify and quantify non- or poorly crystalline phases (chert, opal, organics)
  - Trace phases can be missed do to high detection limit (~2 %)

**QEMSCAN**

- Identification based on chemical composition (and BSE brightness)
- Analysis of drill cuttings, thin sections, RSW core, plugs, core
- Results include
  - Quantitative mineral abundances including trace minerals
  - Textural information
    - Grain Size
    - Shape
    - Depositional and diagenetic textures
- Limitations
  - Some fine grained and layered clays can be difficult to resolve due to the similar chemistries and maximum resolution of EDS (~2µm)

**Presenter's Notes:**

To summarize when we compare XRD and QEMSCAN we see that both provide quantitative mineralogy, and both can analyze the same samples, however XRD requires this sample be pulverised where as QEMSCAN can measure whole rock samples retaining textural and spatial data. While XRD struggles with non crystalline phases such as chert, opal and organics which are all typically present in shale gas systems, QEMSCAN cannot always distinguish fine grained layered clays where the chemistries are similar.

So we can see that both methods have particular strengths and weaknesses and depending on the information we are trying to obtain one method may not always provide the correct result. As we are often looking for multiple variables we often must use two or more techniques to ensure we have obtained accurate results. Besides XRD and QEMSCAN we could use TOC and porosity to correct XRD results, as well I will demonstrate later that SEM data can be integrated with QEMSCAN results to provide a more complete data set.

What we need for shale gas is:

QEMSCAN + XRD (+XRF, +LECO TOC.....)

**Presenter's Notes:**

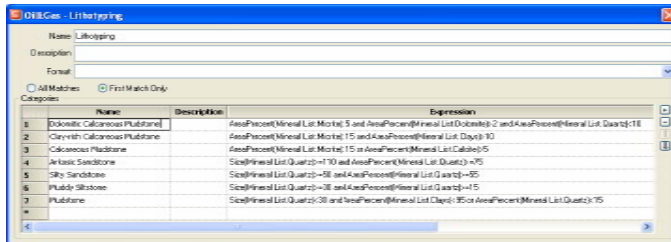
Clearly both systems have distinct advantages

- Silica
  - Detrital versus Pelagic silica
    - Pelagic commonly occurs as opal, which is not always correctly identified or quantified by XRD
  
- Carbonates
  - Texture cannot be measured by XRD
    - Carbonate texture is critical to understanding the effects on hydraulic fracturing
    - Carbonates can be detrimental (carbonate mud), behave like clastics (detrital dolomite) or indicate fracture (fractures healed with calcite)
  
- Organics
  - Neither method can accurately quantify fine organics

**Presenter's Notes:**

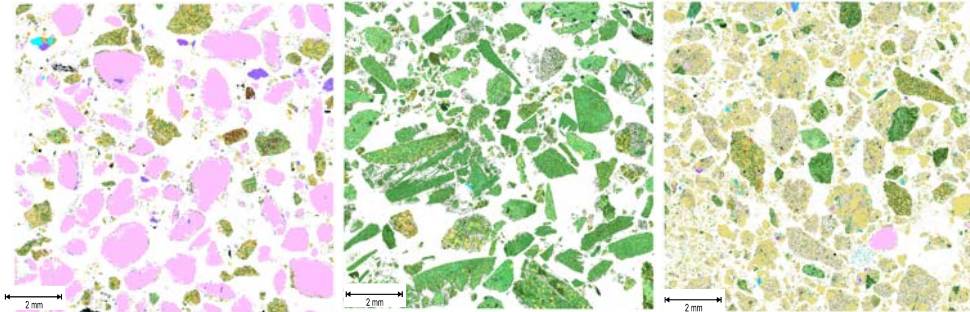
While it has been recognized early on that shales with a high silica content seem to exhibit the best potential for a commercial shale gas it is important to note that not all silica is equal. Biogenic silica is derived from diatoms and radiolarians, these can accumulate as siliceous oozes particularly over areas of coastal upwelling. Biogenic silica is comprised of amorphous silica typically in the form of opal-A. This amorphous silica can be converted to opal-CT and finally quartz with burial and progressive increases in temperature and pressure. This conversion releases water which like the smectite to illite conversion can drive the migration of hydrocarbon. Additionally several authors have pointed out that this biogenic silica provides more sites for the nucleation of syntaxial quartz cements. This is supported by the recent work on the Barnett that suggests there is no intragranular porosity and that the porosity is restricted to the kerogen. Pelagic quartz does however have the advantage of being associated with the organics and original porosity.

- Different rock types identified on basis of mineralogy and/or textures
- Classification of individual cuttings
- Easy creation of lithotype “rules”
- Classification fully generic



**Presenter's Notes:**

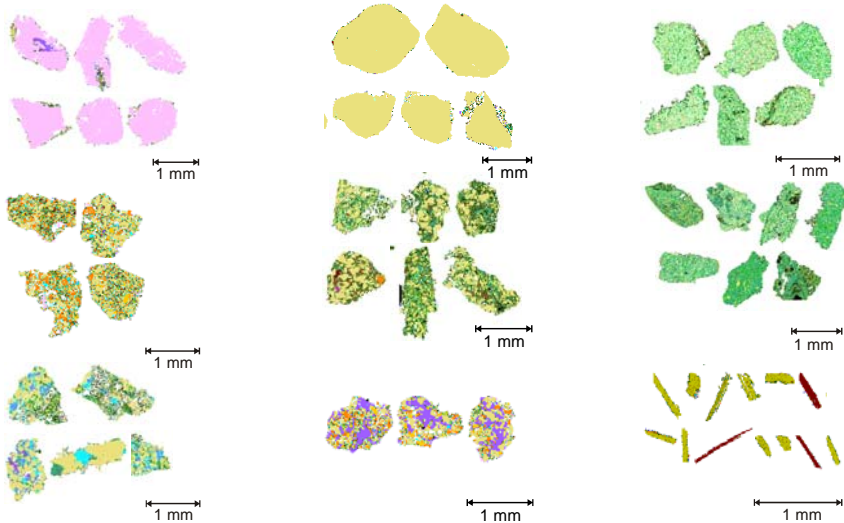
One of the key advantages of capturing the textural and spatial data from the QEMSCAN is that we can then use this data to classify cuttings by rock type or lithotype. We can use a series of rules based on mineralogy, grain size, grain shape and many other properties to classify cuttings. While many have previously thought of shale as homogeneous more recently shales facies have been described for the Barnett and other gas shale. Using these lithotyping rules we can pick facies from cuttings.



Cuttings samples: Individual lithotypes can be identified, categorised and used to characterise lithology and texture through reservoir intervals.

**Presenter's Notes:**

To take a closer look here we the false colour images of several cutting samples, the blocks are approximately 2 cm square. The individual cuttings particles can be separated and then classified on the basis of mineralogy, texture and spatial data.

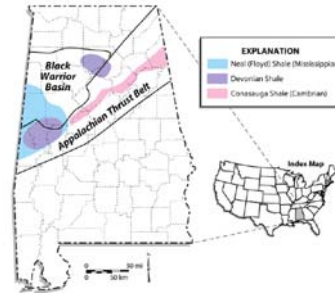


**Presenter's Notes:**

Here we can see the cuttings from the previous slide grouped and classified by lithotype. These lithotypes can then be plotted with depth quickly identify the facies

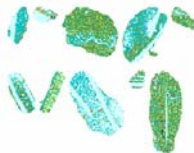


- Client identified 4 facies within the shale, with one being ideal for hydraulic fracturing.
- Shale interval was over 5000' thick, and tectonic antiformal stacking made correlation difficult.
- Coring was not an economic option.
- Well logs were inconclusive
- QEMSCAN cuttings analysis

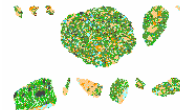




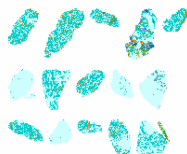
Calcite Filled Fractures



Siliceous Facies



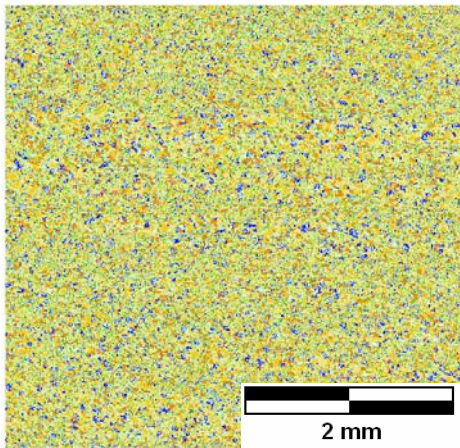
Micritic Limestone



Mushwad



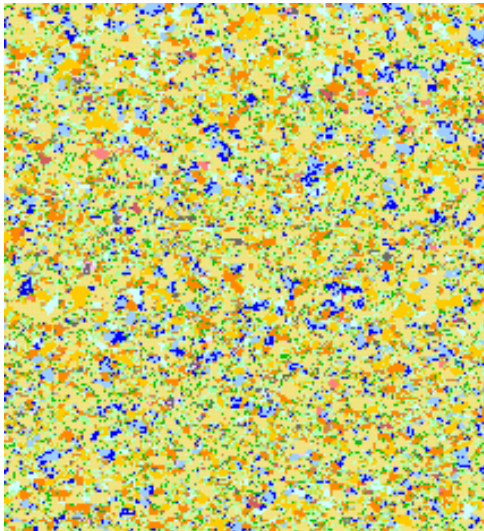




- Mineral Name**
- Background
  - Quartz
  - K Feldspar
  - Plagioclase
  - Muscovite
  - Smectite
  - Kaolinite
  - Chlorite
  - Illite
  - Fe-Illite
  - Illite-smectite
  - Clays+Carbonates
  - Calcite
  - Dolomite
  - Ferroan Dolomite
  - Siderite
  - Fe Oxides
  - Chromite
  - Tourmaline
  - Pyrite
  - Barite
  - Gypsum & Anhydrite
  - Rutile & Ti Silicates
  - Apatite
  - Zircon
  - Organics
  - Undifferentiated

**Presenter's Notes:**

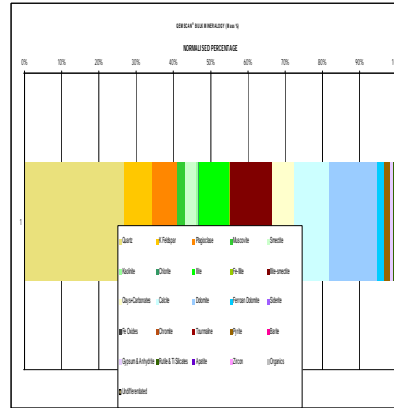
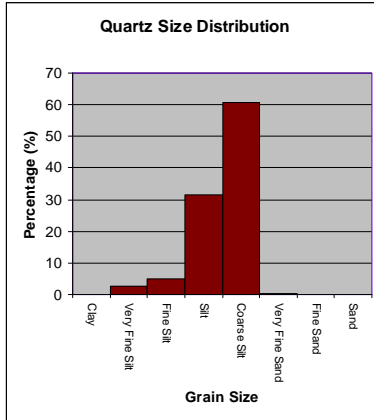
While there is no direct evidence of this the mineralogy data supports this origin. Here we see a section from the upper Montney

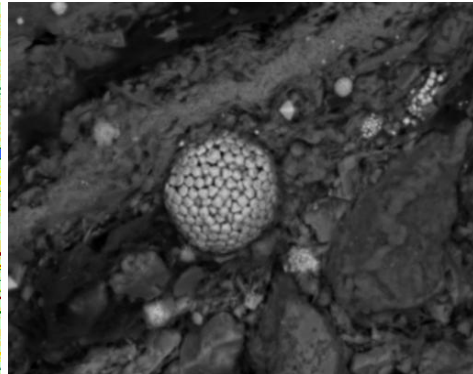
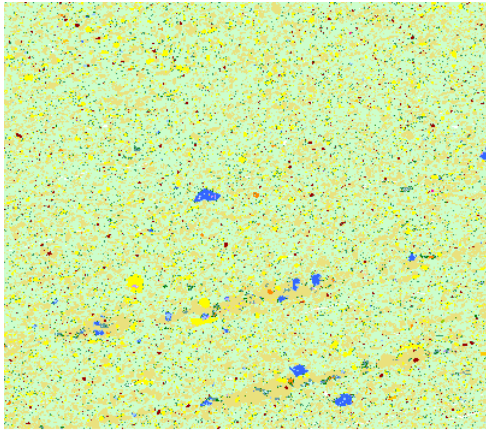


- Mineral Name**
- Background
  - Quartz
  - K Feldspar
  - Plagioclase
  - Muscovite
  - Smectite
  - Kaolinite
  - Chlorite
  - Illite
  - Fe-Illite
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  - Ferroan Dolomite
  - Siderite
  - Fe Oxides
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  - Pyrite
  - Barite
  - Gypsum & Anhydrite
  - Rutile & Ti Silicates
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  - Zircon
  - Organics
  - Undifferentiated

**Presenter's Notes:**

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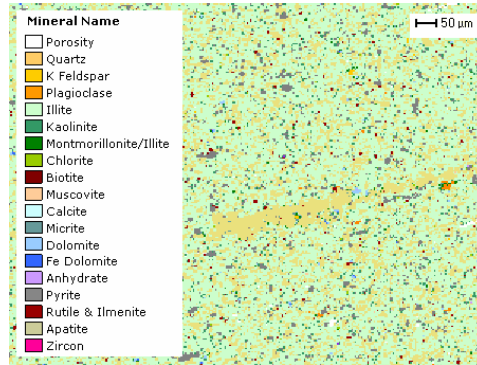
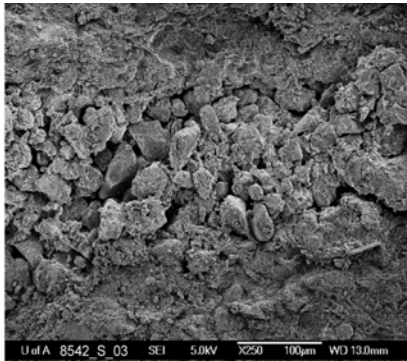




SEM HV: 15.00 kV WD: 18.9390 mm  
View field: 75.40  $\mu\text{m}$  Det: BSE Detector 20  $\mu\text{m}$  VEGAII TESCAN  
Date(m/d/y): 03/24/10 Jon Digital Microscopy Imaging

**Presenter's Notes:**

Often many features in shale gas are beyond the resolution of the QEMSCAN image. In this case we see framboidal pyrite, which contains intragranular porosity, the entire cluster of grains measure just over 10 microns and the pores are much less than a micron. Clearly cannot image this with the QEMSCAN but we can image the pyrite with an SEM, the BSE image can then be imported in to an image analysis package where the intragranular porosity can be calculated. However quantify the porosity contained in an entire section would be to time consuming. Therefore we have used the SEM to find an average porosity value that we can then assign to the pyrite in a QEMSCAN analysis.



**Presenter's Notes:**

Similarly thin silt laminations within a shale can be critical to producibility. In this example we can clearly see the quartz rich layer contains a significant amount of porosity where the QEMSCAN image show a single quartz layer with no clearly defined porosity. However by



# Summary

WHEN YOU NEED TO BE SURE



- Best to use more than one method
  - By utilizing complimentary methods (i.e. QEMSCAN and XRD) it is possible to overcome the limitations of both
- Know the sample and what is important to the results
  - Silica (detrital versus pelagic)
  - Textures (grain size, shape, etc)
  - Shale Facies descriptions
  - Sub-micron details (i.e. intragranular porosity in framboidal pyrite)
- Remember to compensate results
  - Adjust XRD for non-crystalline phases, organics and porosity
  - Calibrate QEMSCAN clays with XRD