# Organic Matter Characterization in Shales: A Systematic Empirical Protocol

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## ABSTRACT

Increased economic interest in shale oil and gas has prompted new exploration into shale reservoirs, subsequently prompting fresh attempts at understanding how storage and delivery works in tight gas sands and shales. Focusing on the organic matter (OM) present in the shales we have developed a process enabling us to study changes in OM after heating and extraction using different solvents. The changes occurring henceforth, being documented by using a Field Emission Scanning Electron Microscope (FE- SEM) at various imaging voltages as deemed necessary.

The work flow is as follows (for Horizontal plugs):

- Obtain mirror end trims
- Subject end trims to: micro-CT scan, thin section preparation, argon ion milling (AIM)
- Crush remainder of material as appropriate for TOC, XRD, SRP, etc.;
- Two large pieces from each crushed sample (A& B) subjected to AIM
- Sample A & B:
- Native state imaging of entire milled area
- Hi resolution imaging of OM and pore types; EDS as appropriate, mark locations on mosaic for repeated post-test imaging
- Sample A
- Extract (appropriate solvents) and reimage
- Retort@ 350 C, image/extract, image
- Retort@ 450 C, image/extract ,image
- Retort@ 650 C, image/extract, image
- Sample B Heating and re-imaging (no extraction)

Our initial (3) samples are from the Eagle Ford Formation with maturities ranging from 0.5-1.2% RO. These would be classified as low, medium and high maturity samples. A fourth sample from an unknown formation, is of intermediate maturity and is clay rich . We will be studying this sample as well to document the differences between clastic and carbonate rich mudrocks.

Our observations illustrate the initial "softening" of sedimentary organic material (SOM) as it is heated. Subsequent extraction of the low-temperature retort samples reveals "cracks" developed in the "softened" regions of SOM. As heating progresses to higher and higher

temperatures, sedimentary organic material is progressively removed from the sample, through generation of organic solvent soluble, liquid range hydrocarbons. The end result are crack-like pores lined with an insoluble organic residue. Similar structures are observed in "naturally matured" subsurface samples. Early formed bitumen in the samples is identified on the basis of structural position (e.g. partially filling large, intra-particle pores). When early formed products are sulfur rich they do not exhibit solubility in organic solvents, nor do they develop typical nano-pores upon further heating.

This study represents an initial foray into the characterization of OM in shales and based on these results we will carry out further experiments. This includes but is not limited to, retort under confining stress and axial load conditions.