

# Fluid Inclusions in Mudrocks

Don Hall  
Scientific Advisor  
Schlumberger  
March, 2018

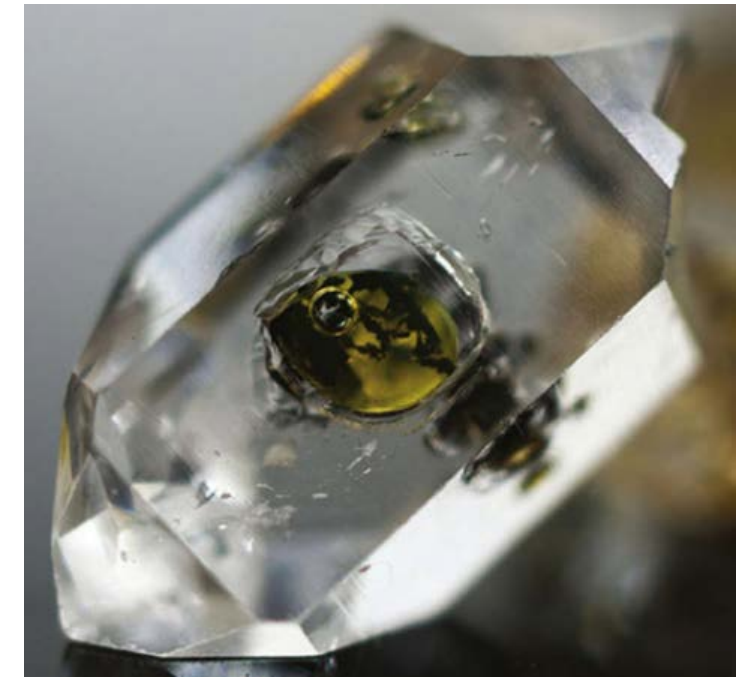
# Fluids “Trapped” in Fine-Grained Rocks: Size, Origin, Content

- Visible (petrographically) – can be non-destructively analyzed
- Invisible (seen on SEM) – destructively analyzed
- Locally generated – mature source rocks
- Migrated – source or non-source of variable maturity; ultimate origin?
- Organic – gas, oil
- Inorganic – aqueous with variable salinity
  - “gases”: CO<sub>2</sub>, H<sub>2</sub>S, N<sub>2</sub>, He, dissolved or free-phase

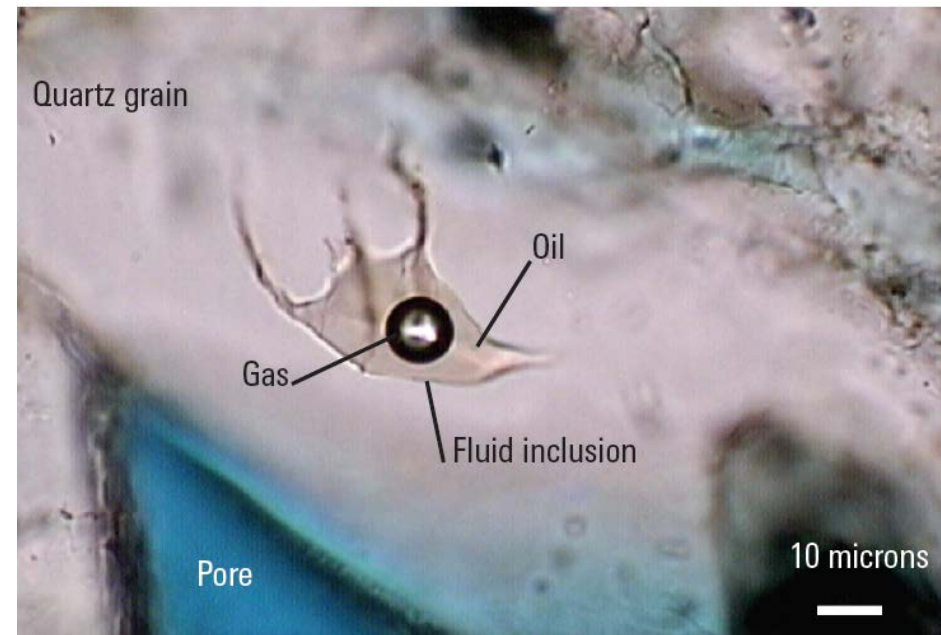
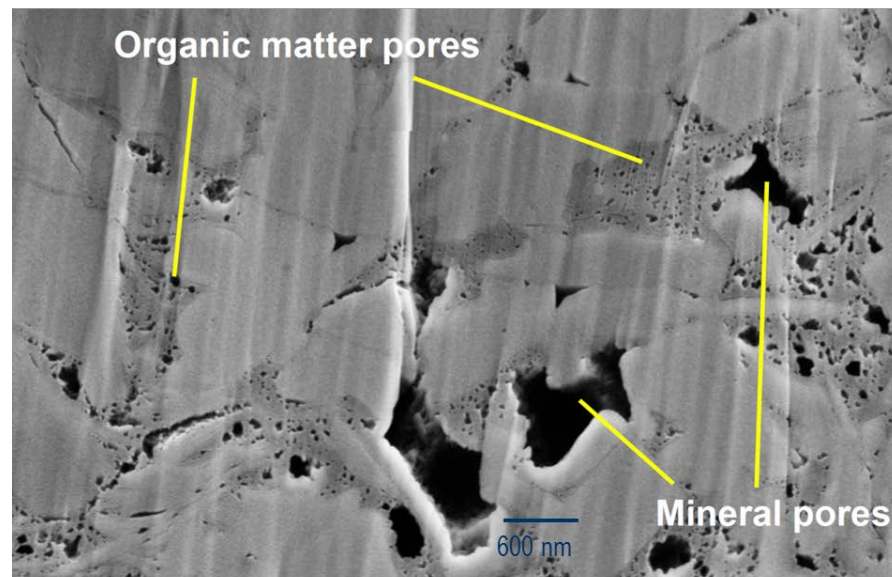
*These fluids provide useful information for both conventional and unconventional petroleum exploration. Combined destructive extraction and classical microscopy can be used to better understand past and present fluid distribution, composition, and associated diagenetic processes.*

# Fluid Inclusion Size and Scale of Observation

- A size continuum exists from things we can see with the naked eye, to things that can be studied with the white light microscope, to sub-micron-sized entities whose contents can only be indirectly inferred



From: Geofluids: Developments in Microthermometry, Spectroscopy, Thermodynamics (2015)  
By Vratislav Huraí, Monika Huraiová, Marek Slobodník, and Rainer Thomas

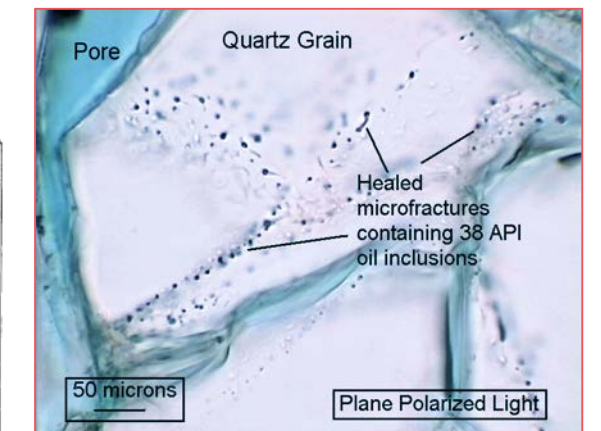
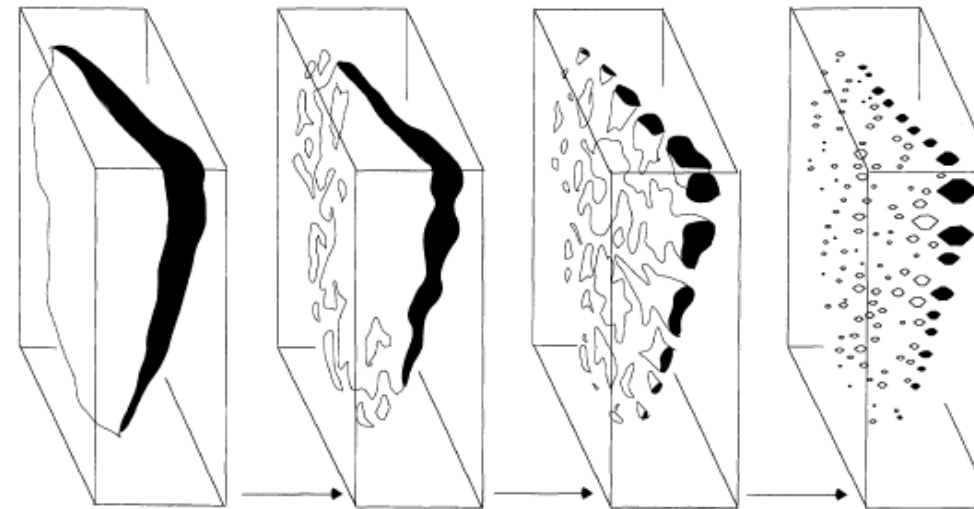
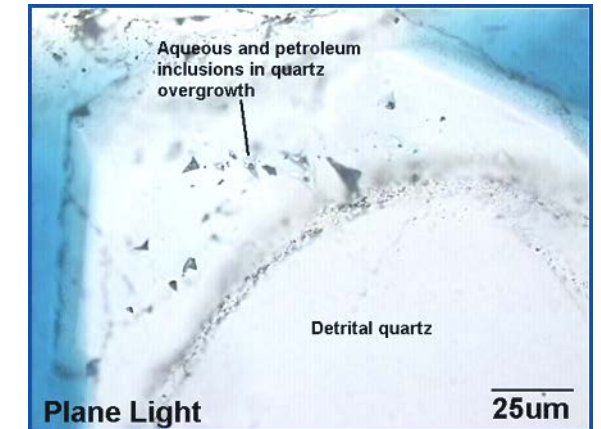
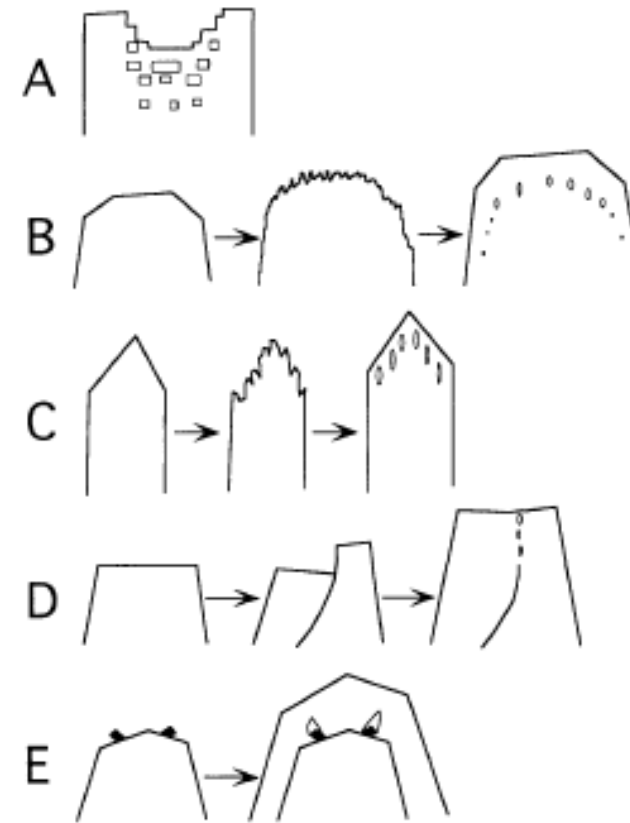


Synthetic oil inclusion in quartz: Dick Larese and the author

Nanopores in Barnett Shale: Ruppel and Louckes, 2006

# Fluid Inclusion Formation and Origin

- Micron-scale, fluid-filled isolated cavities in or between organic or inorganic material in rocks
- Form during subsurface diagenetic processes in which mineral cement is added to intergranular pore space, when microfractures are healed or during kerogen maturation
- Are representative of past or near-present-day pore fluids
- Track movement of aqueous and petroleum fluids
- Formation mechanisms are similar for fine-grained rocks, with the additional possibility of local generation in organic rich mudstones



# Fluid Inclusion Content

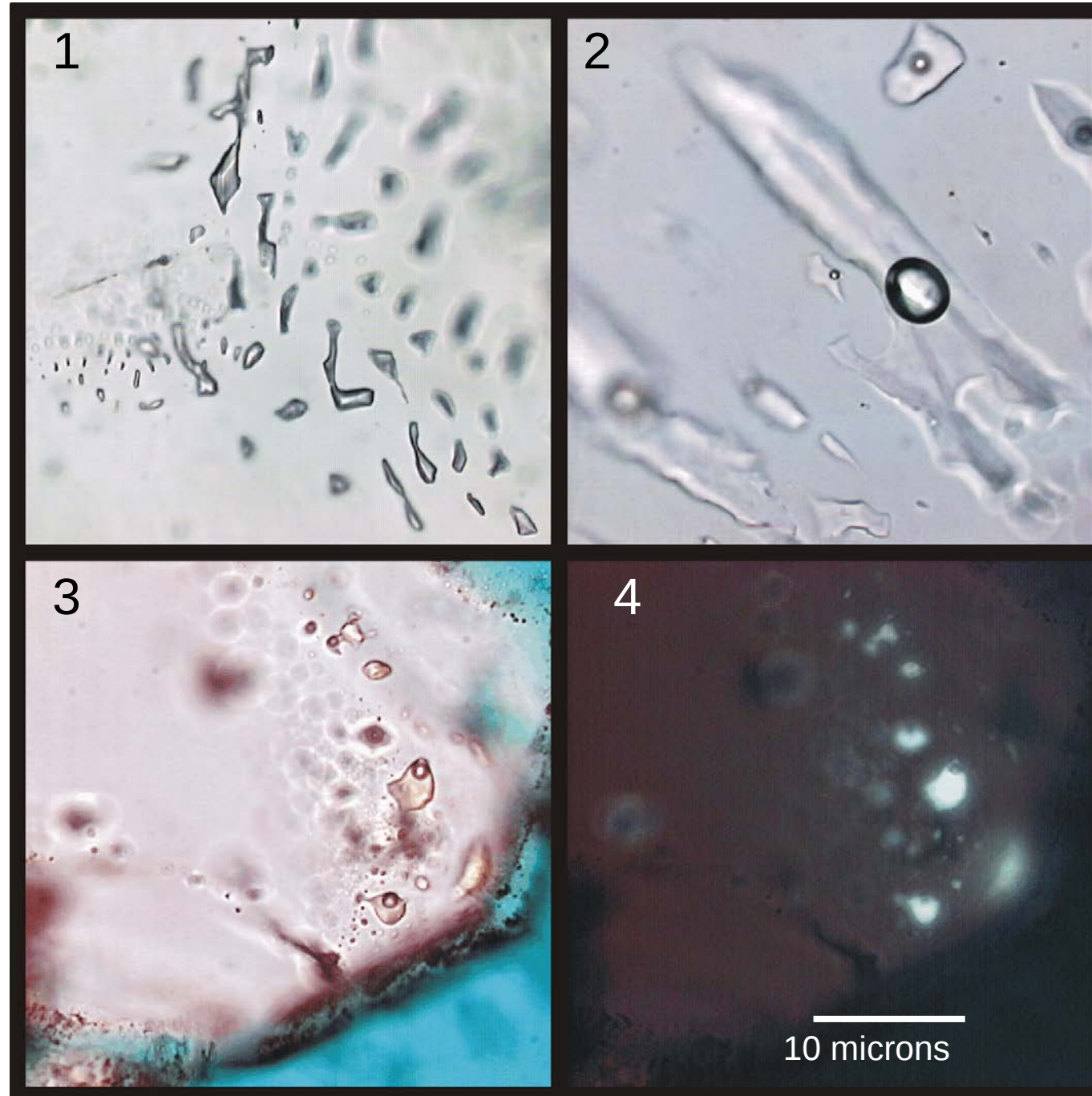
1: Gas

2: Aqueous Fluid

3: Oil (Plane Light)

4: Oil (UV Light)

*All photos are from polished thick sections of sandstone. Inclusions are present along healed microfractures.*



# Where Fluid Inclusions Form in Mudrocks

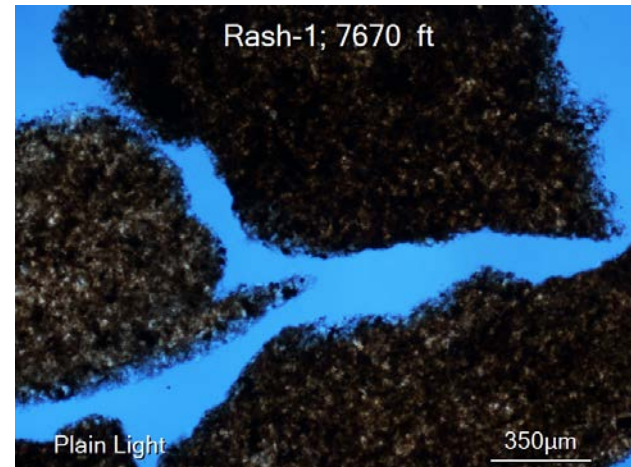
- Diagenetic cements
- Fractures in detrital material (quartz and feldspar grains, fossils)
- Fracture-filling cements
- Secondary nanoporosity created by kerogen maturation

*Remember that fluid inclusions exist as a size continuum, and many are much smaller than even the average grain size of mudrocks.*

# Petroleum Fluid Inclusions in Mudrocks

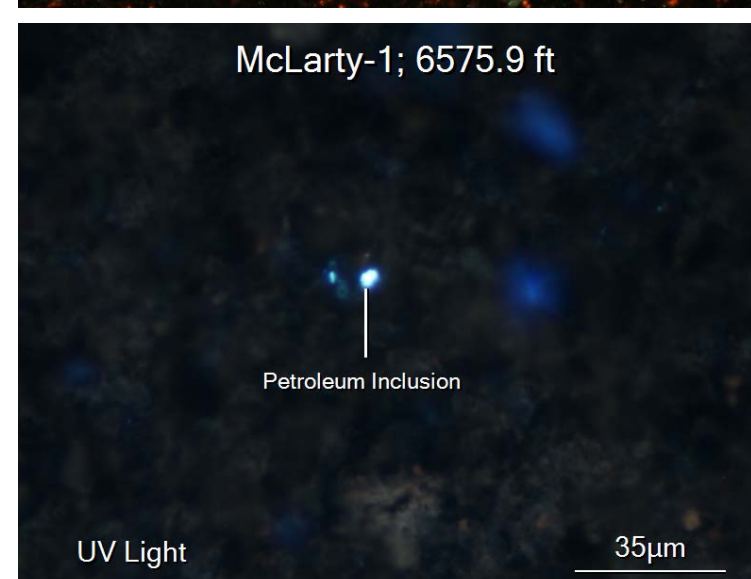
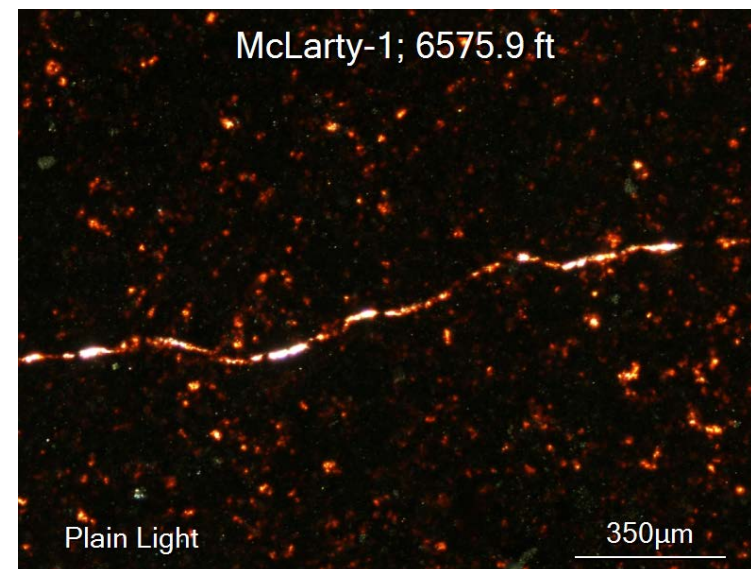
*Bone Springs Fm; Delaware Basin, USA*

*Immature Silty Shale; Dominated by Gas-Prone Kerogen*

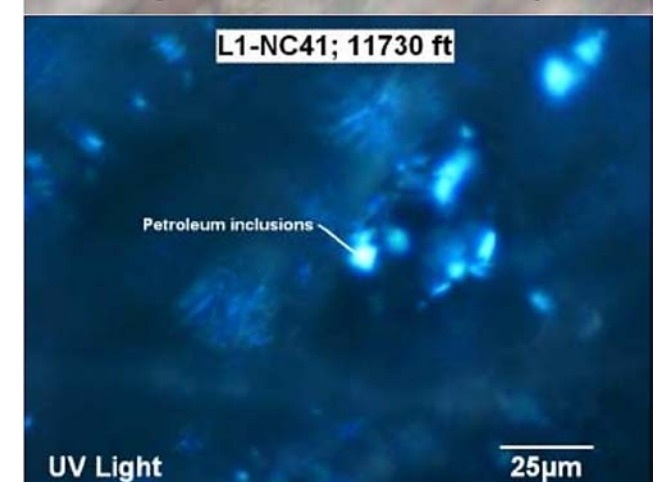
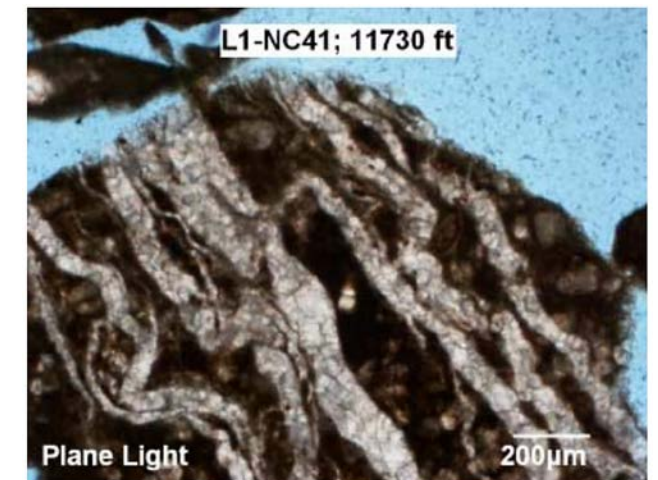


*Goldwyer Fm; Onshore Canning Basin, Australia*

*Organic-Rich Oil-Mature Shale*

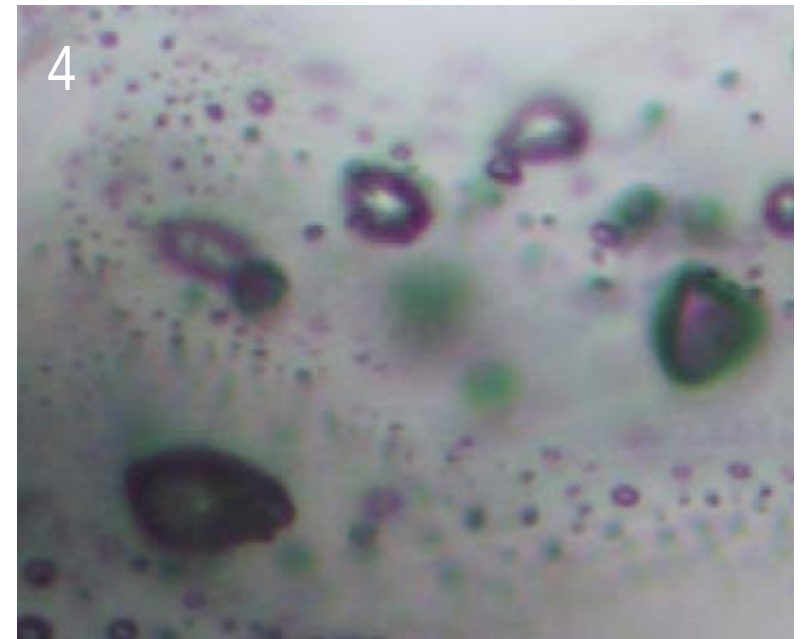
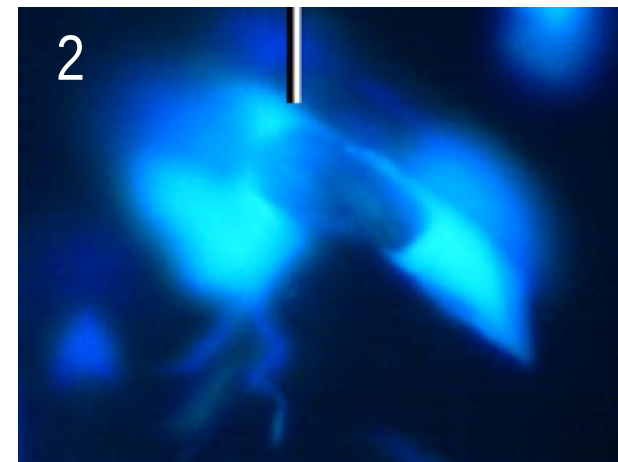
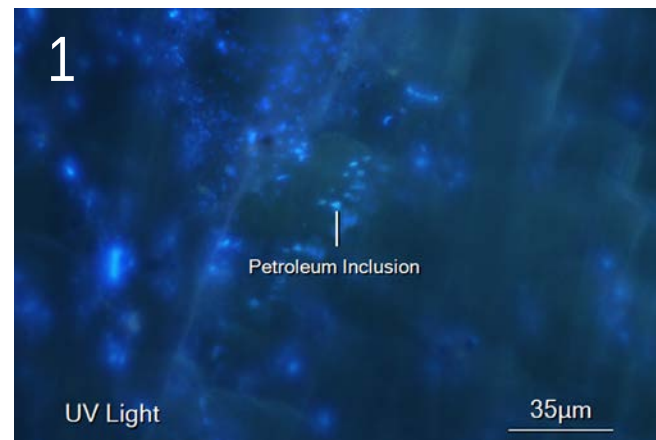
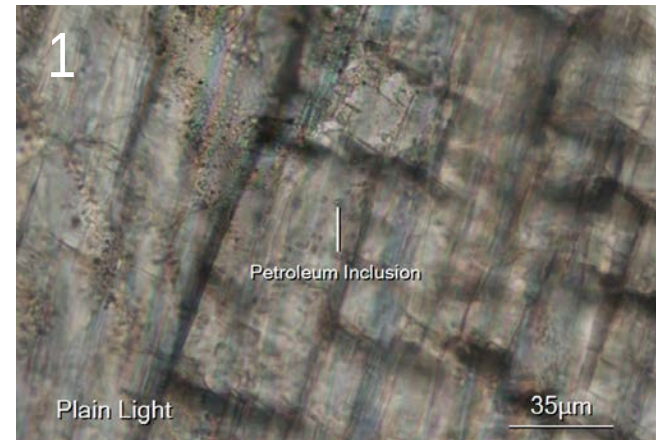


*Paleocene; Offshore Libya*  
*Organic-Lean, Deformed Mudstone*



# Fracture Fill in Organic-Rich Shale

- Bedding parallel and/or high angle veins or mineralized joints are ubiquitous in most mature, organic-rich shales
- In part attributed to anomalous pore pressures generated during kerogen maturation
- They potentially contain the expulsion history of the source rock system in the vicinity

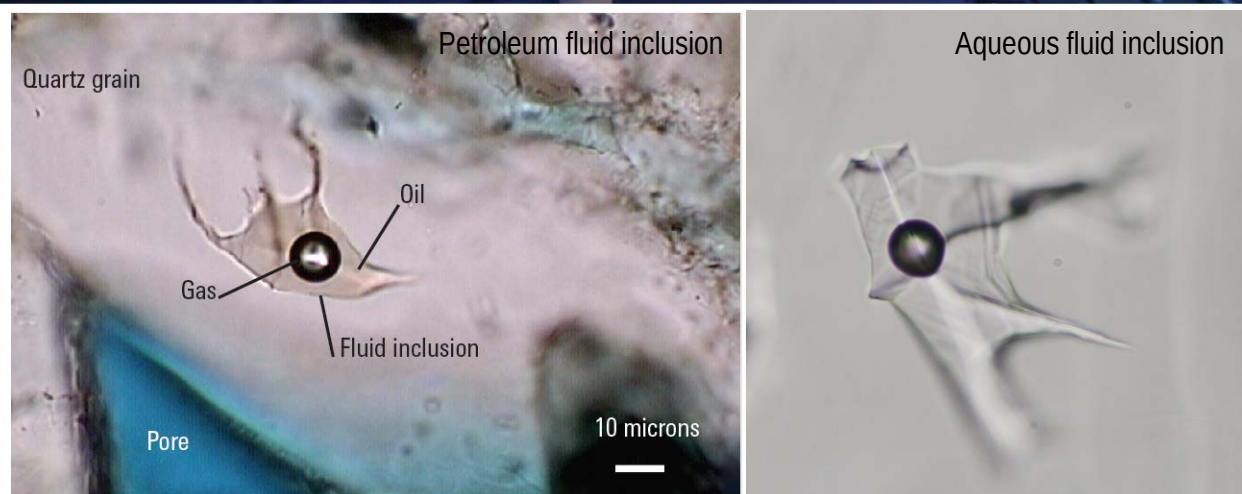


- 1) Monophase condensate inclusions; late condensate window (Vaca Muerta Fm, Argentina)
- 2) Rich gas-condensate / light oil inclusions; late oil / early condensate window (Bakken Fm)
- 3) Pyrobitumen stained gas inclusion; wet gas window (Appalachian Basin)
- 4) Dry gas inclusions; dry gas window (Marcellus Fm)

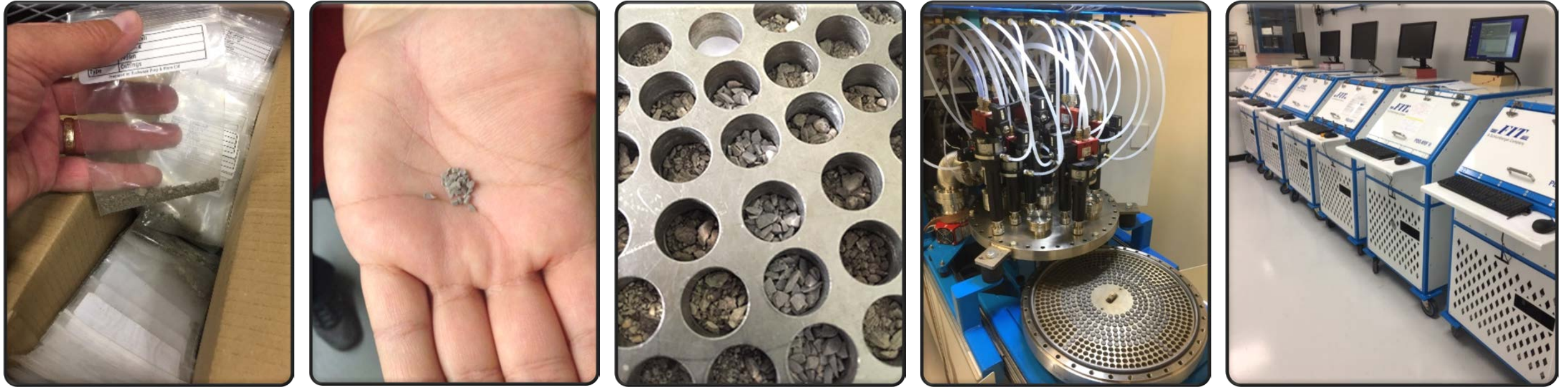
# Fluid Inclusion Petrography & Microthermometry (Non-Destructive)



- Distribution, abundance and attributes of encapsulated fluids
- Temperature, salinity, API gravity
- Evidence for petroleum migration and paleo-accumulations
- Basin model calibration
- Saturation state
- Water saturation calculations



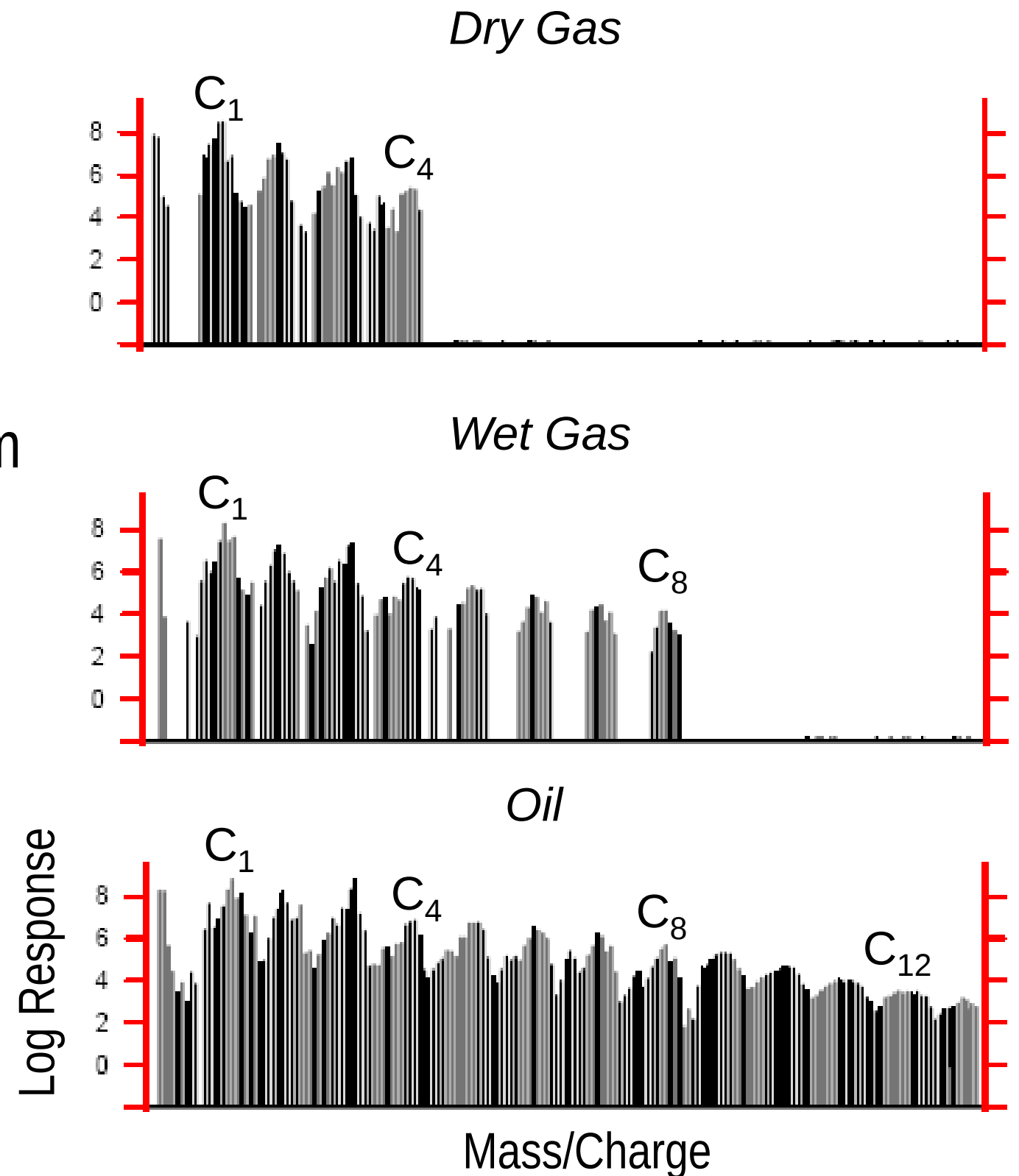
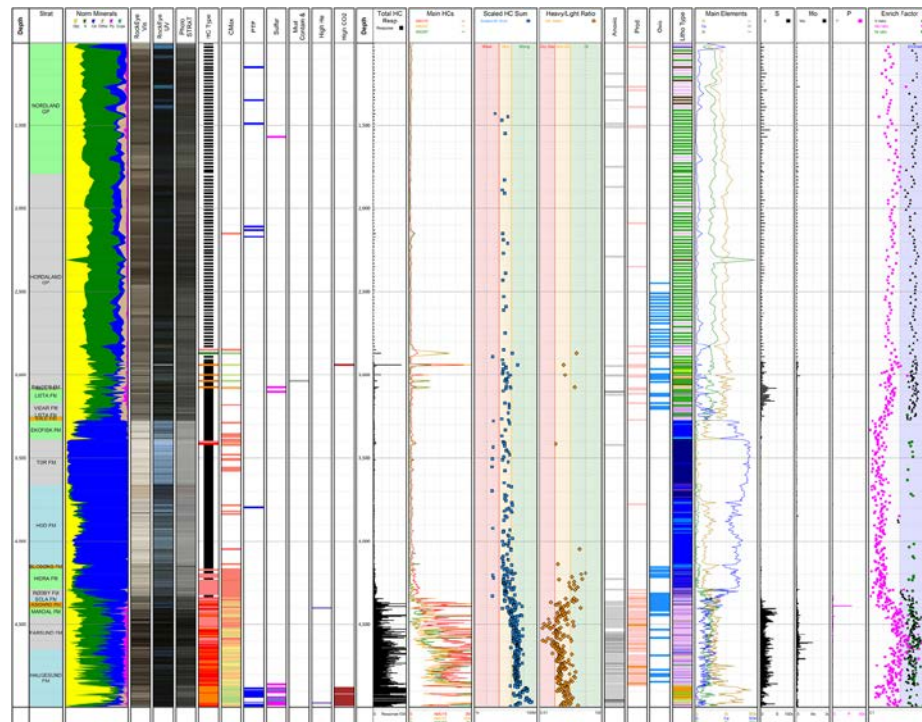
# Destructive Trapped Volatile Measurement (FIS)



- Automated instrumentation developed for rapid, high throughput of cuttings or core
- Requires only a small amount of sample; the same sample is used in each analysis
- No special preservation; no shelf life; generally applicable to all muds and drilling conditions
- Automated interpretation based on a multimillion-sample global database

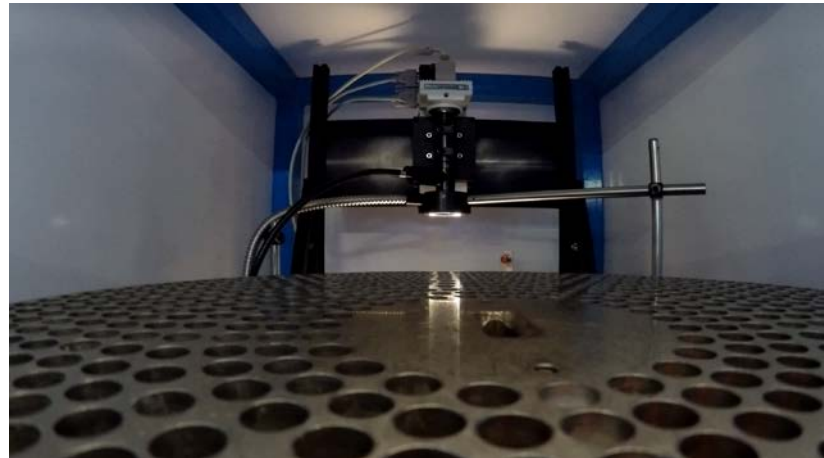
# Trapped Fluid Composition

- Bulk sample crushed in a vacuum system
- Trapped fluid analyzed by direct quadrupole mass spectrometry
- Locally generated (+/- migrated) petroleum type and composition distinguished
- Mass spectra and depth plots

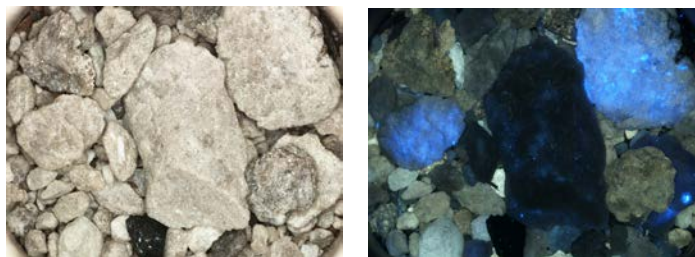


# Extended Automated Cuttings Screening Process

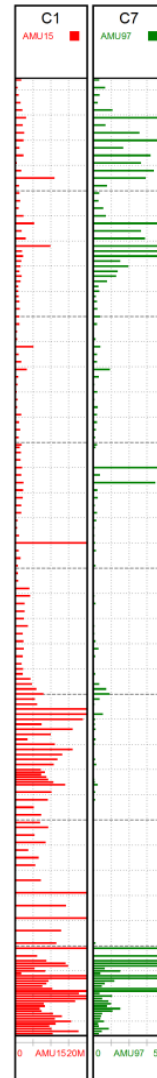
## High-Resolution Photography



- Image in white light and UV
- Images are focus-stacked
- Grain scale details of porosity, texture, rock types, etc.
- Mineral fluorescence correlated with cement or rock types
- Kerogen fluorescence related to maturity

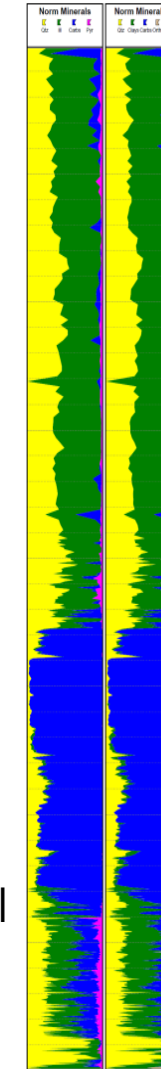


## Fluid Inclusion Stratigraphy (FIS)



- Analysis of trapped organic and inorganic volatiles
- Identifies where petroleum is or was
- Regional picture of petroleum history and prospectivity
- Identify remaining exploration potential in mature areas
- Data for completions

## ED X-Ray Fluorescence



- Lithology and normative mineralogy
- Chemical stratigraphy for correlation
- Depositional environment, facies, provenance
- Anoxia and paleo-surface productivity
- TOC proxies

# Mudrock Questions in Conventional/Unconventional Plays:

## *Petroleum System Questions*

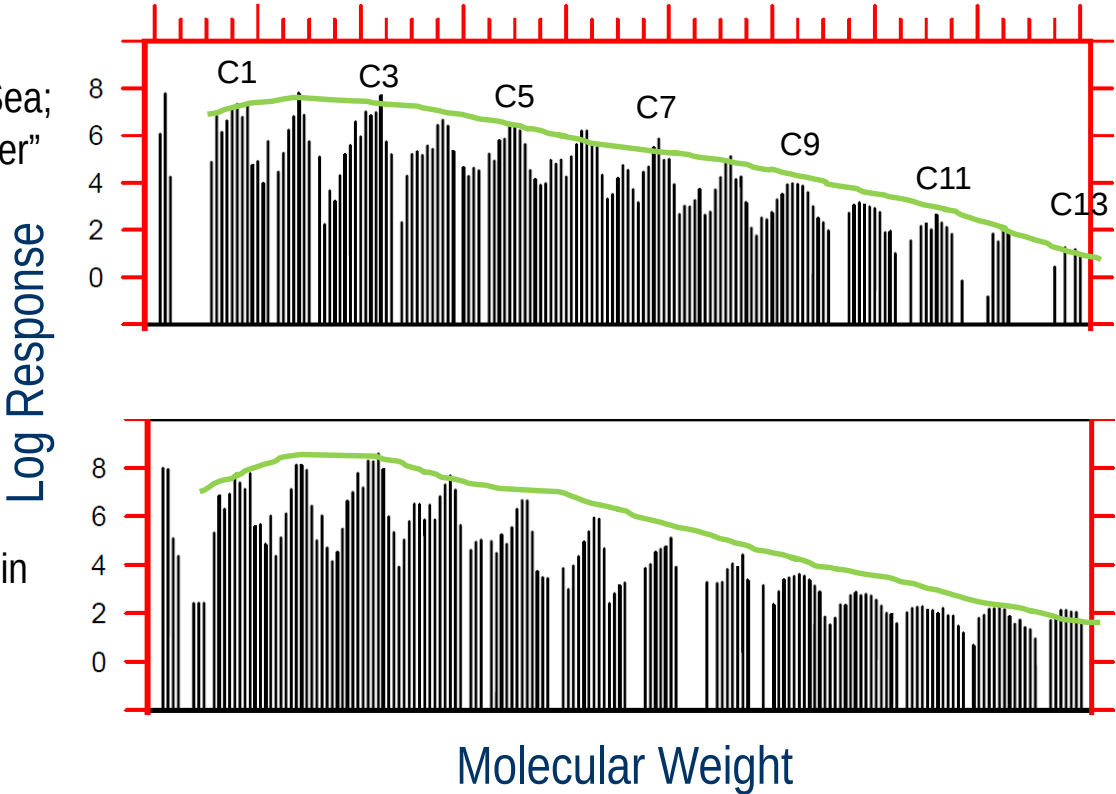
- Effective source rock
- Seal effectiveness
- Fluid composition and quality
- Local vs. migrated petroleum
- Timing

## *Reservoir Characterization Questions*

- Compartmentalization, connectivity
- Fluid distribution and volumes
- Water saturation and composition
- Diagenetic temperatures and maturity
- Vertical and horizontal targets

# Identifying Mature Source Rock FIS Responses

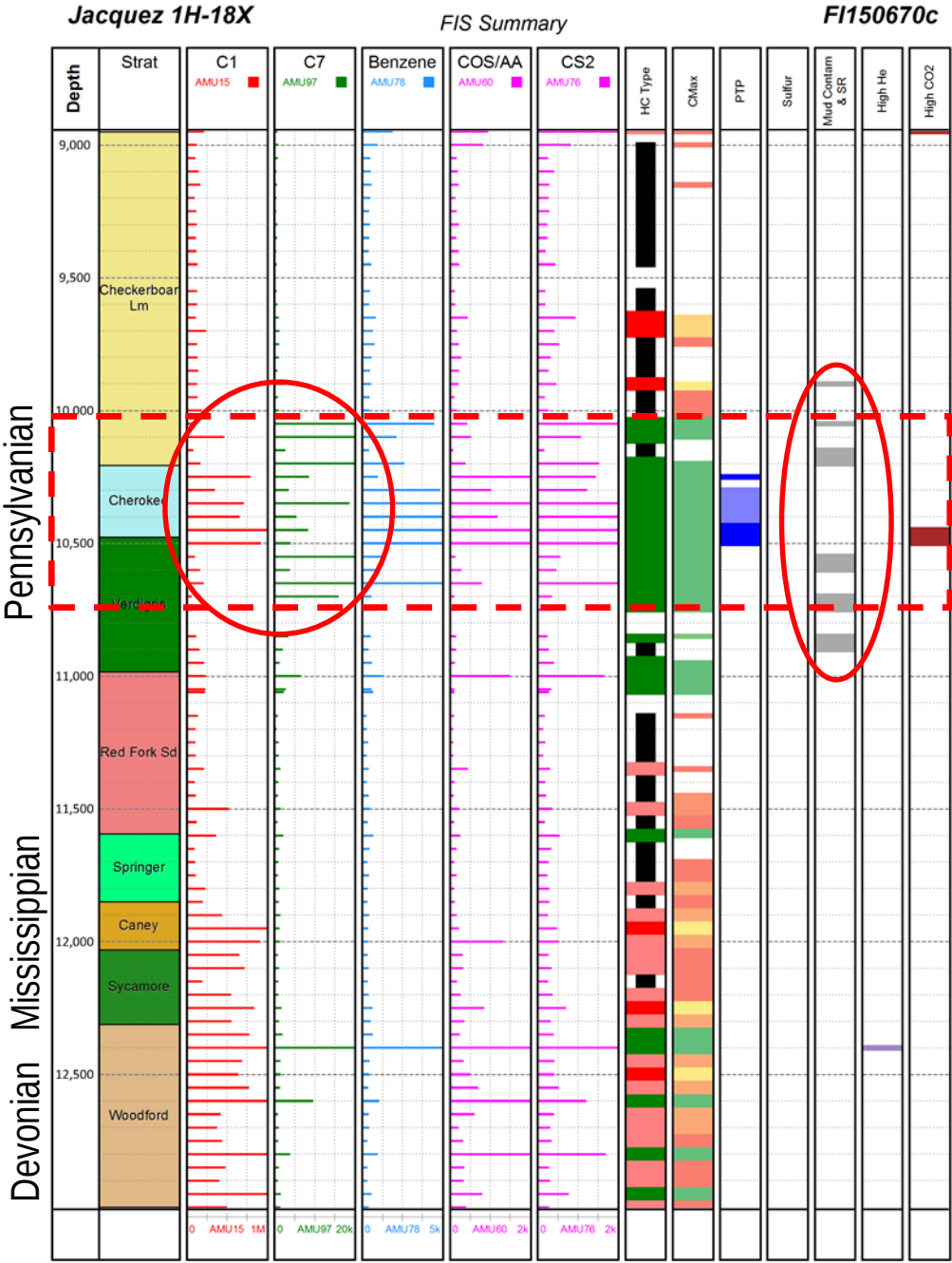
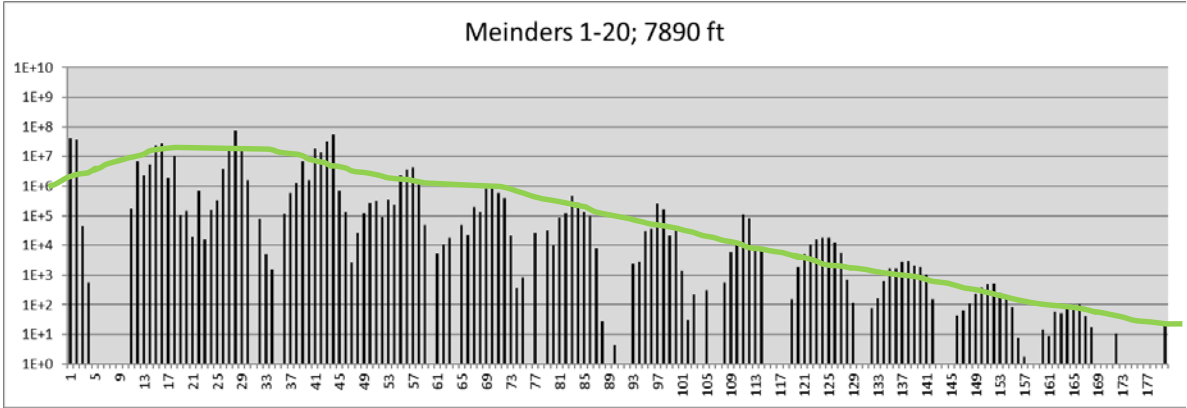
Type II marine shale from North Sea; Oil window. Note light end “rollover” in C1 and C2.



Type III Coal from Asia; Oil window. Note light end “rollover” in C1 and C2.

Decoupled C1 and C7 profiles in FIS logs can be produced either by a dual-stage charge history involving oil and gas; or, as in this case, local generation of liquids and a superimposed migrated gas event.

Type II/III Shale  
Caney Fm SCOOP

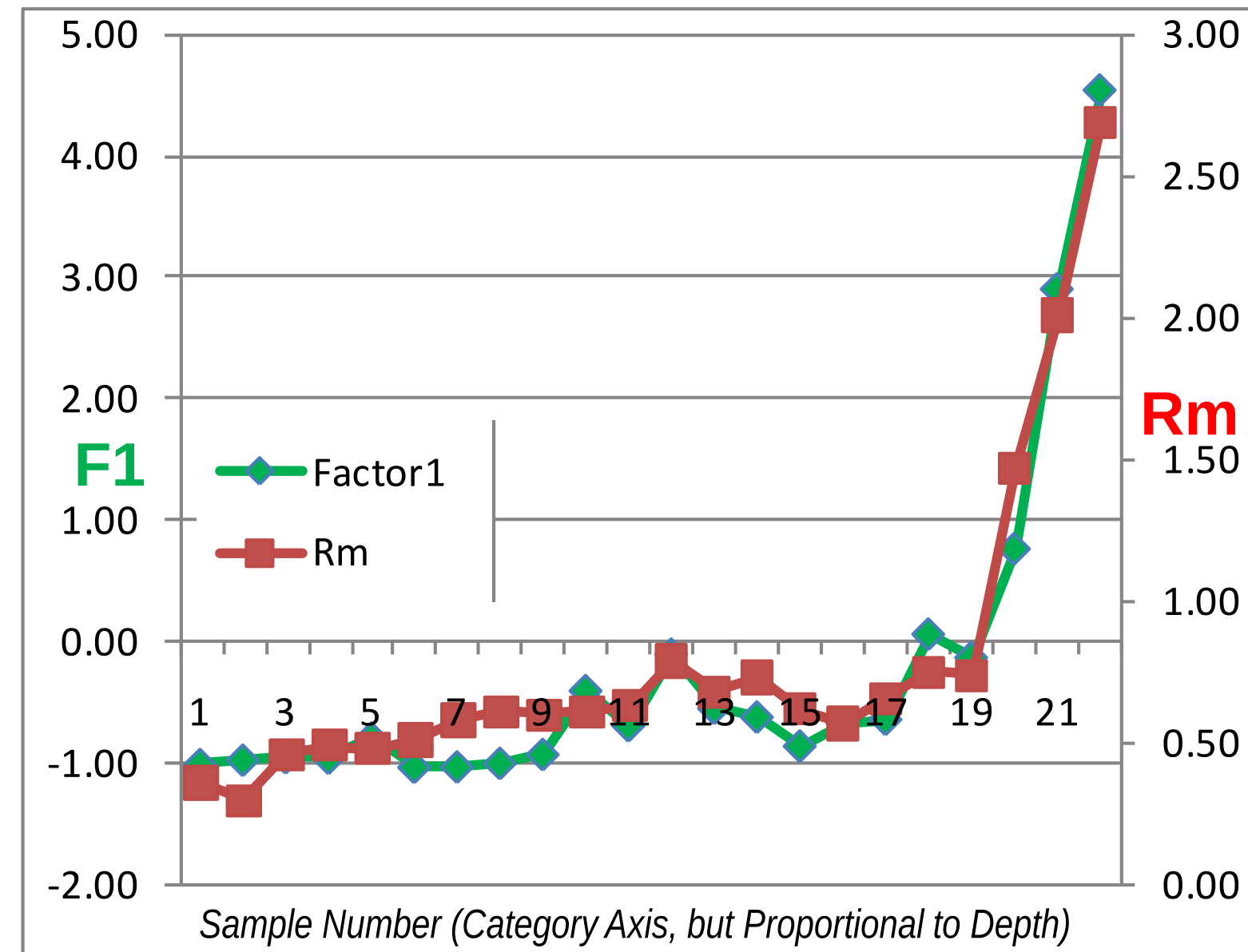


FIS Profile; SCOOP well

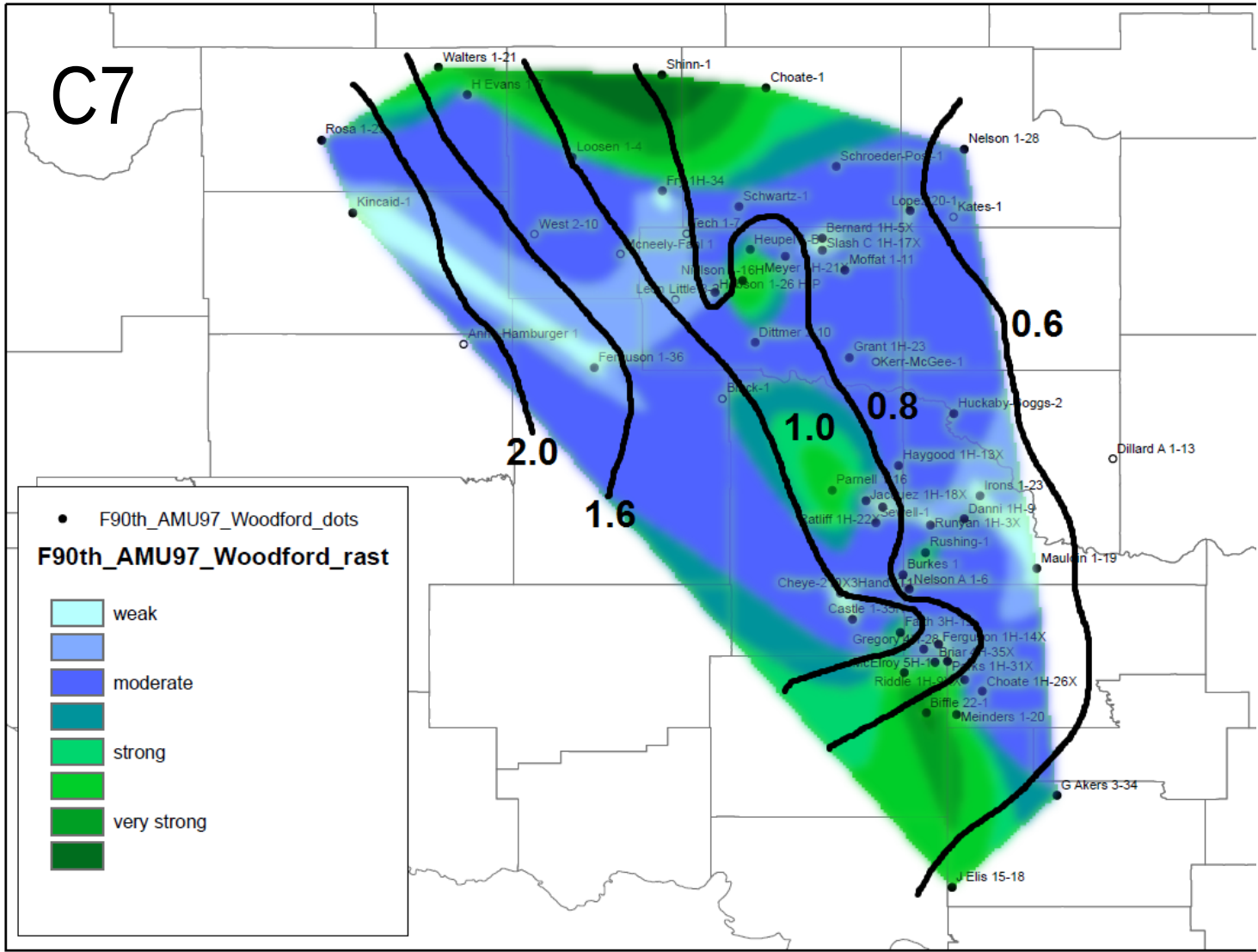
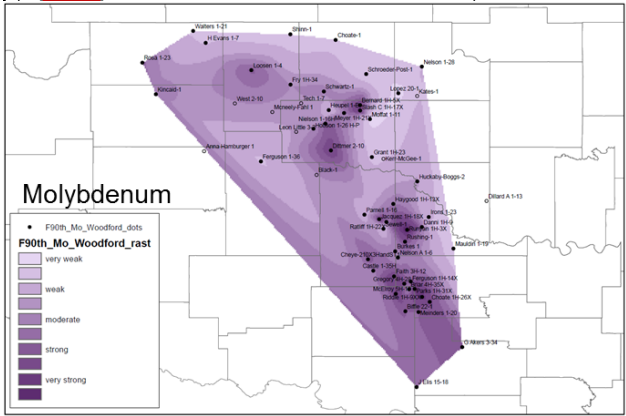
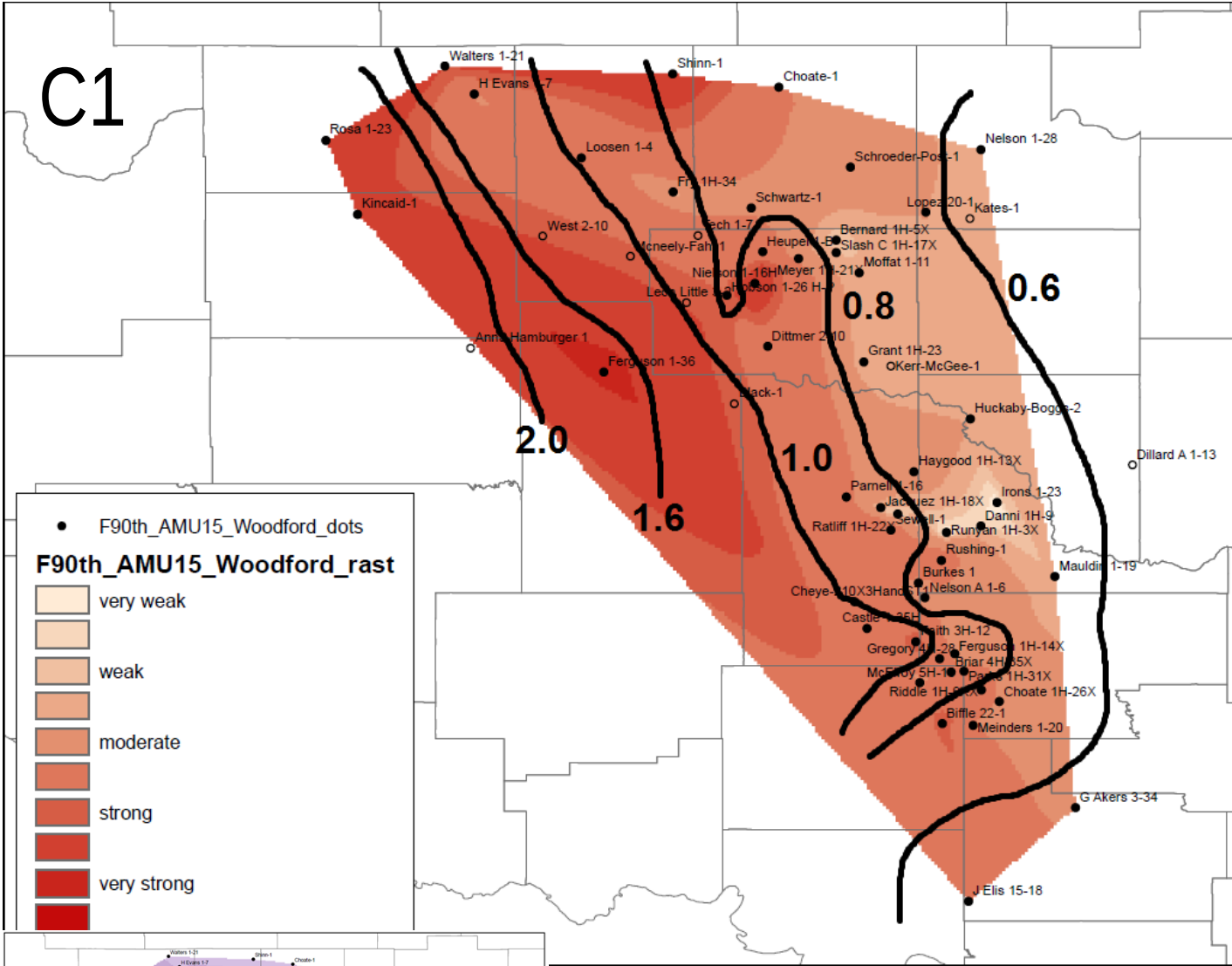
# Chemometric Analysis of FIS Response in Source Rocks for Maturity

- Factor 1 is highly correlated with independently measured thermal maturity data over a large maturity range
- Statistical methods applied to FIS data can be used to map maturity trends and define resource boundaries

*Relationship between PCA Factor 1 (F1) and measured Vitrinite Reflectance (Rm)*

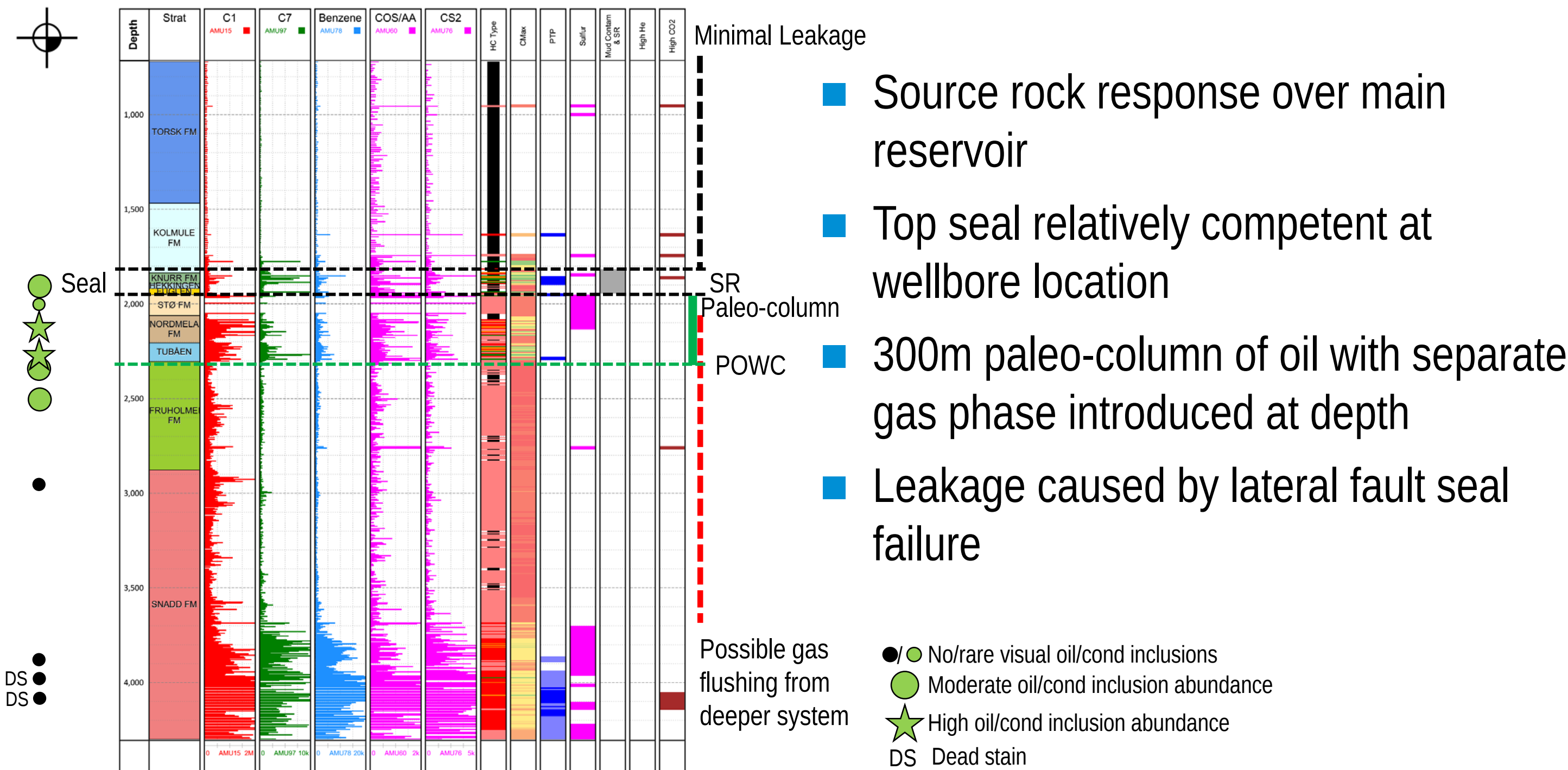


# Regional Oil and Gas Mapping, STACK & SCOOP Plays, OK

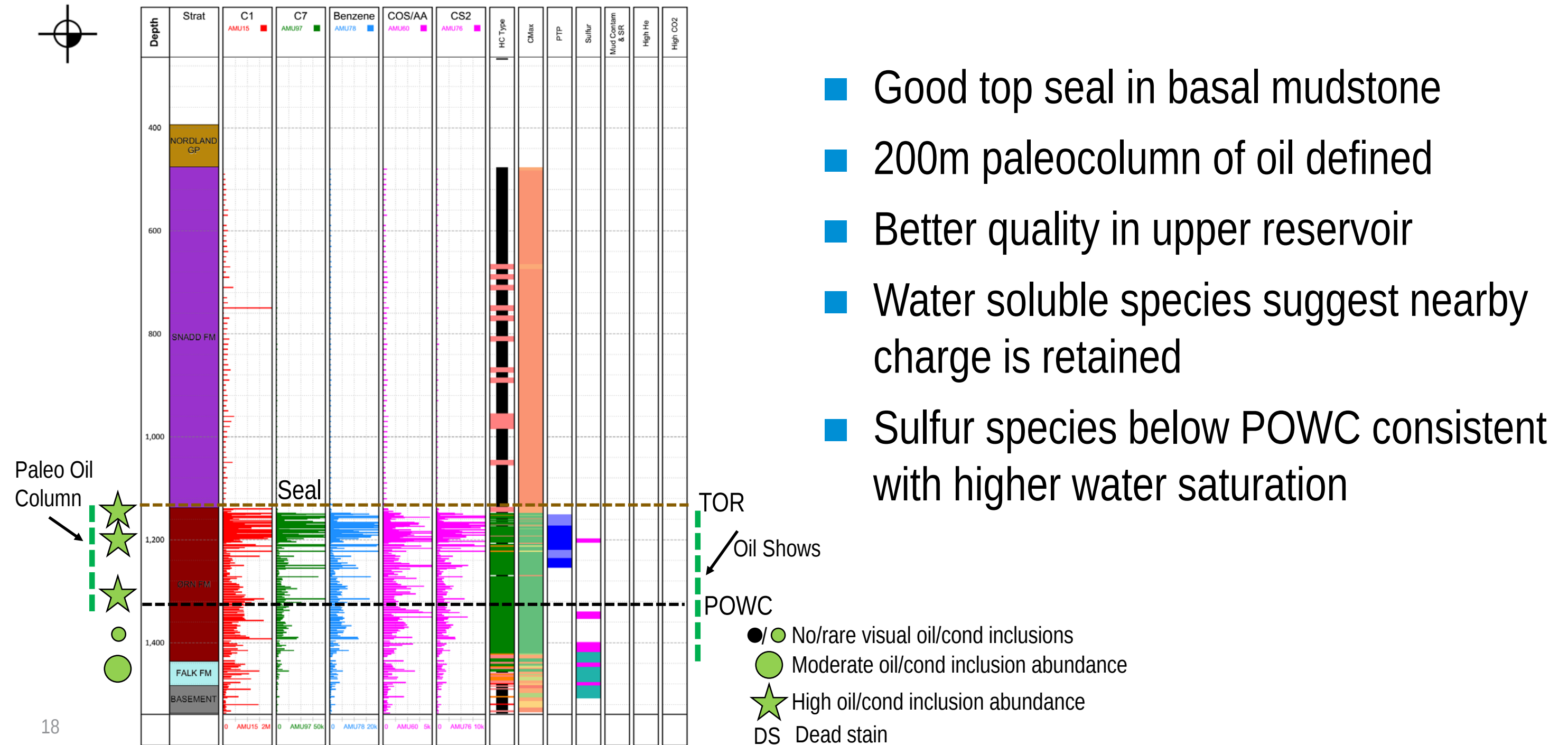


Data indicate strong maturity control on fluid distribution, but with some possible hot spots related to original depositional environment (see XRF-derived molybdenum distribution to the left).

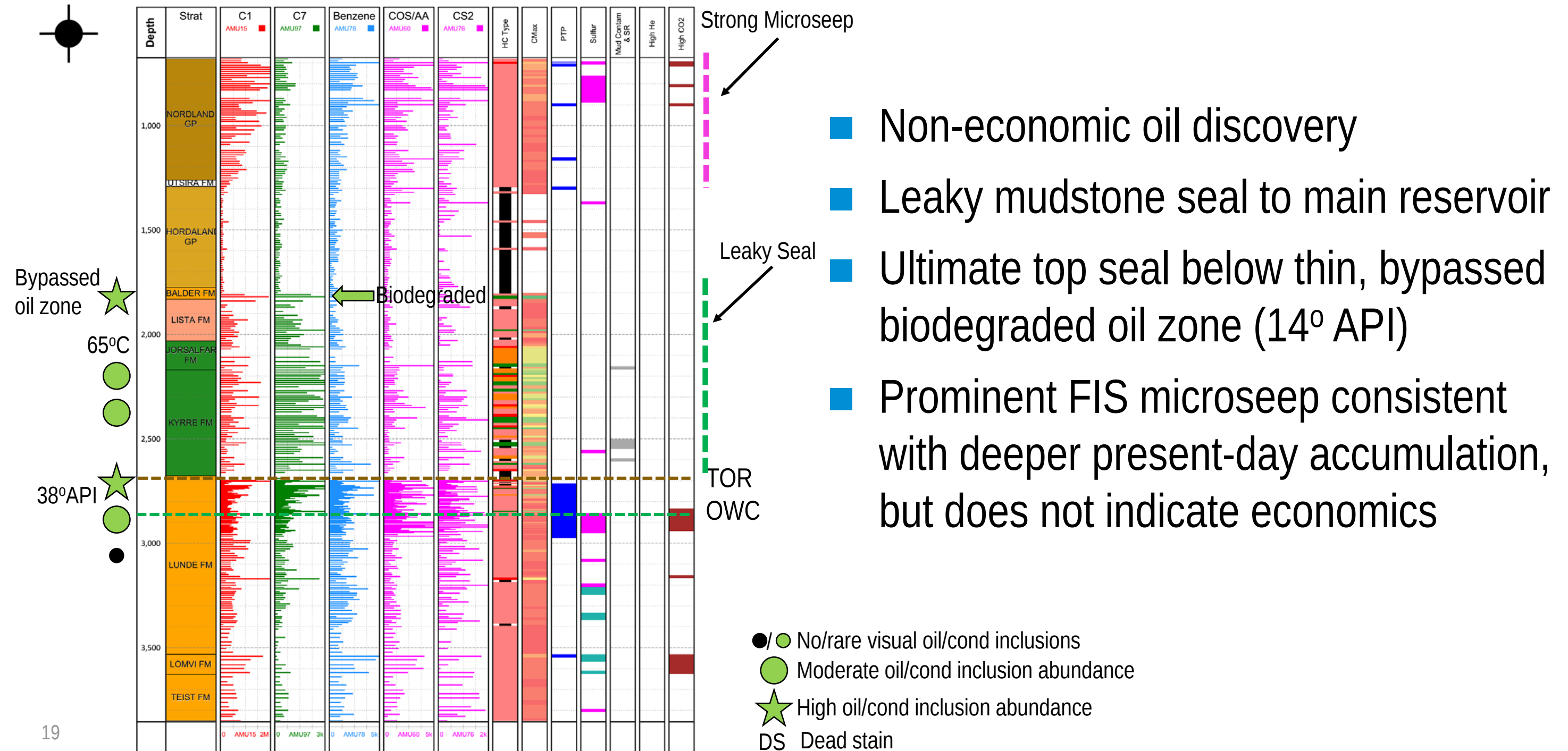
# Seal and Effective Source Rock; Barents Sea



# Good Seal in Non-source Shale; Barents Sea



# Poor Seal in Non-source Shale; Norwegian Sea



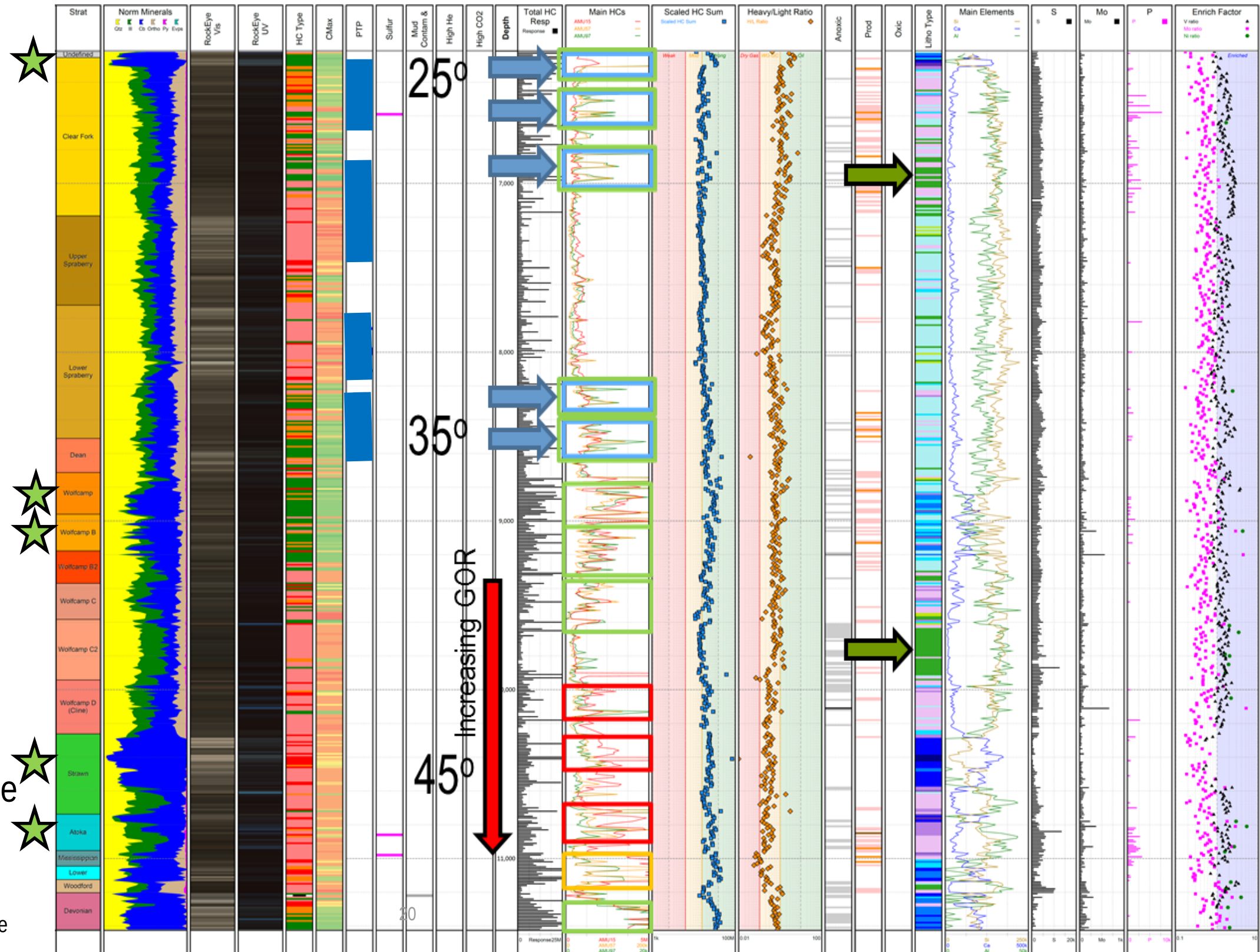
- Non-economic oil discovery
- Leaky mudstone seal to main reservoir
- Ultimate top seal below thin, bypassed biodegraded oil zone (14° API)
- Prominent FIS microseep consistent with deeper present-day accumulation, but does not indicate economics

# Unconventionals

## Midland Basin Well

- Comingled production from vertical wells; horizontal well planning needed
- Hydrocarbon indications throughout varying from dry gas to oil
- Most attractive zones are the Upper Clear Fork (or Glorieta), Lower Spraberry, Dean, Wolfcamp A-B and the Strawn to Devonian
- High visible oil inclusion abundance (stars) is consistent with high oil saturation
- A separate phase of dry gas is noted in the deeper section
- Higher water saturation above the Wolfcamp

★ = High oil inclusion abundance

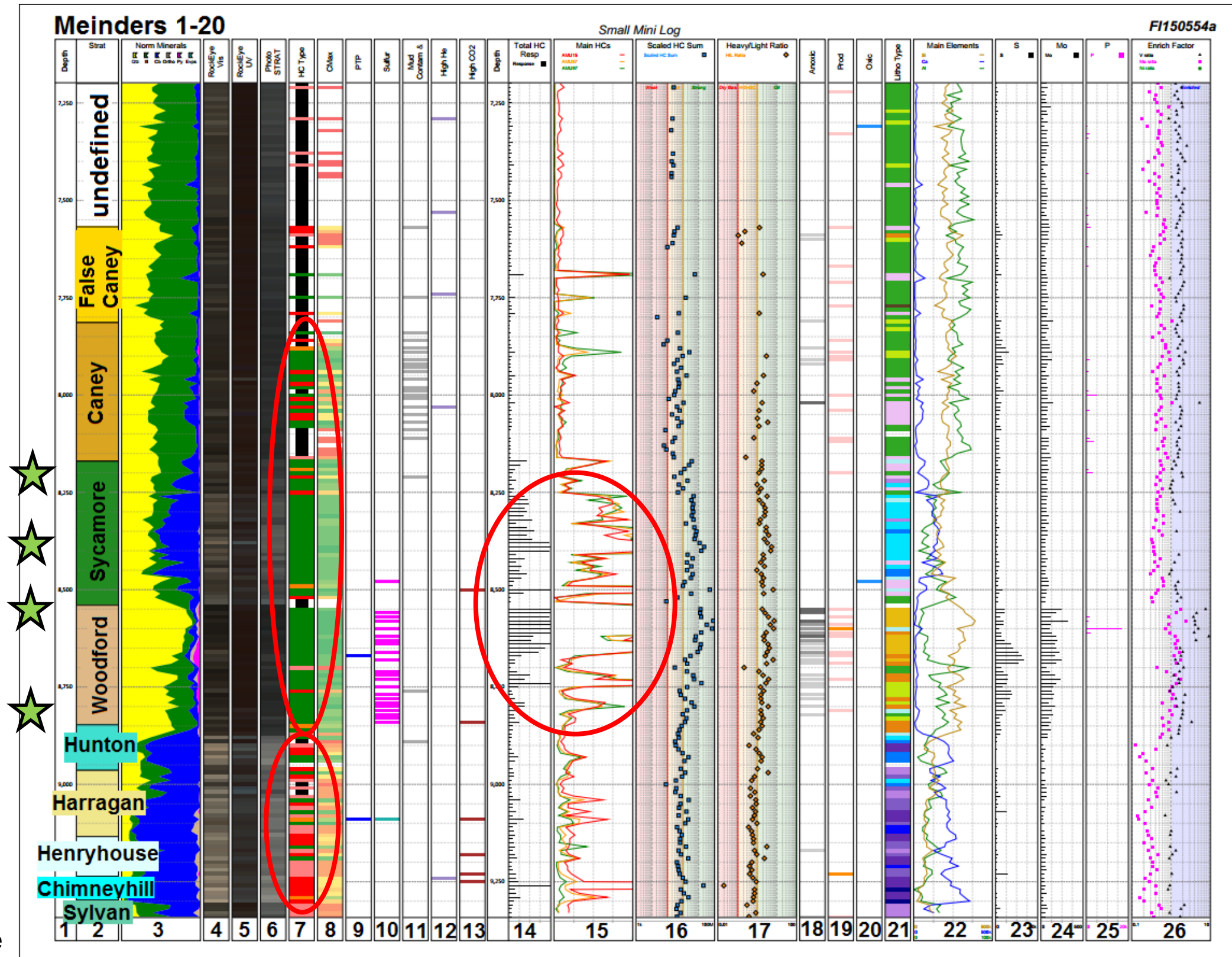


# Unconventionals

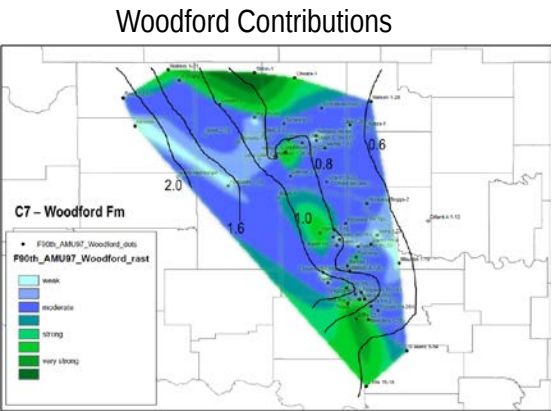
## Anadarko Basin Well

- Gas-condensate to oil-like responses from base of the Woodford to the Caney
- Wet gas to gas-condensate in the underlying Hunton to Sylvan
- The most anomalous zones are in Sycamore to Woodford
- High visible abundance of inclusions in thin section (stars)
- High saturation of migrated oil in the Sycamore and proximally generated oil in the Woodford
- Both are viable liquids targets (38-44 gravity) in this well

★ = High oil inclusion abundance

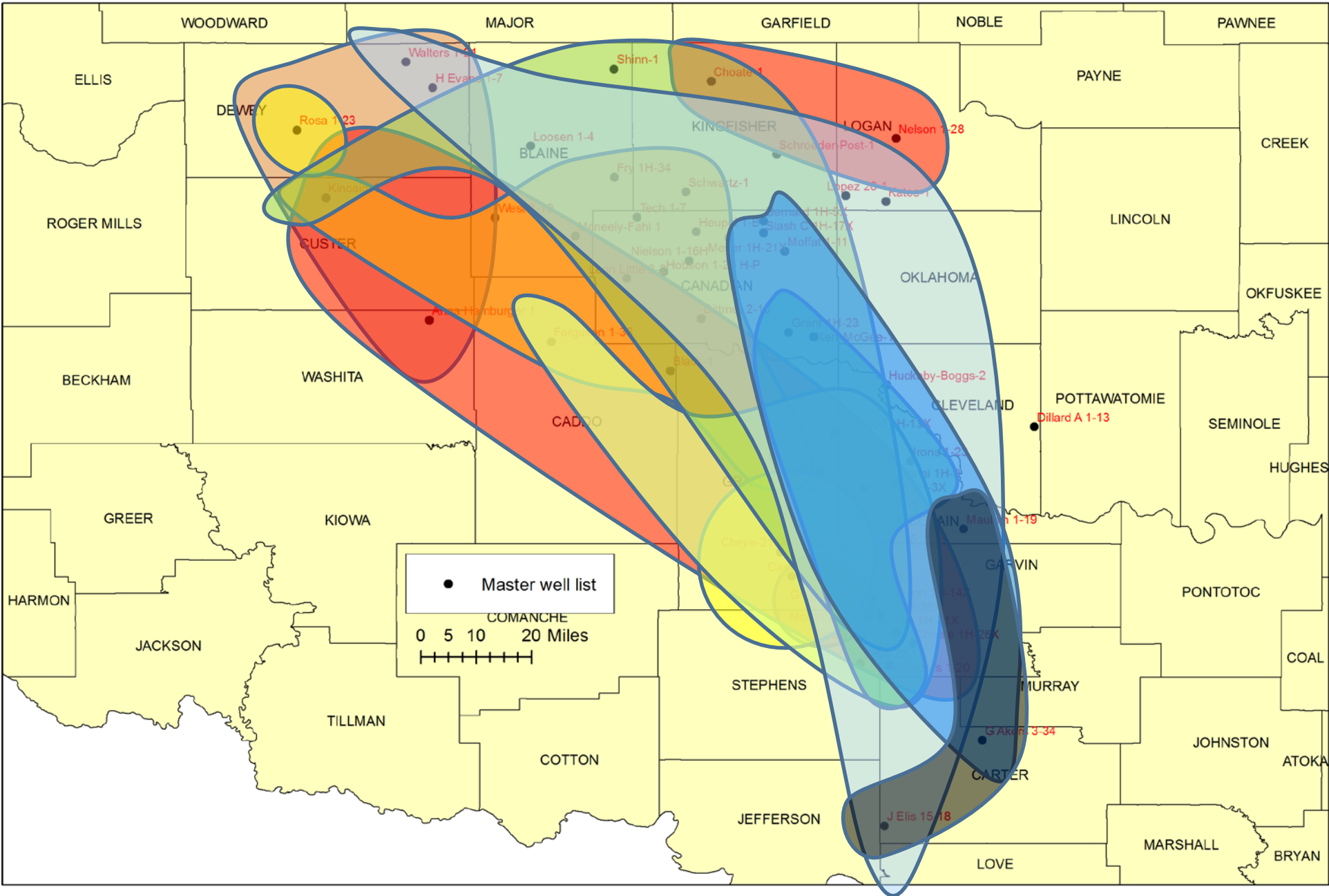


# STACK-SCOOP; Source of Non-Woodford Liquids



Formations Interpreted to have Contributed Liquid Petroleum within the Study Area (Excluding Woodford)

- Morrow
- Springer
- Chester
- Goddard
- Caney
- Meramec
- Sycamore
- Hunton
- Sylvan
- Viola



# Validating Maturity/Fluid Comp.

- Normalized fluid inclusion gas composition ratios and modeled vitrinite reflectance values correlate well with analog Barnett gas composition ratios from produced gas samples.
- Mean fluid inclusion gas compositions of the three wells modeled at 1.2-1.3 % Ro are similar to those of Barnett produced gas from wells also modeled at 1.2-1.3 % Ro.
- The fluid inclusion work helped validate the optical maturation assessment and basin model, and the projected produced gas composition.
- A well drilled in the area qualitatively confirmed the modeled maturity, predicted fluid composition and liquids API.

From Tobey, Schmude, Smagala and Hall, 2010: AAPG Search and Discovery Article #90122

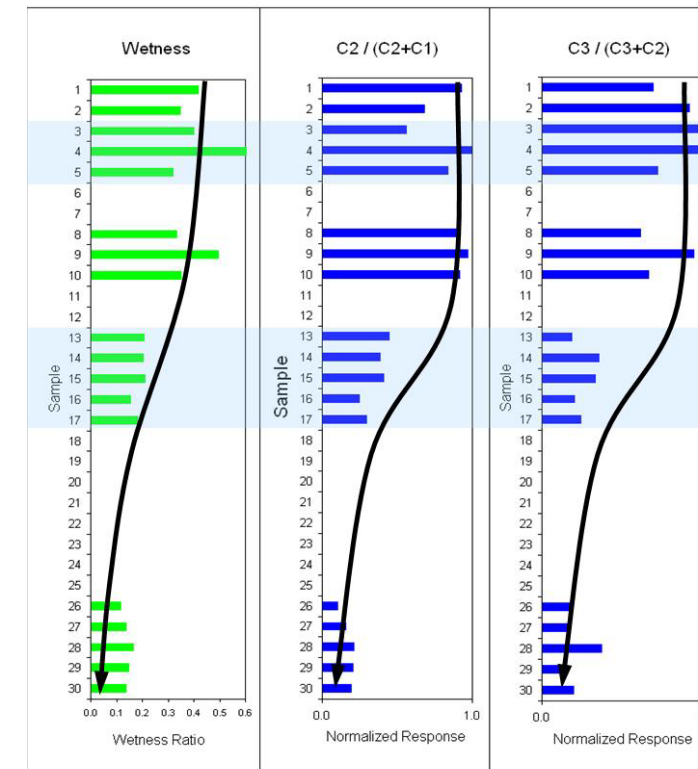
Study Area FIS

Well 1 & 2  
1.20 %Ro

Well 3  
1.30 %Ro

Well 4  
1.45 %Ro

Well 5  
1.80 %Ro



Barnett  
Produced Gas

1.2 %Ro

1.3 %Ro

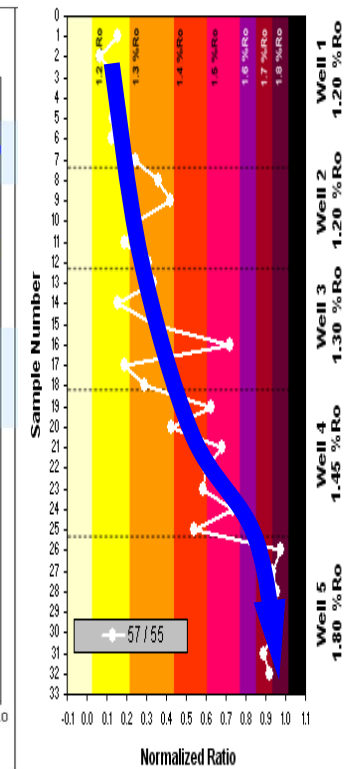
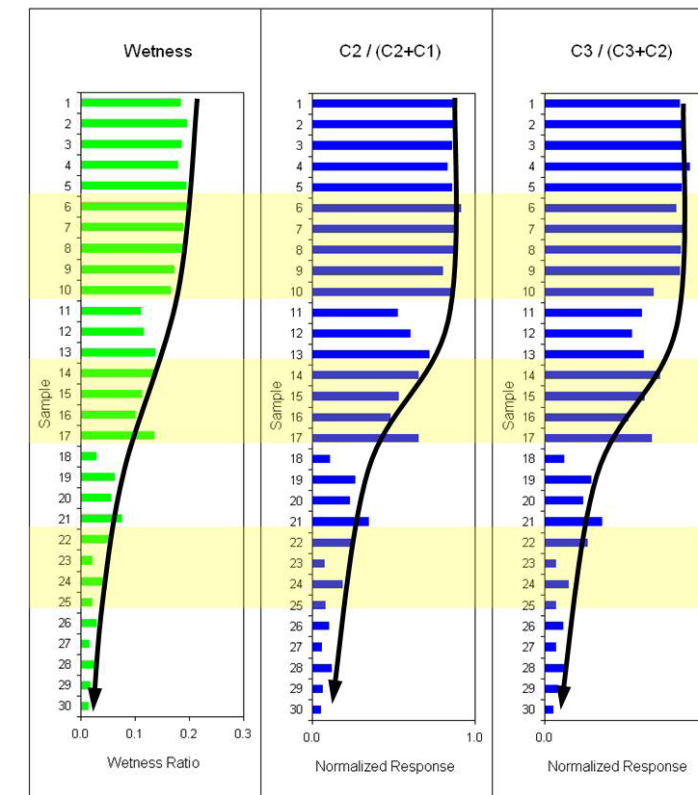
1.4 %Ro

1.5 %Ro

1.6 %Ro

1.7 %Ro

1.8 %Ro

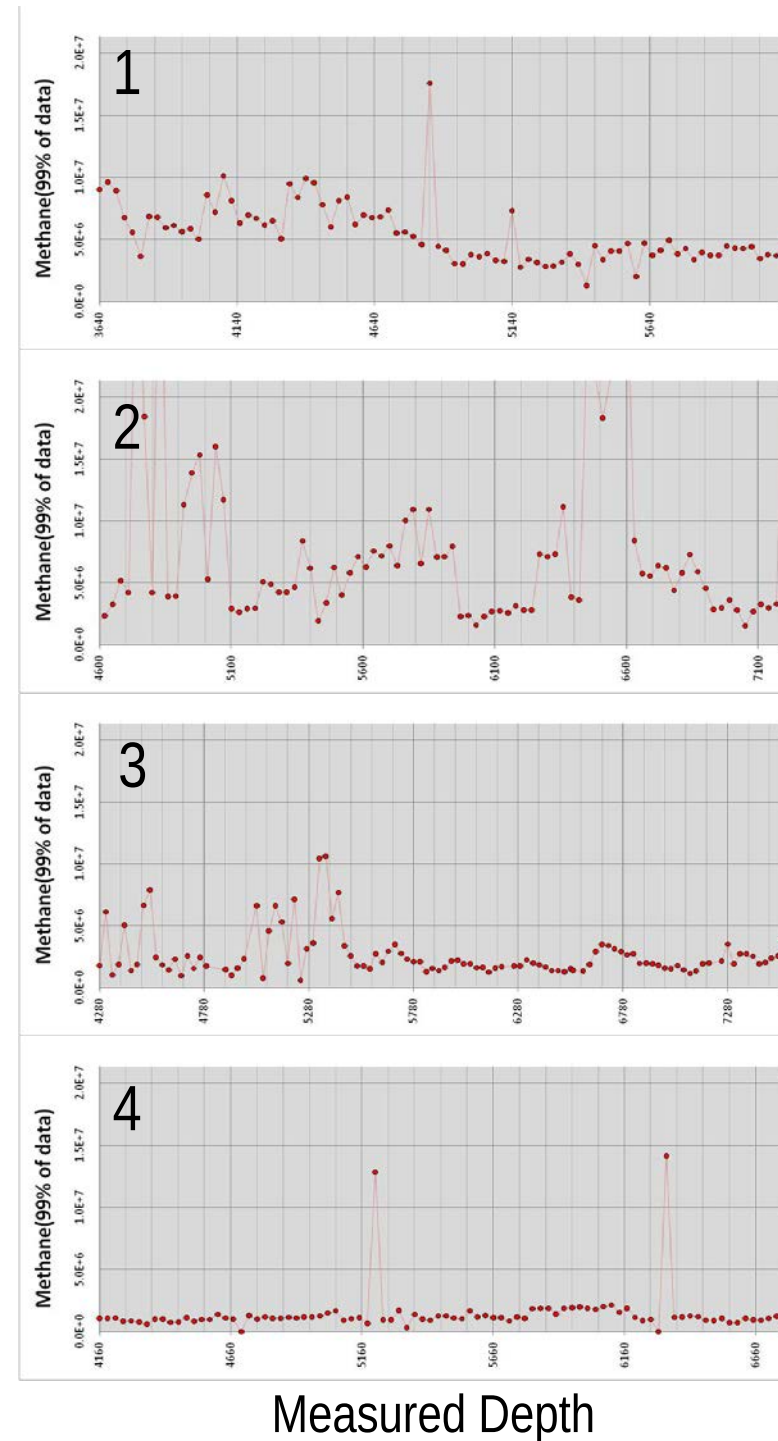


The alkane/ cycloalkane ratio increases with maturity within FIS data.

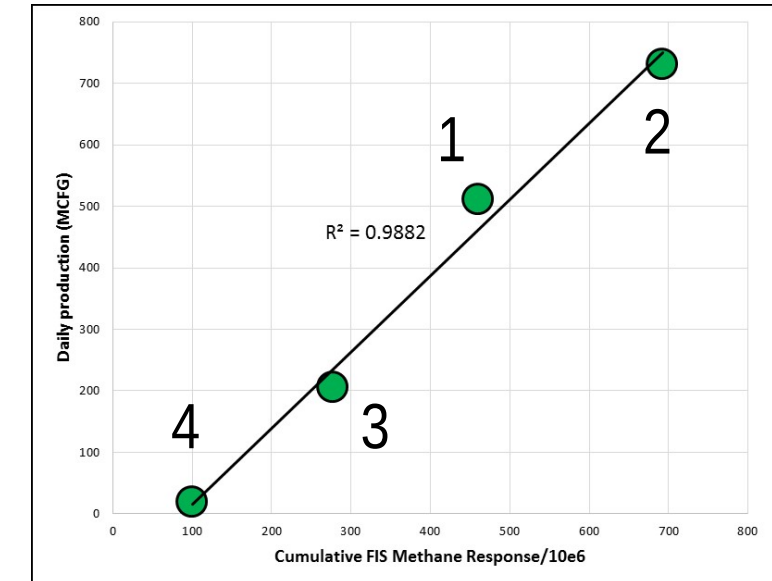
# Production and Compartmentalization, Appalachian Basin

- Four horizontals in an unconventional play
- FIS methane response plotted to same scale
- FIS strength often related to producibility
- Summed response proportional to eventual production
- Rank wells at an early stage
- Optimize completions
- Identify non-economic wells prior to completion

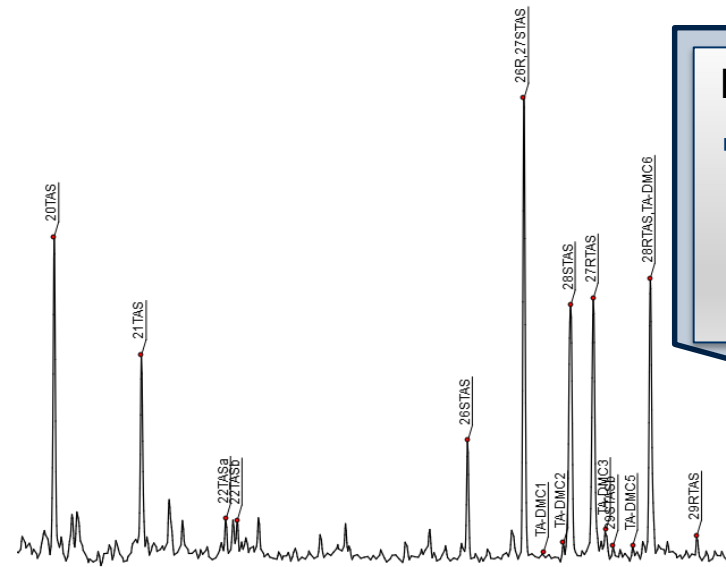
FIS Methane Response



FIS Response vs. Daily Production



# Extraction and Analysis of Petroleum Inclusion Gas and Liquid

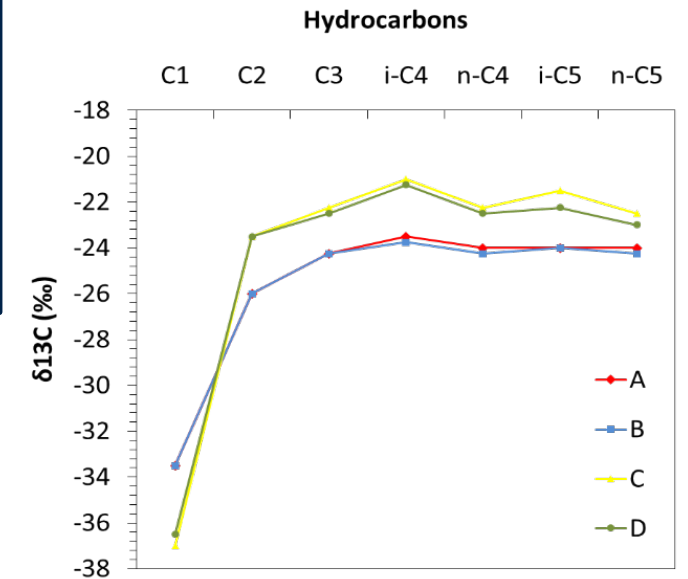


## Biomarker Analysis

- Compositional analysis via gas chromatography and mass spectrometry (GC-MS) of fluid inclusion

## Compound-Specific Isotope Analysis

- Compositional analysis via gas chromatography (GC) and for  $\delta^{13}\text{C}$  via IR-MS of fluid inclusions



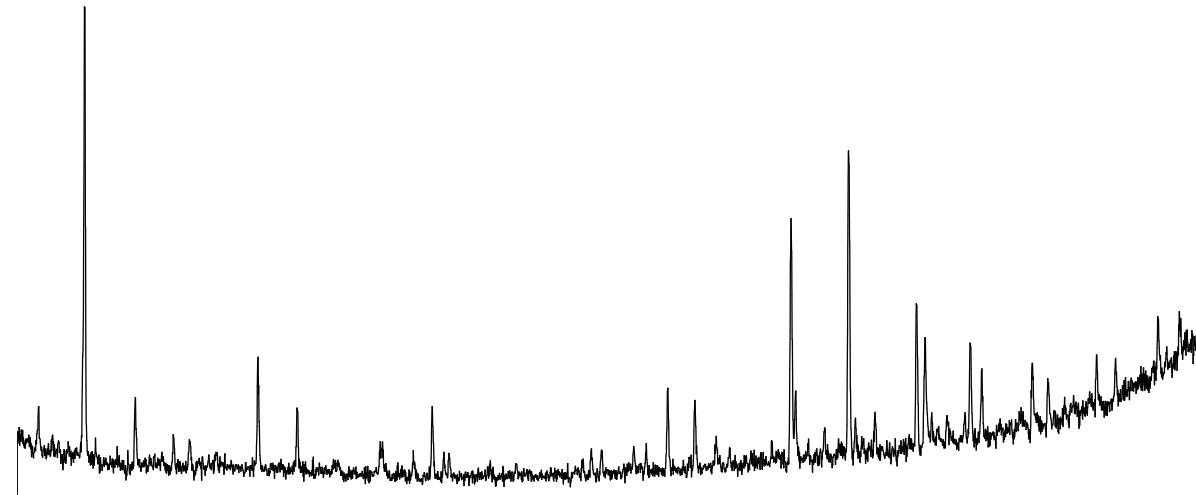
Correlate data from rock material, hydrocarbon samples, and fluid inclusions

- Refine and explain screening results
- Correlate shows and paleo accumulations to source rock
- Conduct maturity comparison
- Assess biodegradation and thermal alteration

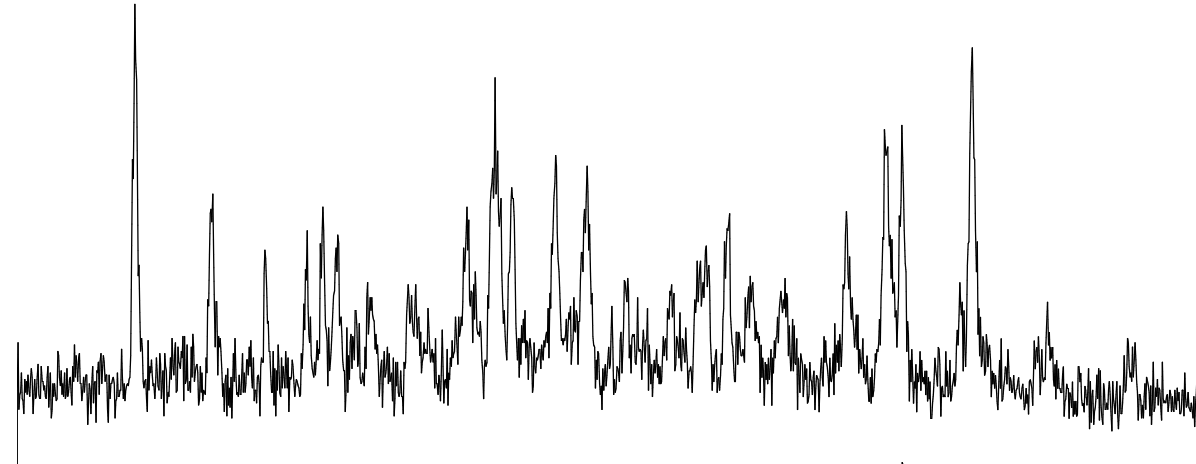
# STACK Play, Oklahoma

GCMS analysis of a fluid inclusion extract from the Kinderhook Shale (10850-10920 ft) indicates a marine carbonate or marly shale with about 0.85-0.9% VRE (late mature). Data suggest that this fluid may have contributions from both the Woodford and Mississippian and may have been “proximally generated”, based on the maturity of the underlying section.

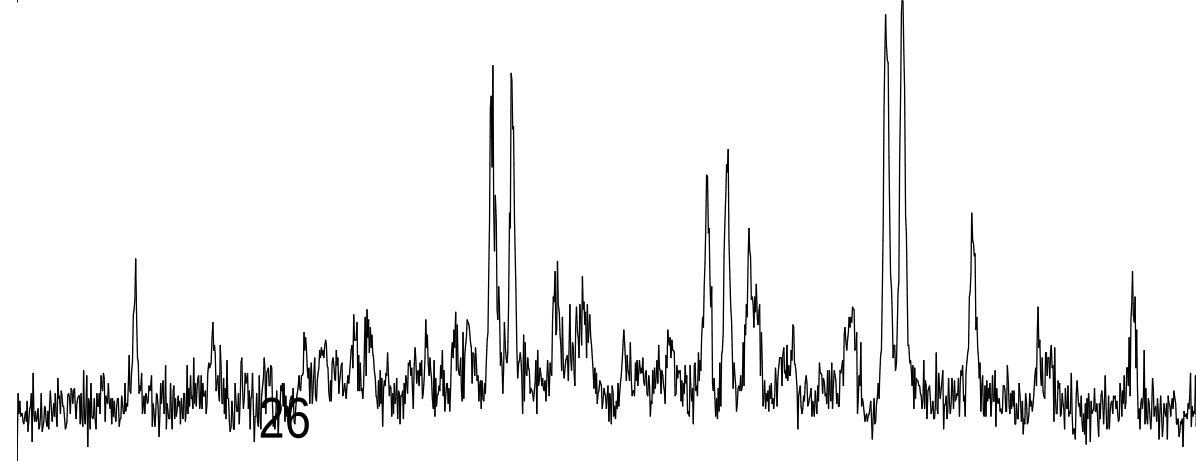
Fluid Inclusion Extract



m/z 191  
Tri- and Pentacyclic  
Terpanes



m/z 217  
Steranes



m/z 218  
BB Steranes

# Comparison of SR and Reservoir Fluid to Deduce Source

## Data from Shale

Th(oil) = 75-90°C  
API gravity = 35-37°  
Th(aq) = 80-95°C  
Salinity = 16-18 wt%

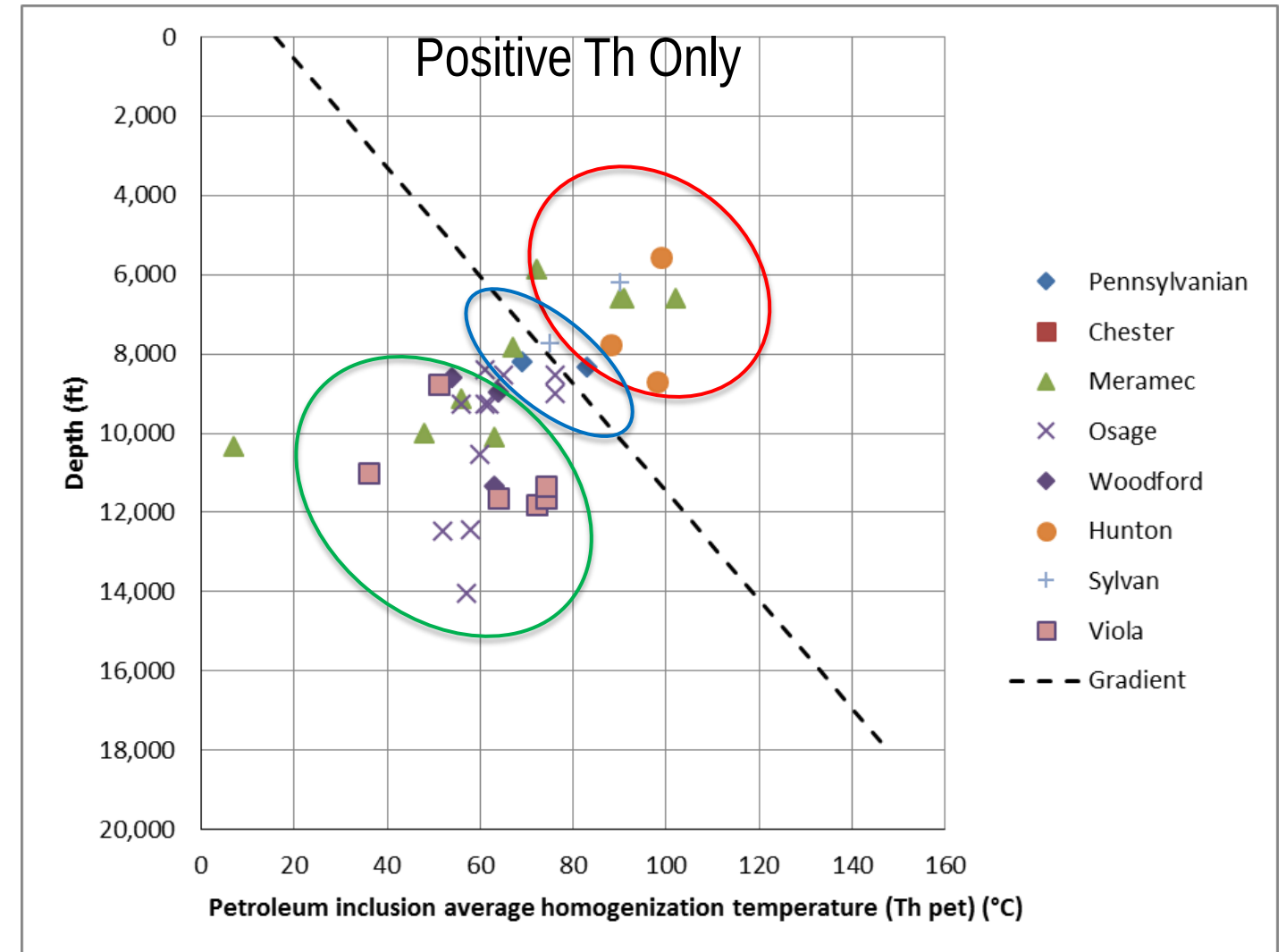
## Data from Underlying Carbonate

Th(oil) = 30-45°C  
API gravity = 45-48°  
Th(aq) = 80-95°C  
Salinity = 18-19 wt%

**Interpretation:** Moderate gravity gas-saturated oil is/was present in the shale. Highly undersaturated upper-moderate to high gravity light oil or condensate in the underlying carbonate was not locally produced, and would require a different source or an equivalent source at higher maturity. Shale maturity is 0.6-0.65 Ro (Th suggests 0.6). Petroleum in carbonate was probably generated at  $\geq 1.0$  Ro. High salinity indicates open system and evaporite-derived pore fluids.

# Saturation State of Reservoir Fluid

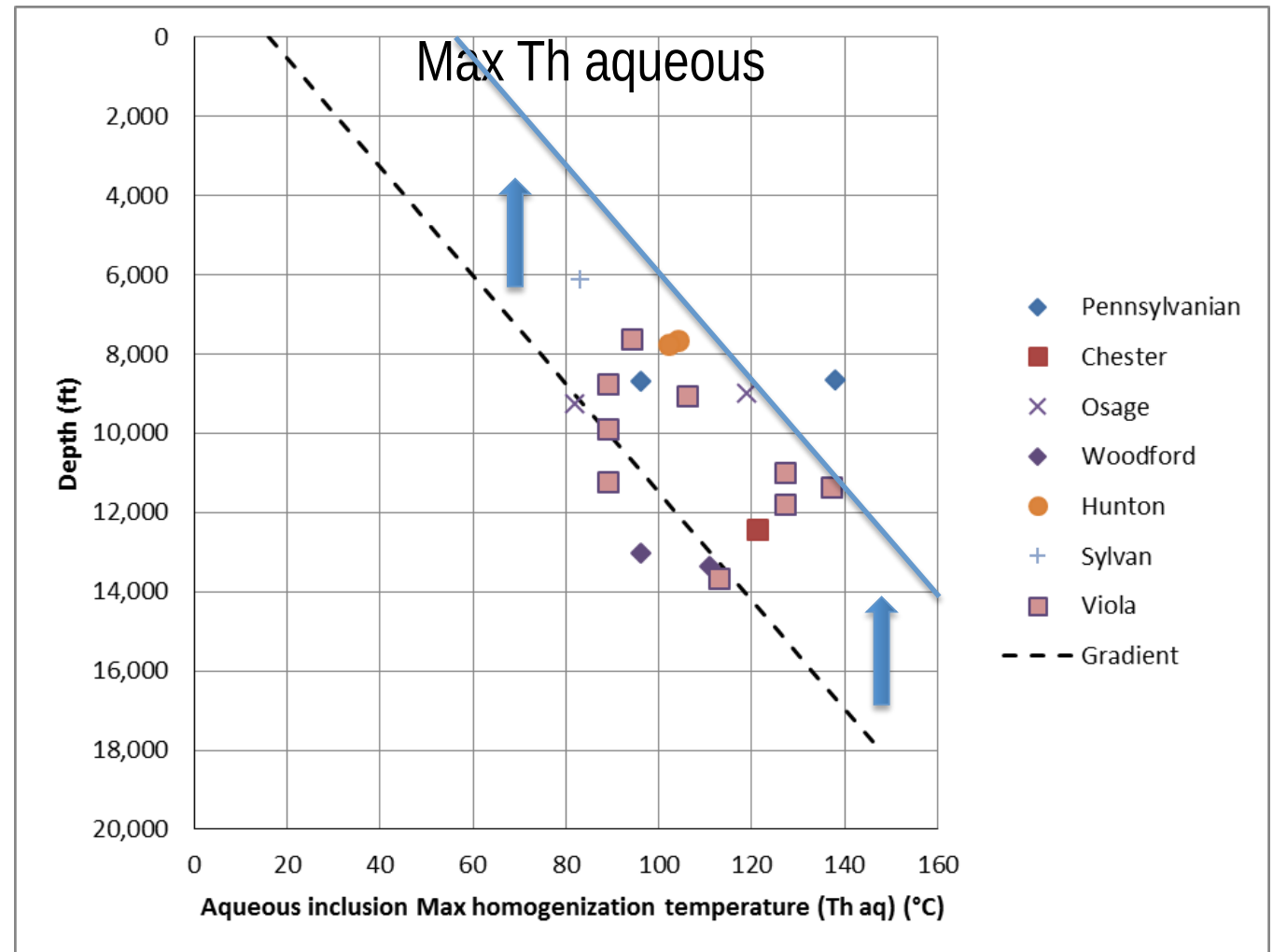
- Temperatures above the gradient line may indicate uplift since inclusion entrapment and suggests dual phase fluid in the reservoir
- Temperatures near the gradient line suggest that fluids may be near saturation and may drop into the two-phase field when produced
- Temperatures well below the gradient line indicate gas-undersaturated fluids



Gradient: 24°C/km with mean annual surface temperature of 16°C

# Uplift

- Th values of aqueous inclusions are probably close to actual trapping temperatures (in the absence of stretching or heterogeneous entrapment of water and gas)
- A number of inclusions have higher measured Th than current estimated subsurface temperatures
- Data suggest up to 5,000 ft of uplift since inclusion formation (assuming a constant geothermal gradient)

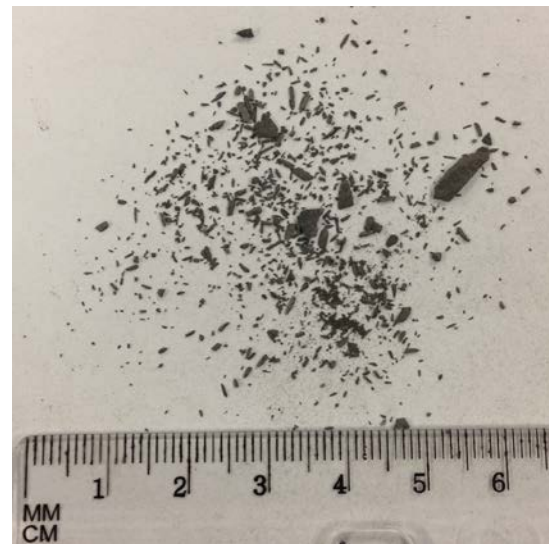
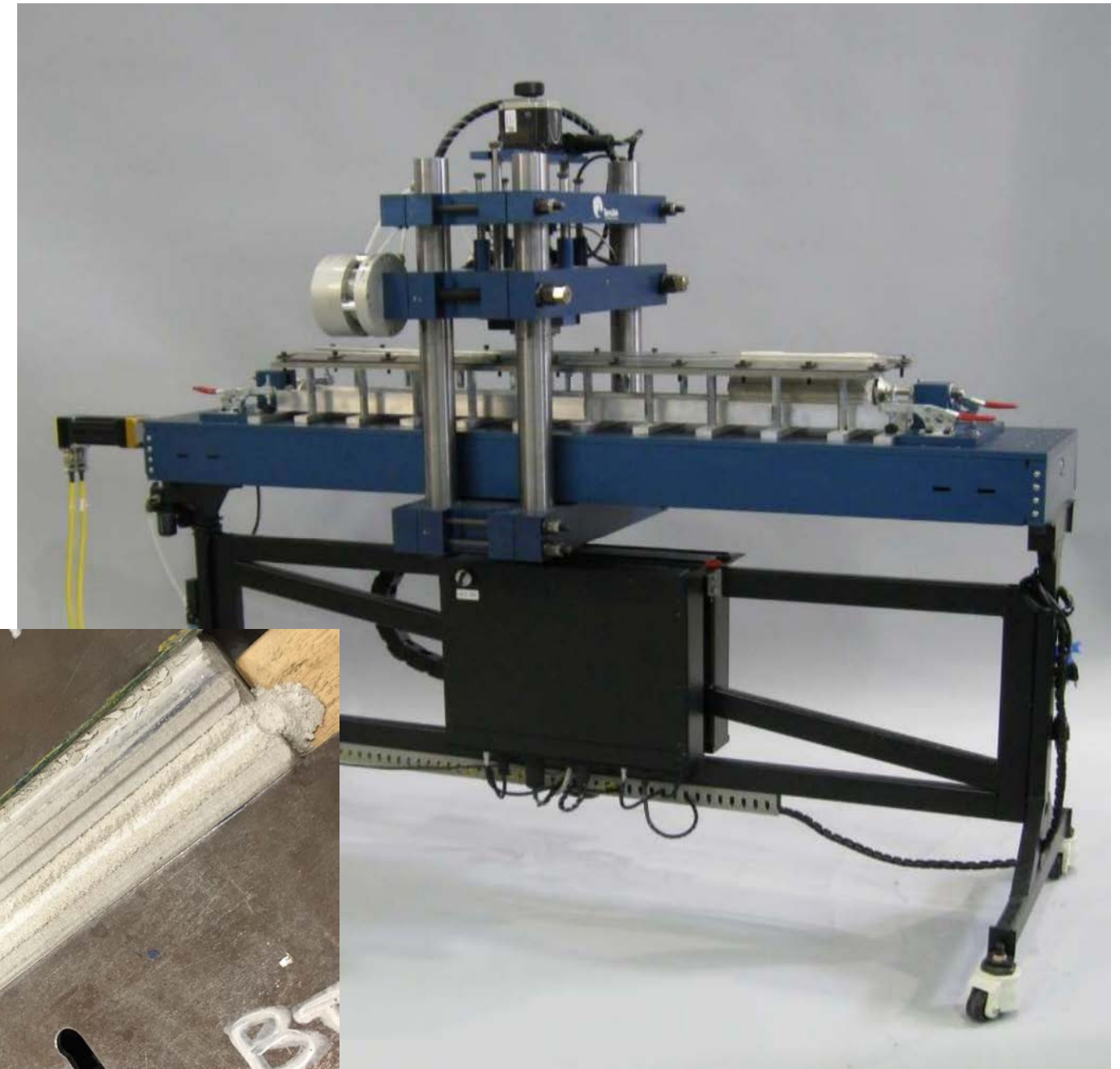


Gradient: 24°C/km with mean annual surface temperature of 16°C

# What's Next: Scratch and DRIFTS

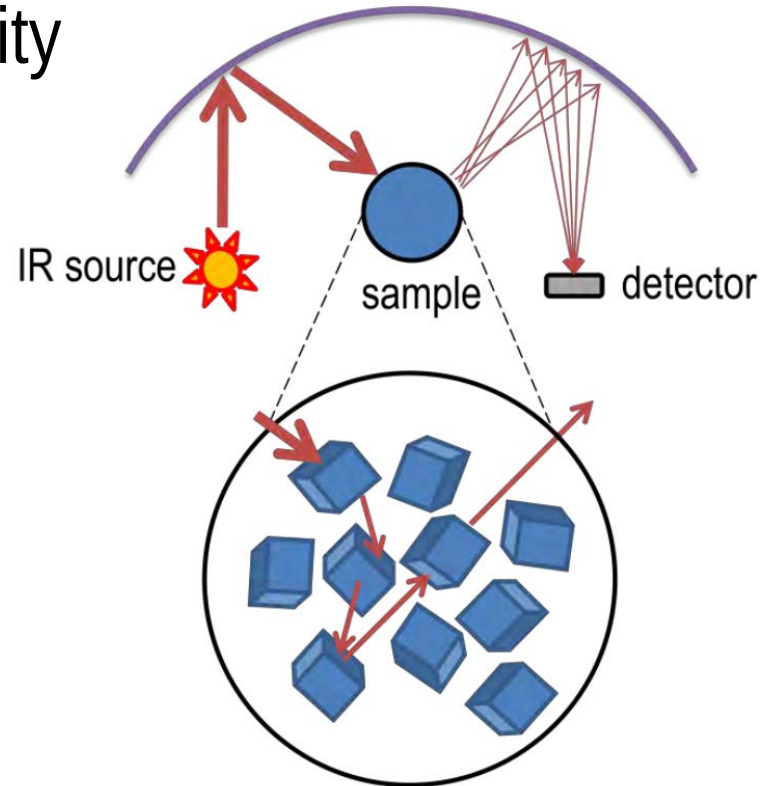
- The scratch machine produces a continuous strength index by measuring the force necessary to cut a groove of constant depth in core
- Provides a means of characterizing mechanical behavior and heterogeneity at a fine scale
- Produces scrapings, which, contain some reasonably-sized material
- This byproduct is usually discarded

Scratch Machine



# What's Next: Scratch and DRIFTS

- Diffuse reflectance infrared Fourier-transform spectroscopy (DRIFTS) provides mineralogical data on powdered material with little sample preparation
- The technique can also be used to estimate TOC and, in some cases, maturity
- DRIFTS has been automated into the current cuttings analytical workflow, although some technical challenges remain

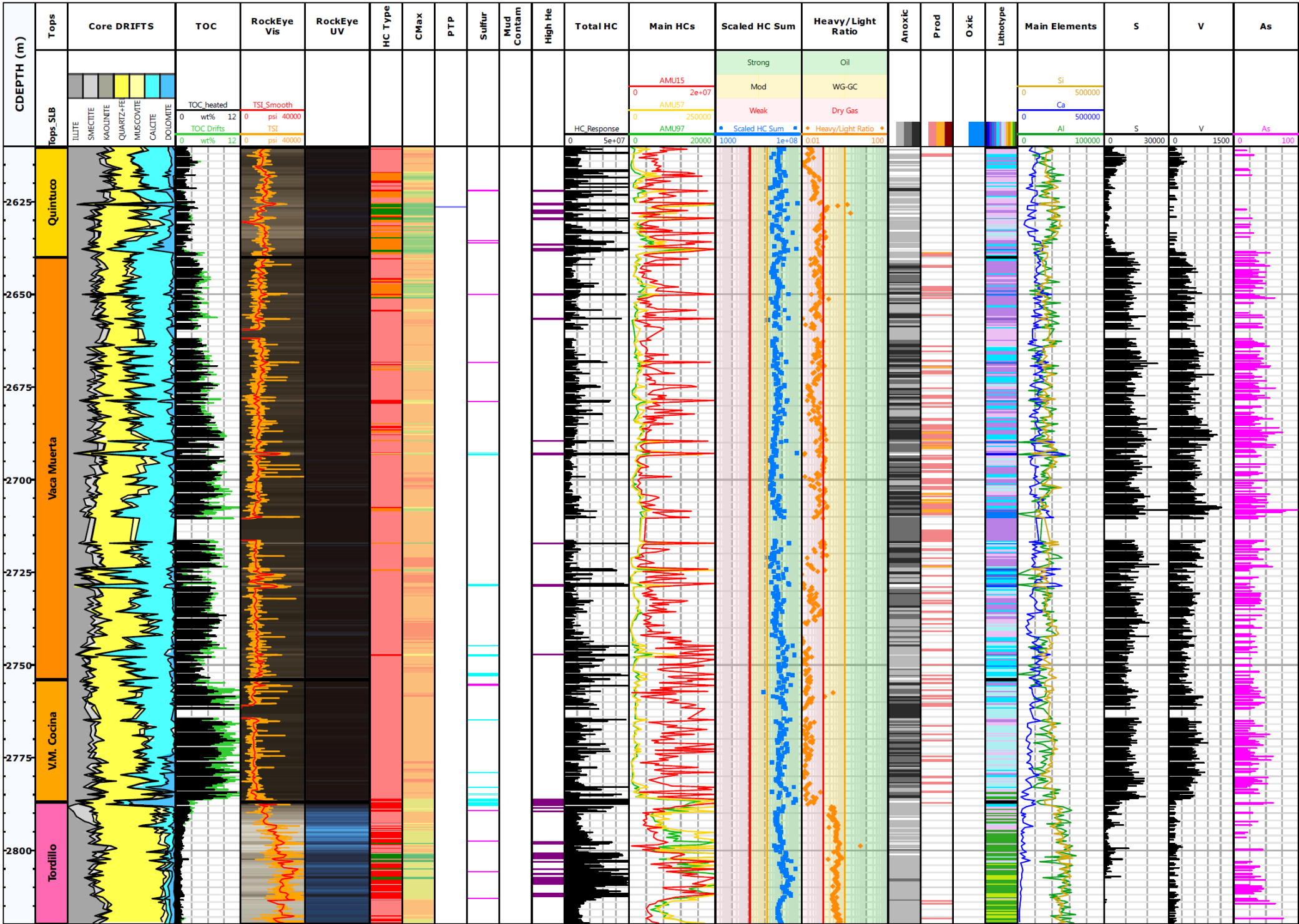


Bruker Alpha DRIFTS



# Vaca Muerta

- Dry to wet, non-associated gas; API >50
- Sulfur species and helium associated with calcite veins; deeper mature gas
- Higher Sw near base of VM
- DRIFTS and XRF-derived TOC is consistent with LECO to within 1-2%
- Free and kerogen-related TOC can be distinguished
- Maturity of 1.0-1.2% VRE
- Ash beds and calcite veins distinguished
- One possible optimum completion zone identified by combination of data



# Conclusions

- Fluid inclusions are prevalent in fine-grained rocks. They may be locally generated or migrated, visible or beyond optical resolution, and can contain a wide range of organic and inorganic species.
- A rapid, automated process using archived, unpreserved rock material is available to destructively analyze these trapped fluids. Classical optical techniques can be used on larger inclusions.
- No lithologic, mud system or age restrictions on application.
- Establish rock-fluid databases with existing well control.
- Impact exploration efforts & reservoir characterization; potential to refine or rewrite petroleum system concepts in a given area.

