Vitrinite reflectance of Cretaceous coals and thermal maturity of the Niobrara Formation, DJ Basin, Colorado, USA

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Outline

- Building a vitrinite reflectance lab from scratch
- Vitrinite reflectance of the J sandstone / maturity of the Niobrara
What is a maceral?

• A maceral is a building block of an organic rock
  – Mineral: rock as maceral: coal
  
  – Main types:
    • Vitritine
      – Cells and cell walls of woody material
    • Liptinite
      – Spores, waxy material, resins
    • Inertinite
      – Fusinite, semi-fusinite
        » Oxidized material
What is vitrinite reflectance?

- Relationship between “grayness” of vitrinite macerals and thermal stress
  - Developed in the coal industry to assess rank
- In a controlled environment, we measure the amount of light reflected off the surface of a vitrinite maceral as a proxy for thermal stress
  - Need to be able to identify vitrinite and distinguish it from other maceral types
Problems

• Commercial reflectance work
  – Heavy backlog
  – Expense
  – Inconsistency between service companies, between petrographers
  – A need to independently verify their work

• Vitrinite reflectance studies from marine shales:
  – The risk is high that measurements have been taken on oxidized/recycled macerals
  – Difficult to identify tiny macerals
  – Small sample sizes
  – Well log-based approximations, Tmax, etc. are less precise than VRo
Solutions

- Bring reflectance work in house
  - “How hard could it be?”
- Measure actual coal rather than dispersed OM in shale
  - In the case of the Niobrara, let’s measure coaly material in the J sandstone rather than “vitrinite” in the Niobrara
Let’s buy a complete vitrinite reflectance set-up...
Then again, maybe not...
In the late 1970s Bob Cluff and others at the Illinois State Geological Survey were studying thermal maturity of the New Albany shale.

On their desks were Leitz microscopes complete with photometers and analog photometer controllers.

“I bet we can recreate that.”
The treasure hunt begins...
“This is gonna be easy!”

- Leitz Orthoplan base and stage (~$1500-3000)
  - Common in silicon wafer inspection, biology
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“Patience is a virtue...”

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- **Oil immersion objective** (~$600-1200)
  - “Unobtainium”
- **Photometer and controller** (~$3000)
  - The hardest part
Lots of other stuff required

- Reflectance standards (~$850 each)
- 546 nm green interference filter
- Desiccator cabinets (~$200-300 each)
- Grinding/polishing wheel
- Pelican foam-lined shipping case (~$250)
- Tons of lab supplies
- Reference books
Macerals

- Vitrinite
Macerals (cont’d)

- Several maceral types co-occurring
Macerals (cont’d)

- Fusinite
Macerals (cont’d)

- Semifusinite
Name that maceral
Search through core...
Collect the good stuff...
Prepare the plugs...

The sample preparation process is based on the recommended practices in ASTM D2797/D2797M-09.
Finally, we can get started!

Leitz Orthoplan microscopes fitted with Leitz MPV photometers

- 500x magnification under oil immersion
- 50+ measurements on randomly oriented vitrinite macerals
- Klein & Becker mono-crystalline standards
- Calibrations before and after analysis to ensure there was not drift
- Bracket the sample with two “nearby” standards
Analysis

(Each image is roughly 500 microns across)
Typical histograms

Marine shale example: Mel Bickling 42-21

Coaly material example Horst 1
Geology

Late Cretaceous paleogeography (~85Ma)

- Niobrara is an organic-rich unit of interbedded chalks and marls
- It has emerged as a significant unconventional resource
- Self-sourced
- Because migration of hydrocarbons in these shale oil systems can be minimal, maturation is crucial for exploration
Study area

Modified from Blakey (2014)
Stratigraphic column

- **Niobrara**: marine unit, poor in terrestrial organic material
- **J-sand**: fluvial/deltaic with thin coaly laminations and “coffee grounds”
- Examined well over 300 J- & D-sand cores
  - About 70% appeared to have coaly material, so a sample was collected
- Ultimately we’d like to know maturity in the Niobrara

(modified from Higley and Cox (2007))
Subsidence profile

Significant basin subsidence would not have occurred until after and during Kn deposition. Therefore, maturation history for the Kj and Kn would be functionally the same.
Quality control

- Multiple petrographers analyzing the same samples
  - 20+% redundancy
  - Measuring on different days, different calibrations
  - Excellent convergence ($R^2 = 0.95$)

- Mean $= 0.85\%$
  - Std Dev $= 0.03$

\[
\begin{align*}
\text{Petrographer 1} & : \text{Mean} = 0.85\% \quad \text{Std Dev} = 0.03 \\
\text{Petrographer 2} & : \text{Mean} = 0.85\% \quad \text{Std Dev} = 0.03
\end{align*}
\]
Quality control (cont’d)

• Cross-verification with a commercial lab
  - 41 samples
  - Excellent correlation ($R^2 = 0.97$)

![Graph showing correlation between TDG measurement and Weatherford Labs measurement with a linear equation $y = 0.9909x - 0.0065$, $R^2 = 0.967$.](image)
Maturity transform

- Reflectance-depth transform based on well in Tainter (1984) study

Raw data from Tainter (1984)
Correction

- Resulted in reduction in VR by an average of 0.03%
VRo contour map

- Over 180 new data points
Comparison to other studies
Conclusions

• Measuring vitrinite reflectance in the Niobrara is inherently problematic
• This can be overcome by measuring vitrinite reflectance on coaly material from the stratigraphically deeper J- and D-sands
• A research-quality vitrinite reflectance microscope can be built from parts on ebay for less than $10,000
  – With patience and perseverance it can probably be done for considerably less
• Over 180 new vitrinite reflectance data points have helped to both expand and refine the maturity profile of the DJ Basin.
• The new data have revealed localized previously-undocumented high and low temperature anomalies.
References cited


