ICCP WORKING GROUP
IDENTIFICATION OF PRIMARY VITRINITE IN SHALE
2014 REPORT

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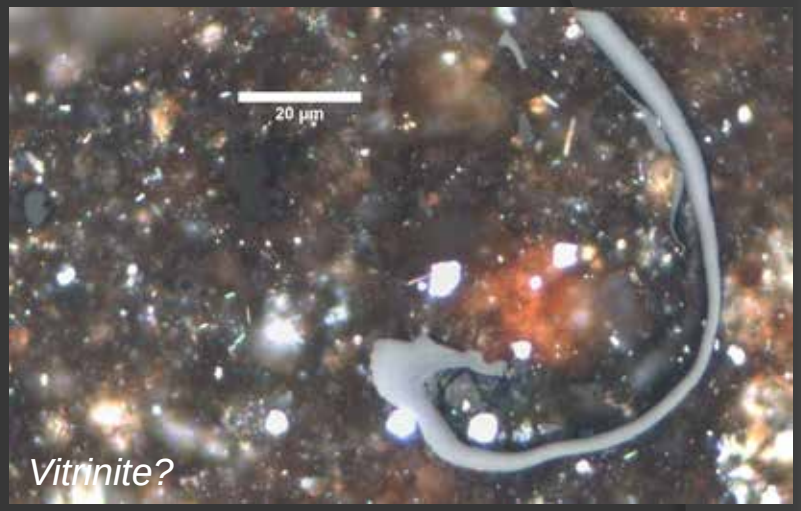
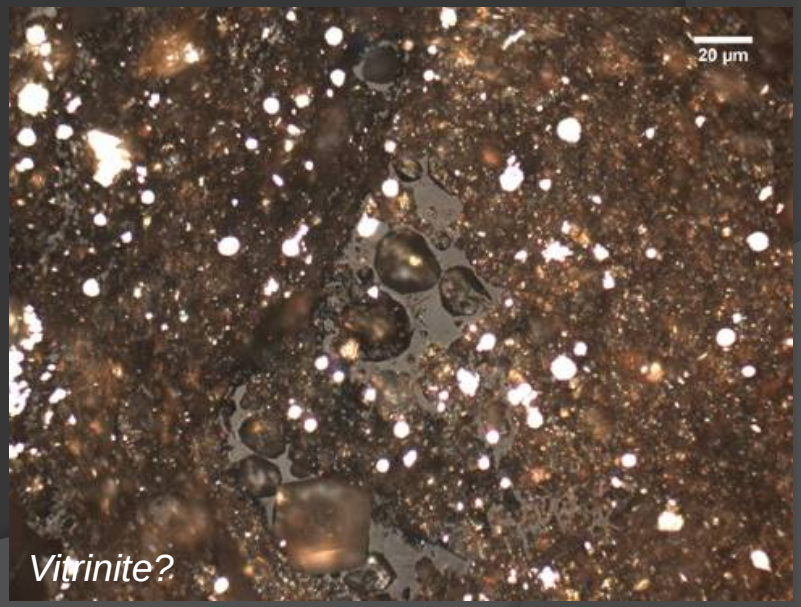
Presented for ICCP Commission II, September, 2014

Outline of this presentation

- Problem to be solved
- History of the working group
- Findings and products to-date
- Discussion and future directions



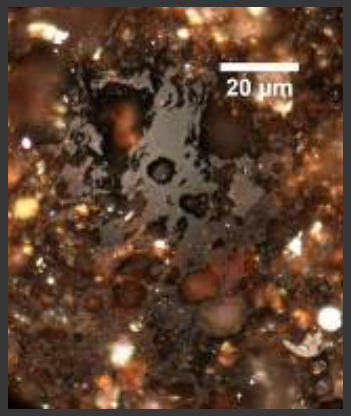


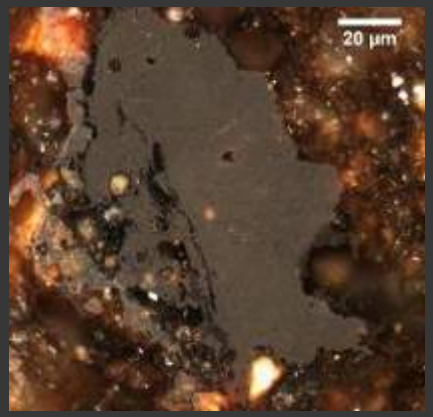
WHAT ARE THE BIGGEST PROBLEMS WITH IDENTIFICATION OF PRIMARY VITRINITE AND REPRODUCIBILITY OF REFLECTANCE MEASUREMENTS?

- Recognition of primary vitrinite and distinguishing it from similar macerals in shale such as bitumen, recycled vitrinite, and low-reflecting semifusinite
- Lack of supporting documentation and data
- Lack of experience – or a particular experience guides interpretation
- Pressure to determine thermal maturity of vitrinite when vitrinite may or may not be present
- Poor polish
- Preparation: whole rock vs. kerogen concentrate

RECOGNITION OF PRIMARY VITRINITE

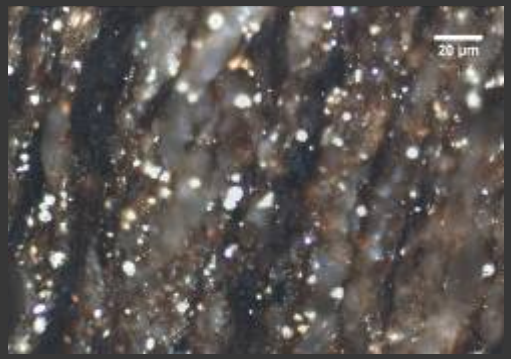
Distinction from bitumens

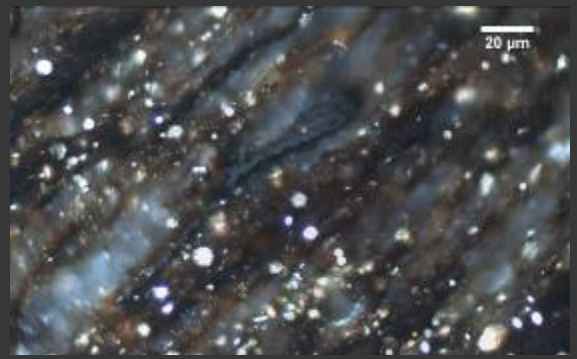




Vitrinite is not pore-filling or anastamosing, is not embayed by authigenic minerals, often is brighter, thicker, boundaries are more distinct, does not have mosaic anisotropy, may occur with other macerals; whereas bitumens cross bedding, can occur as droplets, dissolve in solvents, and may have mosaic anisotropy – rock type, rank, and geologic occurrence may influence expectations

Distinction from bituminite





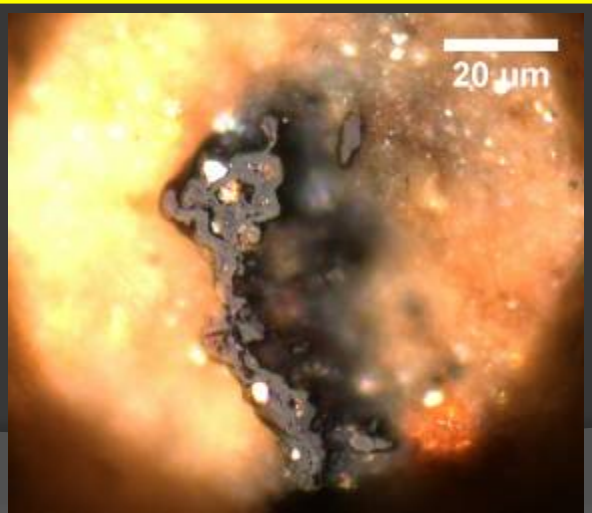
Vitrinite has brighter reflectance, lower fluorescence, more distinct boundaries, is more blocky and evenly colored; whereas bituminite often is observed in association with lamalginite and micrinite, is indistinct and wispy, and is speckled or unevenly colored

RECOGNITION OF PRIMARY VITRINITE

Distinction from recycled/oxidized vitrinite

Primary vitrinite is not as bright, more angular, recycled vitrinite may have bright or dark halos, recycling may be anticipated from geologic context, e.g., orogeny, recycled vitrinite has higher spread of reflectance values

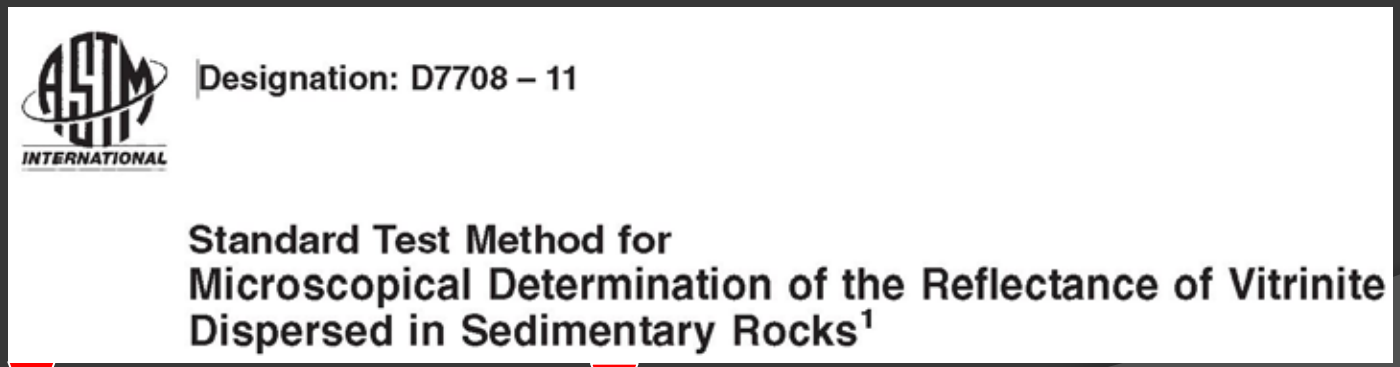
Distinction from low-reflecting semifusinite



Vitrinite is not as bright, has lower relief, is not usually as arcuate, does not have well-preserved cellular structure-lumens, has less distinct grain margins, has a more porous and textured surface; semifusinite may have irregular anisotropy regions

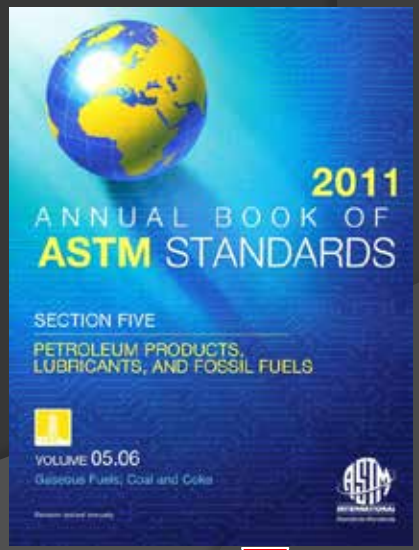
Identification of primary vitrinite: History of the working group

- Working group proposed by Angeles Borrego at September 2008 Oviedo ICCP meeting
- Results of survey about DOMVR analysis and identification of primary vitrinite presented at 2009 Gramado meeting and published in ICCP News No. 48, Nov. 2009
- New ASTM standard for DOMVR published in 2011 Annual Book of ASTM Standards September 2011



Designation: D7708 – 11

Standard Test Method for
Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks[1]



Oviedo 2008 ***Gramado 2009*** ***Belgrade 2010*** ***Porto 2011***

Identification of primary vitrinite: History of the working group cont.

- Six samples used to test ASTM D7708 via interlaboratory study with twenty-two laboratories in 2012-2013
- Round robin results presented to ICCP in Sosnowiec, 2013
- Results presented to oil and gas community at AAPG, Houston, USA, April 2014
- Results published in J. Marine and Petroleum Geology, 2014

Porto 2011 ***Beijing 2012*** ***Sosnowiec 2013*** ***Kolkata 2014***

Results of the 2012-2013 interlaboratory study

Marine and Petroleum Geology 59 (2015) 22–34

Contents lists available at ScienceDirect

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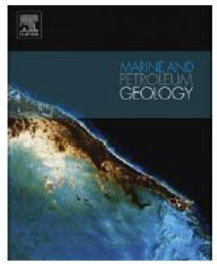


Research paper

Standardization of reflectance measurements in dispersed organic matter: Results of an exercise to improve interlaboratory agreement

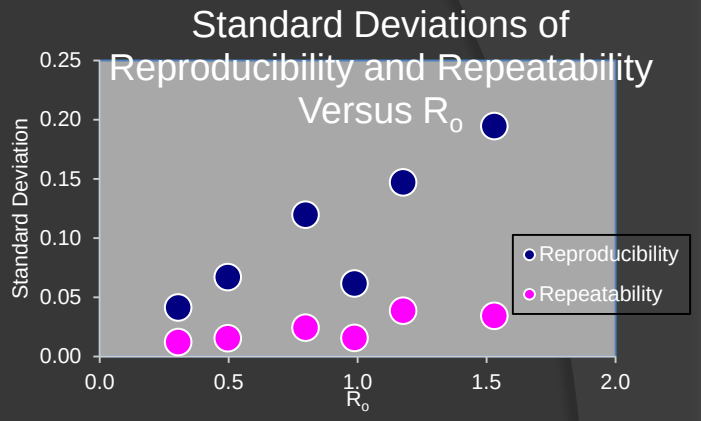
Paul C. Hackley [a, *], Carla Viviane Araujo [b], Angeles G. Borrego [c], Antonis Bouzinos [d], Brian J. Cardott [e], Alan C. Cook [f, 1], Cortland Eble [g], Deolinda Flores [h], Thomas Gentzis [i], Paula Alexandra Gonçalves [h], João Graciano Mendonça Filho [j], Mária Hámor-Vidó [k], Iwona Jelonek [l], Kees Kommeren [m], Wayne Knowles [n], Jolanta Kus [o], Maria Mastalerz [p], Taíssa Rêgo Menezes [b], Jane Newman [q], Ioannis K. Oikonomopoulos [i], Mark Pawlewicz [r], Walter Pickel [s], Judith Potter [t], Paddy Ranasinghe [u], Harold Read [s], Julito Reyes [v], Genaro De La Rosa Rodriguez [w], Igor Viegas Alves Fernandes de Souza [b], Isabel Suárez-Ruiz [c], Ivana Sýkorová [x], Brett J. Valentine [a]

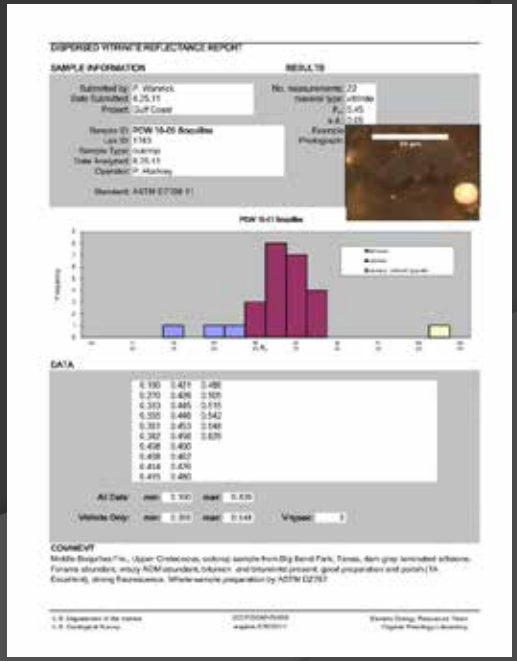
Thirty-one authors, twenty-two laboratories, fourteen countries

Important Findings

- Repeatability and reproducibility limits degraded consistently with increasing maturity and decreasing organic content (except for Type III sample)

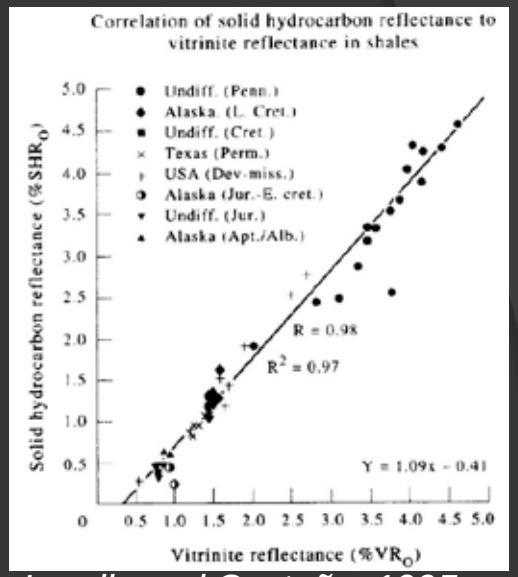
- Operators did not meet reporting requirements of D7708, indicating need for a template



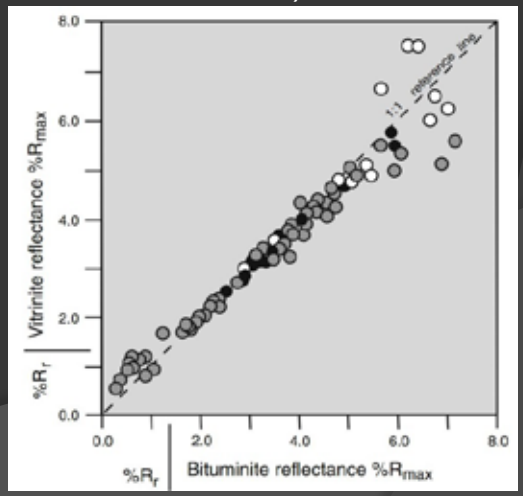


Important Findings

- No statistical difference between Ro from bitumen and vitrinite (contradictory to empirical conversions)
- Reproducibility was improved compared to historical exercises (summarized in Borrego, 2009)



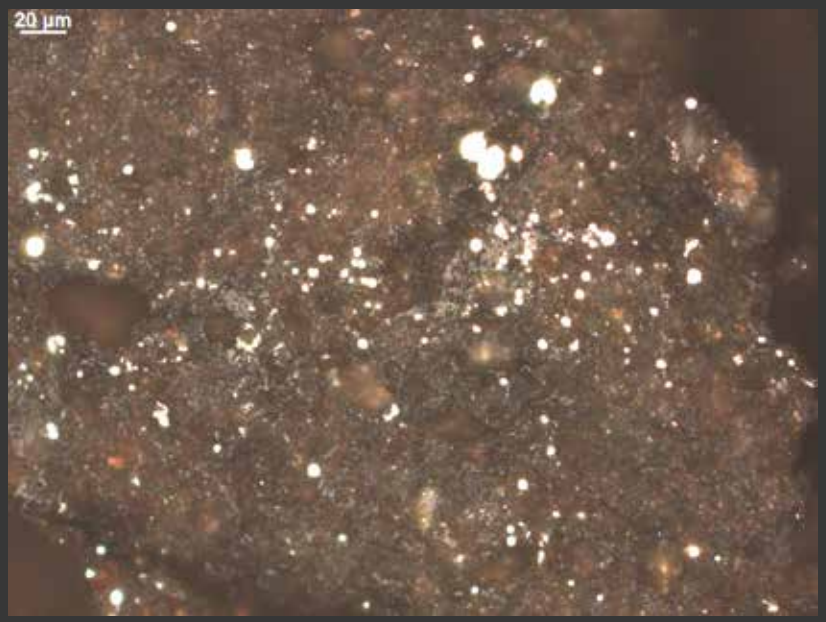
Landis and Castaño, 1995
Also Jacob, 1989 and Schoenherr et al, 2007

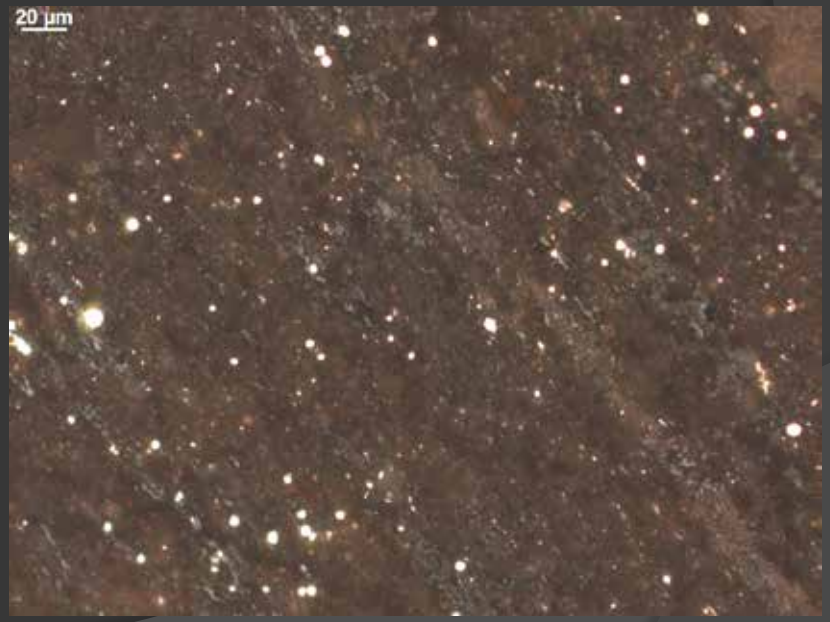


Mählmann and Frey, 2012

DISCUSSION

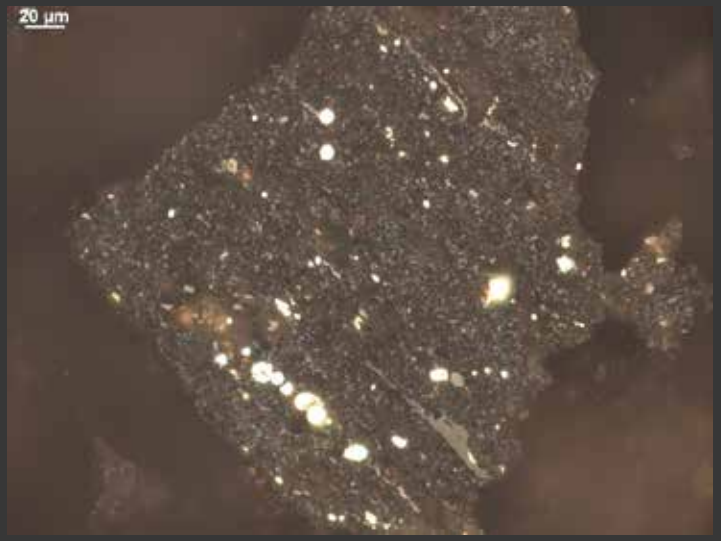
- Ideas for future work using ASTM D7708
  - Quarterly or biannual round robin with committed WG?
  - Send out similar samples – one with supporting information and one without to test the hypothesis that supporting information will improve accuracy of test?
  - **Use high maturity samples with high TOC – current USA shale gas/oil plays: e.g., Eagle Ford, Marcellus, Haynesville**

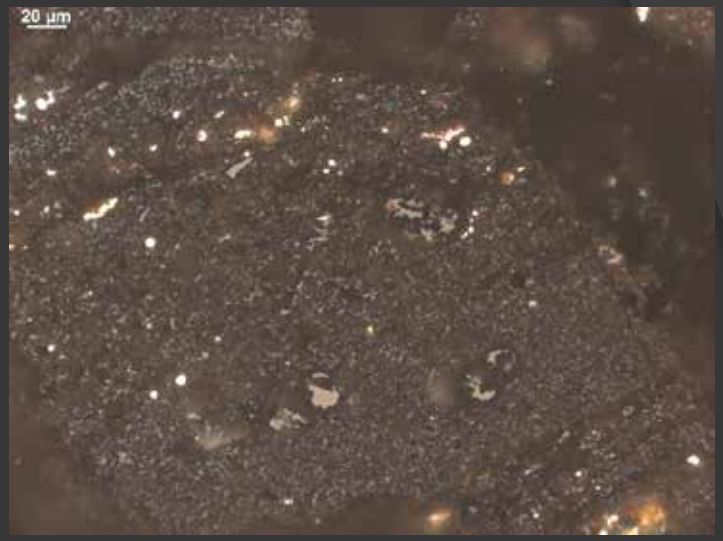




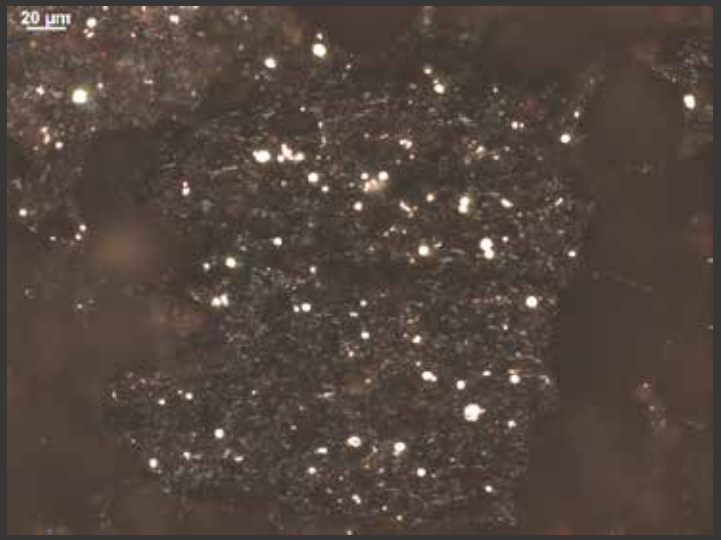
Jurassic shale from important North American shale gas play: TOC 2.66 wt.%, Ro > 1.0%

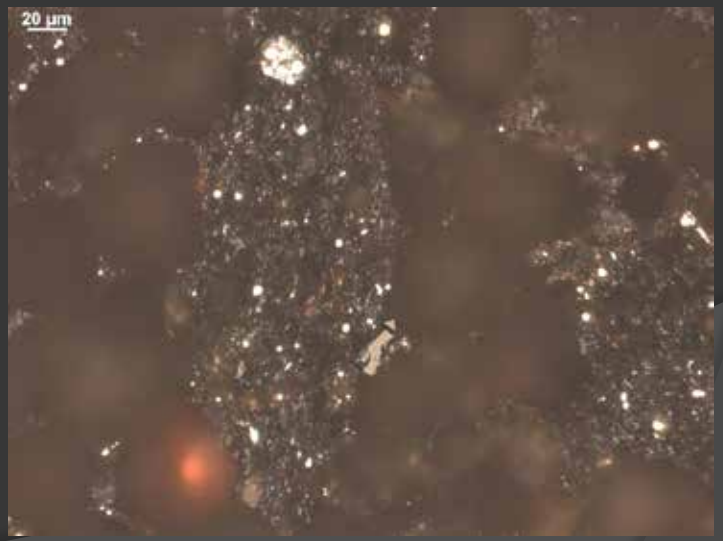
DISCUSSION





Upper Cretaceous shale from important North American shale liquids play: TOC 5.07 wt.%, Ro > 1.0%





Devonian shale from important North American shale gas play: TOC ~2-3 wt.%, Ro > 1.0%

More Discussion

- Using several samples from NA or other shale gas/liquids plays with ‘name recognition’ will generate high impact, additional samples can be obtained from State Geological Surveys
  - full paper for laypersons in Oil & Gas Journal circulation >100,000
  - Generate database of shale Ro information with goals to update ASTM D7708 precision, and eventual follow-on paper to JMPG 2014
- Explore bitumen vs. vitrinite reflectance calibration in more detail? Lacustrine vs. marine?
- How to update the WG webpages!!!!!????
- Other ideas?

Acknowledgments

- Participants in the DOMVR survey of 2009
- All members of the writing committee for ASTM D7708
- Participants in the 2012-2013 ASTM D7708 round robin and contributors to the JMPG paper
- All Commission II members

- **THANKS ICCP!**