# ICCP Working Group
# Identification of Primary Vitrinite in Shale
# 2016 Report

*Paul C. Hackley - U.S. Geological Survey, Reston, Virginia, USA*

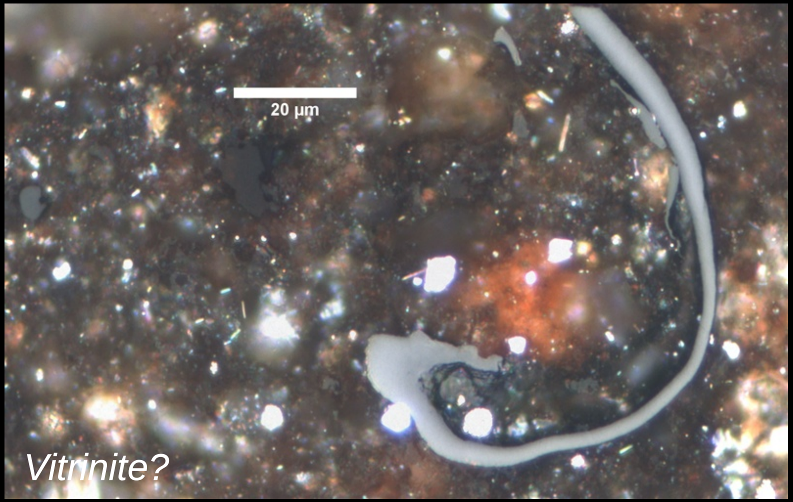
*Presented for ICCP, September, 2016*

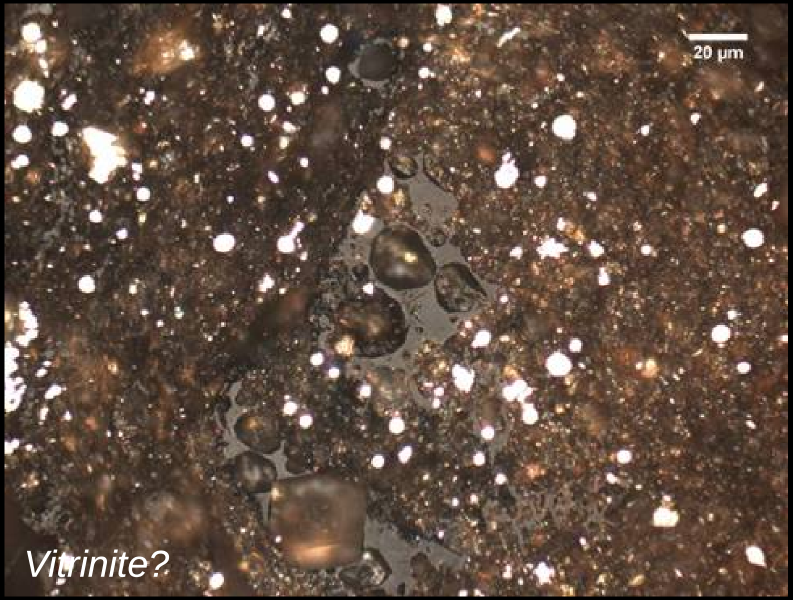
# Acknowledgments


- Owen Scholl, Javin Hatcherian, Brett Valentine (USGS)
- Thomas Gentzis, Humberto Carvajal (Core Laboratories)
- Sample contributors: James Donnelly, Steve Ruppel (BEG), Terry Huber, John Repetski (USGS)
- Participants in the ICCP interlaboratory study
- USGS Energy Resources Program

# Outline

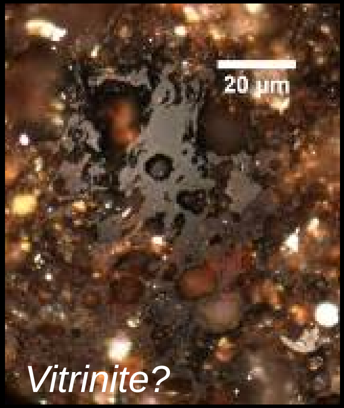
- Problem to be solved
- History of the ICCP working group
- Findings and products to-date
- 2015-2016 exercise and results
- Summary



# Objective of the Working Group

- Provide guidelines for identification of the primary vitrinite population in dispersed organic matter

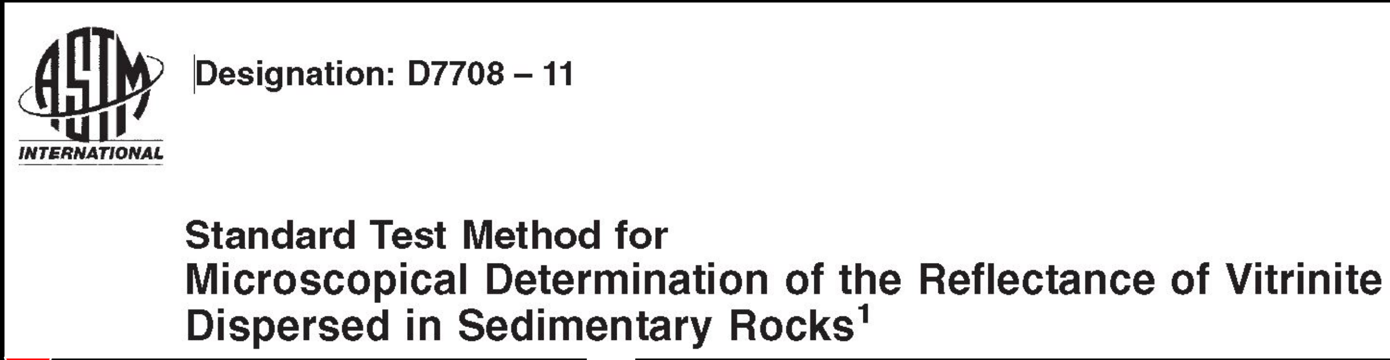




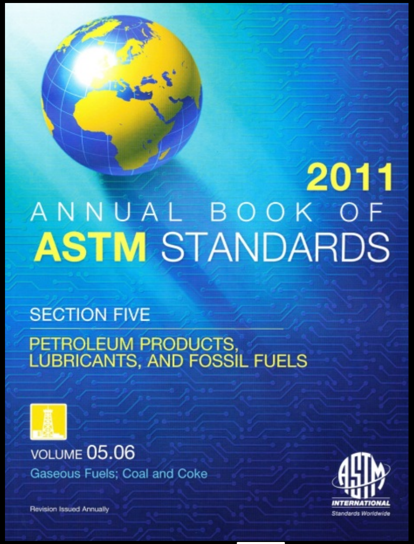



# Identification of primary vitrinite: History of the working group

- Proposed by Angeles Borrego 2008 Oviedo ICCP meeting
- Survey of DOMVR analysis presented 2009 Gramado ICCP meeting, ICCP News No. 48
- ASTM standard D7708 for DOMVR in 2011 Annual Book of ASTM Standards


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.

- Test of ASTM D7708 reproducibility via interlaboratory study in 2012-2013
- 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, 2015

***Porto 2011*** ***Beijing 2012*** ***Sosnowiec 2013*** ***Kolkata 2014*** ***Potsdam 2015***

# Results of the 2012-2013 interlaboratory study

Marine and Petroleum Geology 59 (2015) 22–34

Contents lists available at ScienceDirect

Marine and Petroleum Geology

journal homepage: www.elsevier.com/locate/marpetgeo

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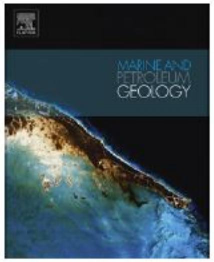



Research paper

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

CrossMark

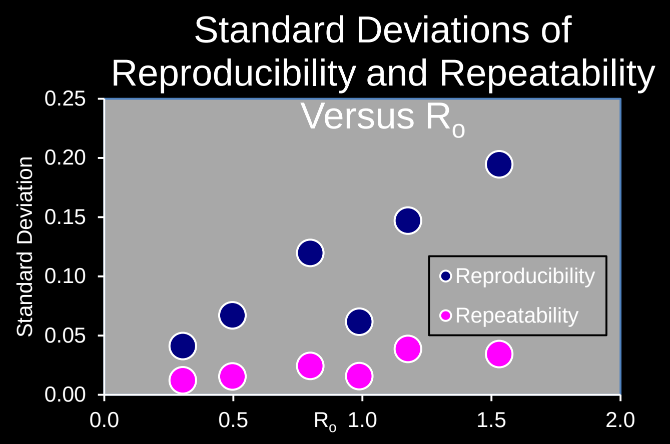
Paul C. Hackley [a,*], Carla Viviane Araujo [b], Angeles G. Borrego [c], Antonis Bouzinos [d], Brian J. Cardott [e], Alan C. Cook [f,1], Cortland Eble [g], Deolinda Flores [h], Thomas Gentzis [i], Paula Alexandra Gonçalves [h], João Graciano Mendonça Filho [j], Mária Hámor-Vidó [k], Iwona Jelonek [l], 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]

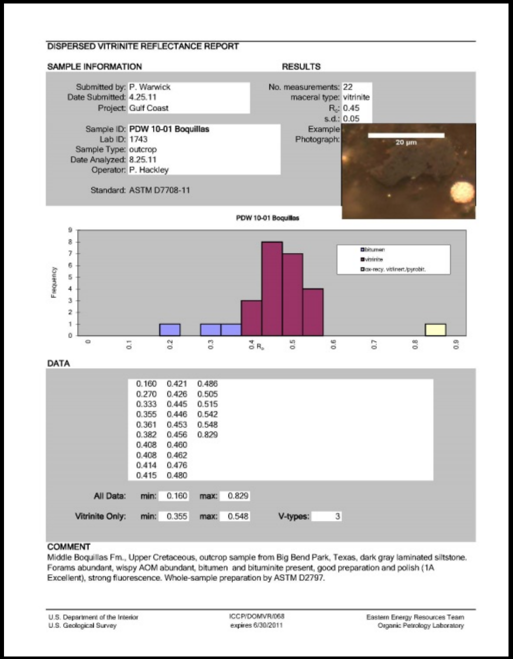
**Thirty-one authors, twenty-two laboratories, fourteen countries**

# Important Findings

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

- Operators did not meet reporting requirements, indicating need for a template to improve data quality



DISPERSED VITRINITE REFLECTANCE REPORT

SAMPLE INFORMATION

Submitted by: P. Warwick
Date Submitted: 4.25.11
Project: Gulf Coast

Sample ID: **PDW 10-01 Boquillas**
Lab ID: 1743
Sample Type: outcrop
Date Analyzed: 8.25.11
Operator: P. Hackley

Standard: ASTM D7708-11

RESULTS

No. measurements: 22
maceral type: vitrinite
$R_o$: 0.45
s.d.: 0.05
Example Photograph:



DATA

| | | |
|---|---|---|
| 0.160 | 0.421 | 0.486 |
| 0.270 | 0.426 | 0.505 |
| 0.333 | 0.445 | 0.515 |
| 0.355 | 0.446 | 0.542 |
| 0.361 | 0.453 | 0.548 |
| 0.382 | 0.456 | 0.829 |
| 0.408 | 0.460 | |
| 0.408 | 0.462 | |
| 0.414 | 0.476 | |
| 0.415 | 0.480 | |

All Data: min: 0.160 max: 0.829

Vitrinite Only: min: 0.355 max: 0.548 V-types: 3

COMMENT

Middle Boquillas Fm., Upper Cretaceous, outcrop sample from Big Bend Park, Texas, dark gray laminated siltstone. Forams abundant, wispy AOM abundant, bitumen and bituminite present, good preparation and polish (1A Excellent), strong fluorescence. Whole-sample preparation by ASTM D2797.

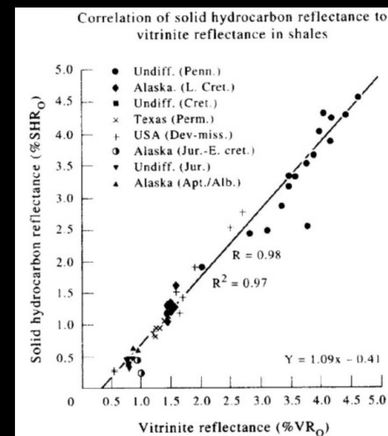
U.S. Department of the Interior
U.S. Geological Survey

ICCP/DOMVR/068
expires 6/30/2011

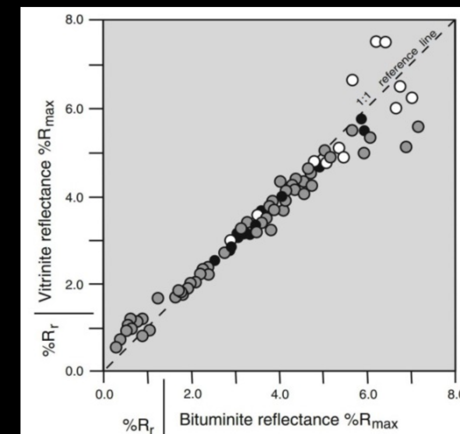
Eastern Energy Resources Team
Organic Petrology Laboratory

# Important Findings

- No statistical difference between Ro from bitumen and vitrinite (contradictory to empirical conversions schemes)




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



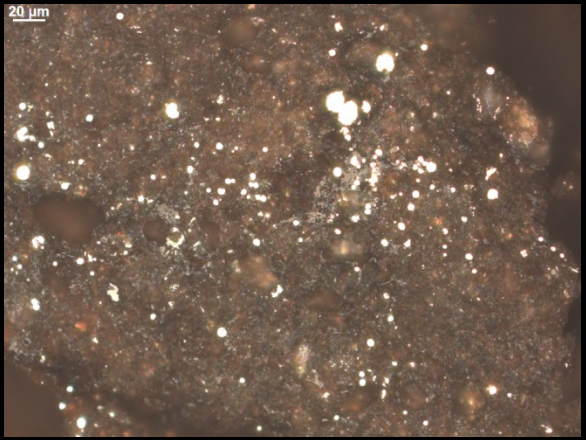

*Mählmann and Frey, 2012*

- Reproducibility was improved compared to historical exercises (summarized in Borrego, 2009)

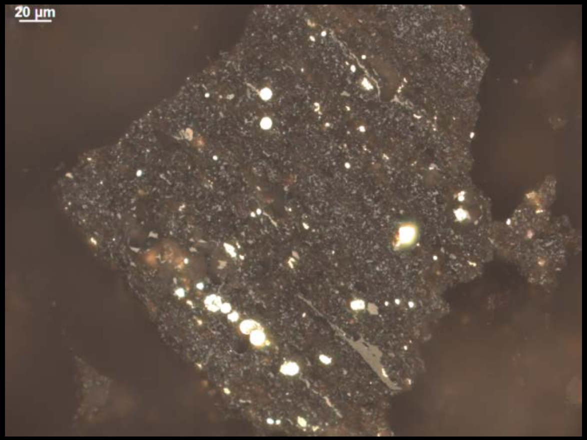
- Poor reproducibility for high maturity sample (R=0.54 for 1.5% Ro) – because low TOC(?)

# Proposal for 2015-2016

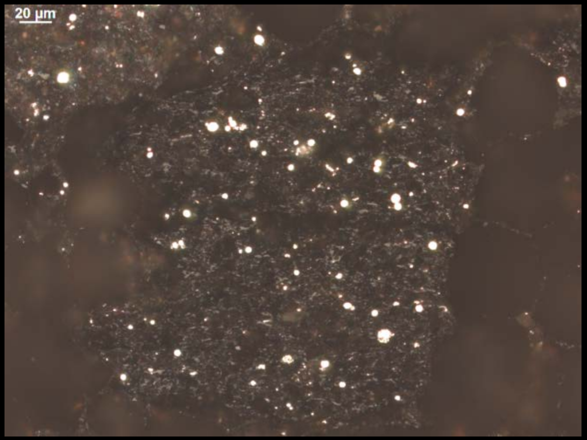
- Use high maturity samples with high TOC – current USA shale gas/tight oil plays: e.g., Eagle Ford, Marcellus, Haynesville, Barnett, Bakken
- Using several samples from NA with 'name recognition' will generate high impact result/paper
- Round robin with 6 samples over 2015-2016




*Jurassic:TOC 2.66 wt.%, Ro > 1.0%*



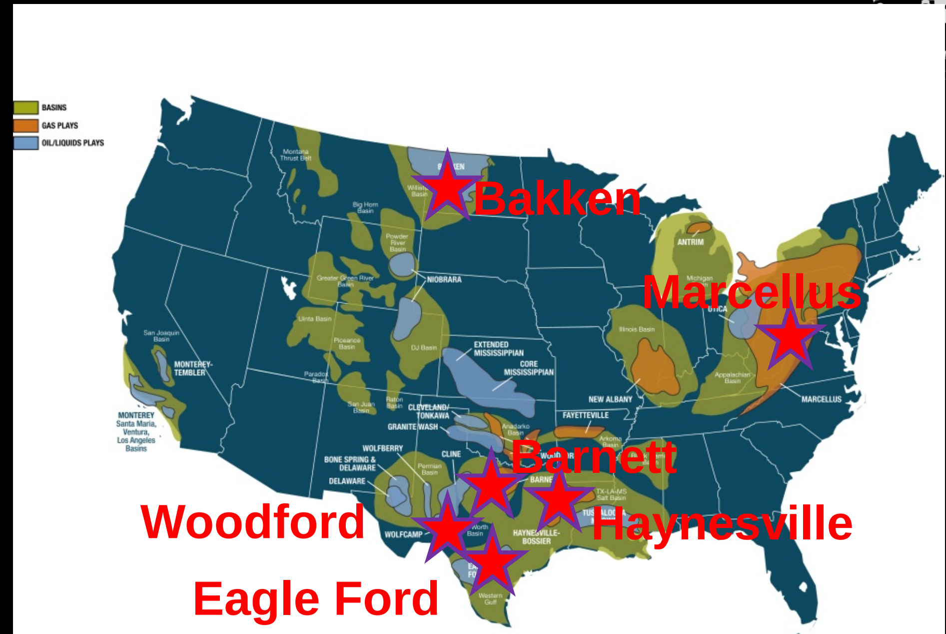

*Upper Cretaceous: TOC 5.07 wt.%, Ro > 1.0%*

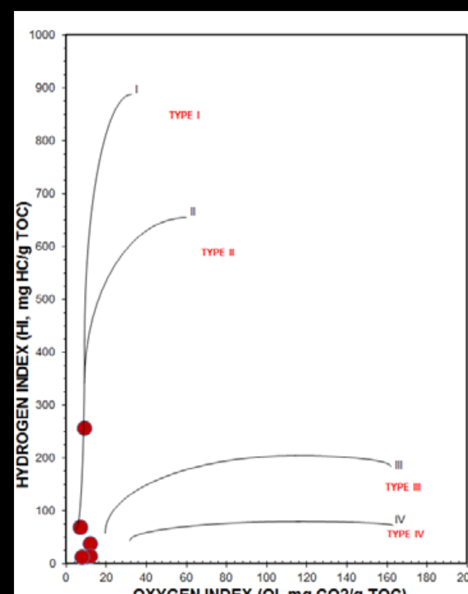


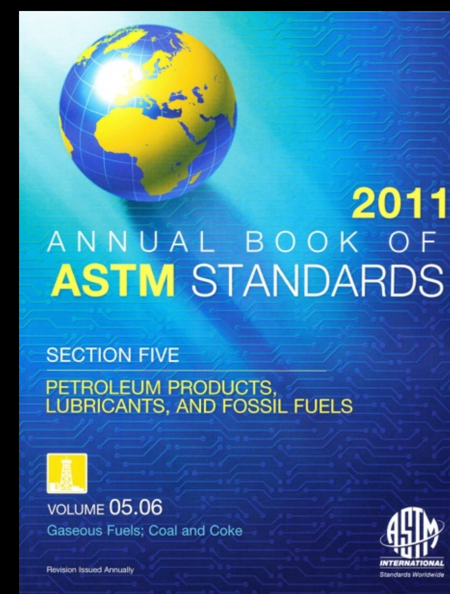

*Devonian: TOC 5.17 wt.%, Ro > 1.0%*

# Samples:

- From core
- High maturity: peak oil, condensate/wet gas, dry gas
- Organic-rich (2.7-11.5% TOC)
- From North America
- 6 shale gas and tight oil plays with ‘name recognition’
- Typical of the shale
- Distributed as crushed rock
- Instructions to follow D7708

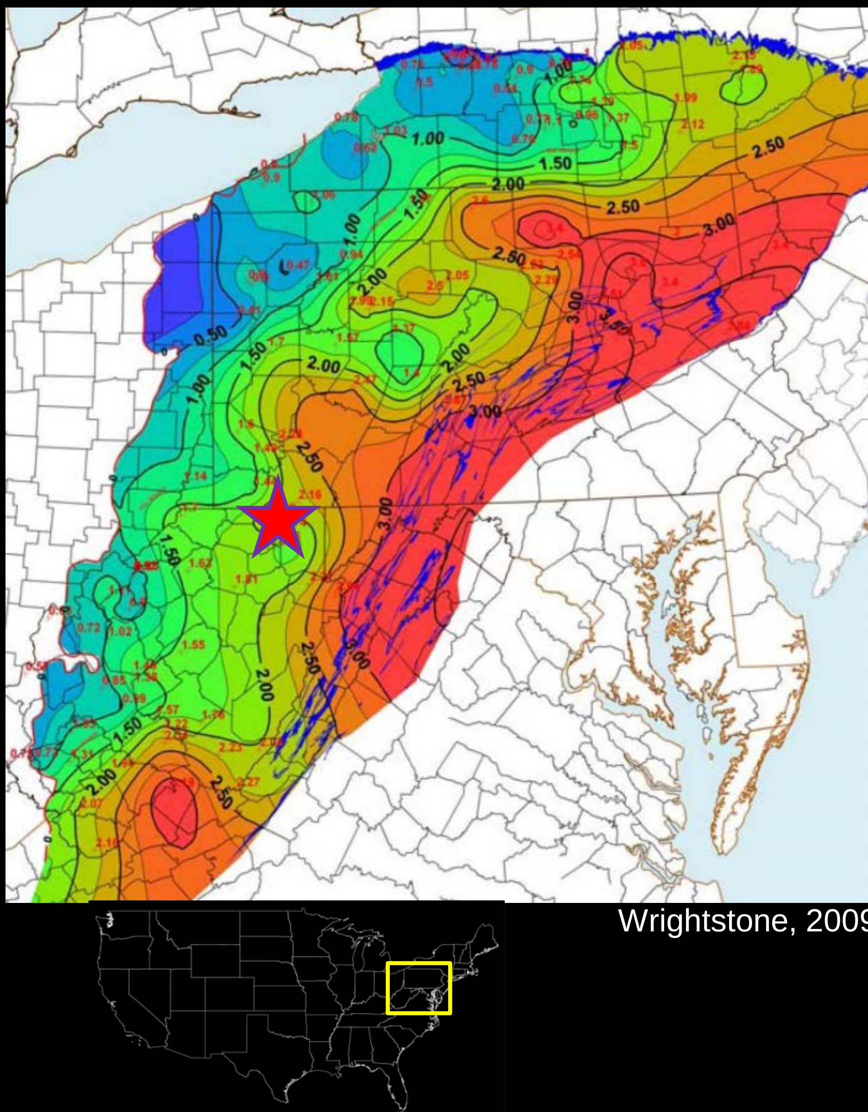


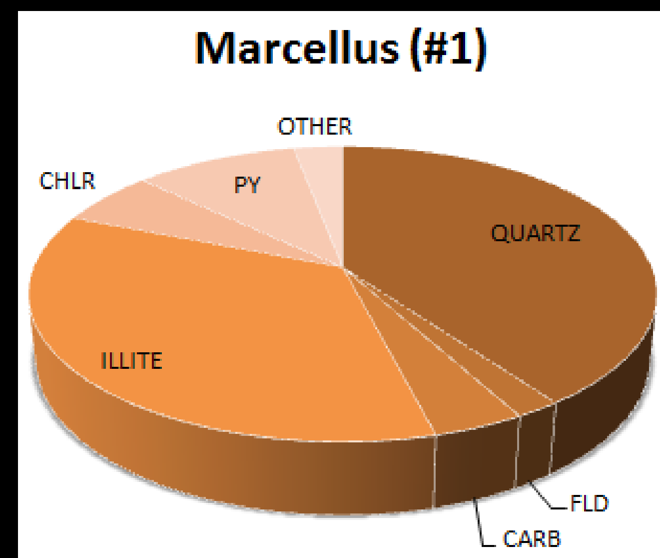

*Presented for ICCP, September, 2016*

# Samples: Marcellus (#1)

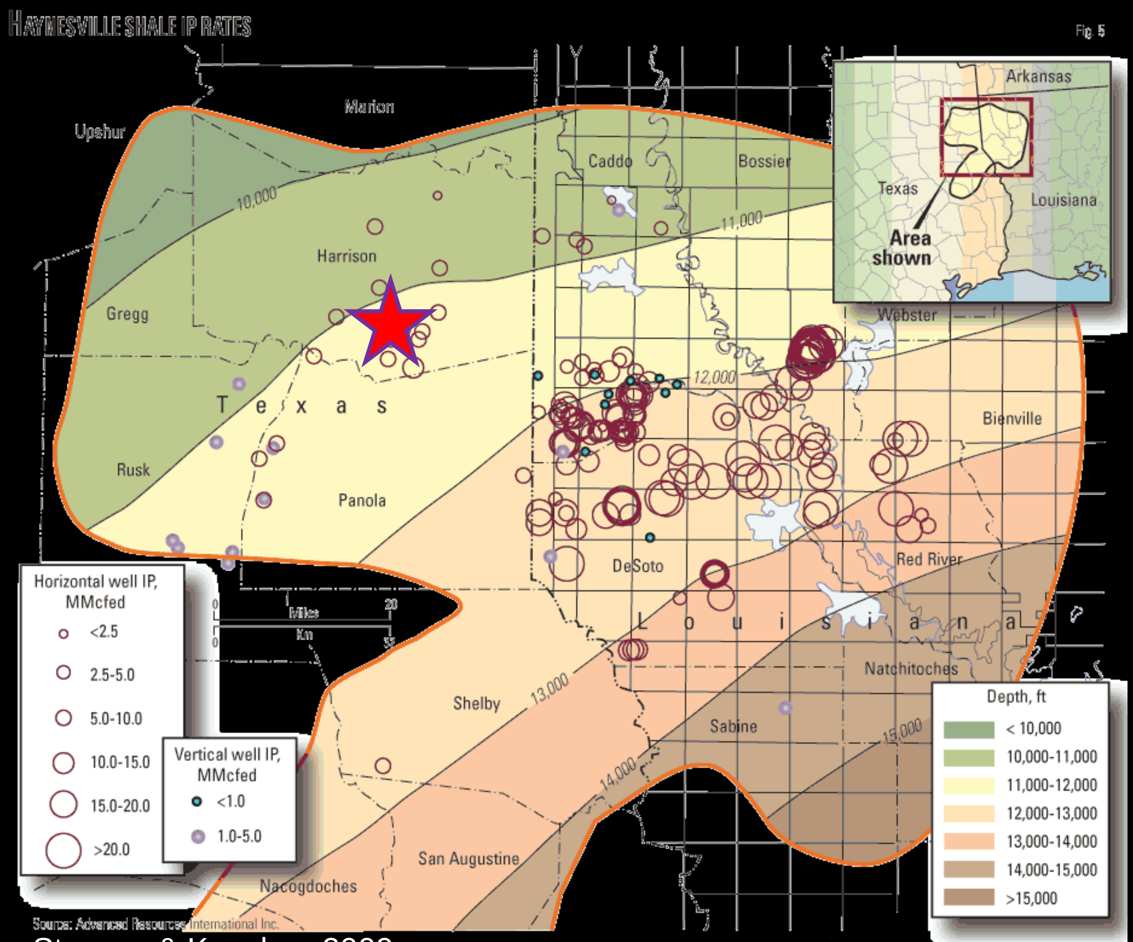



Wrightstone, 2009

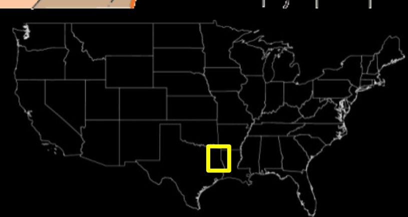
- Appalachian Basin, West Virginia
- Middle Devonian
- Dry gas
- 5.2% TOC



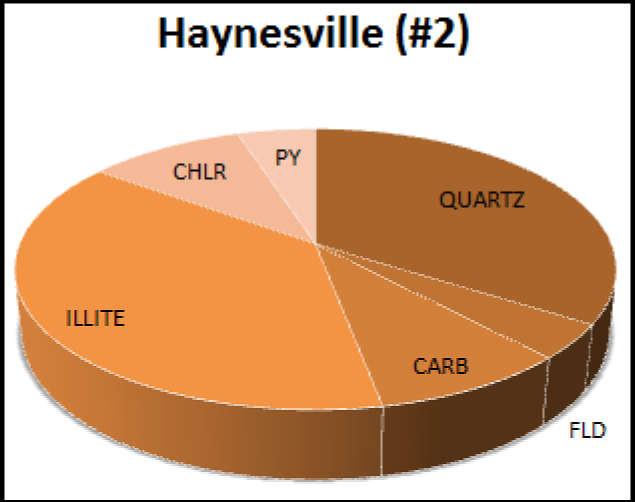
# Samples: Haynesville (#2)



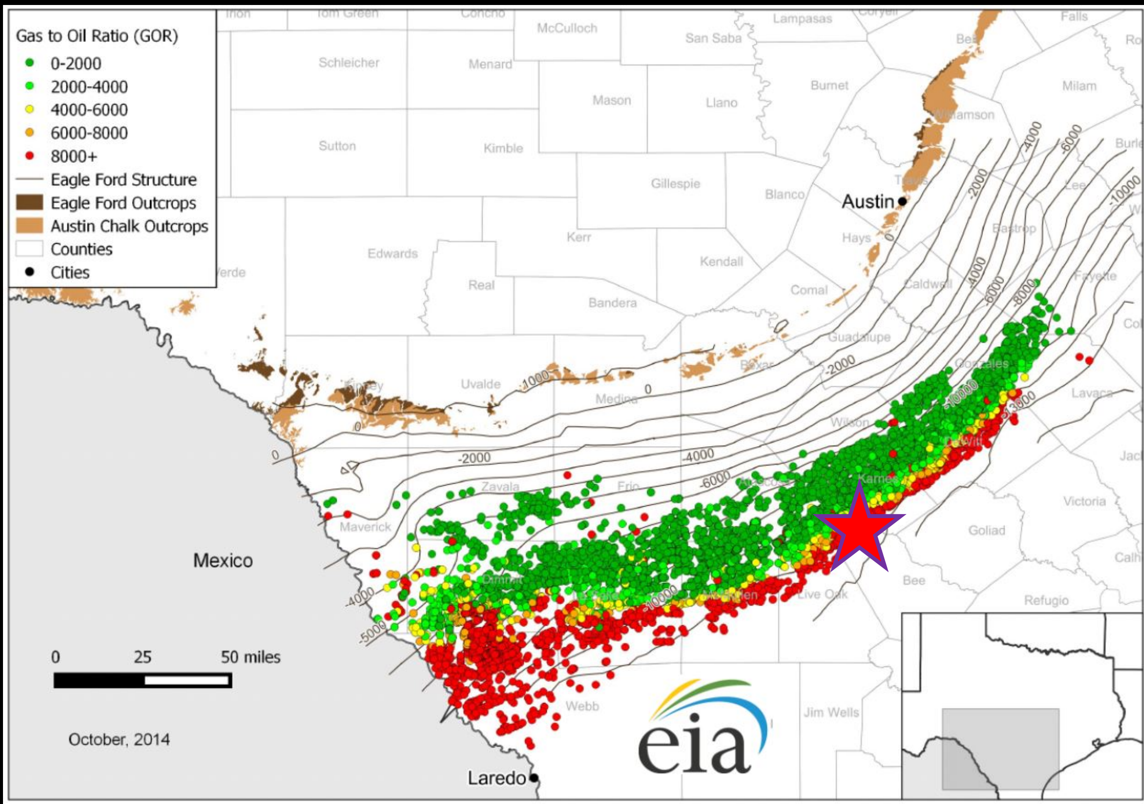

Stevens & Kuuskra, 2009



- Gulf of Mexico Basin, Texas
- Jurassic
- Dry gas
- 2.7% TOC

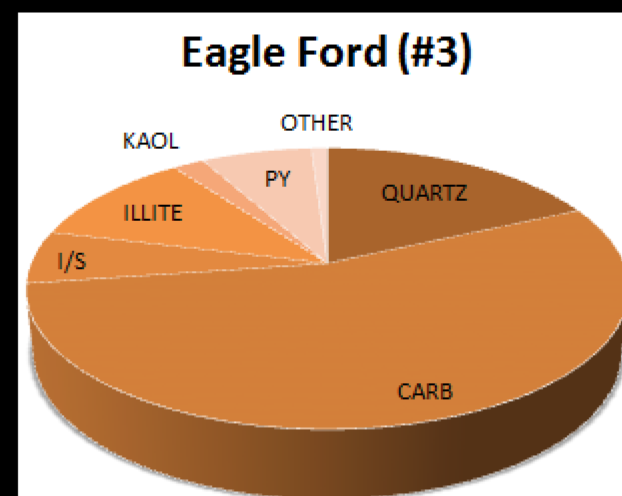


# Samples: Eagle Ford (#3)




Energy Information Administration, 2014

- Gulf of Mexico Basin, Texas
- Upper Cretaceous
- Dry gas
- 5.1% TOC

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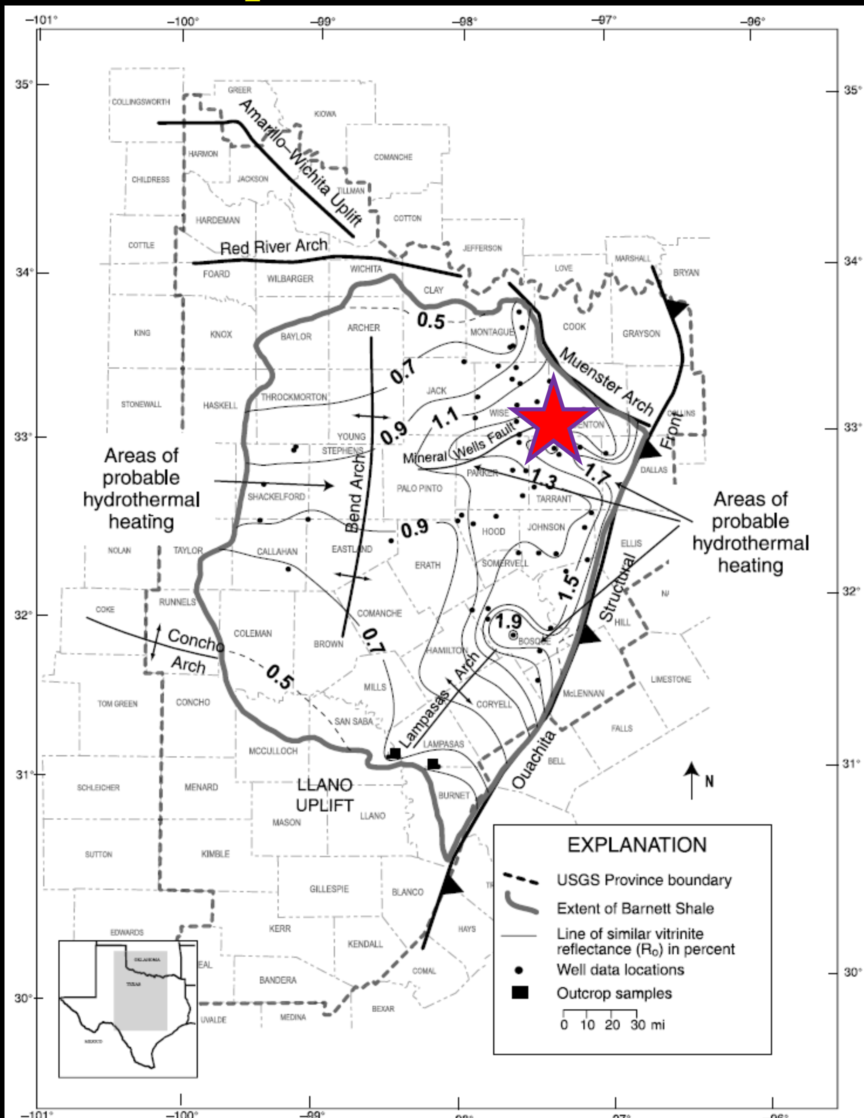
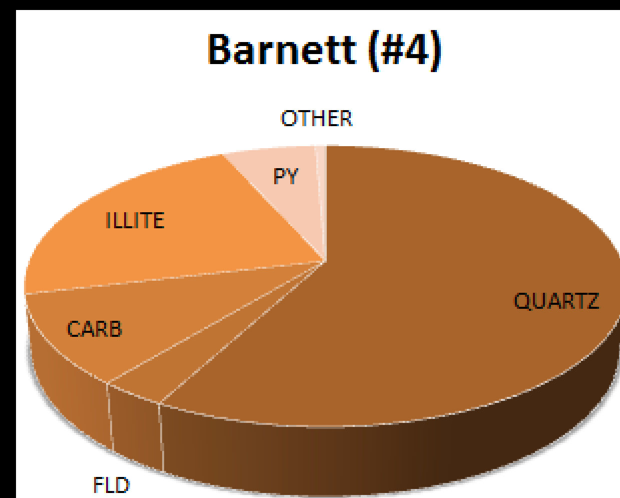
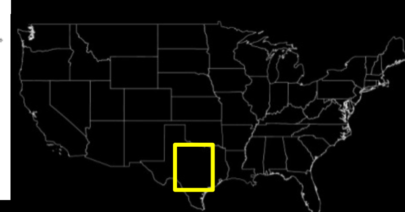
# Samples: Barnett (#4)



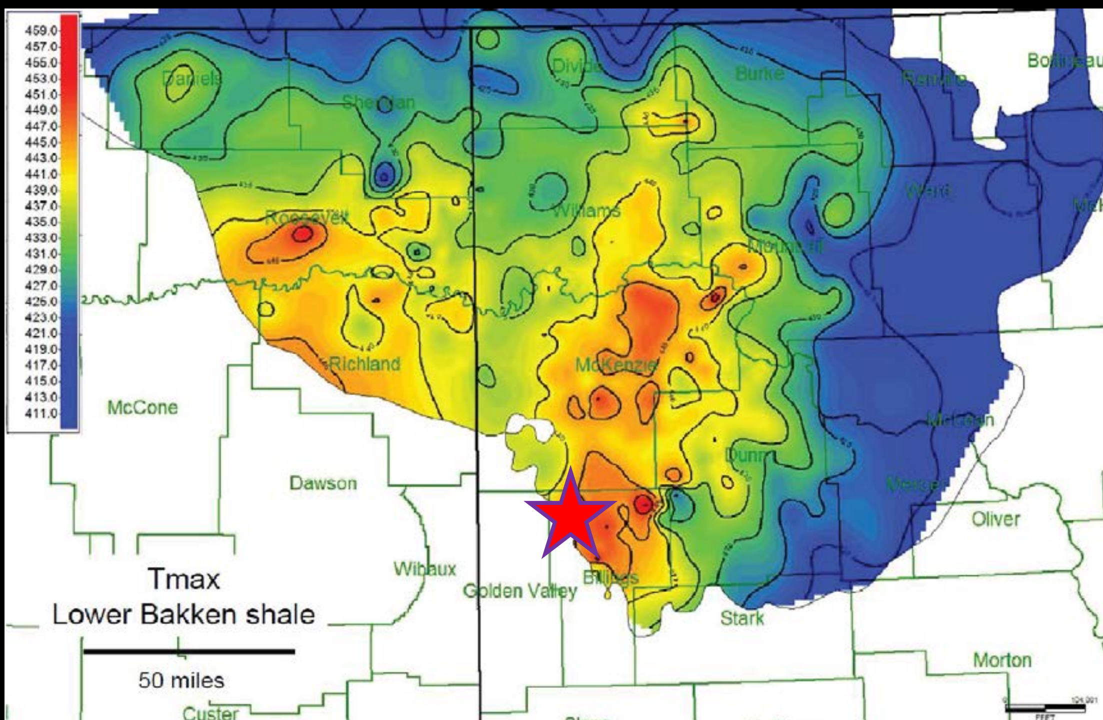

Pollastro et al. 2007

- Ft. Worth Basin, Texas
- Mississippian
- Dry gas
- 3.0% TOC



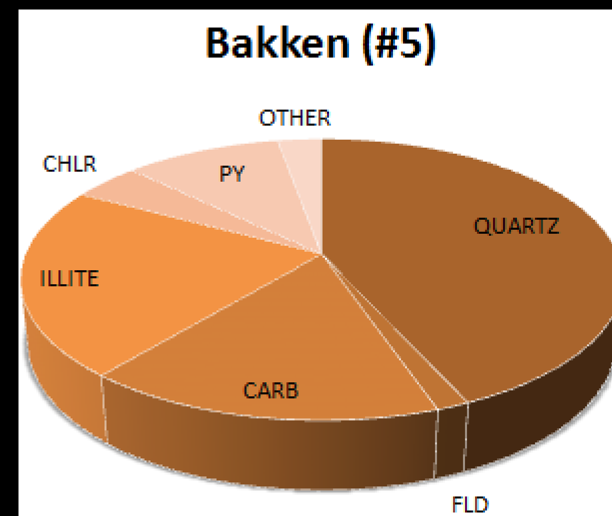



# Samples: Bakken (#5)

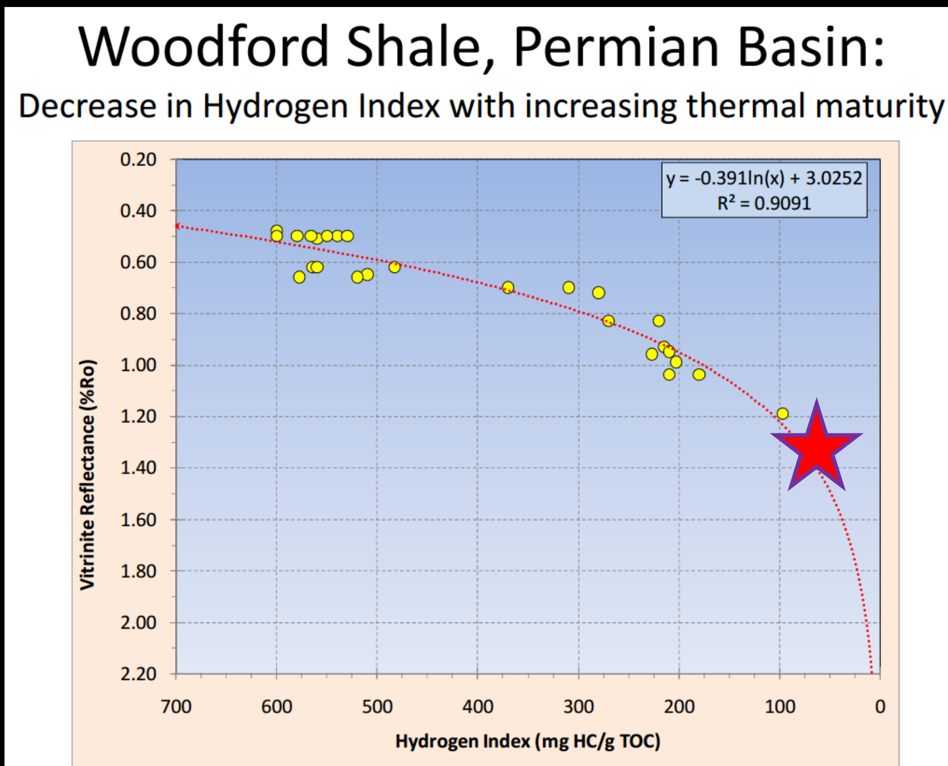



Jin and Sonnenberg, 2012

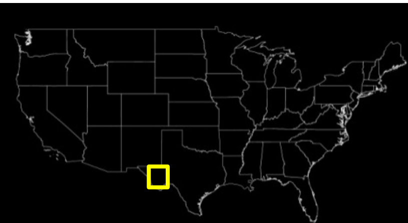
- Williston Basin, North Dakota
- Devonian-Mississippian
- Peak oil
- 10.6% TOC



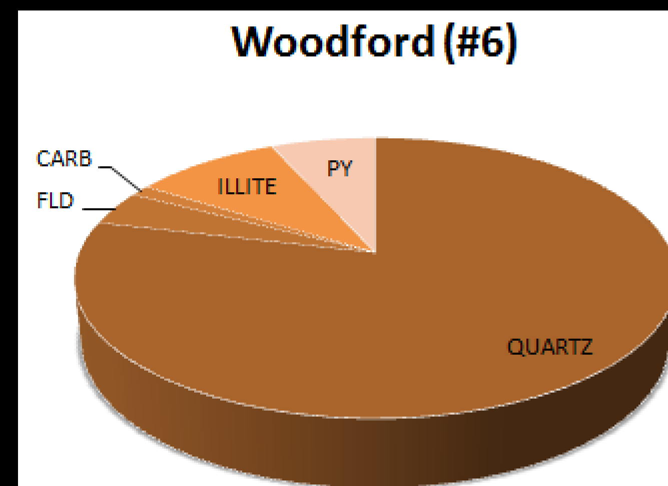
# Samples: Woodford (#6)




Jarvie, 2008



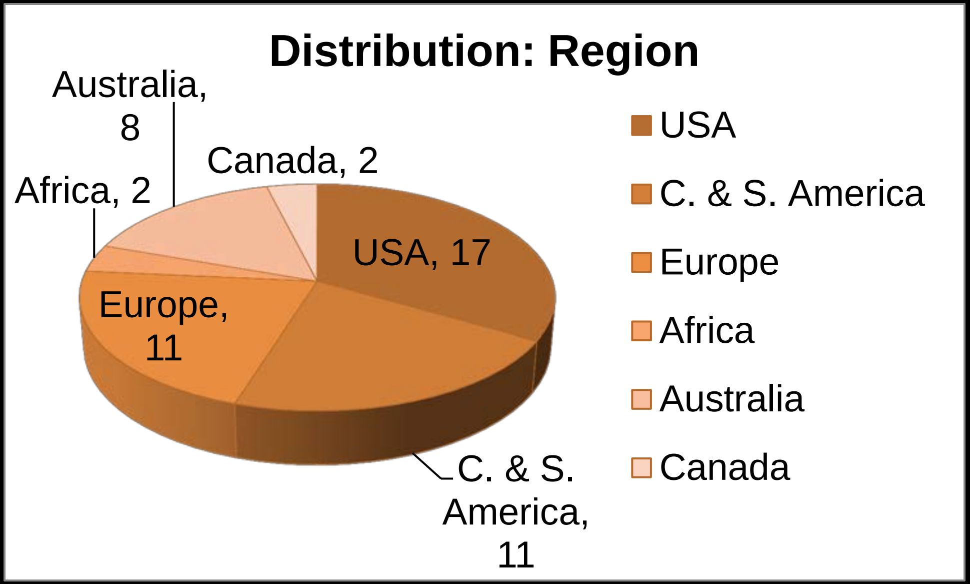
- Permian Basin (Delaware), Texas
- Devonian-Mississippian
- Condensate/wet gas
- 11.5% TOC



# Distribution of Samples

- Samples posted January 2016

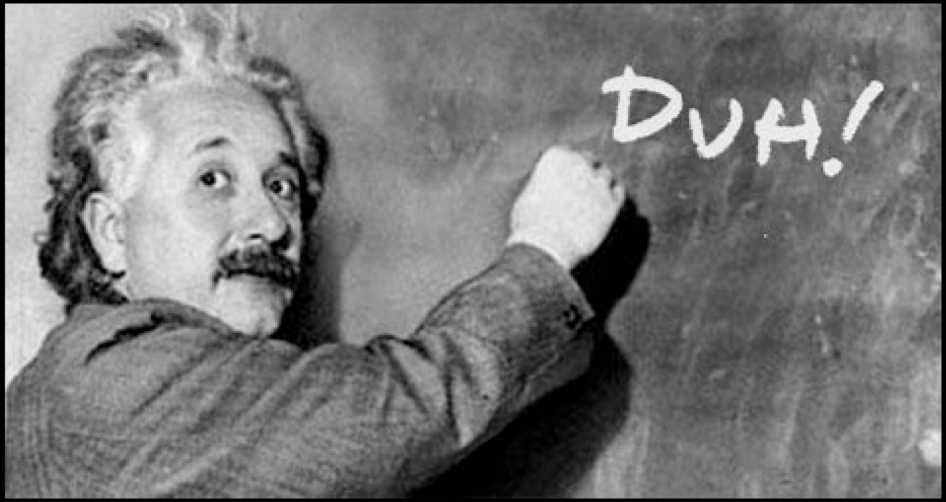
- 51 Petrographers
- 40 Laboratories
- 14 Countries
- 6 Continents



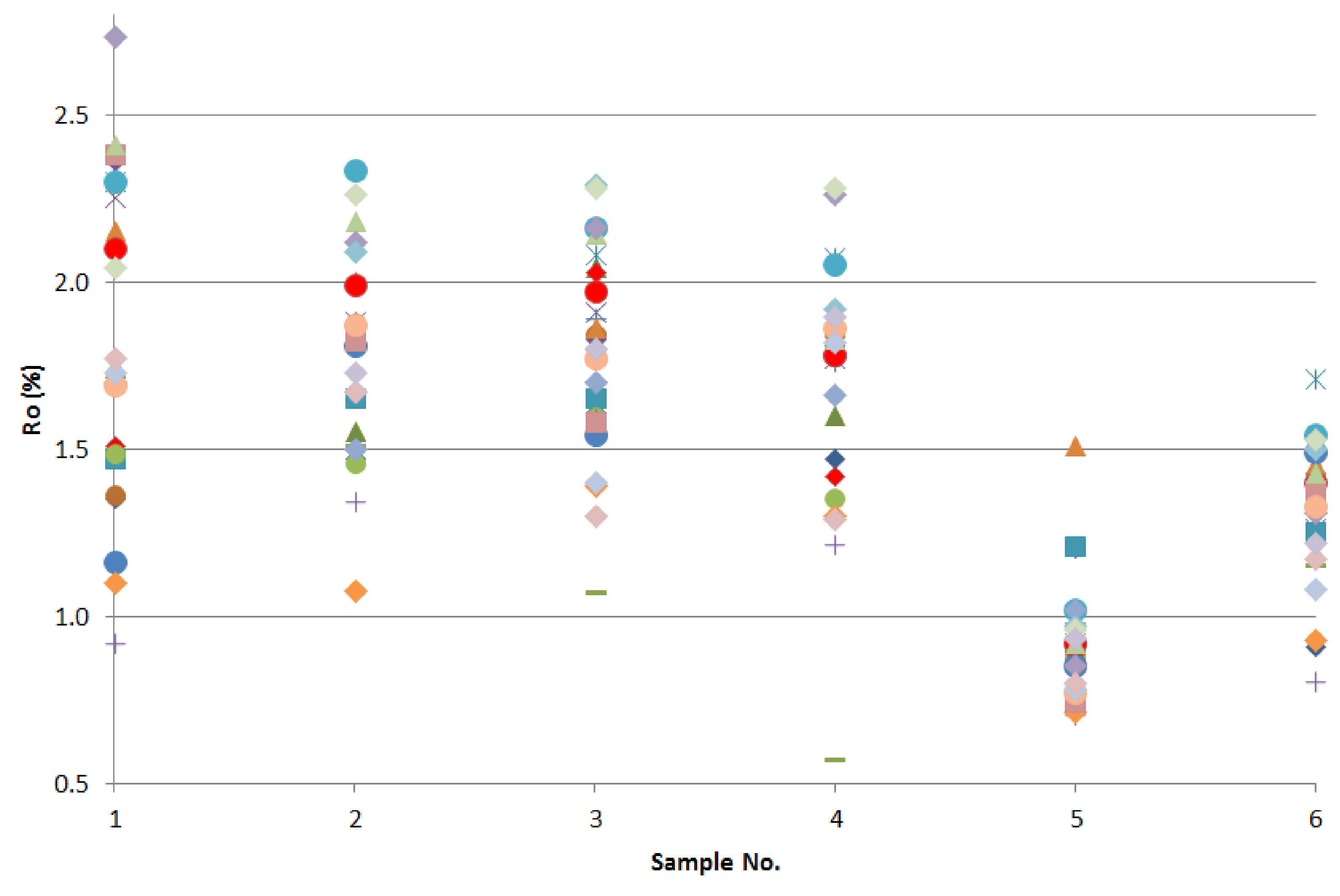

- Instructions: **follow ASTM D7708!**

# Results(?)

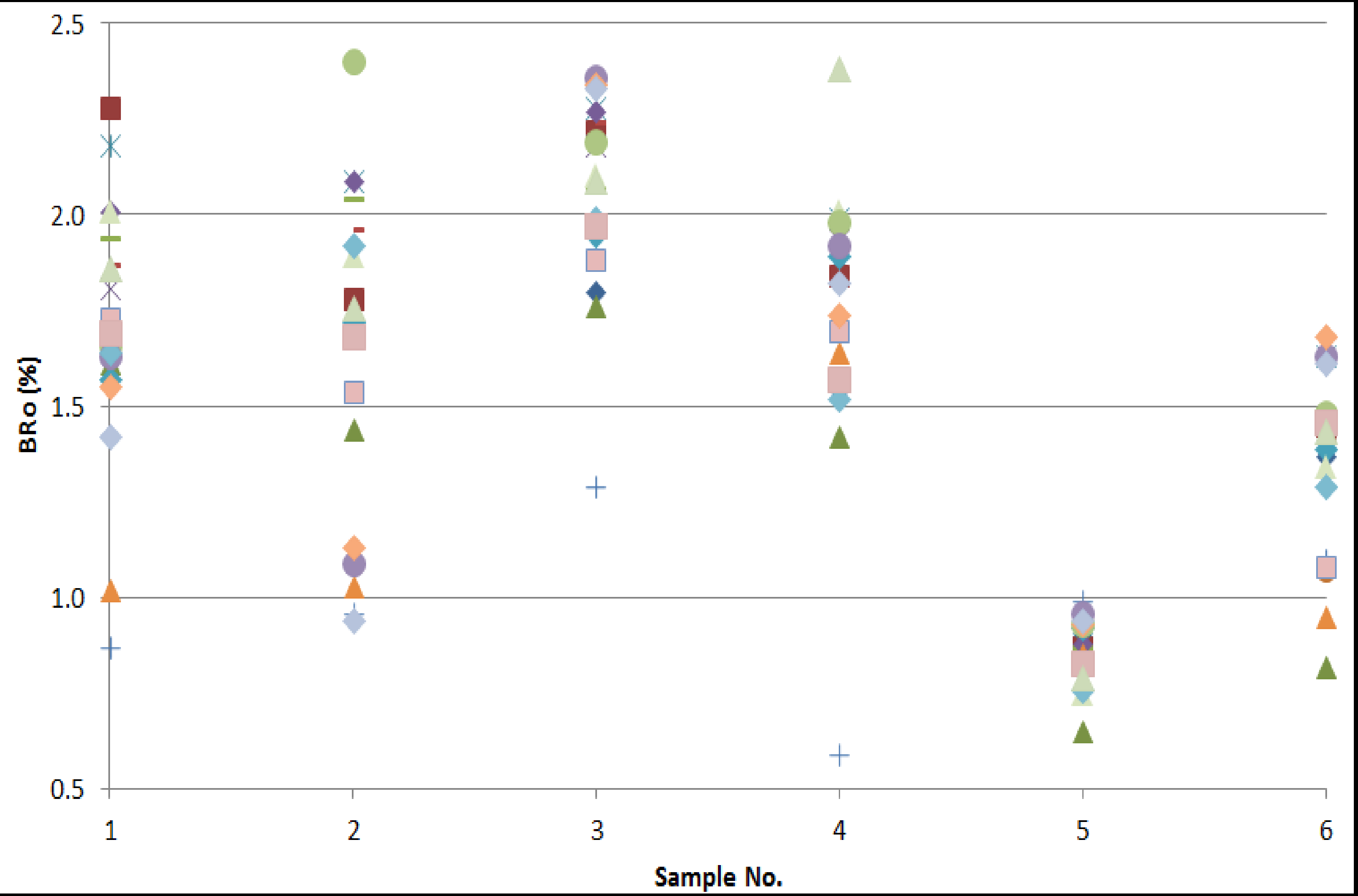
- Two petrographers returned data that could not be included (too few measurements)
- One petrographer could not receive samples (a bribe was extended from customs)
- One petrographer sent results that could not have been obtained from the samples sent to them (data are included)



# Results: vitrinite (n=24-27)

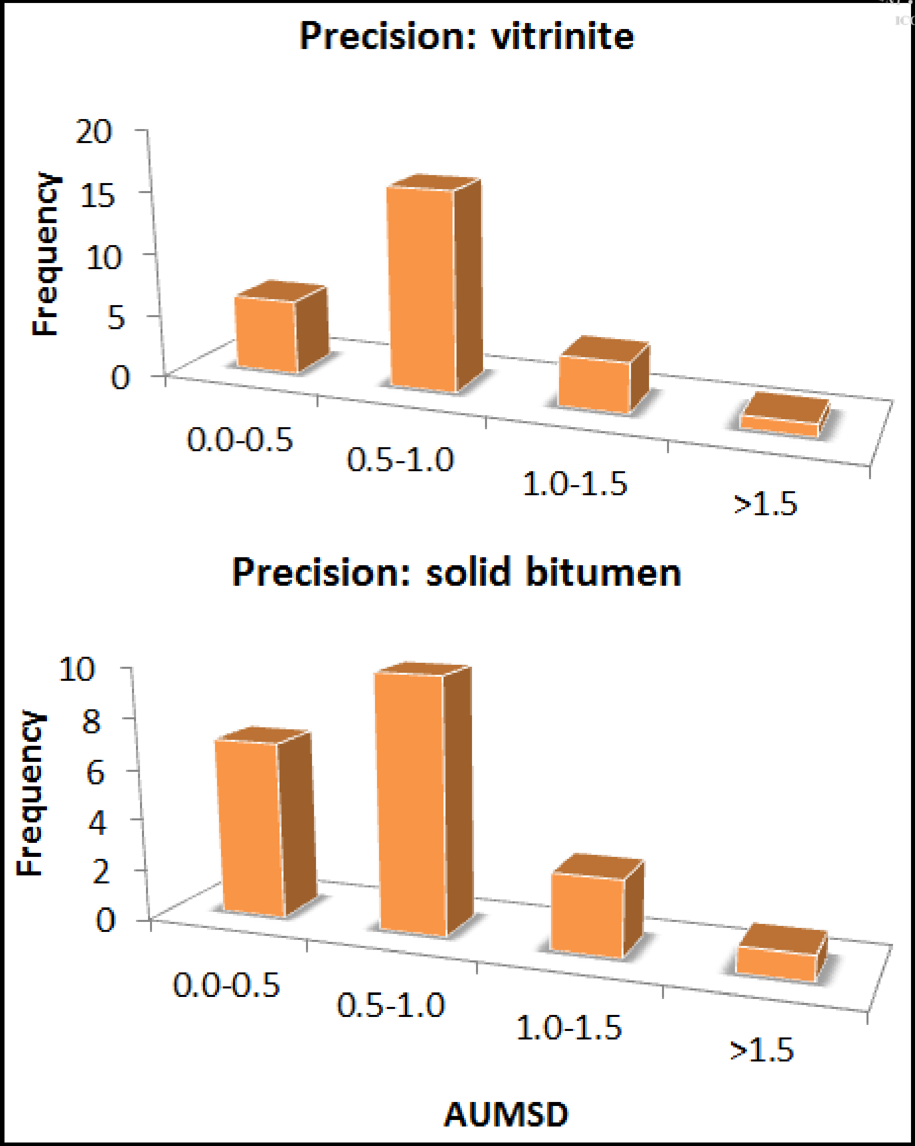


# Results: solid bitumen (n=18-20)

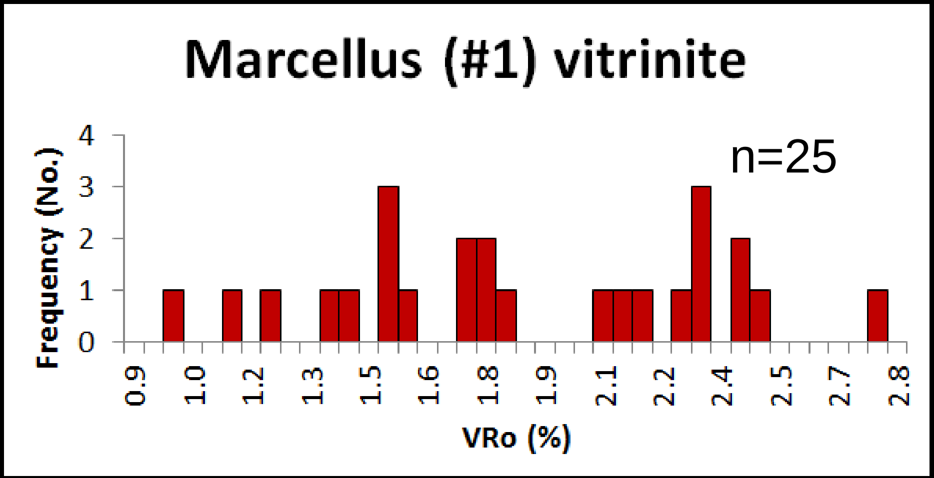


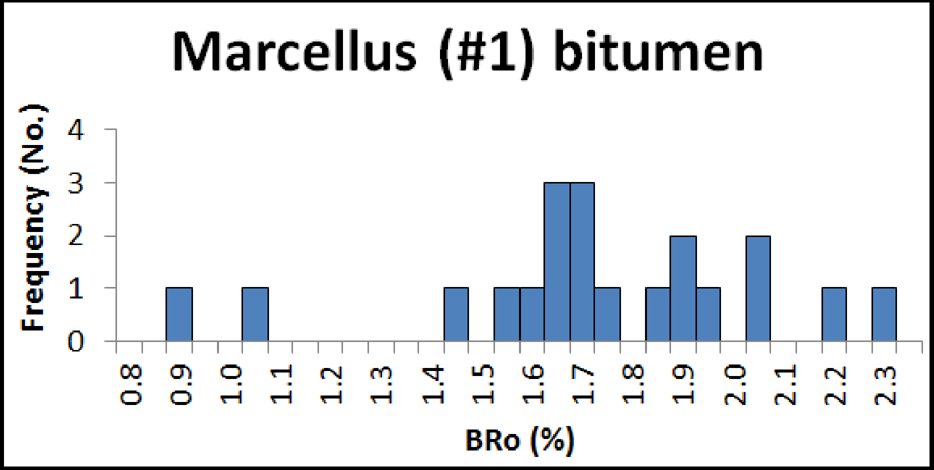
# Results

- 36 petrographers up until September 9th
- 71% (36 of 51) sample recipients returned results
- 28 petrographers held ICCP accreditation in DOMVR
- Accredited vs. non-accredited petrographers performed similarly
- 1 petrographer had AUMSD >1.5 for vitrinite
- 1 petrographer (a different one) had AUMSD >1.5 for solid bitumen
- Most had moderate to high precision



# Results: Marcellus (#1)



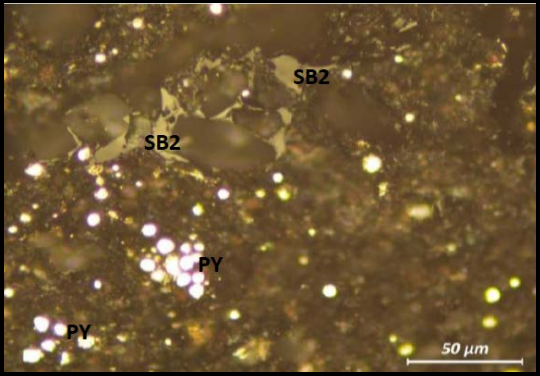




Vitrinite

- Mean: 1.83
- GSD: 0.48
- Skew: -0.07
- Kurt: -0.96
- R: 1.35


Solid Bitumen

- Mean: 1.70
- GSD: 0.34
- Skew: -0.81
- Kurt: -1.38
- R: 0.95

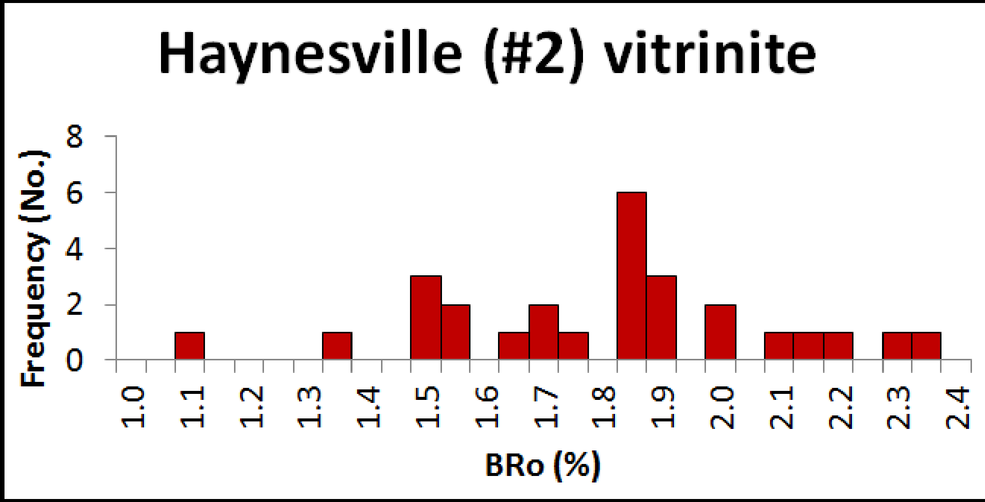



+ SK *tails to the right*
+ KT acute *peak* and fatter tails

- SK *tails to the left*
- *KT lower, wider peak and thinner tails*

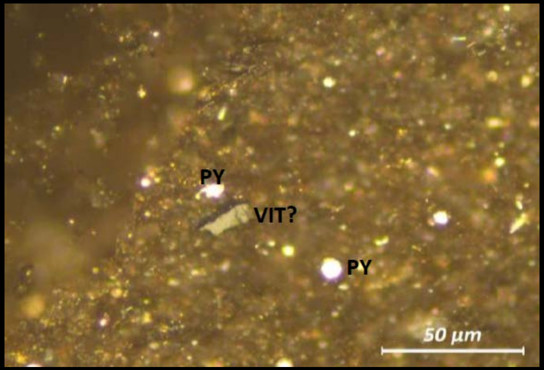
*Presented for ICCP, September, 2016*

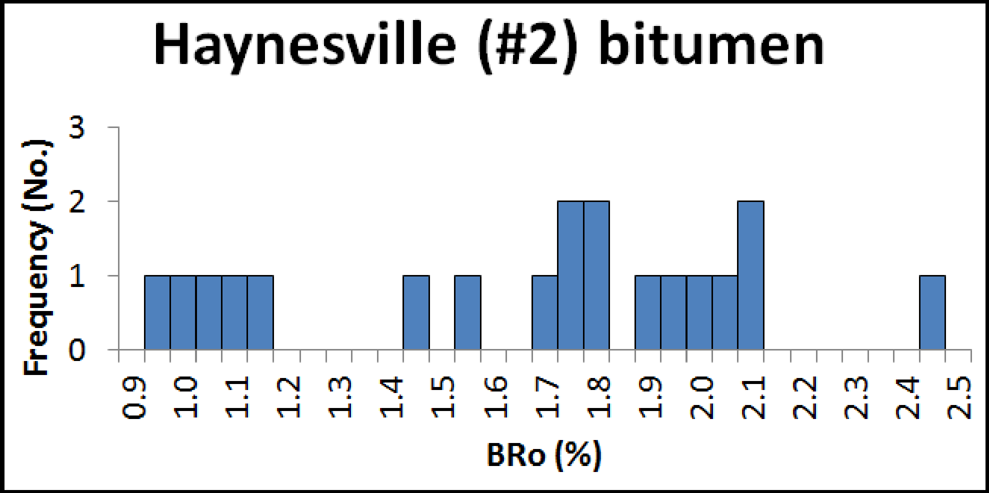
# Results: Haynesville (#2)




<u>Vitrinite</u>

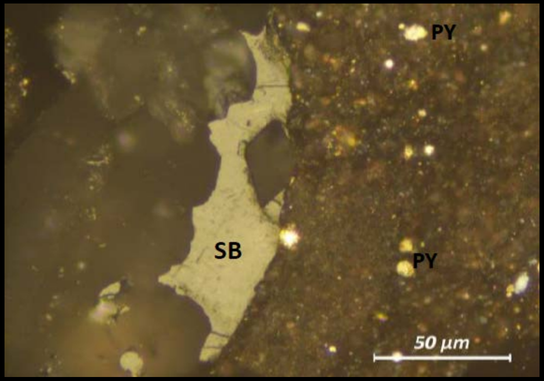
- Mean: 1.79
- GSD: 0.29
- Skew: -0.28
- Kurt: 0.18
- R: 0.81




<u>Solid Bitumen</u>

- Mean: 1.64
- GSD: 0.43
- Skew: -0.34
- Kurt: -0.88
- R: 1.21

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- *KT lower, wider peak and thinner tails*

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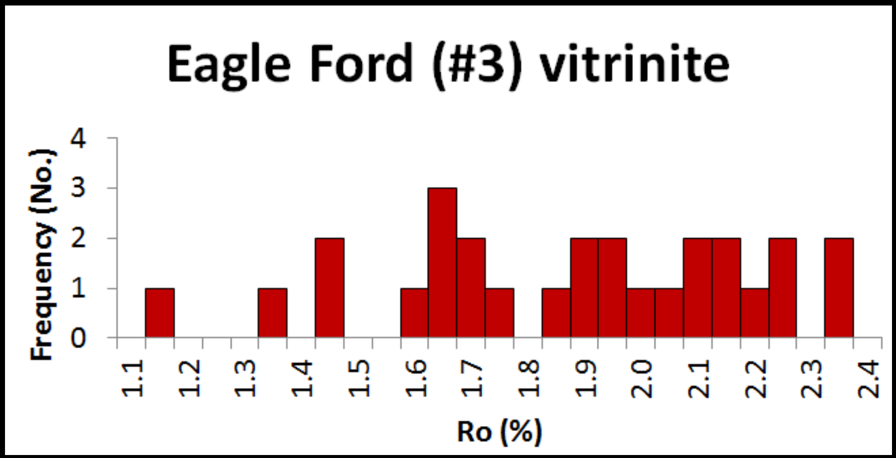
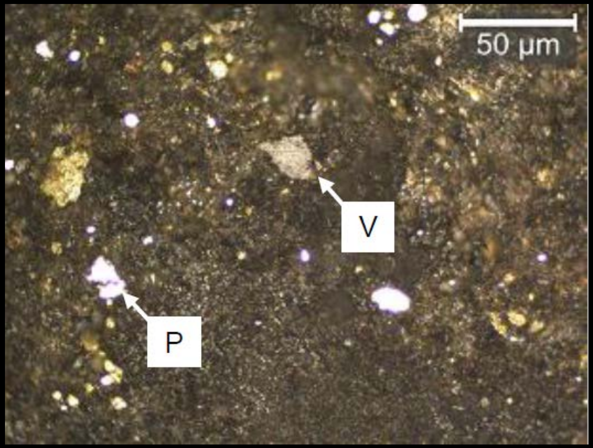
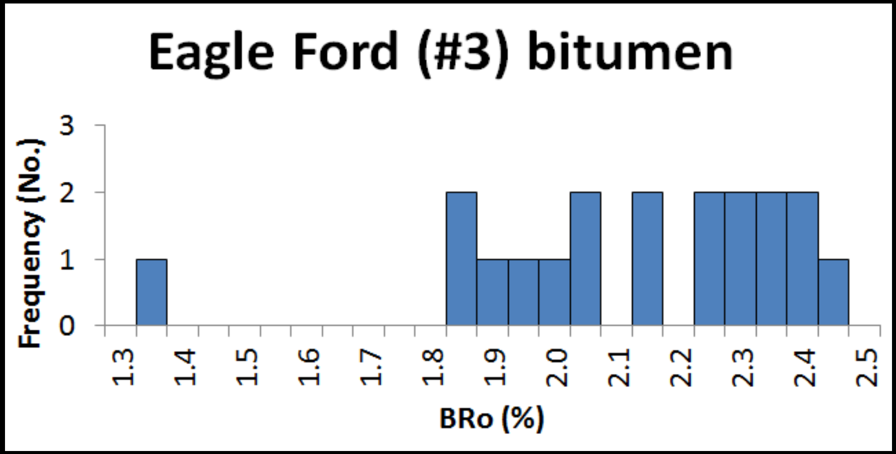
# Results: Eagle Ford (#3)




<u>Vitrinite</u>

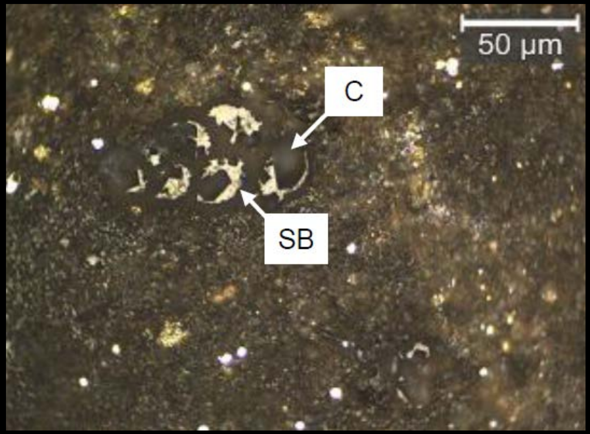
- Mean: 1.81
- GSD: 0.31
- Skew: -0.42
- Kurt: -0.38
- R: 0.88




<u>Solid Bitumen</u>

- Mean: 2.05
- GSD: 0.27
- Skew: -1.28
- Kurt: 2.20
- R: 0.75




+ SK *tails to the right*
+ KT acute *peak* and fatter tails

- SK *tails to the left*
- *KT lower, wider peak and thinner tails*

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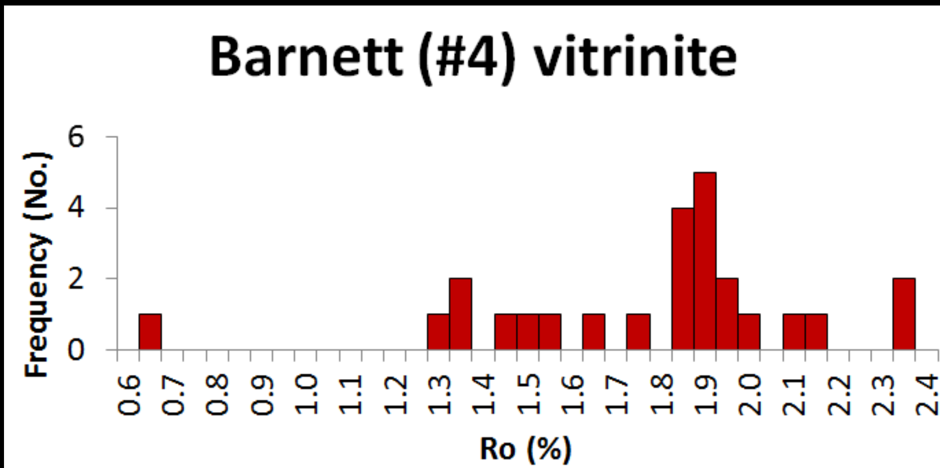
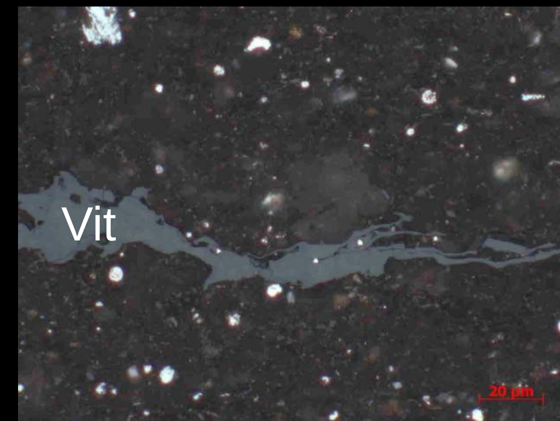
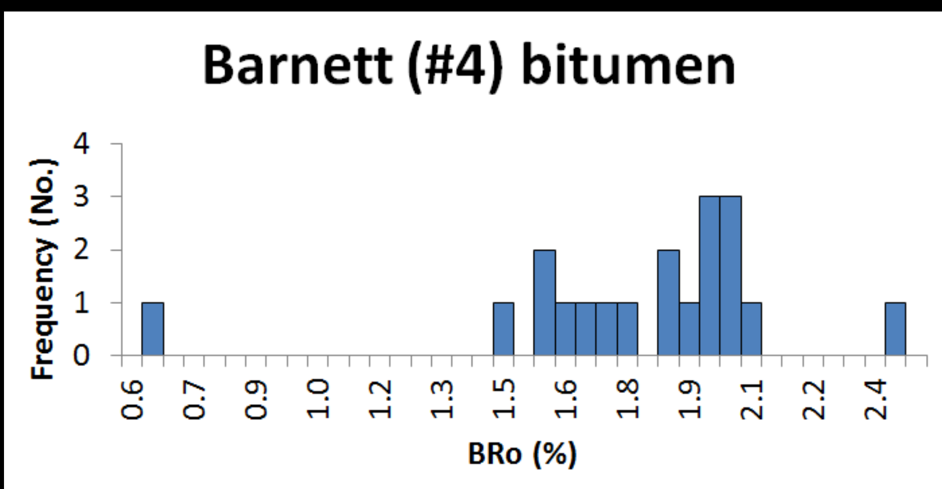
# Results: Barnett (#4)




<u>Vitrinite</u>

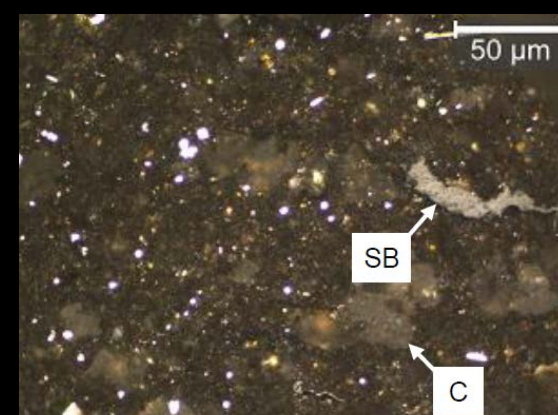
- Mean: 1.70
- GSD: 0.37
- Skew: -1.14
- Kurt: 2.50
- R: 1.03




<u>Solid Bitumen</u>

- Mean: 1.75
- GSD: 0.36
- Skew: -1.77
- Kurt: 5.76
- R: 1.01

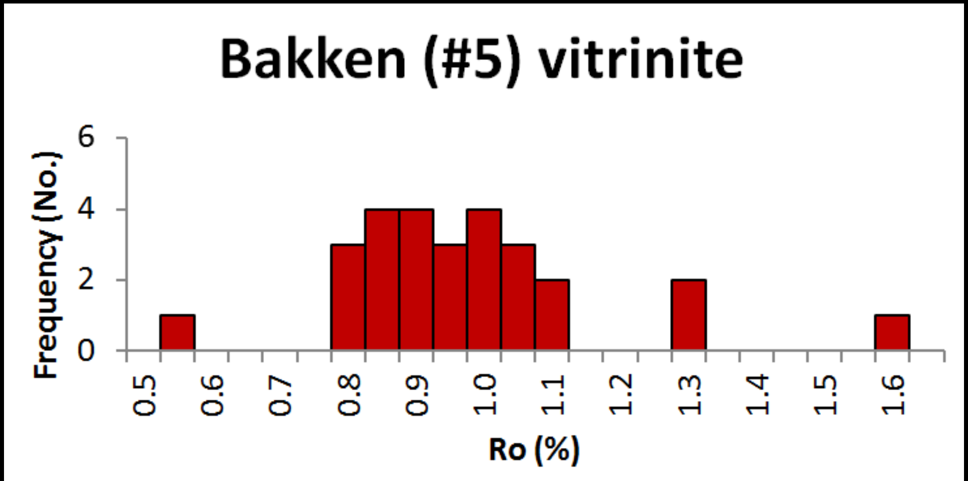



+ SK *tails to the right*
+ KT acute *peak* and fatter tails

- SK *tails to the left*
- *KT lower, wider peak and thinner tails*

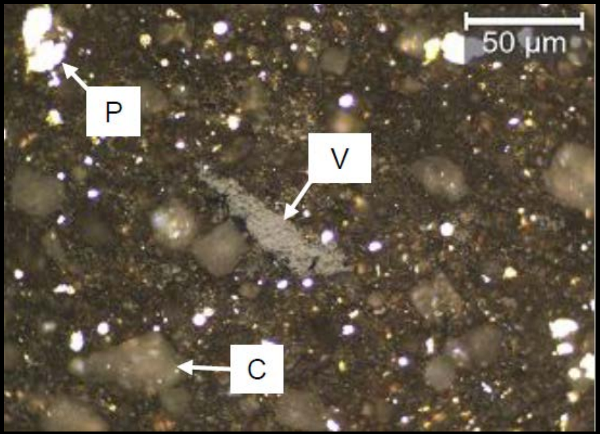
*Presented for ICCP, September, 2016*

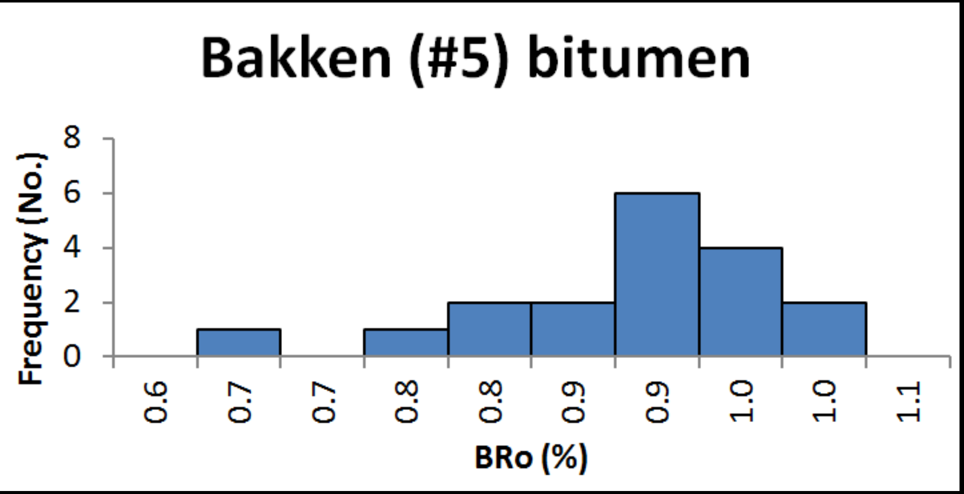
# Results: Bakken (#5)




Vitrinite

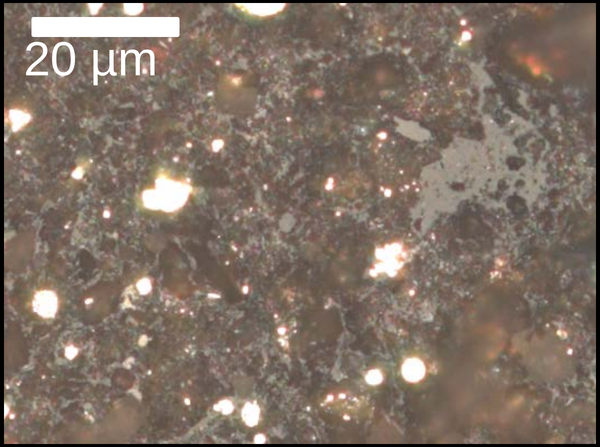
- Mean: 0.90
- GSD: 0.19
- Skew: 1.02
- Kurt: 3.47
- R: 0.54




Solid Bitumen

- Mean: 0.87
- GSD: 0.08
- Skew: -1.00
- Kurt: 1.13
- R: 0.23

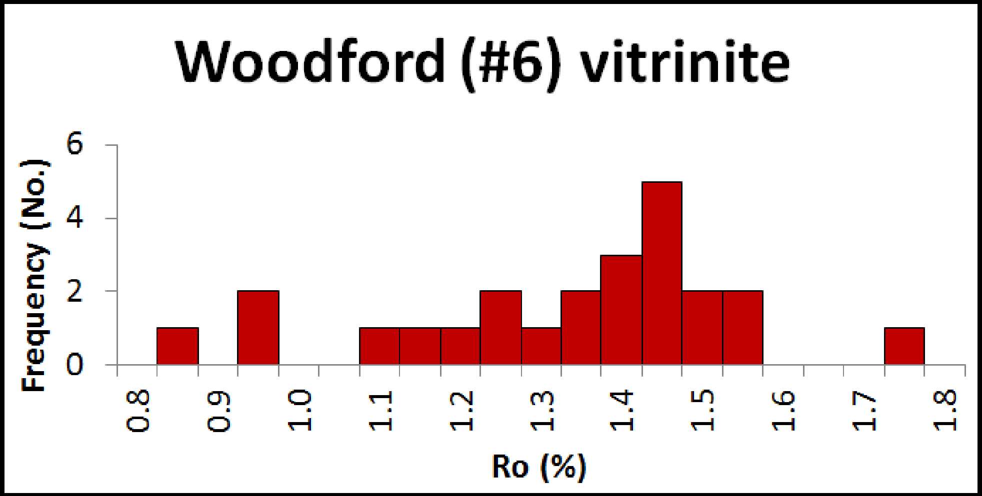



+ SK *tails to the right*
+ KT acute *peak* and fatter tails

- SK *tails to the left*
- *KT lower, wider peak and thinner tails*

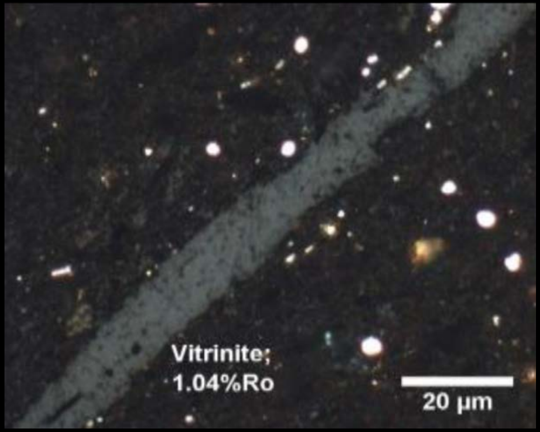
*Presented for ICCP, September, 2016*

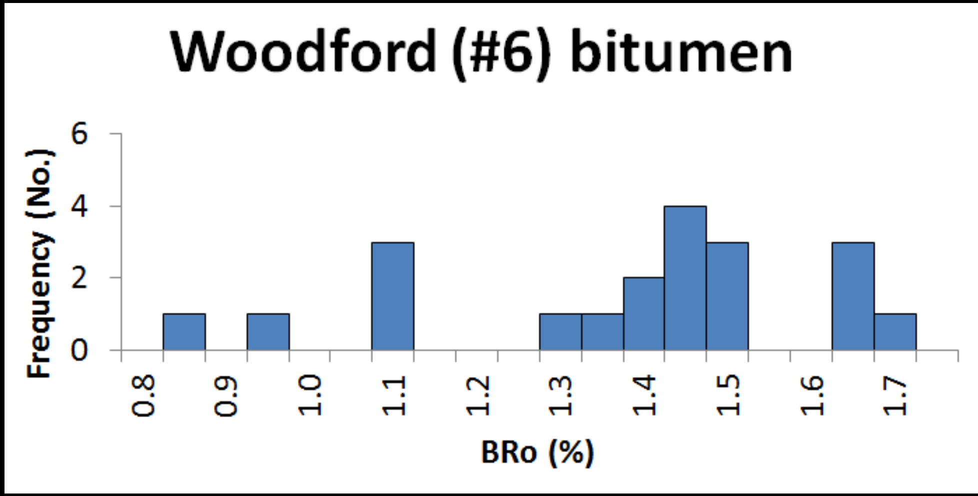
# Results: Woodford (#6)




<u>Vitrinite</u>

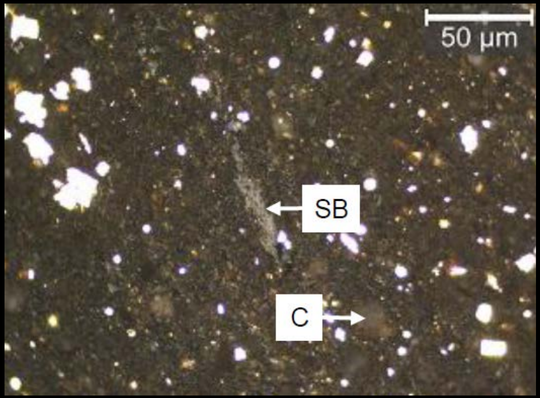
- Mean: 1.31
- GSD: 0.22
- Skew: -0.74
- Kurt: 0.37
- R: 0.61




<u>Solid Bitumen</u>

- Mean: 1.36
- GSD: 0.24
- Skew: -0.80
- Kurt: -0.03
- R: 0.66

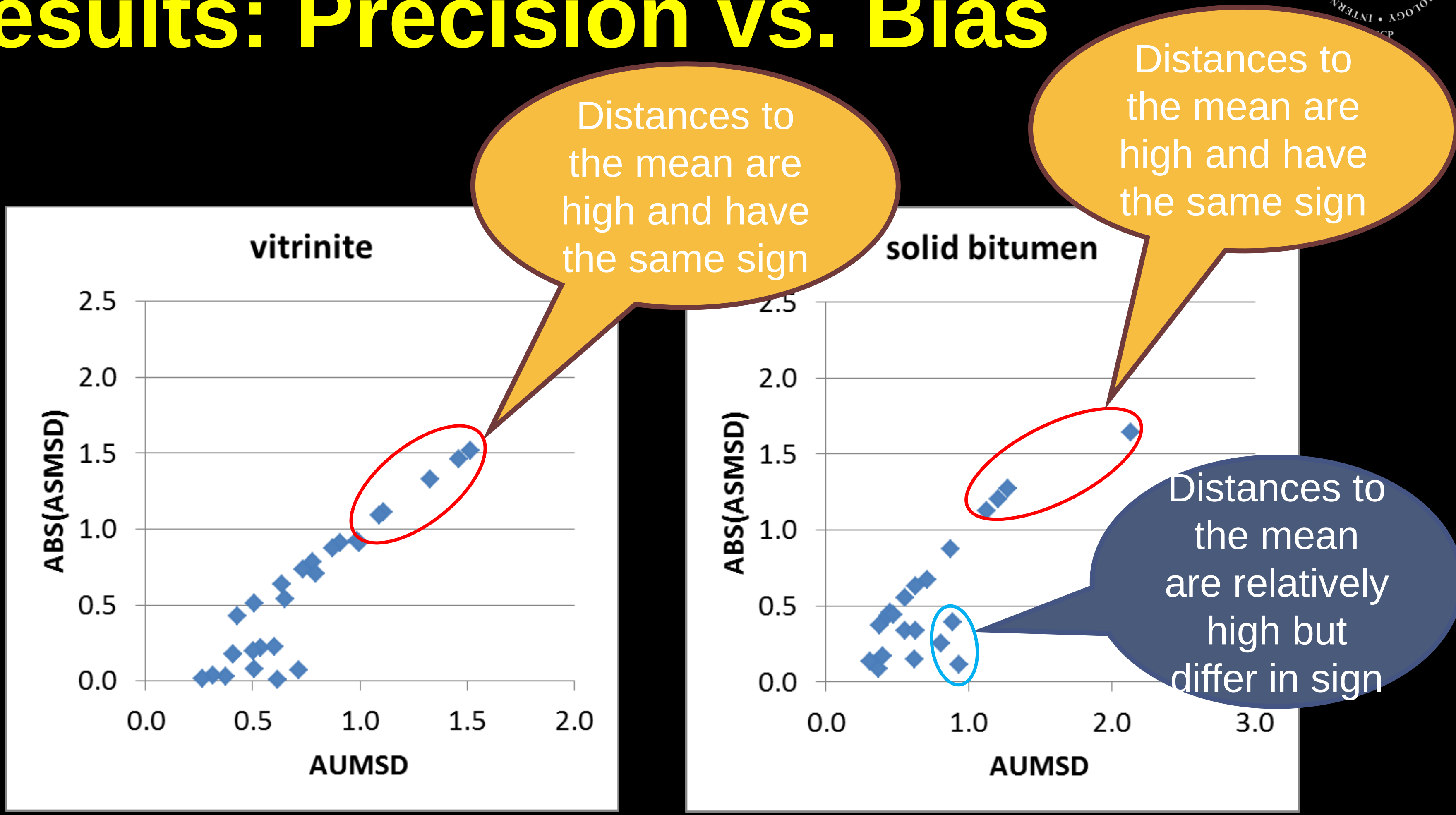



\+ SK *tails to the right*
\+ KT acute *peak* and fatter tails

\- SK *tails to the left*
\- *KT lower, wider peak and thinner tails*

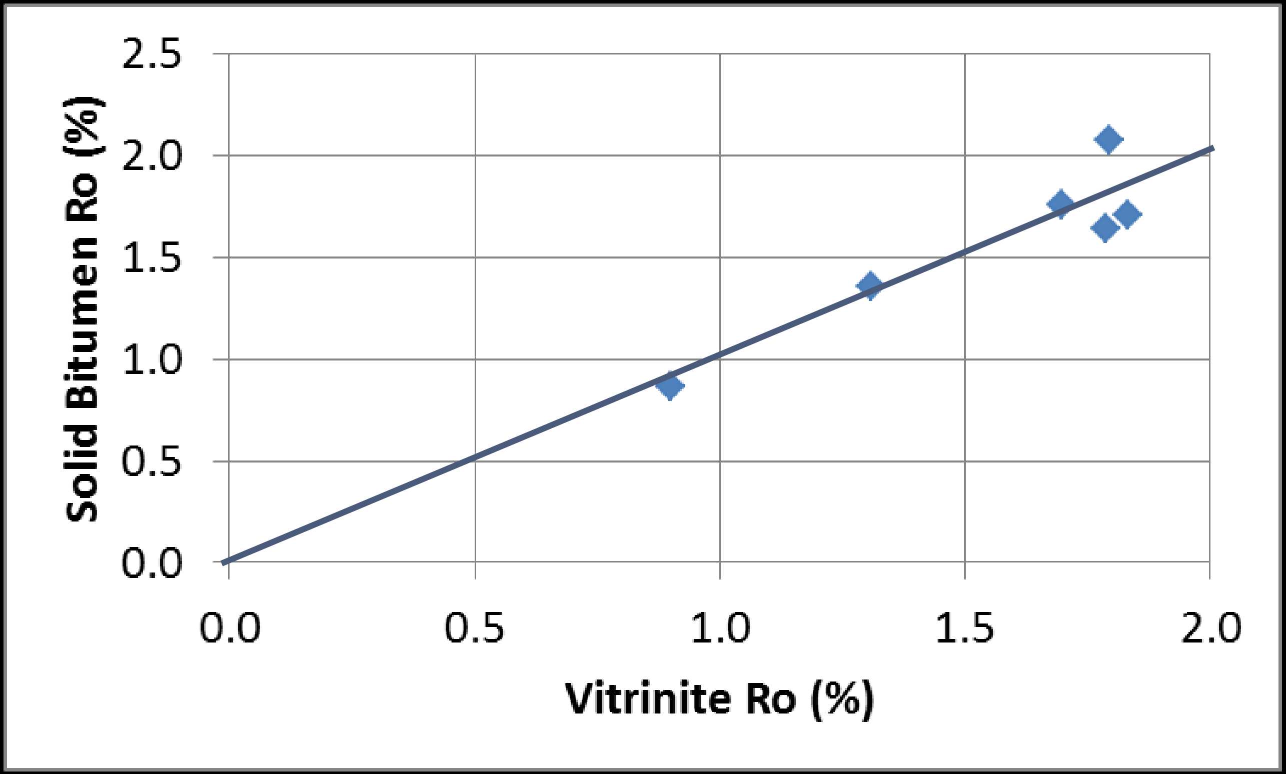
*Presented for ICCP, September, 2016*

# Results: Precision vs. Bias




- Calibration difficulties for high ABS(ASMSD) (?)
- Identification difficulties for high AUMSD and low ABS(ASMSD) (?)

# Results: solid bitumen vs. vitrinite




- No systematic relationship of solid bitumen to vitrinite Ro
- No clear way to differentiate solid bitumen from vitrinite

# Summary

- The results were terrible
- Some statistical method must be used to eliminate outliers
- These results cannot be published, in my opinion
- Solid bitumen vs vitrinite identifications continue to plague organic petrography of NA shales
- These samples were representative of NA shales, and high TOC
- If we cannot measure them, what are we doing?