

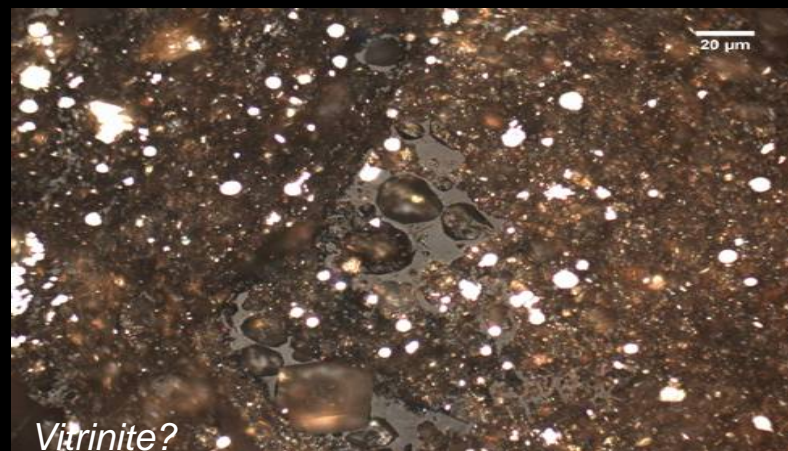
ICCP Working Group Identification of Primary Vitrinite in Shale 2017 Report

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Presented for ICCP Commission II, September, 2017

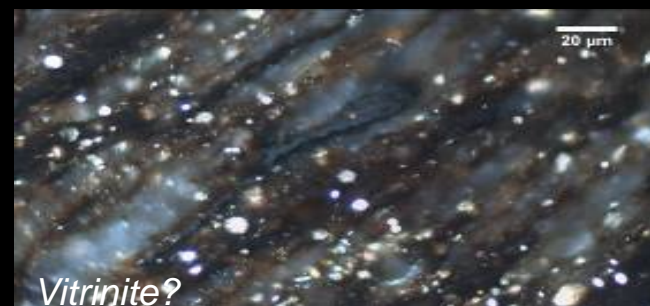
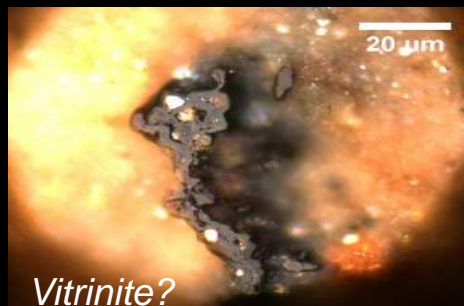
Outline

- 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



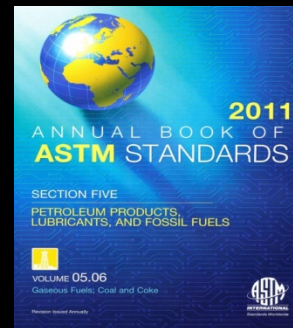
Identification of primary vitrinite: History of the ICCP working group

- Proposed by Angeles Borrego 2008 Oviedo
- DOMVR survey 2009 Gramado, ICCP News No. 48
- ASTM standard D7708 in 2011 ASTM Standards



Designation: D7708 – 11

**Standard Test Method for
Microscopical Determination of the Reflectance of Vitrinite
Dispersed in Sedimentary Rocks¹**



Oviedo 2008

Gramado 2009

Belgrade 2010

Porto 2011

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

- ASTM D7708 interlaboratory study in 2012-2013
- Results presented Sosnowiec 2013
- Results presented AAPG, Houston, USA, 2014
- Results published in J. Marine and Petroleum Geology, 2015

Porto 2011

Beijing 2012

Sosnowiec 2013

Kolkata 2014

Potsdam 2015

2015-2016 Interlaboratory Study

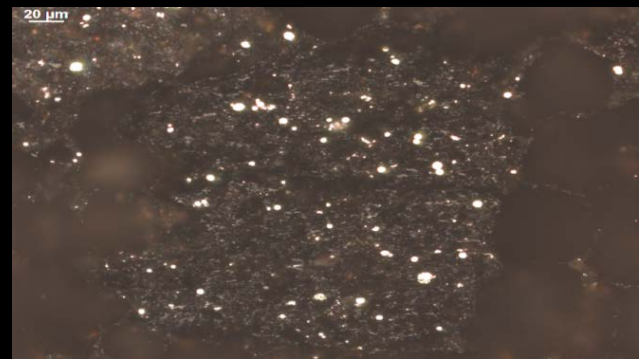
- Six high maturity samples with high TOC – current USA shale gas/tight oil plays: e.g., Eagle Ford, Marcellus, Haynesville, Barnett, Bakken, Woodford



Jurassic: TOC 2.66 wt.%, $R_o > 1.0\%$



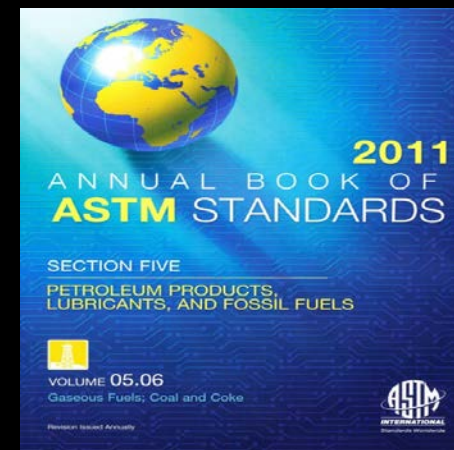
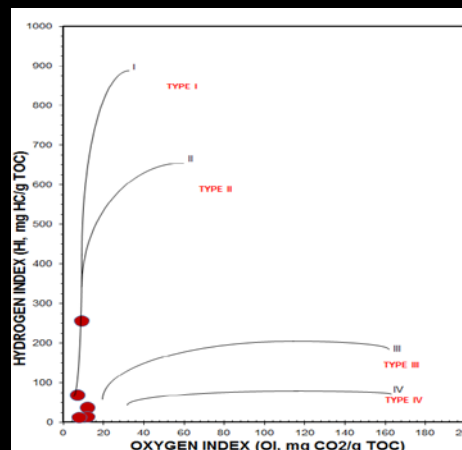
Upper Cretaceous: TOC 5.07 wt.%, $R_o > 1.0\%$



Devonian: TOC 5.17 wt.%, $R_o > 1.0\%$

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
- Vitrinite is rare!
- Distributed as crushed rock
- Instructions to follow D7708



Distribution of Samples

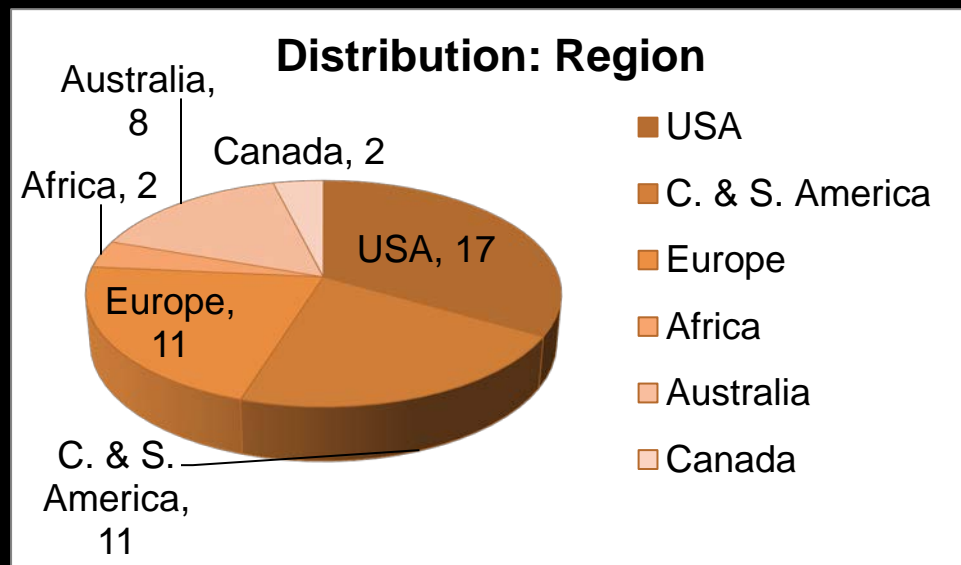
- Samples posted January 2016

- 51 Petrographers

- 40 Laboratories

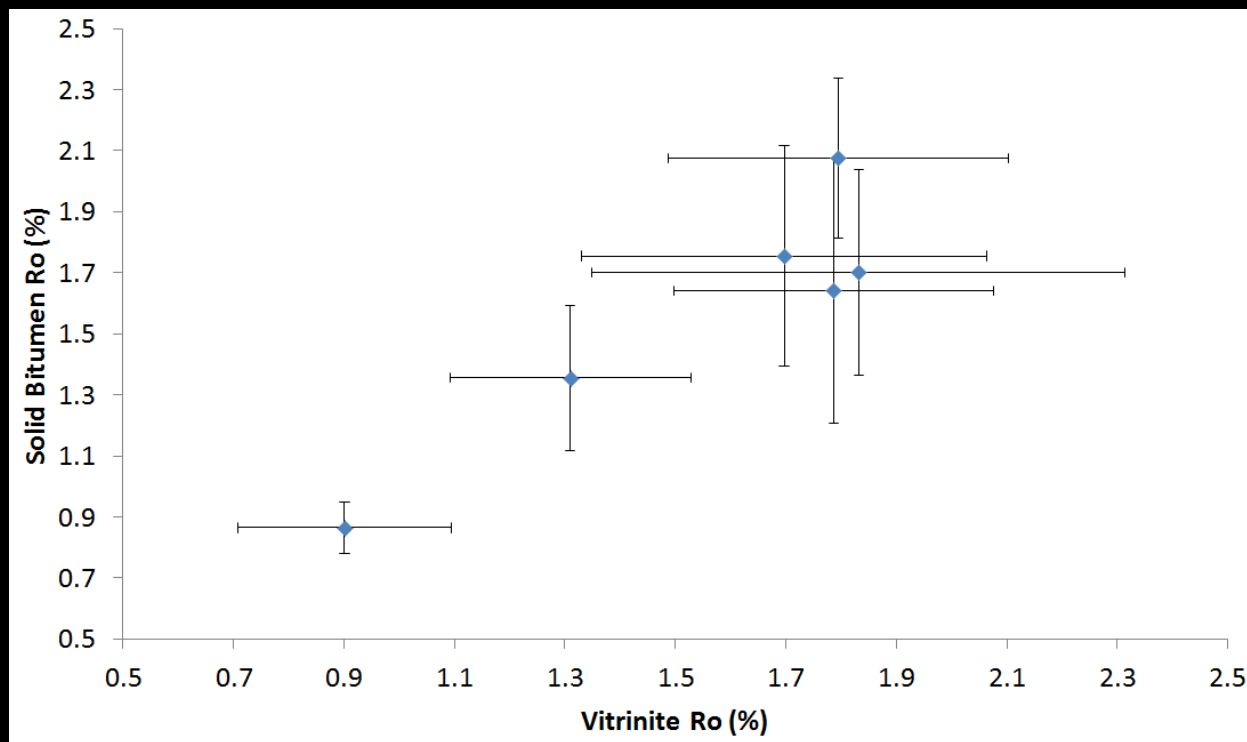
- 14 Countries

- 6 Continents

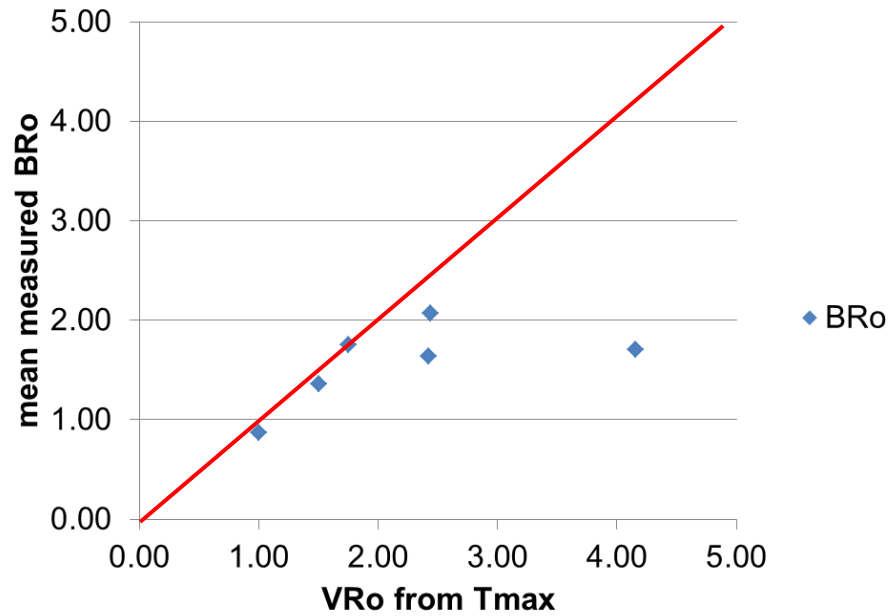
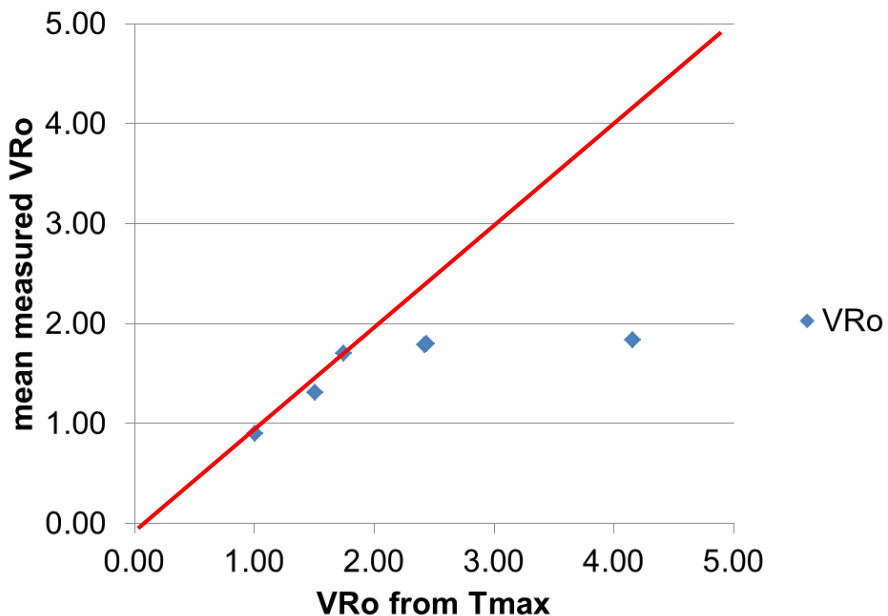


Results: from 36 petrographers

- High standard deviations
- Reproducibility is very poor
- Results need to be refined by some method

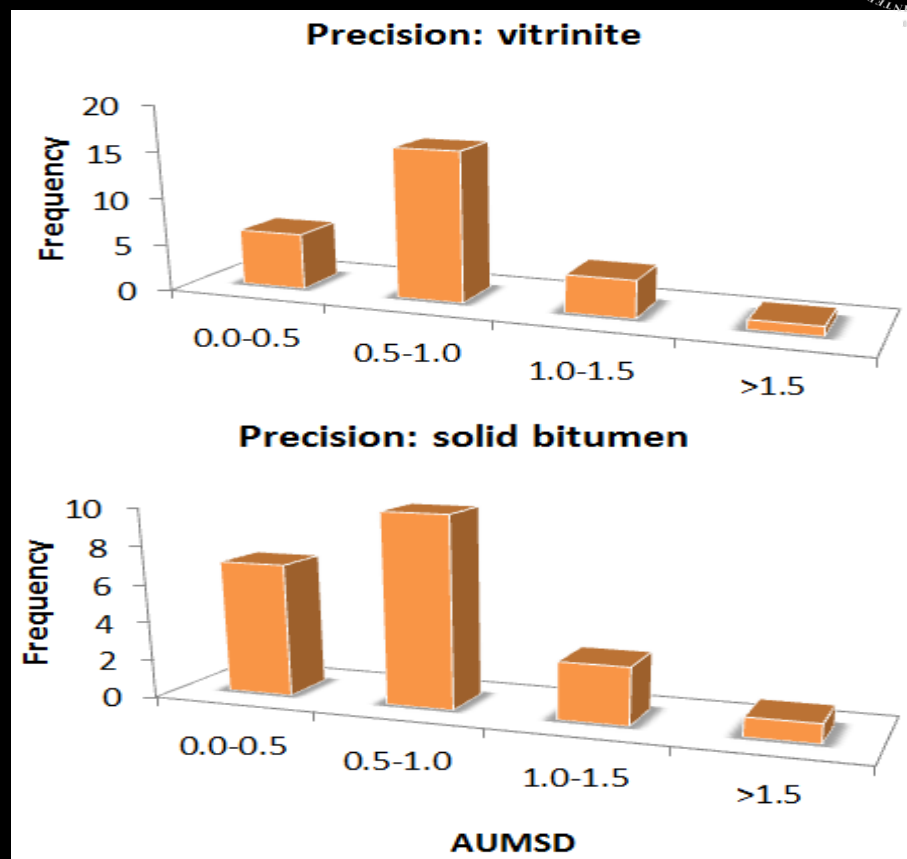


Results: from 36 petrographers

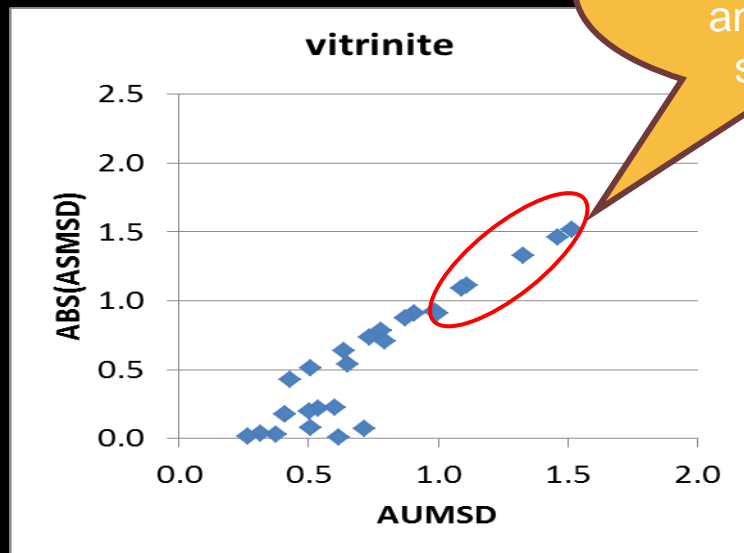


Results

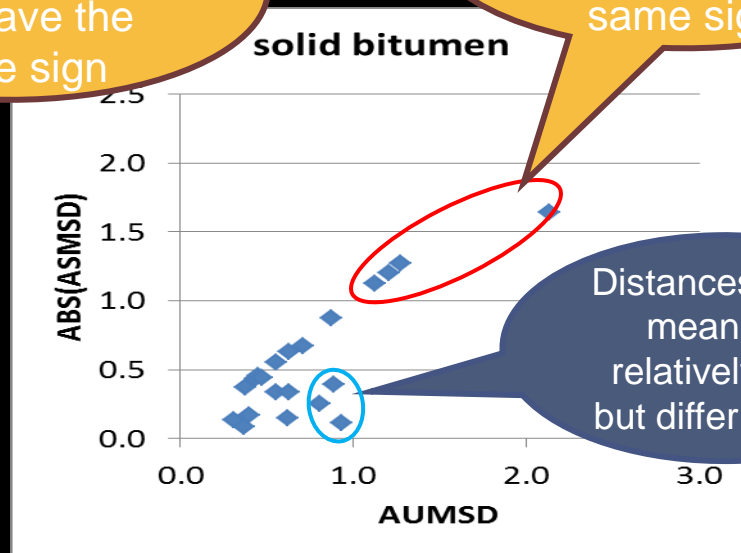
- 36 petrographers up until September 9th, 2016
- 71% (36 of 51) sample recipients returned results
- 28 petrographers held ICCP accreditation in DOMVR
- Accredited vs. non-accredited petrographers performed similarly
- 1 petrographer had AUMSD >1.5 for vitrinite
- 1 petrographer (a different one) had AUMSD >1.5 for solid bitumen
- Most had moderate to high precision (because of high group s.d.)



Results: Precision vs. Bias



Distances to the mean are high and have the same sign



Distances to the mean are high and have the same sign

Distances to the mean are relatively high but differ in sign

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

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?

How to refine results and publish?

- Some objective statistical method or approach must be used to eliminate outliers
- Throw out SMSD and USMD >1.0 ? (5-8)
- Throw out results non-compliant to D7708? (Many!)
- A criterion to limit standard deviation? $0.15 \cdot R_o$
- A photographic round robin to see what people identify as vitrinite?
- Other suggestions?

1

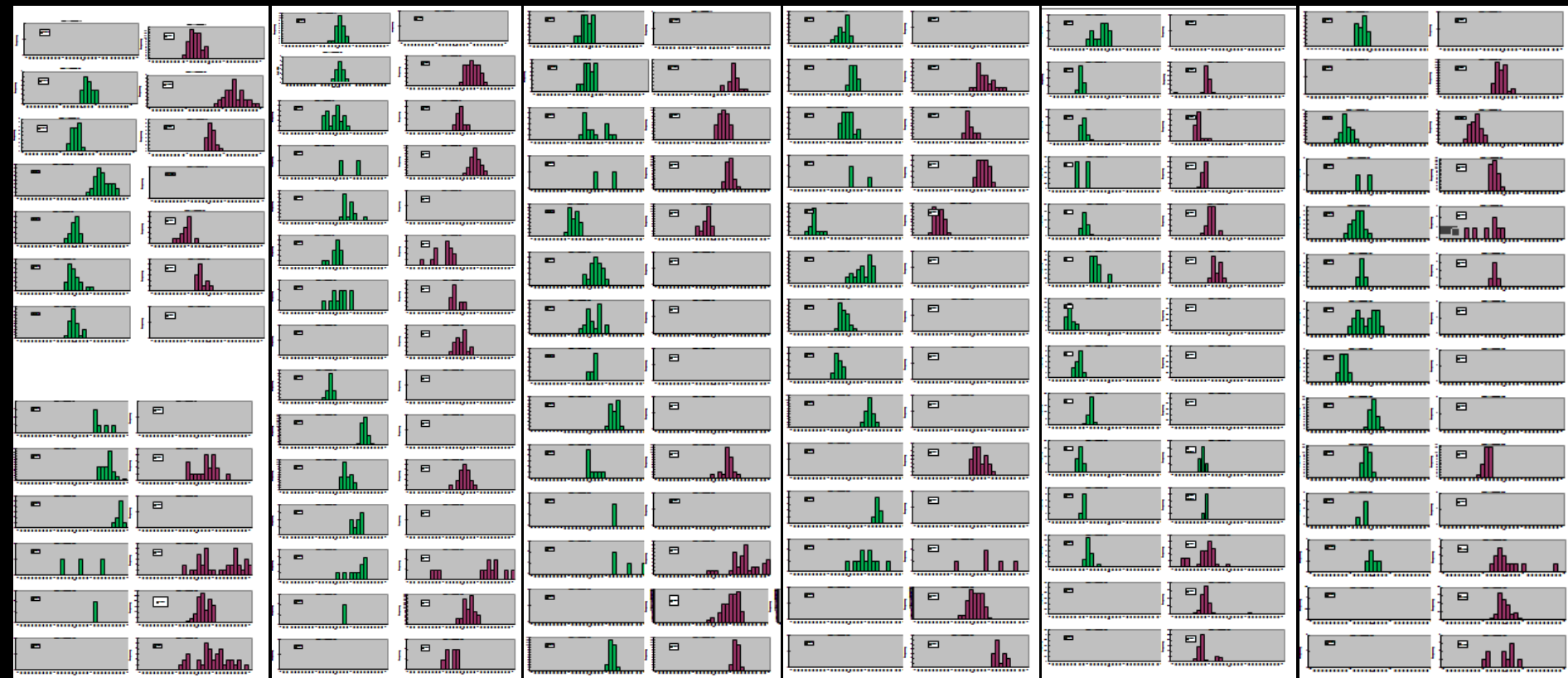
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Acknowledgments

- 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)
- USGS Energy Resources Program
- Participants in the ICCP interlaboratory study