

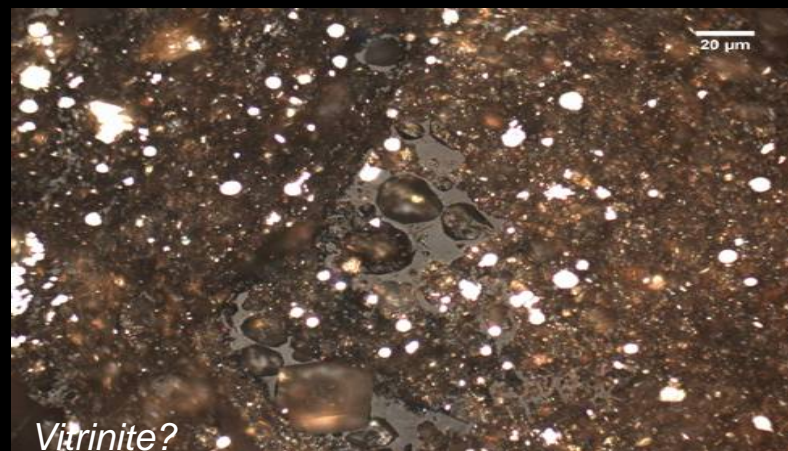
ICCP Working Group Identification of Primary Vitrinite in Shale 2018 Report

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Presented for ICCP Commission II, September, 2018

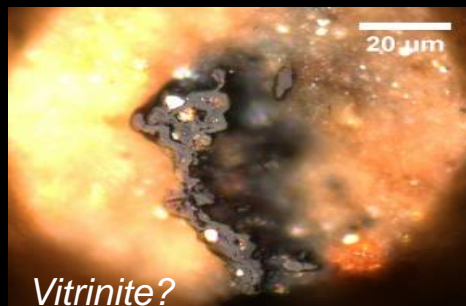
Outline

- Problem to be solved
- History of the ICCP working group
- Findings and products to-date
- 2015-2016 exercise statistics
- Summary & Proposal for New Activities



Objective of the Working Group

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



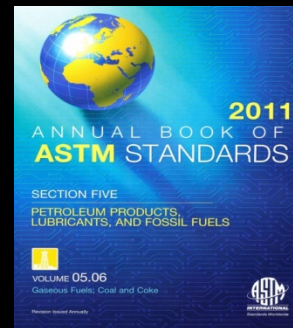
Identification of primary vitrinite: History of the ICCP working group

- 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

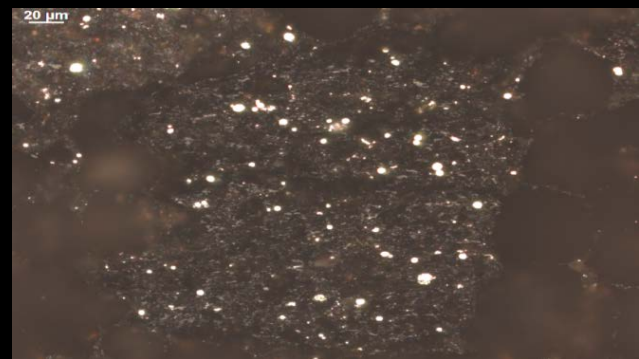
- Six high maturity samples with high TOC – current USA shale gas/tight oil plays: Eagle Ford, Marcellus, Haynesville, Barnett, Bakken, Woodford



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



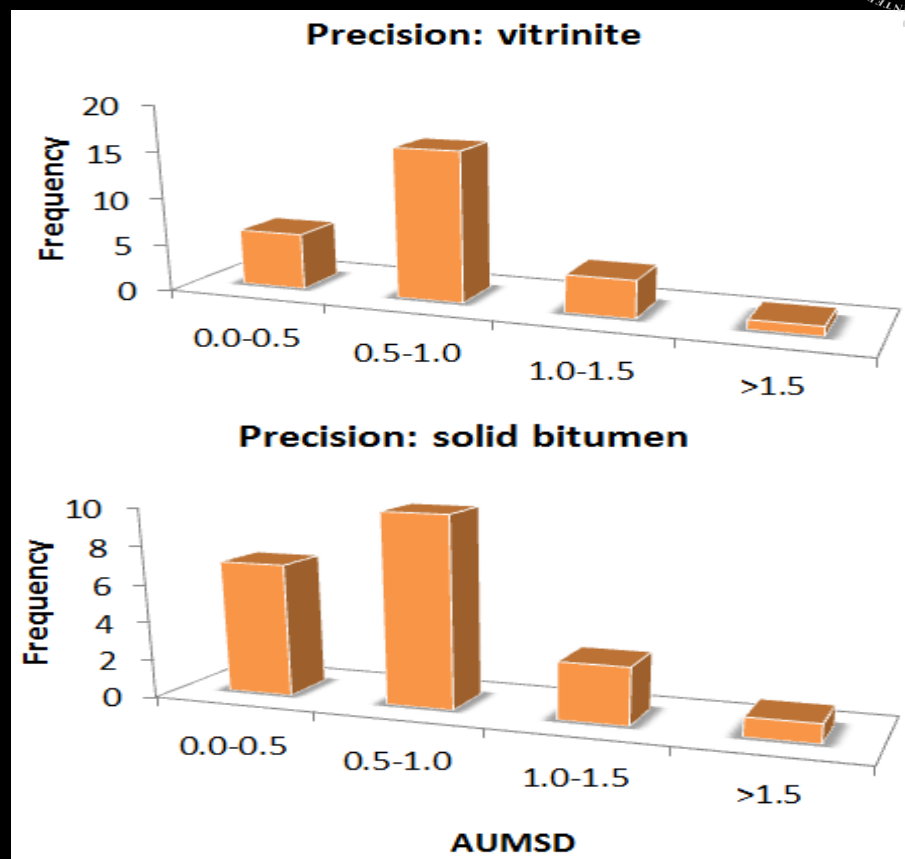
Upper Cretaceous: TOC 5.07 wt.%, $R_o > 1.0\%$



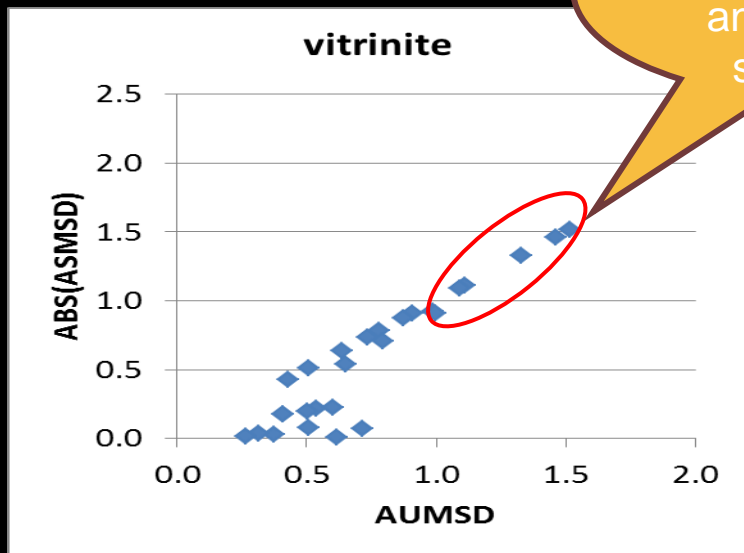
Devonian: TOC 5.17 wt.%, $R_o > 1.0\%$

Results

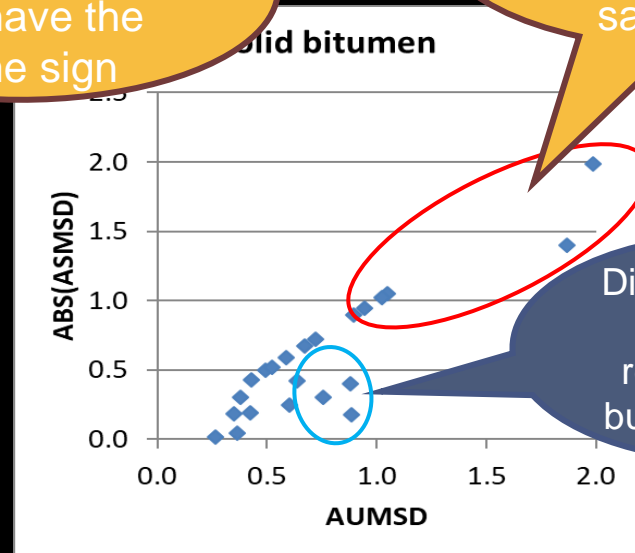
- 37 petrographers up until October 2017
- 73% (37 of 51) sample recipients returned results
- 28 petrographers held ICCP accreditation in DOMVR
- Accredited vs. non-accredited petrographers performed similarly
- 1 petrographer had AUMSD >1.5 for vitrinite
- 2 petrographers (different ones) had AUMSD >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 of 2015-2016 study

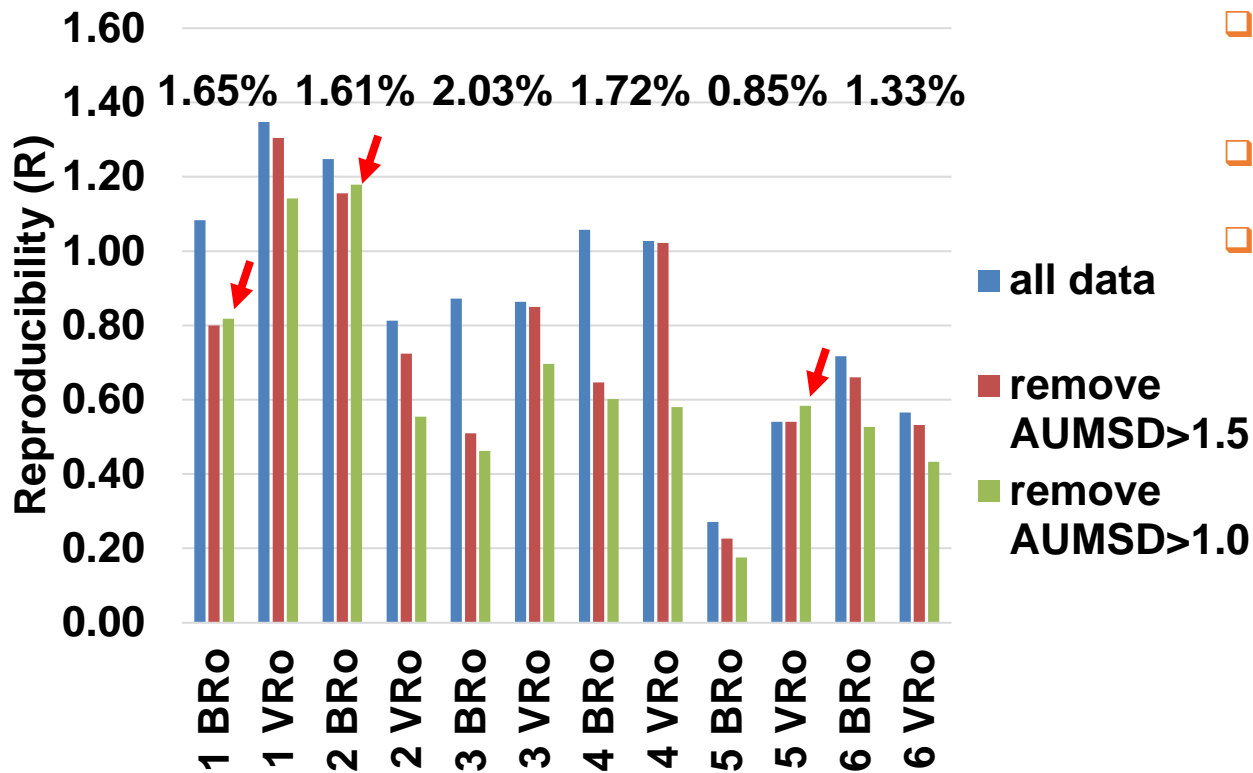
- The results were terrible for reproducibility
- Some statistical method must be used to eliminate outliers
- Solid bitumen vs vitrinite identifications continue to plague organic petrography of NA shales
- These samples were representative of NA shales, and high TOC

How to refine results and publish?

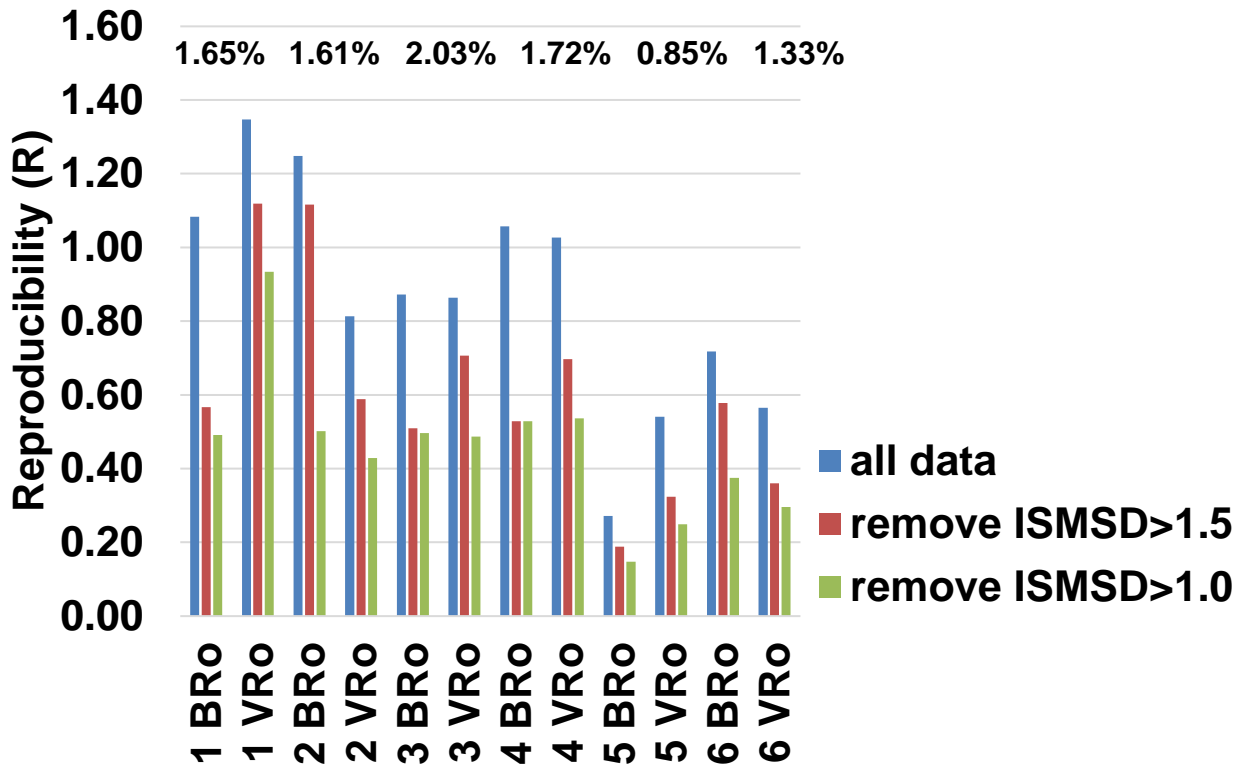
- Remove AUSMD >1.0 ?
- Remove IUSMD >1.0 ?
- Remove results anyway non-compliant to D7708?
- Remove results where $s.d > 0.15 * R_o$
- Remove results where $n < 20$
- Remove results not following D7708 template

Remove AUSMD > 1.0

- Requires pool of data
- Removing >1.5 consistent improvement
- Removing >1.0 inconsistent
- Unacceptable R values

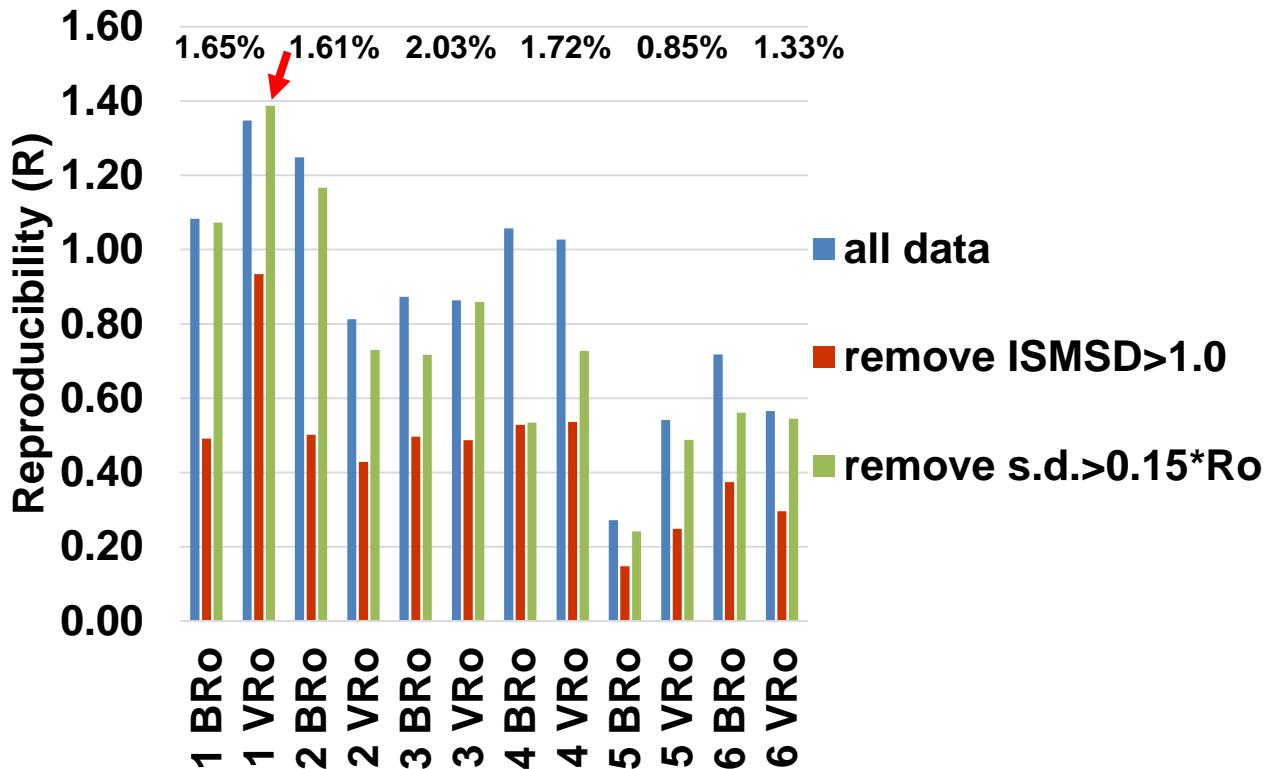


Remove IUSMD > 1.0



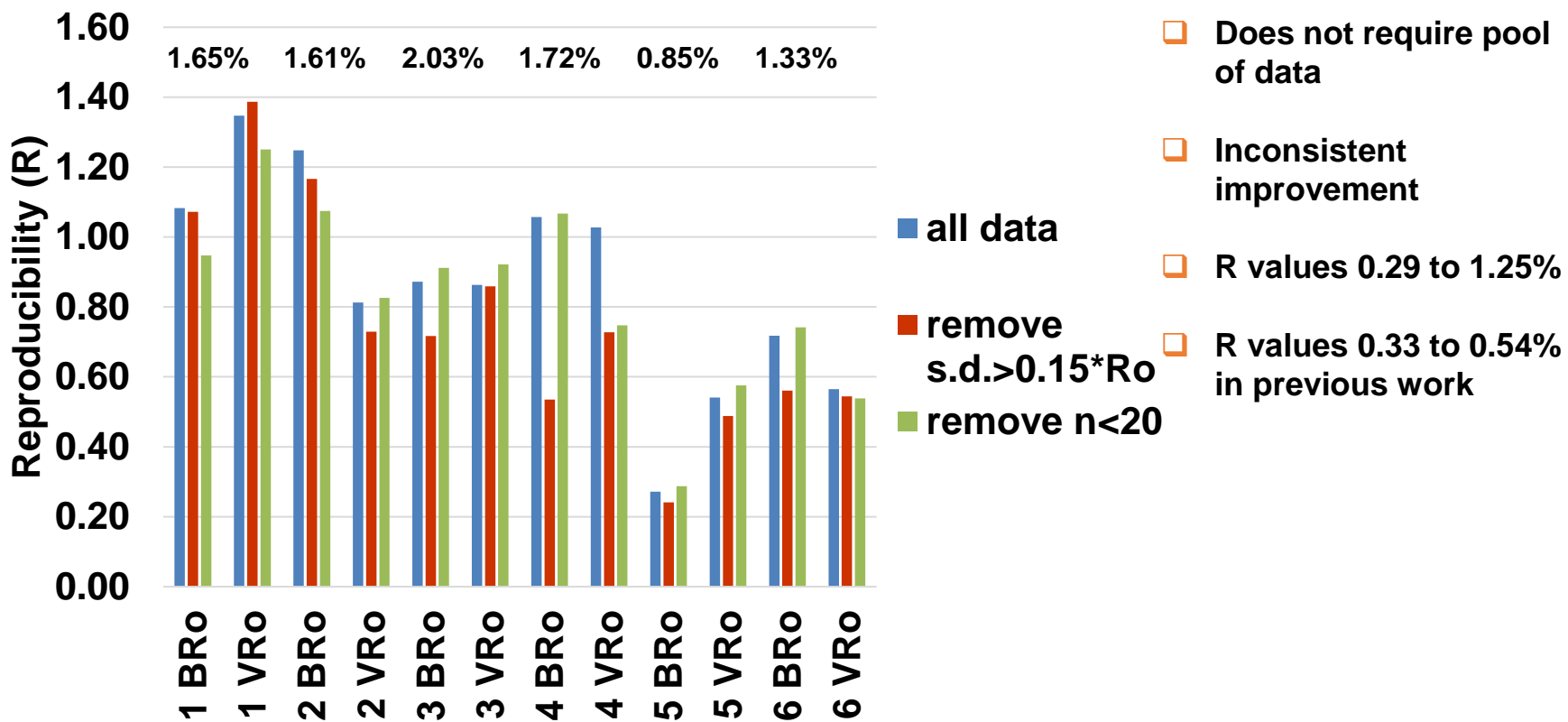
- Requires pool of data
- Removing >1.5 consistent improvement
- Removing >1.0 consistent improvement
- Acceptable R values (0.15 to 0.93%)
- Need pool of data!

Remove s.d.>0.15*Ro

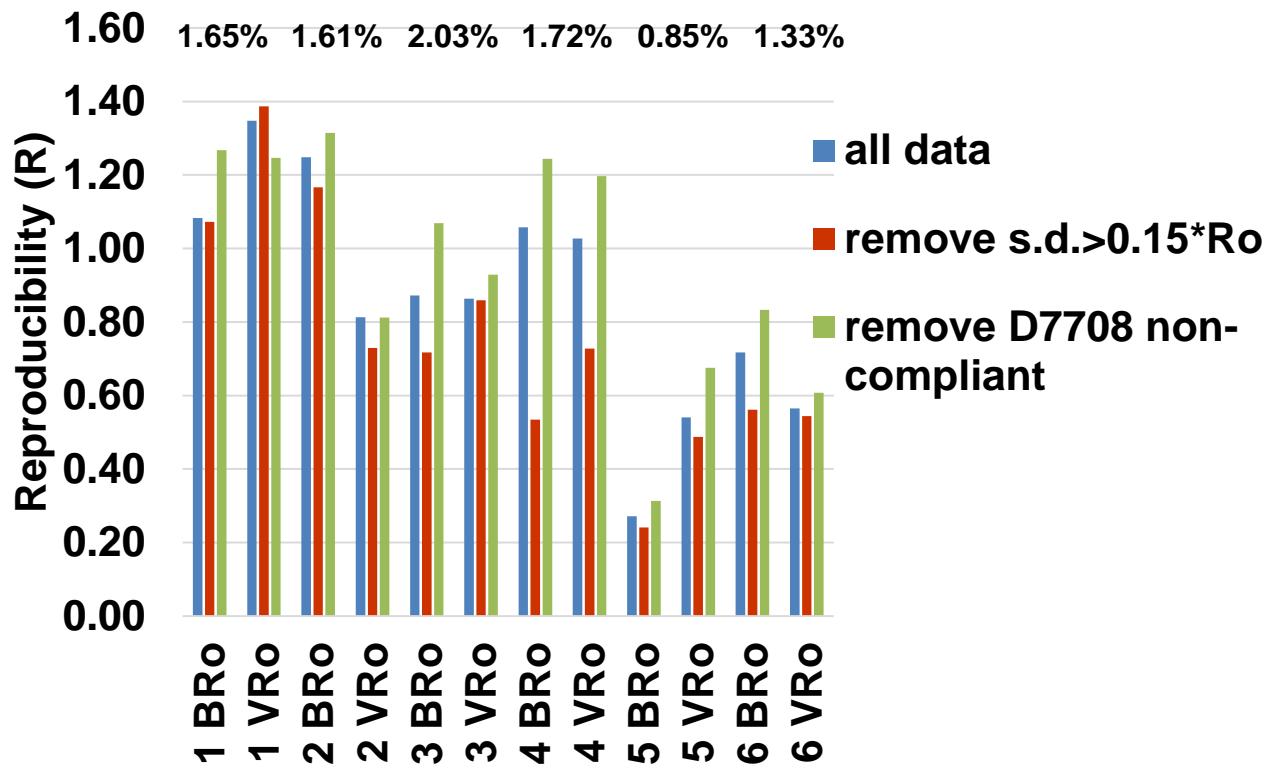


- Does not require pool of data
- Results in mostly consistent minor improvement
- R values (+/- 0.24 to 1.39%)
- R values 0.33 to 0.54% in previous work

Remove n<20

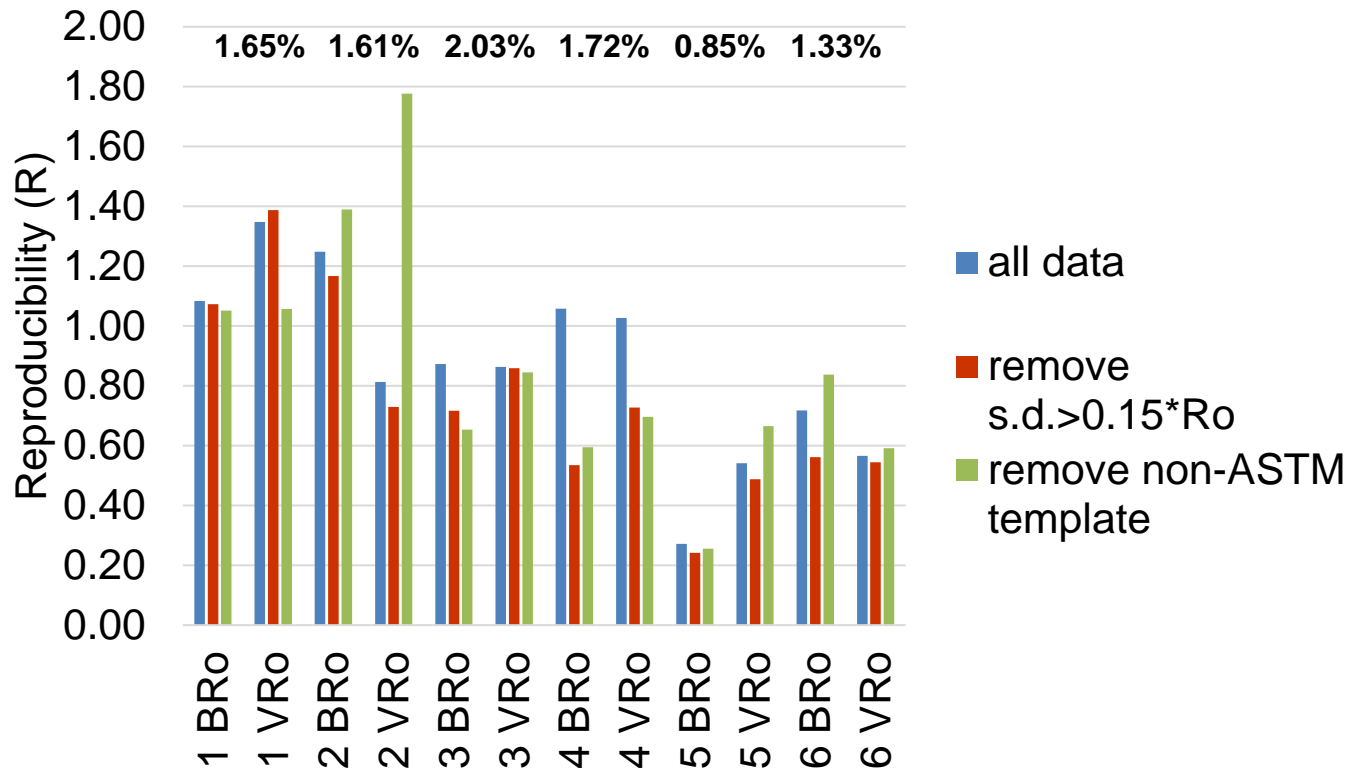


Remove ASTM non-compliant



- Does not require pool of data
- Results in mostly poorer reproducibility
- R values not acceptable (0.31 to 1.31)

Remove non-ASTM template



- Does not require pool of data
- Results are inconsistent
- R values not acceptable (0.26 to 1.78)

■ all data

■ remove s.d. > 0.15 * Ro

■ remove non-ASTM template

Summary

- Remove AUSMD >1.5 : consistent improvement
- Remove IUSMD >1.0 : **best results**
- Remove non-compliant to D7708: no improvement
- Remove s.d $> 0.15 * R_o$: consistent improvement
- Remove $n < 20$: inconsistent
- Remove non-D7708 template: inconsistent

Proposal for New Activities 2018-2019

- A photographic round robin with same samples to see what people identify as vitrinite vs solid bitumen
- Use marked PowerPoint with Excel template for answers

Proposal for New Activities 2020 ...

- New round robin with different (but similar) samples
- Ask for solid bitumen reflectance instead of vitrinite
- Use lessons learned, insist on $s.d. < 0.15 * R_o$
 - Insist $n > 20(?)$
 - Insist ASTM template(?)

Acknowledgments

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- Participants in the ICCP interlaboratory study