

## Research paper

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



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## ABSTRACT

Vitrinite reflectance generally is considered the most robust thermal maturity parameter available for application to hydrocarbon exploration and petroleum system evaluation. However, until 2011 there was no standardized methodology available to provide guidelines for vitrinite reflectance measurements in shale. Efforts to correct this deficiency resulted in publication of ASTM D7708: *Standard test method for microscopical determination of the reflectance of vitrinite dispersed in sedimentary rocks*. In 2012–2013, an interlaboratory exercise was conducted to establish precision limits for the D7708 measurement

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technique. Six samples, representing a wide variety of shale, were tested in duplicate by 28 analysts in 22 laboratories from 14 countries. Samples ranged from immature to overmature (0.31–1.53%  $R_o$ ), from organic-lean to organic-rich (1–22 wt.% total organic carbon), and contained Type I (lacustrine), Type II (marine), and Type III (terrestrial) kerogens. Repeatability limits (maximum difference between valid repetitive results from same operator, same conditions) ranged from 0.03 to 0.11% absolute reflectance, whereas reproducibility limits (maximum difference between valid results obtained on same test material by different operators, different laboratories) ranged from 0.12 to 0.54% absolute reflectance. Repeatability and reproducibility limits degraded consistently with increasing maturity and decreasing organic content. However, samples with terrestrial kerogens (Type III) fell off this trend, showing improved levels of reproducibility due to higher vitrinite content and improved ease of identification. Operators did not consistently meet the reporting requirements of the test method, indicating that a common reporting template is required to improve data quality. The most difficult problem encountered was the petrographic distinction of solid bitumens and low-reflecting inert macerals from vitrinite when vitrinite occurred with reflectance ranges overlapping the other components. Discussion among participants suggested this problem could not be easily corrected via kerogen concentration or solvent extraction and is related to operator training and background. No statistical difference in mean reflectance was identified between participants reporting bitumen reflectance vs. vitrinite reflectance vs. a mixture of bitumen and vitrinite reflectance values, suggesting empirical conversion schemes should be treated with caution. Analysis of reproducibility limits obtained during this exercise in comparison to reproducibility limits from historical interlaboratory exercises suggests use of a common methodology (D7708) improves interlaboratory precision. Future work will investigate opportunities to improve reproducibility in high maturity, organic-lean shale varieties.

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## 1. Introduction and background

Vitrinite reflectance is widely considered the most robust petrographic parameter for determination of thermal maturity in hydrocarbon exploration (e.g., Taylor et al., 1998; Corcoran and Doré, 2005; Dembicki, 2009; Suárez-Ruiz et al., 2012). However, it has been historically difficult to obtain reproducible results for this measurement in interlaboratory studies on samples of non-coal sedimentary rocks (Dembicki, 1984; Borrego, 2009). To address this shortcoming, American Society for Testing and Materials (ASTM) standard test method D7708 (ASTM, 2014a) was developed by an international committee of technical experts from government, academia, industry, and consultancies. This partnership between members of the International Committee for Coal and Organic Petrology (ICCP), the Society for Organic Petrology (TSOP), the American Association of Petroleum Geologists (AAPG), and the ASTM was formed to address the need for reflectance measurement standardization in rocks other than coal, particularly shale. With current global oil and gas industry interest focused on unconventional shale gas and liquids plays (Aguilera and Radetzki, 2013), it is critically important that determination of thermal maturity in these rocks has a codified procedure for measurement.

Development of the standard test method began within ICCP in 2008 with a survey of common practices used by laboratories that routinely measure the reflectance of dispersed vitrinite in shales (ICCP, 2009). The test method writing committee was identified from among the survey respondents, and the existing ASTM coal vitrinite reflectance standard D2798 (ASTM, 2014b) was used as an outline for the new test method. Similar to D2798, the D7708 test method is executed by examining a polished sample of rock material with a microscope-photometer system at high magnification (400–750 $\times$ ) under incident white light with an oil immersion objective. Light reflected from vitrinite or other organic materials is measured and recorded in percent reflectance after calibration to standards of known reflectance.

Significant deviations from the D2798 coal standard included: 1) a specialized terminology to include recycled vitrinite, zooclasts, solid bitumens, and marine algae; 2) discussion of potential for vitrinite suppression and retardation in certain conditions; 3) inclusion of fluorescence observation and resulting changes to equipment

description and procedure; and 4) addition of reporting requirements, including type and quality of sample preparation, observation of fluorescence, and consideration of supporting data and information.

The ASTM D7708 standard test method was first published in 2011 and plans to conduct an interlaboratory exercise for precision were developed during the 2011 and 2012 meetings of ICCP (also see ICCP Commission II website <http://www.iccop.org/workinggroup/identification-of-primary-vitrinite/> for additional information related to standard test method development and ILS planning). During 2012–2013, the interlaboratory exercise was executed and its statistical results were vetted through the ASTM balloting process to include a precision statement in D7708 in 2014.

Users of ASTM D7708 include government, academic, and service laboratories, and it has been adopted as the prescribed method for the dispersed vitrinite reflectance accreditation program of the ICCP, which currently includes approximately 40 laboratories worldwide. The test method is most relevant for shale gas and shale liquids plays where precise information concerning thermal maturity is necessary for successful exploration and development (e.g., Curtis, 2002; Jarvie et al., 2007; Passey et al., 2010; Schlumberger, 2011). This paper describes the 2012–2013 interlaboratory study (ILS) performed to develop precision statistics for D7708 and presents the ILS results.

## 2. Methods

For the ILS, a suite of six rock samples (Table 1) was selected from a larger set of approximately twenty-five potential candidates compiled and characterized by the U.S. Geological Survey (USGS) in consultation with ICCP. Samples were chosen to encompass the range of materials potentially tested by ASTM D7708, based on the criteria of thermal maturity, organic matter type, and organic matter abundance. As part of basic characterization utilized for sample selection, samples were analyzed by Rock-Eval pyrolysis (Rock-Eval II) (Table 2) and total organic carbon (TOC via Leco) (Table 2) in a commercial laboratory (Weatherford Laboratories) according to methods previously described in Barker (1974) and Espitalié et al. (1977). X-ray diffraction (XRD) of low temperature ash residues (Table 3) was performed at USGS via techniques described in Hosterman and Dulong (1989).

**Table 1**  
Sample information.

Sample ID	Formation	Location	Latitude	Longitude	Age	Collector
1	Green River	Colorado, USA	39.5346	−107.9529	Eocene	Justin Birdwell, USGS
2	Boquillas	Texas, USA	29.7097	−101.2362	Upper Cretaceous	Peter Warwick, USGS
3	Ohio/Huron	Virginia, USA	36.8874	−82.7273	Devonian	Cathy Enomoto, USGS
4	Rodiles	Asturias, Spain	43.4823	−5.1316	Jurassic, Pliensbachian	Paul Hackley, USGS
5	Pottsville	Alabama, USA	33.3876	−87.3985	Carboniferous	Richard Carroll, AL Geol. Survey
6	Pearsall	Texas, USA	28.8608	−100.5700	Lower Cretaceous	Paul Hackley, USGS

Rock samples were crushed in a jaw crusher at USGS to approximately 1 mm top size; they were not sieved. Crushed samples were distributed in duplicate to the ILS participants in plastic bags labeled 1–6. Directions sent with the samples instructed participants to further process and prepare the samples as necessary per individual laboratory mounting methods. Participants were instructed to prepare, analyze and report each of the six samples in duplicate according to the ASTM D7708 methodology (analysis in duplicate is not required per ASTM D7708 but was necessary to determine intra-laboratory repeatability for the ILS). Participants were asked to return results according to ASTM D7708 reporting requirements, but in the format preferred by individual laboratories, i.e., no reporting template was suggested. Participants were advised to contact the ILS convener for additional information relevant to the samples (e.g., lithology, age, etc.) if such information was deemed beneficial to sample analysis.

Samples were sent separately to thirty-one analysts; results were received from twenty-eight analysts in twenty-two laboratories located in fourteen different countries. Analysts share authorship on this paper.

### 3. Samples

#### 3.1. Green River Mahogany Ledge

Sample 1 is an immature laminated oil shale from the informal Mahogany oil shale zone of the Eocene Green River Formation. The sample was collected from outcrops at the Anvil Points Mine in western Colorado, USA, in the Piceance Basin. The oil shale sample is highest in total organic carbon (TOC) content of the ILS samples (Table 2) and contains abundant Type I kerogen (Fig. 1), occurring primarily as strongly fluorescent amorphous and lamellar organic matter (Fig. 2A, B). Low-gray reflecting organic matter (OM) resembling vitrinite (huminites per ICCP, 1998) is abundant and sometimes occurs with cellular structure. This sample was deposited in a lacustrine environment (Horsfield et al., 1994); high OM content is interpreted to represent deposition and preservation of algal and microbial biomass in benthic microbial mats (Schieber, 2007).

#### 3.2. Boquillas Formation

Sample 2 is an early mature marl from the Upper Cretaceous Boquillas Formation of west Texas, USA (equivalent to Eagle Ford

**Table 2**  
Rock-Eval pyrolysis (Rock Eval II) and Leco TOC data for the ILS samples.

Sample id	Formation	TOC	S1	S2	S3	T <sub>max</sub>	HI	OI	PI
1	Green River	22.37	2.50	195.00	3.90	439	871	17	0.01
2	Boquillas	4.08	1.16	31.02	1.31	423	761	32	0.04
3	Huron	7.10	0.99	20.54	0.60	435	289	8	0.05
4	Rodiles	2.66	0.48	1.70	1.21	451	64	46	0.22
5	Pottsville	5.00	0.61	6.91	0.46	447	138	9	0.08
6	Pearsall	1.01	0.15	0.29	0.32	426	29	32	0.35

Abbreviations: TOC, total organic carbon; HI, hydrogen index; OI, oxygen index; PI, production index. Units: TOC in weight %; S1, S2 in mg hydrocarbon/g rock; S3 in mg CO<sub>2</sub>/g rock; HI in mg hydrocarbon/g TOC; OI in mg CO<sub>2</sub>/g TOC; T<sub>max</sub> in °C.

Shale of south Texas). The sample was collected from roadside outcrops on U.S. Highway 90 west of Comstock, Texas, in the Val Verde Basin. On the kerogen typing chart (Fig. 1), sample 2 shows a mixed Type I/II kerogen. This rock was deposited in a marine environment (Lock and Peschier, 2006) with relatively low clastic input (high carbonate content, 54 wt.%), and the mixed Type I/II signature probably results from high sulfur concentrations in the OM (Type IIS kerogen; Orr, 1986). Vitrinite, inertinite, and solid bitumen are abundant (Fig. 2C–F). Solid hydrocarbons are variable in presentation, ranging from low-reflecting fluorescent bitumens which fill porosity in planktic foraminifera (Fig. 2E), to higher reflectance bitumens (Fig. 2F), indicating multiple formation and/or alteration processes. Amorphous organic matter (AOM) is dispersed in the mineral matrix and shows moderate fluorescence (Fig. 2D); some discrete algal bodies also are present (Fig. 2D).

#### 3.3. Huron Member of Ohio Shale

Sample 3 is a mature marine shale from the Upper Devonian Huron Member of the Ohio Shale. The sample was collected from roadside outcrops on U.S. Highway 58 in southwestern Virginia, USA, in the Appalachian Basin. This sample plots as a mature Type II kerogen (Fig. 1), consistent with regional studies of the depositional environment and thermal maturity (Ettensohn, 1998; Repetski et al., 2008). The sample contains weakly fluorescent *Tasmanites* telalginites (Fig. 3A, B). Inertinite is present in minor amounts; vitrinite is presumed present in similar low quantities but is difficult to distinguish from solid bitumen (Hackley et al., 2013; Ryder et al., 2013). Solid bitumen (as evidenced by void-filling texture) is the most abundant OM (Fig. 3A).

#### 3.4. Rodiles Formation

Sample 4 is a mature marl from the Jurassic Rodiles Formation in the Asturian Basin of northern Spain, collected from outcrops at Vega Beach. Organic petrography and chemistry of the Rodiles Formation from this area previously was described by Borrego et al. (1997). In the kerogen typing chart (Fig. 1) this sample shows evidence of weathering as indicated by high OI; this is confirmed by petrographic analysis which shows evidence of corrosion and pitting in the OM (Fig. 3C). In addition, this sample contains multiple populations of recycled vitrinite and low-reflecting semisunite which contribute to the high OI. Analysis of vitrinite

**Table 3**  
X-ray diffraction mineralogy of the ILS samples.

Sample	LOI	QTZ	FLD	CARB	Illite	KAOL	CHLR	PY	Other	ΣClay
1	23.64	22	3	51	7	0	0	0	11	7
2	5.65	36	0	54	0	5	0	3	2	5
3	8.78	38	4	0	48	0	1	3	2	49
4	2.37	17	0	41	31	1	5	3	1	38
5	3.41	28	2	0	46	7	15	0	0	68
6	0.54	19	1	64	12	0	0	1	1	12

Abbreviations: LOI, loss on ignition; QTZ, quartz; FLD, feldspar; CARB, carbonate; KAOL, kaolinite; CHLR, chlorite; PY, pyrite.

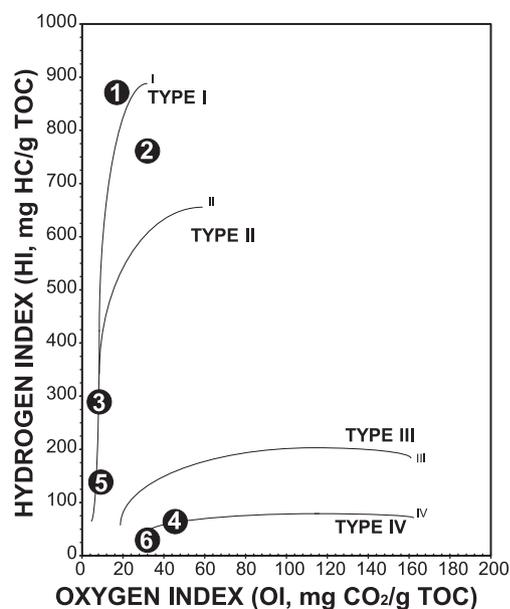


Figure 1. Kerogen typing for ILS samples from Rock-Eval pyrolysis.

reflectance in this sample is challenging because of the presence of void-filling bitumen with the same reflectance as indigenous vitrinite. Char particles are abundant; six and eight spindle calcareous micro-fossils also are present (Fig. 3D).

### 3.5. Pottsville Formation

Sample 5 is a mature carbonaceous mudstone collected from coal measures in the Pennsylvanian Pottsville Formation from the Appalachian Basin in Alabama, USA. The sample was collected from a two-inch borehole cored for shallow coal mining exploration. This sample contains abundant organic matter consisting of structured vitrinite and inertinite (Fig. 3E). On the kerogen typing chart (Fig. 1) this sample plots towards the Type I/II maturation trend; this may be due to high sulfur content in the OM and deposition in a strongly reducing near-shore sapropelic environment (e.g., Orr, 1986).

### 3.6. Pearsall Formation

Sample 6 is an overmature argillaceous carbonate from the Lower Cretaceous Pearsall Formation in the Maverick Basin of south Texas, USA. The sample was collected from three-inch core obtained from conventional oil and gas exploration. Organic petrography and chemistry of the Pearsall Formation from this area previously was described by Hackley (2012). Of the six samples examined for the ILS this was the most challenging to analyze due to low OM content (1.01 wt.% TOC; Table 2) and low S<sub>2</sub> for this sample results in an unreliable  $T_{\max}$  of 426 °C. Vitrinite, if present, grades into low-reflecting semifusinite (Fig. 3F); void-filling bitumens with the same reflectance as vitrinite(?) also are present. Euhedral, internally zoned carbonate [dolomite (?)] rhombs are common.

## 4. Results

Participants submitted their results to the convener (P. Hackley) via email; as results were submitted each participant was provided with preliminary feedback consisting of comparison of their individual result to the current group mean result for each sample

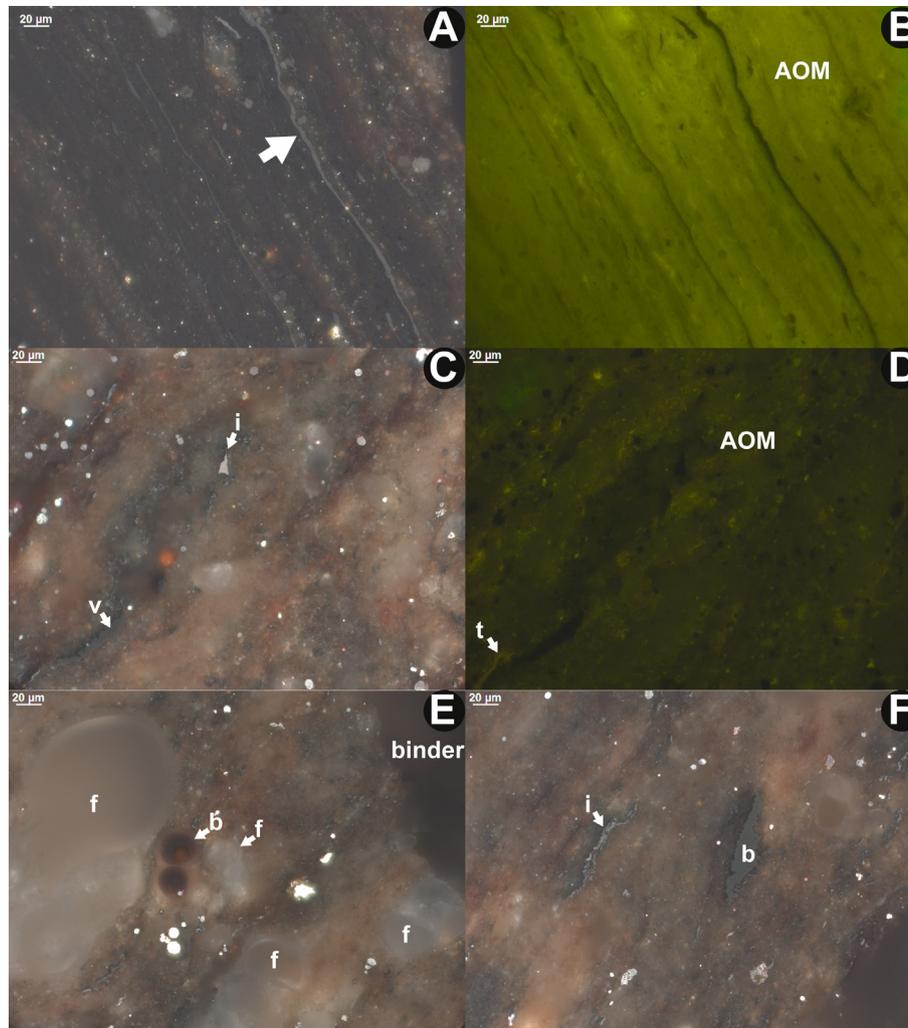
(with the exception that the first petrographer to submit results did not receive feedback). Because of this information exchange, several problems were discovered clearly related to instrument calibration and/or mis-labeling of samples during preparation. In these instances, participants submitted corrected results. In several other cases, participants provided measurement data but did not select a population of measurements representative of thermal maturity, or stated that no material representative of thermal maturity was present to measure. Some petrographers reported results which deviated from the group mean for reasons possibly related to maceral mis-identification; these participants also were given an opportunity to update their data. Four petrographers contacted on the basis of possible maceral mis-identification chose to provide updated results and of these four participants, three provided updated results for one sample only and one provided updated results for two samples. In no case where a petrographer submitted revised data, whether for issues related to calibration, preparation errors or maceral mis-identification, were final results considered 'excessively sanitized.' Two petrographers asked for and were provided supporting sample information (lithology, age, sedimentary basin) by the convener prior to beginning reflectance analysis. Results are compiled in Table 4 and illustrated graphically in Figure 4. Results compiled in Table 4 are not listed in the same order as the authors of this paper. All petrographers reported the same order of increasing thermal maturity in the sample order 1, 2, 3, 5, 4, and 6, with one initial exception (participant initially reported maturity 4 > 6) which was subsequently corrected by re-submission and may have been related to a preparation error (an oily film was present on the examination surface of sample 4 initially). One petrographer (N; Table 4) reported clearly erroneous values for  $R_0$  measured on sample 6 and did not provide updated results or clarification when contacted. As illustrated in Figure 4, relatively low group standard deviations (GSD) of 0.04–0.07 were obtained for samples 1, 2, and 5, whereas higher GSD values of 0.12–0.19 were achieved for samples 3, 4, and 6. Group standard deviation values are plotted as a function of  $R_0$  in Figure 5 showing the expected increase in GSD with advancing thermal maturity and increasing anisotropy of the organic matter. Sample 5 (Fig. 5) falls off this trend; this result also is expected as this is the only terrestrial sample analyzed, containing abundant and relatively homogeneous organic matter from a proximal source.

## 5. Discussion

### 5.1. Vitrinite versus bitumen reflectance

Five of the six samples (all except sample 5, terrestrial Pottsville Formation) contain solid bitumen, a residue of liquid hydrocarbon generation or of its cracking to gas (Curiale, 1986). Solid bitumens typically are distinguished from vitrinite on the basis of void-filling character (ASTM, 2014a). Similar to vitrinite, the reflectance of solid bitumen increases with advancing thermal maturity as the organic molecule condenses and aromatizes (Jacob, 1989). However, empirical calibrations developed to relate vitrinite reflectance to bitumen reflectance (Jacob, 1989; Landis and Castaño, 1995; Schoenherr et al., 2007) differ from each other and therefore applications of conversion factors should a priori be approached with caution.

Some participants acknowledged that some or all reflectance measurements reported from the Green River, Huron, Rodiles, and Pearsall samples were determined on solid bitumens. In these instances, the values reported were indistinguishable from values identified by others to be measured on vitrinite. Therefore, the bitumen reflectance measurements of individual petrographers were included in the evaluation of precision reported below. No



**Figure 2.** Photomicrographs illustrating organic petrographic features of the ILS samples. All photomicrographs under 500 × magnification using oil immersion. A. Sample 1 (Green River Formation, Mahogany Ledge oil shale) under white light illustrating narrow bands of low gray reflecting material measured for this exercise. B. Same field as A under blue light illustrating fluorescent amorphous organic matter (AOM). C. Sample 2 (Boquillas Formation) under white light illustrating small fragments of vitrinite (v) and inertinite (i) dispersed in the mineral matrix. D. Same field as C under blue light illustrating AOM and sparse telalginite (t). E. Sample 2 (Boquillas Formation) under white light illustrating planktic foraminifera (f) occasionally filled with low-reflecting solid bitumen (b). F. Sample 2 (Boquillas Formation) under white light illustrating void-filling solid bitumen (b) with higher reflectance than shown in E.

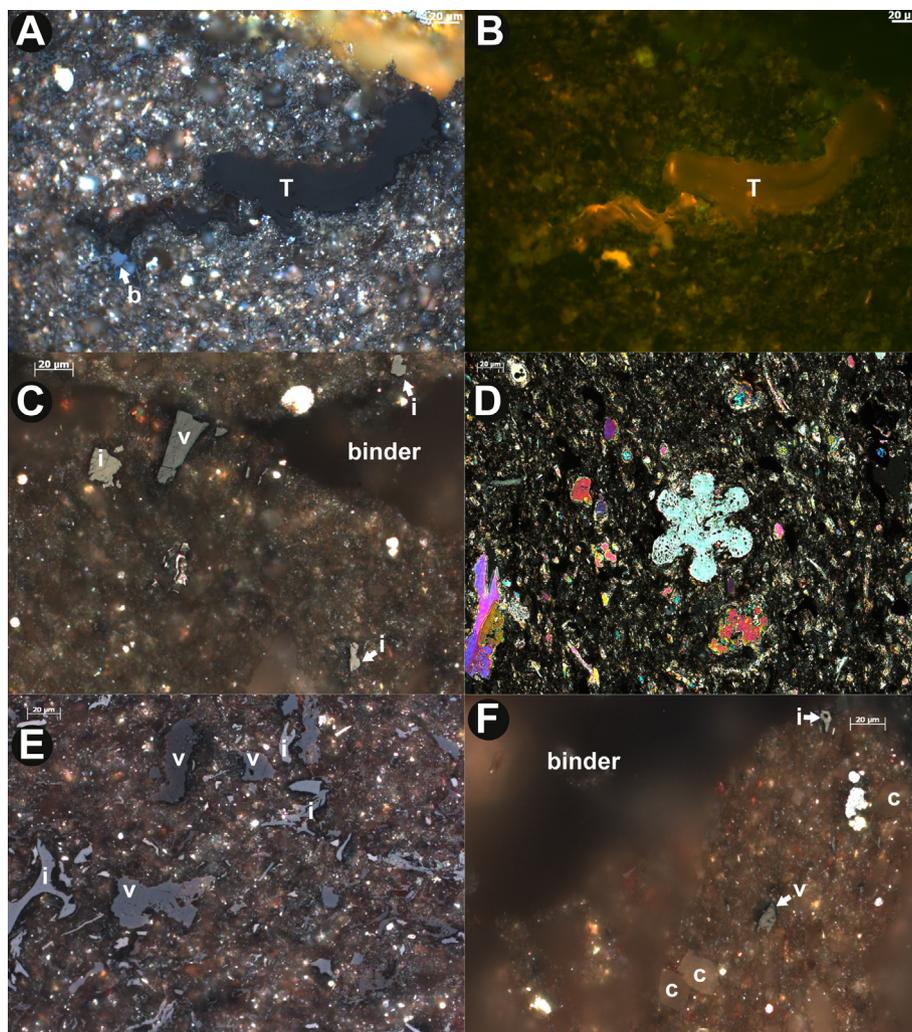
empirical conversion factors were applied to measurements reported on solid bitumens. Void-filling solid bitumens with reflectance clearly lower than co-occurring vitrinite are present in the Boquillas sample; however, no participant measured this material, probably because the solid bitumen is unmistakably void-filling in the primary porosity of foraminifera tests. No petrographer reported the presence of solid bitumen in the Pottsville sample. The observation that measurements determined on material identified as solid bitumen statistically are no different from measurements determined on material identified as vitrinite in this study also calls into question the validity of empirical reflectance conversion schemes. Similar to bitumen measurements, measurements identified as determined from semifusinite in sample 6 showed no statistical difference to measurements reported from vitrinite and were therefore included in the evaluation of precision.

During the planning for this ILS, the question was asked: is an ILS with kerogen concentration to remove solid bitumens (via solvent extraction) necessary to test the precision of D7708? However, at the 2011 and 2012 meetings of ICCP, participants agreed that benefits to identification of primary vitrinite may be

lost if the rock textures are removed by kerogen concentration. In general, the participants herein agree with the assertion by Barker (1996) that whole rock preparations which preserve rock texture are better for identification of primary vitrinite and for distinguishing vitrinite from solid bitumen. Moreover, for most laboratories participating in the ILS, kerogen concentration is a non-routine practice that typically is applied only for research samples and not to every-day work. Finally, solid bitumens are not universally dissolved by organic solvents (e.g., Jacob, 1989) and solvent extraction (as part of kerogen concentration) generally requires grinding of the sample to <200 μm, thereby making subsequent petrographic applications more challenging (Durand and Nicaise, 1980; Barker et al., 2007).

## 5.2. Evaluation of precision

The final results were submitted by the convener to the ASTM ILS Program (<http://www.astm.org/ILS/>) for evaluation via ASTM E691: Standard practice for conducting an interlaboratory study to determine the precision of a test method (ASTM, 2014c). The data



**Figure 3.** Photomicrographs illustrating organic petrographic features of the ILS samples. All photomicrographs under 500 × magnification using oil immersion. A. Sample 3 (Huron Member of the Ohio Shale) showing *Tasmanites* telaginite (T) and void-filling solid bitumen (b) in mineral matrix under white light. B. Same field as A under blue light. C. Sample 4 (Rodiles Formation) under white light illustrating fragments of vitrinite (v) and inertinite (i). D. Sample 4 (Rodiles Formation) in transmitted cross-polarized light showing calcareous micro-fossil. E. Sample 5 (Pottsville Formation) under white light showing abundant structured vitrinite (v) and inertinite (i). F. Sample 6 (Pearsall Formation) under white light showing vitrinite (?) with relief, morphology, and reflectance similar to inertinite (i) and euhedral carbonate [dolomite (?)] rhombohedrons (c).

were examined by ASTM ILS Program staff for consistency via ASTM E691 and several results were flagged as outliers. Outliers were identified by calculation of a between-laboratory consistency statistic,  $h$ , using the formula  $h = d/s_{\bar{x}}$ , where  $d$  = difference of the mean of an individual laboratory's duplicate measurements,  $\bar{x}$ , and the mean of duplicate measurements from all laboratories,  $\bar{\bar{x}}$ ,  $d = \bar{x} - \bar{\bar{x}}$ , and  $s_{\bar{x}}$  = standard deviation of  $d$  values for all laboratories in the ILS. Measurements with calculated  $h$  values greater than 2.63 were excluded as outliers (the value 2.63 is empirically-derived from the number of analysts, 28; see ASTM E691, Table 4) and the measurements which resulted in  $h$  outliers herein are indicated in Table 4 by gray shading. These measurements were not included in the following calculations of repeatability and reproducibility limits for the six tested shales compiled in Table 5.

Repeatability standard deviation ( $s_r$ ) is calculated using the formula (ASTM, 2014c):

$$s_r = \sqrt{\sum_1^p s^2/p}$$

where  $p$  is the number of participating analysts and  $s$  is the standard deviation of each laboratory's duplicate results. A reproducibility standard deviation ( $s_R$ ) is calculated using the formula (ASTM, 2014c):

$$(s_R) = \sqrt{(s_{\bar{x}})^2 + (s_r)^2(n-1)/n}$$

where  $n$  = the total number of measurements for each sample (ASTM, 2014c). Finally, a repeatability limit is calculated by the formula  $r = 2.8 s_r$ , and a reproducibility limit is calculated by  $R = 2.8 s_R$ .

Repeatability is defined by ASTM as precision under conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time (ASTM, 2014c). In other words, the repeatability limit is the acceptable difference in absolute values of reflectance that any individual operator could be expected to obtain from closely-spaced replicate measurements of reflectance for shale thermal maturity using the same equipment. Repeatability limits ranged from low values of 0.03 and 0.04 for lower maturity Green River and Boquillas

**Table 4**  
Reflectance data from ILS participants.

Laboratory		Sample											
		1A	1B	2A	2B	3A	3B	4A	4B	5A	5B	6A	6B
A	R <sub>0</sub>	0.33	0.33	0.51	0.49	0.87	0.85	1.16	1.12	1.09	1.04	1.74	1.74
	s.d.	0.04	0.03	0.07	0.06	0.08	0.09	0.14	0.10	0.07	0.09	0.19	0.21
	n	50	50	50	50	51	30	25	28	47	51	15	44
B	R <sub>0</sub>	0.35	0.36	0.56	0.60	0.77	0.82	1.24	1.19	1.05	1.05	1.56	1.52
	s.d.	0.04	0.04	0.09	0.08	0.09	0.08	0.08	0.11	0.06	0.05	0.15	0.15
	n	25	23	21	22	23	37	21	20	21	31	20	29
C	R <sub>0</sub>	0.35	0.34	0.59	0.58	0.71	0.72	1.01	0.92	0.94	0.93	1.12	1.02
	s.d.	0.02	0.03	0.06	0.05	0.10	0.05	0.08	0.10	0.07	0.08	0.14	0.10
	n	30	30	31	30	30	30	32	32	30	30	32	25
D	R <sub>0</sub>	n.r.	0.38	0.49	0.51	0.92	0.91	1.54	1.54	1.01	1.01	1.63	1.64
	s.d.	n.r.	0.06	0.07	0.08	0.07	0.09	0.19	0.17	0.04	0.05	0.23	0.16
	n	n.r.	8	10	10	15	12	16	23	22	24	20	21
E	R <sub>0</sub>	0.31	0.33	0.54	0.54	0.91	0.97	1.10	1.11	1.09	1.12	1.78	1.82
	s.d.	0.03	0.04	0.06	0.04	0.12	0.15	0.10	0.12	0.08	0.07	0.13	0.14
	n	20	27	27	26	26	22	17	20	42	50	20	24
F	R <sub>0</sub>	0.32	0.33	0.53	0.56	1.08	1.11	1.22	1.28	0.98	0.97	n.r.	n.r.
	s.d.	0.05	0.02	0.08	0.10	0.08	0.09	0.09	0.09	0.17	0.06	n.r.	n.r.
	n	12	6	8	5	4	4	6	4	3	9	n.r.	n.r.
G	R <sub>0</sub>	0.29	0.29	0.40	0.39	0.72	0.73	1.28	1.28	0.96	0.97	1.66	1.69
	s.d.	0.04	0.05	0.08	0.07	0.099	0.08	0.12	0.14	0.07	0.07	0.12	0.11
	n	30	26	30	26	28	13	24	27	30	30	26	13
H	R <sub>0</sub>	0.35	0.37	0.55	0.54	0.78	0.79	1.09	1.09	1.03	1.03	1.56	1.57
	s.d.	0.02	0.04	0.05	0.06	0.04	0.05	0.08	0.07	0.05	0.05	0.09	0.05
	n	52	26	56	51	37	39	26	30	49	51	3	10
I	R <sub>0</sub>	0.38	0.35	0.45	0.43	0.86	0.87	1.01	1.03	0.97	1.01	1.62	1.61
	s.d.	0.00	0.01	0.03	0.02	0.04	0.03	0.03	0.04	0.04	0.04	0.05	0.05
	n	21	19	19	23	9	14	33	21	26	25	12	8
J	R <sub>0</sub>	0.34	0.29	0.53	0.51	0.88	0.88	1.42	1.43	1.07	1.05	1.8	1.81
	s.d.	0.03	0.05	0.04	0.05	0.02	0.04	0.07	0.04	0.04	0.03	0.07	0.06
	n	27	71	28	33	16	47	31	23	67	51	10	17
K	R <sub>0</sub>	0.31	0.31	0.39	0.40	0.70	0.70	1.16	1.20	1.04	1.01	1.34	1.36
	s.d.	0.04	0.05	0.05	0.07	0.08	0.06	0.09	0.08	0.04	0.05	0.08	0.1
	n	23	20	20	22	20	21	20	24	21	22	21	21
L	R <sub>0</sub>	0.26	0.27	0.49	0.49	0.82	0.82	1.19	1.23	0.96	0.95	1.68	1.67
	s.d.	0.05	0.04	0.04	0.05	0.07	0.09	0.08	0.06	0.10	0.11	0.14	0.14
	n	30	30	20	20	20	20	20	20	20	20	20	20
M	R <sub>0</sub>	0.31	0.32	0.46	0.47	0.93	0.93	1.09	1.15	0.97	0.98	1.68	1.67
	s.d.	0.02	0.02	0.05	0.04	0.08	0.09	0.05	0.06	0.05	0.04	0.09	0.08
	n	21	20	22	15	23	24	7	14	30	29	7	5
N	R <sub>0</sub>	0.27	0.27	0.55	0.55	0.69	0.68	0.82	0.82	0.94	0.93	0.33	0.33
	s.d.	0.05	0.05	0.09	0.08	0.09	0.09	0.08	0.10	0.09	0.08	0.10	0.10
	n	124	82	95	95	85	92	28	34	82	100	37	35
O	R <sub>0</sub>	0.26	0.26	0.44	0.45	0.52	0.53	1.12	1.13	0.90	0.89	1.29	1.29
	s.d.	0.04	0.05	0.04	0.06	0.05	0.05	0.04	0.06	0.04	0.05	0.04	0.05
	n	20	20	20	12	10	10	20	20	20	20	11	11
P	R <sub>0</sub>	0.32	0.29	0.80	0.74	0.85	0.78	1.16	1.21	0.92	0.94	1.54	1.41
	s.d.	0.07	0.04	0.24	0.18	0.12	0.06	0.06	0.16	0.06	0.06	0.11	0.59
	n	11	3	8	5	16	12	19	12	19	19	12	3
Q	R <sub>0</sub>	0.25	0.26	0.43	0.39	0.75	0.68	1.02	1.22	0.96	0.91	1.34	1.20
	s.d.	0.03	0.03	0.07	0.06	0.07	0.13	0.10	0.11	0.09	0.08	0.07	0.17
	n	25	20	23	20	9	26	14	13	19	20	17	25
R	R <sub>0</sub>	0.26	0.26	0.46	0.44	0.64	0.66	1.19	1.17	1.04	1.06	1.76	1.74
	s.d.	0.05	0.04	0.04	0.09	0.15	0.15	0.11	0.10	0.06	0.06	0.10	0.20
	n	32	37	22	17	32	31	17	17	45	45	12	20
S	R <sub>0</sub>	0.31	0.30	0.49	0.52	1.00	0.92	1.19	1.13	0.99	0.98	1.63	1.62
	s.d.	0.03	0.03	0.04	0.05	0.08	0.08	0.12	0.16	0.08	0.08	0.09	0.10
	n	20	19	8	12	21	20	5	6	32	30	7	7
T	R <sub>0</sub>	0.20	0.22	0.54	0.54	0.71	0.78	1.16	1.11	1.00	1.03	1.25	1.23
	s.d.	0.03	0.04	0.14	0.15	0.09	0.08	0.11	0.14	0.05	0.06	0.12	0.12
	n	25	21	25	29	32	33	36	33	66	52	23	24
U	R <sub>0</sub>	0.32	0.33	0.51	0.48	0.66	0.65	1.07	1.03	0.87	0.87	1.48	1.49
	s.d.	0.07	0.04	0.10	0.07	0.05	0.06	0.09	0.04	0.07	0.08	0.11	0.10
	n	50	50	50	42	50	50	38	40	50	47	50	27
V	R <sub>0</sub>	0.30	0.31	0.45	0.47	0.80	0.78	1.35	1.32	0.86	0.88	1.50	1.50
	s.d.	0.04	0.04	0.05	0.04	0.08	0.08	0.10	0.10	0.05	0.07	0.08	0.13
	n	40	40	20	20	33	24	22	21	41	47	20	31
W	R <sub>0</sub>	0.29	0.31	0.57	0.56	0.76	0.75	1.20	1.15	0.98	1.01	1.77	1.72
	s.d.	0.04	0.04	0.10	0.08	0.08	0.07	0.10	0.08	0.09	0.07	0.12	0.11
	n	43	30	19	14	34	24	9	19	35	25	32	29
X	R <sub>0</sub>	0.31	0.29	0.37	0.42	0.68	0.70	1.18	1.12	1.02	1.00	1.34	1.35
	s.d.	0.01	0.02	0.04	0.04	0.06	0.07	0.09	0.08	0.03	0.05	0.06	0.08
	n	24	20	20	20	26	21	22	21	25	21	25	22

Table 4 (continued)

Laboratory		Sample											
		1A	1B	2A	2B	3A	3B	4A	4B	5A	5B	6A	6B
Y	R <sub>o</sub>	0.35	0.35	0.67	0.65	0.83	0.83	1.23	1.23	0.95	0.96	1.56	1.53
	s.d.	0.02	0.03	0.05	0.06	0.07	0.07	0.08	0.08	0.03	0.04	0.08	0.09
	n	50	50	50	50	50	50	50	50	50	50	50	50
Z	R <sub>o</sub>	0.31	0.29	0.42	0.44	0.82	0.83	1.15	1.15	1.01	1.03	1.33	1.37
	s.d.	0.02	0.03	0.06	0.06	0.1	0.06	0.07	0.09	0.08	0.06	0.12	0.13
	n	32	34	36	23	37	26	28	39	44	38	43	31
AA	R <sub>o</sub>	0.25	0.26	0.54	0.53	0.81	0.77	1.48	1.44	1.06	1.04	1.67	1.62
	s.d.	0.03	0.04	0.06	0.05	0.06	0.05	0.08	0.08	0.08	0.07	0.08	0.05
	n	22	27	12	16	12	13	13	9	59	81	9	5
BB	R <sub>o</sub>	0.27	0.25	0.52	0.51	0.95	0.94	1.18	1.18	1.06	1.06	1.43	1.42
	s.d.	0.04	0.05	0.08	0.07	0.05	0.04	0.08	0.07	0.04	0.05	0.14	0.12
	n	30	30	30	30	30	30	30	30	30	30	19	30

R<sub>o</sub> in %; s.d., standard deviation; n, number of measurements; n.r., no report; values in bold italic were identified as solid bitumen reflectance; values identified in bold were identified as semifusinite reflectance. Values shaded in gray were identified as outliers by application of ASTM E691 and were not included in the calculations for repeatability and reproducibility limits and other statistics compiled in Table 5. Data are not listed in the same order as participants listed as co-authors.

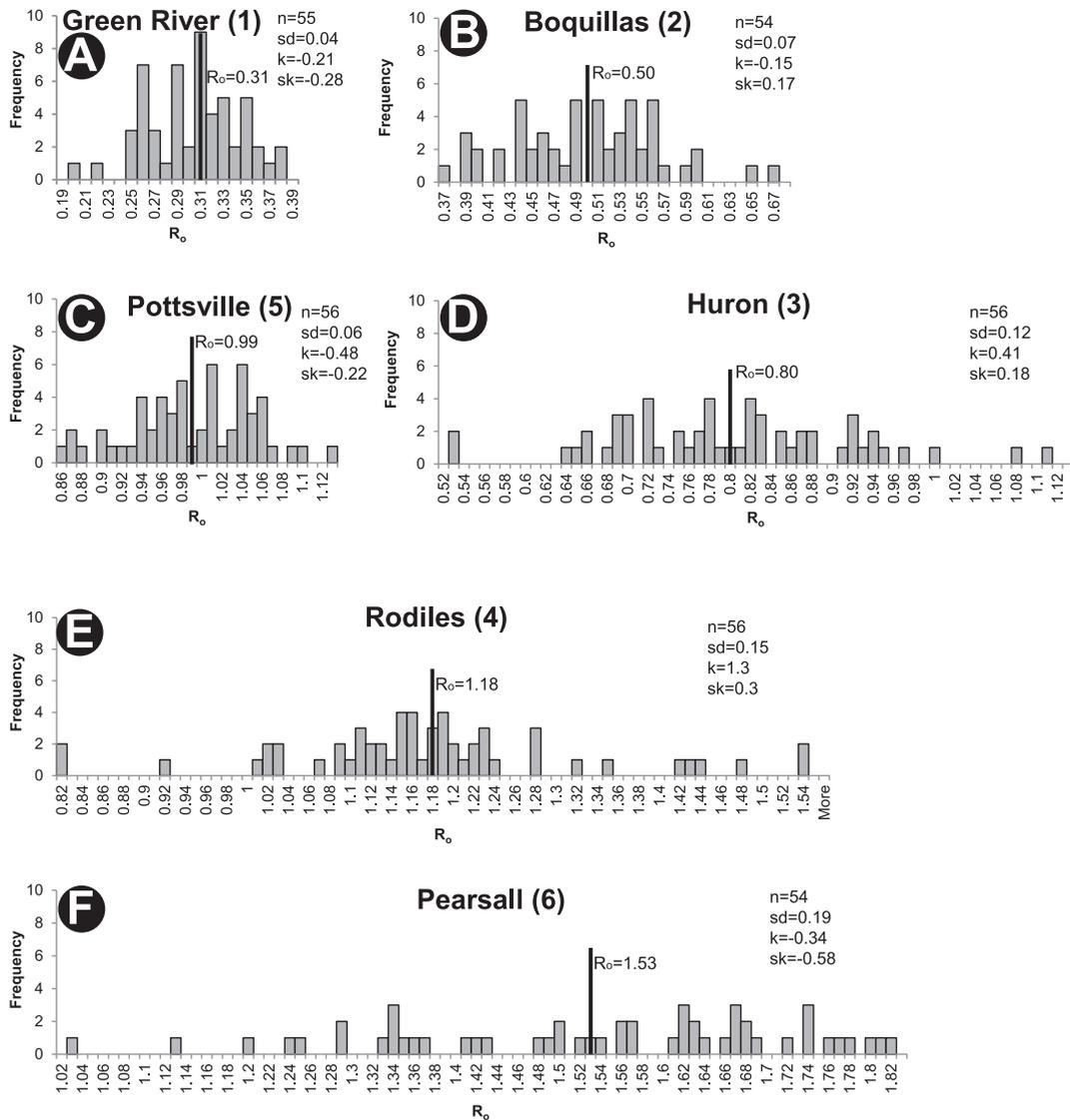
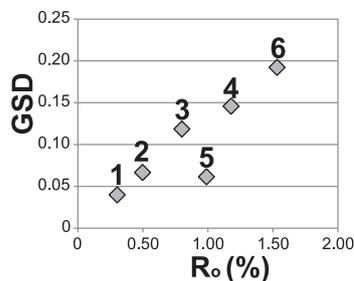


Figure 4. Histograms illustrating reflectance results for the ILS samples. A. Green River Formation (sample 1). B. Boquillas Formation (sample 2). C. Pottsville Formation (sample 5). D. Huron Member of Ohio Shale (sample 3). E. Rodiles Formation (sample 4). F. Pearsall Formation (sample 6). Mean R<sub>o</sub> value shown by thin black vertical line. Abbreviations: n, number of measurements; sd, standard deviation; k, kurtosis; sk, skewness. Outliers (determined via ASTM E691, see text) for Boquillas and Pearsall not shown. The number of measurements (n) results from replicate measurements by twenty-eight analysts (2\*28 = 56) and for cases of n < 56 where an analyst did not report a measurement (e.g., analyst F, sample 6; Table 4) or reported only one measurement (analyst D, sample 1; Table 4).



**Figure 5.** Group standard deviation (GSD) as a function of  $R_0$  for the six samples analyzed in this ILS.

samples, respectively (Table 5) to high values of 0.10 for the Pearsall and 0.11 for the Rodiles. Compare these repeatability limits to that of 0.02% absolute reflectance determined in ILS results for vitrinite reflectance of bituminous coals (ASTM, 2014b). However, it should be noted that vitrinite in coal is autochthonous, and generally homogeneous and abundant, whereas organic matter in shale generally is allochthonous, highly variable and sparse.

Reproducibility is defined by ASTM as precision under conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment (ASTM, 2014c). In other words, the reproducibility limit is the maximum difference in absolute values of reflectance between measurements made in two different laboratories which are acceptable and comparable with each other. Reproducibility limits ranged from low values of 0.11 and 0.17 in the Green River and Pottsville samples, respectively, to high values of 0.41 and 0.54 determined for the Rodiles and Pearsall samples, respectively. Compare these reproducibility limits to that of 0.06% absolute reflectance determined for bituminous coals (ASTM, 2014b). Again, the same statement made above applies regarding the origin, diversity and abundance of organic matter in shale compared to coal. Despite that reproducibility limits compare unfavorably to what is achievable for bituminous coal, the current study shows significant improvement overall when compared to historical round robin exercises performed within ICCP Commission II on the reflectance of dispersed organic matter (Borrego, 2009). Borrego (2009) evaluated the precision of dispersed vitrinite reflectance round robins exercises performed over the years 1981–2008 ( $n = 23$  samples) and found reproducibility limits of 0.09–0.85 (mean reproducibility limit, 0.41) according to the formula  $R = 2.8 \cdot GSD$ . While this is a slightly different computation for reproducibility limit than by ASTM E691, the results herein compute to identical values as derived by ASTM E691 using this alternative formula, and suggest significant improvement in

**Table 5**

Summary precision statistics for reflectance of the ILS samples. Reprinted, with permission, from ASTM D7708 Standard test method for microscopical determination of the reflectance of vitrinite dispersed in sedimentary rocks (ASTM, 2014a), copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. A copy of the complete standard may be obtained from ASTM International, [www.astm.org](http://www.astm.org).

Sample ID	Formation	$R_0$	s.d.	$s_r$	$s_R$	$r$	R
1	Green River	0.31	0.04	0.01	0.04	0.03	0.11
2	Boquillas	0.50	0.07	0.02	0.07	0.04	0.19
3	Ohio/Huron	0.80	0.12	0.02	0.12	0.07	0.33
4	Rodiles	1.18	0.14	0.04	0.15	0.11	0.41
5	Pottsville	0.99	0.06	0.02	0.06	0.04	0.17
6	Pearsall	1.53	0.19	0.03	0.19	0.10	0.54

$R_0$  in %; s.d., standard deviation;  $s_r$ , repeatability standard deviation;  $s_R$ , reproducibility standard deviation;  $r$ , repeatability limit; R, reproducibility limit.

precision over the historical results with a mean reproducibility limit of 0.29% absolute reflectance. Considered only for samples with low anisotropy ( $R_0 < 1.0\%$ ), results herein (mean reproducibility limit, 0.20) are improved significantly compared to historical results (mean reproducibility limit, 0.35). Likewise, for samples with high anisotropy ( $R_0 > 1.0\%$ ) results herein (mean reproducibility limit 0.48) are improved compared to historical results (mean reproducibility limit, 0.68). Considered collectively just in terms of reproducibility limits, this analysis suggests that use of a common methodology (ASTM D7708) may result in better inter-laboratory precision for reflectance measurements of dispersed organic matter in shale. However, it should be emphasized that the ILS studies considered in Borrego (2009) were inhomogeneous and not all were conducted primarily for the purpose of reflectance analysis.

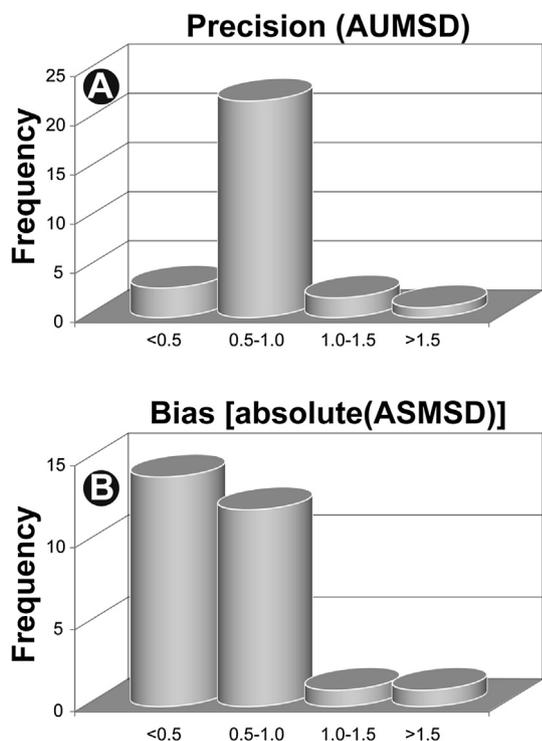
The results of the current ILS also were evaluated by calculation of the signed multiple of the standard deviation, SMSD, according to the formula:

$$\text{SMSD} = (X_i - GM) / \text{GSD}$$

where  $X_i$  is the average reflectance,  $GM$  is group mean, and  $GSD$  is group standard deviation, and by the unsigned multiple of standard deviation (USMD, absolute value of SMSD). The SMSD is a measure of bias whereas the USMD is a measure of precision because both of these statistics evaluate the distance to the mean of any given value. Values for SMSD  $< 1.5$  are considered acceptable (Borrego et al., 2006; Mendonça Filho et al., 2010) and the ICCP Accreditation programs for single coal and dispersed vitrinite reflectance analysis consider values  $> 1.5$  for average USMD (AUSMD) to be failing. For all petrographers, the AUSMD was  $< 1.5$  (Fig. 6A) except for petrographer N (Table 4), who reported low  $R_0$  values of 0.33% for high maturity sample 6 (it is suspected that this was due to measurements of carbonate reflectance). Most participants had values of AUSMD between 0.5 and 1.0 indicating moderate precision. Excluding the  $h$  outliers, the AUSMD for all petrographers for all samples was 0.80, which is slightly higher than the AUSMD of 0.76 for all petrographers for all samples in historical exercises (Borrego, 2009). Six of the twenty-eight participants did not hold a current ICCP accreditation in dispersed vitrinite reflectance measurement; however, no statistical difference was observed between the precision of accredited vs. non-accredited analysts (comparing AUSMD for both groups with  $h$  outliers excluded). Most participants showed low levels of bias (Fig. 6B) as evaluated by the ASMSD (absolute values). A chart showing ASMSD (absolute values) vs. AUSMD (Fig. 7) indicates possible calibration problems for participants N and O where distances to the mean value are high and SMSD is consistently with the same sign. A relatively high value of AUSMD and low ASMSD (absolute) for participant C may indicate potential maceral identification issues.

### 5.3. Other considerations arising from the ILS

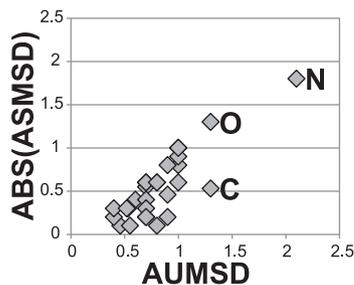
Publications describing application of dispersed vitrinite reflectance analyses to thermal maturity of sedimentary basins first appeared over forty years ago (Teichmüller, 1971; Robert, 1971; Castaño and Sparks, 1974; Bostick, 1974) and according to Mukhopadhyay (1994) the methodology was first applied in 1951–1952 by Marlies Teichmüller. However, experience has shown that reproducibility in dispersed vitrinite reflectance measurements can be extraordinarily difficult to achieve (e.g., Dembicki, 1984; Borrego, 2009). The evaluation of precision presented above shows that improvement has been accomplished by use of a common methodology (ASTM D7708) yet there remains significant room for optimization. For instance, the distribution of



**Figure 6.** A. Histogram of average unsigned multiple of standard deviation (AUMSD) values for participants in the ILS. B. Histogram of absolute values of average signed multiple of standard deviation (ASMSD) for participants in the ILS.

samples without a reporting template in this ILS resulted in widely varying results with respect to the presentation of data, ranging from simple columns of numbers to pages of interpretive material and accompanying photomicrographs. Ultimately, this lack of uniformity in reporting leads to variable data quality. Some participants also had difficulty meeting the minimum reporting requirements as specified in ASTM D7708 section 11, e.g., recording the presence or absence of fluorescence, and/or the distinguishing features of organic matter. To address the variability in reporting and improve the likelihood that users will meet the minimum reporting requirements, the ILS participants agreed that a reporting template was a necessary addition to D7708. An example (Figure 8) will be included in the 2014 version of the D7708 test method. This template is intended as an example report only; users of D7708 do not have to reproduce it exactly.

The ILS participants agreed that the current reporting requirements of D7708 are stringent and difficult to follow; however,



**Figure 7.** Absolute values of average signed multiple of standard deviation (ASMSD) as a function of average unsigned multiple of standard deviation (AUMSD) values, illustrating possible calibration issues for participants O and N and possible identification issues for participant C.

some results of the ILS suggested that additional requirements could be a benefit. For instance, participants whose instrumentation did not allow visualization of fluorescence in sample 3 reported results that diverged significantly from the group mean: one petrographer mis-identified *Tasmanites* as vitrinite because of the apparent absence of fluorescence, while another analyst unable to see *Tasmanites* fluorescence in this sample reported significantly higher values than the group mean reflectance value. While these are cases of mis-identification and bias, respectively, they illustrate that the type and sensitivity of microscope equipment can influence the outcome of a reflectance measurement. The possibility of adding additional reporting requirements to D7708 related to specifying equipment type was considered at the 2013 ICCP meeting but not supported by the majority.

Another consideration discussed at the 2013 ICCP meeting was determination of “acceptable” standard deviation limits for individual dispersed vitrinite reflectance analyses. A wide range in reported standard deviation from some participants, e.g., for samples 4 and 6 (Table 4) may indicate that measurements were taken on different populations of vitrinite or on different components with variable reflectance. While it is difficult to establish fixed limits for “acceptable” standard deviation in different types of samples, this sort of information would be very useful to help to select the indigenous vitrinite population in the sample.

Some participants in the ILS had difficulty obtaining the minimum of 20 measurements for compliance with D7708, in particular for the organic-lean samples 4 (Rodiles) and 6 (Pearsall). Since this is a common-place occurrence and since these rocks are representative of rocks currently being analyzed in the oil and gas industry, participants agreed to add a statement to the reporting requirements that non-compliant values (<20 measurements) can be used as a qualitative thermal maturity indicator.

#### 5.4. Future directions

It was observed by many participants from academic, government, and oil company settings that analysis of the samples in the ILS was more difficult than typical work because of the absence of supporting information such as Rock-Eval, TAI/SCI, and other geological data. To test whether such supporting information will improve precision, a future ILS may use two similar samples, one sent to participants with supporting information and one without.

Poor reproducibility limits derived for the high maturity samples in this ILS may be in part related to abundance of organic matter. As described above, many participants could not find organic matter to determine the minimally compliant number of 20 measurements for samples 4 and 6. To test whether abundance or anisotropy of organic matter is responsible for poor reproducibility limits at high maturity, a future ILS may use high maturity samples with abundant organic matter.

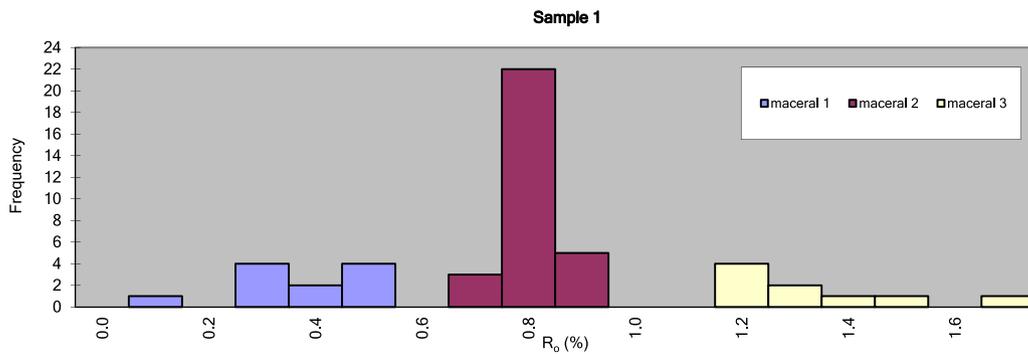
Finally, while this ILS has shown that precision in reflectance measurements can be improved by use of a common methodology, the identification of primary vitrinite (first cycle, indigenous) in dispersed organic matter remains problematic. In particular, primary vitrinite can be difficult to distinguish from similar macerals such as solid bitumen, bituminite, recycled vitrinite, and low-reflecting semifusinite. Zooclasts also can appear similar to vitrinite. To help improve the identification and distinction of these macerals, several efforts to develop a supplemental image atlas of dispersed organic matter in sedimentary rocks currently are underway. The USGS has developed a preliminary online atlas of organic matter in shales and coals (Valentine et al., 2013) and similar efforts to create a petrographic image database are under development by ICCP. The ICCP also offers regular training courses in the petrography of dispersed organic matter, provides well-

**DISPERSED VITRINITE REFLECTANCE REPORT**

**SAMPLE INFORMATION**

**RESULTS**

Submitted by: Anybody	No. measurements: 30
Date Submitted: Any Date	maceral type: maceral 2
Project: Any Project	mean $R_o$ (%): 0.76
	s.d.: 0.04
Sample ID: <b>Sample 1 (Client ID)</b>	Example Photograph:
Lab ID: 1	Place Example Photomicrograph of maceral 2 here
Sample Type: core/cuttings/outcrop	
Date Analyzed: Date of Analysis	
Operator: Analyst 1	
Protocol: ASTM D7708	
non-compliant analyses (<20 measurements) may be used as qualitative thermal maturity indicator	



**DATA**

0.30	0.45	0.73	1.23	0.81	0.74
0.30	0.78	0.79	1.24	0.82	0.76
0.04	0.77	0.80	1.35	0.83	0.71
0.21	0.74	1.12	2.10	0.77	0.71
0.22	0.70	1.13	2.30	0.78	
0.33	0.68	1.70	1.10	0.79	
0.32	0.67	1.45	0.77	0.78	
0.45	0.71	1.78	0.78	0.78	
0.44	0.71	2.10	0.79	0.74	
0.43	0.72	1.18	0.80	0.75	

**All Data:** min: 0.04 max: 2.30

**Maceral 2 only:** min: 0.67 max: 0.83

**COMMENT (reporting requirements referenced to ASTM D7708 section number)**

Organic-rich sample with >20 organic fragments to measure (11.1.1). Maceral 2 represents indigenous thermal maturity (some petrographers list level of confidence, poor, good, etc). Organic fluorescence not present (11.1.3). Prepared via ASTM D2798 (11.1.2), no repolish or additional preparation necessary (11.1.6). Whole rock preparation, black shale, >3 mm top-size, carbonate-poor, quartz-rich (11.1.2). No supporting data (11.1.4 and 11.1.8). Maceral 2 distinguished from macerals 1 and 3 by form, texture and reflectance (11.1.5). Polish quality 1A (11.1.7).

Lab Name	ICCP/DOMVR/##-##	Lab Contact Information
Address	expires 12/31/2014	Lab Contact Information

**Figure 8.** Example reporting template in D7708 to reduce variability and harmonize quality of reflectance data reports from individual laboratories. Reprinted, with permission, from ASTM D7708 Standard test method for microscopical determination of the reflectance of vitrinite dispersed in sedimentary rocks (ASTM, 2014a), copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. A copy of the complete standard may be obtained from ASTM International, [www.astm.org](http://www.astm.org).

characterized sample sets with supporting information and runs an accreditation program in dispersed vitrinite reflectance analysis.

**6. Summary and conclusions**

An international interlaboratory study to develop precision statistics for ASTM D7708 Standard test method for microscopical

determination of the reflectance of vitrinite dispersed in sedimentary rocks was conducted using six shale samples of varied origin and thermal maturity. Twenty-eight analysts from twenty-two laboratories participated. Repeatability limits (intra-laboratory) ranged from 0.03 to 0.11% absolute reflectance, whereas reproducibility limits (interlaboratory) ranged from 0.12 to 0.54% absolute reflectance. The repeatability and reproducibility

limits were poorer for samples at higher maturity and with lower organic content except for a carbonaceous mudstone sample which contained abundant and homogeneous proximal terrestrial kerogens. Petrographic distinction of solid bitumens from vitrinite was difficult when vitrinite was present with reflectance ranges overlapping the other components. However, reflectance values from material reported as bitumen were identical to reflectance values reported from vitrinite, suggesting empirical reflectance conversion schemes should be treated cautiously. The reproducibility limits observed herein compare favorably to reproducibility limits from historical interlaboratory exercises, suggesting use of a common methodology (D7708) improves interlaboratory precision. In some cases, participants had difficulty meeting the reporting requirements of D7708, prompting inclusion of a common reporting template in the test method to improve data quality. Future work is required to improve reproducibility limits in high maturity, organic-lean shales and move towards the development of online photographic image databases to assist in the identification of organic materials in shale.

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Figure 8 and Table 5 are reprinted, with permission, from ASTM D7708 Standard test method for microscopical determination of the reflectance of vitrinite dispersed in sedimentary rocks (ASTM, 2014a). Definitions of repeatability and reproducibility are reprinted, with permission, from ASTM E691 Standard practice for conducting an interlaboratory study to determine the precision of a test method (ASTM, 2014c). ASTM standards are copyrighted by ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Copies of the complete standards may be obtained from ASTM International, [www.astm.org](http://www.astm.org). Philip Godorov and Caitlin Farrell of the ASTM ILS program processed the ILS data and prepared drafts of the precision statement and research report. Frank Dulong (USGS) provided XRD analyses of the samples. Reviews by Ricardo Olea (USGS) and two anonymous journal reviewers improved this manuscript. Justin Birdwell, Peter Warwick, Cathy Enomoto (USGS) and Richard Carroll (Alabama Geological Survey) contributed samples for this work. Alana Bove packaged and distributed the samples for the ILS. Any use of trade, product, or firm names herein is for descriptive purposes only and does not imply endorsement by the U.S. Government. This paper is dedicated to the memory of Alan C. Cook who was always encouraging and fundamentally involved with the initiation and early phases of the current work.

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