Identification of Thermal Maturity -Relevant Organic Matter in Shale Working Group Report 2021

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The Identification of Thermal Maturity-Relevant Organic Matter in Shale Working Group of the ICCP was established in 2008 to provide guidelines for identifying and measuring the reflectance of the population of dispersed organic matter that is relevant to thermal maturity determination. Information products published by the working group include ASTM D7708 Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks (ASTM, 2014) and two interlaboratory studies (ILS) to determine precision statistics for this ASTM test method (Hackley et al., 2015, 2020). Poor reproducibility of solid bitumen and vitrinite reflectance measurements in the second ILS (Hackley et al., 2020) suggested that further work is required to standardize the identification of thermal maturity-relevant dispersed organic matter for reproducible reflectance measurements. Thus, at the 2019 ICCP meeting in The Hague, Netherlands, the working group decided to pursue additional ILS via image-based approaches to improve reproducibility.

Working group activity in 2021 included a pilot-scale, imaged-based study to identify organic matter types in six shale samples from the North American Bakken, Woodford, Eagle Ford, Marcellus, Haynesville, and Barnett unconventional resource plays. A total of fifty incident white light photomicrographs taken at 500x magnification under oil immersion were selected from images which had been previously collected during characterization of these six samples for the second ILS (Hackley et al., 2020). The images were collated into a Microsoft PowerPoint file and marked with two or three arrows each pointing to a maceral (Figure 1) to be identified by the organic petrographer in an accompanying Microsoft Excel file.

The Excel file also contained a minimal amount of supporting information including formation name, total organic carbon (TOC) content, T_{max} (°C), and solid bitumen reflectance (BR_o, %). The PowerPoint and Excel file (with instructions) were circulated between the five active U.S. Geological Survey organic petrographers (Paul Hackley, Javin Hatcherian, Jennifer Rivera, Maggie Sanders, and Brett Valentine) as a pilot-scale study prior to planned wider distribution amongst the ICCP community. The instructions directed the petrographer to identify the selected maceral from a multiple-choice list of potential identifications, which included solid bitumen (sb), vitrinite (v), inertinite (i), micrinite (m), can't determine (?), or other (for which the petrographer could submit a different answer).

The exercise took the petrographers between thirty minutes to two hours to complete, depending on the level of petrographic experience with identifying dispersed organic matter. Perfect agreement (i.e., all five petrographers selected the same identification for a particular location) of organic matter identification was positively correlated with increasing TOC content (Figure 2).

Perfect agreement decreased in order from Woodford (78%), Bakken (50%), Eagle Ford (45%), Marcellus (45%), Haynesville (19%), to Barnett (13%) (Figure 2a). The same order was followed for perfect plus good agreement (i.e., four of five petrographers selected the same identification for a particular location) for the top three samples: Woodford (83%), Bakken (75%), and Eagle Ford (69%). The Woodford and Bakken samples with the highest TOC had the lowest thermal maturity, suggesting that the level of agreement for organic matter identification is affected both by abundance of organic matter and its relative thermal maturity. A high level of agreement for organic matter identification in the Eagle Ford is thought to be due to the common occurrence of void-filling solid bitumen occupying the spherical chambers of planktic *Globigerina* foraminifera, which was almost always correctly identified.

Based on the answers submitted by the petrographers, the selected maceral was sometimes identified as vitrinite if it was larger or visually different than the network or groundmass solid bitumen in the same photomicrograph. This tendency was previ-

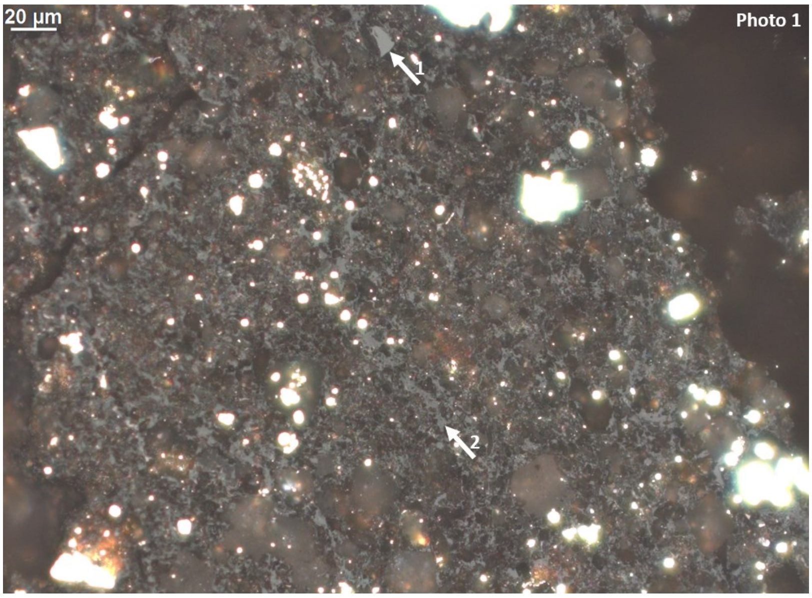

Figure 1. Example image from Bakken shale with two locations requested for organic matter identification.

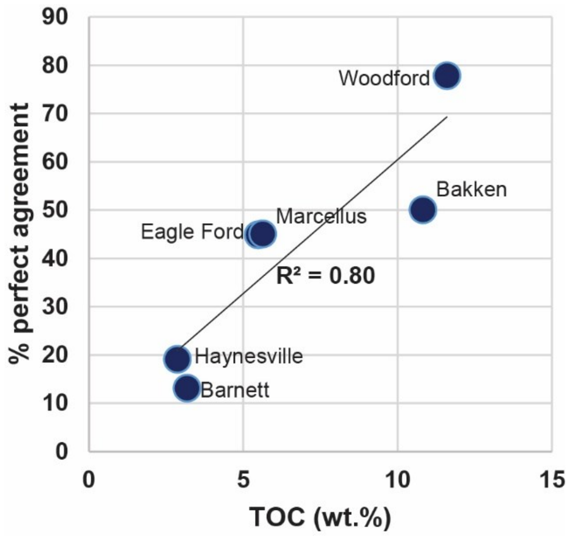

Figure 2. Graph illustrating the percentage of perfect agreement (5 of 5 petrographers) versus total organic carbon (TOC) content.

ously noted by Hackley et al. (2020). If the selected maceral was a small but discrete inertinite fragment it was sometimes identified as micrinite. This identification is perhaps due to the ASTM petrographic standard for metallurgical bituminous coals (D2799, ASTM, 2021) in which the term micrinite is assigned if the inertinite fragment is "generally nonangular, exhibiting no relict plant cell wall structure, smaller than 10 µm and most commonly occurring as particles around 1- to 5-µm diameter." Conversely, the selected organic maceral was sometimes identified as inertinite instead of micrinite if micrinite granularity was not clear in the image. This discrepancy may be due in part to image quality and the inability to resolve granularity. Similarly, if solid bitumen contained a granular texture, it was sometimes identified as micrinite.

Some of the comments provided by the participants suggested a low confidence level in maceral identifications related to distinguishing solid bitumen from vitrinite, which is the main issue examined by this working group. Participants cited a training bias to look for vitrinite, possibly leading to the observation noted above where vitrinite was identified if the selected maceral was larger or visually different than the network or groundmass solid bitumen in the same photomicrograph. Participants also noted differences in white balance among the fifty reflected light images of the exercise (which were collected over the course of about a year), suggesting that improvements could be made to image-based approaches by collecting images under similar microscope working conditions. Participants noted the image-based approach added an additional layer of difficulty in that the sample could not be scanned to locate additional macerals for comparison. To improve the image-based approach, the participants suggested including 1) a higher magnification image of the selection (e.g., 1000x), 2) a reflectance measurement image of the same brightfield image, 3) a comment field in the answer sheet to indicate level of confidence in identification or other general comments, and 4) guidance that not all maceral types are present in every image.

The working group is also engaged in the effort to update and ballot ASTM for the renewal of the D7708 test method, which will be withdrawn if it is not updated in 2022. The ICCP user community has been solicited for comments since 2019 and many suggestions have been received for ballot. Some of these include the explicit disallowance of sample preparation via ion milling, a note on inaccurate calculation of the reflectance of translucent standards by their refractive index, expansion of calibration methodology, removal of the preparation grading scheme (which is almost universally ignored by users), and many other suggestions. Please do not hesitate to contact Paul Hackley (phackley@usgs.gov) if you have thoughts to contribute on updating ASTM D7708. ASTM will ballot modifications suggested by the user community in July 2022.

References

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ASTM, 2021. D2799-21 Standard test method for microscopical determination of the maceral composition of coal: Petroleum products, lubricants, and fossil fuels; Gaseous fuels; coal and coke, sec. 5, v. 05.06. ASTM International, West Conshohocken, PA.

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I am sure we all miss these days!
Looking forward to dancing together in India (2022) or Patras in 2023. Stavros, hoping you can show us how it's done!
Photograph left : 1999, Bucharest ICCP Meeting; right Kolkata, 2014.