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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 ×) 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).