Designation: D7708 – 14

Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks[1]

This standard is issued under the fixed designation D7708; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the microscopical determination of the reflectance measured in oil of polished surfaces of vitrinite dispersed in sedimentary rocks. This test method can also be used to determine the reflectance of macerals other than vitrinite dispersed in sedimentary rocks.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D121 Terminology of Coal and Coke
D388 Classification of Coals by Rank
D2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light
D2798 Test Method for Microscopical Determination of the Vitrinite Reflectance of Coal
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—For definitions of terms, refer to Terminology D121.

3.2 *Abbreviations:*

3.2.1 $R_o ran$—mean random reflectance measured in oil. Other organizations may use other abbreviations for mean random reflectance.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *alginite, n*—a liptinite maceral occurring in structured morphologies, telalginite, and unstructured morphologies, lamalginite.

3.3.2 *bituminite, n*—an amorphous primary liptinite maceral with low reflectance, occasionally characterized by colored internal reflections and weak orange-brown fluorescence, derived from bacterial biomass and the bacterial decomposition of algal material and faunal plankton. Bituminite is equivalent to the amorphous organic matter recognized in strew slides of concentrated kerogen (**1**).[3]

3.3.2.1 *Discussion*—Bituminite may be distinguished from vitrinite by lower reflectance, as well as higher fluorescence intensity if fluorescence is present in vitrinite. Bituminite has poorly-defined wispy boundaries and may be speckled or unevenly colored whereas vitrinite has distinct boundaries and is blockier and evenly colored. The occurrence of bituminite in association with lamalginite and micrinite is common. Rock type, thermal maturity, and geologic occurrence can be used to interpret the potential presence of bituminite; for example, bituminite may be expected to occur in lacustrine or marine settings. It is less commonly present in fluvial or similar proximal depositional environments, where vitrinite may be expected to occur in greater abundance.

3.3.3 *chitinozoan, n*—a group of flask-shaped, sometimes ornamented marine microfossils of presumed metazoan origin which are composed of 'pseudochitin' proteinic material and which occur individually or in chains. Chitinozoan cell walls are thin, opaque to translucent, and range from dark gray to white in reflected white light similar to vitrinite. Chitinozoans are common in Ordovician to Devonian marine shales.

3.3.4 *conodont, n*—the phosphatic, tooth-like remains of marine vertebrate worm-like animals present from the Cambrian through Triassic, composed predominantly of apatite with subordinate amounts of organic matter. Conodont morphology is variable, but often well-defined denticles and blades

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[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.28 on Petrographic Analysis of Coal and Coke.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

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are preserved. In reflected white light examination conodonts range from pale yellow to light brown to dark brown and to black.

3.3.5 *fusinite, n*—an inertinite maceral distinguished principally by the preservation of some feature(s) of the plant cell wall structure, high relief, and reflectance substantially higher than first cycle vitrinite in the same sample. When less than 50-µm in size this maceral is assigned to inertodetrinite. Other organizations may define macerals using different technical specifications.

3.3.6 *graptolite, n*—colonial, chitinous animal which occurs as thin, elongate bodies sometimes showing complex skeletal morphology and with reflective dark gray to white color in reflected white light similar to vitrinite (**2**). Graptolites occur from the Cambrian through Carboniferous.

3.3.7 *huminite, n*—maceral group present in lignite and immature sedimentary rocks with reflectances intermediate to those of associated darker liptinites and brighter inertinites (**3**). Huminite is equivalent to the vitrinite maceral group that occurs in subbituminous and higher rank coals with measured reflectance values greater than 0.5% (**4**).

3.3.8 *inertinite, n*—maceral group with macerals that exhibit higher reflectance than other organic components in the same sample; for example, semifusinite, fusinite, and inertodetrinite. Inertinite macerals generally lack fluorescence and usually retain preserved plant cell wall structure(**5**).

3.3.9 *inertodetrinite, n*—an inertinite maceral occurring as individual, angular, clastic fragments incorporated within the matrix of other macerals (commonly vitrinite) or minerals, and in the size range from 2- to 50-µm. Other organizations may define macerals using different technical specifications.

3.3.9.1 *Discussion*—Inertodetrinite is derived through the disintegration of other inertinite macerals, that is, fusinite and semifusinite, by mechanical abrasion during transport.

3.3.10 *kerogen, n*—dispersed or concentrated organic matter, or both, occurring in sediments and sedimentary rocks that is insoluble in organic solvents.

3.3.11 *lamalginite, n*—an unstructured liptinite maceral with low reflectance distinguished primarily by the presence of bright fluorescence and lamellar character.

3.3.12 *liptinite, n*—maceral group with macerals that exhibit lower reflectance than other organic components in the same sample of sedimentary rocks and coal, appearing black to dark gray in reflected white light and that fluoresce under blue to ultraviolet light in coals ranked medium volatile bituminous and lower. Liptinite maceral fluorescence can be used as a qualitative thermal maturity indicator as fluorescence changes from green to yellow to orange before becoming extinguished at advanced maturity.

3.3.12.1 *Discussion*—Liptinite macerals are observed only in coals of maturity up to approximately the high volatile bituminous to medium volatile bituminous transition, and in sedimentary rocks of equivalent thermal maturity. Liptinite macerals undergo chemical changes during maturation which render their optical distinction from vitrinite and inertinite macerals difficult at maturities higher than medium volatile bituminous.

3.3.13 *maceral, n*—an organic component occurring in sedimentary rocks and coal that is distinguished on the basis of its optical microscopic properties, primarily reflectance and morphology.

3.3.14 *maceral classification, n*—the systematic division of the organic components (macerals) in sedimentary rocks and coal based on their appearance in the optical microscope under incident white and fluorescent light.

3.3.15 *micrinite, n*—an inertinite maceral, generally nonangular, exhibiting no relict plant cell wall structure, smaller than 10 µm and most commonly occurring as granular particles around 1- to 5-µm diameter. Other organizations may define macerals using different technical specifications.

3.3.15.1 *Discussion*—Micrinite is a secondary maceral formed from liptinite macerals during maturation.

3.3.16 *mineral matter, n*—in sedimentary rocks and coal, the non-organic fraction composed of physically discrete particles of minerals such as clays, pyrite, quartz, carbonates, etc., and all elements other than carbon, hydrogen, oxygen, nitrogen and sulfur in the organic fraction.

3.3.17 *recycled vitrinite, n*—vitrinite that has undergone at least one additional cycle of burial, exhumation, and erosion in contrast to first cycle vitrinite which has undergone only one burial cycle. The additional cycle may result in exposure to thermal maturation, chemical or thermal oxidative processes, or both, and mechanical abrasion (sometimes resulting in increased particle rounding) that is not experienced by first cycle vitrinite contained in the same sample.

3.3.17.1 *Discussion*—Recycled vitrinite has higher reflectance than co-occurring first cycle vitrinite, and sometimes is less angular, due to the rounding of grain boundaries experienced during transportation. Recycled vitrinite may have bright or dark halos, representing thermal oxidation and weathering processes, respectively, which are not present in the co-occurring first cycle vitrinite. Recycled vitrinite has a higher variance of reflectance values, representative of the many possible sources and processes occurring during transportation, and may show greater relief than first cycle vitrinite in the same sample. Recycling of vitrinite may be inferred from the geologic context; for example, a higher proportion of recycled vitrinite may be observed in a catchment collecting sediments derived from a growing orogenic belt.

3.3.18 *scolecodont, n*—the chitinous, variably mineralized fossil remains of the jaw elements of polychaete annelid worms, which occur as lamellar to tooth-like structures with spongy, laminated, or granular texture, and with reflective dark gray to white color similar to vitrinite. Scolecodonts occur from the Ordovician to recent.

3.3.19 *semifusinite, n*—an inertinite maceral with morphology like fusinite sometimes with less distinct evidence of cellular structure, and with reflectance ranging from slightly greater than that of the associated vitrinite to that of the least reflective fusinite. Semifusinite may show irregular mosaic texture or satin anisotropy when viewed under polarized reflected white light.

3.3.19.1 *Discussion*—Low-reflecting semifusinite may be distinguished from vitrinite by higher reflectance and relief, and the presence of more arcuate boundaries. The most reliable distinguishing feature of low-reflecting semifusinite is the frequent presence of well-preserved cellular structure or open cell lumens, or both. However, it is not unusual for cell lumens to also remain open in vitrinite when deposited in clay-rich sediments. Semifusinite usually has more distinct particle boundaries, which distinguishes it from vitrinite which has a more porous and textured surface. Geologic context is important; a greater proportion of semifusinite can be expected in sediments or coals associated with more arid locations, climates, and time periods.

3.3.20 *solid bitumen, n*—a secondary maceral associated with hydrocarbon generation from kerogen distinguished primarily by its conformation to pores, voids and fractures in the rock matrix, embayment by authigenic mineral grains, and the absence of features such as cellular structure indicating derivation from precursor plant material. Solid bitumens may show homogenous or granular textures; irregular anisotropic mosaic textures also are common, particularly at advanced stages of thermal maturity (**6**). Solid bitumens may exhibit fluorescence at low thermal maturity.

3.3.20.1 *Discussion*—For the purpose of reflectance measurement it is important to distinguish solid bitumen from vitrinite since both macerals appear gray under reflected white light and the reflectance of both advances with increasing maturity. Several populations of solid bitumen with distinct reflectance ranges can be present in a single whole-rock sample. Solid bitumens are characterized by their pore-filling or anastamosing forms. Boundaries of solid bitumen can be well-defined by textural embayment by authigenic minerals such as calcite and dolomite that commonly form contemporaneously with solid bitumen deposition. However, vitrinite can be replaced by authigenic minerals and therefore textures indicative of embayment or mineral inclusion are not always diagnostic of solid bitumen. Solid bitumen exhibits mosaic anisotropic domains at higher thermal maturity whereas vitrinite does not. Use of cross-polarized light by insertion of a post-sample analyzer into the light path may help to distinguish mosaic bitumens. Solid bitumens may be deposited in voids and fractures with orientations normal to sedimentary bedding. Solid bitumens may occur as droplets and may be translucent (recognized by reflections from pyrite inclusions) and contain pyrite crystals at edges. Rock type, thermal maturity, and geologic occurrence can be used to interpret the potential presence of solid bitumens; for example, bitumens may be present if the sample is or occurs in proximity to a mature hydrocarbon source rock or if the sample is from an exhumed oil reservoir. Solid bitumens can be physically associated with bituminite or other liptinite macerals from which they are derived. Some solid bitumens are soluble in organic solvents and may be distinguished from vitrinite in low maturity source rocks by low magnification observation of fluorescence streaming after pipetted solvation of the examination surface.

3.3.21 *telalginite, n*—a liptinite maceral characterized by strong fluorescence and structured morphologies. Common botanical varieties include Botryococcus, a freshwater indicator, and Tasmanites, a marine indicator. Fluorescence intensity diminishes and fluorescence color shifts toward red wavelengths with increasing thermal maturity.

3.3.22 *thermal maturity, n*—the degree of thermal alteration of the dispersed organic matter contained in sedimentary rocks, synonymous with coal rank. Thermal maturity of sedimentary rocks commonly is defined by vitrinite reflectance, spectral fluorescence, X-ray diffraction crystallography, or by organic geochemical parameters.

3.3.23 *vitrinite, n*—vitrinite dispersed in Upper Silurian and younger age sedimentary rocks is the remains of coalified material from vascular land plants. Vitrinite dispersed in sedimentary rocks may be representative of a large variety of precursor plant materials with differing original chemistries and structures. Vitrinite typically occurs as finely comminuted dark gray to white particles (in reflected white light) of sizes less than 100 µm dispersed throughout the mineral matrix although particles of larger size can also be present. Vitrinite dispersed in sedimentary rocks may occur as fragments of coal which include other macerals, including inertinite and liptinite.

3.3.23.1 *Discussion*—The identification of the primary vitrinite (first cycle vitrinite) population is essential for determining the peak thermal maturity experienced by a sedimentary rock. This can be complicated by: the chemical and structural heterogeneity of dispersed vitrinite reflecting multiple sources; the presence of similar organic components resembling vitrinite, including solid bitumen, bituminite, recycled vitrinite, low-reflecting semifusinite, and zooclasts; vitrinite reflectance retardation or suppression, or both; alteration by oxidation or weathering from sample handling or by exposure to the atmosphere at outcrop; and the potential for contamination such as cavings and drilling mud additives in the case of drill cuttings. The term vitrinite is currently used as both a maceral and maceral group.

3.3.24 *vitrinite reflectance retardation, n*—a reduction in vitrinite reflectance values below thermal maturity levels determined by geochemical or other petrographic parameters. Vitrinite reflectance retardation occurs due to decreased reaction rate and inhibition of the rearrangement of vitrinite molecular structure principally as a result of overpressure.

3.3.24.1 *Discussion*—The presence of vitrinite reflectance retardation can only be assessed if other thermal maturity parameters are available for the same sample or if vitrinite reflectance data from different depths or locations in an area are available for comparison. Vitrinite reflectance retardation cannot be assessed from the reflectance result of a single sample or the appearance of a single vitrinite particle.

3.3.25 *vitrinite reflectance suppression, n*—a reduction in vitrinite reflectance values below thermal maturity levels determined by geochemical or other petrographic parameters arising as a result of atypical hydrogen-rich vitrinite chemistry inherited from the precursor plant material or introduced into the vitrinite by the chemical microenviroments of deposition, diagenesis, and catagenesis.

3.3.25.1 *Discussion*—The presence of vitrinite reflectance suppression can only be assessed if other thermal maturity parameters are available for the same sample or if vitrinite reflectance data from different depths or locations in an area are available for comparison. Vitrinite reflectance suppression cannot be assessed from the reflectance result of a single sample or the appearance of a single vitrinite particle.

3.3.26 *zooclast, n*—faunal relics such as chitinozoans, graptolites, scolecodonts, and conodonts which may show similar optical properties to dispersed vitrinite (reflective dark gray to white color) in incident white light and which increase in reflectance with increasing thermal maturity. The reflectance of zooclasts may be measured and used for thermal maturity information of sedimentary rocks of pre-Upper Silurian age which do not contain vitrinite, or in addition to vitrinite reflectance in Upper Silurian and younger rocks.

4. Summary of Test Method

4.1 In this test method, the reflectance of the vitrinite maceral or other macerals is determined by illuminating the polished surface of a sedimentary rock or thin section, or a particulate preparation of rock core, drill cuttings, or kerogen concentrate covered by immersion oil using a microscope-photometer or other detection system that measures the amount of light reflected from the surface. The reflected light is recorded in percent reflectance after calibration of the photometric or other detection equipment by measuring the reflected light from standards of known reflectance as calculated from their refractive indices (see 6.13, Calibration Standards).

4.1.1 Color photomicrographs of vitrinite and other organic materials dispersed in sedimentary rocks are available from various publications and websites.

5. Significance and Use

5.1 The mean reflectance of the vitrinite maceral in sedimentary rocks as determined by this test method is used as an indicator of thermal maturity, that is, the progressive geochemical alteration of dispersed organic material experienced during diagenesis, catagenesis, and metagenesis. In the case of hydrocarbon source rocks, three major categories of thermal maturity are defined by vitrinite reflectance: immature ($R_o ran \leq 0.5\%$), mature ($R_o ran \approx 0.5\text{-}1.35\%$), and overmature ($R_o ran \geq 1.35\%$) with respect to the generation of liquid hydrocarbons. Thermal maturity as determined by the reflectance of vitrinite dispersed in sedimentary rocks is similar to the rank classification of coals as presented in Classification D388 and measured similarly to the reflectance of vitrinite in coal as presented in Test Method D2798. The mean reflectance of the vitrinite maceral in sedimentary rocks correlates with geochemically determined parameters of thermal maturity and can be used to characterize thermal maturation history, to calibrate burial history models, and to better understand the processes of hydrocarbon generation, migration, and accumulation in conventional and unconventional petroleum systems.

6. Apparatus

6.1 *Microscope*—Any microscope equipped for reflected light microscopy (such as a metallurgical or opaque-ore microscope) can be used. The microscope shall be able to project an image to a photomultiplier tube or other light detection system and to support the photomultiplier tube/light detection system housing.

6.2 *Light Sources*—The white light source used for measuring reflectance shall have a regulated power supply to provide for stable output. White light delivered from a 12 V 100 W tungsten halogen bulb is routinely employed; other illumination devices such as LEDs are acceptable provided they have similar emission spectra to that from tungsten halogen. Some lamps require supplemental voltage-stabilizing transformers if the line voltage fluctuates. The microscope may also be equipped with low wavelength fluorescence illumination from mercury or xenon gas discharge lamps, LEDs, or other devices with similar emission spectra. A beam-splitting mirror is used to switch illumination sources.

6.3 *Polarizer and Illuminator*—The light incident on the vertical illuminator of the microscope can be plane-polarized by a removable prism or sheet polarizer. The vertical illuminator can contain a Berek prism, a Smith illuminator, or a high-quality glass plate. When polarized light is employed, the polarizer shall be oriented at 45° when using a Berek prism or at 0° when using a Smith illuminator or glass plate.

6.4 *Fluorescence Filter Set*—For fluorescence microscopy, the microscope can be equipped with appropriate filter sets designed to observe the fluorescence emission spectra of the sample. Typically, the sets contain a bandpass excitation filter, a long pass beam splitter which serves as the vertical illuminator, and a long pass emission filter.

6.5 *Field Diaphragm*—The light incident on the vertical illuminator of the microscope shall be limited by an adjustable or fixed diaphragm field stop that should close to approximately ⅓ of the field or smaller as projected on the image. An adjustable field stop shall be limited by means of a set screw or similar mechanism so as to close to precisely the same diameter each time it is employed.

6.6 *Objective*—The oil immersion microscope objective shall be constructed of high quality lenses with anti-reflection coatings such that a minimum of stray light enters the light path. The combined magnification of objective and oculars shall permit examination of the specimen at a magnification between 400 and 750×, such that particles of 1µm can be resolved. Objectives of 40× or 50× magnification are routinely employed with oculars of 10× magnification.

6.7 *Stage*—The microscope stage can be capable of rotating through 360° or can be fixed. The mechanical stage attached to the microscope stage shall enable the analyst to move the specimen accurately (within 0.1 mm) to a given field location. A combination of objective and circular stage shall permit centering of the optical path.

6.8 *Measuring Aperture*—A measuring aperture made of non-reflecting, opaque material shall be placed approximately in the focal plane of the ocular at its central axis to restrict light to the photomultiplier tube window so that only a small area of the reflectance standard or sample is sensed. The diameter of the aperture shall be selected to provide an effective field of measurement (sensed spot) of about 5 µm diameter or an area of about $20\text{-}\mu m^2$.

6.9 *Filters*—The light reflected from the surface of the sample or standard shall be converted to monochromatic green by inserting an interference filter, or combination of filters, into the light path. The filters shall have peak transmittance of 546 ± 5 nm and a half-peak transmittance bandwidth of less than 20 nm. The filters shall be inserted into the light path immediately before the photomultiplier tube.

6.10 *Photomultiplier Tube*—In combination with the microscope optical system, light source, and filter used, the photomultiplier photometer shall be capable of detecting the minimum light reflected from the limited portion of the sample. The high voltage supplied to the photomultiplier tube must be within the prescribed range to obtain linearity of response. Photodiode arrays, channeltrons, digital cameras, or other light-measuring devices are acceptable alternatives providing that sufficient gray levels obtainable will enable reliable differentiation of signal equivalent to 0.01% reflectance and that the system is linear in the range of the reflectance measured. Some photometers and recorders require supplemental voltage-stabilizing transformers if the line voltage fluctuates.

6.11 *Photometer Amplifier*—The signal from the photomultiplier tube or detector shall be amplified and displayed by a galvanometer, digital meter, or recorder. When adjusted for operation, the amplifier and meter shall be capable of reliably distinguishing differences in signal equivalent to 0.01% reflectance and shall be linear in the range of reflectance measured.

6.12 *Ocular*—The viewing ocular shall be supplied with a crosshair or grid to be used as a reference to locate precisely the area sampled by the phototube. During measurement, no light shall be permitted to enter the observer's end of the viewing ocular.

6.13 *Calibration Standards*—Prisms constructed of high-index glasses or synthetic minerals shall be used as standards to calibrate the photometer for reflectance measurement. These standards must be durable, isotropic, resistant to corrosion and tarnish, free from internal flaws or fractures, and have negligible light absorption. A prism with sides that form a 30-60-90° triangle is the most effective shape, with the side between the 30° and 90° angles highly polished and used as the reflectance-measuring surface. The prisms shall be enclosed, except for the polished surface, in a durable, light absorbent, water- and oil-resistant mount; polyester or epoxy resin, made light absorbent with a dye or filler, serves adequately. It is desirable to have a number of different standards with calculated reflectance values within 0.1 to 0.15% of those of the vitrinite in the sample being measured; these also serve to check the linear response of the photometer. The reflectance of each standard shall be calculated to the nearest 0.001% by means of the following equation:

$$R_s = 100(n_g - 1.5180)^2/(n_g + 1.5180)^2 \quad (1)$$

where:

R_s = standard reflectance in oil of the glass, % and
n_g = refractive index of the glass at 546 nm wavelength, to the nearest 0.0001 index value.

Many laboratories use the following Bausch and Lomb Co. or Schott Co. optical glasses (the reported refractive index at 546 nm and the calculated standard reflectance in oil are given in parentheses):

Bausch and Lomb	Schott
689 309 (1.6935; 0.299%)	SF8-689-312 (1.6945; 0.303%)
751 278 (1.7566; 0.532%)	SF13-714-276 (1.7477; 0.496%)
827 250 (1.8351; 0.895%)	LaF12-836-423 (1.8400; 0.921%)
850 324 (1.8543; 0.996%)	LaSF9-850-322 (1.8567; 1.009%)
915 213 (1.9235; 1.390%)	LaSF18-913-325 (1.9273; 1.413%)
980 222 (1.9907; 1.817%)	LaSF6-961-249 (1.9670; 1.662%)

Other standards available that can be used include the following:

Leucosapphire	(1.77; 0.59%)
Yttrium aluminum garnet, YAG	(1.84; 0.92%)
Gadolinium gallium garnet, 3G	(1.98; 1.73%)
Silicon carbide	(2.663; 7.52%)

The theoretical standard values should be periodically checked in relation to reliable reference standards such as are available from the manufacturer or from commercial laboratories and scholarly organizations.

6.14 *Immersion Oil*—The oil shall be a non-drying, non-corrosive type that will not react with the sample, does not contain carcinogens, and has a refractive index within the range specified: 1.515 to 1.519 at 546 nm and 23°C. Within this range, the refractive index of the oil is not critical provided the specified value of 1.518 is used in calculating reflectance of standards as specified in 6.13. Periodic checking of the refractive index of the oil is discretionary.

6.15 *Sample-Leveling Press*—A conventional manual leveling device is used to level sample briquettes and glass standards when they are mounted on glass or metal microscope slides with modeling clay.

7. Test Specimen

7.1 Provided that the sample to be measured is representative, whole rock core, cuttings, outcrop, kerogen concentrate, and other sample types are suitable. For processed particulate rock samples, prepare the sample briquette in accordance with Practice D2797. Sections of whole rock core, cuttings, and outcrop can be mounted in polished briquettes as rock chips.

8. Preparation of Apparatus

8.1 The light source and photomultiplier or other light detection device shall be allowed to warm and stabilize prior to commencing vitrinite reflectance measurement; therefore, turn on the light sources and photometer or other light detection device and allow the equipment to warm up for at least ½ h.

9. Calibration and Standardization

9.1 Mount standards and a polished briquette containing the sample on slides using modeling clay and a leveling press (6.15), or use a leveling briquette holder.

9.2 Place the mounted briquette on the stage and observe its fluorescence properties under blue or ultraviolet illumination if the microscope is so equipped. Note and record the fluorescence intensity and color of any fluorescing organic material in the sample. Remove the polished briquette and repeat the final polish and desiccation stages in accordance with Practice D2797.

9.3 Switch illumination to reflected white light, apply immersion oil to the sample, and verify leveling of the mount and stage by checking that there is no systematic focus change when the briquette is moved laterally on the stage. Use Köhler illumination. To minimize glare, restrict the illuminated field by means of the field diaphragm so that the diameter is about one third or less than the size of the full field. The field diaphragm shall be closed as tightly as is feasible permitting that the entire area of the limiting aperture of the photomultiplier is illuminated. Adjust any other provisions of the illuminator to reduce scattered light in the system. Use these limiting settings for all subsequent measurements of reflected light.

9.4 Verify the position of the limiting aperture of the photometer with respect to the field of view. This can be done by moving a small bright object of the sample across the position of the crosshair or reticle that marks the photometer-sensed spot (ascertaining that readings are highest when the bright object is within the sensed area) or by using back-lit illumination of the measuring aperture if so equipped.

9.5 Identify vitrinite in the sample and observe its approximate reflectance value.

9.6 Remove the sample briquette from the stage and clean the immersion oil from the objective with lens tissue.

9.7 Place a standard of reflectance similar to the observed vitrinite covered with clean immersion oil on the microscope stage and focus on the polished surface. Use a parfocal closed field diaphragm, the edge of the standard, or a light scratch on the surface to focus correctly on the surface of the standard.

9.8 With no light reflected from the standard to the phototube, adjust the photometer setting to zero, correcting for dark current.

9.9 Allow the reflected light to impinge on the detector. Adjust the photometer amplifier or the illumination to obtain a meter readout or setting that corresponds to the calculated reflectance of the standard.

9.10 Without changing the settings, measure the reflectance of one or more additional standards to check that the photometer system measures correctly in the range to be studied. Because the photometric system cannot give a linear response to a wide range of light fluctuation, standards with reflectance values close to that of the sample being measured should be used. At least two standards having reflectance values that span the range of the sample being measured should be used.

9.11 Make all standardization measurements under the same conditions to be used for measuring vitrinite reflectance. Measure the same areas of the reflectance standards each time the calibration is made. Standards should be cleaned at least once a week to avoid oxidation and tarnishing and changes in the refractive index of the immersion oil on prolonged exposure to air or light.

10. Procedure for Random Reflectance Measurements

10.1 Random reflectance measurements are determined using non-polarized light; therefore, make sure that there is neither a polarizer nor an analyzer in the light path between the lamp and the photomultiplier tube/light detection system.

10.2 Immediately after calibrating the system, apply immersion oil and place the polished sample briquette on the microscope stage.

10.3 Adopt a systematic scheme of transection of the briquette for selection of areas to be measured. Transect intervals shall be such that the entire surface of the briquette or briquettes will be sampled for the component being measured.

10.4 Find and identify vitrinite or the organic component to be measured. Avoid the measurement of recycled vitrinite or record its reflectance separately. Avoid the measurement of poorly polished or pitted vitrinite or record its reflectance separately to delineate the lower limit of the adequately polished vitrinite range. In whole rock sample preparations, avoid the measurement of isolated organic fragments which have lost context regarding their geologic occurrence or which could be from laboratory contamination.

10.5 Select the location of the organic component to be measured to obtain a scratch-free area of uniform appearance. Observe and record the reflectance value, along with the interpreted maceral identification. Avoid taking measurements of areas that are near highly reflecting mineral grains such as pyrite or highly reflecting macerals such as fusinite. Because some relief and non-planarity can develop during polishing, avoid edges of particles and particles near the edge of the briquette.

10.6 Move the stage to the next area to be measured and repeat 10.5. Continue the location selecting and measuring procedure. After approximately ½ h of operation, remove the briquette, wipe off the immersion oil from the objective with a lens tissue, and recheck the calibration of the instrument by measuring the reflectance of a standard. If the measurement of the standard indicates a drift equivalent to more than 0.01% reflectance of the initial standard reflectance value, discard the last obtained set of measurements on the sample and rerun the measurements after recalibrating the system in accordance with 9.7 – 9.11.

10.7 When determining the reflectance of vitrinite, continue the procedure until at least 20-30 measurements have been obtained (**7**). The number of measurements for any other maceral will vary according to the application of the data.

11. Report

11.1 Report the following information:

11.1.1 Mean and standard deviation of the readings of random reflectance of vitrinite, as percent reflectance in immersion oil, shall be noted. Compute the mean to 0.01% as the sum of the individual measurements divided by the total number of measurements; the standard deviation is the square root of the computed variance. The number of measurements collected shall be noted. If fewer than twenty measurements are collected, prominent notice shall be made to indicate that the overall reflectance determination is not in compliance with this test method due to insufficient collection of measurements, whether due to organic leanness, insufficient sample availability, or unsatisfactory preparation method. Non-compliant analyses (<20 measurements) may be used as a qualitative thermal maturity indicator. The spread of individual reflectance values should be indicated either as a table of the individual reflectance values or as a frequency distribution in the form of a histogram or a table of percents within reflectance classes (V steps) which span 0.1% reflectance intervals, for example 0.90 through 0.99%. The identification of macerals other than vitrinite presented in the reflectance table or histogram shall be noted.

11.1.2 Sample preparations and measuring equipment, or indication of compliance with Test Method D7708 and Practice D2797 shall be noted. Any descriptive information obtained during sample preparation, such as color, mineralogy, size consist, etc., shall be noted. Sample processing technique, for example, kerogen concentration by acid digestion, shall be noted.

11.1.3 When fluorescence observation is employed, the presence or absence of fluorescence in the organic material of the sample including vitrinite shall be noted. In the presence of abundant or intense fluorescence in the organic material of the sample the possibility of vitrinite reflectance suppression shall be noted.

11.1.4 Should the sample represent a particular depth interval in a well, the report may consider reflectance data from elsewhere in the well profile, in particular if abnormal reflectance values in the well profile suggest vitrinite retardation or suppression.

11.1.5 Any distinguishing features of the first cycle vitrinite shall be noted. The presence or absence of organic materials in the sample similar to first cycle vitrinite (for example, recycled vitrinite, bitumen, bituminite, low reflecting semifusinite, zooclasts) shall be noted. Should there be organic materials in the sample similar to vitrinite, their distinguishing features shall be noted, including their reflectance.

11.1.6 Any provisions made to check preparation and polish quality, such as a check of measurements after re-polish or a comparison of measurements from two mounts of the same sample shall be noted.

11.1.7 Report the quality of the sample preparation according to the following alphanumerical scale: Proportion of the sample which has remained coherent at the polished surface:

(1) Organic matter and associated mineral grains have remained coherent with binder and polished with minimal erosion over >90% of the examination surface.

(2) Organic matter and associated mineral grains have remained coherent with binder and polished with minimal erosion over most of the examination surface.

(3) Organic matter and associated mineral grains have remained coherent with binder and polished with minimal erosion over a minority of the examination surface.

Quality of the polish on organic materials:

(A) Within the coherent areas of the examination surface with minimal erosion, >90% of the organic materials are free of pitting, scratching, and excessive relief.

(B) Within the coherent areas of the examination surface with minimal erosion, >50% of the organic materials are free of pitting, scratching, and excessive relief.

(C) Within the coherent areas of the examination surface with minimal erosion, most of the organic materials are pitted, scratched, or have excessive relief.

	1	2	3
A	1A Excellent	2A Very good	3A Poor
B	1B Good	2B Good	3B Very poor
C	1C Poor	2C Poor	3C Unusable

11.1.8 Any accompanying information with the sample shall be noted, such as well depth and stratigraphic information. If accompanying information has influenced the interpretation of vitrinite reflectance information for the sample, such influence shall be noted.

11.1.9 An example report is shown in Fig. 1.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is based on an interlaboratory study of D7708 – Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks, conducted in 2013. Twenty-two laboratories participated in this study. Each laboratory was instructed to report duplicate test results for six different types of shale. Every "test result" reported represents an individual determination. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D05-1043.[4]

12.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the long run, in employing the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.1.1 Repeatability can be interpreted as the maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

12.1.1.2 Repeatability limits are listed in Table 1.

12.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.2.1 Reproducibility can be interpreted as the maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

12.1.2.2 Reproducibility limits are listed in Table 1.

12.1.3 The above terms ("repeatability limit" and "reproducibility limit") are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1.1 and 12.1.2 will have an approximate 95% probability of being

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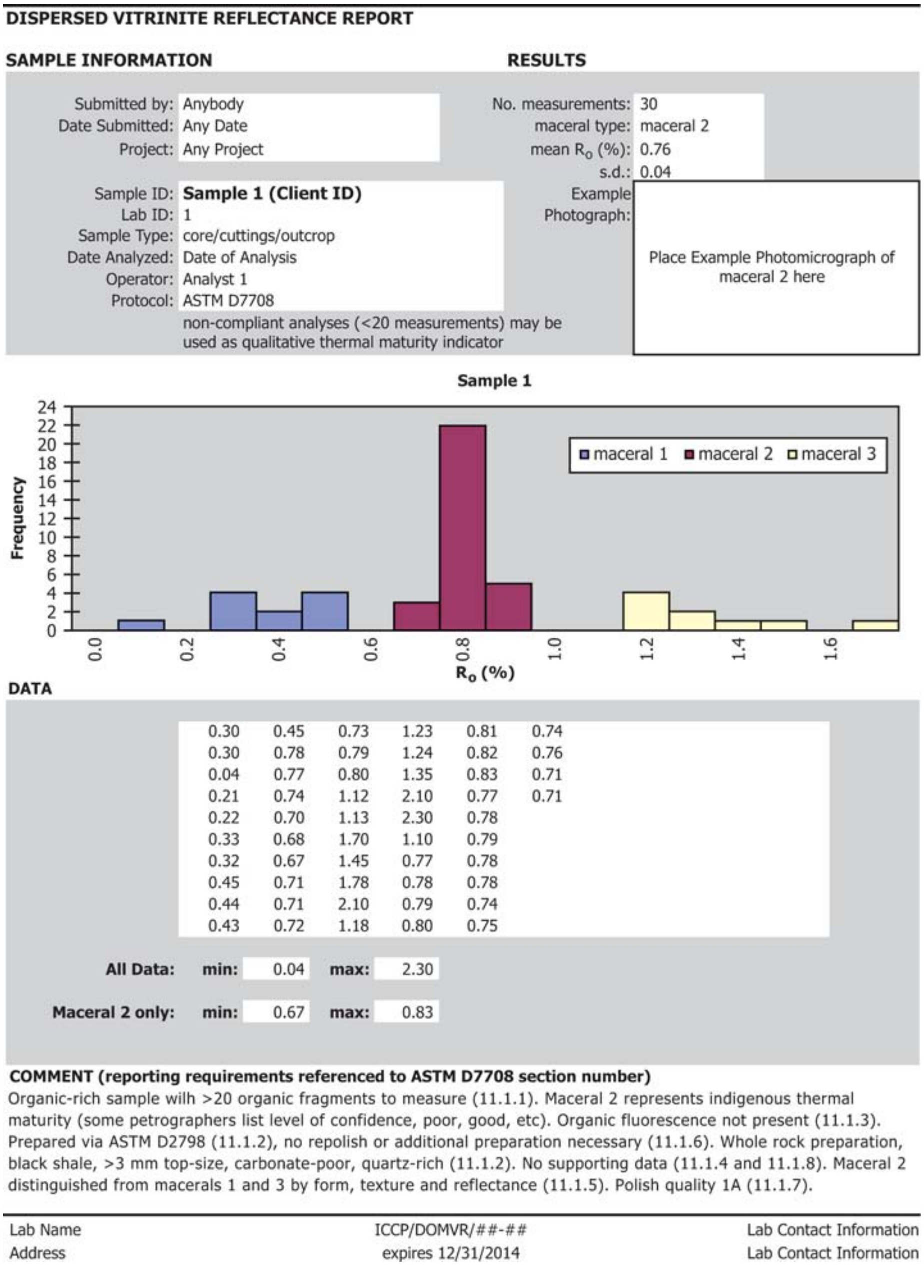
[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1043. Contact ASTM Customer Service at service@astm.org.



DISPERSED VITRINITE REFLECTANCE REPORT

SAMPLE INFORMATION

Submitted by: Anybody
Date Submitted: Any Date
Project: Any Project

Sample ID: **Sample 1 (Client ID)**
Lab ID: 1
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

RESULTS

No. measurements: 30
maceral type: maceral 2
mean R_o (%): 0.76
s.d.: 0.04
Example Photograph: Place Example Photomicrograph of maceral 2 here

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 wilh >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
Address

ICCP/DOMVR/##-##
expires 12/31/2014

Lab Contact Information
Lab Contact Information

FIG. 1 Example Report for D7708.

TABLE 1 Total Organic Carbon (weight %), Organic Matter Type, Average Vitrinite Reflectance (%), and Repeatability and Reproducibility Limits

Material	Total Organic Carbon (wt. %)	Organic Matter Type	Average[A] $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Eocene Shale	22.4	lacustrine	0.31	0.01	0.04	0.03	0.12
Upper Cretaceous Shale	4.1	marine	0.50	0.02	0.07	0.04	0.19
Devonian Shale	7.1	marine	0.80	0.02	0.12	0.07	0.34
Carboniferous Shale	5.0	terrestrial	0.99	0.02	0.06	0.04	0.17
Jurassic Shale	2.7	marine/inert	1.18	0.04	0.15	0.11	0.41
Lower Cretaceous Shale	1.0	marine/inert	1.53	0.03	0.19	0.10	0.54

[A] The average of the laboratories' calculated averages.

correct. The precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested may lead to times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95% probability limit would imply.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made. All aspects of sample preparation (as specified in Practice D2797) and the condition of the reflectance standards can impact reflectance measurement.

12.3 The precision statement was determined through statistical examination of 329 results, from twenty-two laboratories, on six materials. To judge the equivalency of two test results, it is recommended to choose the shale type closest in characteristics to the test material. Precision also can be impacted by type and abundance of organic material present; Table 1 includes these characteristics for the test materials evaluated in this ILS.

13. Keywords

13.1 dispersed organic matter; gas; hydrocarbons; oil; thermal maturity; vitrinite reflectance

APPENDIX

(Nonmandatory Information)

X1. CARE OF STANDARDS

X1.1 Keep immersion oil on standard and store standard in a container with a lid to minimize dust accumulation on the surface of the standard. Desiccating containers prevent moisture accumulation on the sample surface and thereby minimize changes to the refractive index of immersion oils.

X1.2 Periodically remove the oil and carefully clean the surface of the standard with a mild detergent such as used for cleaning optical glass. Application of fine polishing compounds via a sterile cotton swab may assist in removing tarnish buildup. Apply fresh immersion oil to the surface and store in the appropriate container. Some practitioners remove oil and clean standards daily, storing the standards overnight uncovered by immersion oil.

X1.3 Should scratches or irremovable tarnish appear on the standard surface preventing the measurement of reproducible vitrinite reflectance values, it may be necessary to have the surface polished by a professional vendor.

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