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Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite Dispersed in Sedimentary Rocks¹

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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](#)

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:*

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.

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

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

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 *lamalginitite, 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 anastomosing 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 microenvironments 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 1/3 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-µm².

6.9 *Filters*—The light reflected from the surface of the sample or standard shall be converted to monochromatic green