Designation: D 2798 – 09a

Standard Test Method for Microscopical Determination of the Vitrinite Reflectance of Coal [1]

This standard is issued under the fixed designation D 2798; 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 both the mean maximum and mean random reflectances measured in oil of polished surfaces of vitrinite and other macerals present in coals ranging in rank from lignite to anthracite. This test method can be used to determine the reflectance of other macerals.

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]

D 121 Terminology of Coal and Coke

D 388 Classification of Coals by Rank

D 2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light

3. Terminology

3.1 *Definitions*—For definitions of terms, refer to Terminology D 121.

3.2 *Abbreviations:*

3.2.1 R_{max}—mean maximum reflectance measured in oil.

3.2.2 R_m—mean random reflectance measured in oil.

4. Summary of Test Method

4.1 The reflectance of the maceral vitrinite or other macerals is determined in this test method by illuminating a polished surface of a section of coal in immersion oil using a microscopic system that photometrically measures the amount of light reflected from the surface. The reflected light is recorded in percent reflectance after calibration of photometric equipment by measuring the reflected light from standards of reflectance as calculated from their refractive indices.

5. Significance and Use

5.1 The mean maximum reflectance of the vitrinite component in coal as determined by this test method is often used as an indicator of rank as presented in Classification D 388, independent of petrographic composition, and in the characterization of coal as feedstock for carbonization, gasification, liquefaction, and combustion processes.

5.2 This test method is for use in scientific and industrial research.

6. Apparatus

6.1 *Microscope*—Any microscope equipped for reflected light microscopy (such as a metallurgical or opaque-ore microscope) can be used, provided the lens combination of objective and eyepieces permits examination of the specimen at a magnification between 400 and × 750, such that particles of 1 μm can be resolved. The objectives shall be constructed so that samples can be examined in oil with plane-polarized light and have the highest quality of antireflection coatings. The microscope shall be able to project an image at similar magnification to a photomultiplier tube and to support the photomultiplier tube housing. Means shall be provided to position the tube housing laterally to obtain maximum response. The microscope shall have a circular stage that is capable of rotating a specimen through 360°. 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. The viewing eyepiece 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 eyepiece.

6.2 *Polarizer and Illuminator*—The light incident on the vertical illuminator of the microscope shall be plane-polarized by a prism or sheet polarizer. The vertical illuminator can contain a Berek prism, a Smith illuminator, or high-quality

[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.

glass plate. The polarizer shall be oriented at 45° when using a Berek prism or at 0° when using a Smith illuminator or glass plate.

NOTE 1—Photodiode arrays, channeltrons, or other light-measuring devices are acceptable 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.

6.3 *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 coal sample (see 6.8). The high voltage supplied to the photomultiplier tube must be within the prescribed range to obtain linearity of response. This is usually from 300 to 1100 V for side-window tubes and from 1000 to 1500 V for end-window tubes.

6.4 *Photometer Amplifier*—The signal from the photomultiplier tube 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.

6.5 *Recorder or Meter*—The recorder or meter used shall have a response time at full scale of no more than 1 s to detect the maximum reflectance level during rotation of the microscope stage.

6.6 *Light Source*—The light source shall have a regulated power supply to provide for stable output. Some photometers and recorders require supplemental voltage-stabilizing transformers if the line voltage fluctuates.

6.7 *Filters*—The light shall be made approximately monochromatic green by passage through an interference filter or combination of filters with peak transmittance of 546 ± 5 nm and a half-peak transmittance bandwidth of less than 20 nm. The filters can be inserted into the light path between the light source and photomultiplier tube at any convenient position in the optical system.

6.8 *Limiting Aperture*—A limiting aperture made of nonreflecting and opaque material shall be placed approximately in the focal plane of the eyepiece 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 about 20-µm^2 area.

6.9 *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, 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, lightabsorbent, 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 reflectances near those of the vitrinite studied; 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.

NOTE 2—Most coal laboratories in North America 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 %)

6.10 *Immersion Oil*—The oil shall be a nondrying, noncorrosive type that will not react with coal, does not contain carcinogens, and has a refractive index within the range from 1.515 to 1.519 at 546 nm and 25°C. Within the specified range, the refractive index of the oil is not critical provided the specified value of 1.5180 is used in calculating reflectance of standards as specified in 6.9. Periodic checking of the refractive index of the oil is discretionary.

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

7. Test Specimen

7.1 Prepare the sample briquette in accordance with Practice D 2797.

8. Setting Up and Calibrating the Apparatus

8.1 Turn on the photometer and light source and allow equipment to warm up for at least ½ h.

8.2 Mount the glass standards and a polished briquette containing the sample on slides using modeling clay and a leveling press or use a leveling briquette holder.

8.3 Place the mounted briquette on the stage, apply immersion oil, 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. Adjust any other provisions of the illuminator to reduce scattered light in the system.

8.4 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