Designation: D 2798 – 99

Standard Test Method for Microscopical Determination of the Reflectance of Vitrinite in a Polished Specimen 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 *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:*

D 121 Terminology of Coal and Coke[2]

D 388 Classification of Coals by Rank[2]

D 2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light[2]

3. Terminology

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

3.2 *Abbreviations: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. If mean maximum reflectance is used as a rank indicator, the types of vitrinites measured shall be specified.

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

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[2] *Annual Book of ASTM Standards*, Vol 05.05.

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contain a Berek prism, a Smith illuminator, or high-quality 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 coalsample (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.

7.2 Place the sample briquettes in the desiccator at least 15 h before measuring the reflectance as stated in the Moisture Control section of 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 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.

8.5 Using a small, distinctive feature of the sample as a guide, adjust the microscope so that the axis of rotation of the stage is coincident with the photometer-sensed spot. This is accomplished by adjusting the centering screws of the objective or stage. The purpose is to eliminate movement of the object grain or area from the sensed spot when the stage is rotated.

8.6 Adjust the polarizer to a 45° position when using a Berek prism or 0° when using a Smith illuminator or glass plate. Place a glass standard covered with clean immersion oil on the microscope stage and focus on the polished surface.

8.7 With no light reflected from the standard to the phototube, adjust the photometer zero setting or dark current. Place on the microscope stage a briquette of opaque resin that has a hole 5 mm in diameter and 5 mm deep which is filled with immersion oil. Measure the reflectance of the hole to ensure that a reflectance of 0.00 ± 0.03 % is obtained thereby ensuring that parasitic reflectances of the objective are minimal. If the reflectance of the hole exceeds the stated limits, then another objective having a higher quality antireflection coating shall be used.

8.8 Then allow the reflected light to impinge on the tube. Adjust the photometer amplifier or the illumination to obtain a meter or recorder scale setting that conveniently represents the calculated reflectance of the glass standard (see Appendix X1).

8.9 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.

NOTE 3—Because the photometric system cannot give a linear response to a wide range of light flux, standards with reflectance values close to that of the coal being measured should be used. At least two standards having reflectances that span the range of the coal being measured should be used.

8.10 Make all standardization measurements under the same conditions used in measuring vitrinite reflectance. When measuring mean maximum reflectance of vitrinite, rotate the stage through 360° and note the maximum reflectance value of the glass reflectance standard. If values change during rotation more than 0.03 %, then the system alignment shall be checked.

8.11 Measure the same areas of the glass reflectance standards each time the calibration is made.

8.12 Glass standards should be cleaned at least once a month to avoid oxidation and changes in reflectances.

9. Procedure for Measuring Maximum Reflectance of a Sample

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

9.2 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 are briquettes will be sampled for the component being measured. The transect spacing shall be suitable for a total of 100 measurements.

9.3 Using the procedure specified in 9.2, select the location to be measured. Slight adjustments to the maceral position may be made to obtain a scratch-free area of uniform appearance. Rotate the circular stage slowly (approximately 4 r/min) through 360°. Reflectance will vary progressively from a maximum value to a minimum value as the stage is rotated. Observe and record the maximum value. If the effective field of measurement does not remain on the component being measured when the stage is rotated, then recenter the objective or stage as described in 8.5. Avoid taking measurements of areas that are near highly reflecting grains such as pyrite. Because some relief and nonplanarity may develop during polishing, avoid edges of particles and particles near the edge of the briquette.

9.4 Move the stage to the next area to be measured and repeat 9.3. Continue the location selecting and measuring procedure. After approximately ½ h of operation, remove the briquette and recheck the calibration of the glass standards. If this value indicates a drift equivalent to more than 0.01 % reflectance of the initial standard reflectance value, discard the set of readings on the coal sample and rerun the measurements after recalibrating the system in accordance with 8.7.

9.5 When determining the reflectance of vitrinite, continue the procedure until at least 100 measurements have been obtained. The number of measurements for any other maceral will vary according to the application of the data.

9.6 For blends that contain coals of different rank, 150 measurements are necessary to determine the mean maximum reflectance.[3]

NOTE 4—Although the term "maximum reflectance" is used, the actual value obtained in this method may not represent the true maximum reflectance axis of the reflectance indicatrix, an imaginary surface that defines a coal's three-dimensional distribution of reflectances.[4,5] The reflectance indicatrices of most coals approximate those of uniaxial negative optical materials. All particles of such coals, regardless of orientation, will display a true maximum reflectance in at least one direction in polarized light. However, some higher rank coals, especially anthracites, can have biaxial optical properties. In these cases, the mean value obtained by this ASTM method is a mean apparent maximum rather than a mean true maximum reflectance. The apparent maximum reflectance is intermediate between the true maximum and the true intermediate reflectance. Determination of the true maximum reflectances of biaxial coals can be obtained by (1) measurements on three different oriented surfaces of a polished coal block[4] or (2) a graphical method applied to measurements from particulate samples.[5]

10. Measuring Random Reflectance of a Sample

10.1 Assure that there is neither a polarizer nor an analyzer in the light path between the lamp and the photomultiplier tube.

[3] Research Report D05: 1021.

[4] Levine, J. R. and Davis, A., "Reflectance Anisotropy of Upper Carboniferous Coals in the Appalachian Foreland Basin, Pennsylvania, U.S.A.," *International Journal of Coal Geology*, Vol 13, 1989, p. 341.

[5] Kilby, W. E., "Recognition of Vitrinite with Non-Uniaxial Negative Reflectance Characteristics," *International Journal of Coal Geology*, Vol 9, 1988, p. 267.

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

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. The transect spacing shall be suitable for a total of 100 measurements.

10.4 Using the procedure specified in 10.3, select the location to be measured. Slight adjustments to the maceral position may be made to obtain a scratch-free area of uniform appearance. Observe and record the reflectance value. Avoid taking measurements of areas that are near highly reflecting grains such as pyrite. Because some relief and nonplanarity can develop during polishing, avoid edges of particles and particles near the edge of the briquette.

10.5 Move the stage to the next area to be measured and repeat 10.4. Continue the location selecting and measuring procedure. After approximately ½ h of operation, remove the briquette and recheck the calibration of the glass standards. If this value indicates a drift equivalent to more than 0.01 % reflectance of the initial standard reflectance value, discard the set of readings on the coal sample and rerun the measurements after recalibrating the system in accordance with 8.7.

10.6 When determining the reflectance of vitrinite, continue the procedure until at least 100 measurements have been obtained. The number of measurements for any other maceral will vary according to the application of the data.

10.7 More than 100 measurements are necessary to test blends that contain coals of different rank.

NOTE 5—Automated microscopy systems are used to measure random reflectance and estimate mean maximum vitrinite reflectance and coal blend percentages. Users should be aware that the particle size consist, briquette polishing quality, surface leveling procedure, binder/coal ratio used, and maceral composition will significantly affect the final reflectogram generated by automatic systems. If a correction to mean-maximum reflectance is made, results obtained by using an automated microscope system should be statistically equivalent to manually derived vitrinite reflectance results as determined in Section 9.

11. Report

11.1 Report the following information:

11.1.1 Mean and standard deviation of the readings of maximum or random reflectance of vitrinite, as percent reflectance in immersion oil, shall be noted. Compute the mean 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. It is suggested that the spread of individual reflectance values be indicated either as a table of the individual maximum reflectance values or as a frequency distribution in the form of a histogram or a table of percents within reflectance classes.

NOTE 6—Classes commonly span 0.1 % reflectance intervals, for example 0.60 through 0.69 %.

NOTE 7—For informational purposes, the relationship between mean maximum and mean random reflectance has been determined through regression analysis from the 1991 to 1995 interlaboratory round-robin exercise. The equation developed was found to be as follows for single seam coals between 0.7 and 1.7 mean maximum reflectance:

$$\text{Mean Maximum Reflectance} = -0.034 + 1.09\ (\text{Mean Random Reflectance}) \quad (2)$$

11.1.2 Sample preparations and measuring equipment, or indication of compliance with Test Method D 2798 and Practice D 2797 shall be noted. Specify the particular type of vitrinite measured when the mean maximum reflectance is to be used as an indicator of rank.

11.1.3 Any provisions made to check polish quality, such as a check of measurements after repolish or comparison of measurements from two mounts of the same sample shall be noted.

12. Precision and Bias

12.1 *Precision*—The following criteria should be used for judging the acceptability of results on representative minus 850-µm (No. 20) subsamples within the mean maximum reflectance range of 0.7 and 1.7 %.

12.1.1 *Repeatability*—Duplicate results by the same laboratory, using the same operator and equipment, should not be considered suspect unless the results differ by more than 0.02 % actual reflectance.

NOTE 8—This test method does not require duplicate determinations to be made. The repeatability value was developed because most laboratories occasionally monitor and check within laboratory precision as part of internal quality control practices.

12.1.2 *Reproducibility*—The results submitted by two or more laboratories, using different equipment, operators, date of test, and different representative subsamples of the same sample, should not be considered suspect unless the results differ by more than 0.06 % actual reflectance.

12.2 *Bias*—Because there is no accepted reference material for obtaining the bias for the procedure in this test method for measuring reflectance of vitrinite, no specific statement is being made. All aspects of sample preparation (as specified in Practice D 2797) and the condition of the glass reflectance standards can impact reflectance measurement.

NOTE 9—Based on the results from the interlaboratory round-robin study conducted by the twelve participating laboratories to establish the precision values during 1991 to 1994, it was found that; (1) the difference between laboratories for reflectance was not significant when comparing briquettes prepared and polished by a common laboratory to briquettes made and polished by the twelve paticipating individual laboratories and (2) relectance measurements determined on a common set of glass standards were found to closely parallel measurements determined on glass standards used by the twelve participating laboratories.

13. Keywords

13.1 coal; maceral; microscopy; rank; reflectance; vitrinite

APPENDIX

(Nonmandatory Information)

X1. CARE AND CALIBRATION OF GLASS STANDARDS

X1.1 Care of Glass Standards

X1.1.1 Keep immersion oil on glass standard and store standard in a container with a lid to minimize dust accumulation on the surface of the standard.

X1.1.2 Periodically remove the oil and carefully clean the surface of the polished glasses with a mild detergent such as used for cleaning optical glass. Apply fresh immersion oil to the surfaces and store in the appropriate container.

X1.2 Preparation of a Matrix of Determined Reflectance Values for the Glass Set

X1.2.1 Set up the microscope system in accordance with 8.1 through 8.7.

X1.2.2 Place the first glass standard under the microscope objective and move to the center area of the glass surface.

X1.2.3 Record the position of the mechanical stage.

X1.2.4 Adjust voltage on the photometer to the calculated reflectance value of the glass standard as the maximum value obtainable as the stage is rotated 360°.

X1.2.5 Record the calculated value of the first glass standard in a table as shown in Table X1.1.

X1.2.6 Read and record the stage position and the maximum reflectance of the centers of all other glass standards without adjusting the photometer system.

X1.2.7 Place the second glass standard under the microscope objective and move to the recorded coordinates for that glass surface. Repeat X1.2.3 through X1.2.6.

X1.2.8 Repeat X1.2.7 for each remaining glass standard.

X1.2.9 Using the recorded values in the table, disregard those values associated with highly inconsistent readings (greater than 0.2 % reflectance difference) such as shown for glass No. 5 in Table X1.1.

X1.2.10 Average all other readings except the adjusted value that corresponded to the calculated reflectance and record the determined average value. This value is the one for the specified area of the glass that is to be used for instrument calibration as given in 8.7.

TABLE X1.1 Example of Calibration Matrix for Glass Standards

Glass Number	1	2	3	4	5	6
Calibration on No. 1	0.303[A]	0.508	0.914	1.014	1.351	1.649
Calibration on No. 2	0.302	0.496[A]	0.906	1.002	1.360	1.654
Calibration on No. 3	0.304	0.504	0.921[A]	1.007	1.363	1.650
Calibration on No. 4	0.307	0.508	0.924	1.008[A]	1.386	1.670
Calibration on No. 5	0.324	0.554	0.928	1.049	1.413[A]	1.703
Calibration on No. 6	0.307	0.509	0.914	1.014	1.361	1.662[A]
Determined average	0.305	0.507	0.915	1.009	n.u.[B]	1.656
Stage coordinates	3 × 118	3 × 110	3 × 103	12 × 118	12 × 110	12 × 103

[A] Adjusted photometric reading from calculated reflectance values of glass standards.
[B] n.u. = values not used for calculation of other reflectances or calibration of equipment because readings were inconsistent with others during construction of matrix.

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