Designation: D2799 – 13

Standard Test Method for Microscopical Determination of the Maceral Composition of Coal[1]

This standard is issued under the fixed designation D2799; 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 equipment and techniques used for determining the physical composition of a coal sample in terms of volume percent of the organic components and of mineral matter, if desired.

1.2 The term *weight* is temporarily used in this test method because of established trade usage. The word is used to mean both force and mass and care must be taken to determine which is meant in each case (the SI unit for force is newton and for mass, kilogram).

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
D2797 Practice for Preparing Coal Samples for Microscopical Analysis by Reflected Light
D2798 Test Method for Microscopical Determination of the Vitrinite Reflectance of Coal
D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal
D4239 Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion
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 *Classification*—The classification of the microscopic constituents into groups of similar properties in a given coal is as follows:

Maceral Group	Maceral
Vitrinite	—
Liptinite or (exinite)	alginite
	cutinite
	resinite
	sporinite
Inertinite	fusinite
	inertodetrinite
	macrinite
	micrinite
	funginite
	secretinite
	semifusinite

3.3 Many laboratories associated with the coke-making industry use the following simplified classification for petrographic analysis of bituminous coal:

vitrinite
liptinite (other than resinite)
resinite
semifusinite
micrinite
fusinite
mineral matter

3.4 *Definitions of Terms Specific to This Standard:*

3.4.1 *alginite, n*—a liptinite maceral that is generally spherical or ovoid, frequently having a crenulated border and somewhat irregular reflectance and sometimes occurring in clusters reflecting an origin from *Botryococcus* algae.

3.4.1.1 *Discussion*—Alginite often occurs as degraded fragments derived from colonial or unicellular bodies.

3.4.2 *cutinite, n*—a liptinite maceral in the form of a sheet reflecting its origin from leaf- or twig-covering plant cuticle, frequently exhibiting reticulation in planar section and a serrated edge in cross section.

3.4.3 *exinite, n*—Deprecated term. Use preferred term **liptinite**; sometimes has also been used as a synonym for sporinite.

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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.

3.4.4 *funginite, n*—an inertinite maceral occurring as round or ovoid bodies, frequently containing voids, reflecting an origin from fungal sclerotia; also occurs (especially in lower rank coals) as interlaced, stringy materials derived from fungal hyphae.

3.4.5 *fusinite, n*—an inertinite maceral distinguished principally by the preservation of some feature(s) of the plant cell wall structure, and with a particle size greater than 50 µm except when it occurs as a fragment within the binder matrix; see also **semifusinite**.

3.4.6 *inertinite, n*—macerals that exhibit higher reflectance than other organic substances in the coal.

3.4.6.1 *Discussion*—In any coal ranked lower than anthracitic, inertinite reflectance commonly spans the range from only slightly higher than associated vitrinite to very high reflectance (often as high as $R_o max \geq 6$ %). In anthracitic rank coals, inertinite reflectance may be lower than that of vitrinite, and is then recognized by its morphology and form of anisotropy. Highly reflecting inertinite commonly exhibits relief on polished surface. Its name derives from the fact that most varieties behave inertly in the thermoplastic deformation during the coking process (except in its lowest reflecting manifestation). The volatile matter yield of inertinite is lower than that of other macerals in the same coal.

3.4.7 *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.

3.4.8 *liptinite, n*—macerals that exhibit lower reflectance than other organic substances in a coal, appearing black to dark gray and that fluoresce under blue to ultraviolet light in coals ranked high volatile bituminous and lower.

3.4.8.1 *Discussion*—The fluorescence of liptinite distinguishes fine-grained liptinite from similar sized, low reflectance, nonfluorescing clay minerals. Liptinite is derived principally from lipid substances forming skins (exines) and resinous secretions or exudates of plants. Liptinite is subclassified on the basis of morphology inherited from plant structure. In coals in which vitrinite reflectance exceeds about 1.4 %, liptinite can be indistinguishable from vitrinite. Liptinite has the highest volatile matter yield of the macerals in a coal.

3.4.9 *maceral, n*—an organic substance in coal that is distinguished and classified (see **maceral classification**) on the basis of its optical microscopic properties.

3.4.9.1 *Discussion*—Macerals originate from plant tissues, secretions, and exudates that have been altered by geological processes and may contain up to several weight percent of inorganic elements in microscopically indistinguishable form.

3.4.10 *maceral classification, n*—The systematic division of the organic substances (macerals) in coal based on their appearance in the optical microscopic.

3.4.10.1 *Discussion*—Although macerals may be identified in translucent, thin sections using criteria not defined herein, this test method deals only with identification and classification based on microscopic appearance on polished surfaces according to Practice D2797. Three major maceral groups are recognized on the basis of relative reflectance in white light, specifically: vitrinite—moderately reflecting (intermediate gray), liptinite—poorly reflecting (black to dark gray), and inertinite—highly reflecting (light gray to white). Each group can be subdivided on the basis of other microscopically distinctive features such as: reflectance contrasts (relative shades of gray); morphology, that is, shape and size (morphologic distinctions in definitions contained herein are idealized because morphologic appearance depends on the initial form of the source material, its state of preservation, including granulation, and on the orientation of the cross section presented on the polished preparation); spatial association with other substances; fluorescence properties (color, intensity) in blue to ultraviolet light; relief; color tinges; internal reflections; and anisotropic properties.

Microscopic criteria provide classification capability without any implication of absolute chemical composition or physical behavior, although some properties relative to other macerals in the same coal can be inferred broadly. Substances classified as the same maceral by microscopic criteria can differ chemically, physically, and behavioristically in coals of different ranks. Some properties can be estimated by the measurement of reflectance (Test Method D2798).

See 3.3 for the classification used by most practitioners of this test method.

3.4.11 *macrinite, n*—an inertinite maceral, generally nonangular, exhibiting no relict plant cell wall structure and larger than 10 µm.

3.4.12 *micrinite, n*—an inertinite maceral, generally nonangular, exhibiting no relict plant cell wall structure, smaller than 10 µm and most commonly occurring as particles around 1- to 5-µm diameter.

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

3.4.14 *resinite, n*—a liptinite maceral occurring as rounded, ovoid, or rod-like bodies assuming the shape of an enclosing cell lumen or as irregular shapes filling cracks in the coal.

3.4.15 *secretinite, n*—an inertinite maceral occurring as round, ovoid, or oblong bodies, without obvious plant structure, vesicled to non-vesicled, sometimes containing characteristic fractures, slits, or a notch.

3.4.15.1 *Discussion*—Secretinite is considered to be derived by the oxidation of plant resin secretions or humic gels. Vesicular and non-vesicular secretinite was formerly included in sclerotinite of fungal origin. Secretinite is a common maceral in medium- and high-rank Permian and Carboniferous coals.

3.4.16 *semifusinite, n*—an inertinite maceral with morphology like fusinite sometimes with less distinct evidence of cellular structure, but with reflectance ranging from slightly greater than that of associated vitrinite to some value intermediate to that of the brightest fusinite. The particle size is also greater than 50 µm except when it occurs as a fragment within the binder matrix.

3.4.16.1 *Discussion*—The precise reflectance boundary between semifusinite and fusinite has not been universally defined, although some practitioners place the division at $R_o max = 2.0$ %; hence, semifusinite is somewhat vaguely defined as "fusinite with low reflectance."

3.4.17 *sporinite, n*—a liptinite maceral exhibiting various lenticular, oval, or round forms that reflect the cross-sectioning of a flattened, hollow, ovoid body; sometimes exhibits rod-like projections that are small relative to the size of the total body.

3.4.17.1 *Discussion*—Sporinite originated as a lipid substance that covered, as a skin, ovoid spore or pollen grains which commonly ranged from around ten to several hundred micrometres in diameter. Sporinite often occurs as fragments derived from these initially ovoid bodies.

3.4.18 *vitrinite, n*—the predominant maceral in most coals of intermediate reflectance occurring as substantial volumes of more or less uniformly reflecting material or as a matrix enclosing particles of other macerals and mineral matter or as particles or bands intermixed with other maceral fragments.

3.4.18.1 *Discussion*—Because most vitrinite is derived from the cellular, structural tissues of plants, it may exhibit relict cell structure. The reflectance of vitrinite is related to the rank of the coal in which it is found. Reflectance increases (from around $R_o max = 0.3$ % in lignitic coals) in parallel with the increase in fixed carbon yield associated with increasing rank. Because many of the properties of typical coals reflect the properties of the dominating vitrinite, it is common practice to estimate coal properties and process behaviors by measuring the reflectance of a representative sampling of vitrinite in the specimen according to procedures described in Test Method D2798.

Pseudovitrinite, a certain variety of vitrinite, is differentiated by some practitioners. It exhibits slightly higher reflectance than most of the vitrinite in the coal and is commonly slitted, with indistinct remnant cell structure and angular or jagged edges. Pseudovitrinite has been postulated to be less thermoplastic in the coking process.

The term vitrinite is currently used as both a maceral and maceral group.

4. Summary of Test Method

4.1 The components in a representative crushed coal sample, prepared as prescribed in Practice D2797, are identified under a microscope according to their reflectance, other optical properties, and morphology. The proportions of these components in a sample are determined by observing a statistically adequate number of points, and summing those representative of each component. Only area proportions of components are determined on a surface section of a sample. However, the area and volume proportions are the same when the components are randomly distributed throughout the sample.

4.1.1 Color photomicrographs of the maceral components of bituminous coals are available from various publications and websites.[3]

5. Significance and Use

5.1 The volume percent of physical components of coal is used as an aid in coal seam correlation and in the characterization of coals for their use in carbonization, gasification, liquefaction, and combustion processes.

5.2 This test method is for use in scientific and industrial research, not compliance or referee tests.

6. Apparatus

6.1 *Microscope*—Any microscope with a mechanical stage and a vertical illuminator (that is, metallurgical or opaque-ore microscope) may be used, provided that the lens combination of objective and eyepiece permits resolution of objects on the order of 1 to 2 µm. A minimum magnification of approximately 400 diameters is recommended. Either a prism or a partially reflecting glass plate may be used in the illuminator. One eyepiece of the microscope should be fitted with a graticule or crosshair.

6.1.1 *Eyepiece Disk*—If other than crosshairs are used, the eyepiece disk shall contain a Whipple graticule or one of such design that four points are visible, lying at the corners of a square covering nearly all of the field of view. The minimum effective distance between the points, referred to the plane of the specimen, shall be 0.1 mm.

6.1.2 *Mechanical Stage*—The mechanical stage shall be of such type that the specimen can be quickly advanced by definite fixed increments in two perpendicular directions. If an electrically operated stage is used, increment steps in one direction across the specimen may be actuated by the counter switches.

6.2 *Counter*—Counters shall be used to count components.

7. Test Specimen

7.1 Prepare sample briquets in accordance with Practice D2797.

8. Procedure

8.1 In accordance with present practice, maceral components counted shall be as defined in Section 3. The specific application will determine the degree of detail and maceral components identified.

8.2 When a graticule is used, count the components lying under each of the four points described in 6.1.1 in each microscopic field. When a crosshair disk is used, count the component lying under the intersection of the crosshairs.

[3] U.S. Geological Survey, ASTM International Maceral Composition of Coals available from: http://energy.er.usgs.gov/coal_studies/organic_petrology/photo_atlas.html.

8.3 Advance the specimen in steps of 1.0 mm when a graticule is used and 0.5 mm when a crosshair is used, until the desired length of the specimen in that direction has been covered. Then advance the specimen one similar step at right angles and repeat the first procedure in reverse. Do not count any of the field points that fall on the briquet binder.

8.4 Total mineral content may be point counted or calculated from ash and sulfur contents determined in accordance with Test Methods D3174 and D4239. The percent of mineral matter calculated as in the Parr formula (1.08 ash % + 0.55 sulfur %, dry basis) on a weight basis shall be converted to a volume basis using a density of 2.8 g/cm^3 for mineral matter and 1.35 g/cm^3 for organic components, unless more specific information about density is available. Use the following formula to calculate volume percent mineral matter:

$$MM = \frac{100[(1.08\,A + 0.55\,S)/2.8]}{[100 - (1.08\,A + 0.55\,S)]/1.35 + (1.08\,A + 0.55\,S)/2.8} \quad (1)$$

where ash and sulfur values are on a dry basis.

8.5 Count a minimum of 1000 points on one briquette or 500 points on each of two briquettes. As much as possible of the briquette surface should be covered during these counts. Calculate the volume percent of each component to the nearest 0.1 % from the proportionate number of counts.

9. Report

9.1 Report microscopical data in one of two ways. If only organic components are determined, report these on a basis of 100 %. If mineral matter is included, mineral matter plus organic components should total 100 %. The reporting must clearly indicate whether the mineral matter is counted or calculated.

9.2 The total number of points counted during the analysis shall be specified. The precision of determining the volume percent of physical components in coal is partially related to the number of points counted during the analysis. In general, increasing the number of points counted can achieve increased precision.

10. Precision and Bias

10.1 *Precision*—The precision for the determination of the maceral groups vitrinite, inertinite and liptinite in coal by test method D2799, is given in Table 1.

10.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

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

10.1.4 Any judgment in accordance with statements 10.1.1 and 10.1.2 would have an approximate 95% probability of being correct.

10.2 *Bias*—There are no accepted reference materials suitable for determining the bias for this test method, therefore no statement on bias is being made.

10.3 The information in Table 1 was calculated per Practice E691 using 8 sets of data for each of 12 coal samples from a commercially available inter-laboratory proficiency test program. The coals used in this study varied in mean maximum vitrinite reflectance between 0.6% and 1.8%. Details and supporting information are given in Research Report RR:D05-1037.[4]

TABLE 1 Limits of Repeatability and Reproducibility for Maceral Groups

Maceral Group	Range (percent)	Repeatability Limit r	Reproducibility Limit R
Vitrinite	57.6 – 86.9	3.6	7.1
Inertinite	8.4 – 41.7	3.8	6.8
Liptinite	0.2 – 9.5	0.84 + 0.23xValue[A]	1.71 + 0.38xValue[A]

[A] Value = average value of the two test results being compared.

11. Keywords

11.1 coal; exinite; fusinite; maceral; micrinite microscopy; mineral matter; organic components; petrographic analysis; resinite; semi-fusinite; vitrinite

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1037. Contact ASTM Customer Service at service@astm.org.

ANNEX

A1. PRECISIONS STATISTICS

A1.1 The precision of this test method, characterized by the repeatability (S_r, r) and Reproducibility (S_R, R) has been determined for the following materials as listed in Tables A1.1-A1.4.

TABLE A1.1 Limits of Repeatability and Reproducibility for Maceral Groups

Coal Sample	Approximate Coal Rank	General Sample Location
48227	Medium volatile bituminous (mvb)	British Columbia, CA
48624	High volatile B bituminous(hvBb)	Illinois, USA
51851	High volatile A bituminous(hvAb)	Virginia, USA
52556	mvb	Virginia, USA
53407	Borderline: hvBb / hvAb	Ohio, USA
54683	hvAb	state not known, USA
55194	hvAb	West Virginia, USA
58770	Borderline: hvAb / mvb	West Virginia, USA
59323	Low volatile bituminous (lvb)	West Virginia, USA
59806	mvb	West Virginia, USA
60586	hvAb	West Virginia, USA
61173	lvb	West Virginia, USA

TABLE A1.2 Vitrinite (percent)

Coal Sample ID	Average $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
48227	57.994	0.927	3.972	2.596	11.120
48624	85.988	1.125	1.882	3.149	5.270
51851	63.000	1.085	2.026	3.037	5.673
52556	71.244	1.295	1.979	3.627	5.541
53407	80.438	1.252	3.218	3.506	9.011
54683	86.931	1.244	2.644	3.482	7.404
55194	68.719	1.323	1.721	3.705	4.818
58770	57.625	1.969	3.225	5.513	9.031
59323	73.769	1.185	1.747	3.318	4.893
59806	68.194	0.819	1.860	2.293	5.209
60586	65.250	0.994	2.799	2.782	7.837
61173	65.881	1.660	1.963	4.649	5.496

TABLE A1.3 Inertinite (percent)

Coal Sample ID	Average $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
48227	41.713	0.966	3.977	2.706	11.137
48624	9.900	1.137	1.762	3.185	4.933
51851	29.763	0.969	1.507	2.713	4.219
52556	25.969	1.733	2.228	4.853	6.239
53407	15.531	1.010	2.997	2.827	8.391
54683	8.369	1.213	2.463	3.397	6.896
55194	23.163	1.450	1.835	4.061	5.138
58770	38.225	1.846	3.431	5.168	9.607
59323	25.950	1.189	1.612	3.329	4.514
59806	29.919	0.958	1.754	2.681	4.912
60586	25.213	1.583	2.272	4.433	6.362
61173	33.913	1.785	2.075	4.997	5.811

TABLE A1.4 Liptinite (percent)

Coal Sample ID	Average $\bar{x}$	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
48227	0.294	0.135	0.350	0.377	0.979
48624	4.119	0.786	1.613	2.201	4.516
51851	7.244	0.714	1.213	1.998	3.397
52556	2.788	0.902	0.902	2.526	2.526
53407	4.031	0.525	1.101	1.470	3.084
54683	4.700	0.677	1.557	1.896	4.361
55194	8.119	1.163	1.823	3.257	5.104
58770	4.150	0.551	1.317	1.543	3.689
59323	0.281	0.066	0.330	0.185	0.923
59806	1.881	0.503	1.380	1.409	3.865
60586	9.538	1.018	1.699	2.850	4.758
61173	0.206	0.530	0.530	1.483	1.483