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Standard Test Method for Microscopical Determination of Volume Percent of Physical Components of Coal[1]

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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:*

D 121 Terminology of Coal and Coke[2]

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

D 2798 Test Method for Microscopical Determination of the Reflectance of Vitrinite in a Polished Specimen of Coal[2]

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal[2]

D 3177 Test Method for Total Sulfur in the Analysis Sample of Coal and Coke[2]

3. Terminology

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

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
	sclerotinite
	semifusinite

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *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.

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

3.3.2 *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.

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

3.3.4 *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**.

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

3.3.5.1 *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 Ro $\geq$ 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

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

manifestation). The volatile matter yield of inertinite is lower than that of other macerals in the same coal.

3.3.6 *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.

3.3.7 *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.

3.3.7.1 *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.

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

3.3.8.1 *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.

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

3.3.9.1 *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 D 2797. 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 D 2798).

See 3.2 for the classification used by most practitioners of this test method.

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

3.3.11 *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.

3.3.12 *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.

3.3.13 *sclerotinite*, *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.

3.3.14 *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.

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

3.3.15 *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.

3.3.15.1 *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.

3.3.16 *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.

3.3.16.1 *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 Ro = 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 D 2798.

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. The subcommittee is actively working on defining subcategories of the maceral group-vitrinite.

4. Summary of Test Method

4.1 The components in a representative crushed coal sample, prepared as prescribed in Practice D 2797, 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.

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*—The counter shall be capable of recording counts for at least six components.

7. Test Specimen

7.1 Prepare sample briquets in accordance with Practice D 2797.

8. Procedure

8.1 In accordance with present practice, maceral components counted shall be as defined in Section 3.

NOTE 1—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.

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. Generally, only organic components are counted. Pyrite and other visible mineral matter may be counted.

8.4 A better approximation of the total mineral content may be calculated from ash and sulfur contents determined in accordance with Test Methods D 3174 and D 3177. 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:

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

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.

10. Precision and Bias

10.1 *Precision*—The precision of the procedure in this test method is being determined.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

NOTE 2—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. Other factors also affect the precision of this test method.

11. Keywords

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