Designation: D 5061 – 92 (Reapproved 1997)

Standard Test Method for Microscopical Determination of Volume Percent of Textural Components in Metallurgical Coke[1]

This standard is issued under the fixed designation D 5061; 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 procedures used for determining the types and amounts of coke carbon forms and associated recognizable coal- and process-derived textural components in metallurgical coke in terms of volume percent. This test method does not include coke structural components such as coke pores, coke wall dimensions or other structural associations.

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 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analyses[2]

D 3997 Practice for Preparing Coke Samples for Microscopical Analysis by Reflected Light[2]

3. Terminology

3.1 *Definitions*—For additional definitions of terms used in this test method, refer to Terminology D 121.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *anisotropic*, *adj*—exhibiting optical properties of different values when viewed with an optical microscope having mutually exclusive polarized light, for example, crossed nicols.

3.2.2 *binder phase*, *n*—a continuous solid carbon matrix formed during the thermoplastic deformation of those coal macerals that become plastic during carbonization.

3.2.2.1 *Discussion*—The binder phase material is formed from the thermoplastic deformation of reactive (vitrinite and liptinite) and semi-inert (semifusinite) coal macerals of metallurgical bituminous coals. During thermoplasticity, the inert coal maceral and mineral are partly or wholly incorporated into the binder phase. Also, most of the coke pores are located in the binder phase.

3.2.3 *carbon form*, *n*—microscopically distinguishable carbonaceous textural components of coke, but excluding mineral carbonates.

3.2.3.1 *Discussion*—Carbon forms are recognized on the basis of their reflectance, anisotropy, and morphology. They are derived from the organic portion of coal and can be anisotropic or isotropic.

3.2.4 *domain*, *n*—a region of anisotropy in a carbon form that is distinctively marked by its isochromatic boundary and cleavage.

3.2.5 *circular anisotropic phase*, *n*—a group of binder-phase anisotropic carbon textures that are distinguished by approximately circular domains (that is length equals width) and composed of fine circular (0.5 to 1.0-µm), medium circular (1.0 to 1.5-µm), and coarse circular (1.5 to 2.0-µm) size categories.

3.2.6 *coke pore*, *n*—a microscopically distinguishable void that is a structural element of coke.

3.2.6.1 *Discussion*—Coke pores are considered to be nearly spherical-shaped voids created by the entrapment of gaseous volatiles during the solidification of thermoplastic coal. However, other types of voids can be distinguished in coke that include fractures or cracks, interconnected and elongated pores, and the open cell lumens of fusinite and semifusinite. The size and shape of the voids are coal rank and grade, and to some degree, process dependent. Pore sizes vary from tens of angstroms to tens of millimetres in any given coke.

3.2.7 *coke reactivity*, *n*—a measure of the mass loss when coke, held at a designated temperature, is contacted with gaseous carbon dioxide over a specific time interval.

3.2.8 *coke wall*, *n*—a predominantly carbonaceous layer that encloses a coke pore and which is a structural element and essence of coke.

3.2.9 *depositional carbon*, *n*—a group of carbon forms that are formed from cracking and nucleation of gas-phase hydrocarbon molecules during coal carbonization.

3.2.9.1 *pyrolytic carbon*, *n*—an anisotropic carbon form that is formed by the deposition of carbon parallel to an inert substrate causing the resulting texture to appear ribbon-like.

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[1] This test method is under the jurisdiction of ASTM Committee D-5 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] *Annual Book of ASTM Standards*, Vol 05.05.

3.2.9.2 *sooty carbon, n*—an isotropic carbon form comprised of approximately spherical particles of less than 1-µm diameter sometimes referred to as combustion black.

3.2.9.3 *spherulitic carbon, n*—a spherical anisotropic carbon form sometimes referred to as thermal black that is formed by the deposition of carbon concentrically around a nucleus.

3.2.10 *filler phase*, *n*—a discontinuous solid formed from coal macerals and minerals that do not deform thermoplastically during carbonization.

3.2.10.1 *Discussion*—The filler phase material is formed from coal macerals that are inert with respect to development of thermoplasticity (inertinite), the inorganic components of coal (minerals), as well as normally reactive coal entities that are noncoking or have been rendered inert by thermal oxidation, natural weathering or brecciation. These inert materials possess their original morphologies, but their reflectance and chemical properties have been altered prior to or during carbonization.

3.2.11 *green coke*, *n*—carbonaceous binder or filler phase material that has exceeded the temperature of thermoplasticity, but has not obtained the temperature of metallurgical coke.

3.2.11.1 *Discussion*—Green coke is recognized on the basis of relative reflectance in comparison to fully carbonized coke. Green coke exhibits varying degrees of lower reflectance than fully carbonized coke.

3.2.12 *incipient anisotropic phase, n*—a binder-phase carbon texture having a domain size (less than 0.5 µm) that is near the measuring resolution of the light microscope.

3.2.13 *isotropic phase*, *n*—a binder-phase carbon texture that exhibits optical properties that are the same in all directions when viewed with an optical microscope having mutually exclusive polarized light, for example, crossed nicols.

3.2.14 *lenticular anisotropic phase*, *n*—a group of binder-phase anisotropic carbon textures distinguished by their lens-shaped domains (that is, length (L) to width (W) ratio of $2W < L < 4W$) and subdivided based on domain widths as fine lenticular (1.0 to 3.0-µm), medium lenticular (3.0 to 8.0-µm), and coarse lenticular (8.0 to 12.0-µm) size categories.

3.2.15 *ribbon anisotropic phase*, *n*—a group of binder-phase anisotropic carbon textures distinguished by their ribbon-like domains (that is, length (L) to width (W) ratio of $L > 4W$), and subdivided based on domain width as fine ribbon (2.0 to 12.0-µm), medium ribbon (12.0 to 25.0-µm), and coarse ribbon (>25.0-µm) size categories.

3.2.16 *textural component*, *n*—the collective term used to describe carbon forms and recognizable coal- and process-derived components (binder-phase, filler-phase, and miscellaneous material) in coke.

3.2.17 *vitrinite type*, *n*—reflectance classes of vitrinite which span 0.1 % reflectance intervals.

3.2.17.1 *Discussion*—This term is commonly referred to as V-Type. For example, V-type 6 includes vitrinite reflectance values from 0.6 through 0.69 %.

4. Summary of Test Method

4.1 The textural components of coke (coke carbon forms and associated coal- and process-related components) in a representative crushed particulate coke sample, prepared in the form of a briquetted, polished specimen as described in Practice D 3997, are identified under a microscope according to their degree of anisotropism, carbon form domain sizes, boundary size, color of individual isochromatic domains, their morphology, relative reflectance, and other optical properties. The proportions of these textural 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 observed on the briquette surface. 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 determination of the volume percent of the textural components in coke is useful to characterize the optical properties of coke as it relates to utilization. Specifically, the technique has been used as an aid in determining coal blend proportions (after correcting for coke yield), and recognition of features present in the coke that can be responsible for coke quality or production problems such as reduced coke strength or difficulty in removing coke from commercial coke ovens, or both. The study of coke textures is also useful in promoting a better understanding of coke reactivity, and the relationship between coal petrography and its conversion to coke.[3]

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

6. Apparatus

6.1 *Microscope*—A high quality reflected-light microscope with a vertical illuminator and rotating mechanical stage is used, provided that the objective and eyepiece lenses permit resolution of objects on the order of 0.5 µm. The objective lens shall be of such construction that samples can be studied in oil with plane-polarized light. A minimum total magnification of approximately 500 diameters is recommended. Use of an accessory plate (quartz, gypsum, or mica), an analyzer, and polarizer combination is recommended to achieve optimum optical effect for discriminating among the various textural components. Either a prism or a partially reflecting glass plate may be employed in the illuminator. One eyepiece of the microscope must be fitted with a special ruled graticule disc.

6.1.1 *Eyepiece Disc*—The eyepiece shall contain a ruled graticule disc to enable size estimations and to provide a field-of-view grid for point counting. The design may be a squared pattern (10 by 10 squares) containing a bolder crosshair with one of the squares near the center crosshair intersection divided into 25 subsquares. The ruled portion of the disc shall cover at least one third of the field of view.

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 (referred to as the X and Y directions).

6.2 *Counter*—The counter shall be capable of recording counts for at least eight components (preferably twelve or

[3] Gray, R. J., and DeVanney, K. F., "Coke Carbon Forms: Microscopic Classification And Industrial Applications," International Journal of Coal Geology, Vol 6, pp. 277–297.

more) equipped with a totalizer. The counter design can either be mechanical or electrical.

6.3 *Immersion Oil*—The oil shall be a nondrying, noncorrosive, noncarcinogenic type having similar properties as used for coal microscopic techniques.

7. Organization of Analysis[3]

7.1 Textural components are grouped into three major categories; (*1*) binder phase carbon forms, (*2*) filler phase carbon forms (including coal-related inorganic material), and (*3*) miscellaneous materials. These categories are shown in summary form in Table 1. Volume percent of the various types of binder phase carbon forms should be determined during the first microscopic analyses. The volume percent of the filler phase (including coal-related inorganic material) should be determined as a second analysis. The miscellaneous materials are commonly determined during analysis of the filler phase.

7.1.1 *Binder Phase Carbon Form Determinations*—The components counted and kept separate shall be the following: isotropic, incipient, circular anisotropic (fine), circular anisotropic (medium), circular anisotropic (coarse), lenticular anisotropic (fine), lenticular anisotropic (medium), lenticular anisotropic (coarse), ribbon anisotropic (fine), ribbon anisotropic (medium), ribbon anisotropic (coarse). These binder phase categories relate to parent coal rank. When other components (filler phase, including coal-related inorganic material, and miscellaneous materials) are encountered, they are to be allocated to the appropriate binder phase category within which they are incorporated.

7.1.2 *Filler Phase Carbon Form Determinations (Including Coal-Related Inorganic Material)*—The components counted and kept separate shall be the following: one category for all binder phase carbons (no discrimination to specific binder phase components is necessary), organic inerts (fine), organic inerts (coarse), inorganic inerts (fine), inorganic inerts (coarse), miscellaneous inerts (by type, such as noncoking vitrinite, coked oxidized coal, coked brecciated coal, etc.), and others. Filler phase materials relate back to the parent coal type.

7.1.3 *Miscellaneous Materials Determination*—The components counted and kept separate shall be the following: depositional carbons (by type, that is, sooty, spherulitic, or pyrolytic) and any other observations such as additives (breeze, anthracite, petroleum coke, etc.), coal, and green coke (by relative degree). Due to the normally small occurrences or absence of these components, they are commonly counted during the filler phase counting procedure described in 7.1.2. These miscellaneous materials generally relate to coke plant processes and operational practices not directly related to parent coal rank or type.

NOTE 1—The degree of detail necessary will dictate the specific components quantified. The specified components to be counted in this test method assume that the operator is generating a detailed analysis. It may be practical to lump some components together. This is up to the discretion of the operator or based on agreement between such parties involved.

8. Procedure

8.1 Mount the coke briquette on a glass slide containing modeling clay, level using a specimen leveling press, and place on the stage of the microscope. Use a few drops of immersion oil on the briquette surface.

8.2 Adjust the microscope polarizer and analyzer to a crossed polarized position. Mount the accessory plate between the polarizer and the analyzer to the position that yields optimal retardation and color enhancement.

8.3 *Binder Phase Counting*—Position the coke briquette by means of the mechanical stage to the starting position. Identify four points per field under the special graticule or whipple disc (the intersection at each of the outermost corners). The exact directions traversed on the briquette are up to the preference of the operator. An example of one type of surface traverse is to move the mechanical stage 1 mm to the next field in the *X* direction and identify four points. Movement can be from left to right. Continue movement and counting in the *X* direction until the edge of the specimen is reached then move the specimen by means of the mechanical stage down 1 mm in the *Y* direction and begin traversing from the left to the right in the *X* direction. Continue this until a total of at least 1000 binder phase points (500 points on each of two different briquettes) are counted.

8.4 *Filler Phase and Miscellaneous Counting*—Position the coke briquette by means of the mechanical stage to the starting position. Identify four points per field under the special graticule or whipple disc (the intersection at each of the outermost corners). The exact directions traversed on the briquette are up to the preference of the operator. An example of one type of surface traverse is to move the mechanical stage 1 mm to the next field in the *X* direction and identify four points. Movement can be from left to right. Continue movement and counting in the *X* direction until the edge of the specimen is reached then move the specimen by means of the mechanical stage down 1 mm in the *Y* direction and begin traversing from the left to the right in the *X* direction. Continue this until a total of at least 1000 filler phase, and miscellaneous materials points are counted. It is preferred to count 500 points on each of two different briquettes. Carry one category for all binder phase carbon forms. Identification of the specific binder phase component is not necessary.

8.5 Do not count any of the field points that fall on the binding matrix (epoxy) used to make the coke briquette or coke pores (see 3.2.6).

9. Report

9.1 Report the following information:

9.1.1 The total number of points counted for microscopical analysis and volume percent of each category. Volume percents shall be rounded and reported to nearest 0.1 %.

9.1.2 The microscopical data using one of the following two options.

9.1.2.1 If carbon forms for estimating blend proportions are of major interest and it is desirable to see the three major categories separate, report the binder phase carbon forms, filler phase carbon (including coal-related inorganic material), and miscellaneous materials (refer to Table 2, Option A) each equal to 100 % separately.

9.1.2.2 As an alternative method of reporting the data, compute the volume percent of all components summed to equal 100 % (refer to Table 2, Option B).

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 of the procedure in this test method, bias has not been determined.

11. Keywords

11.1 carbon forms; coke; coke texture; microscopy

TABLE 1 Classification of Coke Textural Components

Binder Phase				
	Domain Dimensions		Parent Coal Vitrinite Type[A]	Bituminous Coal (Volatility)
	Width, µm	Length (*L*) to Width (*W*) Relation		
Isotropic	0.0	None	6, 7	high
Incipient (anisotropic)	0.5	$L = W$	8	high
Circular (anisotropic)				
Fine circular	0.5–1.0	$L = W$	9	high
Medium circular	1.0–1.5	$L = W$	10	high
Coarse circular	1.5–2.0	$L < W$	11	high to medium
Lenticular (anisotropic)				
Fine lenticular	1.0–3.0	$L \geq 2W$, $L < 4W$	12	high to medium
Medium lenticular	3.0–8.0	$L > 2W$, $L < 4W$	13	medium
Coarse lenticular	8.0–12.0	$L > 2W$, $L \leq 4W$	14	medium to low
Ribbon (anisotropic)				
Fine ribbon	2.0–12.0	$L > 4W$	15	medium to low
Medium ribbon	12.0–25.0	$L > 4W$	16	low
Coarse ribbon	25.0 +	$L > 4W$	17, 18	low
Filler Phase				
	Size, µm	Precursor Material		
Organic inerts:				
Fine	<50	micrinite, macrinite, inertodetrinite		
Coarse	≥50	semifusinite, fusinite, macrinite		
Miscellaneous inerts:				
Oxidized coal (coke)	...	oxidized coal		
Brecciated coal (coke)	...	brecciated coal		
Noncoking vitrinite (coke)	...	vitrinite too high or low in rank		
Inorganic inerts:	...	various types of mineral matter		
Fine	<50	coal mineral matter and bone coal		
Coarse	≥50	coal mineral matter and bone coal		
Miscellaneous Materials				
		Description		
Depositional carbon	...	sooty carbon spherulitic carbon pyrolytic carbon		
Additive carbons	...	coke breeze anthracite petroleum coke others		
Miscellaneous observations	...	green coke burnt or reacted coke others		

[A] Unusual coking conditions such as very fast (>30 mm (or 1.2 in.) per hour) or slow (<20 mm (or 0.8 in.) per hour) coking rates can cause the binder phase carbon forms produced to deviate from the expected coal rank category.

TABLE 2 Example of Reporting Coke Textural Components

Option A		Option B	
Binder Phase Carbon Forms	Volume %	Binder Phase Carbon Forms	Volume %
Isotropic	0.2	Isotropic	0.2
Incipient anisotropic	2.4	Incipient anisotropic	1.9
Circular anisotropic (fine)	50.6	Circular anisotropic (fine)	40.0
Circular anisotropic (medium)	12.1	Circular anisotropic (medium)	9.6
Circular anisotropic (coarse)	12.1	Circular anisotropic (coarse)	9.6
Lenticular anisotropic (fine)	2.0	Lenticular anisotropic (fine)	1.6
Lenticular anisotropic (medium)	4.1	Lenticular anisotropic (medium)	3.3
Lenticular anisotropic (coarse)	4.1	Lenticular anisotropic (coarse)	3.2
Ribbon anisotropic (fine)	9.0	Ribbon anisotropic (fine)	7.1
Ribbon anisotropic (medium)	2.3	Ribbon anisotropic (medium)	1.8
Ribbon anisotropic (coarse)	1.1	Ribbon anisotropic (coarse)	0.9
Total	100.0	Total	79.2
Filler Phase Carbon and Related Forms	Volume %	Filler Phase Carbon and Related forms	Volume %
Binder phase carbons	79.6	Organic inerts (fine)[A]	8.8
Organic inerts (fine)[A]	8.8	Organic inerts (coarse)[A]	7.2
Organic inerts (coarse)[A]	7.2		
Miscellaneous inerts:		Miscellaneous inerts:	
Noncoking vitrinite	0.0	Noncoking vitrinite	0.0
Coked oxidized coal	0.6	Coked oxidized coal	0.6
Coked brecciated coal	0.0	Coked brecciated coal	0.0
Inorganic inerts (fine)	3.2	Inorganic inerts (fine)	3.2
Inorganic inerts (coarse)	0.2	Inorganic inerts (coarse)	0.2
Pyritic minerals	0.4	Pyritic minerals	0.4
Total	100.0	Total	20.4
Miscellaneous Materials[B]	Volume %	Miscellaneous Materials	Volume %
Normal coke	98.9		
Depositional carbons:		Depositional carbons:	
Sooty	0.0	Sooty	0.0
Spherulitic	0.0	Spherulitic	0.0
Pyrolytic	0.4	Pyrolytic	0.4
Additives:		Additives:	
Breeze	0.0	Breeze	0.0
Anthracite	0.0	Anthracite	0.0
Petroleum coke	0.0	Petroleum coke	0.0
Other	0.0	Other	0.0
Noncoal related mineral matter	0.0	Noncoal related mineral matter	0.0
Other observations:		Other observations:[C]	
Coal	trace	Coal	(trace)
Green coke	0.7	Green coke	(0.7)
Other	0.0	Other	(0.0)
Total	100.0	Total	0.4

[A] Anisotropic organic inerts (semi-inerts) can also be kept separate in the filler phase category.
[B] The miscellaneous materials category listed in Table 2, Option A are not included in the 100 volume % totals of the binder phase or filler phase because they are not directly related to the original coal composition.
[C] Other observations with the volume percents in brackets listed in Table 2, Option B are not included in the volume percent totals.