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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Determination of Crystallite Size (L_c) of Calcined Petroleum Coke by X-Ray Diffraction¹

This standard is issued under the fixed designation D 5187; 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 determination of the mean crystallite thickness of a representative, pulverized sample of calcined petroleum coke by interpretation of a X-ray diffraction pattern produced through conventional X-ray scanning techniques.

1.2 Calcined petroleum coke contains crystallites of different thicknesses. This test method covers the determination of the average thickness of all crystallites in the sample by empirical interpretation of the X-ray diffraction pattern. The crystallite diameter (L_a) is not determined by this test method.

1.3 The values stated in SI (metric) units are to be regarded as the standard. The inch-pound units given in parentheses are provided for information purposes only.

1.4 *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 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis²

D 2013 Method for Preparing Coal Samples for Analysis²

D 2234 Test Methods for Collection of a Gross Sample of Coal²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *crystallites*—stacks of graphitic carbon platelets located parallel to one another.

3.1.2 L_c —the mean or average thickness of crystallites in a sample. It is expressed as a linear dimension in angstrom units, Å (10^{-1} nm).

3.1.3 $hkl(002)$ —the Miller indices of the crystalline planes

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² Annual Book of ASTM Standards, Vol 05.05.

³ Annual Book of ASTM Standards, Vol 05.02.

of graphite corresponding to a lattice spacing (d) of 3.35 Å.

3.1.4 Θ —the glancing angle produced when a parallel beam of uniform X-rays impinges upon a crystalline lattice. This angle is measured by the X-ray goniometer and is usually expressed in $^\circ 2\Theta$.

4. Summary of Test Method

4.1 A packed sample pulverized to less than 75 μm is subjected to a monochromatic X-ray beam and rotated to produce a diffraction pattern under specific conditions. The location and shape of the peak with $hkl = (002)$ at $d = 3.35$; Å is used to calculate the L_c by manual interpretation of the peak or by computer simulation.

5. Significance and Use

5.1 The crystallinity of petroleum coke, as reflected by the L_c value, is a general measure of quality affecting suitability for end use and is a function of the heat treatment.

5.2 The crystallite thickness is used to determine the extent of such heat treatment, for example, during calcination. The value of the L_c determined is not affected by coke microporosity or the presence of foreign, non-crystalline materials such as dedust oil.

6. Apparatus

6.1 *X-Ray Powder Diffractometer*, equipped with an X-ray source, a monochromator or filter for restricting the wavelength range, a sample holder, a radiation detector, a signal processor, and readout (chart or computer memory). The diffractometer must be capable of rate scanning at 1° per minute or incrementally step scanning at 0.2° per step.

6.2 *Sample Holders*, as specified by the manufacturer of the diffractometer that enables packing of a pulverized sample of sufficient thickness to expose a level, smooth surface to the X-ray beam.

6.3 *Briquetting Press*, capable of generating pressures up to 10 000 psi (69 MPa).

6.4 *Compressible Aluminum Caps*, used as a support for producing a briquetted sample.

6.5 *Silicon or Quartz Sample*, available from National Institute of Standards and Technology (NIST).

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be