



Standard Test Method for Determination of Crystallite Size (L_c) of Calcined Petroleum Coke by X-Ray Diffraction¹

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^{ε1} NOTE—Editorial changes were made throughout in December 2015.

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:²

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D2013 Practice for Preparing Coal Samples for Analysis

D2234/D2234M Practice for Collection of a Gross Sample of Coal

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E691 Practice for Conducting an Interlaboratory Study to

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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.

Determine the Precision of a Test Method

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

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

3.1.2 L_c , n —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 $hkl(002)$, n —the Miller indices of the crystalline planes of graphite corresponding to a lattice spacing (d) of 3.35 Å.

3.1.4 *Theta angle*, Θ , n —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 degrees TwoTheta ° (2Θ).

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