



Standard Test Method for Microscopical Analysis by Reflected Light and Determination of Mesophase in a Pitch¹

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1. Scope

1.1 This test method covers laboratory procedures for the preparation of granular and melted samples for microscopic analysis using reflected light to identify and estimate the amount and size of the mesophase.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

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

D329 Specification for Acetone

D1160 Test Method for Distillation of Petroleum Products at Reduced Pressure

D2318 Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch

D3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)

D4296 Practice for Sampling Pitch

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E562 Test Method for Determining Volume Fraction by Systematic Manual Point Count

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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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.

3.1.1 *cenospheres*—usually a minor component of coal tar pitch. They are formed by the rapid pyrolysis of unconfined coal particles that are carried over from the coke oven to the tar. Microscopically, they appear like hollow spheres or segments thereof (see Fig. 1), and are typically sized from about 10 to 500 μm . In polarized light (crossed polarizers), a cenosphere may be optically active. The size of the anisotropic pattern or mosaic depends upon the rank of the coal carbonized. Cenospheres are harder than the continuous phase and polish in relief (see Fig. 1).

3.1.2 *coke-oven-coke*—usually a minor component of coal tar pitch. It originates in carry-over from the coke oven to the tar side. It differs from cenospheres only in terms of its shape and porosity. Coke-oven-coke is angular and less porous.

3.1.3 *isotropic phase*—usually the predominant, and continuous, phase. It is a complex mixture of organic aromatic compounds composed mainly of carbon and hydrogen. At room temperature, the isotropic phase is a glass-like solid. It is optically inactive in polarized light (see Fig. 1 and Fig. 2).

3.1.4 *mesophase*—an optically anisotropic liquid crystal carbonaceous phase that forms from the parent liquor when molecular size, shape, and distribution are favorable. In the early stages of its development, mesophase usually appears as spheroids. The planar molecules are lined up equatorially as shown schematically in Fig. 3. This equatorial arrangement may be distinguished in crossed polarized light. Under crossed polarizers, the distinctive mesophase spheroids, with their complex extinction patterns shown in Fig. 2, can be readily seen.³

3.1.4.1 *spheroids*—At magnifications of 400 $\times$ and 500 $\times$, the minimum spheroid size which can be resolved with confidence is 4 μm in diameter. At magnifications of 1000 to 1800 $\times$, the minimum spheroid size that can be resolved with confidence is about 2 μm in diameter. Typically, the upper size may be 100 μm . Mesophase spheroids are relatively soft and do not form relief structures (see Fig. 4). Quinoline insoluble particles often aggregate at the interface between the continuous isotropic phase and mesophase.

³ A more complete discussion will be found in a paper by Honda, H., Kimura, H., and Sanada, Y., "Changes of Pleochroism and Extinction Contours in Carbonaceous Mesophase," *Carbon*, 9, 1971, pp. 695–697.