



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 the 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:

D 329 Specification for Acetone²

D 1160 Test Method for Distillation of Petroleum Products at Reduced Pressures³

D 2318 Test Method for Quinoline-Insoluble (QI) Content of Tar and Pitch³

D 3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)³

D 4296 Practice for Sampling Pitch⁴

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

E 562 Test Method for Determining Volume Fraction by Systematic Manual Point Count⁶

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 and Lubricants and is the direct responsibility of Subcommittee D02.05.OF on Industrial Pitches.

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² Annual Book of ASTM Standards, Vol 06.04.

³ Annual Book of ASTM Standards, Vol 05.01.

⁴ Annual Book of ASTM Standards, Vol 05.02.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Annual Book of ASTM Standards, Vol 03.01.

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 *mineral matter*—formed when minute particles of the coke oven charge are carried over into the coke oven collecting main during the charging operation. The tiny coal particles are digested in the collecting main tar, resulting in a residue that is rich in mineral matter. This mineral matter is identified under bright field illumination by its high reflectivity, in the case of pyrite, and its low reflectance in the case of clay, quartz, and carbonates. The association of mineral matter with insoluble organic matter from coal aids in its identification.

3.1.4 *refractory*—usually a minor component that originates from the coke oven walls, doors, and patches due to wear and degeneration; another component is charge hole sealant. It can be recognized under the microscope through optical properties, hardness, shape, and associated minerals.

3.1.5 *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).

3.1.6 *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

