



Standard Practice for Collection of Calcined Petroleum Coke Samples for Analysis¹

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1. Scope

1.1 This practice covers procedures for the collection of calcined petroleum coke samples from conveyor belts or transfer points. These samples may be used for physical and chemical analyses.

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:²

D 4702 Guide for Inspecting Crosscut, Sweep-Arm, and Auger Mechanical Coal Sampling Systems for Conformance with Current ASTM Standards

D 5709 Test Method of Sieve Analysis of Petroleum Coke

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *accuracy*—generally, a term used to indicate the reliability of a sample, a measurement, or an observation. Specifically, a measure of closeness of agreement between an experimental result and the true value.

3.1.1.1 *Discussion*—Example: the observed and true sulfur content of a calcined petroleum coke consignment. This measure is affected by chance errors as well as by bias.

3.1.2 *analysis sample*—the reduced and divided representative portion of the bulk sample, prepared for use in the laboratory.

3.1.3 *gross sample*—a sample representing one lot of petroleum coke and composed of a number of increments on which no reduction or division has been performed.

3.1.4 *increment*—a small portion of a lot collected by one operation of a sampling device and normally combined with other increments from the lot to make a gross sample.

3.1.5 *lot*—a quantity of coke represented by a gross sample.

3.1.6 *representative sample*—a sample collected in such a manner that the size consist is the same as the lot.

3.1.7 *sample*—a quantity of petroleum coke taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity.

3.1.8 *sample preparation*—the process that may include drying, crushing, division, and mixing of a gross sample for the purpose of obtaining an unbiased analysis sample.

3.1.9 *size consist*—the particle size distribution of a material; for example, petroleum coke.

3.1.10 *top size*—the size of the smallest opening of one sieve of a series upon which is cumulatively retained a total of less than 5 % of the sample. This defined top size is not to be confused with the size of the largest particle in a lot.

4. Significance and Use

4.1 Data obtained from calcined petroleum coke samples are used in commercial transactions, controlling plant operations, and allocating production costs. Use of standard sampling procedures facilitates the task of obtaining a sample to represent an entire lot of calcined petroleum coke.

4.2 This practice gives general procedures for the collection of calcined petroleum coke samples and is intended to provide useful methodology for the collection of a sample to represent a lot of calcined petroleum coke. The variety of calcined petroleum coke handling facilities and sampling applications preclude the publication of detailed procedures for every sampling situation.

5. Organization of Sampling Operation

5.1 Place of Sampling:

5.1.1 Sample calcined petroleum coke while it is being loaded into or unloaded from railcars, ships, barges, or trucks, or when discharged from kilns, supply bins, grab buckets, belt conveyors, or other conveying equipment. The sample should be collected as close to the point of interest as possible.

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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.

5.1.2 Samples collected from the stationary surface of calcined petroleum coke in stockpiles, bins, railcars, trucks, ships, or barges may be unreliable because of size segregation and shall not be used for determining conformance to specifications unless the purchaser and the seller agree. If it is necessary to collect a stationary sample of calcined petroleum coke, use a sampling method mutually acceptable to all involved parties.

5.2 Collection of Gross Sample:

5.2.1 Collect increments systematically, and with such frequency to produce a representative sample mutually acceptable to all involved parties.

5.2.2 It is recommended the gross sample contain at least the required minimum number of increments as outlined in Table 1.

5.2.3 The quantity of sample to be taken assumes that the top size of the coke is 25 mm (1 in.) or less.

5.3 Four Conditions of Increment Collection are Recognized:

5.3.1 *Condition A (stopped-belt cut)*, in which a loaded conveyor belt is stopped and a full cross-section cut with parallel sides is removed from the calcined petroleum coke stream. The distance between the parallel faces shall not be less than three times the normal top size of the calcined petroleum coke.

5.3.2 *Condition B (full-stream cut)*, in which a full cross section cut is removed from a moving stream of calcined petroleum coke either by manual or mechanical means.

5.3.3 *Condition C (part-stream cut)*, in which a portion, not a full cross section, is removed from a moving stream of calcined petroleum coke.

5.3.4 *Condition D (stationary sampling)*, in which a portion of calcined petroleum coke is collected from a pile, a rail car, a barge, truck, or a shiphold.

5.4 Size of Increments:

5.4.1 Collect increments using a specially designed tool or mechanical device capable of taking increments of equal mass. Increments comprising a gross sample shall not weigh less than 2 kg (4.4 lb) each.

5.5 Quantities Represented:

5.5.1 Collect a gross sample for each 2000 metric tons or fraction thereof, or in case of larger tonnage, as may be mutually agreed upon by all involved parties. Practical experience, however, indicates the maximum size of a lot of calcined petroleum coke to be represented by one gross sample shall not exceed 15 000 metric tons (see Table 1). Separate samples shall be taken from each lot of calcined petroleum coke arising from a different source or known to be of different quality or size.

5.6 Distribution of Increments:

5.6.1 It is essential that the increments be distributed throughout the lot to be sampled. This distribution is related to the entire volume of the lot, not merely its surface or any linear direction through it or over it. If circumstances prevent the sampler from applying this principle, then the lot is sampled only in part, and the gross sample is representative only of this part. The spacing of increments shall be varied if the possibility exists that increment collection may get in phase with the sequence of calcined petroleum coke variability. Example: routine sampling where increment collection from a continuous stream (conveyor belt), and where increment collection is automatic and its sequence coincides with the highs or lows in the content of fines.

6. Sampling Requirements

6.1 *Dimensions of Sampling Device*—The opening of the sampling device shall be at least three times the top-size of the calcined petroleum coke. For practical reasons, however, it is recommended that the opening of any sampling device be not less than 32 mm (1¼ in.), regardless of the top size of the calcined petroleum coke. The sampling device shall be of sufficient capacity to completely retain or entirely pass the increment without loss or spillage at the maximum flow rate of material.

6.2 *Movement of Sampling Device*—In sampling from moving streams of calcined petroleum coke, the sampling device, either mechanical or manual, shall be designed to minimize disturbance of the calcined petroleum coke, thereby avoiding separation of various densities and sizes, or both.

6.3 Mechanical Sampling Devices:

6.3.1 *Cross-stream Samplers*—The cross-stream sampler is designed to extract increments from falling streams of calcined petroleum coke at the end of conveyors or at transfer points between two conveyors, or both. Increments are obtained by a single passage of the cross-stream cutting device through the falling stream. Cross-stream cutter speeds must be carefully set and maintained so as to prevent segregation and rejection due to disturbance of the falling stream. Cutter speeds of 454 mm/s (18 in./s) and less have been shown to produce acceptable results. Additionally, the cutter must traverse the stream at a constant velocity and also for each pass.

6.3.2 *Sweep-arm Samplers*—The sweep-arm sampler is designed to extract increments directly from the conveyor surface. Increments are obtained by a single rotation of the sweep-arm cutting device across the moving, loaded conveyor in a path perpendicular to its material flow. Practical experience indicates that the velocity with which the sweep-arm cutter travels across the conveyor shall be as high as possible. The conveyor belt curvature shall be reasonably profiled to match that of the bottom of the sweep-arm cutter as it rotates across the material flow. Any blades, brushes, or skirts fitted to the back of the sweep-arm cutter shall be routinely inspected and adjusted so that they maintain close contact with the surface of the conveyor to further insure complete increment extraction. Exercise care to ensure that only material collected by the cutter is retained in the sample.

6.4 Mechanical System Features:

6.4.1 It is essential that mechanized systems as a whole, including sampling devices, chutes, feed conveyors, crushers,

TABLE 1 Minimum Increments Required Per Lot

Sample Lot, metric tons	Number of Increments
1 to 2000	15
2001 to 5000	20
5001 to 10 000	25
10 001 to 15 000	30

and other devices be self-cleaning and non-clogging and be designed in a manner that facilitates routine inspection, maintenance, and performance testing.

6.4.2 If a secondary sampler is used in conjunction with the primary sampler (cutter), it is recommended to collect a minimum of three secondary increments per primary.

6.5 *Criteria of Satisfactory Performance*—A satisfactory sampling arrangement is one that produces a sample with accuracy acceptable to all involved parties. One fundamental characteristic of such an arrangement is that the size consist of the sample will adequately represent the true size consist of the calcined petroleum coke. Mechanical sampling systems will be performance tested initially and at regular intervals thereafter to determine that the sample adequately represents the calcined petroleum coke under the normal range of conditions expected. Performance testing can include, but is not limited to: (1) Statistically sound comparisons of sample analysis obtained from the sampling system with stopped-belt samples of the same calcined petroleum coke; (2) inspections by qualified personnel; (3) comparison of actual versus calculated increment weights; and (4) comparison of the weight or volume of collected sample with that of the total flow of calcined petroleum coke to assure proper and consistent extraction criteria. In all cases, performance testing will be conducted utilizing consistent practice and execution acceptable to all interested parties.

NOTE 1—See Guide D 4702 which can serve as a reference for the inspection of mechanical sampling systems.

6.6 *Preservation of Moisture*—In cases where determination of sample moisture is important, increments obtained by sampling shall be protected from changes in composition due to exposure to rain, snow, wind, sun, contact with absorbent materials, extremes of temperature, and excessive air flow through the sampling system. Such samples shall be stored in moisture-tight containers. Metal cans with airtight lids, or heavy vapor-impervious bags, properly sealed, are satisfactory for this purpose.

7. Processing of the Test Sample

7.1 Since some analyses must be performed on the unprepared calcined petroleum coke and others on crushed or pulverized material, prior knowledge of the analytical requirements is necessary before beginning sample preparation.

7.2 Process the samples collected for sieve analysis of calcined petroleum coke in accordance with the procedure given in Test Method D 5709.

7.3 Process the samples taken for the determination of total moisture and chemical analysis in accordance with the procedure given in D 6969, “Draft Standard Practice for the Preparation of Calcined Petroleum Coke Samples for Analysis.”

8. Keywords

8.1 calcined petroleum coke; sample preparation; sampling

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