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Standard Test Method for Sieve Analysis of Petroleum Coke[1]

This standard is issued under the fixed designation D5709; 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 details a procedure for performing particle size distribution analysis by dry sieve testing on green petroleum coke with a topsize of no more than 75 mm and calcined petroleum coke with a topsize of no more than 25 mm. Size fractions go down to and include 4.75 mm for green petroleum coke and 75 µm for calcined petroleum coke.

NOTE 1—To convert units, see Table 1 on nominal dimensions in Specification E11. For example, 75 mm is approximately equivalent to a nominal sieve opening of 3 in. and 25 mm to a nominal sieve opening of 1 in. Likewise, 4.75 mm can be converted to approximately 0.187 in. and 75 microns to 0.0029 in.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. The sieve size is reported as U.S.A. standard test series in any units listed in Table 1 on nominal dimensions of Specification E11, or their commercial size equivalents.

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]

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

D4749 Test Method for Performing the Sieve Analysis of Coal and Designating Coal Size

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *bulk sample*—the reduced and divided representative portion of the gross sample as prepared for shipment to and received by a laboratory, to be prepared for analysis.

3.1.2 *gross sample*—the original, uncrushed, representative portion taken from a shipment or lot of coke.

3.1.3 *lot*—a quantity of coke to be represented by a gross sample.

3.1.4 *representative sample*—a sample collected in such a manner that every particle in the lot to be sampled is equally represented in the gross sample.

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

4. Summary of Test Method

4.1 A representative coke sample is divided into ranges of particle size by the use of a series of square-holed sieves.

5. Significance and Use

5.1 The test method concerns the sieving of coke into designated size fractions for the purpose of characterizing the material as to its particle size distribution. It requires the use of standard sieves, standard sampling methods, standard sample preparation methods, and a minimum initial sample mass based on lot topsize. Suggestions are given for industry typical sieve stacks for both green and calcined petroleum coke.

5.2 Particle size distribution is significant in that many physical characteristics of a coke are related to such a distribution including bulk density and surface area. Nuisance characteristics, such as excessive fines in a lot, can also be controlled.

5.3 Results from this test method are useful in determining whether a coke lot meets purchase specifications, for classification purposes, and for quality control. The results of this test

[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 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.

TABLE 1 Industry Typical Sieves

Calcined Petroleum Coke	Green Petroleum Coke
25.0 mm	25.0 mm
19.0 mm	12.5 mm
12.5 mm	4.75 mm
4.75 mm	–4.75 mm
3.35 mm	
2.36 mm	
1.18 mm	
600 µm	
300 µm	
212 µm	
150 µm	
75 µm	
–75 µm	

TABLE 2 Initial Minimum Test Sample Mass Requirements for Sieve Analysis

Topsize	Type of Coke	Initial Sample Mass Requirement, g	Expected Relative Error, %
75 mm	Green	50,000	6
50 mm	Green	30,000	6
25 mm	Green/Calcined	1,500	3
19 mm	Green/Calcined	1,300	1
12.5 mm	Green/Calcined	1,000	1
4.75 mm	Green/Calcined	800	1
2.36 mm	Green/Calcined	700	1
1.18 mm	Green/Calcined	500	1
600 µm	Green/Calcined	300	1
300 µm	Green/Calcined	100	1
150 µm[A]	Green/Calcined	50	1

[A] For topsize less than 150 µm, use an initial sample mass requirement of 50 g.

method can also be used to predict the performance of a particular lot of coke in a process.

6. Interferences

6.1 A sieve analysis is very sensitive to the sieve cloth and sieve cloth-frame integrity. Minor separations of the sieve cloth from the frame such as one broken sieve wire, and slight distortions of sieve wires, can cause serious inaccuracies in the final results of a sieve analysis.

6.2 Blinding of or a reduction in the number of openings in a sieve due to a collection of particles caught in the mesh can introduce errors.

6.3 Flooding or overloading of any sieve with particles reduces the probability of any given particle encountering an opening in the sieve.

7. Apparatus

7.1 *Sieves*:

7.1.1 Sieves will be used in a descending size opening sequence, larger mesh openings above smaller.

7.1.1.1 Typical sets of sieves to be used are listed in Table 1.

NOTE 2—Sets of sieves are often modified. Typically, specifications on sets of sieves are negotiated between the buyer and the seller. The actual sequence used by the operator performing the analysis can vary. For example, intermediate sieves can be chosen to avoid sieve flooding and to make the sieving operation more efficient. Table 1 on nominal dimensions in Specification E11 is to be used as a guide.

7.1.2 A topsize sieve shall be used.

7.1.3 Wire sieve cloth and frames used will conform to Specification E11.

7.1.4 Wire composition and types of frames must be sized properly for potential sieving operations. Stainless steel sieve cloth is very resistant to distortion and preferred over softer metals.

7.1.5 Collecting pans and sieve covers designed to fit the sieves are required.

7.1.6 Check Specification E11 for more details on standard sieves, service checks, and calibration.

7.2 *Sieve Shaker*:

7.2.1 Use a batch type sieve shaker.[3]

7.2.2 For sieving small quantities of coke or very fine coke (below 70 mesh), use a laboratory type sieving machine.[4]

8. Sample Preparation

8.1 A representative gross sample of the coke lot must be collected using appropriate procedures from Practice D346, Test Methods D2234/D2234M, or Practice D4057. (**Warning**—The gross sample must not be crushed or reduced in topsize during the gross sample collection process or during subsequent divisions of the sample.)

8.2 The gross sample is divided into a smaller bulk sample following guidelines in Test Methods D2234/D2234M. The bulk sample must remain representative including no loss in topsize. Keeping in mind the initial sample mass requirements (see Table 2), the bulk sample must be at least twice the largest minimum mass that you estimate will be required for the analysis.

8.3 Upon delivery of the bulk sample to the laboratory, the sample shall be stored in a safe, dry location. Prevent any size degradation, loss of mass, or contamination of the sample until needed for the sieve analysis.

8.4 Immediately prior to the sieve analysis, examine the bulk sample determining whether it is dry and free flowing. If not, use the air drying apparatus and drying procedure of Practice D2013.

8.5 Determine the initial minimum test sample mass required for the analysis from Table 2.

[3] The sole source of supply of the model number TS-1 Gilson Testing Screen machine known to the committee at this time is Gilson Company, Inc., P. O. Box 677, Worthington, OH 43085-0677. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] The sole source of supply of the Ro-Tap Testing Sieve Shaker known to the committee at this time is W. S. Tyler, Inc., 3200 Bessemer City Rd., P. O. Box 8900, Gastonia, NC 28053-9065. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

TABLE 3 Suggested Starting Points for Sieving Time

Time, min	Size of Coke, mm
5	coarse (greater than 12.5)
10	medium (1.18 to 12.5)
15	fine (less than 1.18)

8.6 Reduce the bulk sample to the recommended minimum test sample mass required using the division methods outlined in Test Methods D2234/D2234M or D4749.

9. Procedure

9.1 Accurately weigh the minimum test sample mass (see 8.6) before sieving with a precision equal to or better than 0.5 % of the fraction being weighed. This mass is M_i (initial test sample mass).

9.2 Start with the sieve having the largest required opening.

9.3 All sieving is to be done using a batch type sieve shaker.

NOTE 3—It is recommended that coke 50 mm in diameter and larger be hand sieved by the methods outlined in Test Method D4749 rather than attempting the use of a mechanical sieving device.

9.4 Clean sieves prior to each use following the method recommended by the sieve manufacturer. If this information is unavailable, thoroughly brush the sieves using an appropriate bristle or soft metal brush. Do not distort or damage sieves during this process.

9.5 Limit the portions of coke used for each sieving so that all coke particles will be in direct contact with the mesh at the completion of sieving on each successive sieve.

9.5.1 To determine the length of sieving time, refer to Table 3 for an estimated starting point. Use a sample divider as described in Practice D2013 to form four subsamples from a gross sample of a coke similar to that being tested. Sieve one of these for the time given as an appropriate starting point, a second for starting point plus 1 min, a third for starting point plus 2 min, and a fourth for starting point plus 3 min. Tabulate the results of these tests by the percentages retained on each sieve (see Section 10), and the length of sieving time required to stabilize the sieving result without particle size degradation should be readily apparent and can be established. If necessary, keep adding additional minutes until the percentages are stable.

9.6 Sieve until all portions of the sample are used. Combine all separately sieved material representing a particular size fraction, but obtained from sieving separate portions of the same sample.

9.7 Continue sieving with successive sieves which have the desired size openings until the sieve having the smallest desired size opening is used. Combine all the pan contents that have passed through this smallest size opening and consider these a particle size fraction.

NOTE 4—When larger particles are present that can physically affect the dimensional stability of sieve openings or possibly damage the sieve cloth, use a cover sieve (protective sieve of a larger mesh) to keep coarse particles off the surface of the finer sieve.

9.8 Sieving can be done by grouping sieves having the desired size openings, always stacking larger sieve openings above smaller, thus accomplishing the sieving in fewer operations. This is known as nesting sieves.

9.9 Always use sieve covers and collecting pans to prevent loss of fines and larger coke particles.

9.10 Weigh each size fraction of sieved coke including the bottom pan size fraction with a precision equal to or better than 0.5 % of the fraction being weighed. These masses are m_f (final size fraction mass).

9.11 Be aware that the objective of mechanically shaking sieves is to place all of the given particles of a given size on the appropriate sieve while avoiding size degradation of any of these particles. Larger coke particles are especially susceptible to particle degradation; avoid excessive sieving time (see 9.5.1).

10. Calculation

10.1 Calculate the sum of the size fractions including pan fraction (see 9.9) and call the sum M_f (combined final mass).

10.1.1 Convert all masses to the same units before calculation, that is, kilograms or grams.

10.1.2 Convert and utilize the masses of the size fractions by both multiplying and making proper use of significant figures. For example, if a size fraction weighed 11.25 kg another 204 g, and another 148 g, determine all the masses to the nearest 0.01 kg (since 11.25 kg is reported to the nearest 0.01 kg) before proceeding with calculations, as follows:

(1)

$$\begin{array}{r} 11.25\ \text{kg} \\ 0.20\ \text{kg} \\ 0.15\ \text{kg} \\ \hline 11.60\ \text{kg} \end{array}$$

10.2 If the percentage mass loss or gain is over 1 %, reject the analysis and make another test. The formula for the calculation of the percentage mass loss or gain is as follows:

$$\%\ M = \frac{(M_f - M_i)100}{M_i} \tag{2}$$

where:

M_f = combined final mass (10.1), g or kg,
M_i = initial test sample mass (9.1), g or kg, and
$\%M$ = % mass loss or gain upon sieving, g or kg.

A mass gain will result in a positive percent while a mass loss will result in a negative percent.

10.2.1 If the variation is greater than the above tolerance of 1 %, recheck the figures for possible errors in determining mass, calculating, blinding of the sieve openings, or accidental spillage. If a calculation, transcription, or other error is detected and correctable, correct the error. If the resulting variation from initial sample weight is within the 1 % tolerance, accept and report the corrected results. If the source of error is not detected, or if it is detected but uncorrectable, repeat the test.

10.3 Convert the mass m_f (see 9.8) of an individual size fraction to a percentage basis by dividing the mass of that portion by the combined final mass M_f, or by the initial test sample mass M_i as follows:

$$\%\ \text{m} = \frac{M_f}{M_f o_r M_i} * 100 \tag{3}$$

where:

m_f = final size fraction mass (9.9), g or kg,
M_f = combined final mass (10.1), g or kg,
M_i = initial test sample mass (9.1), g or kg, and
% m = % mass of size fraction, g or kg.

Calculate each mass % to the nearest 0.01 % and then round to the nearest 0.1 %.

10.3.1 The sum of the fractional masses, rather than the original sample mass, can be used as a 100 % for calculation of the sieve analysis percentages. However, the percent mass loss or gain must be stated in the report, and it must be stated that the sum of the fractional masses rather than the original sample mass was used to force the total of the fractional mass percentages to equal 100 %.

10.4 Calculate cumulative percent retained figures by adding the percentages of each individual size fraction from the largest size to the smallest size.

10.5 Calculate cumulative percent passing figures by adding the percentages of each individual size fraction from the smallest size to the largest size.

11. Report

11.1 The topsize, sieve opening (mm or µm) or U.S.A. standard sieve number of any other unit listed in Table 1 on nominal dimensions in Specification E11 or their commercial size equivalents.

11.2 The % mass loss or gain, in grams or kilograms, if needed.

11.3 The individual size fractions as percentages (10.3), in grams or kilograms.

11.4 The cumulative percent retained (10.4), in grams or kilograms.

11.5 The cumulative percent passing (10.4), in grams or kilograms.

11.6 Further coke characterization tests are frequently required and these results may also be reported. For example, particle shape, an estimate of the particle shape distribution when shapes are not uniform, non-coke particles or debris found on the sieves. This information should be readily available upon visual observation of sized material lying on the surface of the sieve.

12. Precision and Bias

12.1 No precision statement (reproducibility) has been developed for this test method because of the impracticality of obtaining, transporting, and handling representative splits of the materials in the quantities that would be needed to establish the precision statement. The precision (repeatability) of this test method has not been determined at this time.

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, bias has not been determined.

13. Keywords

13.1 calcined petroleum coke; green petroleum coke; particle size analysis; screening; sieve