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An American National Standard

Standard Practice for Preparation of Calcined Petroleum Coke Samples for Analysis[1]

This standard is issued under the fixed designation D 6969; 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 practice covers the preparation procedures necessary for the reduction and division of calcined petroleum coke samples in order to generate appropriate analytical samples upon which physical and chemical analytical tests will be performed.

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 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)

D 2638 Test Method of Real Density of Calcined Petroleum Coke by Helium Pycnometer

D 4292 Test Method of Determination for Vibrated Bulk Density of Calcined Petroleum Coke

D 4422 Test Method of Ash in the Analysis of Petroleum Coke

D 4930 Test Method for Dust Control Material on Calcined Petroleum Coke

D 4931 Test Method for Gross Moisture in Green Petroleum Coke

D 5004 Test Method of Real Density for Calcined Petroleum Coke, Xylene Displacement

D 5056 Test Method for Trace Metals in Petroleum Coke by Atomic Absorption

D 5187 Test Method for Determination of Crystallite Size (L_c) of Calcined Petroleum Coke by X-Ray Diffraction

D 5600 Test Method for Trace Metals in Petroleum Coke by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D 5709 Test Method for Sieve Analysis of Petroleum Coke

D 6376 Test Method for Determination of Trace Metals in Petroleum Coke by Wavelength Dispersive X-ray Fluorescence Spectroscopy

3. Terminology

3.1 *Definitions:*

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

3.1.2 *composite sample*—a sample, representative of an entire consignment of calcined petroleum coke, generated by mixing portions of gross samples from different lots together in mass fractions proportioned to the consignment.

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

3.1.4 *intermediate sample*—a sample, representative of a gross sample, upon which no analysis is to be performed, yet required for generation of analysis samples after undergoing further division and reduction.

3.1.5 *lot*—a quantity of calcined petroleum coke to be represented by a gross sample.

3.1.6 *riffle*—a manual sample divider which splits the sample stream into a number of alternate elements.

3.1.7 *sample division*—the process whereby a sample is reduced in mass without change in particle size.

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 *sample reduction*—the process whereby a sample is reduced in particle size by crushing or grinding without significant change in chemical properties.

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.

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[1] This practice 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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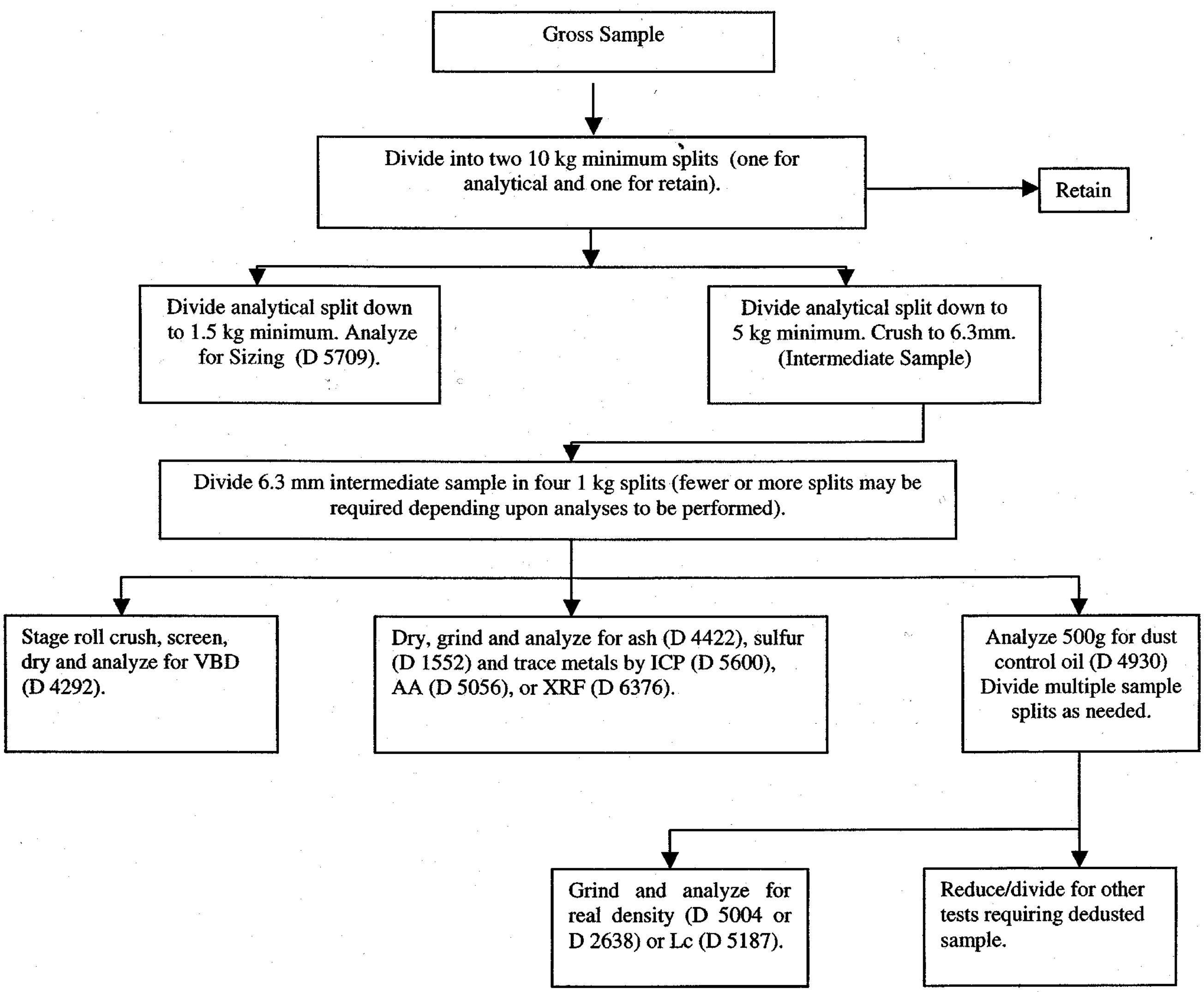
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Example of Mixing Gross Samples to Generate a Weighted Average Vessel Composite Sample

	Lot 1 Hold 1	Lot 2 Hold 2	Lot 3 Hold 3	Lot 4 Hold 4	Lot 5 Hold 5	Lot 6 Hold 6	Lot 7 Hold 7	Total
Lot tonnage (short tons)	7800	7500	8200	7500	8000	7800	7900	54 700
Target sample mass mixed into composite (grams)	1426 ± 29	1371 ± 27	1499 ± 30	1371 ± 27	1463 ± 29	1426 ± 29	1444 ± 29	10 000 ± 200



NOTE—The tasks outlined in this figure are not necessarily required for analysis.

FIG. 1 Preparation of Calcined Petroleum Coke

4. Significance and Use

4.1 This practice provides field personnel and laboratories with standard procedures for dividing, reducing, and mixing gross samples and intermediate samples, such that the resulting prepared analysis samples are more uniform from laboratory to laboratory. Adherence to these guidelines is expected to provide significant reduction in inter-laboratory variability.

5. Organization of Sample Preparation

5.1 *Initial Division of Gross Sample*:

5.1.1 Determine the required analyses for the lot and manage the division steps accordingly to achieve enough analytical sample to perform all required analyses in duplicate (see flowchart in Fig. 1 as an example). Divide the gross sample by use of a riffle or rotary sample divider to an intermediate sample of sufficient mass.

5.1.2 Exercise care in the division operation to preserve the particle size distribution of the gross sample. Riffle openings must be at least three times the top size of the calcined petroleum coke being divided. A feed hopper or vibratory feeder, or both, are recommended to feed the coke into the riffle. Enclosed riffles are recommended to minimize dusting and loss of sample.

5.2 *Subsequent Reduction and Division*:

5.2.1 Crushing processes are to be carried out such that the apparatus does not contribute significant impurities into the analysis sample. For example, if trace metal analyses are to be performed on a number 60 mesh analysis sample, a ring and puck mill with tungsten carbide grinding components is recommended to minimize the metallic impurities of analytical interest that may be added to sample.

5.2.2 If trace metals are not required for testing on a number 60 mesh analysis sample, a bench-top hammer mill or ball mill are adequate for performing the grinding operation.

5.3 Refer to actual test methods for size and mass of samples required for analysis.

5.4 Removal of dedusting oil may be required for analytical purposes and for subsequent analyses such as real density or porosity. Oil may be extracted and quantified by Test Method D 4931 or thermally removed.

5.5 Mixing of gross samples to generate a composite sample representing more than one lot is frequently required. Mixing must be planned such that the final composite sample has a mass of no less than 10 kg so that all the required analyses may be performed.

5.5.1 Divide each gross sample down into an intermediate sample weighing no less than 1.5 kg such that each intermediate sample is still representative for sizing determination by Test Method D 5709.

5.5.2 Each lot sample will be proportionately represented in the composite, as the lot tonnage was representative of the total cargo. An example of a large seven-hold vessel cargo represented by a 10 kg vessel composite sample is illustrated in Table 1. To calculate the individual target sample mass, use the following formula:

$$\text{target sample mass} = \frac{A}{B} \times C \tag{1}$$

where:

A = mass of individual lot in tons,
B = mass of total vessel in tons, and
C = mass of composite sample in grams.

5.5.3 Adjust the mass of the intermediate sample to within 2 % of the target mass contribution to the composite sample by removing material from the intermediate sample. Use appropriate means to preserve the particle size distribution of the original gross sample.

6. Keywords

6.1 calcined petroleum coke; sample division; sample preparation; sample reduction