



Standard Test Method for Volatile Matter in Green Petroleum Coke Quartz Crucible Procedure¹

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1. Scope

1.1 This test method covers the determination of the volatile matter produced by pyrolysis or evolved when petroleum coke is subjected to the specific conditions of the test method.

1.2 The interlaboratory study for precision covered materials with a volatile matter concentration ranging from about 8 to 16 %.

1.3 Samples having a thermal history above 600°C are excluded.

1.4 This test method is empirical and requires the entire test procedure to be closely followed to ensure that results from different laboratories will be comparable.

1.5 This test method is not satisfactory for determining de-dusting material content.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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 to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards*:²

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

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

3.1.2 *analysis time*—period test samples are placed in the furnace, as determined in Section 9 (5 to 10 min).

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

3.1.4 *green petroleum coke*—same as raw petroleum coke

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

3.1.6 *petroleum coke*—a solid, carbonaceous residue produced by thermal decomposition of heavy petroleum fractions or cracked stocks, or both.

3.1.7 *raw petroleum coke*—petroleum coke that has not been calcined.

3.1.8 *test sample*—the weighed portion of the analysis sample actually used in a test.

3.1.9 *volatile matter*—the mass loss on heating expressed as a percent of the moisture free sample used.

4. Summary of Test Method

4.1 Volatile matter of a moisture free petroleum coke is determined by measuring the mass loss of the coke when heated under the exact conditions of this test method.

5. Significance and Use

5.1 The volatile matter of petroleum coke affects the density of coke particles and can affect artifacts produced from further processing of the coke.

5.2 The volatile matter can be used in estimating the calorific value of coke.

6. Interferences

6.1 Moisture has a double effect. The mass loss is increased and the moisture free sample weight is decreased by the amount of moisture actually present in the test sample.

6.2 *Particle Size Effect:*

6.2.1 The particle size range of the analysis sample affects the volatile matter. The coarser the analysis sample, the lower the reported volatile matter will be. Crush analysis sample (see **Annex A1**) to pass a 0.250-mm opening (No. 60) sieve but do

not overcrush. A 0.125-mm opening (No. 120) sieve should retain 40 to 55 % of the sample. Do not obtain the analysis sample by scalping and discarding a portion of the sample.

6.2.2 Any segregation of particle sizes within the analysis sample shall be corrected by rebinding the sample just prior to weighing the test sample.

6.3 The furnace shall recover to $950 \pm 20^\circ\text{C}$ before the time limit is reached. If the furnace does not recover to the starting temperature within a maximum of 10 min, reduce the number of samples being analyzed until recovery is achieved within 10 min (see Section 9).

7. Apparatus

7.1 *Electric Muffle Furnace*—Calibrated (Test Method E220) and regulated to maintain a temperature of $950 \pm 20^\circ\text{C}$ (1742°F), as measured by a thermocouple mounted inside the furnace. Its heat capacity shall be such that the initial temperature is regained after the introduction of a cold rack of samples within 10 min.

7.2 *Quartz Crucibles*, self-sealing, 10 mL capacity with covers.

7.3 *Rack*, stainless steel, designed to hold up to 20 crucibles.

7.4 *Rifflers*, with hoppers and closures.

7.5 *Jaw Crusher and Roll Crusher*—Other style crushers that allow control over particle size without contamination are acceptable (see Annex A1).

7.6 *Balance*, capable of weighing to 0.1 mg.

7.7 *Dessicator*.

7.8 *Sieves*, meeting Specification E11.

7.9 *Timers*, stopwatch or second timer.

7.10 *Heat Sink*, 1/2 in. plate steel or other heat resistant material large enough to hold the crucible rack.

8. Precautions

8.1 Effusion of gaseous products, including soot and various hydrocarbons, and the increase of heat associated with the test can make the use of a hood desirable.

9. Furnace Calibration

9.1 Avoiding segregation of particles, transfer 1 g (± 0.05 g) of an analysis sample (see Annex A1) to each crucible in the rack, weigh each crucible with its contents, and cover to the nearest 0.1 mg. Place the cover on the crucible, and place in sample rack.

9.2 Record the furnace temperature at equilibrium ($950 \pm 20^\circ\text{C}$).

9.3 Place the full rack of samples in the furnace, and start the timer.

9.4 Monitor the furnace temperature. Record the elapsed time required to return to the equilibrium temperature recorded in 9.2. If the time exceeds 10 min, repeat the procedure, reducing the number of crucibles in the rack until temperature recovery is completed within 10 min of the introduction of the sample rack.

9.5 When temperature recovery is achieved within the 10 min period, record the number of crucibles in the rack. This number of crucibles shall be in the rack for all future runs (fill empty spaces in the stand with empty crucibles when necessary).

9.6 Round the recorded elapsed time to the next higher minute. This rounded time will be the *analysis time* for future runs (5 min minimum).

10. Procedure

10.1 Ignite the quartz crucible and cover at $950 \pm 20^\circ\text{C}$ for 5 min periods to constant mass (± 0.5 mg). Do not cover the crucible at this time to allow the carbon to burn off. Cool to ambient temperature in a desiccator before weighing the crucible and cover.

NOTE 1—Placing the empty crucibles in the inverted cover when igniting will reduce breakage and chipping.

10.2 Avoiding segregation of particles, transfer 1 g ± 10 mg of the analysis sample (see Annex A1) to the crucible, weigh the crucible with its contents, and cover to the nearest 0.1 mg. Place the cover on the crucible, and place in sample rack.

10.3 Set the timer for the *analysis time* (5 to 10 min). Place the rack in the furnace. Start the timer.

NOTE 2—Place the rack in the center of the furnace. Do not allow the rack to touch the walls of the furnace.

10.4 After the *analysis time* (± 5 s), remove the rack from the furnace and immediately place on a heat sink.

10.5 Weigh the crucible after it has cooled to room temperature and before it has been at room temperature for longer than 20 min.

NOTE 3—If cool down is not in a humidity controlled area, desiccation is required.

11. Calculation

11.1 Calculate the volatile matter of the moisture free prepared coke as follows:

$$\text{Volatile matter, mass \%} = ((A - B)/C) \times 100 \quad (1)$$

where:

A = weight of crucible and prepared sample prior to analysis, g,

B = weight of crucible and prepared sample after analysis, g, and

C = weight of prepared sample, g.

12. Report

12.1 Report to one decimal the average of duplicate determinations that agree within 0.5 %. When this agreement is not met, the values are considered suspect and another duplicate set shall be run. Report the average of all results agreeing within 0.5 %. If the second set also fails to agree within 0.5 %, report the average of all four values.

13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows:

13.2 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method, exceed the following values only one case in twenty.

$$\text{Repeatability} = 0.01905 \times (X + 2.826) \quad (2)$$

where: X is the average of two results in weight percent.

13.3 Reproducibility—The difference between two single and independent results obtained by different operators working in different laboratories on identical material will, in the long run, in normal and correct operation of the test method, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.06662 \times (X + 2.826) \quad (3)$$

where: X is the average of two results in weight percent.

13.4 Bias—Since there is no accepted reference material for determining the bias for this test method for measuring the volatile matter in green petroleum coke, no statement on bias is being made.

14. Keywords

14.1 analysis time; green petroleum coke; volatile matter

ANNEX

(Mandatory Information)

A1. PREPARATION OF SAMPLES

A1.1 Preparation of Bulk Sample

A1.1.1 The bulk sample is prepared by reducing the gross sample in such a manner as to obtain a representative bulk sample.

A1.1.2 The bulk sample can vary in weight from 22.7 to 90.6 kg (50 to 200 lb) and will vary in top particle size from 6 to 75 mm (¼ to 3 in.), depending upon the particle size of the gross sample.

A1.1.3 Air dry the bulk sample, if needed, so that it can be properly ground and fed through riffles. Air dry using drying pans 38-mm (1½-in.) deep made of noncorroding metal and of sufficient size that the sample may be spread to a depth of approximately 25 mm (1.0 in.). The maximum depth of the sample shall be no more than twice the diameter of the largest particles. The room shall be free of dust and excessive air currents. Avoid excessive drying time.

A1.1.4 An air drying oven can be used to pass slightly heated air over the sample. The oven is to be capable of maintaining a temperature of 30 to 40°C. Air changes should be at a rate of 1 to 4 min. Avoid over-heating and excessive drying.

A1.1.5 A drying floor can be used to dry bulk samples. This is a smooth clean floor in a room free from dust and excessive air current.

A1.1.6 Any modification of the above that will not overheat or contaminate the bulk sample is permitted.

A1.1.7 After the bulk samples have been air dried, grind the entire sample to pass a 6.7 mm sieve, using a jaw crusher. Do not overgrind. Keep a maximum amount of sample as coarse as possible.

A1.2 Preparation of Analysis Sample

A1.2.1 Using a riffle, split the bulk sample to obtain a 700-g portion.

A1.2.2 Pass the coke through the riffle from a feed scoop, feed bucket, or riffle pan length or opening equal to the full length of the riffle. When using any of the above containers to feed the riffle, spread the coke evenly in the container, raise the container, and hold it with its front edge resting on the top of the feed trough; then slowly tilt it so that the coke flows in

uniform streams through the hopper straight down the chutes into the pans. Under no circumstances shovel the coke into the riffle or dribble the coke into the riffle from a small mouth container. Make sure the riffle has a chute opening of at least 2 to 2½ times greater than the top size particle of the coke being riffled.

A1.2.3 Grind the sample to pass a 3.35-mm (No. 6) sieve using a roll crusher or other suitable crushing device that yields the result specified in **A1.2.8**. Do not overgrind (see **A1.2.8**).

A1.2.4 Using a 3.35-mm sieve (No. 6), separate the coarse particles to be crushed. This is called scalping.

A1.2.5 Set the rolls far enough apart to crush only the largest particles of the sample. Turn the roll crusher on, and pour the coarse portion of the sample very slowly through the rolls. Once more scalp off the coarse particles that are to be ground through the roll crusher again. Close the rolls not more than 1.5 mm (⅛ in.) for each pass of the scalped sample. Repeat the crushing and scalping of the sample until all of the sample has passed through the 3.35-mm sieve. Make sure all particles are well blended and none of the sample has been discarded.

A1.2.6 Riffle the sample to obtain two 350-g portions. Do not discard any of the sample.

A1.2.7 Using a procedure analogous to **A1.2.3**, grind one of the 350-g samples in the roll crushers to pass a 0.250-mm sieve (No. 60). Do not overgrind.

A1.2.8 About one half of the sample at this point shall be retained on a 0.125-mm sieve (No. 120). If this condition is not met, go back to **A1.2.5**, and grind the second 350-g portion. Adjust the roll crusher in smaller increments to increase the amount of sample retained on the 0.125-mm sieve. Likewise, use large increments to decrease the amount of sample on the 0.125-mm sieve.

A1.2.9 Recombine the 350-g sample, and dry it to constant weight at 95 to 105°C. This dried sample is now ready for analysis and can be stored in a closed container within a dessicator. Samples shall be redried on the fourth day. Excessive heating can result in higher volatile matter values because of oxidation of some cokes.

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