



Designation: D 6374 – 99 (Reapproved 2004)

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 the 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

E 11 Specification for Wire-Cloth Sieves for Testing Purposes

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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 Materials.

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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.

3.1.2 *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 sample 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 is 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 reblending 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 E 220) 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 E 11.

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 $1 \text{ g} \pm 10 \text{ mg}$ 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.