



Designation: D4931 – 06 (Reapproved 2017)

## Standard Test Method for Gross Moisture in Green Petroleum Coke<sup>1</sup>

This standard is issued under the fixed designation D4931; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers both the preparation procedure for samples containing free water (air drying loss (ADL) on gross moisture samples) and the determination of the gross moisture content of green petroleum coke.

1.2 The values stated in SI units are to be regarded as standard.

1.2.1 *Exception*—The values given in parentheses are for information only.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D2013 Practice for Preparing Coal Samples for Analysis  
D2234/D2234M Practice for Collection of a Gross Sample of Coal

D3302 Test Method for Total Moisture in Coal

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 *air drying, n*—a process of partial drying of a green petroleum coke sample to bring it to near equilibrium with the

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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<sup>2</sup> 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.

atmosphere in the room in which further reduction/division of the petroleum coke sample is to take place.

3.1.2 *air dry loss, ADL, n*—the loss in mass, expressed as a percentage, resulting from each air drying operation.

3.1.3 *free water, n*—visible unbound water in the sample.

3.1.4 *green petroleum coke, n*—same as raw petroleum coke.

3.1.5 *gross moisture, n*—that moisture determined as the loss in mass in an air atmosphere under rigidly controlled conditions of temperature, time, and air flow.

3.1.5.1 *Discussion*—Test Method D3302 prescribes the above controlled conditions.

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

3.1.7 *residual moisture, n*—that moisture remaining in the sample after air drying.

3.1.8 *total moisture, n*—synonym for *gross moisture*.

### 4. Summary of Test Method

4.1 This test method is based on the loss in mass of a green petroleum coke sample in an air atmosphere under controlled conditions of temperature, time, and air flow.

4.1.1 *Preparation Procedure* shall be used when the petroleum coke sample contains free water. The gross moisture sample is weighed and air dried to equilibrate it with the atmosphere. Determination of residual moisture is that determined using the *Drying Oven Method*. Air drying and residual moisture losses are combined to report gross moisture.

4.1.2 *Drying Oven Method* shall be used in routine commercial practice when the sample does not contain *free water*. The sample is crushed to at least minus 25 mm (1 in.) top sieve size and divided into analytical portions of at least 500 g each. Determination of total gross moisture is calculated by summing the results of the *Drying Oven Method* and the results of the *Preparation Procedure*.

### 5. Significance and Use

5.1 Moisture adds mass to the coke and serves no useful purpose. A knowledge of the moisture content is important in the purchase and sale of green petroleum coke, both of which are conducted on a dry basis.

5.2 The collection of the sample as specified for the *Drying Oven Method* is intended for the express purpose of determining the total moisture of green petroleum coke. The standard is available to producers, sellers, and consumers for determination when other techniques or modifications are not mutually agreed on.

5.3 The *Preparation Procedure* is used only when sample contains free water. Obtaining a representative sample of a coke source is compounded by the presence of free water.

**6. Apparatus**

**6.1 Ovens:**

6.1.1 *Air Drying Oven*—A device for passing slightly heated air over the sample. The oven shall be capable of maintaining a temperature of 10 °C to 15 °C (18 °F to 27 °F) above ambient with a maximum oven temperature of 40 °C (104 °F) unless ambient temperature is above 40 °C (104 °F), in which case ambient temperature shall be used. Air changes are to be at the rate of 1 to 4/min. A typical oven is shown in Fig. 1.

6.1.2 *Drying Oven* (for residual moisture on minus 25 mm (1.0 in.) samples sieve USA standard—forced-air type). It shall be capable of maintaining a temperature of 105 °C ± 5 °C and

so constructed that fresh air is introduced to all parts of the oven to ensure the removal of moisture-laden air. Air flow shall be maintained at sufficiently low velocity to prevent loss of fine particles, (for example, one exchange per minute).

**6.2 Drying Pans:**

6.2.1 Pans for the gross moisture sample (*Preparation Procedure*) shall be noncorroding, mass-stable at temperature used, and large enough so that the sample can be spread to a depth of not more than twice the diameter of the largest particles, or not more than 25 mm (1.0 in.) depth for smaller coke. The pan sides shall be 50 mm to 75 mm (2 in. to 3 in.) high.

6.2.2 Pans for crushed and divided sample shall be noncorroding, mass-stable at temperature used, and large enough so that the sample can be spread to depth of not more than 50 mm (2 in.). The sides shall be not more than 75 mm (3 in.) high.

**6.3 Balances:**

6.3.1 *Balance (Gross Sample)*—A balance with a minimum capacity of 10 kg and sensitive to 1 g.

6.3.2 *Balance (Crushed Sample)*—A balance with a minimum capacity of 4 or sufficient to weigh the pan, sample, and container and sensitive to 0.1 g.

6.4 *Laboratory Sample Containers*—Noncorroding cans with airtight, friction top or screw top sealed with a rubber gasket and pressure-sensitive tape for use in storage and transport of the laboratory sample. Glass containers, sealed with rubber gaskets, can be used, but care must be taken to avoid breakage in transport.

**6.5 Sieves**—Meeting Specification E11.

**7. Sampling**

7.1 The principles, terms, and procedures as set forth in Practice D2234/D2234M shall apply to the collection of the total moisture sample. Particular attention is directed to the section on Sampling Coal for Total Gross Moisture Determination.

7.2 Proceed with determination of moisture without unnecessary delay to minimize the loss (or gain) of moisture to air or the walls of the sample container. Visible condensation on the walls of the sample container indicates a moisture loss by the sample. Determine the moisture on the gross sample and include the sample container in the drying process adding the mass loss of the container to *G* (mass of sample) to determine the total moisture.

**8. Procedure**

8.1 *Preparation Procedure*—When the sample contains free moisture, a conditioning step is needed before determining gross moisture. If the sample does not contain free water, go to 8.2.

8.1.1 Distribute the required number of increments of the gross moisture sample in a series of preweighed pans. The sample is spread to a depth of not more than 50 mm (2 in.) or twice the diameter of the largest particle for cokes smaller than 25 mm (1.0 in.).

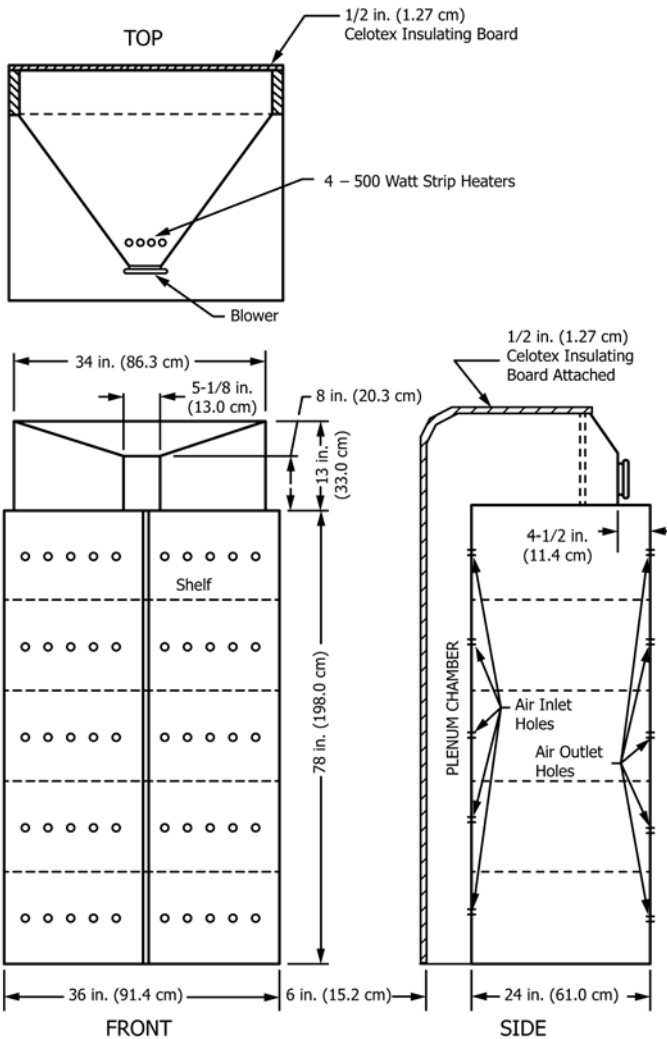


FIG. 1 Air Drying Oven