Designation: D4931 – 06

Standard Test Method for Gross Moisture in Green Petroleum Coke[1]

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 (ε) 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. 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 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 weight 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

[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.

***A Summary of Changes section appears at the end of this standard.**

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 to 15°C (18 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 ± 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).

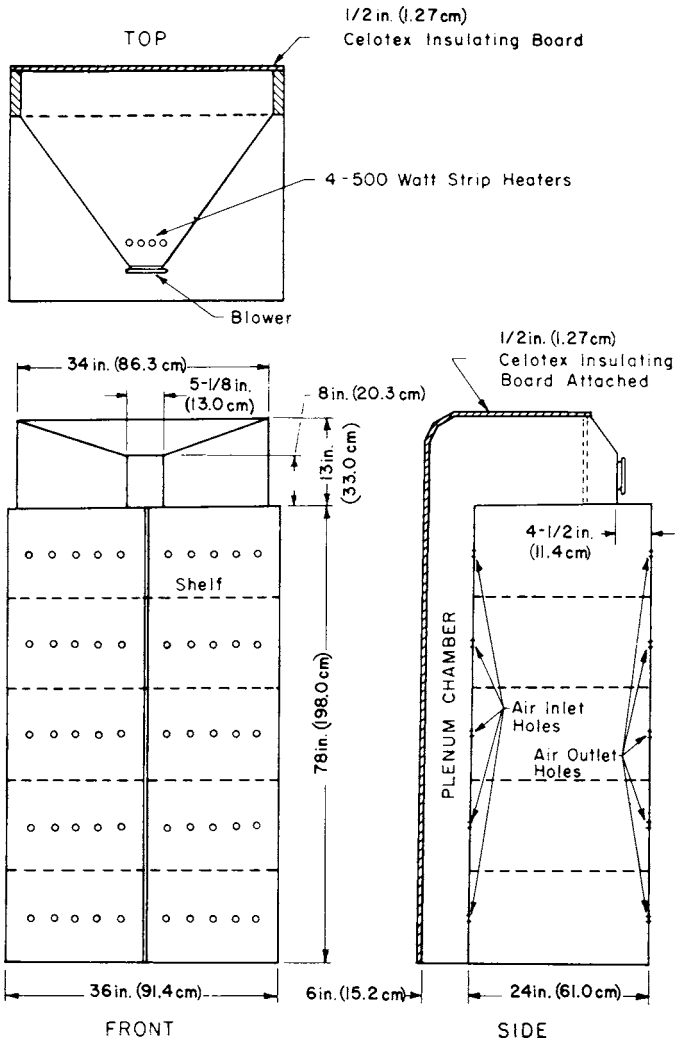

FIG. 1 Air Drying Oven

6.2 *Drying Pans*:

6.2.1 Pans for the gross moisture sample (*Preparation Procedure*) shall be noncorroding, weight-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 to 75 mm (2 to 3 in.) high.

6.2.2 Pans for crushed and divided sample shall be noncorroding, weight-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 weight 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.).

8.1.2 Weigh each pan with sample as it is filled from the gross sample and record the weights. Place each in an air drying oven that has been adjusted to maintain temperature no more than 15°C (27°F) above ambient to a maximum of 40°C (104°F). Maintain air circulation through oven at a rate of at least one air exchange per minute, but in no case is it to be so high as to blow fine particles from the pans. Gently stir the sample occasionally to ensure uniform drying.

8.1.3 When the coke surfaces appear dry, remove the sample pans from the oven, cool to ambient, weigh, and record mass to the nearest gram. Return the pans with sample to the oven and repeat the drying and weighing at two hour intervals until the weight loss is less than 0.1%/ h.

8.1.4 Allow the sample to reach equilibrium with ambient temperature and humidity before the final air dry weight is recorded. Avoid excess drying. Calculate the air dry moisture loss as shown in Step 9.1.1.

8.1.5 Proceed with sample reduction to at least 25 mm (1 in.) and division into minimum 500 g lots in accordance with the procedures listed in Practice D2013. Use enclosed equipment where possible to minimize moisture change.

8.1.6 The crushed and divided sample, 500 g minimum shall remain in an airtight container with minimum unused volume until starting the determination for Residual Moisture on Prepared Sample.

8.2 *Residual Moisture on Prepared Sample (Drying Oven Method)*:

8.2.1 Determine and record the mass of the drying pan and sample container. Transfer the crushed and weighed sample, including sample container and pan, to an oven maintained at 105 ± 5°C. Dry sample to a constant weight (see Note 1).

NOTE 1—Time required to achieve constant weight is estimated to be approximately 1 h per 100 g of sample. However, do not assume 5 h is sufficient to dry a 500 g sample to constant weight.

9. Calculation

9.1 *Total Moisture*:

9.1.1 Calculate air dry losses (ADL) as follows:

$$ADL = [(L2 - L1)/G] \times 100 \quad (1)$$

where:
ADL = air dry loss, mass %,
$L1$ = mass of sample after air drying,
$L2$ = mass of sample before air drying, and
G = mass of gross sample.

9.1.2 Calculate the percent residual moisture, R, as follows when the Air Drying Procedure is used:

$$R = [(L2 - L1)/G] \times 100 \quad (2)$$

where:
R = residual moisture,
$L1$ = mass of sample after drying,
$L2$ = mass of sample before drying, and
G = mass of sample.

9.1.3 Calculate the percent total moisture, M, as follows when the Air Drying Procedure has been used:

$$M = [R(100 - ADL)/100] + ADL \quad (3)$$

where:
M = total moisture, of sample mass %,
ADL = air dry loss, of sample mass %, and
R = residual moisture.

9.2 Calculate the percent total moisture, M, as follows when the air drying stage is not used:

$$M = [(WL2 - WL1)/G] \times 100 \quad (4)$$

where:
M = total moisture, mass %,
$WL1$ = mass of sample after drying,
$WL2$ = mass of sample before drying,
G = mass of sample.

10. Precision and Bias [3]

10.1 *Precision*—The precision of total moisture as determined in 9.1.3 by the statistical examination of the interlaboratory test results is as follows:

10.1.1 *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.

$$\text{Repeatability} = 0.34\% \quad (5)$$

10.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.58\ \% \quad (6)$$

NOTE 2—The values in the statements were determined in a cooperative program following D02-1007.[3]

10.2 The precision for ADL moisture as determined by Step 8.1 was not determined in this program. It is believed that there would be no difference in the precision.

10.3 The interlaboratory program included six duplicate petroleum coke samples with nine laboratories participating.

10.4 *Bias*—Since there is no accepted reference material for determining the bias for this test method for measuring the gross moisture in green petroleum coke, no statement on bias is being made.

11. Keywords

11.1 air drying loss (ADL); free water; green petroleum coke; residual moisture; total moisture

[3] Supporting data are available from ASTM International Headquarters. Request D02-1007.

SUMMARY OF CHANGES

Subcommittee D02.05 has identified the location of selected changes to this standard since the last issue (D4931–92(2002)) that may impact the use of this standard.

(*1*) Revised 3.1.4.

(*2*) Added 3.1.6.