Designation: D409/D409M – 09a$^{\varepsilon 1}$

Standard Test Method for Grindability of Coal by the Hardgrove-Machine Method[1]

This standard is issued under the fixed designation D409/D409M; 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.

This standard has been approved for use by agencies of the Department of Defense.

ε^1 NOTE—Footnote 2 was editorially corrected in June 2010.

INTRODUCTION

This test method is structured into the following parts:

The body of the test method discusses the scope, referenced documents, significance and use, apparatus, gross samples, preparation of test samples, procedure, calculation and report, precision and bias, and keywords.

Annex A1 contains the method to obtain and prepare coal feedstock for potential use as HGI RMs (reference materials).

Annex A2 contains the method to divide and containerize the candidate HGI RMs (candidate HGI RMs) from the feedstock prepared in Annex A1.

Annex A3 contains the method for homogeneity testing of the candidate HGI RMs containerized in Annex A2.

Annex A4 contains the method for determining the Hardgrove grindability index (HGI) to be assigned to each lot of the candidate HGI RMs.

Annex A5 contains the method used to calibrate a Hardgrove grindability machine using the primary or secondary HGI RMs.

Annex A6 contains the method for determining the moisture content of the 1.18 × 0.60 mm (No. 16 × 30) test sample.

1. Scope

1.1 This test method[2] covers the determination of the relative grindability or ease of pulverization of coals in comparison with coals chosen as standards. A prepared and sized sample receives a definite amount of grinding energy in a miniature pulverizer, and the size consist of the pulverized product is determined by sieving. The resultant size consist is used to produce an index relative to the ease of grinding [Hardgrove Grindability Index (HGI)].

1.1.1 Some coals, such as some high-volatile bituminous, subbituminous, and lignite coals, can undergo physical change as the natural or seam moisture is released during handling and preparation. This change is often sufficient to alter the grindability characteristics that will be reported when tested in the laboratory and could produce different indices dependent on the conditions of drying and the moisture level of the 1.18 × 0.60 mm (No. 16 × 30) (see Test Method D4749) materials used for the test. Therefore, the repeatability and reproducibility cited in this test method may not apply for these high-volatile bituminous, subbituminous, and lignite coals.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of D05.07 on Physical Characteristics of Coal.

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[2] For information concerning the experimental work on which this test method is based, see paper by Hardgrove, R. M., "Grindability of Coal," *Transactions*, American Society of Mechanical Engineers, Vol 54, F.S.P., p. 37, 1932.

D05.07 on Physical Characteristics on Coal is conducting ongoing investigations in regard to quality control during preparation, distribution, and use of standard reference materials and during preparation and testing of actual samples.

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:*[3]

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
D4749 Test Method for Performing the Sieve Analysis of Coal and Designating Coal Size
D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
D6883 Practice for Manual Sampling of Stationary Coal from Railroad Cars, Barges, Trucks, or Stockpiles
D7430 Practice for Mechanical Sampling of Coal
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Significance and Use

3.1 This test method develops a measurement of grinding or pulverizing characteristics that can be used to evaluate the yield, or energy input, or both, required in a grinding or pulverizing process, which can impact a wide variety of processes, including combustion, coke-making, liquefaction, and gasification.

4. Apparatus

4.1 *Air-Drying Oven [for air-drying the 1000 g, 4.75 mm (No. 4) sample]*—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 room temperature with a maximum oven temperature of 40 °C [104 °F] unless ambient temperature is above 40 °C, in which case ambient temperature shall be used. In the case of easily oxidized coals, the temperature shall not be over 10 °C [18 °F] above room temperature unless ambient temperature is above 37 °C [100 °F] in which case ambient temperature shall be used. Air changes shall be at the rate of 1 to 4 air volumes of oven capacity per minute.

4.2 *Drying Pans [for air-drying the 1000 g, 4.75 mm (No. 4) sample]*—Drying pans of sufficient size so that the sample may be spread to a depth of not more than 25 mm [1.0 in.] with sides not more than 38 mm [1.5 in.] high. The pans shall be nonreactive with coal and unaffected by the method of air drying selected.

4.3 *Balance [for weighing the 1000-g sample before and after air-drying and the sieve fractions formed during stage-crushing]*—With a sensitivity of at least 0.5 g in 1000 g and sufficient capacity to weigh a sample with a mass of greater than or equal to 1000g along with the drying pan.

4.4 *Balance [for weighing the 50-g test sample and the sieve fractions formed during milling]*—With a sensitivity of at least 10 mg and a minimum capacity of 800 g.

NOTE 1—A single balance meeting the specifications of 4.3 and 4.4 is permitted.

4.5 *Calibration Weights*—These calibration weights shall be used periodically to monitor the response of each balance over the working range.

4.6 *Sample Divider*— An enclosed riffle divider with feed chute as described in Practice D2013 or a rotary sample divider, which has been proven to be free of significant bias, may be used.

4.7 *Standard Sieves*—A working set of circular, standard testing sieves, which are 203 mm [8 in.] in diameter and conform to Specification E11, are required in the following sizes, together with cover and catch pan (receiver):

TABLE 1 Standard Sieves for HGI Testing

E11 Specification	U.S.A. Standard Sieve Series Designation
16.0 mm	5/8 in.
4.75 mm	No. 4
2.36 mm	No. 8
1.18 mm	No. 16
600 µm	No. 30
75 µm	No. 200

4.7.1 These working sieves must be periodically inspected for wear or damage. Any excessively worn or damaged sieves (for example, sieves with holes, tears, cracks; etc.) must be replaced immediately, and the HGI calibration procedure must be repeated after the damaged sieves are replaced (see Annex A5). Since the HGI depends upon the sieve analysis and since the 75-µm (No. 200) sieve is fragile, it is recommended that at least one set of working sieves be obtained, identified, and used exclusively for HGI determinations and that only this exclusive set of sieves be used for determining HGI.

4.7.1.1 Excessive wear may be indicated by poor repeatability or by failure of quality control checks (A5.4.1.1) to agree reasonably with the initial calibration data.

4.7.2 Normal wear on sieves is compensated by the use of primary HGI standard reference samples (HGI RMs) and proper calibration of equipment; excessive wear (such as holes or tears in the sieve cloth, and so forth) is not compensated by HGI RMs. Because excessive wear is unacceptable, inspect sieves carefully before each test to ensure the absence of excessive wear. For the 75- and 600-µm (No. 200 and 30) sieves, use only a soft-bristled brush (such as short-bristled camel hair) for brushing and cleaning.

4.7.2.1 It is good practice to keep a separate primary 75-µm (No. 200) check sieve to be used only to check the 75-µm (No. 200) working sieves described in 4.7.1 whenever the working sieve becomes suspect. This check is done by running a comparative HGI test between the two 75-µm (No. 200) sieves. Use of the working sieve for HGIs should be discontinued if HGI difference between the sieves is greater than 1 HGI unit.

4.8 *Plate Mill Crusher*—A laboratory plate mill capable of reducing 4.75-mm (No. 4) sieve size coal particles with the

[3] 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.

production of a minimum of minus 0.60-mm (minus No. 30) sieve size material. The crusher plates shall be serrated and about 100 mm [4 in.] in diameter. The distance between the plates shall be adjustable, and the relative speed of rotation of the plates shall not exceed 200 r/min.

4.8.1 Evidence from HGI standard reference sample user-response forms (A5.4.4) indicates that the median value of % Yield (8.3) of the 4.75-mm (No. 4) material as 1.18 × 0.60 mm (No. 16 × 30) material, regardless of the HGI level, is about 55 %. Use this median value to gage the effectiveness/efficiency of the stage-crusher and of the stage-crushing process, while noting that, in some situations, the % Yield has been as low as 45%.

4.8.2 The most frequently used plate mill can be equipped with "fine" or "coarse" plates. One independent study[4] has found evidence of differences in percent recovery resulting from use of different types of plate. In order to obtain the maximum % Yield, only the coarse plate should be used in preparation of both HGI calibration standards and unknown samples.

4.9 *Mechanical Sieving Machine*—The mechanical sieving machine shall accept an assembly of vertically nested circular sieves of 203 mm [8 in.] in diameter, together with cover and receiver. The machine shall simulate the motions given testing sieves during hand sieving by imparting a horizontal oscillatory motion of approximately 28-mm [1.1-in.] amplitude at a rate of approximately 300 oscillations or cycles per minute (cpm). Simultaneously, the top of the oscillating assembly is struck at a rate of approximately 150 blows per minute by a mass of 1.9 kg [4.2 lb] moving through a vertical distance of approximately 28 mm [1.1 in.] under the influence of gravity.

4.9.1 Whenever a mechanical sieving machine other than the reference machine described above is used, the method may be shown to be equivalent by one of two methods *(1)* side x side equivalency testing of the alternative machine vs. the reference machine (with both calibration and test samples that cover the working range), using the equivalency testing statistical technique of D6708 or *(2)* acceptable performance in an appropriate proficiency test program that covers the working range.

4.10 *Grindability Machine*—The Hardgrove Grindability Machine such as is shown in Fig. 1 is required for this test. Essential tolerances and specifications are shown in Fig. 2. The grindability machine includes a stationary grinding bowl of polished cast iron, with a circular horizontal track that holds eight polished steel balls, each 25.40 ± 0.13 mm [1.000 ± 0.005 in.] in diameter. The balls are driven by an upper grinding ring which is rotated at 20 ± 1 r/min by means of the upper spindle and which, in turn, is driven by an electric motor through reduction gears or, in newer models, belts. Weights are added to the driving spindle so that the total vertical force on the balls as a result of the weights, shaft, top grinding ring, and gear is equal to 29.0 ± 0.2 kg [64 ± 0.5 lb]. The machine is equipped with a counter and automatic device, which can be properly adjusted for stopping the machine after 60 ± 0.25 revolutions.

4.10.1 It is good laboratory practice to have the dimensions and the tolerances components that are subject to wear verified on a periodic basis.

4.10.2 The position of the counter trip mechanism before and after the test must be used to determine the number of revolutions completed. With some machines, it may be necessary to position strategically the counter trip mechanism at the beginning of the test to accomplish the desired number of revolutions (that is, during a set-up period, watch where the trip mechanism is initially, count the number of revolutions from the point of origin, determine where the trip mechanism is at the end of the test, and determine if the specified 60 ± 0.25 revolutions are obtained. If not, adjust the position of the trip mechanism at the beginning of the test until the specified number of revolutions are obtained). Periodically, verify that the machine is operated to obtain the specified number of revolutions per test.

5. Gross Samples

5.1 Collect a gross sample of coal, representative of the material from which it is taken. The sample may be collected, in accordance with Practice D2234/D2234M, D6883, or D7430 (or equivalent other international or national standard), and prepared in accordance with Practice D2013 (or equivalent other international or national standard) except that the sample topsize shall not be reduced beyond the 4.75-mm (No. 4) sieve and the sample shall have a mass of at least 1000 g.

5.2 If the topsize of the final sample product produced from mechanical sampling system is smaller than 4.75mm (No. 4), the sample does not meet the requirements of this test procedure.

6. Preparation of Test Samples

6.1 Prepare a 1.18 × 0.60 mm (No. 16 × 30) test sample for establishing the HGI of HGI RMs (Annex A4), for calibration (Annex A5), or for routine determination of HGI.

6.2 When necessary, divide the quantity of 4.75-mm (No. 4) sieve size coal to not less than 1000-g lots using an enclosed riffle or rotary sample divider, and air dry each sample in conformance with Test Method D3302. To calculate the % Yield (8.3), record the mass of the air-dried sample (m_1).

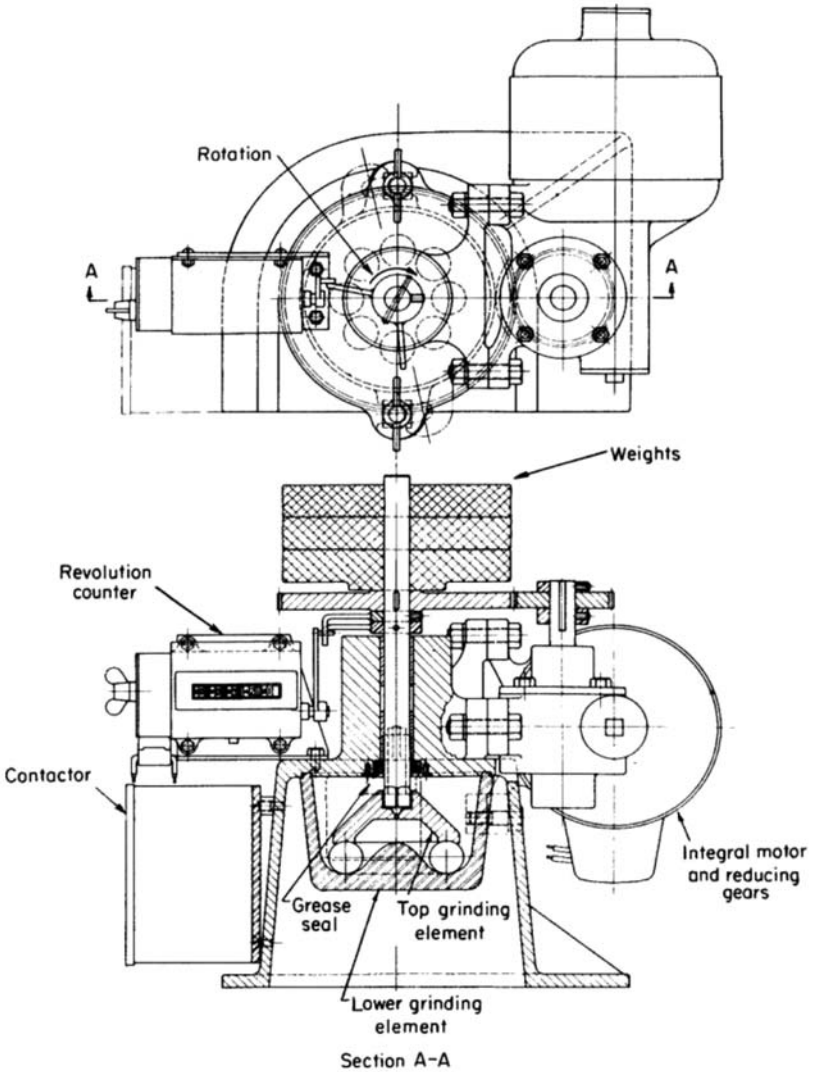
6.3 Sieve the entire amount (1000 g) of air-dried 4.75-mm (No. 4) sieve size sample in lots of no greater than 250 g for 2 min ± 10 s in the mechanical sieving machine. Use a set of nested sieves consisting of a 1.18-mm (No. 16) sieve on top of a 0.60-mm (No. 30) sieve. Weigh and record the mass of each sieve size.

6.3.1 Include a 4.75-mm (No. 4) and a 2.36-mm (No. 8) sieve in this nest of sieves to ease separation, determine and record the "natural" particle size distribution, and to verify that the topsize of the sample is appropriate for this test. Sieving more than 250 g at a time on the 200-mm [8-in.] diameter sieve may not allow each particle to "see" the sieve surface and have an opportunity to pass or to be retained by the sieve.

6.4 Combine and stage crush all of the sieve fractions greater than the 1.18-mm (No. 16) sieve with the crusher adjusted so that only the largest particles are crushed. Sieve the crushed material in portions of no greater than 250 g for 2 min ± 10 s in the mechanical sieving machine. Return the oversize

[4] D05.07 Minutes, J. Gardner, Georgia Power and G. Linton, October 1990.

FIG. 1 Hardgrove Grindability Machine



Item	Mass, kg	Mass, lb
Lead	25.9	57.0
Shaft and Gear	20.0	4.5
Top Ring	1.1	2.5
Total	29.0 ± 0.2	64.0 ± 0.5

to the crusher, after setting the crusher so that only the largest particles are crushed. Continue the stage crushing and sieving procedure until all the material passes the 1.18-mm sieve. Retain the 1.18 × 0.60 mm (No. 16 × 30) material. After all stage-crushing is completed, to calculate the % Yield (8.3), record the mass of the 1.18 × 0.60 mm (No. 16 × 30) fraction (m_2).

6.5 Mix well all the 1.18 × 0.60 mm (No. 16 × 30) material accumulated from the stage crushing and sieving process and divide the quantity using an enclosed riffle or rotary sample divider to obtain approximately 120 ± 10 g.

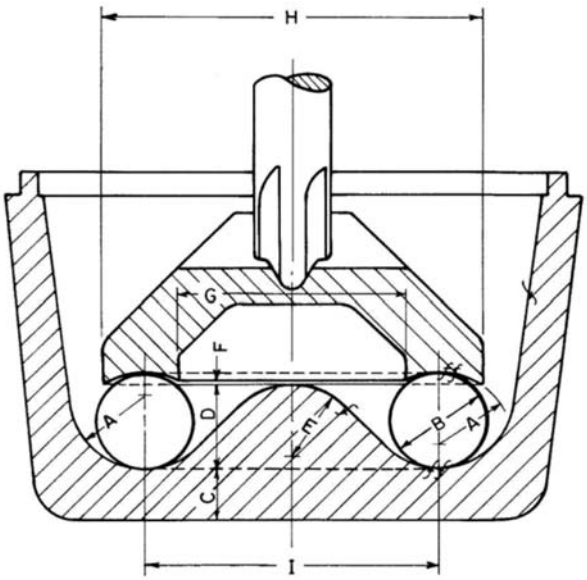
6.6 As the final step in preparation of the test sample, dedust the 120 ± 10-g sample from 6.5 by sieving on a 0.60 mm (No. 30) sieve for 5 min ± 10 s using the mechanical sieving machine.

6.6.1 Determining the mass of each sieve fraction after each stage-crushing step will allow the rate of reduction to be calculated and evaluated as a contributor to achieving adequate % Yield.

7. Procedure

7.1 Clean the grindability machine thoroughly, place the machine on a level surface, and space the balls as evenly as possible around the grinding bowl. Set the automatic stopping device so that the motion of the grindability machine will stop after 60 ± 0.25 revolutions of the upper grinding ring.

7.1.1 If the grindability machine makes a different number of revolutions other than the specified 60 ± 0.25 revolutions, the machine must be adjusted to within the tolerance specified.



A = 19.05 ± 0.13 mm [0.750 ± 0.0005 in.] radius
B = 25.40 ± 0.13 mm [1.000 ± 0.0005 in.] diameter
C = 12.70 mm [0.50 in.]
D = 22.23 mm [0.875 in.]
E = 19.05 mm [0.750 in.]
F = 1.42 mm [0.078 in.]
G = 60.33 mm [2.375 in.]
H = 98.43 mm [3.875 in.]
I = 76.20 mm [3.000 in.]
f = smooth machine surface
ff = fine machined surface and polished

FIG. 2 Grinding Elements of Hardgrove Machine

(This is normally done by strategically placing the counter tripper arm at the start of the test. See 4.10.2.)

7.2 Weigh 50 ± 0.01 g of the 1.18 × 0.60 mm (No. 16 × 30) sieve size dedusted material from 6.6 and distribute it evenly in the grinding bowl, brushing any material that falls on the elevated section of the lower grinding element into the lower grinding element, which contains the balls. Fasten the bowl in position and make sure the load is fully applied to the driving spindle.

7.3 Operate the machine for the specified 60.00 ± 0.25 revolutions.

7.4 Remove the bowl from the machine, lift out the upper grinding ring, and carefully brush adhering coal dust from the bowl and grinding ring onto a 16-mm [⅝-in.] sieve nested on a 75-µm (No. 200) sieve and a closely fitting receiving pan. Carefully empty the contents of the bowl onto the 16-mm [⅝-in.] sieve. Brush off material adhering to the balls and remove the balls from the 16-mm [⅝-in.] sieve. Brush material adhering to the 16-mm [⅝-in.] sieve, including the underside of the sieve, onto the 75-µm (No. 200) sieve. Replace the 16-mm [⅝-in.] sieve with a close-fitting cover and shake the nested 75-µm (No. 200) sieve; cover and pan for 10 min ± 10 s in a mechanical sieving machine. After the 10-min ± 10-s period, carefully brush coal dust from the underside of the 75-µm (No. 200) sieve into the receiving pan using a soft-bristled brush to avoid damaging the sieve. Repeat the shaking of the ball-milled material and the cleaning of the underside of the 75-µm (No. 200) sieve for two more periods each of 5-min ± 10-s duration.

7.4.1 Before emptying the contents of the lower grinding bowl onto the 16-mm [⅝-in.] sieve, the tops of the grinding balls may be brushed and then the balls may be removed from the lower grinding bowl by use of a magnetic extraction tool. Each grinding ball is brushed off and set aside; then, the pulverized coal is emptied from the bowl onto the 16-mm [⅝-in.] sieve.

7.5 Weigh separately to the nearest 0.01 g the coal retained on the 75-µm (No. 200) sieve (m_3) and the coal passing the 75-µm (No. 200) sieve (m_4).

7.5.1 Alternatively, predetermine the tare masses of the 75-µm (No. 200) sieve (m_5) and the catch pan (m_6). After the sieving of 7.4, weigh the 75-µm (No. 200) sieve and its contents (m_7). Subtract the tare mass to obtain the mass of the +75-µm (+No. 200) material = $m_3 = m_7 - m_5$. Weigh the catch pan and its contents (m_8). Subtract the tare mass to obtain the mass of the –75-µm (–No. 200) material = $m_4 = m_8 - m_6$.

7.6 If the sum of the +75-µm (+No. 200) (m_3) and the -75-µm (-No. 200) (m_4) sieve fraction masses differs by more than 0.50 g from the initial mass of 50 ± 0.01 g (m_9, see 7.2), reject the test and repeat. Use the calculated mass of the coal passing the 75-µm (No. 200) sieve (m_{10}), determined by subtracting the mass retained on the 75-µm (No. 200) sieve (m_3) from the test specimen mass (m_9), in determining the sum-of-least-squares fit (A5.4.3), in preparation of the calibration chart (A5.4.3.2) or in calculation of the grindability index of the sample (8.1) $m_{10} = m_9 - m_3$.

8. Calculation and Report

8.1 Calculate the grindability index corresponding to the calculated mass of test specimen passing the 75-µm (No. 200) sieve (7.5) directly from the equation of the line prepared in A5.4.3 and report to the nearest whole number.

8.2 For ease of comparison between or among laboratories, calculate and report the percent residual moisture in the 1.18 × 0.60 mm (No. 16 × 30) sample (Annex A6).

8.3 Calculate the % Yield of 1.18 × 0.60 mm (No. 16 × 30) material generated from the initial 1000 g of 4.75-mm (No. 4) top-size sample and compare the actual value to a typical median value (4.8.1) to gage the effectiveness/efficiency of the stage-crusher and of the stage-crushing process.

$$Y = \frac{m_2}{m_1} \times 100 \quad (1)$$

where:
Y = % Yield,
m_1 = mass of air dried sample (6.2), and
m_2 = mass of 1.18 × 0.60 mm (No. 16 × 30) material (6.4).

8.4 If other than primary HGI RMs are used for calibration of the test apparatus and method, report the source of the calibration standards used.

8.5 An example of a typical HGI preparation log sheet that has found application for record keeping during preparation of samples to be used for HGI determination is shown in Fig. 3.

Sample I.D.: _____ Date: _____ Analyst: _____

Sample Description: _____

AIR-DRYING

Air-dry pan mass, (a) :	_____ g	Ambient Temp :	_____ °C [°F]
Initial sample and air-dry pan mass, (b) :	_____ g	Time into oven :	_____
Final sample and air-dry pan mass, (c) :	_____ g	Time out of oven :	_____
Air-dried sample mass, c - a, (d) :	_____ g	Temperature :	_____ °C [°F]
Mass loss on drying, b - c, (e) :	_____ g	Heat on at :	_____
Initial sample mass, b - a, (f) :	_____ g	Heat off at :	_____
% Mass loss on drying, e/f x 100%, (g) :	_____ %	Fan on at :	_____
		Fan off at :	_____

INITIAL SIEVED MASSES

+4.75 mm (+No.4), (h) :	_____ g	1.18 x 0.600 mm (No.16 x 30), (k) :	_____ g
4.75 x 2.36 mm (No.4 x 8), (i) :	_____ g	-0.600 mm (-No.30), (l) :	_____ g
2.36 x 1.18 mm (No.8 x 16), (j) :	_____ g	Topsize :	_____
Total recovered mass after sieving, (h + i + j + k + l), (m) :	_____ g		
Mass loss on sieving, d - m, (n) :	_____ g		
% Mass loss on sieving, (n/d) x 100%, (o) :	_____ %		

REDUCTION OF +1.18 mm (+No. 16)

	Mass, +1.18 mm (+No. 16)		Mass, +1.18 mm (+No. 16)
Initially, h + i + j, (p) :	_____ g	>6th crushing of oversize, (v) :	_____ g
>1st crushing of oversize, (q) :	_____ g	>7th crushing of oversize, (w) :	_____ g
>2nd crushing of oversize, (r) :	_____ g	>8th crushing of oversize, (x) :	_____ g
>3rd crushing of oversize, (s) :	_____ g	>9th crushing of oversize, (y) :	_____ g
>4th crushing of oversize, (t) :	_____ g	>10th crushing of oversize, (z) :	_____ g
>5th crushing of oversize, (u) :	_____ g		
Final total mass of 1.18 x 0.600 mm (No.16 x 30), (A) :	_____ g		
Final total mass of -0.600 mm (-No.30), (B) :	_____ g		
Final total recovered mass, (A+B), (C) :	_____ g		
Loss on crushing, (m-C), D :	_____ g		
%loss on crushing, (D/d) x 100%, (E) :	_____ %		
%Yield of 1.18 x 0.600 mm (No.16 x 30), A/d x 100%, (F) :	_____ %		

DEDUSTING 120 g OF 1.18 x 0.60 mm (No.16 x No.30) and Milling

Mass of 1.18 x 0.60 mm (No.16 x 30) dedusted sample placed into HGI bowl, (G) :	_____ g	Number of revolutions, (I) :	_____
No. of increments collected, (H) :	_____	Seconds to mill, (J) :	_____ sec
Start position :	_____	RPM, [I/(J/60)] :	_____ RPM
Stop position :	_____		
Start time :	_____		
Stop time :	_____		

SIEVING OF MILLED PRODUCT

Tare mass of catch pan, (L) :	_____ g	Mass of -75 µm (-No.200), (N - L), (P) :	_____ g
Tare mass of 75 µm (No.200) sieve, (M) :	_____ g	Mass of +75 µm (+No.200), (O - M), (Q) :	_____ g
Mass of catch pan and -75 µm (-No.200), (N) :	_____ g	Total recover, (P + Q), (R) :	_____ g
Mass of 75µm (No.200) sieve and +75 µm (+No.200), (O) :	_____ g	Loss (≤0.50 g), (G - R), (S) :	_____ g
Calculated mass of -75 µm (-No.200), (G - Q), (T) :	_____ g		

ANALYTICAL RESULTS

HGI Formula (from Least Squares Regression) :	_____	
HGI (U) :	_____	
% moisture on 1.18 x 0.600 mm (No.16 x 30) material :	_____ %	*See the ANNEX, Method for Determining the Moisture Content of the 1.18x0.600mm (No.16x30) Test Sample*

FIG. 3 HGI Log Sheet is for Example Only and is Non-Mandatory

9. Precision and Bias

9.1 *Precision*:

9.1.1 The precision of this test method for the determination of Hardgrove Grindability Index of Coal, whenever primary HGI reference materials are used for calibration, is shown in Table 2.

TABLE 2 Repeatability and Reproducibility for HGI of Coal

Repeatability Limit r	Reproducibility Limit R
2	3

9.1.2 For the HGI method, the Repeatability Limit (r) in Table 2 is the value which the absolute difference between two

test results of separate and consecutive test determinations, carried out on dedusted 1.18 × 0.60 mm (No. 16 × 30) samples in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous 4.75 mm (No. 4) material, may be expected to occur with a probability of approximately 95 %.

9.1.3 For the HGI method, the Reproducibility Limit (R) in Table 2 is the value which the absolute difference between two test results, carried out in different laboratories on riffled splits of the 4.75 mm (No. 4) analysis sample, may be expected to occur with a probability of approximately 95 %.

9.1.3.1 The precision of this test method for the determination of Hardgrove Grindability Index of Coal, whenever national (secondary) HGI reference materials are used for calibration, must be determined and reported by each pertinent ISO national member body (NMB) and/or their national organization responsible for obtaining and preparing national (secondary) HGI RMs. The precision limits for this test method, whenever these national (secondary) RMs are used, will be as large or larger than the precision limits stated in Table 2.

9.2 *Bias*—Since this test method (using a calibration procedure) is an empirical standard, the degree of absolute bias cannot be determined.

10. Keywords

10.1 grindability; Hardgrove Grindability Index; HGI; pulverization

ANNEXES

(Mandatory Information)

A1. METHOD TO OBTAIN AND PREPARE HGI REFERENCE MATERIAL FEEDSTOCK (HGI RM Feedstock)

A1.1 Scope

A1.1.1 This method describes the procedures used to obtain and prepare Hardgrove Grindability Index (HGI) reference material feedstock (HGI RM feedstock).

A1.1.1.1 Authority to obtain and prepare HGI RM feedstock for use as candidate primary HGI RMs is given by ASTM Committee D05 on Coal and Coke, which maintains oversight responsibility for these activities.

A1.1.1.2 Authority to obtain and prepare HGI RM feedstock for use as candidate secondary HGI RMs is given by the pertinent ISO national member body (NMB), which maintains oversight responsibility for these activities.

A1.1.1.3 Authority is given on the basis of the producer's capability, experience, and expertise in these activities.

A1.2 Apparatus

A1.2.1 *Sieves; 4.75 mm (No. 4) and 2.36 mm (No. 8)*—Square mesh sieves meeting the requirements of Test Method D4749, used to verify that the candidate HGI RMs are 4.75-mm (No. 4) topsize.

A1.2.2 *Sieve Shaker*—Meeting the requirements of Test Method D4749, used to verify that the candidate HGI RMs are 4.75-mm (No. 4) topsize.

A1.2.2.1 No specifications are given for the sieve shaker used in the processing of the feedstock; as long as the final product meets the specification of being 4.75-mm (No. 4) topsize, there is no need to specify production equipment.

A1.2.3 *Crusher*—Commensurate with the requirement to maximize the 1.18 × 0.60-mm (No. 16 × 30) size fraction while maintaining a 4.75-mm (No. 4) topsize.

A1.3 HGI RM Feedstock Requirements

A1.3.1 Four coals shall be collected and used as the feedstock for the HGI RMs. One feedstock shall have an HGI of approximately 40 (typically between 35 and 45); a second feedstock shall have an HGI of approximately 60 (typically between 55 and 65); a third feedstock shall have an HGI of approximately 80 (typically between 75 and 85); and a fourth feedstock shall have an HGI of approximately 100 (typically between 90 and 110).

A1.3.2 Each HGI RM feedstock shall have a minimum mass of 80 kg [175 lb]. Where 80 kg [175 lb] is not adequate to produce the desired number of candidate RMs, an adequate mass to produce the desired number of candidate HGI RMs shall be obtained. Each candidate RM sample shall have a minimum mass of 1000 g.

A1.3.3 Any feedstock will be acceptable as long as the final production lot meets the homogeneity requirements found in Annex A3.

A1.3.4 To minimize the potential rejection of a production lot of HGI RMs after the lot has been processed, each feedstock coal should have the following characteristics: *(1)* be from a single seam, containing no blended materials, *(2)* be double-sieved to pass a 63-mm [2.5-in.] square-hole sieve while being retained on a 3.35-mm [⅛-in.] square-holed sieve, and *(3)* be mechanically cleaned, having been through a preparation or wash plant to remove non-coal material.

A1.3.5 Each feedstock coal shall be visually inspected upon its receipt to assure the absence of foreign material. If any foreign material (wood, rock, slate, steel, and so forth) is present, the entire shipment shall be rejected.

A1.3.6 Each feedstock coal shall be visually inspected upon its receipt to assure the absence of more than 5 % of undersized −3.35-mm [-⅛ in.] or of oversized +63- mm [+2.5 in.] particles. If undersized −3.35-mm [-⅛ in.] or oversized +63-mm [+2.5 in.] material constitutes greater than 5 % of the lot, after air-drying, the entire shipment shall be sieved to remove the undersize or oversize prior to further processing.

A1.4 Preparation of Each Candidate HGI RM

A1.4.1 Each feedstock is prepared according to the instructions in this section.

A1.4.2 Spread the feedstock coal (onto a smooth, flat, non-contaminating surface) to a thickness of no more than three times the particle topsize. A floor fan and periodic stirring can accelerate drying. Air dry the feedstock at room temperature for at least 12 h.

A1.4.2.1 If, after air drying, a feedstock contains greater than 5 % undersize (–3.35-mm) [-⅛ in.] or greater than 5 % oversize +63-mm [+2.5 in.], sieve the entire feedstock coal to remove the undersize or oversize.

A1.4.3 Reduce the air-dried feedstock to 4.75-mm (No. 4) top size. Use a crusher and a technique that will maximize the 1.18 × 0.60 mm (No. 16 × 30) size fraction while maintaining a 4.75-mm (No. 4) topsize.

A1.4.3.1 Verify that each reduced feedstock is a 4.75-mm (No. 4) topsize by obtaining a sample from each feedstock and conducting a size analysis [at 4.75 and 2.36 mm (No. 4 and 8)] according to the requirements of Test Method D4749. No more than 5 % of the sample may be retained on a 4.75-mm (No. 4) sieve and no less than 5 % of the sample may be retained on a 2.36-mm (No. 8) sieve (cumulative retained basis).

A1.4.4 Dedust each feedstock by removing and discarding the –0.30-mm (–No. 50) material.

A1.4.5 If the feedstock is to be transported or held for subsequent processing, package the material in a rigid, non-contaminating container. If necessary, line the container with double plastic liners to preserve the feedstock integrity and avoid excessive handling.

A2. METHOD TO DIVIDE AND CONTAINERIZE CANDIDATE HGI REFERENCE MATERIALS (Candidate HGI RMs)

A2.1 Scope

A2.1.1 This method describes the procedures used to divide and containerize candidate Hardgrove Grindability Index (HGI) reference materials (candidate HGI RMs).

A2.1.2 Authority to divide and containerize candidate primary HGI RMs for use in determining the HGI of coal (or for calibrating national HGI machines) is given by ASTM Committee D05 on Coal and Coke, which maintains oversight responsibility for these activities.

A2.1.3 Authority to divide and containerize candidate secondary HGI RMs for use in determining the HGI of coal is given by the pertinent ISO national member body (NMB), which maintains oversight responsibility for these activities.

A2.1.4 Authority is given on the basis of the producer's capability, experience, and expertise in these activities.

A2.1.5 Other methods for the division of the candidate HGI RMs (for example, riffle division or incremental division) may be used as long as the resulting candidate HGI RMs meet the homogeneity requirements of Annex A3.

A2.2 Apparatus

A2.2.1 *Blender/Mixer*—A rotating device is typically used to blend each feedstock before the feedstock is divided into candidate HGI RMs; typically, the blender is a double-cone or v-type blender or designed like a cement mixer. [Multiple mixing of the material through a riffle or rotary sample divider is acceptable, as long as the material passes the homogeneity test (Annex A3).]

A2.2.2 *Hopper/Feeder*—A device that holds the blended feedstock and allows the feedstock to be feed at a uniform rate into the containers, which are along the perimeter of the rotary sample divider. [A riffle or other type of mechanical divider that does not require a hopper / feeder may be used, as long as the material passes the homogeneity test (Annex A3)]

A2.2.3 *Rotary Sample Divider*—A rotating wheel upon which is fixed a level platform, which rotates at a constant speed and around which perimeter is located a series of uniformly spaced devices, which hold containers into which the candidate HGI RMs are evenly distributed. [A riffle or other type of mechanical divider may be used, as long as the material passes the homogeneity test (Annex A3)]

A2.3 Blending and Division of Each Feedstock

A2.3.1 Each feedstock is blended and divided according to the instructions in this section (A2.3).

A2.3.2 Thoroughly mix the feedstock for at least 30 min in a blender with a capacity to contain the entire feedstock at once.

A2.3.3 Divide the blended feedstock coal into candidate HGI RMs.

A2.3.3.1 Feed the feedstock at a uniform rate from the stationary hopper/feeder into containers located on the perimeter of a rotary sample divider (RSD), which is rotating at a steady rate. Assign container number "1" to the container that is located at a defined position on the RSD and number all remaining containers counterclockwise in consecutive order (that is, 1, 2, 3, …, x-1, x). Locate container "1" at such a position that, once the flow starts from the feeder into the containers, container "1" is at or near the location that receives the first increment (that is, start the RSD and, once the RSD is rotating at a steady rate, then start the feeder in such a manner that container "1" receives the first increment). Adjust the feed rate such that each container receives a maximum number of increments, but, in no case, shall less than 34 increments be collected per container.

A2.3.3.2 A stopwatch or timer should be used during the production of each feedstock lot to determine the time required to empty the hopper/feeder, which time, in conjunction with the known constant rpm of the RSD, can be used to calculate the number (and average mass of) increments going into each container.

A2.3.3.3 The number of containers shall be at least 24, but may be more, depending upon the configuration of the processing equipment.

A2.3.3.4 After all of the material has been fed into the containers, seal and label the containers according to the lot and the container number.

A2.3.3.5 Ideally, the containers will be labeled before being placed in order onto the RSD; alternatively, if assurance is such that the order of the containers remains secure after division, labels may be attached after division.

A2.3.3.6 Weigh and record the mass of each sealed container and the container number.

A2.3.3.7 From the masses of the packets determined in A2.3.3.6, the average mass per packet is calculated.

A2.3.3.8 The tare mass of a container may be determined individually or a representative number of the containers may be counted and weighed all at once and the average mass per container used to calculate the mass of HGI RM per packet.

A2.3.3.9 The ordered masses from A2.3.3.7 will be used to monitor the consistency of the process and to determine if the mass of material in each container is at least 1000 g.

A2.3.3.10 As an example of how these masses may be used, criteria such as the following is established by each authority manufacturing candidate HGI RMs: The average mass per packet is within ± 10 g of the target mass (that is, within the range of 1100 to 1120 g whenever the target mass is 1110 g per packet). The relative standard deviation of all of the packet masses is less than 2.50 %.

A3. METHOD FOR HOMOGENEITY TESTING OF CANDIDATE HGI REFERENCE MATERIALS (Candidate HGI RMs)

A3.1 Scope

A3.1.1 This method describes the procedures used to test the homogeneity of the candidate international Hardgrove Grindability Index (HGI) standard reference coal samples (candidate HGI RMs).

A3.1.2 Authority to test the homogeneity of the candidate primary HGI RMs that will subsequently be used in determining the HGI of coal (or for calibrating national HGI machines) is given by ASTM Committee D05 on Coal and Coke, which maintains oversight responsibility for these activities and for this standard method.

A3.1.3 Authority to test the homogeneity of the candidate secondary HGI RMs that will be subsequently used in determining the HGI of coal is given by the the pertinent ISO national member body (NMB), which maintains oversight responsibility for these activities.

A3.1.4 Authority is given on the basis of the producer's capability, experience, and expertise in these activities

A3.2 Other Documents

Pure Applied Chemistry, Vol. 78, No. 1, pp. 145–196, 2006; The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories; Prepared for publication by Michael Thompson, Stephen L. R. Ellison, and Roger Wood.; Appendix 1, Recommended Procedure for Testing a Material for Sufficient Homogeneity.

A3.3 Apparatus

A3.3.1 All of the apparatus and equipment shall be as prescribed in Section 4 and shall include the following:

A3.3.1.1 *Standard Sieves*—Set of circular, standard testing sieves to sieve the stage-crushed sample and the milled sample.

A3.3.1.2 *Crusher*—To reduce the 4.75 mm (No. 4) sample to 1.18 × 0.60 mm (No. 16 × 30) test sample.

A3.3.1.3 *Mechanical Sieving Machine*—To sieve the stage-crushed sample and the milled sample.

A3.3.1.4 *Balance*—For weighing the sieve fractions formed during stage-crushing.

A3.3.1.5 *Grindability Machine*—To test the samples for HGI.

A3.3.1.6 *Balance*—For weighing the 50-g test sample and the sieve fractions formed during milling.

A3.3.1.7 *Calibration Weights*.

A3.4 Determination of the Homogeneity of Each of the Candidate HGI RM Lots

A3.4.1 A lot of a particular candidate HGI RM is the total number of HGI RM containers produced from one process batch of a feedstock through the production process.

A3.4.2 Randomly select at least 10 % of the containers from each lot.

A3.4.2.1 In the special case of whenever the number of containers in the lot is 24, the homogeneity testing may be conducted at the same time as and as an intimate part of the process defined in Annex A4.

A3.4.2.2 In the special case of whenever a lot contains 24 two-kg containers, only 2 containers need to be randomly selected for homogeneity testing, but the material in each of the two containers is divided into two subsamples, producing four 4.75 mm (No. 4) topsize samples.

A3.4.3 Prepare the material in each selected container to 1.18 × 0.60 mm (No. 16 × 30).

A3.4.4 Test each prepared 1.18 × 0.60 mm (No. 16 × 30) test sample in duplicate.

A3.4.5 Conduct homogeneity testing as outlined in Pure Applied Chemistry, Vol. 78, No. 1, pp. 145–196, 2006, Appendix 1. The lot is acceptable for determination and assignation of HGI values, if this homogeneity test is passed.

A4. METHOD FOR DETERMINING THE HARDGROVE GRINDABILITY INDEX TO BE ASSIGNED TO EACH LOT OF THE CANDIDATE HGI REFERENCE MATERIALS (HGI RMs)

A4.1 Scope

A4.1.1 This method describes the procedures used to test the candidate Hardgrove Grindability Index (HGI) reference materials (candidate HGI RMs) to obtain the HGI value to be assigned to that lot.

A4.1.2 Authority to test the candidate primary HGI RMs for use in determining the HGI of coal (or for calibrating national HGI machines) is given by ASTM Committee D05 on Coal and Coke, which maintains oversight responsibility for these activities and for this standard method.

A4.1.3 Authority to test the candidate secondary HGI RMs for use in determining the HGI of coal is given by the pertinent ISO national member body (NMB), which maintains oversight responsibility for these activities.

A4.1.4 Authority is given on the basis of the producer's capability, experience, and expertise in these activities.

A4.2 Apparatus

A4.2.1 All apparatus and equipment is essentially as prescribed in Section 4 but with the following special precautions being taken:

A4.2.1.1 A mechanical sieving machine (4.9) and a grindability machine (4.10) are both to be reserved for use only for determining HGI of the candidate HGI RMs.

A4.2.1.2 At least four specially calibrated, stainless steel 75-µm (No. 200) square-mesh sieves shall be set aside for use only in determining the HGI of the HGI RMs [that is, in determining the amount of material passing the 75-µm (No. 200) sieve in 7.5]. One of the sieves (the primary "master" sieve) is used only to check the secondary sieve, as needed. The secondary sieve is used to check the two (or more) working sieves at least annually. The two (or more) working sieves are to be used only for determining HGI to be assigned to the the candidate HGI RMs.

A4.3 Determination of Grindability Index of Each Lot of HGI Reference Materials

A4.3.1 Randomly select four of the containers remaining after the process defined in Annex A3 has been completed and the lot has been deemed to be homogeneous.

A4.3.1.1 In the special case of whenever the number of containers in a lot is 24, randomly select and riffle-divide two containers from each given lot of candidate HGI RMs resulting from the process defined in Annex A2. Each of the four resulting samples is tested for HGI according to the method described in the body of this test method. (Two different technicians shall each test one portion obtained from each of the two selected samples.)

A4.3.2 Prepare a test sample from each of the four subsamples in accordance with Section 6.

A4.3.3 Process each of the four subsamples in the grindability machine in accordance with Section 7 and note the mass of the original sample (50 g) minus the mass of the material retained on the 75-µm (No. 200) sieve.

A4.3.4 *(Primary HGI RMs)*—To the nearest 0.1 HGI unit, determine the value of Hardgrove Grindability Index for each subsample from the equation of the line (least squares regression line) or from the standardization graph derived from the correlation of data between the standardization equipment reserved exclusively for HGI RM standardization purposes and the original equipment previously used by the Babcock & Wilcox Co. (the "Hardgrove" sieve) for standard samples.

A4.3.5 *(Secondary HGI RMs)*—To the nearest 0.1 HGI unit, determine the value of Hardgrove Grindability Index for each subsample from the equation of the line (least squares regression line) or from the standardization graph derived from the correlation of data between the standardization equipment reserved exclusively for HGI RM standardization purposes and the set of four primary HGI RMs obtained from the primary HGI RM producer.[5]

A4.3.6 Calculate the average and the standard deviation of the four values obtained in A4.3.4 or A4.3.5 and report the HGI to the nearest whole number and the standard deviation to the nearest 0.1 unit. Assign this average whole-number HGI value to the remaining HGI RMs in that lot.

A4.3.7 (*Primary HGI RMs)*—Report the average value and the standard deviation (as well as individual analyses, analyst identity, container identity, sieve identity, etc.) to the chairman of the ASTM D05.07 Producer Liaison Task Group.

A4.3.8 *(Secondary HGI RMs)*—Report the average value and the standard deviation (as well as individual analyses, analyst identity, container identity, sieve identity, etc.) to the chairman of the pertinent NMB oversight committee.

[5] The sole source of supply of the primary HGI reference materials (HGI RMs) known to the committee at this time are available from The Pennsylvania State University. Contact Brad Maben; Address: EMS Energy Institute, Pennsylvania State Univeristy, C-211 CUL, University Park, PA US 16802; Telephone: +1-814-865–3899 (back up Telephone: +1-814-863-8896); Fax: +1-814-863-7432; email: bam17@psu.edu; web-site: www.energy.psu.edu/HGI/index.html. ASTM Subcommittee D05.07 and the ASTM/DOE Liaison Task Group maintains oversight for production of only the HGI RMs. Secondary standard reference samples are available from other sources. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

A5. CALIBRATION OF HARDGROVE MACHINE (HGI Machine)

A5.1 Scope

A5.1.1 This method describes the procedure used to calibrate Hardgrove machines.

A5.1.2 Such Hardgrove machines can be used by pertinent ISO national member bodies (NMB) to assign values to lots of candidate secondary HGI RMs. (See Annex A4.)

A5.1.3 Such Hardgrove machines can be used by coal-analysis laboratories, which are then used for determining the HGI of coal.

A5.2 HGI Reference Materials (HGI RMs) for Calibration[5]

A5.2.1 Four HGI RMs prepared especially for this purpose (in accordance with Annex A1-Annex A4) and representing grindability indices of approximately 40, 60, 80, and 100 shall be used for calibration.

A5.2.2 Upon receipt, each of the HGI RMs should be examined for the following:

A5.2.2.1 Preparation date (no more than six months old).

A5.2.2.2 Oversize (material retained on a 4.75-mm (No.4 in.) sieve). {Less than 5 % +4.75 mm (No. 4.) and no particle on a 6.30-mm [¼-in. square-holed sieve]}.

A5.2.2.3 Foreign, noncoal material (no wood, rock, slate, and so forth).

A5.2.2.4 Excessive fines [more than 30% −0.60-mm (−No. 30) material].

A5.2.3 Whenever any of these parameters are unacceptable, contact the agency responsible for production of the HGI RMs and report the data on the response form accompanying the HGI RMs (see A5.4.4).

A5.3 Apparatus

A5.3.1 All apparatus and equipment is that described in Section 4.

A5.4 Calibration

A5.4.1 Calibrate each grindability machine, together with all ancillary equipment including sieves (4.7) and plate mill crusher (4.8), that will be used for this test when new, modified, repaired, suspected of being defective, or when operated by new personnel.

A5.4.1.1 HGI RMs shall not be used for calibration beyond 18 months from their preparation date. It is good practice to check the calibration of the HGI apparatus periodically with at least one of the HGI RMs (quality control check sample) even if none of the conditions in A5.4.1 apply. This will ensure that the system is still in control and it will allow for detection of problems previously unnoticed or not then occurring, and it will allow for use of the HGI RMs before their expiration date.

A5.4.2 For each calibration, process each of the four HGI RMs with indices of approximately 40, 60, 80, and 100 as described in Sections 6 and 7 and use the results to determine the equation of the line by use of the sum of least squares method (A5.4.3) and, if desired, to prepare a calibration chart (A5.4.3.1).

A5.4.3 By the method of the sum of least squares, determine the equation of the line that best fits the analytical results obtained from the four HGI RMs. An example HGI calculation from HGI RM data, shown in Table A5.1, illustrates this method.

A5.4.3.1 *Example—Sum of Least Squares Fit*—The equation of the line for the sum of least squares takes the form of:

$$Y = a + bX \tag{A5.1}$$

where:

Y = HGI,
a = y axis intercept,
b = slope of the regression line, and
X = calculated −75-µm (−No. 200) material.

(1) a and b can be determined by use of the following table and equations:

Y	X	X^2	XY
40	4.35	18.92	174.00
58	7.14	50.98	414.12
83	10.44	108.99	866.52
100	13.38	179.02	1338.00
$\Sigma Y = 281$	$\Sigma X = 35.31$	$\Sigma(X^2) = 357.91$	$\Sigma(XY) = 2792.64$

$$a = \frac{\Sigma Y \Sigma X^2 - \Sigma X \Sigma XY}{n\Sigma X^2 - (\Sigma X)^2} \tag{A5.2}$$

$$b = \frac{n\Sigma XY - \Sigma X \Sigma Y}{n\Sigma X^2 - (\Sigma X)^2} \tag{A5.3}$$

where:

ΣY = 281,
ΣX = 35.31,
$\Sigma(X^2)$ = 357.91,
$\Sigma(XY)$ = 2792.64,
$(\Sigma X)^2$ = 1246.80, and
n = 4.

(2) By doing these calculations, $a = 10.63$ and $b = 6.75$. Therefore, the equation of the line for this database is HGI = 10.63 + 6.75 (calculated −75-µm (−No. 200) material).

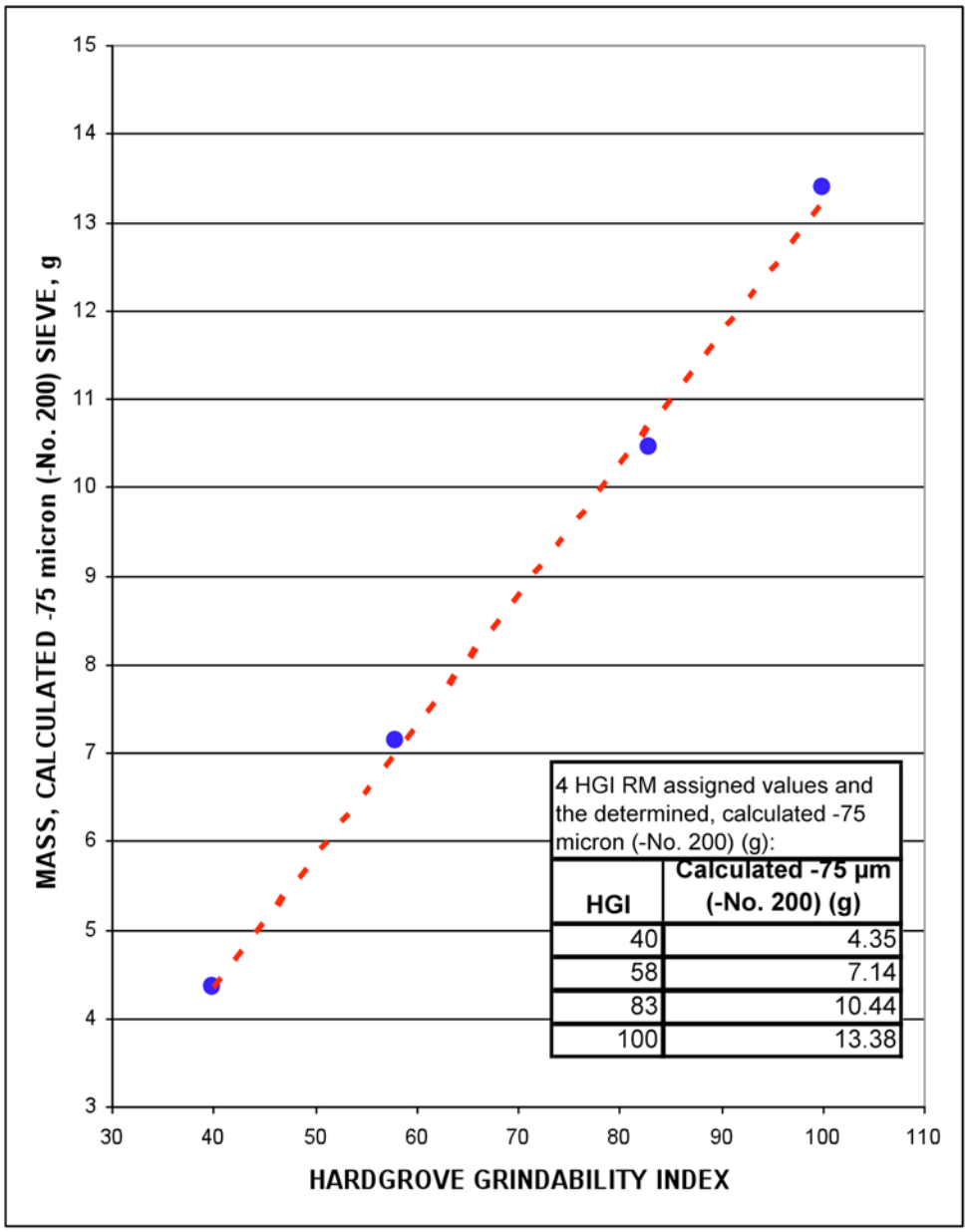
A5.4.3.2 Use results from HGI RMs processed in accordance with Sections 6 and 7 in determining the equation of the line by the sum of least squares method and (optionally) in preparing the calibration chart (see Fig. A5.1).

A5.4.4 *(Primary HGI RMs)*—A standard form for reporting data obtained during calibration is received with each set of HGI RMs received from the producer of the primary HGI

TABLE A5.1 Example HGI Data Calculation—Actual Mass of Materials, g

HGI[A]	mass, i[B]	+75 µm (+No. 200)	−75 µm (−No. 200)	Recovered	Lost	Calculated, −75 µm (−No. 200)
40	50.00	45.65	4.27	49.92	0.08	4.35
58	50.00	42.86	7.11	49.97	0.03	7.14
83	50.00	39.56	10.23	49.79	0.21	10.44
100	49.99	36.61	13.25	49.86	0.13	13.38

[A] Value issued with HGI RMs.
[B] Initial mass, g.



NOTE—Example: Use certified HGI RM and develop a similar calibration chart(s) for each HGI apparatus, or sieve sets, or both.

FIG. A5.1 Example Calibration Chart

RMs.[5] This form shall be completed and returned to the producer of the primary HGI RMs as noted on the form. A copy of this form is sent by the producer of the primary HGI RMs to the ASTM D05 Producer Liaison Task Group for use in monitoring the primary HGI RMs.

A5.5 Verification/Recalibration

A5.5.1 At a period of no longer than 36 months, the calibration of a national Hardgrove machine (with its calibration chart/equation) shall be verified against primary HGI reference materials (HGI RMs). Where the calibration has changed, the national Hardgrove machine will be recalibrated against the primary HGI RMs and a new calibration curve/equation of the line determined and used.

A6. METHOD FOR DETERMINING THE MOISTURE CONTENT OF THE 1.18 × 0.60 mm (No. 16 × 30) TEST SAMPLE (Moisture Content)

A6.1 Scope

A6.1.1 This method describes the procedures used to determine the moisture content of the 1.18 × 0.60-mm (No. 16 × 30) test sample.

A6.2 Apparatus

A6.2.1 *Drying Oven, Minimum Free Space [for moisture in the 1.18 × 0.60-mm (No. 16 × 30) sample]*—The oven shall be so constructed as to have a uniform temperature in all parts, have a minimum of air space, and be capable of temperature regulation between the limits of 104 and 110 °C [220 and 230 °F]. Provision shall be made for renewing the preheated air in the oven at a flow-rate of between approximately 2 to 4 times the oven volumes per minute.

A6.2.2 *Weighing Dish [for moisture in the 1.18 × 0.60-mm (No. 16 × 30) sample]* —Shallow, of glass, porcelain, fused silica, or of corrosion-resistant metal, with a well-fitting cover, of such a size that the coal layer does not exceed 0.20 g/cm^2. The dish should be as shallow as possible, consistent with convenient handling.

A6.2.3 *Desiccator, or similar cooling vessel*—Adequate for allowing cooling of the dried, 10-g, 1.18 × 0.60-mm (No. 16 × 30) sample after the 90-min heating period and before weighing back of the dried sample; the desiccator must be adequately designed and maintained as to prevent moisture reabsorption/readsorption during cooling.

A6.2.4 *Analytical Balance [for moisture in the 1.18 × 0.60-mm (No. 16 × 30) sample]*—With an approximate capacity of at least 100 g, capable of weighing to the nearest 0.1 mg.

A6.2.5 *Calibration Weights*—These calibration weights shall be used to monitor the response of each balance over the working range, as required by the laboratory's quality system.

A6.3 Reagents

A6.3.1 *Dry Air*—Air used to purge the drying oven should be dried to a moisture content of 1.9 mg/L or less (to a dew point of 10 °C or less, that is, 30 % relative humidity or less). Any desiccant or drying method capable of achieving this degree of dryness is suitable.

A6.3.2 *Desiccants*—Materials suitable for use in the desiccator may be chosen from the following:

A6.3.2.1 *Anhydrous Calcium Sulfate*, (0.004 mg/L).

A6.3.2.2 *Silica Gel*.

A6.3.2.3 *Magnesium Perchlorate*, (0.0005 mg/L).

A6.3.2.4 *Sulfuric Acid, Concentrated*, (0.003 mg/L).

NOTE A6.1—The desiccant must be kept fresh enough to assure that the air in the desiccator is dry to a moisture content of 1.9 mg/L or less. Values in parentheses ( ) are literature values for the residual amount of moisture in air at equilibrium with these desiccants.

NOTE A6.2—**Warning:** Sulfuric acid is corrosive and can cause severe damage to eyes, skin, and clothing. Magnesium perchlorate is a strong oxidant and can react violently with organic materials.

A6.4 Moisture in the 1.18 × 0.60-mm (No. 16 × 30) Test Sample

A6.4.1 Heat an empty weighing dish under the conditions at which the 10-g sample is to be dried, place the cover on the weighing dish, cool the cover and dish in the desiccator until the dish has cooled to ambient temperature, and weigh the dish and cover to ± 0.1 mg (m_{11}).

A6.4.2 Place the cover on the balance and the weighing dish on the cover. Tare the balance (so the balance reads 0.0000 g). With a spatula, collect increments from the unused 1.18 × 0.60-mm (No. 16 × No. 30) sieve size dedusted material until approximately 10 ± 0.1 g of the sample is in the weighing dish. Distribute the 10 g of sample evenly in the weighing dish. Weigh the dish, cover, and sample (the "assembly") at once to ± 0.1 mg (m_{12}).

A6.4.3 Place the assembly near the drying oven, remove the cover, and place the uncovered weighing dish and sample into the preheated drying oven 107 ± 3°C [225 ± 5°F]. Close the oven at once and heat for 90 min.

A6.4.4 After 90 minutes, open the drying oven, quickly remove and cover the weighing dish and dried sample, place the assembly into the desiccator, cool the assembly in the desiccator, and weigh the assembly to ± 0.1 mg (m_{13}) as soon as the assembly has cooled to ambient temperature.

A6.5 Calculation and Report

A6.5.1 For ease of comparison between or among laboratories, calculate and report the percent moisture in the 1.18×0.60 mm (No. 16 × 30) sample to the nearest 0.01%.

A6.5.2 Calculate percent moisture in the 1.18 × 0.60-mm (No. 16 × 30) material, *M*, as follows:

$$M = \left[\frac{m_{12} - (m_{13} - m_{11})}{m_{12}}\right] * 100 \qquad \text{(A6.1)}$$

where:

M = percent moisture in the 1.18 × 0.60-mm (No. 16 × 30) sample, %,

m_{11} = the mass of the empty weighing dish and cover, g,

m_{12} = the mass of the 1.18 × 0.60-mm (No. 16 × 30) sample used, g, and

m_{13} = the mass of the assembly after heating and cooling, g.