National Institute of Standards & Technology

Report of Investigation

Reference Material 8441a

Wheat Hardness

(kernel form)

This Reference Material (RM) is intended primarily for use in calibrating instruments used for the determination of hardness of single-kernel wheat. RM 8441a was prepared and analyzed by the Federal Grain Inspection Service (FGIS) program, Grain Inspection Packers and Stockyards Administration (GIPSA) of the United States Department of Agriculture (USDA). The USDA is the sole authority for all of the information provided in this report, including reference values and other technical information. A unit of RM 8441a consists of fifty pouches, five pouches each of five hard wheats and five soft wheats. Each pouch contains 20 g of material.

Reference Concentration Values: Reference values for hardness are provided in Table 1. Reference values are noncertified values that are the best estimate of the true values based on available data; however, the values do not meet NIST criteria for certification [1] and are provided with associated uncertainties that may reflect only measurement reproducibility and may not include all sources of uncertainty.

Expiration of Value Assignment: RM 8441a is valid, within the measurement uncertainty specified, until **03 January 2022**, provided the RM is handled and stored in accordance with the instructions given in this report (see "Instructions for Storage and Use"). This report is nullified if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM: The FGIS will monitor representative samples from each of the lots of wheat comprising this RM. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Technical aspects involved in the preparation and issuance of this RM were coordinated by K.E. Sharpless of the NIST Special Programs Office.

RM 8441a was characterized by the USDA FGIS. The cleaning, dividing, packaging, and characterization of each wheat sample were performed at the FGIS under the direction of A.C. Johnson.

Statistical consultation was provided by J. Yen of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this RM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR STORAGE AND USE

This RM, in unopened pouches, should be stored at 2 °C to 5 °C. Each pouch should be allowed to warm to room temperature (23 °C ± 2 °C) immediately before use.

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Report Revision History on Page 3

Steven J. Choquette, Acting Director
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WARNING TO USERS

RM 8441A IS INTENDED FOR RESEARCH USE; NOT FOR HUMAN OR ANIMAL CONSUMPTION.

PREPARATION AND ANALYSIS[(1)]

Sources and Preparation of Materials: A set of wheat samples was designated by FGIS in 1996 as wheat hardness reference samples (WHRS). This set of samples is made up of 10 separate lots of wheat (three pure varieties of hard red winter, two pure varieties of hard red spring, two pure varieties of soft red winter, and three pure varieties of soft white wheat) purchased from commercial sources. All lots were cleaned with a Carter-Day dockage tester and split into 15.9 kg (35 lb) sub lots using a Gamet rotator divider. The samples were stored in 19-liter (5 gallon) pails that were sealed with lids and stored at 2 °C to 5 °C. A sub lot of each of the WHRS was taken from storage and split into 20-gram portions using a Boerner divider and these sub samples were packaged in barrier-film aluminized pouches. Such sub lots were used to prepare RM 8441, which was issued in 1997, and RM 8441a, issued in 2004.

Single-Kernel Reference Values and Uncertainties: RM 8441 and RM 8441a were prepared as transfer standards for linking the measurements of Single-Kernel Characterization System (SKCS) 4100 instruments to the National FGIS wheat hardness reference scale. The single-kernel hardness values assigned to the WHRS are the grand averages of the hardness values that were found for these samples using 13 single-kernel characterization system instruments (SKCS 4100). The SKCS instruments were calibrated to give a mean hardness reading of 72.6 for the five hard WHRS and they were calibrated to give a mean hardness of 31.2 for the five soft WHRS. Each WHRS was measured in each of the 13 SKCS instruments in triplicate. The standard deviation of the 13 instruments' mean hardness values for a particular WHRS was taken as the uncertainty in the hardness values assigned to that WHRS (Table 1).

The hardness values reported in this document for RM 8441a are the hardness values that were assigned to the WHRS samples in 1996 (issued in 1997). To verify that the single-kernel hardness values of the WHRS had not changed in RM 8441a, single-kernel SKCS 4100 hardness measurements were made on ten sets of RM 8441a. The SKCS instrument was sloped and biased such that the mean of the hardness results for the hard RM 8441a was 72.6 and the mean of the hardness results for the soft RM 8441a was 31.2. Ten sets of three replicates of each of the 10 RM 8441a wheats were tested for hardness values.

The grand average of the means of the hardness values for the single-kernel SKCS 4100 hardness measurements for a particular RM 8441a material was calculated. The single-kernel hardness values found for RM 8441a were within the acceptance limits (i.e., the value found deviated from the assigned value by less than three times the uncertainty values listed in Table 1) of the corresponding hardness values that were assigned to those samples in 1996. Therefore, the single-kernel hardness values that are assigned to the RM 8441a material in this document (Table 1) are values that were originally assigned to these samples in 1996.

The wheat hardness values listed in Table 1 are the values that the user should obtain using an SKCS 4100 instrument that has been sloped and biased to RM 8441a, following the instructions in the SKCS 4100 Operation Manual [3], such that the mean of the hardness values measured for the five hard RM wheats (M_{H1} to M_{H5}) is 72.6 and the mean of the hardness scores measured for the five soft RM wheats (M_{S1} to M_{S5}) is 31.2. On a properly sloped and biased SKCS 4100 instrument, the hardness value measured for each wheat should be within three times the uncertainty values ($3 \times u_c$) listed in Table 1. If RM 8441a is used for standardizing instruments other than SKCS 4100 instruments, the hardness values measured may be different from the values listed in Table 1.

[(1)]Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for this purpose.

Table 1. Mean Single-Kernel Hardness Values for RM 8441a Wheats
(SKCS 4100 Instrument)

Wheat	Hardness Score	Uncertainty (u_c)
Hard-1 (M_{H1})	79.0	1.1
Hard-2 (M_{H2})	66.3	0.9
Hard-3 (M_{H3})	68.5	1.0
Hard-4 (M_{H4})	63.5	1.2
Hard-5 (M_{H5})	85.5	0.8
Soft-1 (M_{S1})	24.7	0.5
Soft-2 (M_{S2})	26.1	0.7
Soft-3 (M_{S3})	34.4	0.6
Soft-4 (M_{S4})	34.2	0.6
Soft-5 (M_{S5})	36.6	0.9

The computational procedure for SKCS 4100 standardization using RM 8441a is provided in the appendix of this Report of Investigation.

Comments and inquiries from users of RM 8441a will be welcome and should be directed to:

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REFERENCES

[1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G..; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136; U.S. Government Printing Office: Gaithersburg, MD (2000); available at http://www.nist.gov/srm/publications.cfm (accessed May 2016).

[2] AACC 39-70A; American Association of Cereal Chemists: Approved Methods AACC, 16th ed; St. Paul, MN (1995).

[3] Instruction Manual, *Single-Kernel Characterization System, Model SKCS 4100*; Perten Instruments, Inc.: 6444 South Sixth Street Road, Springfield, IL 62707.

Report Revision History: 05 May 2016 (Change of expiration date; editorial changes.); 24 March 2011 (Removed values for bulk hardness measurements; extension of the expiration date; minor editorial changes.); 19 May 2004 (Original report date.)

Users of this RM should ensure that the Report of Investigation in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

Computation Procedure for the Standardization of SKCS 4100 Instruments Using RM 8441a.

The process by which the mean of the hardness values measured for the five hard RM 8441a wheats is adjusted to the fixed value, H_B, and the mean of the hardness values measured for the five soft RM 8441a wheats is adjusted to the fixed value, S_B is called standardization. For single-kernel hardness measurements, H_B and S_B are fixed at 72.6 and 31.2, respectively.

Let M_{H1}, M_{H2}, M_{H3}, M_{H4}, and M_{H5} be the means of the hardness values for two or more hardness measurements performed on Hard 1, Hard 2, Hard 3, Hard 4, and Hard 5 RM wheats, respectively, and let M_{S1}, M_{S2}, M_{S3}, M_{S4}, and M_{S5} be the means of the hardness values for two or more hardness measurements performed on Soft 1, Soft 2, Soft 3, Soft 4, and Soft 5 RM wheats, respectively.

Then $M_H = (M_{H1} + M_{H2} + M_{H3} + M_{H4} + M_{H5}) \div 5$

and $M_S = (M_{S1} + M_{S2} + M_{S3} + M_{S4} + M_{S5}) \div 5.$

An SKCS 4100 instrument is standardized to RM 8441a if $M_H = H_B$ and $M_S = H_S$. If $M_H \neq H_B$ and/or $M_S \neq H_S$, then the hardness values measured for the RM using this instrument are sloped and biased such that $M_H = H_B$ and $M_S = H_S$.

The following relationship holds true for H_B, S_B, M_H, and M_S: $H_B = a + (b \times M_H)$ and $S_B = a + (b \times M_S)$, where, a and b are the bias and slope factors used to adjust the measured hardness values to the appropriate fixed values.

The slope factors can be computed as follows: $b = (H_B - S_B) \div (M_H - M_S)$.

The bias factor can be computed from: $a = H_B - (b \times M_H)$ or $a = S_B - (b \times M_S)$.

Therefore, the standard hardness value (SHV) for each wheat can be determined using the following relationship: SHV = a + (b × old HV) where old HV is the hardness measured for RM 8441a.

Example: Suppose that the SKCS 4100 yielded the hardness values listed in Table A1 for RM 8441a.

Table A1. Example Hardness Values Measured for RM 8441a Using the SKCS 4100

	Replicate			
Sample	R1	R2	R3	Mean
Hard-1 (M_{H1})	76.5	76.4	76.2	76.4
Hard-2 (M_{H2})	65.0	63.9	63.5	64.1
Hard-3 (M_{H3})	64.4	64.7	67.1	65.4
Hard-4 (M_{H4})	61.3	60.5	61.0	60.9
Hard-5 (M_{H5})	82.3	82.7	83.4	82.8
Soft-1 (M_{S1})	24.4	23.4	23.4	23.7
Soft-2 (M_{S2})	25.3	24.2	24.1	24.5
Soft-3 (M_{S3})	32.3	33.1	32.5	32.6
Soft-4 (M_{S4})	32.6	33.4	31.6	32.5
Soft-5 (M_{S5})	33.7	34.7	34.6	34.3

$M_H = (M_{H1} + M_{H2} + M_{H3} + M_{H4} + M_{H5}) \div 5$
$= (76.4 + 64.1 + 65.4 + 60.9 + 82.8) \div 5$
$= 69.9$

$M_S = (M_{S1} + M_{S2} + M_{S3} + M_{S4} + M_{S5}) \div 5$
$= (23.7 + 24.5 + 32.6 + 32.5 + 34.3) \div 5$
$= 29.5.$

For SKCS 4100, $H_B = 72.6$ and $S_B = 31.2$

$b = (H_B - S_B) \div (M_H - M_S)$
$= (72.6 - 31.2) \div (69.9 - 29.5)$
$= 41.4 \div 40.4$
$= 1.02475$

$a = S_B - (b \times M_S)$
$= 31.2 - (1.02475 \times 29.5)$
$= 1.0.$

SHV = a + (b × old HV)

so that

$M_H = a + (b \times \text{old HV}M_H)$
$= 1.0 + (1.02475 \times 69.9)$
$= 72.6$

and

$M_S = a + (b \times \text{old HV}M_S)$
$= 1.0 + (1.02475 \times 29.5)$
$= 31.2.$

Also,

SHV (Hard 1) = a + (b × old HV Hard 1)
= 1.0 + (1.02475 × 76.4)
= 79.2

and

SHV (Soft 1) = a + (b × old HV Soft 1)
= 1.0 + (1.02475 × 23.7)
= 25.2

and so on.