

## Report of Investigation

### Reference Material 8441

#### Wheat Hardness

United States Department of Agriculture Federal Grain Inspection Service

This Reference Material (RM) is intended primarily for use in calibrating instruments used for the determination of hardness of bulk or single kernel wheat. RM 8441 was prepared and analyzed by the Federal Grain Inspection Service (FGIS) program, Grain Inspection Packers and Stockyards Administration 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. One unit of RM 8441 consists of fifty pouches, five pouches each of five hard wheats and five soft wheats. Each pouch contains 20 g of material.

Sources and Preparation of Materials: Ten 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) were purchased from commercial sources. All lots were cleaned with a Carter-Day<sup>1</sup> dockage tester and split into 15.9 kg (35 lb) sublots using a Garnet<sup>1</sup> divider. A Boerner<sup>1</sup> divider was then used to further split these sublots into 20 g portions, which were then packaged in barrier film aluminized pouches.

**CAUTION:** This RM is for laboratory use only; IT IS NOT INTENDED FOR HUMAN OR ANIMAL CONSUMPTION.

Stability and Use: For the intended use of this RM the wheats, in unopened pouches and stored at 2 °C to 5 °C, are considered stable for five years from the date of shipment from NIST. Each pouch should be allowed to warm to room temperature (23 °C  $\pm$  2 °C) immediately before use. The FGIS will monitor representative samples from each of the lots of wheat comprising this RM and if any changes occur that invalidate this Report of Investigation, NIST will notify customers.

RM 8441 was characterized by the U.S. Department of Agriculture Federal Grain and Inspection Service (FGIS). The cleaning, dividing, and packaging of each wheat was done at the FGIS under the direction of A.C. Johnson.

The technical and support aspects involved in the preparation and issuance of this Reference Material were coordinated through the Standard Reference Materials Program by D.G. Friend and N.M. Trahey.

<sup>1</sup>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.

Gaithersburg, MD 20899

Certificate Issue Date: 5 December 1997

Thomas E. Gills, Chief Standard Reference Materials Program

RM 8441 Page 1 of 5

Bulk and Single Kernel Reference Values and Uncertainties: RM 8441 was prepared as a transfer standard for linking the measurements of Near-Infrared Reflectance (NIR) instruments and Single Kernel Characterization System (SKCS) 4100 instruments to the National FGIS wheat hardness reference scale. The bulk hardness values assigned to RM 8441 are the scores that were obtained using two reference NIR instruments that had been sloped and biased to the Standard Hardness Samples (SHS). Hardness scores were obtained from ten sets of measurements that were made on each RM 8441 sample over a period of five weeks (two sets of measurements per week). Each set of measurements consisted of determining the hardness values for five ground portions of a sample in the two NIR instruments. The bulk hardness score assigned to the RM 8441 unit is the grand average of one hundred hardness measurements made on that unit (Table 1). The uncertainty in measuring the bulk hardness values is estimated from the standard deviation among the averages of all measurements made in the measuring period. The mean hardness scores listed in Table 1, are the values that the user should obtain using an NIR instrument standardized to RM 8441 using AACC Method 39-70 [1], such that the mean hardness value measured for the five hard RM wheats ( $M_{\rm HI}$  to  $M_{\rm HS}$ ) is 76.7 and the mean of the hardness measured for the five soft RM wheats ( $M_{\rm SI}$  to  $M_{\rm SS}$ ) is 30.5. On a properly standardized NIR instrument, using AACC Method 39-70, the bulk hardness value measured for each wheat should be within three times the uncertainty values (3 x  $u_{\rm c}$ ) listed in Table 1.

Table 1. Mean Bulk NIR Hardness Scores for RM 8441 Wheats (NIR Instrument; AACC Method 39-70)

Wheat #	Hardness Score	Uncertainty $(u_c)$	
Hard-1 (M <sub>H1</sub> )	74.7	1.9	
Hard-2 (M <sub>H2</sub> )	75.8	1.9	
Hard-3 $(M_{H3})$	63.7	1.6	
Hard-4 (M <sub>H4</sub> )	77.5	2.1	
Hard-5 (M <sub>H5</sub> )	91.8	2.8	
Soft-1 (M <sub>S1</sub> )	30.0	0.7	
Soft-2 $(M_{S2})$	29.9	1.3	
Soft-3 (M <sub>S3</sub> )	31.1	0.9	
Soft-4 (M <sub>S4</sub> )	29.8	1.1	
Soft-5 (M <sub>S5</sub> )	31.5	1.5	

The wheat hardness scores listed in Table 2 are the values that the user should obtain using a SKCS 4100 instrument that has been sloped and biased to RM 8441, following the instructions in the SKCS 4100 Operation Manual [2], such that the mean of the hardness values measured for the five hard RM wheats ( $M_{\rm HI}$  to  $M_{\rm HS}$ ) is 72.6 and the mean of the hardness scores measured for the five soft RM wheats ( $M_{\rm SI}$  to  $M_{\rm SS}$ ) is 31.2. On a properly sloped and biased SKCS 4100 instrument, the hardness value measured for each RM should be within three times the uncertainty values ( $3 \times u_c$ ) listed in Table 2.

Table 2. Mean Single Kernel Hardness Scores for RM 8441 Wheats (SKCS 4100 Instrument)

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

RM 8441 Page 2 of 5

General Comments: The bulk and single kernel hardness methods give different values for the individual reference wheats and for  $M_H$  and  $M_S$ . This is because two very different techniques are used to determine wheat hardness. The NIR instruments are responding to differences in the particle size of the ground material while the SKCS instruments depend on crushing force, moisture content, mass, and size per kernel. The computational procedure for NIR and SKCS 4100 standardization using RM 8441 as provided in the appendix of this Report of Investigation.

If RM 8441 is used for standardizing other than NIR and SKCS 4100 instruments, the hardness values measured may be different from the values listed in Tables 1 and 2.

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

Dr. Aarion C. Johnson USDA-FGIS Technical Center 10383 N. Executive Hills Blvd. Kansas City, Missouri 64154

Telephone: (816) 891-0468 FAX: (816) 891-7314 E-mail: ajolmson@tsd.fgiskc.usda.gov

#### REFERENCES

- [1] Method 39-70A issued by the American Association of Cereal Chemists (AACC).
- [2] Instruction Manual, Single Kernel Characterization System, Model SKCS 4100, Perten Instruments, Inc., P.O. Box 7398, 5303 Louis Lane #1, Reno, NV 89511.

RM 8441 Page 3 of 5

### Appendix A

# Computation Procedure for the Standardization of NIR and SKCS 4100 Instruments Using RM 8441

The process by which the mean of the hardness values measured for the five hard RM 8441 wheats is adjusted to the fixed value,  $H_B$ , and the mean of the hardness values measured for the five soft RM 8441 wheats is adjusted to the fixed value,  $S_B$  is called standardization. For bulk hardness measurements,  $H_B$  and  $S_B$  are fixed at 76.7 and 30.05, respectively. 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 NIR or SKCS 4100 instrument is standardized to RM 8441 if  $M_H = H_B$  and  $M_S = H_S$ . If  $M_H \neq H_B$  and  $M_S \neq H_S$ , then the hardness values measured for the RM using these instruments 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 RM can be determined using the following relationship:  $SHV = a + (b \times Old\ HV)$  where old HV is the hardness measured for RM 8441.

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

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

		Replicate		
Sample	R1	R2	R3	Mean
Hard 1	76.5	76.4	76.2	76.4
Hard 2	65.0	63.9	63.5	64.1
Hard 3	64.4	64.7	67.1	65.4
Hard 4	61.3	60.5	61.0	60.9
Hard 5	82.3	82.7	83.4	82.8
Soft 1	24.4	23.4	23.4	23.7
Soft 2	25.3	24.2	24.1	24.5
Soft 3	32.3	33.1	32.5	32.6
Soft 4	32.6	33.4	31.6	32.5
Soft 5	33.7	34.7	34.6	34.3

RM 8441 Page 4 of 5

$$M_{H} = (M_{H1} + M_{H2} + M_{H3} + M_{H4} + M_{H5}) \div 5$$
  
= (76.4 + 64.1 + 65.1 + 60.9 + 82.8) \div 5  
= 69.9

$$M_S = (M_{S1} + M_{S2} + M_{S3} + M_{S4} + M_{S5}) \div 5$$
  
=  $(23.7 + 24.5 + 32.6 + 32.5 + 34.3) \div 5$   
=  $29.6$ 

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 - 29.6)
= 41.4 \div 40.3
= 1.02729

$$a = S_B - (b \times M_S)$$
  
= 31.2 - (1.02729 × 29.6)  
= 0.8

$$SHV = a + (b \times old HV)$$

so that,

$$SHVM_H = a + (b \times old HVM_H)$$
  
= 0.8 + (1.02729 × 69.9)  
= 72.6

and,

SHVM<sub>S</sub> = 
$$a + (b \times old HVM_S)$$
  
=  $0.8 + (1.02729 \times 29.6)$   
=  $31.2$ 

also,

SHV (Hard 1) = 
$$a + (b \times old HV Hard 1)$$
  
=  $0.8 + (1.02729 \times 76.4)$   
=  $79.3$ 

and,

SHV (Soft 1) = 
$$a + (b \times old HV Soft 1)$$
  
=  $0.8 + (1.02729 \times 23.7)$   
=  $25.1$ 

and so on.

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.