DETERMINATION OF FALLING NUMBER FOR WHEAT

Contents

1. PURPOSE

This directive provides procedures for determining falling number (FN) and certifying the results for wheat only. The service is provided upon request under the authority of the Agricultural Marketing Act of 1946, as amended (AMA). Testing services are available at selected Agricultural Marketing Service (AMS), Federal Grain Inspection Service (FGIS) field offices, official agencies, and the Technology and Science Division (TSD).

This directive also provides, upon request of the applicant, FN test results on a United States Grain Standards Act (USGSA) inspection certificate. This certification procedure is applicable only to FN tests that are performed in conjunction with other testing services performed under USGSA. FN testing performed independent of USGSA services must be reported on an AMA inspection certificate.

2. REPLACEMENT HIGHLIGHTS

This directive supersedes FGIS Program Directive 9180.38, Falling Number for Wheat, dated May 20, 2013. The following, significant changes were made:

- Adobe Acrobat is required to open the attachments in this document.
- Updated the format to be consistent with FGIS Administrative Directive 3010.2;
- Added a section for definitions;
- Revised the list of equipment and supplies to be more specific and reflect equipment currently supported by Perten Instruments;
- Added section for equipment quality control procedures;
- Required the use of specific digital barometer and thermometer models;
- Required barometer accuracy to be verified by TSD and the thermometers to have a National Institute of Standards and Technology (NIST) traceable calibration;
- Removed the requirement to calibrate the barometer using the nearest weather station information;

- Eliminated the use of mercury thermometers;
- Changed the tolerance for measured water temperature to account for measurement uncertainty;
- Required collecting ground wheat in a plastic bag, closing, and shaking to mix prior to taking a test portion;
- Eliminated requirement to run grinder for at least one minute after each sample and inserted requirement to clean the grinder chamber and cyclone after each sample;
- Required addition of the homogenized ground wheat to the viscometer tube prior to addition of water;
- Clarified the FN tube scraping procedure and added the explanation that failure to scrape causes a lower FN;
- Incorporated FGIS Policy Bulletin #264 – “Certification of Falling Number Based on Composite Samples”;
- Increased the time of sample preparation before placing the FN tube in the water bath from 40 to 60 seconds, bringing the time into agreement with that stated in the American Association of Cereal Chemists (AACC) International method 56-81.03, “Determination of Falling Number”;
- Incorporated three Microsoft Excel 2016 worksheets as attachments to perform all calculations previously requiring lookup tables;
- Removed the lookup tables associated with altitude-corrected FN values, converting between FN and liquefaction numbers, predicting percent moisture in ground wheat samples, and the boiling point of distilled water referenced to barometric pressure.
- Required the use of the Shakematic for shaking samples and eliminated the option of shaking samples by hand;
- Implemented a new procedure for adjustment of FN based on barometric pressure instead of altitude;

- Changed contact for inquiries to the Falling Number Program, Analytical Chemistry Branch;
- Inserted the option to use deionized water;
- Required the use of a bottle-top dispenser in place of a graduated cylinder or automatic pipette;
- The troubleshooting section was included as attachment 4.

3. BACKGROUND

The FN method is an indirect measurement of the amount of alpha-amylase activity in wheat. High alpha-amylase activity is associated with the natural germination process that results in the breakdown of starch and ultimately in sprouting of the wheat kernel. High alpha-amylase activity adversely affects the end-use quality of wheat, and as a result, assessment of this factor is important in the domestic and international trade of wheat.

The FN result is the time in seconds required for the instrument to mix the sample with a viscometer-stirrer, release the viscometer-stirrer, and allow it to fall through the hot aqueous mixture. A high FN indicates a high viscosity of the mixture and low alpha-amylase activity. A low FN indicates a low viscosity of the mixture and high alpha-amylase activity.

4. DEFINITIONS

a. Wheat – whole-kernel, wheat grain as defined in book II, chapter 13 of the FGIS Grain Inspection Handbook.

b. Gram (g) – a metric system unit of measurement for mass.

c. Milliliter (mL) – a metric system unit of measurement for volume.

d. Barometric Pressure (P) – atmospheric pressure as measured by a barometer. For the analytical method herein, the barometric pressure is the **absolute pressure** reported in inches of mercury (inHg).

5. EQUIPMENT AND SUPPLIES

This section specifies the equipment and supplies needed to perform the FN test. In certain cases, other supplies may be substituted with written approval from TSD.

a. Equipment and Supplies from Perten Instruments North America (Springfield, IL).

(1) Falling Number Instrument. Model No. FN 1000, FN 1500, FN 1700, or FN 1900 (FN 1400, FN 1600, and FN 1800 models are no longer supported by Perten Instruments and replacement with a supported model is recommended).

(2) Cooling Tower 1000. Fits all FN Models. Part No. 011050.

(3) Plastic tubing for cooling system. Part No. 30718.

(4) Spiral hose collector. Part No. 31037.

(5) Flow indicator. Part No. 106571.

(6) Hose clamps for cooling tubing. Part No. 30099.

(7) Bottle. For refilling water bath. Part No. 30690.

(8) Hose Kit for Water Bath Refill System. Part No. 31105.

(9) Viscometer Tubes. Certified for falling number test, Part No. 10.03.00.

(10) Viscometer Tube Rack. Part No. 100800.

(11) Plastic Funnel. Part No. 100600.

(12) Cassette Stand. Part No. 160810.

(13) Stand for Holding Cassettes and Tubes. Part No. 160440.

(14) Viscometer-stirrer. Part No. 160400.

(15) Shakematic 1095. Part No. 011090.

(16) Shakematic Tube Stopper. Part No. 109070.

(17) Mains Power Cable for Shakematic. Part No. 100119 (115 V).

(18) Spolett Tube Cleaner. Part No. 191100.

(19) Grinder. Laboratory Mill Model 3100, with 0.8 mm sieve. Part No. 031011 (115V, 60 Hz).

(20) Cyclone. Used with grinder for sample collection. Part No. 310500.

(21) Plastic Baton. Used to clean grinding chamber of LM 3100. Part No. 310430.

(22) Grinding Chamber Brush. Used to clean grinding chamber of LM3100. Part No. 311440.

(23) Cylcone Brush. Used to clean cyclone attachment. Part No. 310260.

b. Other Equipment and Supplies

(1) FGIS-approved divider.

(2) GAC2500-UGMA or Perten AM520 moisture meter. For additional information refer to the FGIS Moisture Handbook.

(3) Digital Barometer. Accuracy ± 0.12 inHg and Precision ± 0.03 inHg or better. Testo 511, Part No. 0560 5011 (Testo, Inc., Sparta, NJ) or Fisher Scientific Barometer, Part No. 14-650-118 (Fisher Scientific, Hampton, NH).

(4) Digital Thermometer with NIST-Traceable Calibration. Accuracy ± 0.4°C and Precision ± 0.1°C, Part No. 06-664-27 (Fisher Scientific).

(5) Balance. A precision-class balance accurate to within ± 0.02 g.

(6) Distilled or Deionized Water. Used for water bath and addition to sample test portion.

(7) Water Dispenser. Bottle-top dispenser, 2.5–25 mL, with discharge tube, telescoping filling tube, spare dispensing cartridge, 45/33mm and 45/38 mm thread polypropylene adapter, and operating manual, Accuracy ± 1.2% and Precision ± 0.2%, Part No. 13-688-247 (BrandTech, Essex, CT).

(8) 2L Bottle. For use with the bottle-top water dispenser with 45/33 mm or 45/38 mm threads (bottles of other volumes may be used). Use of an amber-colored bottle is preferred for inhibiting microbial growth.

(9) Weighing boats or metal weighing pan.

(10) Plastic bag. Any sealable plastic bag large enough to contain the ground sample being no more than 2/3 full and thick enough to resist tearing.

(11) Test tube brush. Used to clean viscometer tubes.

6. EQUIPMENT QUALITY CONTROL PROCEDURES

a. Falling Number Instrument.

CAUTION: Use only distilled or deionized water in the water bath.

CAUTION: Do not add chemicals to adjust the water bath temperature as this will result in erroneous results.

(1) Water Level. Periodically check the water bath level while running FN tests. Some instrument models have an overflow tube on the water bath. The water level should be at the overflow level when viscometer tubes are in place. Immediately after a FN test in which the water level was at the overflow outlet, mark the new water level on the water level indicator. Maintain the water height at the mark, so that when a set of FN tubes is inserted, the water will rise to the overflow outlet.

(2) Water Bath Temperature Check. Before the initial test of each day and after adding distilled water to the bath, verify that the bath temperature is correct for the barometric pressure in the lab.

(a) Measure the barometric pressure using the barometer and measure the temperature of the water bath by inserting the thermometer in the center of an opening to the bath to a depth of 3.5 inches. The probe may be lightly etched for referencing the distance.

(b) Enter the barometric pressure (*P*) and temperature measurements into the attachment 1. This worksheet is set

to calculate the difference of the measured temperature from the predicted temperature using equation 1.

Equation 1. Water Bath Check

$$Difference\ (\%) = Measured\ Temp. - (0.9586 \times P + 71.696)$$

(c) If the measured temperature differs from the predicted temperature by more than ± 0.5°C, correct the problem before proceeding. Refer to the attachment 4 for troubleshooting tips to help in resolving the problem.

b. Viscometer Tubes. Replace when worn and scratched.

c. Grinder. Replace the sieve (Part No. 310230) at least every 1,000 samples or sooner whenever it appears damaged.

d. Digital Barometer. The barometer must be set to read absolute pressure using **inHg**. New barometers must be sent to TSD for verification of accuracy and every 2 years thereafter.

e. Digital Thermometer. The accuracy of the thermometer must be verified to be within the specification at least once every 2 years. The thermometer may be purchased with a NIST-traceable calibration, and calibration services are commercially available.

f. Balance. Verify accuracy of balance daily using a 5-g reference weight. The mass must be 4.98–5.02 g to be considered acceptable.

g. Bottle-top Dispenser. Operate according to the manufacturer's manual. Check the accuracy of the water dispenser at the beginning of each day falling number testing is performed. Set the water dispenser to 25.0 mL and dispense the nominal amount of water into a tared container. The mass should be in the range of 25.0 ± 0.3 g based on the density of water being 0.998 g/mL at room temperature (20–23°C). If the mass is not in this range, calibrate the water dispenser per the manufacturer's instructions.

7. BASIS OF DETERMINATION

The FN test is based on a 7 g representative portion taken from 250 g of ground wheat. Sufficient wheat must be ground to yield 250 grams of flour. Remove dockage and stones from the sample before grinding. See section 10.b. for more details.

NOTE: All FN determinations are performed in duplicate and reported, on the official certificate, as the average of the two measurements.

FN results are reported on a 14 percent moisture basis unless the applicant specifies another moisture basis. The applicant may request to have the FN results reported on the "as is" moisture basis, dry matter (zero percent moisture) basis, or any other specified moisture basis. Only one moisture basis is reported on any one certificate.

There are three certification options available for export lots:

(1) Individual sublot samples.

(2) Average of sublots using the approved method (see section 12 for more details).

(3) Composite sample representing the entire lot (see section 13 for more details).

NOTE: Applicants must specify the certification option to use prior to loading.

8. SAMPLING

Refer to the FGIS Sampling Handbook for official sampling procedures. Use an FGIS-approved divider for obtaining the amount required for the FN test.

9. PREPARATION OF GROUND WHEAT

a. Moisture Determination.

(1) Determine the moisture content of the wheat using a GAC2500-UGMA or Perten AM 5200-A moisture meter. Refer to the Moisture Handbook for more details.

(2) If the moisture is above 15.9 percent, air dry the sample, using an open pan at room temperature, to a moisture content of 15.9 percent or less before grinding.

(a) Air drying to 15.9 percent moisture is not required for “as is” certification.

(b) Air dry enough so that the weight after drying is at least 250 g.

b. Grinding and Homogenization.

(1) Remove dockage and stones from the wheat before grinding.

(2) Grind a minimum of 250 g of wheat using a Perten Lab Mill 3100.

(3) Collect the sample in a plastic bag. Do not reuse the plastic bag.

(4) Close the bag and shake the sample for at least 10 seconds to homogenize the ground wheat prior to taking a test portion.

(5) Clean the grinding chamber and cyclone between samples using a brush and vacuum.

10. FALLING NUMBER DETERMINATION

CAUTION: Steps a, b, and c, must be completed and the FN instrumental analysis (step d) started within 60 seconds after adding the water. Perform all FN determinations in duplicate as follows:

a. Weighing Test Portions and Addition of Water.

(1) Weigh 7.00 ± 0.05 g of the homogenized ground wheat into a clean, dry weighing boat or metal weighing pan and then transfer the entire test portion to the viscometer tube using a plastic funnel. Then lightly tap the funnel a few times to get all dry matter into the tube. Repeat for the second test portion.

(2) Tilt the viscometer tube and tap or rotate the tube so that a portion of the tube bottom is not covered by the wheat. This will allow the water to reach the bottom of the tube and help prevent clumping.

(3) While the tube is tilted, dispense 25.0 ± 0.3 mL distilled or deionized water and straighten the tube as it fills.

(4) Complete steps 2 through 3 for the second viscometer tube and immediately proceed to step b, Shaking the Viscometer Tube.

b. Shaking the Viscometer Tube.

(1) Open the doors on the Shakematic by pressing the [OPEN] button. The [START] button will blink green.

(2) Insert a Shakematic tube stopper into the viscometer tube.

(3) Insert the bottom of the tube into the hollow of the holder, and then push the upper part of the tube into the top of the holder.

(4) Close the doors manually. The [START] button will become a steady green light.

(5) Press the [START] button. The shaking starts and the green [START] button will turn red. The Shakematic will stop after a few seconds, and the doors will automatically open when the shaking is complete.

(6) Carefully remove the viscometer tubes by first pulling the upper part out of its holder. Ensure no clumps or dry matter are visible in the tubes. If clumps or dry matter are present, start the process over with a new sample.

(7) Immediately proceed to step c, Scraping the Stopper and Viscometer Tube.

c. Scraping the Stopper and Viscometer Tube.

CAUTION: Failure to scrape the slurry residue from the stopper into the tube and from the tube walls down into the slurry will decrease the FN test result.

(1) Remove the stopper from the viscometer tube.

(2) Scrape the slurry coating fully from the stopper into the tube.

(3) Scrape the slurry coating the wall of the tube down to the top of the slurry with the viscometer-stirrer. Make sure scraping is thorough to ensure the slurry is scraped cleanly off the sides of the tube.

(4) Repeat step 1 through 3 for the second viscometer tube.

(5) Immediately proceed to step d, FN Instrument Analysis.

d. FN Instrumental Analysis.

(1) Place the tube and the viscometer-stirrer into the FN instrument's water bath tube holder and start the instrument.

(2) Once the instrument is finished, record the FN readings from the instrument.

There is very little alpha-amylase activity in samples with a FN result above 400 seconds at sea level (29.92 inHg). Thus, when the FN results exceed 400 seconds at sea level, record the result on the certificate as "more than 400 seconds." Each official service provider will need to determine FN reading that is equivalent to 400 seconds at sea level entering the measured barometric pressure at their location into attachment 2. If the applicant requested the actual FN results or the actual result is needed for sublot analysis, allow the test to continue until completion.

di. Cleaning the Viscometer Tubes.

CAUTION: Do not use soap or detergent to clean the viscometer tubes. Clean the tubes only per the manufacturer's recommendation.

Clean the viscometer thoroughly between samples.

(1) Hold the viscometer tube under running water and remove the viscometer-stirrer.

(2) Using a Spolett or brush, clean the viscometer-stirrer and viscometer tube thoroughly, making certain that all gelled material is removed from inside the black, ebonite neck of the viscometer-stirrer.

dii. FN Data Entry, Measurement Agreement, and Correction.

Attachment 2 has a table for data entry. Each row is for recording test information for a sample. Enter the date of inspection, instrument serial number, sample ID, measured moisture, specified moisture basis, and the two FN measurements. The file can be saved locally for use.

The table is set up to calculate the average FN result, agreement of the duplicate measurements, and pressure corrected FN under "as-is"

moisture basis and specified moisture basis according to the following procedures.

(1) Average FN. The average FN is calculated according to equation 2.

Equation 2. Average Falling Number

$$FN_A = (FN_1 + FN_2) \div 2$$

where FN_A is the average FN, and FN_1 and FN_2 are the two results from duplicate analysis of the sample

(2) Agreement of Duplicate Measurements. For samples having a reading 400 seconds or less, the maximum allowable difference of the two results from the average is 5.00 percent. This requirement does not apply to samples for which both readings are greater than 400 seconds.

(a) If the first set of duplicate results are within ± 5.00 percent from the average of the two tests, certify the results. If the duplicate results differ by more than ± 5.00 percent from the average of the two tests, proceed to the next step.

(b) Repeat the test with the same ground sample. If the results are within tolerance, certify the results. If not, proceed to the next step.

NOTE: If the results from the second set are not within the tolerance, inform TSD.

(c) Grind a new portion of the sample and perform the falling number test again. If the results are within tolerance, certify the results. If not, proceed to the next step.

(d) Repeat the falling number test. If the results are within tolerance, certify the results. If not, consult the troubleshooting section and fix any instrument issues. If instrument issues cannot be resolved, use another instrument (if available) or contact Perten Instruments.

(e) After resolving the issue, repeat testing with original ground sample and check the measurement agreement again.

(3) FN Pressure Correction. The average FN is corrected to sea level using the barometric pressure according to equation 3. The equation is applicable to elevations from 0–5000 ft.

Equation 3. Pressure Corrected Falling Number

$$FN_{PC} = 60 + 10^{[log(FN_A - 60) + (-0.0214 \times (29.9213 - P))]}$$

where FN_{PC} *is the pressure corrected FN,* FN_A is the average FN, and P is the barometric pressure measured in inHg.

NOTE: The pressure corrected FN derived above is the FN for the "as-is" moisture basis. No moisture correction is applied.

(4) Moisture Correction. The pressure corrected FN is adjusted for the moisture basis of 14.0 percent as the default. Another value for the moisture basis may be used, if specified by the applicant. The FN for a particular moisture basis (FN_{MB}) other than "as-is" is determined according to equation 4.

Equation 4. Falling Number Moisture Correction

$$FN_{MB} = FN_{PC} \times (100 - MB) \div (100 - (1.904606 + (0.81661 \times M)))$$

where FN_{MB} is the FN at a specified moisture basis, FN_{PC} is the pressure corrected FN, MB is the moisture basis, and M is the percent moisture determined by the GAC2500-UGMA or Perten AM 5200-A moisture meter.

11. AVERAGING SUBLOTS

Enter the pressure corrected FN for each sublot into attachment 3. A special formula is used to average the sublot results, because FN values do not have additive properties. Low FN results have a pronounced affect in lowering the average FN result when combined with a sample having a high FN reading. The average sublot FN is calculated as follows:

a. Each sublot FN is converted to its Liquefaction Number (LN) equivalent using equation 5.

Equation 5. Conversion of Falling Number to Liquefaction Number

$$LN = 6{,}000 \div (FN_{PC} - 50)$$

where FN_{PC} is the pressure corrected FN.

b. The total (i.e., sum) and average of the sublot LN values are calculated.

c. The average sublot LN value is converted to its FN equivalent using equation 6 and displayed to nearest whole number.

Equation 6. Conversion of Average Sublot Liquefaction Number to Average Sublot Falling Number

$$FN_{SA} = (6{,}000 \div LN_{SA}) + 50$$

where FN_{SA} is the sublot average FN and. LN_{SA} is the sublot average LN.

12. COMPOSITE SAMPLE ANALYSIS

Criteria for composite sample analysis of export and domestic shipments not graded under the CuSum plan are as follows.

If a lot of up to 5 railcars, 15 trucks, or 20 containers is receiving a single certificate, the same group of carriers may also have FN determined from a composite sample, or from a combination of the individual carrier results, and reported on the same certificate. Carriers may not be grouped differently for FN than for other factors. Please note that FN must not be averaged arithmetically. Each FN must be converted to its LN, the average LN calculated, and the average LN converted back to a FN using equations 5 and 6 in section 12. Attachment 3 can be used for performing the calculations.

When carriers are receiving individual certificates, separate certificates may be issued for falling numbers for groups of up to 5 railcars, 15 trucks, or 20 containers, and composite samples may be used for the FN test. These separate certificates may be USGSA certificates, which also report official criteria such as aflatoxin or protein, or they may be AMA certificates that report only FN.

An entire train can receive a FN from a single composite sample, regardless of whether or not the lot was graded under the CuSum plan.

13. CERTIFICATION

a. AMA Certification.

Report FN results on the inspection certificate (lot or sample) in accordance with chapter 6 of the Processed Commodities Handbook.

On the inspection certificate, describe the commodity as "Wheat." Under "Results of Inspection," report the FN result with the applicable statement below. FN results are reported for certification to the nearest whole number.

(1) "Falling Number:_____, percent moisture basis."

Use this statement to report FN results on any specified moisture basis.

(2) "Falling Number:_____, dry matter basis."

(3) "Falling Number:_____, as is moisture basis."

Divided lot inspection certificates are available (for lot inspections only) upon request of the applicant.

b. Optional (USGSA) Certification.

(1) At the request of the applicant, FGIS will provide FN results in the “Results” section of the USGSA inspection certificate.

(2) When FN results are reported on a USGSA inspection certificate the following qualifying statement must be entered in the “Remarks” section of the certificate.

“Falling Number results provided under the authority of the Agricultural Marketing Act (AMA) of 1946.”

NOTE: This certification procedure is applicable only to FN tests that are performed in conjunction with other testing services performed under the USGSA. FN testing performed independent of USGSA services must be reported only on an AMA inspection certificate.

14. RETEST AND APPEAL

A retest and/or appeal inspection is available upon request by the applicant. A retest is performed on the basis of the whole grain file sample, and an appeal is performed on the basis of the whole grain file sample or a new sample (AMA Part 868.33 and 868.35).

a. A retest inspection may be performed by the same office that performed the original inspection.

b. An appeal inspection, when requested by the applicant, may be performed by an FGIS field office, TSD, or Federal/State office that monitored the office that performed the original inspection or retest.

Official personnel shall not perform a retest or appeal inspection of a sample of which they participated in a previous inspection, unless there is only one authorized person available at the time and place of the requested service. Also, whenever possible, the retest or appeal inspection should be performed on a different FN apparatus than the one used for the original inspection.

The applicant may also request a retest and appeal inspection of individual sublots without separate certification. That is, a single inspection certificate for a shiplot may use original, retest, and appeal results for different sublots for the final certification.

The retest or appeal inspection shall be performed according to the procedures specified in this directive for original FN determinations.

15. QUALITY CONTROL

a. Quality control has been included in this directive by establishing procedures for checking barometers, thermometers, balances, water dispensers, and water bath temperatures to ensure proper operation of equipment.

b. Monitoring and proficiency testing will be performed according to FGIS Program Directive 9180.84, National Falling Number Quality Assurance Program.

c. New FN instruments and repaired instruments must undergo a validation according to FGIS Program Directive 9180.84 before use in delivering official certification of FN.

16. TROUBLESHOOTING

For troubleshooting tips, refer to attachment 4.

17. REFERENCES

- American Association of Cereal Chemists (AACC) International Method 56-81.03.

- Delwiche, S.R.; Rausch, S.R.; Vinyard, B.T. Correction of Wheat Meal Falling Number to a Common Barometric Pressure at Simulated Laboratory Elevations of 0 to 1500 Meters. Cereal Chemistry **2018**, 00, 1–8.

18. INQUIRIES

Direct any technical inquiries regarding this directive to the Falling Number Program, Analytical Chemistry Branch by phone at (816) 891-0401 or FGISFallingNumber@usda.gov. Direct any policy questions regarding this directive to Gregory J. Giese, Policies, Procedures, and Market Analysis Branch, at Gregory.J.Giese@usda.gov.

19. ATTACHMENTS

Some browsers may not show a navigation pane needed to open attachments. If the browser being used does not display a navigation pane on the left side of the browser, a small triangle at center of the extreme left edge of the page, or the Adobe symbol at the bottom of the page, the document should be saved locally (on a computer or thumb drive) as a pdf. After saving locally, open the pdf and look for the navigation pane (usually on the left of the display). Attachments are accessed through an icon resembling a paperclip. Click on the paperclip to see the attachment(s). Double click on the attachment of interest.

Attachment 1: Water Bath Check
Attachment 2: Falling Number Data Entry, Validation, and Correction
Attachment 3: Average Sublot or Carrier Falling Number
Attachment 4: Troubleshooting Guide