

Program Notice

FGIS-PN-14-12

7/24/2014

SUNFLOWER OIL CALIBRATION ADJUSTMENT AND UPDATED CALIBRATION PROCEDURES FOR NUCLEAR MAGNETIC RESONANCE (NMR) INSTRUMENTS

1. PURPOSE

This notice announces that beginning July 24, 2014, the Grain Inspection, Packers and Stockyards Administration (GIPSA) has updated the calibration procedure for approved Nuclear Magnetic Resonance (NMR) instruments and implemented new Sunflower Seed Standards (SSS) for the official testing services of sunflower oil at a 10 percent moisture basis under the United States Grain Standards Act (USGSA).

2. BACKGROUND

GIPSA recently completed a review of the official NMR sunflower oil program. The performance of the NMR instruments compared to the standard reference method, Oil Extraction, was also reviewed. Based on the results of the review, GIPSA determined that the current calibration procedure consisting of a three-point calibration be updated to a four-point calibration using three new SSS to more closely align the official NMR sunflower oil measurements with the reference Oil Extraction method. In addition, the four-point calibration will provide a more robust calibration that is less affected by sample variability and will provide increased reproducibility of the results.

3. ANTICIPATED EFFECT

The effect of the new SSS and updated calibration procedures will be to more closely align the official NMR oil measurements with the Oil Extraction reference method, based on the average for the master instruments maintained by the Technology and Science Division (TSD). The anticipated effects of the adjustment will be to raise the oil result by 0.10 percent across the full oil range for High Oleic sunflower samples and to raise the oil result by 0.20 percent across the full oil range for NuSun sunflower samples. Results may vary on a sample-by-sample and instrument-by-instrument basis.

4. OXFORD BENCHTOP NMR ANALYZER MQC-5

- a. Instrument Setup.

NOTE: Contact TSD about new SSS samples before placing newly purchased NMR instruments in service. TSD will provide instructions to check the accuracy of the instrument and correct any noted deficiencies before the instrument is placed into official service. If problems are identified the checkout process may take several days to complete; therefore, contact TSD as soon as possible. Do not use newly purchased instruments for official NMR oil testing until the instrument has been checked and accepted by TSD.

(1) Setup Information.

If the instrument is already powered on and the temperature of the magnet is already 40.0 degrees Celsius (°C), please proceed to section (2) Calibration.

After the instrument is powered on, open the “MQC System Setup” icon on the desktop. A series of tests will be carried out to confirm the instrument communications and the functionality of the console unit. Please follow all computer prompts and consult the MQC-5 user manual “Section 4 – Using the Instrument” for any questions. The set up process may take up to 24 hours.

(2) Calibration.

NOTE: The instrument software for the Oxford NMR supports a four-point calibration. The points will be the high, medium, low, and blank-valued SSS.

The NMR instrument must be calibrated before testing market samples and when room or sample temperature changes by $\pm 1.0^{\circ}$ C or more.

The following steps describe the calibration process:

- (a) Double click the “EasyCal Applications” icon on the desktop. Select the “Oil Content in Seeds” application. In the window at the top right hand corner, verify the magnet temperature is at 40.0 degrees Celsius (°C).
- (b) Name the new calibration: (GIPSA [LN or NS or HO]MMDDYY) where “[LN or NS or HO]” is an abbreviation of the class (LN for linoleic, NS for NuSun, or HO for high oleic), "MM" is the month, "DD" is the day, and "YY" is the year. An example of this would be GIPSANS101512, indicating that the calibration is for NuSun seed created on October 15, 2012.

- (c) Click “Start”.
 - (d) Following the prompts, insert the oil tuning sample for analysis. Remove the tuning sample when prompted.
 - (e) Click “Continue”.
 - (f) Enter the Sample ID, mass and oil percent for the high oil percent SSS with the information provided on the label.
 - (g) Click “Ok”.
 - (h) Following the prompts, insert the high oil SSS sample for analysis.
 - (i) Remove the high oil SSS when prompted.
 - (j) Click “Yes” when asked to scan another standard.
 - (k) Repeat steps (f) through (j) for the medium, low and blank SSS standards.
 - (l) Click “No”, when asked to scan another standard.
 - (m) After the computer calculates the calibration parameters, record the slope, intercept, correlation coefficient (R^2), room temperature to 0.1°C , and the original and corrected/ calculated values for all SSS on the calibration log. The correlation coefficient must be greater than 0.9900. If not, a new calibration will be needed. Repeat steps (b) through (o) again using a new calibration name following this format: GIPSA[LN or NS or HO]MMDDYYx where “x” is the number of the recalibration (1 for the first recalibration, 2 for the second recalibration, etc.). Contact TSD if the correlation coefficient is not greater than 0.9900 after recalibration.
 - (n) Click on “Automatic Calibration” to complete the calibration process.
 - (o) Click on “Exit” then confirm by clicking “Yes”. Exit out of any remaining unnecessary windows.
- (3) Calibration Check.
- (a) Double click the “EasyCal Calibration” icon located on the desktop.
 - (b) Choose the appropriate calibration being checked.

- (c) Click “Start”.
- (d) Insert the oil tuning sample when prompted.
- (e) Remove the oil tuning sample when prompted.
- (f) Enter the sample name for the high SSS standard.
- (g) Enter the sample mass and click “Ok”.
- (h) When prompted insert the high oil SSS. The instrument will automatically begin the analysis.
- (i) Remove the high oil SSS when prompted.
- (j) Record the oil value and the room temperature to 0.1° C on the Calibration/Check Sample Log and calculate the difference between the reported and calculated value for the SSS. If the difference is greater than 0.3percent, repeat the analysis. If the repeat analysis result difference is still greater than 0.3 percent, recalibrate the instrument.
- (k) Repeat steps (f) through (j) for the medium, low and blank SSS standards.
- (l) Click on “Cancel”. Click on “Exit” then, confirm by clicking “Yes.”. Exit out of any unnecessary windows.

NOTE: Test the SSS as a market sample to check the NMR instrument accuracy after calibration, when the room temperature changes by ± 0.5 °C, after every 30 - 40 samples have been analyzed, or every two hours, whichever comes first. Maintain a record (electronic or written) of the calibration checks using the NMR Calibration Monitor Log and Continuation Sheet (Attachments 1 & 2, NMR Handbook, Chapter 3, 7/24/2014) as a template.

b. Sample Preparation

All samples must be prepared according to Chapter 3- “Sample Preparation” of the NMR Handbook for dried or non-dried sample with the exception that the portion size used to determine the NMR oil for the Oxford MQC-5 must not extend over 50 mm in height using a 51-mm diameter NMR tube.

c. Sample Analysis.

Once the instrument has been properly calibrated and the calibration check has been completed, begin analyzing market samples.

(1) Testing Market Samples.

(a) *Dried and Non-dried Samples*

- i. Double click the “EasyCal Calibration” icon located on the desktop to open the program.
- ii. Choose the appropriate calibration for the sample being tested.
- iii. Click “Start”.
- iv. Insert the oil tuning sample when prompted.
- v. Remove the oil tuning sample when prompted.
- vi. Enter the sample ID.
- vii. Enter the sample mass and click “Ok”.
- viii. When prompted insert the sample. The instrument will automatically begin the analysis.
- ix. Remove the sample when prompted.
- x. Record the percent oil for dried samples or uncorrected percent oil for non-dried samples.
- xi. Repeat steps (vi) through (x) for any additional samples of the same seed type. For samples of a differing seed type, click on “Cancel”. Click on “Exit” then confirm by clicking “Yes.”. Repeat steps (ii) through (x).

NOTE: To repeat the analysis of a single sample, you must remove the sample from the magnet and allow it to equalize to room temperature (10-15 minutes). Repeat analysis steps (vi) through (x) when the sample has returned to room temperature.

(b) *Non-dried NuSun (Mid-oleic) or Linoleic Samples Only*

Once the uncorrected percent oil on a non-dried NuSun (mid-oleic) or linoleic sample has been determined by analysis, the result must be corrected using the GIPSA-provided software.

- i. Double click on the desktop icon "SHORTCUT TO SUNFLWR3." Minimize other windows if needed.
- ii. Enter the sample ID.
- iii. Enter the uncorrected percent oil value for the sample.
- iv. Enter the percent moisture obtained using an approved moisture meter.

NOTE: After testing a sample for moisture content with an official moisture meter, place the sample in a closed container until oil testing is performed. This will minimize changes in moisture content of the sample.

NOTE: If the official moisture meter result is not measured within 24 hours prior to the oil measurement, or the official moisture meter result is not between 4.5 percent and 16 percent, the undried sample oil measurement procedure cannot be used. Samples that do not qualify for the undried oil measurement procedure must be tested after drying (air-oven method).

- v. Click on the appropriate oil type.
- vi. Record the "oil, 10 percent M Basis".
- vii. Click "Save".

d. Reporting Results.

Record and report the percent oil on the pan ticket and inspection log, and certify to the nearest tenth percent using the standard FGIS rounding procedures.

5. BRUKER MINISPEC MQ 7.5 and BRUKER MINISPEC MQ-ONE SEED ANALYZER XL

Please follow these procedures when setting up and operating the Bruker Minispec 7.5 or the Bruker Minispec MQ-One XL using the Minispec Plus instrument software.

a. Instrument Setup.

(1) Setup Information.

If the instrument is already on and the temperature of the magnet is already 40.0 degrees Celsius ($^{\circ}\text{C}$), please proceed to step (b). Consult the manufacture's manual for the proper indication of magnet temperature.

- (a) After the instrument is powered on, a series of tests are carried out to confirm the instrument communications and the functionality of the console unit.
- (b) Start the Minispec software on the personal computer. Enter password information if required.
- (c) At the start of the day, select the "Daily Check" icon and run the auto-validation procedure.
- (d) Insert the daily check sample then "Start". After following the prompts, the instrument will perform a series of tests.
- (e) If no error message appears, remove the check sample and the instrument is ready for calibration (Section 5.a.(2) - Calibration).
- (f) If an error message appears, select "Update Settings" from the Minispec menu.
- (g) Click "OK" to proceed to update all instrument settings.
- (h) After the instrument updates the settings repeat the Daily Check. If the instrument repeatedly fails the Daily Check, contact Bruker's technical support for assistance. After use of the "Updated Settings", all previous calibrations become void and the instrument needs to be recalibrated.

(2) Calibration.

NOTE: A four-point calibration will be established with high, medium, low, and blank-valued SSS.

The NMR instrument must be calibrated before testing market samples and when room or sample temperature changes by $\pm 1.0^{\circ}\text{C}$ or more.

The following steps describe the calibration process:

- (a) Click on the “Calibrate” button, opening the “Available Calibrations” window. Click the “New Calibration” button at the bottom left of the window.
- (b) Name the new calibration GIPSA[LN or NS or HO]MMDDYY where “[LN or NS or HO]” is an abbreviation of the class (LN for linoleic, NS for NuSun, or HO for high oleic), "MM" is the month, "DD" is the day, and "YY" is the year. An example of this would be GIPSANS101512, indicating that the calibration is for NuSun seed created on October 15, 2012.
- (c) Select the “Bruker Oil in Seeds” application. Change the reference values to “Mass Percent” and click “Apply”.
- (d) Click the “Calibrate” button and you will be prompted to insert the high oil SSS to tune the receiver gain. Once the SSS is inserted, click on “Tune Gain”. Once completed, the calibration procedure screen will appear.
- (e) Provide the sample name (SSS###), sample mass, and sample reference values (percent oil of the SSS) from the SSS label. Insert the next standard as necessary.
- (f) Press “Measure”.
- (g) When the analysis is complete, remove the high oil SSS. Press “Next Sample” if another standard or the blank needs to be measured.
- (h) Repeat steps (e) through (g) for the medium, low, and the blank SSS standards.
- (i) Once completed, click on “Calibration Results” then on “Show Calibration”. A graph and calibration statistics will be displayed. Record the slope, intercept, correlation coefficient (R^2), room temperature to 0.1° C, and the original and corrected/ calculated values for all SSS on the calibration log. The correlation coefficient must be greater than 0.9900. If not, a new calibration will be needed. For a new calibration, click on “Return to Main Menu” and repeat steps (a) through (h) again using a new calibration name following this format: GIPSA[LN or NS or HO]MMDDYYx where “x” is the number of the recalibration (1 for the first recalibration, 2 for the second recalibration, etc.). Contact TSD if correlation coefficient is not greater than 0.9900 after recalibration.

NOTE: It is possible to hide older calibrations from view so they may not be used by the operators

- (j) If the calibration is acceptable, click “Sign” to accept the calibration. Enter any password information if required. The calibration will not be available for use without signing.
 - (k) Click on “Return to Main Menu”.
- (3) Calibration Check.
- (a) Select the “Measure” button and a new window will appear.
 - (b) Provide an appropriate batch name. This can be any identification the operator or location determines is appropriate. Press the enter key.
 - (c) Select the appropriate calibration being used.
 - (d) Insert the high oil SSS.
 - (e) Enter the sample name.
 - (f) Enter the sample mass.
 - (g) Enter any comments needed.
 - (h) Select the “Measure” button.
 - (i) When the analysis is complete, remove the high oil SSS.
 - (j) Record the oil value and the room temperature to 0.1° C on the Calibration/Check Sample Log and calculate the difference between the reported and calculated value for the SSS. If the difference is greater than ± 0.3 percent from the calculated value, repeat the analysis. If the repeat analysis result difference is still greater than ± 0.3 percent from the calculated value, recalibrate the instrument.
 - (k) Repeat steps (d) through (j) for the medium, low, and blank SSS standards. If needed, click on “Next Sample”.

NOTE: Test the SSS as a market sample to check the NMR instrument accuracy after calibration, when the room temperature changes by ± 0.5 °C, after every 30 - 40 samples have been analyzed, or every two hours, whichever comes first. Maintain a record (electronic or written) of the calibration checks using the NMR Calibration Monitor Log and Continuation Sheet (Attachments 1 & 2, NMR Handbook, Chapter 3, 7/24/2014) as a template.

(l) Click on the return arrow button.

b. Sample Preparation.

All samples must be prepared according to Chapter 3- "Sample Preparation" of the NMR Handbook for dried or non-dried sample with the exception that the portion size used to determine the NMR oil for the Oxford MQC-5 must not extend over 50 mm in height using a 51-mm diameter NMR tube.

c. Sample Analysis.

Once the instrument has been properly calibrated, the Daily Check has been validated, and calibration check completed, begin analyzing market samples.

(1) Testing Samples.

- (a) Select the "Measure" button.
- (b) Provide an appropriate batch name. This can be any identification the operator or location determines is appropriate.
- (c) Select the appropriate calibration for the sample being tested.
- (d) Insert the sample.
- (e) Enter the sample name.
- (f) Enter the sample mass.
- (g) Enter any comments needed.
- (h) Select the "Measure" button.

- (i) When the analysis is complete, remove the sample and record the value. Click on “Next Sample” if needed.
- (j) Repeat steps (d) through (i) for the additional samples of the same seed type. For samples of a differing type, press the return arrow button and repeat steps (a) through (i).

NOTE: To repeat the analysis of a single sample, remove the sample from the magnet and allow it to equalize to room temperature (10-15 minutes). Repeat analysis steps (d) through (i) when the sample has returned to room temperature.

d. Reporting Results.

Record and report the percent oil on the pan ticket and inspection log, and certify to the nearest tenth percent using the standard FGIS rounding procedures.

6. FILING

Retain a copy of this program notice with the NMR Handbook until the handbook is revised to include the information herein.

7. QUESTIONS

Please direct any questions regarding this program notice to Greg Giese, Inspection Instrumentation Branch at (816) 891-0460 or Gregory.J.Giese@usda.gov.

/s/Robert Lijewski

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