

# **Program Notice** FGIS-PN-14-04 11-21-13

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## **APPROVAL OF NEW NUCLEAR MAGNETIC RESONANCE (NMR) INSTRUMENTS**

### **1. PURPOSE**

This notice announces that the Grain Inspection, Packers and Stockyards Administration (GIPSA) has approved two additional instruments for official testing of sunflower oil at a 10 percent moisture basis under the United States Grain Standards Act as amended (USGSA).

### **2. BACKGROUND**

Testing sunflower seed for oil content as “official criteria” is authorized under Section 7(b) of the USGSA. GIPSA is responsible for officially determining and certifying oil content of sunflower seed, monitoring the accuracy of official sunflower seed oil results, and approving the official equipment. The instruments currently approved to officially determine oil percent in sunflowers use pulsed nuclear magnetic resonance (NMR) technology. Currently three instruments using pulsed NMR technology are approved for the official determination of sunflower oil. In order to meet market demands and maintain current technology, GIPSA has tested and approved two new models to be implemented within the official system.

### **3. APPROVED INSTRUMENTS**

- a. Oxford Benchtop NMR Analyzer MQC-5 - is approved for the determination of sunflower oil in linoleic, mid-oleic (Nusun), and high oleic seeds using the dried method. It is also approved for linoleic and mid-oleic (Nusun) using the non-dried method, as described in Chapter 3 of the GIPSA NMR Handbook.
- b. Bruker Minispec MQ-One Seed Analyzer XL - is approved for the determination of sunflower oil in linoleic, mid-oleic (Nusun), and high oleic seeds using the dried method described in Chapter 3 of the GIPSA NMR Handbook.

**NOTE: These instruments must be operated according to the operator’s manual supplied by the manufacturer, and must use officially approved methods and calibrations for the sample procedures for all sunflower samples as described in the NMR Handbook.**

#### 4. OXFORD BENCHTOP NMR ANALYZER MQC-5

##### a. Instrument Setup.

**NOTE: Contact Technology and Science Division (TSD) about new sunflower seed standard (SSS) samples before placing newly purchased NMR instruments in service. TSD will provide instruction to check the accuracy of the instrument and correct 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 on and the temperature of the magnet is already 40.0 degrees Celsius ( $^{\circ}\text{C}$ ), 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 three-point calibration. The first two points will be the high and low valued SSS, while the third sample will be a blank, or an empty NMR tube. The mass and oil percent used for the set-up of the blank will be 30.00 grams and 0.00 percent oil.**

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) 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 ( $^{\circ}\text{C}$ ).

- (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 low oil SSS.
- (l) Repeat steps (f) through (i) for the blank standard.
- (m) Click “No”, when asked to scan another standard.
- (n) Record the slope, intercept, correlation coefficient ( $R^2$ ), and the room temperature to 0.1° C 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.
- (o) Click on “Automatic Calibration” to complete the calibration process.

- (p) 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 values and the room temperature to 0.1° C on the Calibration/Check Sample Log and calculate the difference between the reported and known value for the SSS. If the difference is greater than 0.3 percent, 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 Low Oil SSS.
  - (l) Repeat steps (f) through (i) for the blank.
  - (m) 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  $\pm 0.5^{\circ}$  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 Check Sample Log in the Attachment 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 50 mm in a 51 mm 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 type. For samples of a differing 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, inspection log, and certify to the nearest tenth percent using the standard FGIS rounding procedures.

## 5. BRUKER MINISPEC MQ-ONE SEED ANALYZER XL

**NOTE: The following set up and operating procedures will also apply to the Bruker Minispec 7.5.**

### a. Instrument Setup.

#### (1) Setup Information.

If the instrument is already on and the temperature of the magnet is already 40.0 degrees Celsius (°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: The instrument software for the Bruker NMRs requires a three-point calibration. The first two points will be the high and low valued sunflower seed standard (SSS), while the third sample will be a blank, or an empty NMR tube. The mass and oil percent used for the set-up of the blank will be 30.00 grams and 0.00 percent oil.**

The NMR instrument must be calibrated before testing market samples and when room or sample temperature changes by  $\pm 0.5^{\circ}$  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 a sample name (SSS###), samples mass, and sample reference values (percent oil of the SSS) from the SSS label. Insert the next standard if 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 low value SSS and the blank. The mass and oil percent used for the set-up of the blank will be 30.00 grams and 0.00 percent oil.

- (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 ( $R^2$ ), and the room temperature to 0.1° C 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 SSS.

- (j) Record the oil values and the room temperature to 0.1° C on the Calibration/Check Sample Log and calculate the difference between the reported and known value for the SSS. If the difference is greater than 0.3 percent, repeat the analysis. If the repeat analysis result difference is still greater than 0.3 percent, recalibrate the instrument.
- (k) Repeat steps (d) through (j) for the Low Oil SSS. 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  $\pm 0.5^{\circ}$  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 Check Sample Log in the Attachment 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 55 mm in a 51 mm 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.

**NOTE: All Samples must be prepared according to Chapter 3 - “Sample Preparation” of the NMR Handbook for dried sample analysis.**

- (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 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, 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 email at [Gregory.J.Giese@usda.gov](mailto:Gregory.J.Giese@usda.gov).

*/s/Robert Lijewski*

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