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Nuclear Magnetic Resonance (NMR) Handbook

Program Handbook

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Nuclear Magnetic Resonance Testing for Sunflower Seed Oil

Foreword

The Nuclear Magnetic Resonance (NMR) Handbook has been established to provide official personnel with procedures to follow for the testing and certification of sunflower seed oil using the NMR method.

All official inspection personnel authorized or licensed to perform NMR testing must reference this handbook for procedures.

This handbook supersedes the FGIS Nuclear Magnetic Resonance (NMR) Handbook dated May 12, 2003.

/s/ Robert S. Lijewski
Robert S. Lijewski, Acting Director
Field Management Division

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CHAPTER 1

GENERAL INFORMATION

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1.1 SCOPE

Testing sunflower seed for oil content as “official criteria” is authorized under Section 7(b) of the United States Grain Standards Act (USGSA), as amended. All official sunflower seed oil analysis under the USGSA is performed in accordance with procedures prescribed in this handbook by authorized Federal Grain Inspection Service (FGIS) employees or licensed personnel employed by delegated/designated agencies.

The continuous wave and pulsed low resolution Nuclear Magnetic Resonance (NMR) instruments are the only approved instruments for official sunflower seed oil determination. The NMR method is based on the principle of activating hydrogen atoms in the oil using electromagnetic radiation and a magnet. The NMR reading is a measure of the number of activated hydrogen atoms. Technical Services Division (TSD) will use the petroleum ether oil extraction method (AOCS method number Ai 3-75) as a standard to which the NMR instruments are calibrated and referenced.

This handbook establishes procedures for officially determining and certifying oil content of sunflower seed, monitoring the accuracy of official sunflower seed oil results, and maintaining sunflower seed oil equipment accuracy.

1.2 DEFINITIONS

Check Samples - Sunflower seed samples tested by TSD and distributed to all specified service points for monitoring the uniformity of field results.

Collaborative Study - A study designed to compare NMR oil values determined by different laboratories.

Constituent - Compounds for which an analysis is made in a product, i.e., oil in sunflower seed.

Continuous Wave NMR - A technique used for determining the oil content of a sunflower seed sample by measuring the number of electromagnetically activated hydrogen atoms present in liquid oil. Continuous Wave (CW) instruments continuously apply external radio frequency energy to samples while simultaneously scanning the magnetic field. The signal is recorded by measuring the absorption of radiation by the nuclei at their resonant frequencies.

Correlation - The interdependency of one variable on another, i.e., the amount of oil extractable from a sample using petroleum ether and NMR response.

Monitor Samples - Sunflower seed samples randomly selected from the market which are analyzed and compared to a monitoring office. **NMR Response** - A measure of the number of activated hydrogen atoms within a magnetic field.

Oil - A mixture of a glyceride ester of fatty acids widely occurring in organic tissues that are liquid at room temperature.

Petroleum Ether Oil Extraction - A chemical determination of percent oil in a sample.

Pulsed NMR - A technique used for determining the oil content of a sunflower seed sample by measuring the number of electromagnetically activated hydrogen atoms present in liquid oil. Pulsed NMR instruments apply external radio frequency energy to samples as short pulses lasting a few microseconds. The pulses simultaneously excite all of the nuclei in a sample and the signal (called a free induction decay) is measured after the pulse. All modern NMR instruments use pulsed techniques.

Reference Value - An oil value determined by TSD for each of the Sunflower Seed Standard samples and Sunflower Seed Check samples.

Slope - The degree of slant of the regression line.

Specified Service Point - A city, town, or other location specified by an agency for the performance of official inspection or Class X or Class Y weighing services.

Sunflower Seed Standard (SSS) - Dried and sealed sunflower seed samples with established weights and NMR oil values. SSS are prepared by TSD and distributed to specified service points to calibrate NMR instruments.

Tuning Sample (TS) - A sample (sunflower seed oil or relaxed mineral oil) giving a signal large enough to tune the NMR instrument.

1.3 RESPONSIBILITIES

The general responsibilities for the sunflower seed oil testing program are as follows:

a. Responsibilities of the Technology & Science Division.

- (1) Maintain the standard reference petroleum ether oil extraction laboratory for FGIS and create calibrations for approved NMR instruments used for official NMR oil testing.
- (2) Establish the official oil content of all Sunflower Seed Standards (SSS).
- (3) Provide SSS samples to field offices and all service points providing official NMR sunflower seed oil determination.
- (4) Monitor the capability of the official sunflower seed oil testing program.
- (5) When necessary, review sunflower seed oil analysis procedures at FGIS field offices and specified service points.

- (6) Recommend corrective and follow-up action when problems are detected.
- (7) Provide technical support and training to official personnel in matters relating to oil analysis.
- (8) Initiate and/or conduct and report collaborative and/or special studies as needed.
- (9) When needed, perform calibration studies and make recommendations.
- (10) Provide Board appeal inspection, and where applicable, appeal inspection for sunflower seed oil testing.
- (11) Issue certificates and assess fees for Board appeal, and where applicable, appeal inspection service.
- (12) Where applicable, coordinate and maintain the sunflower seed NMR oil testing program within the circuit.

b. Responsibilities of FGIS Field Office Managers.

- (1) Coordinate and maintain the sunflower seed NMR oil testing program within the circuit.
- (2) At domestic locations, perform or make arrangements to perform appeal inspections for sunflower seed oil testing services within the field office circuit.
- (3) At export locations, provide original, reinspection, and appeal sunflower seed oil testing services in areas not assigned to an official agency.
- (4) Forward file samples for Board appeal testing services to TSD.
- (5) Select and forward samples for monitoring to TSD.
- (6) Monitor the performance of specified service points within the circuit.
- (7) Review oil testing procedures at specified service points within the circuit.
- (8) Immediately inform TSD of problems detected in the circuit and initiate corrective and follow-up action.
- (9) Provide technical support and training to official inspection personnel.
- (10) Assist TSD in conducting collaborative and/or special studies.

C. Responsibilities of Official Agency Managers/State Cooperators.

- (1) Coordinate and maintain a sunflower seed oil testing program within the assigned geographic area.
- (2) Perform original and reinspection NMR sunflower seed oil testing services within the assigned geographic area and forward file samples for appeal sunflower seed oil testing services to TSD.
- (3) Select and forward samples for monitoring to TSD.
- (4) Routinely review oil analysis procedures at specified service points within the assigned geographic area.
- (5) Permit only official personnel who are trained and licensed for sunflower seed oil testing to perform such activities.
- (6) Provide technical support and training to licensed inspection personnel within the assigned geographic area.
- (7) Assist TSD in conducting collaborative and/or special studies.
- (8) Inform the monitoring field office manager and/or TSD, as applicable, of problems detected within the assigned geographic area and initiate corrective and follow-up action.

CHAPTER 2

NMR TESTING EQUIPMENT

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2.1 PURPOSE

To ensure that the FGIS sunflower oil program is applied uniformly by all specified service points, that only approved continuous wave or pulsed NMR equipment and procedures are used, that equipment maintenance and testing schedules are followed, and that laboratory setups conform to FGIS specifications.

2.2 APPROVED EQUIPMENT

The following equipment is approved for official NMR sunflower oil determinations.

Aluminum Dishes - Aluminum dishes with lids, approximately 90 millimeters (mm) in diameter and 50 mm deep with closely fitted slip-on cover.

Balance - An approved electronic balance with 0.01 gram precision and RS-232 output (Mettler PM-200 or equivalent).

Bottle Brushes - Bottle brushes for cleaning NMR sample tubes.

Desiccating Cabinet - An airtight cabinet which can be equipped with a desiccant material to maintain a very low relative humidity environment.

Desiccant - Silica gel for drying, indicating, 6-16 mesh.

Electronic Power Conditioner (Optional) - An electronic power line conditioner (Sola model No. EPC 150-60, Tripp Lite model LS-604 or equivalent).

Forced Air Convection Oven - A forced air convection oven (Blue M Model OV-490A-2, or equivalent).

NMR Instruments

- a. Oxford Instruments pulsed NMR models MQA 6005 and MQA 7005 equipped with 5 Mega Hertz (Mhz) magnet and 150 milliliters (ml) magnet coil assembly.
- b. Bruker minispec mq7.5 pulsed NMR equipped with 7.5 Mhz magnet and 150 ml magnet coil assembly. (Bruker Canada Ltd., Milton, Ontario L9T 1Y6, Canada, phone 1-905-876-4641).
- c. The Newport Analyzer Models Mark III-A and Oxford 4000 model (continuous wave wide-line NMR) equipped with type 10 magnet (Watson 10) and 150 ml magnet coil assembly. (Oxford Instruments Inc., 130-a Baker Avenue Extension, Concord, MA 01742, phone 1-800-447-4717).
- d. Universal Maran Ultra pulsed NMR equipped with 5 Mhz magnet and 150 ml magnet coil assembly. (Universal Systems, 29500 Aurora Road, Unit 16, Solon, OH 44139, phone 1-440-349-3210).

Note: The Newport Mark III-A, Oxford 4000 and Maran Ultra (with 150 ml magnet coil assembly) models are approved for official use but are not in service at the current time. Therefore, the handbook does not contain step-by-step procedures for instrument operation.

NMR Sample Tubes - NMR sample tubes for 150 mL NMR magnet coil assembly.

Rubber Stoppers - Rubber stopper for NMR sample tube, size No. 10.

Thermometers - Thermometer with 1° to 51°C scale and 0.1°C divisions.

2.3 DISCLAIMER CLAUSE

The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned. Except for the NMR instrument, equivalent equipment may be used in place of the items listed.

2.4 OIL TESTING FACILITIES

Equipment location and environmental factors can affect the performance of NMR sunflower seed oil testing equipment. The space and facilities used by official personnel must meet the specifications outlined below.

a. Location of Equipment.

The method used for determining the oil content of sunflower seed samples utilizes a magnet with predetermined electromagnetic strength to activate the hydrogen atoms present in the sample. Metal objects or a strong magnet placed near the NMR equipment may interfere with the electromagnetic field and produce erroneous results. Therefore, do not place metal objects adjacent to the NMR equipment. **Do not place the NMR instrument on a steel table.**

A vibration-free table should be used to support equipment. Also, when more than one electronic instrument is located in the same work area, maintain at least 60 centimeters (approximately 2 feet) distance between instruments.

In addition, NMR equipment must be placed in a location conducive to a stable environment and shielded from electrical or electromagnetic interferences. NMR equipment must be protected from drafts, heating and cooling vents or devices, and preferably be kept away from outside walls and windows.

b. Temperature.

Maintain a constant temperature between 18°C (65°F) and 30°C (85°F) in the room where the NMR instrument is located. Record room temperature using a calibrated thermometer located near the NMR instrument.

Insert the thermometer into a small glass or plastic bottle filled with sunflower seed to reduce erroneous readings. The depth of the sunflower seed must be sufficient to cover the insertion level of the thermometer. Fluctuation in room or sample temperature adversely affects analysis results. After calibration, a change in room or sample temperature greater than +0.5°C will require re-calibration using the SSS. Therefore, locate the NMR equipment in a room where the temperature remains very stable to minimize the need for re-calibration.

c. Power Supply.

The power for NMR instruments shall be supplied by a 120 + 10 VAC 15-20 amp dedicated circuit. A maximum of two electronic instruments and associated printers may be placed on one dedicated circuit (2 NMR or 1 NMR and 1 Near Infrared Transmittance (NIRT) instrument). To reduce interference from other sources, do not place other equipment on the circuit.

d. Dust.

Accessible surfaces of the NMR instrument, balance, and surrounding area shall be maintained essentially free from contaminants. Use a vacuum cleaner and brush for maintaining a clean and dust-free environment in the sunflower seed testing area. Do not use compressed air for clean-up purposes.

At locations where a dust collection system is not available, place the NMR instrument and balance in a room separate from all dust producing equipment such as grinders, dockage testers, and dividers.

2.5 INITIAL LABORATORY SETUP

Official service providers must observe certain guidelines when establishing new testing laboratories and/or placing new equipment in service.

a. New Laboratories.

Upon request, TSD will assist agencies in planning and preparing laboratories for official oil testing service. Agency managers must notify the field office manager, where applicable, or TSD that a new laboratory is being planned and provide a diagram of the proposed laboratory design. The diagram should contain the proposed locations of NMR oil testing equipment, location of major inspection equipment (e.g., dockage testers, dividers, etc.), and a description of the power supply. Any additional information regarding the laboratory setup or equipment should also be discussed.

Where applicable, the monitoring field office will forward a copy of all submitted information to TSD for review. Upon receipt, TSD will review the information and make recommendations to the agency and, where applicable, the monitoring field office to facilitate the laboratory setup.

b. New Equipment.

Notify TSD before placing newly purchased NMR instruments in service. TSD will provide instructions to check the accuracy of the instrument and correct any 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.

CHAPTER 3

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3.1 BASIS OF DETERMINATION

Determine sunflower seed NMR oil on a representative portion cut from the sample after the removal of foreign material. Report the sunflower seed NMR oil percent on a 10 percent moisture basis (mb).

3.2 CLEANING SAMPLES

Use a Boerner divider to obtain a representative sample portion of approximately 50 to 55 grams. Mechanically clean the sample portion using a Carter Dockage Tester operated as specified in the Grain Inspection Handbook, Book II. Sunflower seeds and dehulled seeds that pass over the riddle and the material that passes over the No. 3 and over the No. 8 sieves are combined to form the mechanically cleaned sample. Handpick the mechanically cleaned sample portion to remove all matter other than sunflower seed and dehulled seeds.

3.3 PORTION SIZE

The sample used to determine the NMR oil must not extend above the top of the Radio Frequency (R.F.) coil. For a 150 ml NMR sample tube, a depth of 50.8 to 63.5 mm (2 to 2 1/2 inches) must be observed.

Before drying the handpicked sample portion, check the sample volume using a marked NMR sample tube. If the sample volume does not fall within the required range, adjust the sample size until it does. If the deviation is large, use a Boerner divider to adjust the sample size.

3.4 DRYING SAMPLES

The technique used to determine sunflower seed oil is based on measuring the number of electromagnetically activated hydrogen atoms in a sample. Therefore, liquid hydrogen atom sources other than oil, such as moisture, must be removed prior to NMR oil determination.

NOTE: If the sample is comprised of Linoleic or Mid-Oleic (NuSun) type sunflower seeds and the GAC-2100 moisture result is between 4.5% and 16.0%, the undried analysis method can be used for the Oxford pulsed instruments only. However, if the sample is comprised of High-Oleic sunflower seeds or if the GAC 2100 moisture results for Linoleic or Mid-Oleic sunflower seeds are outside the 4.5%-16.0% range, the drying procedures must be applied.

Perform the following procedures for drying the sunflower seed sample to remove moisture:

- a. Preheat the moisture oven to 130°C and check the oven temperature with a calibrated thermometer. The oven temperature must be 130°C + 2°C before drying samples.

- b. Place the handpicked sunflower seed sample in a moisture dish and record the dish ID number.
- c. Place the sample in the preheated moisture oven with the lid under the dish. Dry the sample for 3 hours. Begin timing the drying once the temperature reaches $130^{\circ}\text{C} + 2^{\circ}\text{C}$.

3.5 COOLING SAMPLES

After drying, the sunflower seed samples must be stabilized to room temperature before performing NMR oil determination. In addition, room and sample temperature must be within $+ 0.5^{\circ}\text{C}$ from the temperature recorded during restandardization.

Allow samples to cool to room temperature using one of the following procedures:

a. Desiccating Cabinet Method.

- (1) Place a calibrated thermometer inside the desiccating cabinet.
- (2) Immediately after removing the samples from the drying oven, place the lid on the dish and place the dish in the desiccating cabinet. Monitor the desiccating cabinet temperature.
- (3) When the room and desiccating cabinet temperature are within $+0.5^{\circ}\text{C}$, samples are ready for NMR oil determination. The cooling time will be a minimum of 3 hours.

b. NMR Sample Tube Method.

A thermometer inserted through a rubber stopper is needed to measure the temperature of at least 1 sample in a rack (maximum 10 samples per rack) as they cool. The thermometer must be inserted into the rubber stopper so that approximately 25 mm of the thermometer will be immersed in the sunflower seed sample during the cooling period.

- (1) Immediately after removing each sample from the drying oven, pour the sample into a marked NMR sample tube and seal with a rubber stopper. Each rack must have at least one rubber stopper with a thermometer inserted.
- (2) Place the NMR sample tube into a rack. The rack should keep sample tubes separated by approximately 20 mm.
- (3) When room temperature and the thermometer reading are within $+ 0.5^{\circ}\text{C}$, the samples are ready for NMR oil determination. The cooling time will be at least 3 hours.

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4.1 SEED TYPE DECLARATION

Applicants requesting sunflower seed oil determination must declare the seed type as part of the request for service. Written confirmation or a verbal declaration may be required at the discretion of official personnel.

Applicants may make blanket declarations for all lots or a specific type of service. For example, "All inbound lots of sunflower seed are of high-linoleic-type unless specified otherwise."

The official certificate shall include a qualifying statement reflecting applicant declaration (see section 4.3 c).

4.2 EXPORT LOTS

Applicants for service may request official sunflower seed oil determination on the basis of subplot samples or a composite sample. The type of service and the quantity of sunflower seed to be loaded must be indicated on the load order document.

a. Sublot Basis.

- (1) Obtain a representative sample for each subplot.
- (2) Follow the official procedure to determine NMR oil content for each subplot sample. Record the oil percentage on the inspection log to the nearest tenth percent.
- (3) Using the standard FGIS rounding procedures, calculate the average NMR oil content for all sublots.

Determine the highest and lowest NMR oil percent for all sublots.

Report the high, low, and the average percent NMR oil (10% mb) to the nearest tenth percent on the certificate.

b. Ship Composite Basis.

- (1) Based on the number of sublots being loaded, determine the amount of sample portion (in proportionate size) to be taken from each subplot that will provide a 1,000-gram composite sample.
- (2) At the completion of ship loading, obtain a representative sample of at least 100 grams from the composite using a Boerner divider.
- (3) Determine the NMR oil content using the official procedures.
- (4) Report the percent NMR oil (10% mb), as determined on the composite sample, to the nearest tenth percent on the certificate.

4.3 CERTIFICATION

Record the percent oil of the sunflower seed using the appropriate approved statement in the “Remarks” section of the official grade certificate. Results may be reported on the same certificate with official grade and factor results, or reported on a separate certificate. When certifying oil alone (without official grade and factors) do not enter a grade on the “Grade” line.

a. Approved Statements for Export Lots.

(1) Sublot Basis

“Oil content of sublots range from (lowest) % to (highest) %.”

“Average oil content for all sublots %, 10 percent moisture basis.”

(2) Composite Basis

“Oil content %, 10 percent moisture basis. Determination based on a composite sample.”

b. Approved Statement for Domestic Lots.

“Oil content %, 10 percent moisture basis.”

c. Approved Qualifying Statements.

The applicant for service must declare the type of seed for NMR oil testing. Include one of the following statements on the certificate.

Insert the appropriate sunflower seed (linoleic, mid-oleic, high-oleic) to complete the statement.

Note: The term NuSun may be used in lieu of mid-oleic.

“(_____) type sunflower seed, per applicant statement.”

“Applicant states sunflower seed is (_____) type.”

Field office managers can approve minor modifications to the statements provided the meaning and intent of the statements have not changed.

CHAPTER 5

MONITORING PROGRAM

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5.1 GENERAL INFORMATION

The sunflower seed oil monitoring program is designed to monitor the accuracy of official sunflower seed oil determinations. Several methods are utilized to monitor particular elements in the sunflower seed oil testing process. These methods include monitoring by TSD, check samples, intermarket sample exchanges, and special studies or collaboratives.

Monitoring information is used by field offices and/or inspection agencies for evaluating the performance of their local oil testing programs. TSD uses the information to evaluate the capability of the national sunflower seed oil testing program and the performance of the calibration standards.

Monitoring by TSD will identify service points having questionable oil results. Field offices and/or agencies must initiate follow-up and corrective action whenever oil testing problems are detected. Follow-up and corrective action includes investigating, identifying, and correcting oil problems. Intermarket sample exchanges, opinion samples, and special studies or collaboratives, are tools which are used to help identify the cause of discrepancies. TSD will assist the field in identifying the cause(s) of oil testing problems.

5.2 MONITORING FILE SAMPLES

Oil testing performance is evaluated through TSD monitoring of official file samples. Sunflower seed oil program performance is determined by comparing original NMR oil results with monitoring results.

Specified service points are required to select and forward 10 samples per month to TSD for monitoring.

a. Selecting Samples.

Specified service points providing official NMR oil testing service shall use the following procedures to select monitoring samples.

- (1) Select 10 sunflower seed samples per month representing the range of oil values observed during the month. Do not select all samples from the same day. When less than 10 samples are tested during a month, select all samples tested.

Service points that perform a low volume of official oil determinations may, with TSD concurrence, periodically make up sets of 10 samples to check the accuracy of their instrument.

- (2) Mail the samples to TSD by close of business on the last business day of the month.

Note: TSD may request additional samples for monitoring purposes.

b. Preparing Samples.

- (1) Using a Boerner divider, obtain a portion weighing approximately 200 grams from each sample selected for monitoring. If a location frequently receives submitted samples of insufficient size to provide a 200-gram portion, TSD may approve a smaller monitor portion.
- (2) Place each sample portion in a clear plastic bag and close securely.
- (3) Include a completed monitoring data worksheet with the samples. Indicate any unusual conditions observed in the “remarks” section of the worksheet.

c. Packaging Samples.

- (1) Place the selected sample portions and completed monitoring data worksheet in a suitable mailing bag or box.
- (2) Prepare the appropriate mailing tag (business reply tag for official agencies, metered tag for FGIS) by indicating “SF Oil Monitoring” on the tag, and securely attach it to the bag or box.

d. Monitoring Results.

TSD will prepare summary reports and transmit the sunflower seed oil monitoring results to the appropriate specified service points for their review and follow-up action.

e. Evaluating Results.

TSD and specified service point managers are responsible for evaluating the monitoring results. If the results indicate unacceptable performance, they must initiate follow-up action, take the necessary corrective measures, and document any action taken to resolve differences between original and monitoring data.

Documentation may be placed directly on the report, in a ledger or notebook, and must indicate the nature of the problem and the corrective action taken.

5.3 CHECK SAMPLES AND SPECIAL STUDIES

Check samples may be issued by TSD periodically to identify potential intermarket differences between service points, and to track the capability of the national oil testing program in relation to the reference method. Special studies are designed to resolve differences in oil results either within or between markets.

a. Check Samples.

TSD will select bulk samples of sunflower seeds representing the range of oil values typically seen in the market and prepare multiple sets of representative portions. On an as-needed basis, a portion of each of these samples will be distributed to specified service points, FGIS field offices, and the TSD reference method lab to be tested for oil content. Participants must complete the oil testing within 7 working days of receipt of the samples and transmit the results to TSD.

TSD will tabulate, plot, and analyze the data and prepare a report of the average results of all locations compared to TSD. Plots and statistical analysis will be transmitted to all participants and supervising FGIS offices.

Field office and/or specified service point managers must initiate corrective action and follow-up when needed. The TSD will assist the field offices in resolving intermarket differences and investigate and take necessary action when excessive differences between the NMR oil and reference methods are indicated.

b. Special Studies or Collaboratives.

Special studies or collaboratives, conducted at the discretion of TSD, are designed to resolve differences in NMR oil results within or between markets. Because special studies are normally of an urgent nature, an expedient resolution of the problem is essential. Therefore, all participants must perform the requested tests and report the results to TSD within 5 working days.

5.4 INTERMARKET SAMPLE EXCHANGE

An inter-market sample exchange is used to isolate oil differences between inspection points. Oil testing laboratories will determine oil results on separate portions obtained from the same sample. Oil results are then compared to determine whether significant differences between locations exist. This procedure is particularly useful when there are sunflower seed shipments between two specified service points or an individual applicant is routinely receiving service from two service points.

There are no restrictions as to which offices may exchange samples. Specified service points are encouraged to exchange samples with other service points for the purpose of investigating and resolving inter-market inspection differences. A copy of the results of the exchange must be provided to TSD for review.

CHAPTER 6

OXFORD MQA 6005 & 7005

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6.1 INSTRUMENT SETUP

a. Setup Information.

Operators must read the user's manual and familiarize themselves with this instruction before operating the NMR instrument. The operation of the OXFORD MQA 6005 and MQA 7005 pulsed NMR instruments is controlled by a personal computer. After the instrument is powered on, a series of automatic internal tests are carried out to confirm proper instrument functionality. Any error messages should be referenced in the Operator's manual.

The magnet is maintained at a temperature of 40°C Degrees Centigrade. The unit must not be used for a period of 6 hours after being switched on to allow the magnet temperature to stabilize. After being switched-on the instrument should remain on.

b. Activation Of Undried Sample Oil Conversion Software.

Upon turning on the instrument, one of the icons displayed is "SHORTCUT TO SUNFLWR3." Using the "mouse" activate the icon and a conversion window will appear on the screen. Again using the "mouse," activate the "-" button located in the upper right corner of the window. This will shrink the window into a "MQA OIL CORRECTION" button located on the lower task bar of the screen. This button is activated by the operator each time the instrument is used to measure the oil content of undried samples.

This setup procedure for activating the undried sample oil conversion software must be repeated each time the power is turned on.

6.2 INITIAL INSTRUMENT CALIBRATION

The NMR instrument must be initially calibrated, using the GIPSA Sunflower Seed Standards (SSS). Once the instrument has been calibrated, the instrument is re-standardized (drift corrected) daily using the SSS or when the room temperature changes by + 1° C.

Follow the procedures listed below for calibrating instruments.

- a. Select "GIPSA Oil in Dried Seeds" method from the MQA Methods menu.
- b. Select Optimize (F8) from the Oxford MQA Methods main menu to activate the Optimize screen.
- c. Select Frequency and Pulse Times using the check boxes.
- d. Click on "Go" (green triangle) or press F5.

- e. At the prompt, insert the Oxford Test Sample (mineral oil tuning sample) and click on “Go” (green triangle) or press F5 to start the frequency optimization.
- f. At the prompt, click on “Go” (green triangle) or press F5 to start the pulse times optimization.
- g. If no error messages are displayed, click on “Accept” (green check) or press F2 to validate the optimization. Remove the test sample.

NOTE: If an error message is displayed, contact Oxford technical support.

- h. Select “GIPSA Oil in Dried Seeds” method from the MQA Methods menu.
- i. Select “Calibrate” or press F7 from the Oxford MQA Methods main menu to display the calibration editor.
- j. In the Data set panel then double click on the “CALMM/DD/YYYY” Data set.
- k. Rename the Data set by replacing the “MM/DD/YYYY” with the current date.

NOTE: The “CALMM/DD/YYYY” data set should be the only data set present on a new instrument. Additional calibration data sets can be added by clicking on the blue plus (+) button. Name new calibrations using the “CALMMDDYYYY” format.

- l. Click on “Accept” (green check) or press F2 to exit the Data set editor.
- m. Note the SSS temperature to 0.1° C.
- n. Click on “Go” (green triangle) or press F5 to start measurement for the first calibration standard.
- o. Insert the Low SSS calibration standard into the instrument.
- p. When the measurement is complete, remove the SSS from the magnet.
- q. Enter the SSS ID, SSS Mass and Reference Oil value from the SSS label. During this process the point appears on the calibration graph, lashing inside a gray dashed box.
- r. Check the “Calib. Standard” box and the “Setup Standard” box.
- s. Click “Accept” (green check) or press F2 to validate the acquisition of this point.

NOTE: If Reject (F3) is selected, the point will be lost. If the reference value for either component is left as zero then the software will ask for confirmation.

- t. Click on “Go” (green triangle) or press F5 to start measurement of the second calibration standard.
- u. Insert the High SSS calibration standard into the instrument and repeat steps p through s listed above.

NOTE: Once a minimum of two calibration standards has been measured, a solid calibration line is shown.

- v. Click “Accept” (green check) or press F2 to exit the calibration program.

6.3 DAILY INSTRUMENT RESTANDARDIZATION

- a. Daily Re-standardization.

When the instrument is first put into service, it must be calibrated following the procedures in section 6.2. Once the calibration has been established, the following procedures are used daily to optimize the instrument and correct for drift due to temperature changes.

- (1) From the main menu, select the “GIPSA Oil in Dried Seeds” method.

NOTE: If only one method is installed, it will be automatically selected.

- (2) Select “Optimize” (F8) from the Oxford MQA Methods main menu to activate the Optimize screen.
- (3) Select “Frequency”, “Pulse Times” and “Re-standardization” (check boxes).
- (4) Click on “Go” (green triangle) or press F5 and click “OK” to confirm that you wish to re-standardize.
- (5) Record the SSS temperature to 0.1° C on the Check Sample Log.
- (6) When prompted, insert the Oxford Test Sample (mineral oil tuning sample) and click on “Go” (green triangle) or press F5 to optimize the resonance frequency.
- (7) When the frequency optimization is complete, click on “Go” (green triangle) or press F5 again to optimize the pulse times.
- (8) When prompted, remove the Oxford Test Sample, insert the Low SSS, and click on “Go” (green triangle) or press F5.
- (9) When prompted, remove the Low SSS, insert the High SSS, and click on “Go” (green triangle) or press F5.
- (10) If no error messages are displayed, click on “Accept” (green check) or press F2, and remove the High SSS. If an error message is displayed, contact Oxford technical support.

b. Calibration Check Using the SSS.

Test the SSS as a market sample to check the NMR instrument accuracy after daily re-standardization or when the room temperature changes by + 1° C or after every 30-40 samples have been analyzed or hourly, whichever comes first. Maintain a record (electronic or written) of the calibration checks using the Calibration/Check Sample Log as a template.

- (1) From the Oxford MQA Methods menu, select “GIPSA Oil in Dried Seeds” method, then “Analyze” (F5) to display the routine analysis screen.
- (2) Click on “Go” (green triangle) or press F5 and insert the Low SSS.
- (3) Enter the SSS ID and mass.
- (4) When the analysis is complete remove the SSS from the magnet, record the reference value on the Daily Calibration Check Log, and click on “Accept” (green check) or press F2 to validate the result.
- (5) Click on “Go” (green triangle) or press F5 and insert the High SSS.
- (6) Enter the SSS ID and mass.
- (7) When the analysis is complete remove the SSS from the magnet, record the reference value on the Daily Calibration Check Log, and click on “Accept” (green check) or press F2 to validate the result.
- (8) Record the SSS temperature to 0.1° C on the Check Sample Log. Compare the current temperature reading to the temperature recorded at step a (5). Repeat the re-standardization and check sample test, if a change in temperature of + 1° C has occurred.
- (9) Calculate the difference between the SSS results obtained and the Reference Values (RV) and record this value on the NMR Check Sample Log. If the difference for either SSS exceeds + 0.3 retest the SSS. If the difference still exceeds + 0.3, re-standardize the instrument and re-test the SSS.

NOTE: If the values repeatedly exceed the tolerances, you may need to re-calibrate the instrument. Contact TSD for technical support.

6.4 ANALYZING MARKET SAMPLES

a. Testing Market Samples.

After the instrument has been re-standardized, analyze market samples as follows:

- (1) If you are already in analysis mode, go to step 2. If not, from the Oxford MQA Methods menu, select “GIPSA Oil in Dried Seeds” method, then “Analyze” (F5) to display the routine analysis screen.
- (2) Click on “Go” (green triangle) or press F5 and insert the sample into the magnet assembly.
- (3) Enter the sample identifier (ID) and mass while the sample is being analyzed.

NOTE: For users employing a Mettler balance and using the automatic sample weight transfer option only the sample identifier is entered.

The procedures required for using the automatic sample weight transfer option are as follows:

- (a) The MQA-6005/7005 must have the enable external balance activated.
- (b) Prior to making a set of method measurements remove the sample cup from the scale and tare. Observe that the lower weight display is approximately 0.000.
- (c) Place the empty sample cup on the scale and wait until the weight display shows the cup tare weight.
- (d) Fill the sample cup with the sample from the sample cell and wait until the weight display shows the sample weight in red.
- (e) Remove the sample cup from the scale, refill the sample cell, and measure the oil content of the sample using the MQA-6005/7005. When the sample cell is inserted into the magnetic module the weight will automatically be entered. Repeat steps (c) through (e) for additional samples.

NOTE: During analysis, interim results are returned after the mass has been entered. Those interim results appear in white. When the analysis is complete the final result appears in black. In the event of a data entry error, the mass value can still be re-entered at this stage.

- (f) For undried samples, press the “MQA OIL CORRECTION” button on the computer lower left display task bar and a conversion window will appear. Enter the MQA-6005 or MQA-7005 oil and GAC-2500-UGMA or Perten AM 5200-A moisture, and the percent oil at 10% moisture is automatically displayed.

Press the “Enter” button to store the data in a text file (c:\qt\reports\sunflwr.txt). After recording the oil at 10% moisture, press the upper right corner “-” button to make the conversion window disappear and to reset the conversion software.

NOTE: After testing a sample for moisture content with the GAC-2500-UGMA or Perten AM 5200-A instrument, 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 GAC-2500-UGMA or Perten AM 5200-A moisture result is not measured within 24 hours prior to the oil measurement, or the GAC-2100 moisture result is not between 4.5% and 16%, 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).

- (4) When the measurement is complete, click on “Accept” (green check) or press F2 to validate the result. To cancel the measurement, click on “Reject” (red X) or press F3.
- (5) 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). Once the sample has returned to room temperature repeat steps (b) to (d).

NOTE: Once the result has been accepted it is added to the report file and added to the trend graph. The trend plot can be viewed by clicking on the Trend Graph tab. The data is also sent to a temporary print file, which will print when a page of data has accumulated or when you exit the program.

b. 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.

CHAPTER 7

BRUKER MINISPEC 7.5

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7.1 INSTRUMENT SETUP

a. Setup Information.

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. The magnet is controlled at a temperature of 35.5 degrees Centigrade. The unit must not be used until two green lights are illuminated on the front of the magnet. This may take up to three hours.

- (1) Start the minispec software on the personal computer.
- (2) At the start of the day, select the “Daily Check” icon and run the auto-validation procedure.
- (3) Insert the tuning sample (100 grams of vegetable oil). After the tuning sample is inserted the instrument will perform a series of tests.
- (4) If no error message appears the instrument is ready for calibration.
- (5) If an error message appears, select “Update Settings” from the minispec menu.
- (6) Click “OK” to proceed to update all instrument settings.
- (7) After the instrument updates the settings repeat the Daily Check. If the instrument repeatedly fails the Daily Check, contact technical support at Bruker.

b. Calibration.

The NMR instrument must be calibrated, using SSS, before testing market samples and when room or sample temperature changes by + 0.5° C or more.

NOTE: To recalibrate while analyzing market samples, at the prompt to enter or transfer from the balance the next sample weight, press the <ESC> key or with the mouse click the <STOP> button at the bottom of the screen. This terminates the <MEASURE> loop. With the mouse, select the <CALIBRATE> icon at the bottom of the screen or press ALT + C.

The following steps describe the calibration process:

- (1) Highlight the most recent calibration in the quick select list and select duplicate application from the file menu.
- (2) Save a copy of the file using the following file name format (calMMDDYY) where “MM” is the month, “DD” is the day, and “YY” is the year.

- (3) Select "Open" from the file menu and open the file created in step (2) above.
- (4) Click on the "Calibrate" button.
- (5) Enter the calibration name (calMMDDYY) for the result box title and click "OK".
- (6) Enter the weight of the low value SSS and click "OK".
- (7) Enter the oil content of the low value SSS and click "OK".

NOTE: The weight and percent oil content of the seed contained in the SSS are recorded on the SSS label.

- (8) Insert the low value SSS into the sample compartment and click "OK".
- (9) A visual progress thermometer and digital countdown of the number of scans will appear in the bottom of the program window and will proceed to count down to 0. Only one measurement will be required.
- (10) When the analysis is complete, remove the SSS.
- (11) Repeat steps (6) - (10) for the high value SSS.
- (12) Click "Cancel" when prompted for data on sample 3.
- (13) The instrument will display the calibration statistics. Record the slope and intercept values on the calibration log.
- (14) Click the "Continue" button, then click "OK" to print and terminate the calibration process.
- (15) Record room temperature to 0.1° C on the calibration log.

c. Calibration Check.

Check the calibration by testing the SSS as market samples before beginning routine sample analysis.

NOTE: The minispec software allows the user to select from several sample data entry interfaces. The following instructions are based on using the “spreadsheet” interface for entering sample data. To configure the application to use the spreadsheet mode click on the “Configuration Table” icon (top of screen). If the “File Name” input box opens, select the configure application. Check the “Database Table Mode” in the option table.

- (1) Clear any previous data from the spreadsheet by highlighting the data and pressing the “Delete” key.
- (2) Enter the ID and weight of the Low Sunflower Seed Standard (SSS) into the appropriate columns of the spreadsheet.
- (3) Repeat step (2) for the High SSS.
- (4) Use the mouse to select the <Measure> button.
- (5) Insert the Low SSS and click “OK”.
- (6) When the measurement is complete (16 seconds), remove the Low SSS.
- (7) Insert the High SSS and click “OK”.
- (8) When the analysis is complete remove the High SSS.
- (9) Record the oil values on the Calibration/Check Sample Log and calculate the difference between the reported and known values for the SSS. If the difference is greater than 0.3%, repeat the analysis. If the repeat analysis result difference is still greater than 0.3%, recalibrate the instrument.

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 Check Sample Log in Appendix A as a template.

7.2 SAMPLE ANALYSIS

Once the instrument has been properly validated (with the Daily Check procedure) and the instrument is calibrated, begin analyzing market samples.

NOTE: The minispec software allows the user to select from several sample data entry interfaces. The following instructions are based on using the “spreadsheet” interface for entering sample data. To configure the application to use the spreadsheet mode click on the “Configuration Table” icon (top of screen). If the “File Name” input box opens, select the configure application. Check the “Database Table Mode” in the option table.

a. Analyzing Samples.

- (1) Clear any previous data from the spreadsheet by highlighting the data and pressing the “Delete” key.
- (2) Enter the ID’s and weights of all of the samples to be tested into the appropriate columns of the spreadsheet.

NOTE: The instrument will also accept weight directly from an electronic balance connected to the RS-232 port. If a balance is connected, place an empty container on the balance and zero the balance. Pour the dry sample into the tin and press the <PRINT> key on the balance. The sample weight will be transferred automatically as soon as the weight stabilizes.

- (3) Use the mouse to select the “Measure” button.
- (4) Transfer the first sample to a 150-ml NMR sample tube, insert into the magnet, and click “OK”.
- (5) When the measurement is complete, remove the sample from the magnet.
- (6) Repeat steps (4) and (5) for the remaining samples.

NOTE: The data will automatically be save to a Microsoft Access database. The program will also print the date and time, sample ID, sample weight, and the NMR percent oil to an attached printer.

b. 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.

CHAPTER 8

REVISION HISTORY

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Change No: 3	October 1, 2009	2
Change No: 2	May 12, 2003	2

Change No: 3 October 1, 2009

This update superseded the FGIS Nuclear Magnetic Resonance (NMR) Handbook dated May 12, 2003. This handbook is updated to reflect organizational changes in the monitoring system, procedural changes, instrument changes, and a change in certification to compliment CRT.

Change No: 2 May 12, 2003

The Nuclear Magnetic Resonance (NMR) Handbook was revised to include procedures for testing sunflower seed oil with pulsed wave NMR instruments (Oxford MQA 6005 and MQA 7005, Resonance MARAN Ultra, and Bruker Minispec 7.5). Additionally, the instructions for selecting monitoring samples have been revised to reflect current selection criteria.