

CHAPTER 6

INSTRUMENT SETUP AND SAMPLE ANALYSIS - OXFORD MQA 6005 AND
MQA 7005 PULSED NMR ANALYZERS

<u>Section Number</u>	<u>Section</u>	<u>Page Number</u>
6.1	INSTRUMENT SETUP	6-1
6.2	INITIAL INSTRUMENT CALIBRATION.....	6-1
6.3	DAILY RESTANDARDIZATION	6-3
6.4	ANALYZING MARKET SAMPLES	6-5

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 $\pm 1^{\circ}$ 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, flashing 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 $\pm 1^{\circ}$ 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 $\pm 1^{\circ}$ 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 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-2100 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-2100 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.