

CHAPTER 6

INSTRUMENT SETUP AND SAMPLE ANALYSIS  
OXFORD 4000 CONTINUOUS WAVE NMR ANALYZER

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## 6.1 INSTRUMENT SETUP

The operation of the OXFORD 4000 NMR analyzer is controlled by a microprocessor and instructions will appear on the display at each stage of the operation.

Operators must read the user's manual and familiarize themselves with this instruction before operating the NMR instrument.

**NOTE: The OXFORD/FGIS EPROM program, which allows for a separate number of repeat parameters for calibration and market sample analysis, must be installed in order to use the instrument for official oil determinations.**

The oil values assigned to the SSS used in a two-point calibration are stated on a 10 percent moisture basis. As a result, the oil values reported by the instrument are also stated on a 10 percent moisture basis.

### a. Instrument Setup.

After the NMR instrument power is turned on, it automatically carries out diagnostic tests on the instrument hardware. Allow the instrument to warm up for at least 2 hours. Once the NMR instrument is turned on, it may be left on. However, if the NMR instrument is turned off, a 2 hour warmup is required before calibration and/or sample analysis.

If an error message is displayed, consult the user's manual.

All instructions must be answered by entering the requested information via the keypad and pressing the "ENTER" key. Press the "YES" or "NO" keys to answer questions.

- (1) ENTER YEAR.
- (2) ENTER MONTH.
- (3) ENTER DAY.
- (4) ENTER HOUR.
- (5) ENTER MINUTE.
- (6) INSERT TUNING SAMPLE. Insert the tuning sample (TS). The instrument will automatically perform the tuning function.

b. Programmed Mode Setup.

To prepare the instrument for a new programmed mode, the operator must key in and enter the secret code number for the instrument when the message "ENTER MODE NUMBER" appears on the display.

The instrument will print "PROGRAM MODE" and "DATE and TIME." Follow the instruction as it appears on the display and enter the requested information.

The following steps describe how to setup a programmed mode.

- (1) **ENTER R.F. LEVEL.** Enter the FGIS approved R.F. level of **225**. Press the "**ENTER**" key.
- (2) **ENTER A.F. GAIN.** Enter the FGIS approved A.F. gain of **500**. Press the "**ENTER**" key.
- (3) **ENTER GATE WIDTH.** Enter the FGIS approved width of **1.5 gauss**. Press the "**ENTER**" key.
- (4) **TEMPERATURE CONTROL REQUIRED?** Temperature control is not required. The NMR instrument will be re-calibrated before proceeding with NMR measurements if the sample or room temperature fluctuates more than "0.5EC. Press the "**NO**" key.
- (5) **MASS INPUT REQUIRED?** The dry sample weight is necessary to calculate the signal per gram ratio. Press the "**ENTER**" key.
- (6) **TWO POINT CALIBRATION?** Two SSS with known oil and dry sample weights are required to determine the calibration slope. Press the "**YES**" key.
- (7) **ENTER PERCENT CONCEN FOR REF XXA.** Enter the oil percent for the low value SSS. Press the "**ENTER**" key.
- (8) **ENTER PERCENT CONCEN FOR REF XXB.** Enter the oil percent for the high value SSS. Press the "**ENTER**" key.

**NOTE: Mode numbers are sequentially assigned by the program. XX is the number assigned to this mode.**

- (9) **ENTER ANALYSIS TIME.** Enter the FGIS approved analysis time of **32** seconds. Press the "**ENTER**" key.

- (10) **ENTER ANALYSIS REPEATS.** Enter **1** for the number of measurements for official NMR oil testing (initial analysis plus one repeat). Press the "**ENTER**" key.
- (11) **ENTER CALIBRATION TIME.** Enter the FGIS approved calibration time of **128** seconds. Press the "**ENTER**" key.
- (12) **ENTER CALIBRATION REPEATS.** Enter **2** for the number of measurements for instrument calibration (initial analysis plus two repeats). Press the "**ENTER**" key. The printer will now list the selected parameters.
- (13) **USE MODE?** Press the "**YES**" key to enter the analysis mode and try the selected parameters before permanently storing it. If the "**NO**" key is pressed then execution continues at the next question.
- (14) **STORE MODE DETAILS?** Press the "**YES**" key to store the programmed mode parameters in the permanent memory. Programs stored in the permanent memory can be recalled and used at a future time by entering the designated code number. The operation will continue at step 16. To discard the program, press the "**NO**" key, and the operation will continue at step 15.
- (15) **ARE YOU SURE?** Press the "**NO**" key to return to step 13 above or press the "**YES**" key to discard the program.
- (16) This mode is now programmed and stored. Record the programmed mode number and parameters in the instrument record book.
- (17) **ENTER MODE NUMBER.** Enter a programmed mode number to proceed to calibration and analysis.

c. Tuning the NMR Instrument.

The instrument will automatically perform the tuning function during setup and calibration. The operator may tune the NMR instrument at any time a signal separation appears on the oscilloscope by performing the following:

- (1) Insert the TS in the magnet coil assembly.
- (2) Press the tuning button located above the on/off switch on the back of the instrument.

- (3) If necessary, repeat step 2 above until no signal separation appears on the oscilloscope.

## 6.2 INSTRUMENT CALIBRATION

### a. Daily Calibration.

The NMR instrument must be calibrated using the SSS before testing market samples, when room or sample temperatures change by "0.5EC or more, and when the result for SSCS exceeds the established tolerance.

When using a mode for the first time, the instrument will automatically initiate the calibration function.

When changing modes to a previously calibrated mode, press "YES" to enter the calibration function when prompted to "RE-STANDARDIZE."

To re-calibrate while analyzing market samples, enter "0" for the sample ID and press "ENTER."

The following steps describe the calibration process:

- (1) **RECORD ROOM TEMPERATURE.** Record to 0.1EC on the Daily Calibration/Check Sample Log.
- (2) **INSERT TUNING SAMPLE.**
- (3) **ENTER REF XXA MASS.** Enter the weight of the low value SSS. The weight of the sample contained in the SSS is recorded on the SSS label.
- (4) **REF XXA INSERTED?** Insert the low value SSS into the magnet coil assembly. Press the "YES" key.
- (5) **MEASUREMENT IN PROGRESS.** The measurement time, in seconds, will appear in the top of the display and will proceed to count down to zero. The measurements will be repeated as specified in the programmed mode.
- (6) **ENTER REF XXB MASS.** Enter the weight of the high value SSS. The weight of the sample contained in the SSS is recorded on the SSS label.
- (7) **REF XXB INSERTED?** Insert the high value SSS into the magnet coil assembly. Press the "YES" key.

- (8) **MEASUREMENT IN PROGRESS.** The measurement time in seconds will appear in the top of the display and will proceed to count down to zero. The measurements will be repeated as specified in the programmed mode.
- (9) After taking the last NMR reading, check the room temperature. Disregard and repeat the calibration if a change in room temperature of greater than "0.5EC has occurred.
- (10) Examine the NMR responses. If the difference between the high and low NMR responses is 0.70 NMR units or less, test the SSS as a market sample. If the difference between the high and low NMR responses is greater than 0.70 NMR units, disregard the readings and repeat calibration. If the problem persists, contact the BAR.

b. Accuracy 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 "0.5 ° C or after every 15-20 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) Enter the SSS ID and seed weight. The instrument will perform two measurements and determine the oil content.
- (2) When the analysis is complete examine the NMR responses. If the difference between the NMR responses is 0.60 NMR units or less, accept the results. Otherwise, disregard and repeat the measurements. Record the oil value on the Calibration/Check Sample Log.
- (3) Record the SSS temperature to 0.1 ° C on the Calibration/Check Sample Log. Compare the current temperature reading to the temperature recorded at step 5.2 a.(1). Repeat the calibration and check sample test, if a change in temperature of "0.5 ° C has occurred.
- (4) Calculate the difference between the SSS results obtained and the Reference Values (RV) and record this value on the Calibration/Check Sample Log. If the difference for either SSS exceeds "0.3 retest the SSS. If the difference still exceeds "0.3, re-calibrate the instrument and re-test the SSS.

## 6.3 ANALYZING MARKET SAMPLES

### a. Testing Market Samples.

Once the instrument has been properly checked and calibrated, begin analyzing market samples as follows:

- (1) **USE MODE XX?** Press the "YES" key to continue with the current mode or "NO" to use a different programmed mode. If you change modes, you will be required to calibrate the new mode before using it.
- (2) **ENTER SAMPLE NUMBER.** Key in the sample identification. Press the "ENTER" key.
- (3) **ENTER SAMPLE XXXXX MASS.** Key in the sample weight (dry weight). Press the "ENTER" key.

**NOTE: The instrument will also accept sample weight directly from an electronic balance connected to the RS-232 port. If a balance is connected, place an empty moisture tin on the balance and zero the balance. Pour the dry sample into the bin. After the balance reading reaches a constant value, press the "YES" key on the NMR instrument to enter the sample weight.**

- (4) **SAMPLE XXXXX INSERTED?** Transfer the dry sample to a 150 ml NMR sample tube and insert it into the magnet coil assembly. Press the "YES" key.
- (5) **EXAMINE NMR RESPONSES.**
  - (a) The instrument will print the sample ID, date, time, NMR response for each of the two measurements, mean NMR response, signal mass ratio, and NMR oil percent on a 10 percent moisture basis.
  - (b) If the difference between the two NMR responses is 0.60 units or less, accept the results.
  - (c) If the difference exceeds 0.60 units, disregard the results and repeat the analysis of the sample. If the problem persists, re-calibrate the NMR instrument using the SSS.
  - (d) If the range of measurements between NMR responses is continually above 0.60 units, contact TSD.

b. Reporting Results.

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

## 6.4 INSTRUMENT DIAGNOSTIC

If an NMR instrument is functioning properly, less than 5.0 percent of the results should be rejected because the difference between the high and low NMR responses exceeds 0.60 or 0.70 NMR unit tolerance for two and three-cycle analyses, respectively. A significantly higher rate of rejected analyses indicates a problem with the NMR instrument.

a. Erratic NMR Response.

The most common cause of erratic responses is a poor connection between the coil and the console. If tightening the connector does not eliminate the problem, disconnect the cable, clean the contacts with a clean pencil eraser, and reconnect. If the problem still persists, contact Oxford technical support.

b. Distorted or Weak Oscilloscope Signal.

A distorted or flickering oscilloscope signal is generally due to a poor connection between the coil and the console. Check the connector at the console and tighten if necessary. If tightening the connector does not eliminate the problem, disconnect the cable, clean the contacts with a clean pencil eraser, and reconnect. If the problem still persists, contact Oxford technical support.

A weak or dim oscilloscope signal may indicate that the cathode ray tube is near failure.

When the instrument is left on but not in use, the brightness control should be turned to the lowest level to extend the life of the cathode ray tube.

c. Consistently High or Low Results.

Consistently high results are generally due to incomplete drying of the sample. If incompletely dried seeds are measured, the NMR signal will include the total amount of water and oil present and will give an inflated indication of oil content.

High results can also occur because of differences in temperature between the SSS at the time of calibration and the market sample. If the market samples are significantly cooler than the temperature of the SSS at the time of calibration, the indicated oil content will be inflated.

Consistently low results are commonly caused by not allowing the market samples to cool to room temperature. If the market samples are significantly warmer than the temperature of the SSS at the time of calibration, the predicted oil content will be lower than the actual oil content.

Low results can also be caused by contamination by ferrous materials. Small amounts of ferrous materials present in samples will reduce the signal extensively.