CHAPTER 7

INSTRUMENT SETUP AND SAMPLE ANALYSIS - BRUKER MINISPEC 7.5 PULSED NMR ANALYZER

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 $\pm$ 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.