AVIII 400 NMR User Guide

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PREFACE

Purpose of This Document

This user guide aims to familiarize you with data acquisition using TopSpin software on the AVIII 400 NMR Spectrometer. The document does not cover all the basic information and details of the application.

Intended Audience

This document is intended for users of the AVIII 400 NMR spectrometer who are familiar with the basic principles of the spectrometer operation. It will enable you to understand the details of basic operations. This document is *not* intended to replace any form of official Bruker user guides or related Bruker documents.

1 Introduction

The AVIII400 NMR spectrometer is equipped with a Bruker BBFO probe. This instrument is used for analyzing time and air sensitive samples. It is also suitable for the study of reaction kinetics of chemical reactions and for a variable temperature (VT) NMR experiment. Prior user training is required to operate the spectrometer. Additional training is available for the VT experiment. The web reservation is required to use the AVIII 400 NMR spectrometer. The spectrometer is running on a Bruker TopSpin software.

1.1 Safety

Iron and other ferro magnetic objects must **NOT** be brought into the vicinity of the magnets. The strong magnetic fields may erase the information of credit card, student ID card, and other magnetic media. No admission is allowed for persons with pacemakers and other metallic implants.

1.2 NMR Sample Preparation

- Always use clean and dry sample tubes
- Use medium to high quality sample tubes
- Filter sample solution if particles are present
- Keep the sample volume approximately 0.5 – 0.6 mL.
- Wipe the sample tube clean before inserting into magnet
- The sample tube should sit tightly inside the spinner
- Use a sample depth gauge to adjust position of sample spinner

1.3 NMR Probe

The AVIII 400 MHz NMR spectrometer is equipped with a BBFO probe. The BBFO probe has two radio frequency channels (^{1}H and X-nucleus) with built-in capability of automatic tuning and match (ATM). The high frequency channel is tuned for ^{1}H observe and decoupling. The observe channel (X) covers a frequency range from ^{109}Ag to ^{19}F. The probe temperature range is from -150 to 150 °C. The commonly used acquisition files for this probe are prefixed with "*bbfo*"

1.4 Login

Once NMR training has been completed, username and password will be provided by the NMR spectroscopist. In general, the username is the same as the university email address, therefore, one must first obtain a university email address before an NMR account is created.

1.5 Workstation

The NMR workstation OS is a Linux system. Unlike Windows, please do not use special characters such as *, space, $, %, etc. in the filenames. To separate concepts, one may consider using a dash or an underscore.

2 TopSpin NMR Software

2.1 TopSpin Interface

To start the TopSpin (TS) NMR software, click on the Topspin icon on the desktop. A TopSpin interface (Figure 1), the lock display window (Figure 2), and the BSMS panel (Figure 3) will appear.

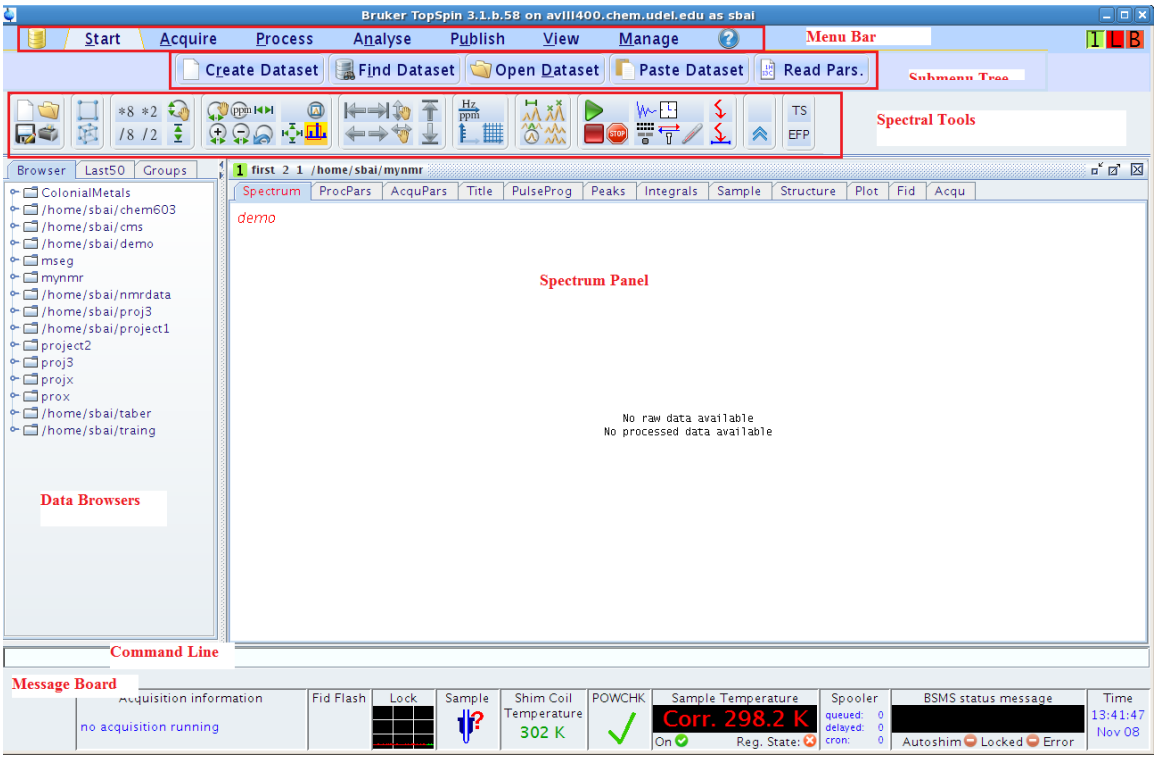

Figure 1: TopSpin Interface

The TopSpin interface contains several portions: the Menu Bar (on the top) with the buttons of **Start, Acquire, Process** etc. The Submenu Tree (the second row) shows special functions such as **Create Dataset, Find Dataset**, etc., corresponding to the **Start,** is located below the Menu Bar. Below the submenu, there are two rows of commonly used data manipulating tools. The left panel provides a number of ways to browse for data. The right panel (Spectrum panel) contains **Spectrum** tab, **ProcPars** (Processing parameter) tab, **AcquPars** (acquisition parameter) tab etc. Below the Spectrum panel, a command line is located. A command line allows user to enter a command manually. Below the command line, a message board provides real-time experiment information. The bottom section of the interface contains a number of status indicators, such as *Lock*, *Sample*, *Sample Temperature* etc.

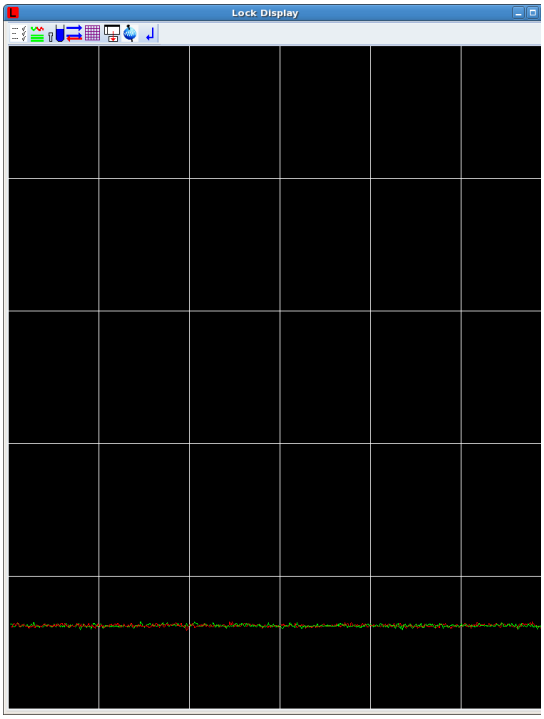

Figure 2: Lock Display

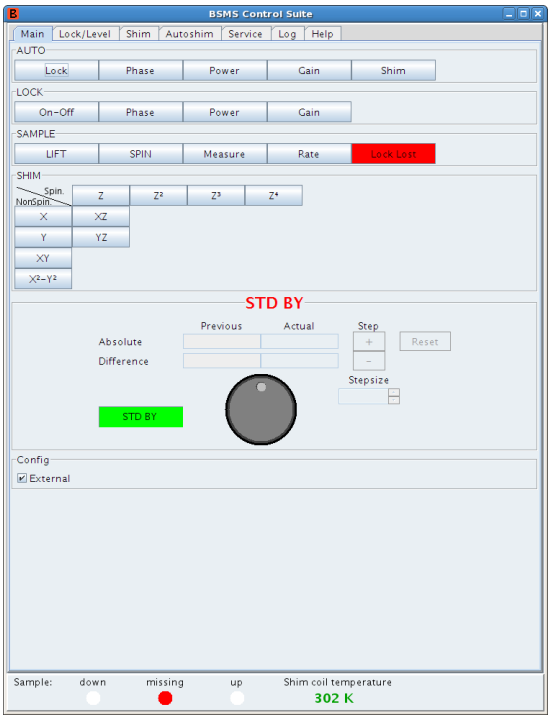

Figure 3: BSMS Panel

The lock display window shows ^{2}H signal profile of deuterium frequency lock and the BSMS panel allows user to execute the sample related commands, such as sample insertion and ejection, manual shimming, locking, lock gain adjusting, etc.

2.2 Recommended Preferences

When TopSpin is started for the first time, the following preferences are recommended:

1. In the command line, type *edc* and a window called "New…" opens. Fill in the Name field with a text such as "first". Assign both EXPNO and PROCNO to a numeric number such as "1". In the Experiment field, select a proton experiment acquisition file, such as "*bbfo1hstd*". The data directory field (DIR) specifies data storage location (/home/username/data_directory). As an example shown in Figure 4, the username is *sbai* and data_directory is *proj1*. *Note*: the directory path /home/username is fixed sequence as user's home directory, which cannot be changed. The data_directory, however, can be set by user. When all fields are filled in, click on **OK** to proceed.

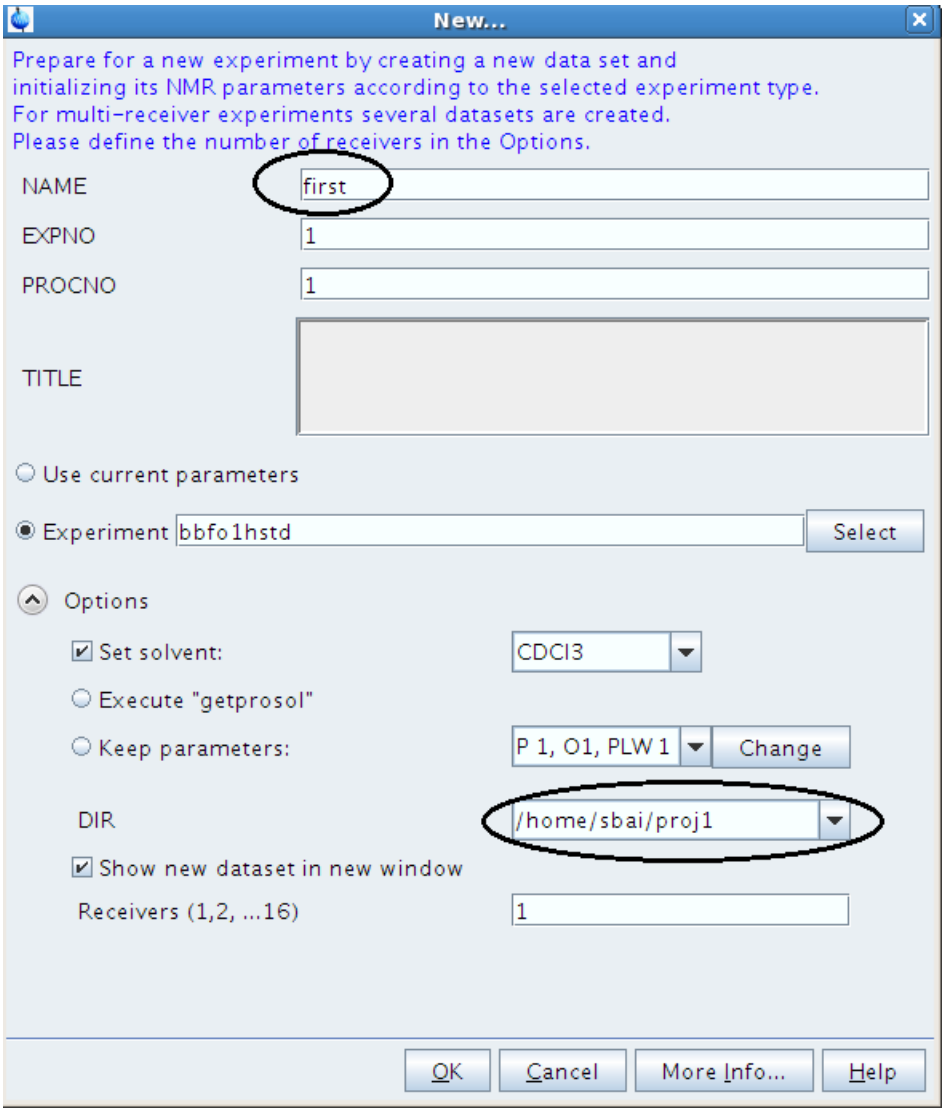

Figure 4: Fill in the "New…" Window for First Time

2. Type *set* in the command line, the User Preference window opens. Make sure "*Auto-open last used dataset when restarting TopSpin*" is checked as shown in Figure 5

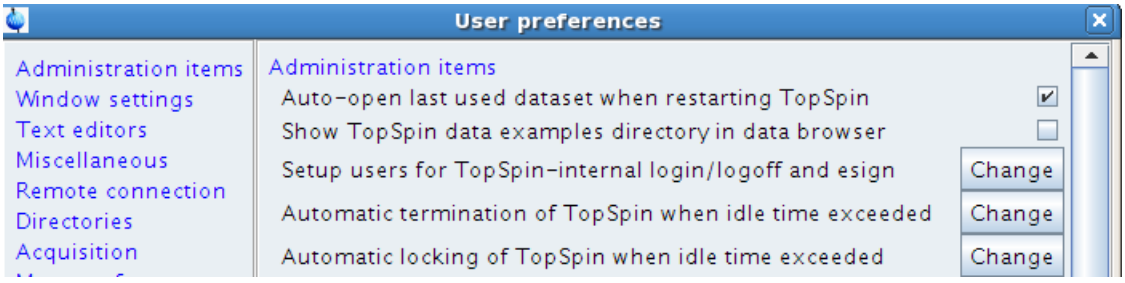

Figure 5: User Preferences – Part I

3. Also in the User Preference window, under **Acquisition**, make sure four items ("*Show "ased*" …; "*Overwrite…*"; "*Display Digital…*"; and "*Auto Open Acquisition…*" are checked as shown in Figure 6.

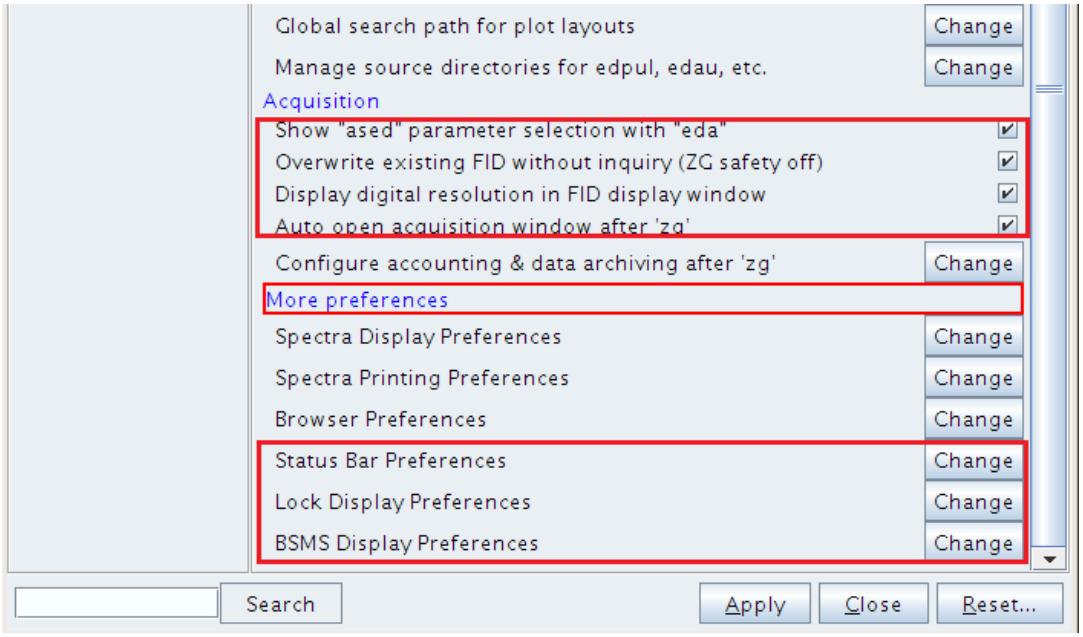

Figure 6: User Preferences – Part II

4. Then click on **Change** associated with the **Status Bar Preferences**, a window opens as shown in Figure 7. Make sure the items in Figure 7 are checked. Click on **Apply** then on **Back**.

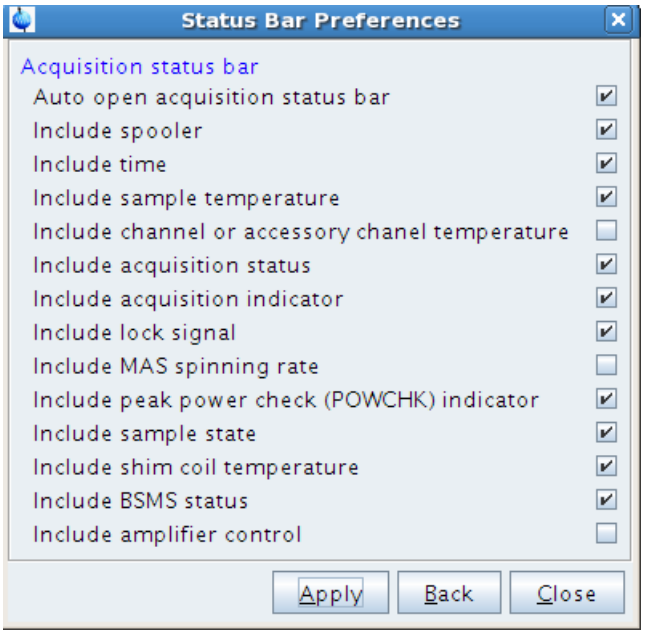

Figure 7: Status Bar Preferences

5. Then click on **Change** associated with **Lock Display Preferences**. In the pop-up window (Figure 8), make sure "*Auto open LOCK display*" is checked. Click on **Apply** and then on **Back**.

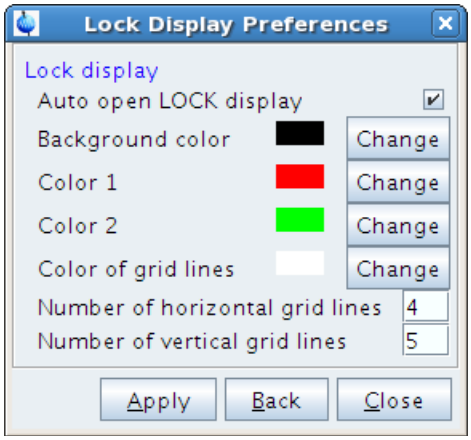

Figure 8: Lock Display Preferences

6. Finally click on **Change** button of BSMS Display Preferences, check "auto open BSMS display" and "External BSMS display". Click on **Apply** and then on **Back**.

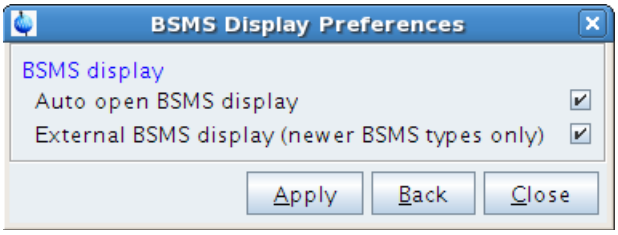

Figure 9: BSMS Display Preferences

7. Back to the User Preference window, click on **Apply** (*c.f.* Figure 6) and close the window.

8. Pointing the cursor to the bottom portion of TopSpin Interface, right click the mouse, and select "*Show Status Bar*". The status bar, including *Acquisition Information, Lock, Sample, Sample temperature* etc. as shown in Figure 10, now open.

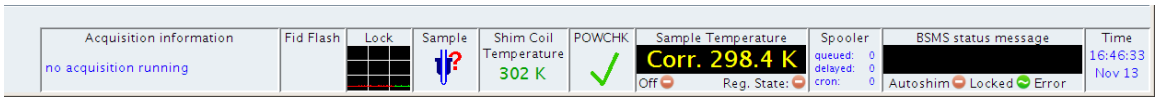

Figure 10: Status Bar

3 Step-by-step Guide for ^{1}H NMR

Follow these steps to obtain a ^{1}H NMR spectrum:

1. Click on **Start** in the menu bar. Then click on **Create Dataset** button in its submenu bar. A "**New…**" window opens up.

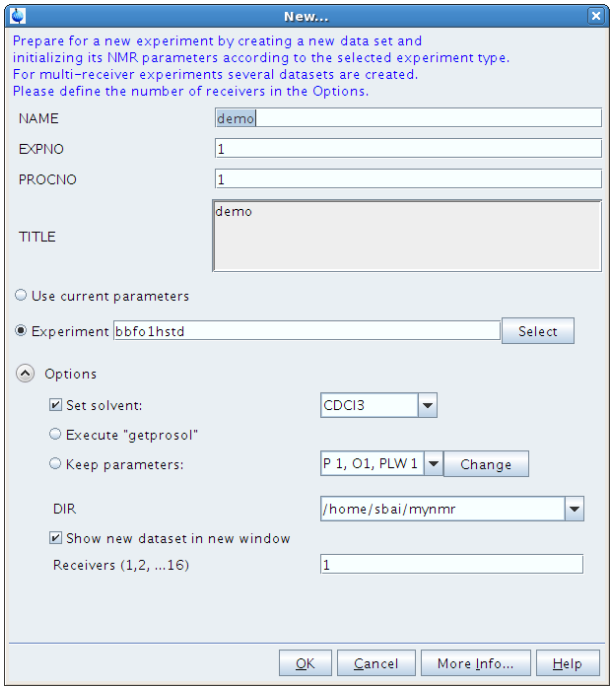

Figure 11: The "New dataset" Window

2. Fill in the fields of NAME, EXPNO, PROCNO, and TITLE. *Note*: *that only numerical numbers are allowed in the fields of EXPNO and PROCNO*.

3. Click on **Select** associated with the **Experiment** field, a "*rpar*" window opens:

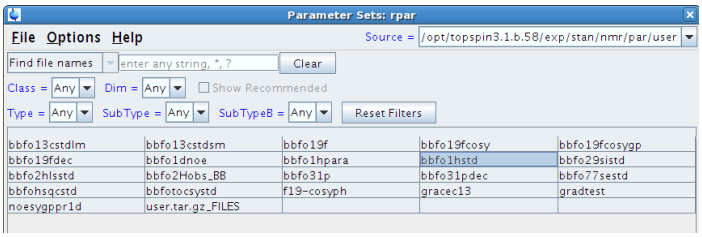

Figure 12: The "rpar…" Window

4. Highlight the acquisition file "*bbfo1hstd*" and then click on **Set Selected Item in Editor**.

5. Go back to "**New…**" window, select appropriate solvent from the '*solvent*' pull-down menu, and make sure '**getprosol**' is checked. Click on **OK** button to finish dataset creation.

6. Click on **Acquire** in the menu bar, select **Turn on Sample Lift Air** by clicking on the green triangle sign of the **Sample** button in the submenu tree. This will turn on the lifting air for sample insertion. Alternatively, type *ej* in the command line to turn on the lifting air. When lifting air is on, place the NMR sample with spinner on the top of magnet. Click on **Sample** in the submenu again and select **Turn off Sample Lift Air**. Or simply type "*ij*" in the command line. The sample will be lowered into the probe. *Note*: *in the bottom section of the TS interface,* ***Sample*** *icon is changed as indicated in* Figure 14.

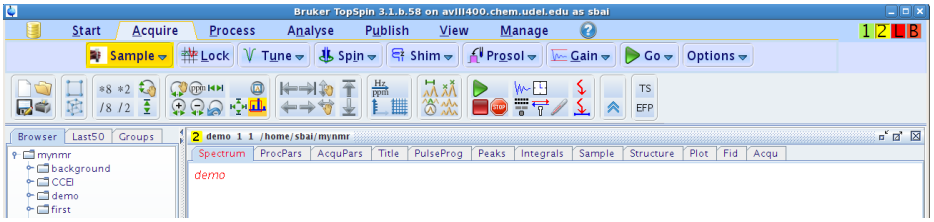

Figure 13: Sample Insertion

Figure 14: Sample Icon in Status Bar

7. Click on **Lock** in the **Acquire** submenu. A **Solvent Table** will appear. Select appropriate solvent, and the click on the "**OK**" button.

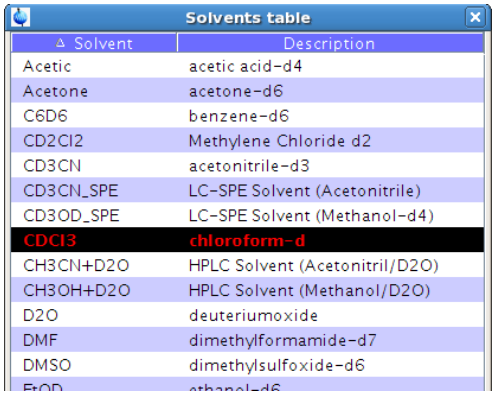

Figure 15: Solvent Table

8. Locking signal is then changed as indicated in Figure 16.

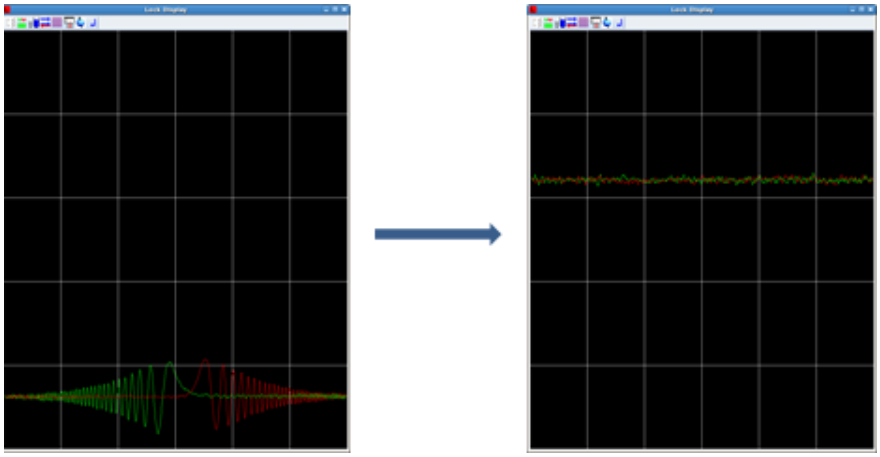

Figure 16: Lock Display Before and After Lock

9. Click on **Tune** in the **Acquire** submenu tree to perform a probe automatic tuning and matching. Or type *atma* in the command line. When the probe tuning starts, a tuning curve (Figure 17) appears and soon disappears in **Acqu** panel.

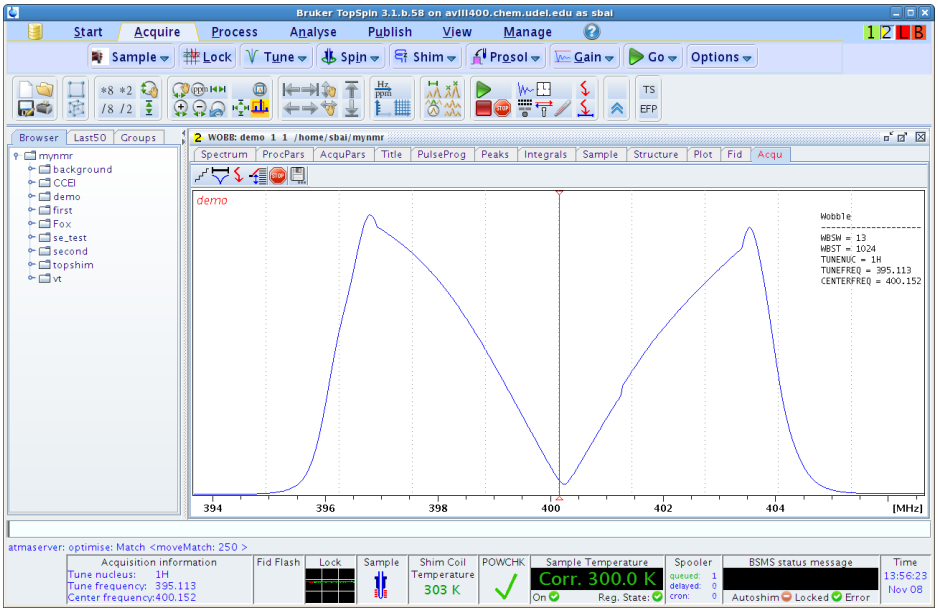

Figure 17: Probe Tuning Curve

10. After the probe tuning, click on **Shim** (or type *topshim* in the command line) to do an automatic shimming which may take 30 seconds or longer.

11. Click on **Gain** (or type *rga* in the command line) to automatically adjust receiver gain.

12. When receiver gain adjustment is finished, click on **Go** (or type *zg* in command line) to acquire proton NMR data. *Note: the status bar shows the progress of data acquisition*.

13. When data acquisition is in progress, the **Acqu** Tab of the spectrum panel is activated and shows the FID data or spectrum data depending on which button is depressed as shown in Figure 18.

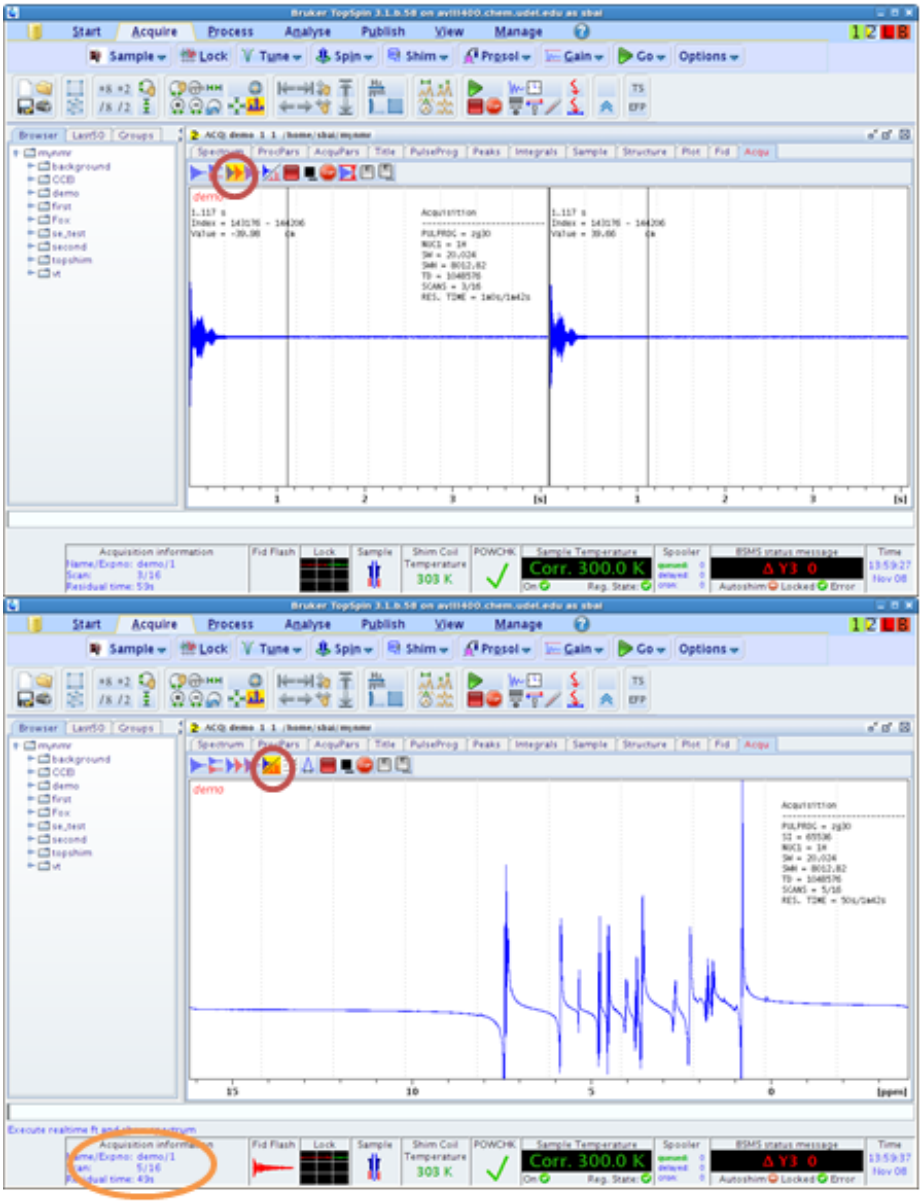

Figure 18: Data Acquisition in Progress

14. After data acquisition is finished, click on **Spectrum** tab in the spectrum panel. A message of "*1D raw data available and no processed data available*" is shown.

Click on **Process** in the menu bar followed by clicking on **Proc. Spectrum** in **Process** submenu tree. A processed spectrum is shown in **Spectrum** tab.

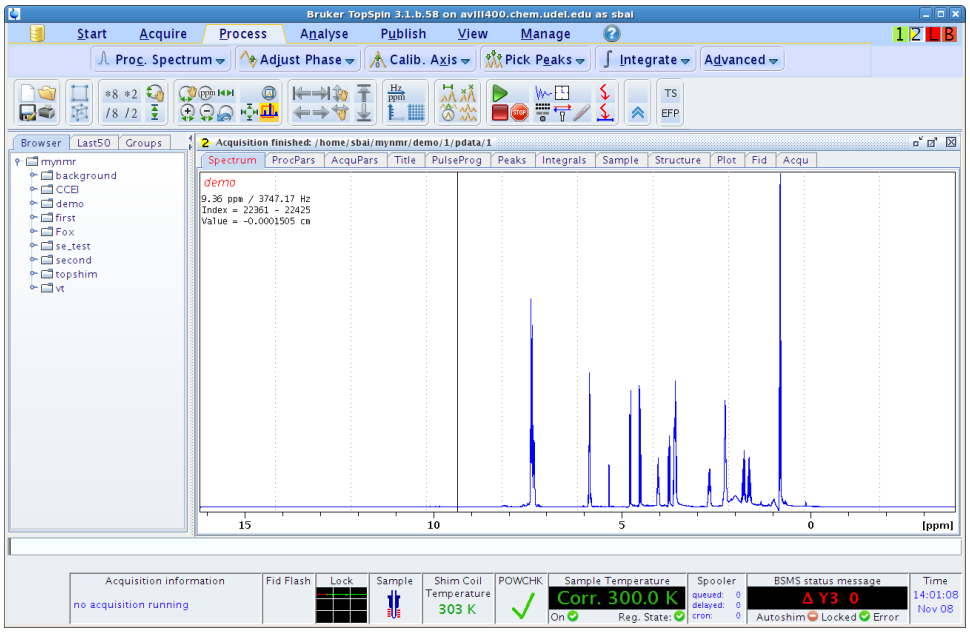

Figure 19: Processed 1H NMR spectrum

15. Use the left button of the mouse to click and drag a region that covers all resonances, and then release the button. The spectrum will be expanded as defined by vertical cursors.

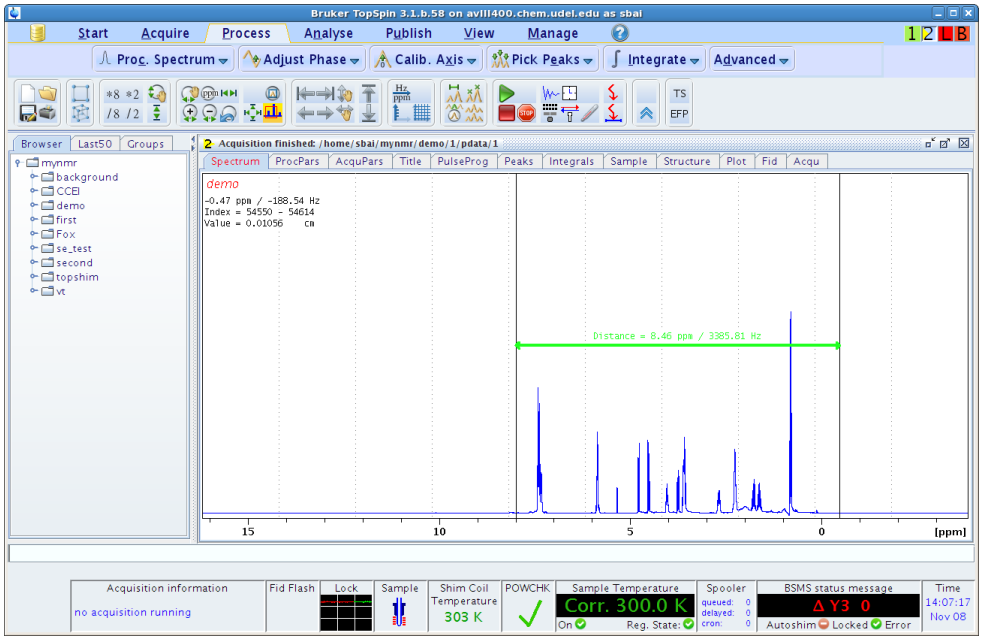

Figure 20: Click and Drag a Spectral Region

16. Click **Peak Picking** in **Process** submenu tree, and make sure the selection icon (circled in Figure 21) in the **Peak Picking** submenu is highlighted. Then click and drag a green box, which defines the minimum and the maximum limits of peak picking, as shown in Figure 21.

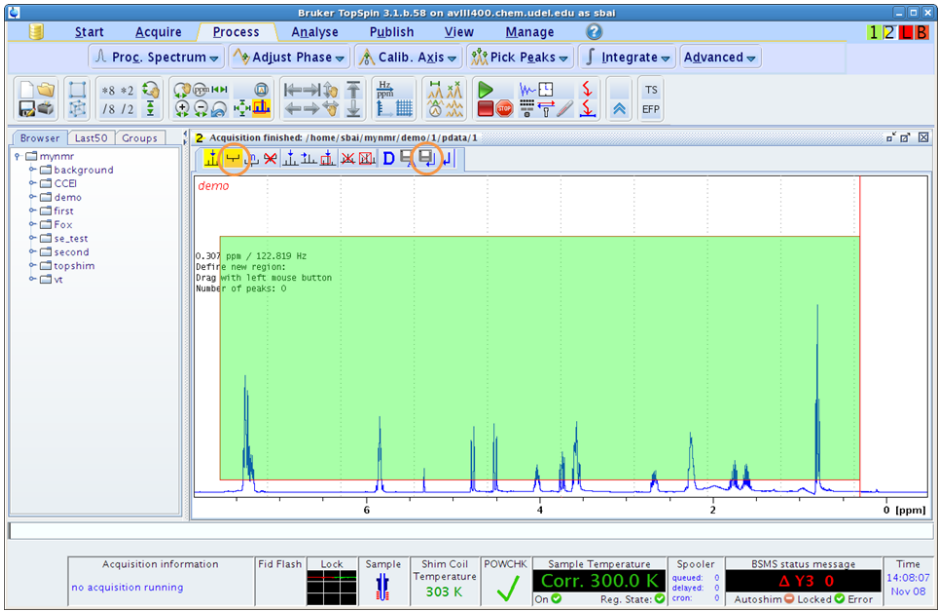

Figure 21: Click and Drag to Define a Region of Peak Picking

17. Click on **Save and Return** as circled in Figure 21 to save all peak pickings. A spectrum with peak pickings is shown in Figure 22.

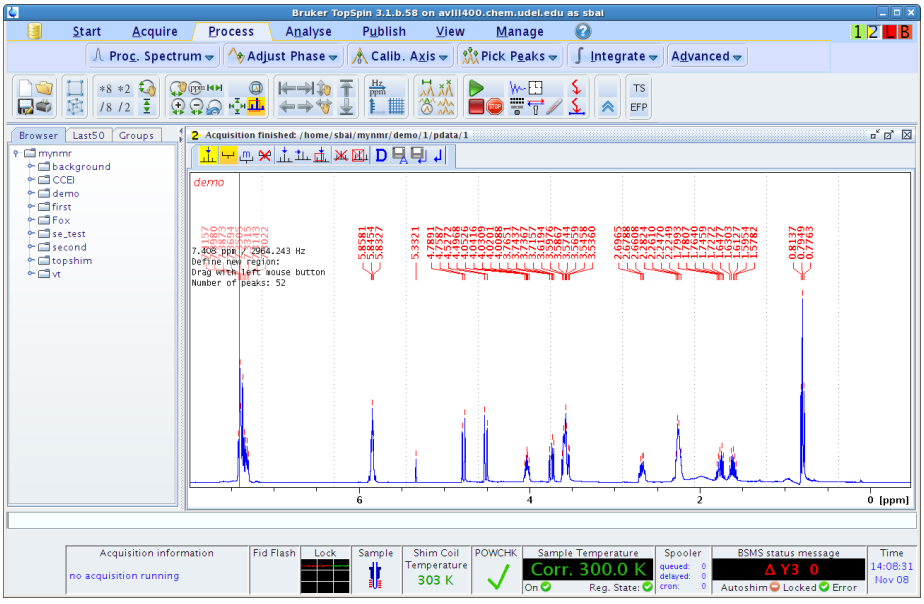

Figure 22: Spectrum with Peak Pickings

18. To integrate spectrum, click on **Integrate** in the submenu tree of **Process**. Make sure the selection tool is highlighted (as circled in Figure 23), then click and drag an integral range around a peak to be integrated. Continue on the rest of the resonances.

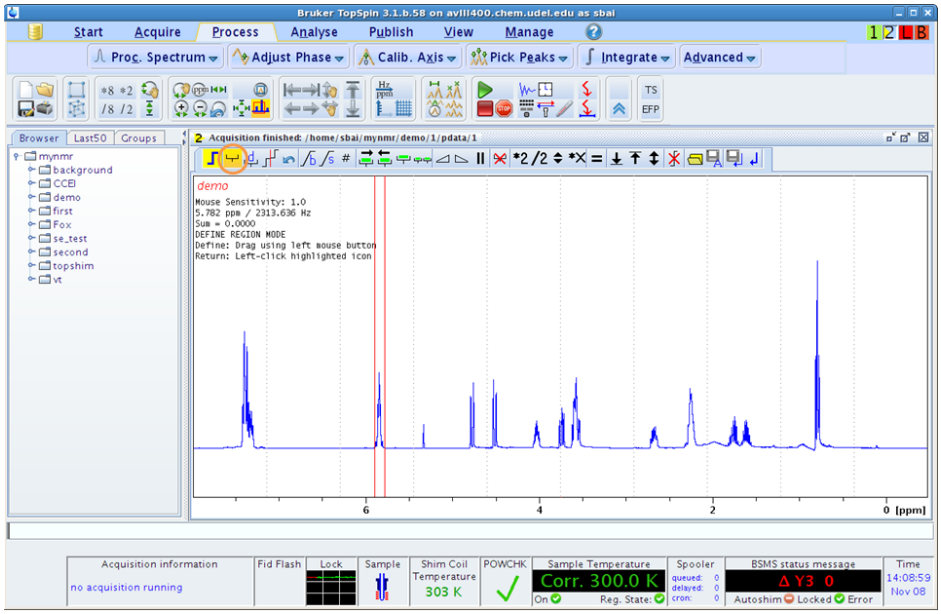

Figure 23: Spectrum Integration

19. To calibrate integral, right click over the integral on the screen and select **Calibrate**, fill in appropriate value in the pop-up box as shown in Figure 24. Finally click on '**Save and Return'** to save the integrations and return to spectrum panel.

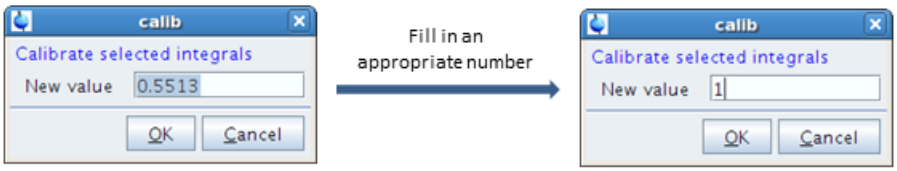

Figure 24: Calibration of Integral

20. The final processed ^{1}H spectrum is shown in Figure 25.

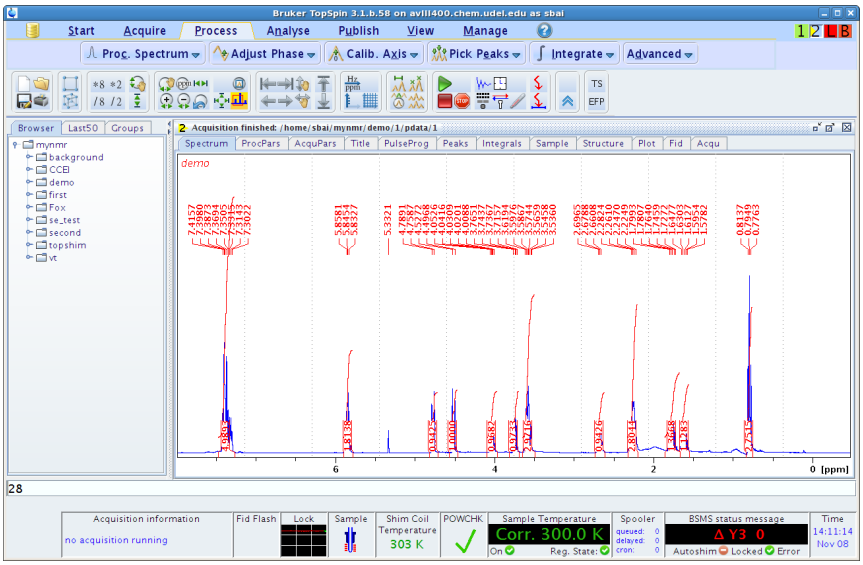

Figure 25: Processed ^{1}H Spectrum

21. Finally, to print out the spectrum, click on **Publish** in the menu bar, and then click on **Print** in the submenu, as indicated as the red circled tool buttons below.

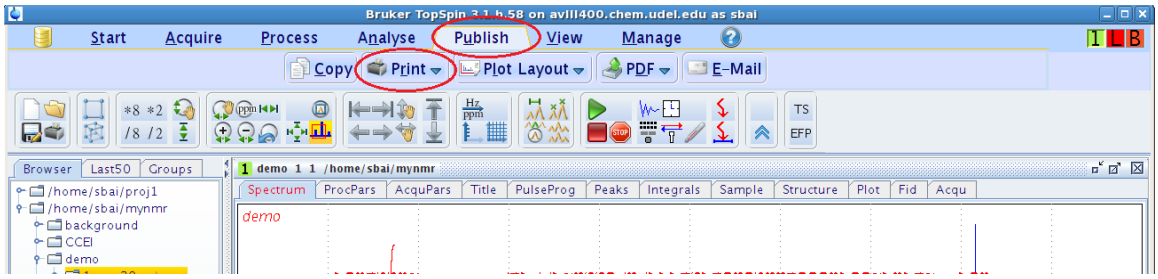

Figure 26: Printing Spectrum

22. In the BSMS panel, click on a green button "**Lock ON-OFF**" to turn off the frequency lock. Then click on a green button "**Lift**" to lift the sample from the probe. Remove the sample from the magnet, click on the **Lift** again to turn off the lifting air.

23. Exit the TopSpin program by clicking on the "x" sign as circled in Figure 27.

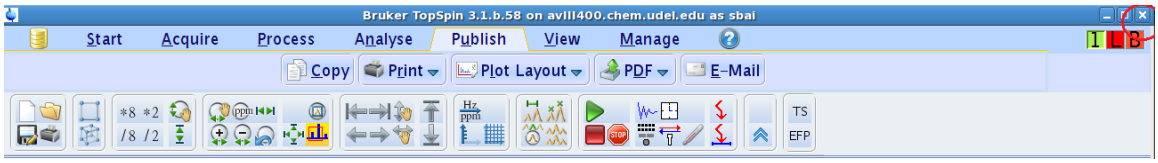

Figure 27: Exit TopSpin

4 Step-by-step Guide for ^{13}C Spectrum

We assume a proton NMR spectrum has been acquired by following the steps described in section 3. In this case, the frequency locking and shimming have been done. We can now follow these steps to obtain a ^{13}C spectrum.

1. First, create a new dataset for ^{13}C spectrum by first clicking on **Start** in the menu bar, followed by clicking on **Create Dataset** in its submenu tree. Change EXPNO from 1 to 2, and select an appropriate ^{13}C acquisition parameter file (*bbfo13cstdsm*) in **Experiment** pull-down menu. Select appropriate solvent in **Solvent** pull-down window. Make sure **Execute "getprosol"** is checked. Click on **OK** to continue.

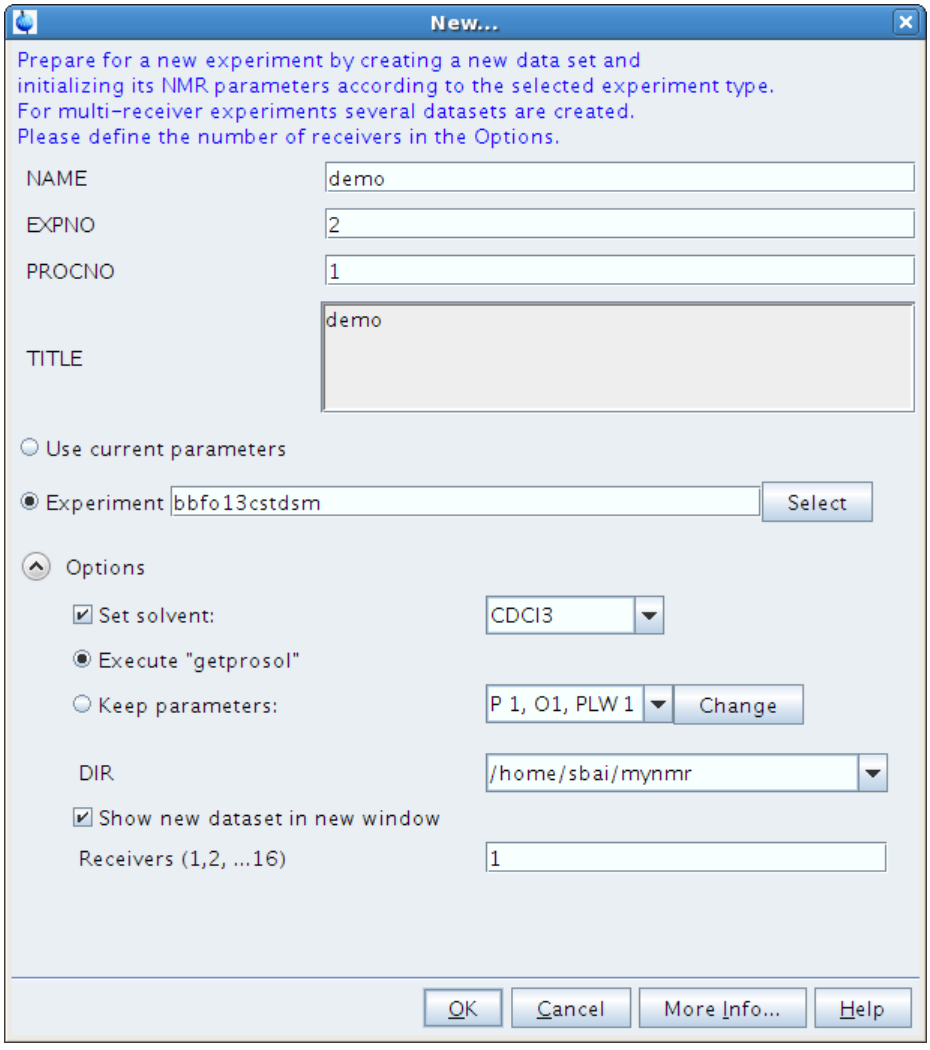

Figure 28: Create a New Dataset for ^{13}C Spectrum

2. Once a new dataset is created, click on **Acquire** in the menu bar and then on **Tune** in its submenu tree. Automatic tuning and matching starts momentarily. The tuning curves are shown for ^{13}C frequency tuning and then for ^{1}H frequency sequentially (Figure 29).

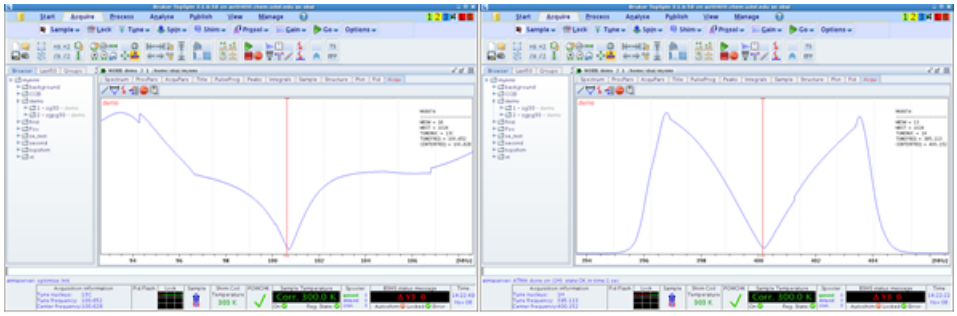

Figure 29: Probe Tuning and Matching for ^{13}C and ^{1}H Frequencies

3. Click on **Spin** in the **Acquire** submenu tree to spin the sample. Wait until **Sample** icon in the status bar changes as indicated in Figure 30.

Figure 30: Sample Icon in Status Bar

4. Click on **Go** in **Acquire** submenu tree (or type *zg* in the command line) to start the data acquisition. Make sure the *Spectrum* icon (circled in) is highlighted in **Acqu** panel. When signal-to-noise ratio reaches to a satisfactory level, type *halt* in the command line to pause the data acquisition. *Note*: *if* **Stop** *button is depressed, the experiment will be terminated without saving the data*.

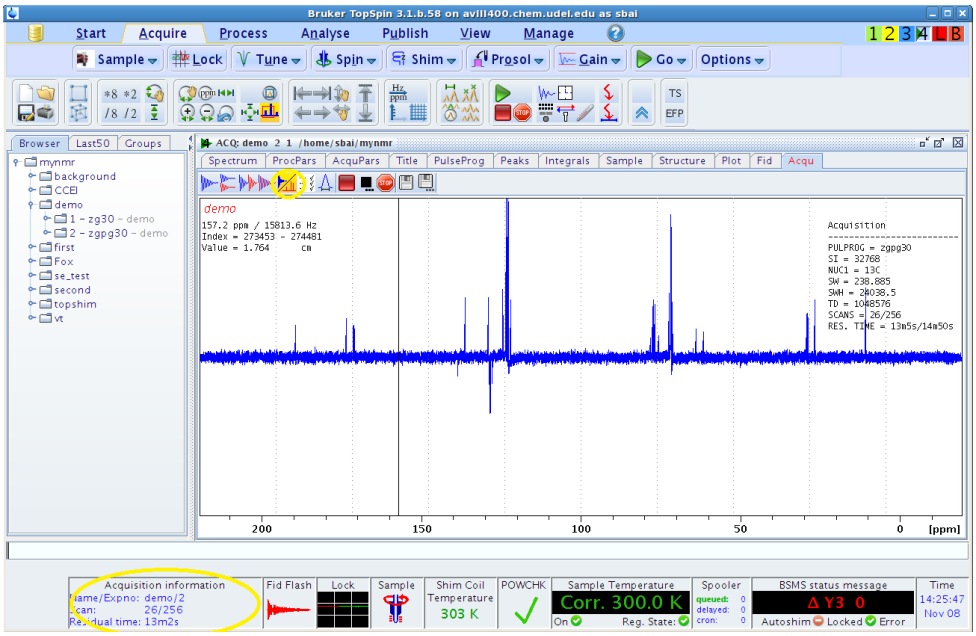

Figure 31: Acquisition Window When Spectrum Button is Highlighted

5. After the experiment is terminated, click on **Process** in the menu bar, followed by clicking on **Proc. Spectrum** in its submenu tree. A processed ^{13}C spectrum is shown in Figure 32.

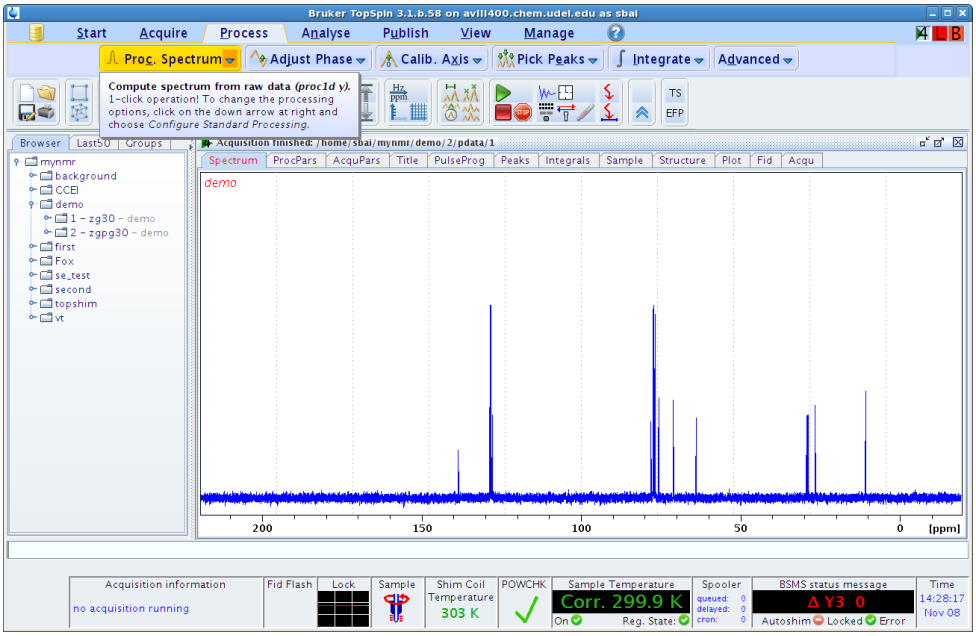

Figure 32: Processed ^{13}C Spectrum

6. Repeat steps 16 and 17 in section 3 (on page 14) to perform peak picking before printing the spectrum as described in step 21 of the section 3 (on page 16).

7. In the BSMS panel, click on green button "**Spin"** to stop sample spinning, click on **Lock ON-OFF** button to turn off frequency lock, and finally click on green button "**Lift"** to eject sample. After the sample is removed from the magnet, click on **Lift** again to turn off the lifting air.

8. Exit the program as described in step 23 of section 3.