



# Fourier

NMR experiments  
User Manual

Version 002

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# 1 Introduction

## 1.1 General

---

This manual was written for Fourier systems running TopSpin 3.1 and should be used as a guide through the set up process for some experiments. The success of running the experiments in this manual is under the assumption that all parameters have been entered in to the prosol table.

## 1.2 Disclaimer

---

This guide should only be used for its intended purpose as described in this manual. Use of the manual for any purpose other than that for which it is intended is taken only at the users own risk and invalidates any and all manufacturer warranties.

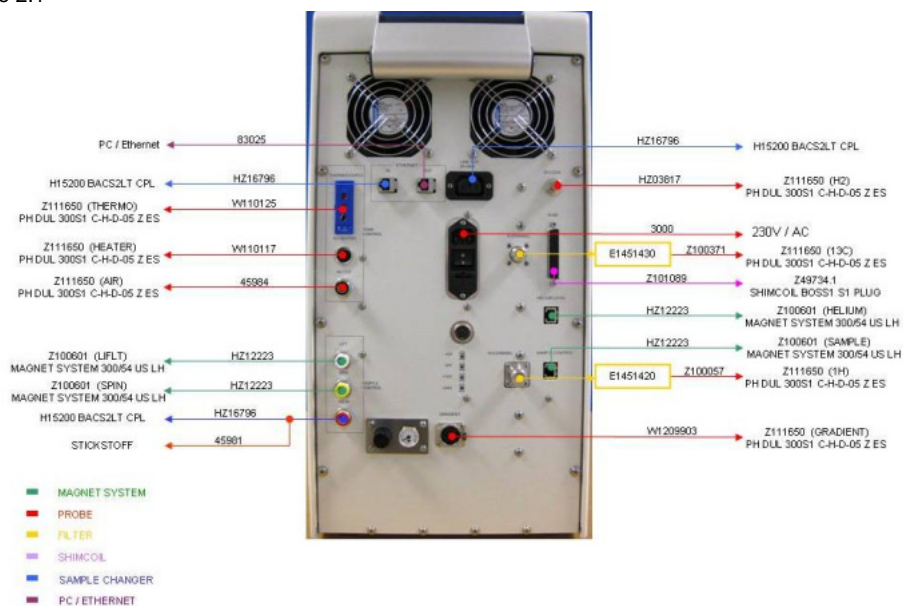
Some parameter values, especially power levels suggested in this manual may not be suitable for all Fourier systems and could cause damage to the unit. Therefore only persons trained in the operation of the a Fourier systems should operate the unit.



# 2 Spectrometer Basics

## 2.1 Fourier Connection Overview

Figure 2.1



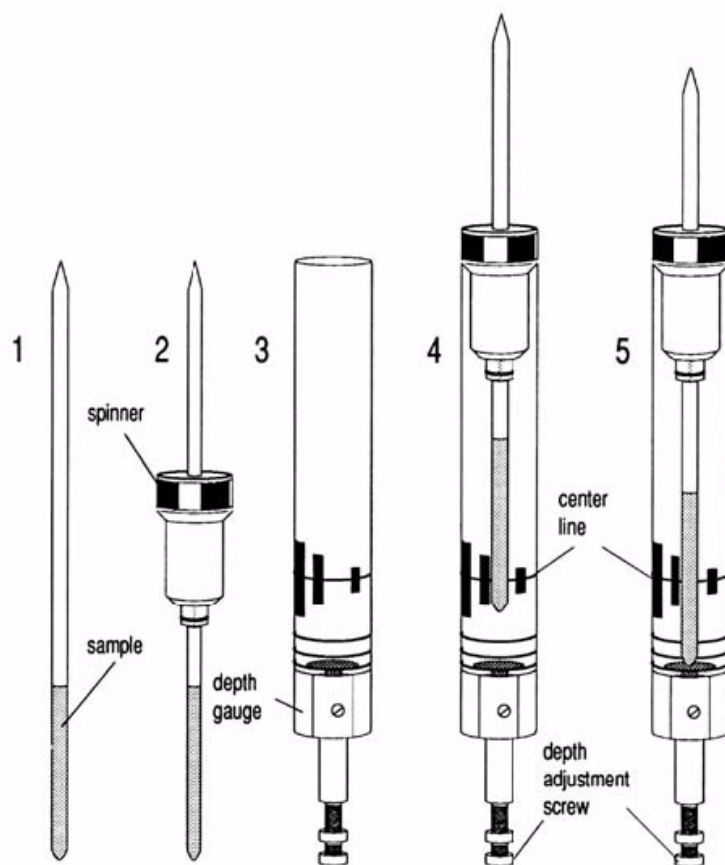
## 2.2 Sample preparation

- Use clean and dry sample tubes
- Use medium to high quality sample tubes
- Always filter the sample solution
- Always use the same sample volume or solution height
- Filling volume of a 5 mm tubes is 0.6 ml or 5 cm
- Use the sample depth gauge to adjust the sample depth
- The sample tube should sit tightly inside the spinner
- Wipe the sample tube clean before inserting into magnet
- Turn on lift air to insert the sample into the magnet

## 2.3 Inserting the Sample with Spinner into the Magnet

The raising and lowering of the sample is controlled by a stream of pressurized air. BOSS-1 shim systems are designed not to enable the LIFT if the magnet bore is plugged. Furthermore, make sure that the air flow is present (it is quite audible) before placing a sample onto the top of the bore.

Figure 2.2



To insert the sample with spinner into the magnet use the following procedure:

1. If present, remove the BST cap from the top of the magnet bore
2. Activate the LIFT. A flow of air will be heard and if a sample is already in the magnet it will be raised and suspended on a cushion of air at the top of the magnet bore.
3. Remove the old sample and place the new sample onto the air cushion
4. Turn off the LIFT. The sample will gently drop into the magnet and will settle at a precise position within the probe. You should be able to hear a clicking sound.

## 2.4 Spinning the Sample

A second function of pressurized air is to enable the sample to rotate. The spinning of the sample serves to “even-out” some of the inhomogeneities that may exist in the magnetic field at the center of the magnet.

---

NOTE: A suggested spin rate of 20Hz is used to spin the sample. The spinner is turned off for experiments such as 1D NOE, T1 and all 2D, to reduce unwanted artifacts from spinning the sample.

---

## 2.5 Locking the sample

---

Open up the lock display. This is a window in which the lock trace appears.

The most convenient way to lock is to use the Lock button in the Topspin menu bar in the the 'Acquire' tab or type **lock** in the command line. To start the lock-in procedure, select the appropriate solvent from the menu. Alternatively, enter the solvent name together with the lock command, e.g., **lock cdcl3**. During lock-in, several parameters such as the lock power, the field value, and the frequency shift for the solvent are set according to the values in the 'edlock' table. This table can be edited using the command edlock. Note that the lock power listed in this table is the level used after the sample has been locked. The field-shift mode is then selected and autolock is activated. Once lock-in is achieved, the lock gain is set so that the lock signal is visible in the lock window. At this point the message "lock: finished" appears in the status line at the bottom of the window.

The lock-phase adjustment by monitoring the sweep wiggles (i.e., while the field is not locked but is being swept) is recommended because autolock may fail. If the original phase is reasonably close to the correct value, lock-in can be achieved and the phase can be adjusted using the lock level. Note that the lock phase for the probe is stored in the edlock table. In some cases, the lock power level listed in the edlock table is set too high leading to a saturation of the lock signal. Usually, lock-in can be achieved, but the signal oscillates due to saturation. A quick fix is simply to reduce the lock power manually after lockin. However, it is better to change the power level in the edlock table.

---

NOTE: The appropriate lock power level depends on the lock solvent, the field value, and the probehead. Any value changes in the edlock table should only be done by experts.

---

## 2.6 Shimming the sample

---

The following is intended to be a practical guide for adjusting the room temperature shim system (BOSS-1). The purpose of shimming is to maximize the magnetic field homogeneity, which depends somewhat on probehead and sample geometry. In general, it is necessary to shim the magnetic field after a sample change, and occasionally between changes to correct for any magnet drifts.

Optimal shim settings may vary from samples and solvents; however, provided the sample has been adjusted to the correct depth and the solvent volume is the same for all samples, the shim values for a sample will be fairly reproducible. Thus, shimming time can be greatly reduced if a good shim setting is stored as a shim file on the computer. When necessary, the shim file can be read in and then final adjustments can be made to these shim values to correct for system drifts, and to account for the geometry of the particular sample being used.

The BOSS shim system consists of a number of shim coils arranged in the room temperature bore of the magnet. During shimming, the currents in these shim coils are adjusted so that the small magnetic field gradients produced cancel the residual inhomogeneity of the main magnetic field ( $H_0$ ) as completely as possible.

## 2.6.1 Shimming on the Lock Signal

When the spectrometer is locked, the vertical offset of the lock trace on the graphics display corresponds to the amplitude of the lock substance signal, assuming constant lock DC, gain, correct phase and power levels. The lock level, then, serves as useful guide for basic shim adjustment. The goal in shimming on the lock signal is to adjust the shims so that the lock trace appears as high on the graphics display as possible. This lock level corresponds to the highest possible lock substance signal amplitude.

## 2.6.2 Shimming on the FID (Free Induction Decay)

The shape of the FID, and especially the beginning of the FID, indicates the shape of the transformed signal line, while the length of the FID tail is important to the overall resolution. For good line shape and high resolution, the shim controls must be adjusted so that the FID envelope is truly exponential with the longest possible decay time.

## 2.6.3 Shimming using Gradshim

Gradshim is a tool designed for easy and automatic shimming.

## 2.7 Optimizing Resolution and Lineshape

The standard sample for measuring the proton lineshape and resolution specifications is, 3%  $\text{CHCl}_3$  in Acetone- $d_6$ .

For measuring the  $^{13}\text{C}$  resolution and lineshape test the standard sample ASTM (60% Dioxane in 40%  $\text{C}_6\text{D}_6$ ) sample may be used.

For both tests the line shape is measured at 50%, 055% and 0.11% of the peak. The Bruker standard parameter sets to use for this tests are PRORESOL and C13RESOL.

Figure 2.3 and 2.4 are illustrating the influence of the On-axis shims on the lineshape.

Figure 2.3

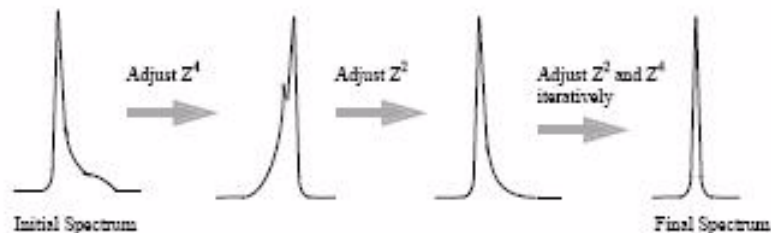
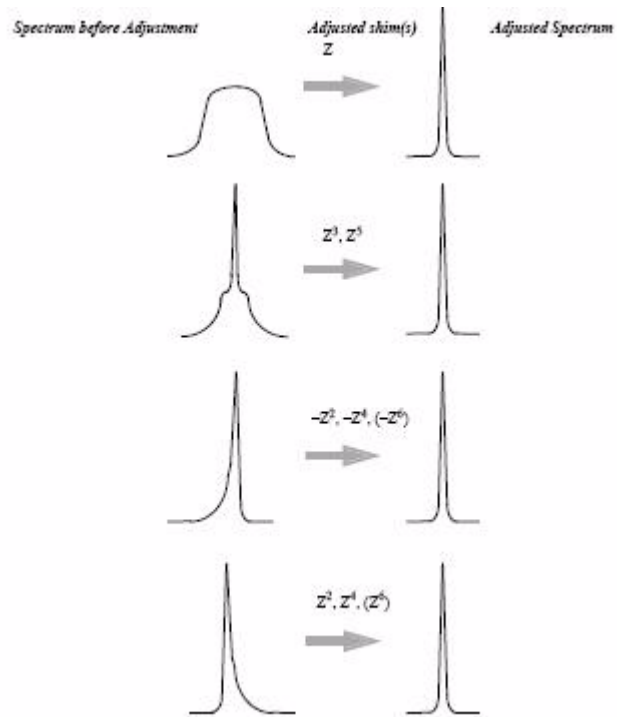


Figure 2.4



## 2.8 Observations

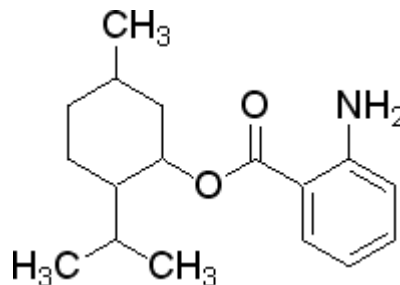
---

# 3 1-D Proton experiment

## 3.1 Sample

A sample of **30mg Menthyl Anthranilate in DMSO-d6** is used for the experiment in this chapter

Figure 3.1

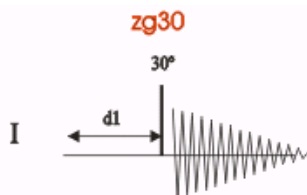


## 3.2 1-D Proton experiment

### 3.2.1 Introduction

Section 3.2 describes the acquisition and processing of a one-dimensional <sup>1</sup>H NMR spectrum using the standard Bruker parameter set **PROTON**. The pulse sequence **zg30**, Figure 3.2 consists of the recycling delay, the radio-frequency (RF) pulse, and the acquisition time during which the signal is recorded. The pulse angle is shown to be 30 degrees. The two parameters, D1 and P1, correspond to the length of the relaxation delay, and the length of the 90 degree RF pulse, respectively.

Figure 3.2



The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

### 3.2.2 Experiment setup

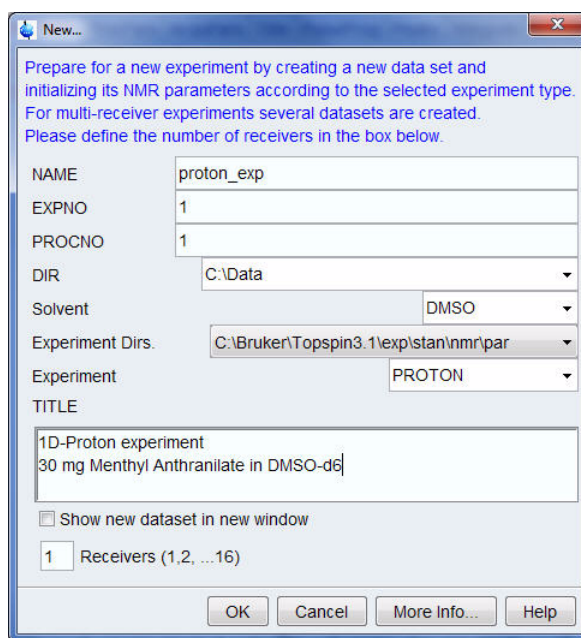
1. Click on the **'Start'** tab in the TopSpin Menu bar

Figure 3.3



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 3.4



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 3.4 above. Click on the down arrow button to browse for a specific directory.

4. Click on **OK**
5. Click on the **Acquire** tab in the TopSpin menu bar

Figure 3.5



6. Select **Sample** by clicking on it

Figure 3.6



7. Select 'ej' by clicking on it

---

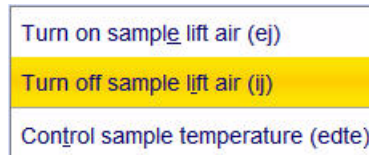
NOTE: Wait till the sample lift air is turned on and remove any sample which may have been in the magnet.

---

8. Place the sample on to the top of the magnet

9. Select  **Sample** by clicking on it

Figure 3.7



10. Select 'ij' by clicking on it

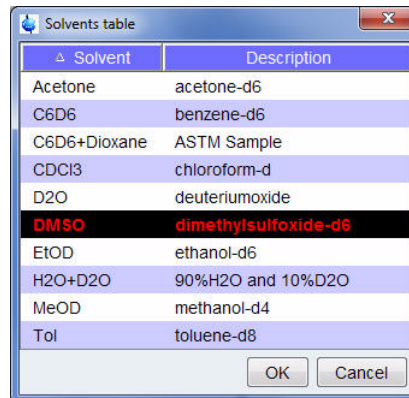
---

NOTE: Wait till the sample is lowered down in to the probe and the lift air is turned off. A clicking sound may be heard.

---

11. Select  **Lock** by clicking on it

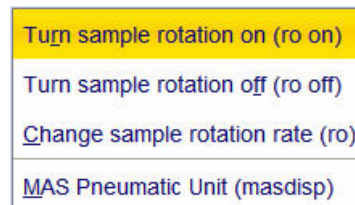
Figure 3.8



12. Select '**DMSO**' by clicking on it

13. Select  **Spin** by clicking on it

Figure 3.9



14. Select '**ro on**' by clicking on it

15. Select  by clicking on it

16. Select  by clicking on it

---

NOTE: This will load the pulse width and power levels in to the parameter set.

---

## 3.2.3 Acquisition

---

1. Select  by clicking on it

2. Select  by clicking on it

---

NOTE: The experiment will take ~2 minutes, using the default number of 16 scans.

---

## 3.2.4 Processing

---

1. Click on the '**Process**' tab in the TopSpin Menu bar

Figure 3.10



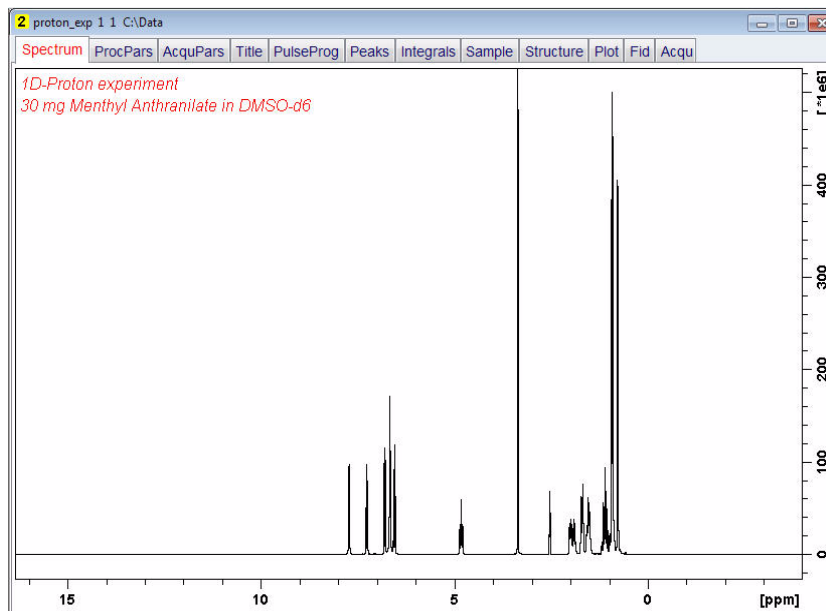
2. Click on 

---

NOTE: This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**' button.

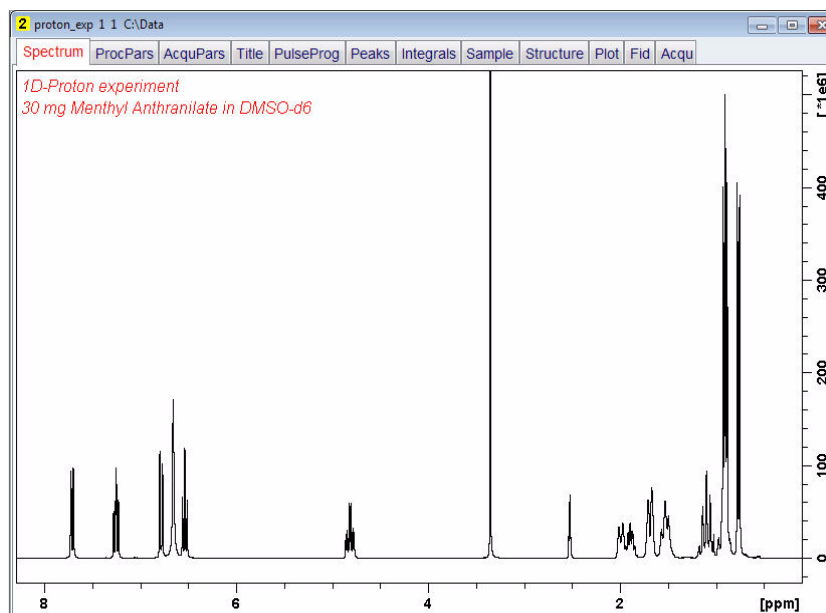
---

Figure 3.11



3. Expand the spectrum to include all peaks

Figure 3.12

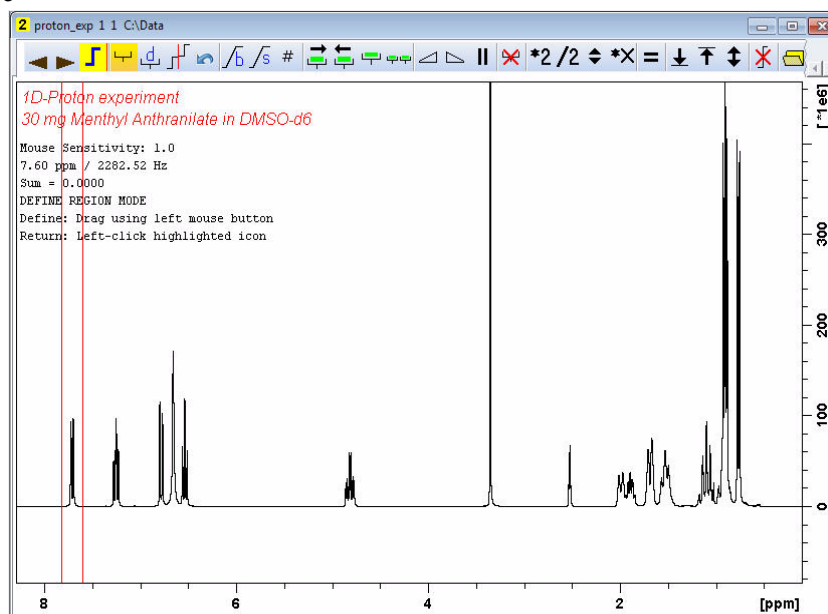


4. Click on 

NOTE: This enters the manual Integration mode. Other options are available by clicking on the down arrow inside the 'Integrate' button.

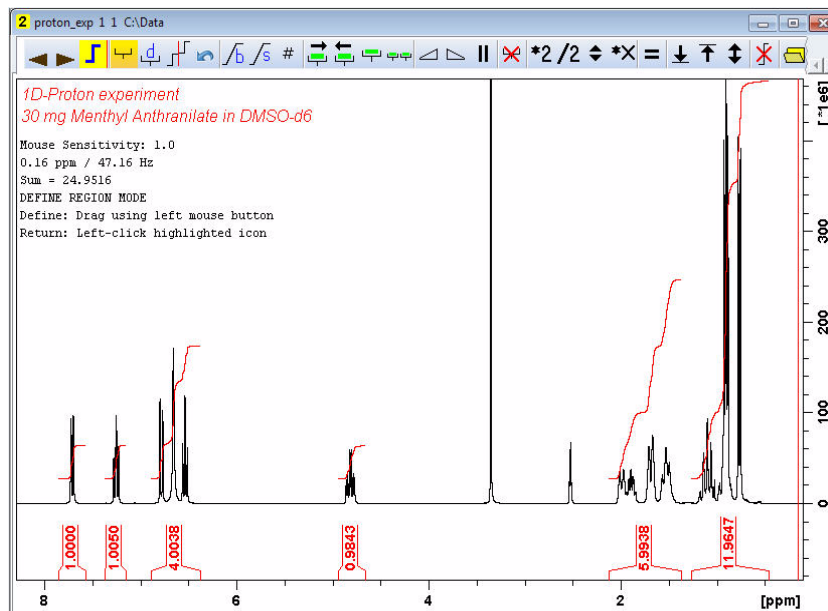
5. Set the cursor line, starting at the left of the spectrum, to the left of the first peak to be integrated, click the left mouse button and drag the cursor line to the right of the peak, then release the mouse button


Figure 3.13



6. Repeat step13 for the remainder of the peaks

Figure 3.14



7. Click on  to save the integration regions

## 3.2.5 Plotting the 1D Proton spectra


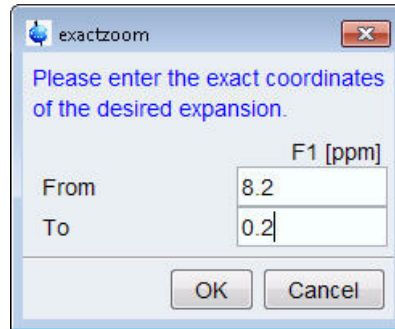
1. Expand the spectrum (all peaks in display)
2. Click on 
3. Type the following F1 [ppm] values:  
From = 8.2  
To = 0.2

Figure 3.15



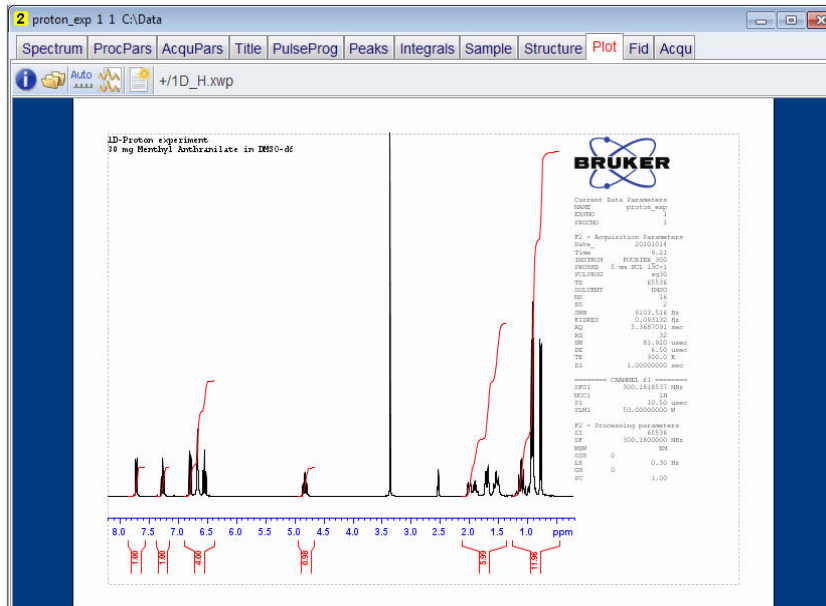
4. Click on 
5. Click on the 'Publish' tab in the TopSpin Menu bar

Figure 3.16




6. Click on 


Figure 3.17



---

NOTE: If desired, any changes can be administered by clicking on the  icon to open the Plot Editor.

---

7. Click on the  to plot the spectrum

## 3.2.6 Observations

---



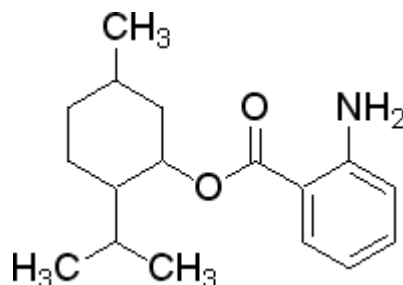
# 4 2-D Homonuclear experiments

## 4.1 Sample

---

A sample of **30mg Menthyl Anthranilate in DMSO-d6** is used for the experiments in this chapter

Figure 4.1



## 4.2 2-D gradient COSY

---

### 4.2.1 Introduction

---

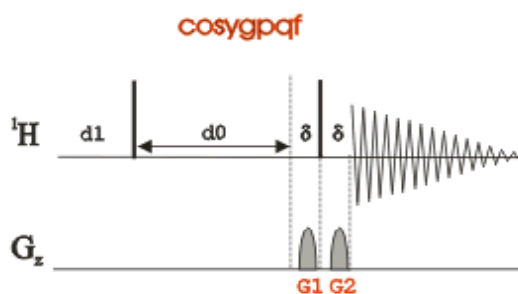
The COSY experiment relies on the J-coupling to provide spin-spin correlation, and whose cross peaks indicate which  $1\text{H}$ 's are close to which other  $1\text{H}$ 's through the bonds of the molecule. Typically proton which are up 3 bonds away can be observed.

The signals acquired with one of these experiments have absorptive and dispersive line shape contributions in both F1 and F2 dimensions. This means that it is impossible to phase the spectrum with all peaks purely absorptive, and, as a consequence, the spectrum must be displayed in magnitude mode. A typical spectral resolution of 3 Hz/pt is sufficient for resolving large scalar couplings. In order to resolve small J-couplings fine digital resolution is required, which significantly increases the experimental time. In general, the DQF-COSY experiment is recommended if a higher resolution is desired.

Using pulsed field gradients (PFG), the coherence pathway selection and the axial peak suppression can be achieved with only one scan per time increment. Thus, if enough substance is available, a typical gradient COSY experiment with 128 time increments can be recorded in 5 minutes.

Section 4.2.3 describes the acquisition and processing of a two-dimensional  $1\text{H}$  gradient COSY. The standard Bruker parameter set is **COSYGPSW** and includes the pulse sequence **cosygpqf** shown in Figure 4.2. It consists of the relaxation delay, two radio frequency (RF) pulses, separated by the increment delay  $D_0$  and the acquisition time during which the signal is recorded. Both pulses have a 90 degrees angle. Two gradient pulses are applied before and after the second pulse in the sequence. Purge pulses are applied before  $d_1$ .

Figure 4.2

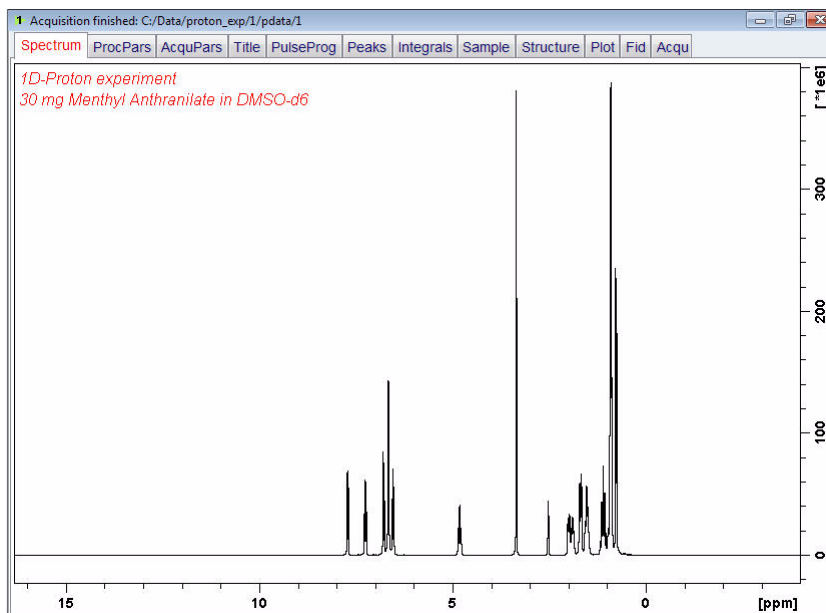


The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

## 4.2.2 Preparation experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 3, 1-D Proton experiment, Paragraph 3.2.2 Experiment setup** through **3.2.4 Processing**.

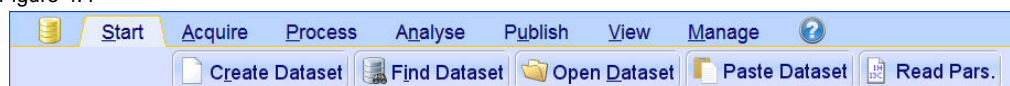
Figure 4.3



## 4.2.3 Setting up the COSY experiment

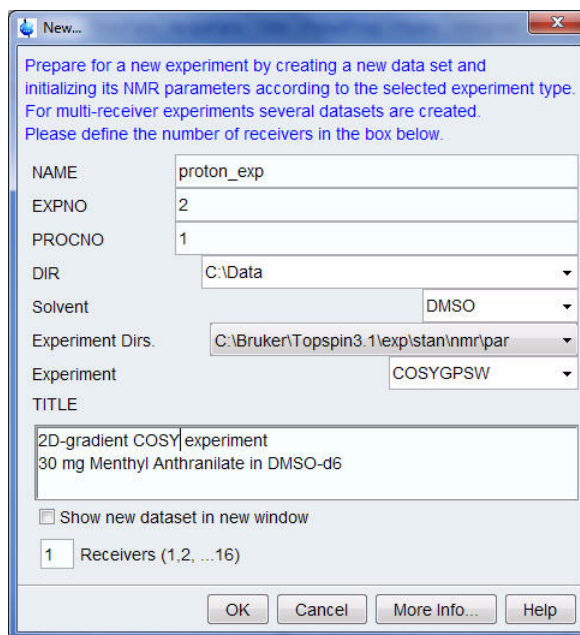
1. Click on the **'Start'** tab in the TopSpin Menu bar

Figure 4.4



2. Select  **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 4.5



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 4.5 above. Click on the down arrow button to browse for a specific directory.


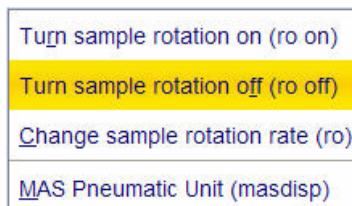
4. Click on 
5. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 4.6



6. Select  **Spin** by clicking on it

Figure 4.7



7. Select '**ro off**' by clicking on it

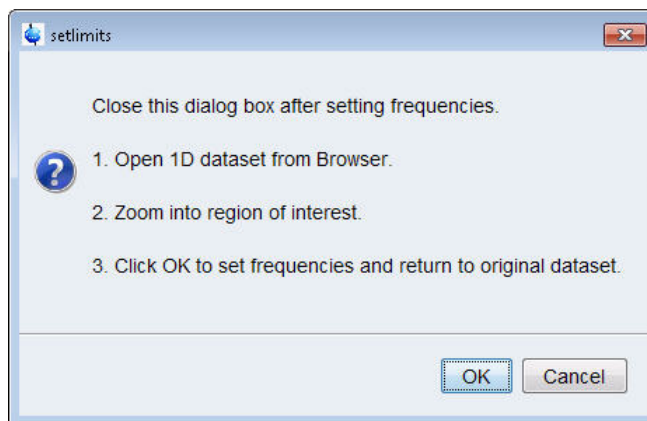
NOTE: 2-D experiments should always be run without rotation.

8. Select  by clicking on it

NOTE: This will load the pulse width and power levels in to the parameter set.

9. Select  by clicking on it

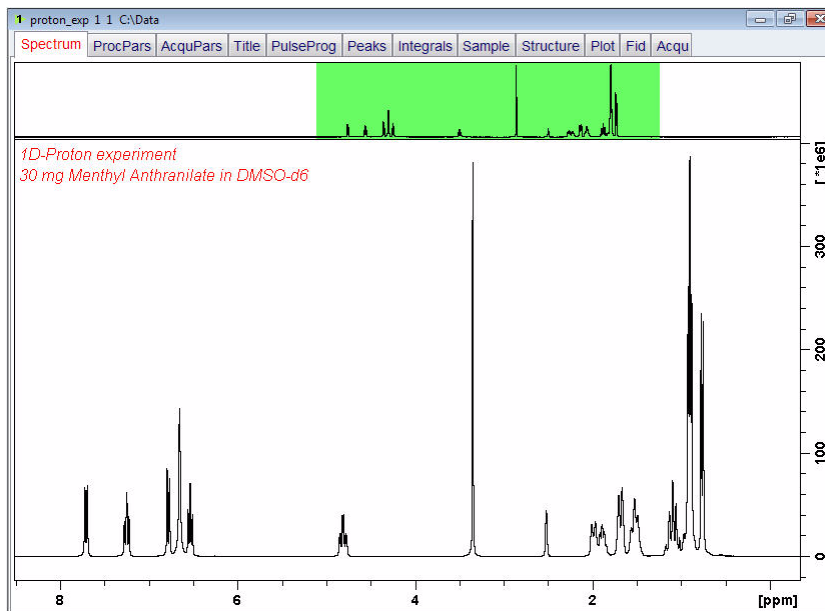
Figure 4.8



10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton\_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window

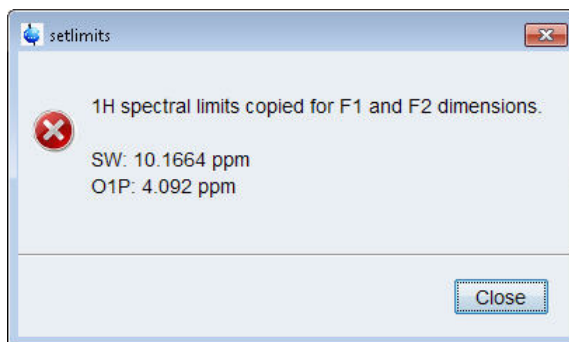
11. Expand the spectrum to display all peaks, leaving ca. 0.2 ppm of baseline on either side of the spectrum

Figure 4.9



12. Click on  to assign the new limit

Figure 4.10



13. Click on 

---

NOTE: The display changes back to the 2D data set.

---

#### 4.2.4 Acquisition

1. Select  by clicking on it
2. Select  by clicking on it

---

NOTE: The experiment will take ~5 minutes, using the default number of 1 scan and 128 increments.

---

#### 4.2.5 Processing

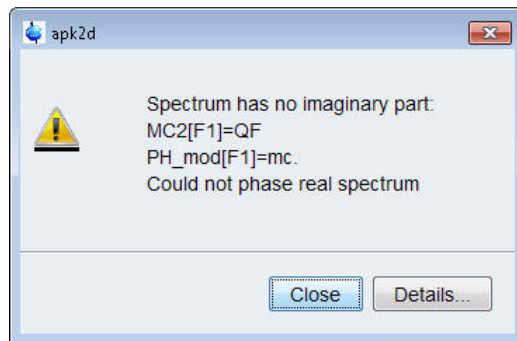
1. Click on the **'Process'** tab in the TopSpin Menu bar

Figure 4.11



2. Select  by clicking on it

Figure 4.12



NOTE: This executes a standard processing program **proc2**. The message shown in Figure 4.12 pops up in case of a magnitude 2D experiment and the apk2d option is enabled. To configure the processing program follow the steps below.


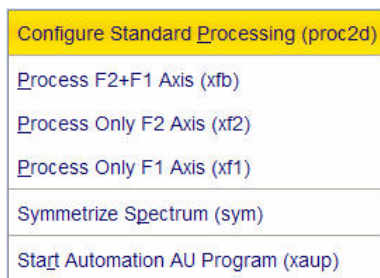
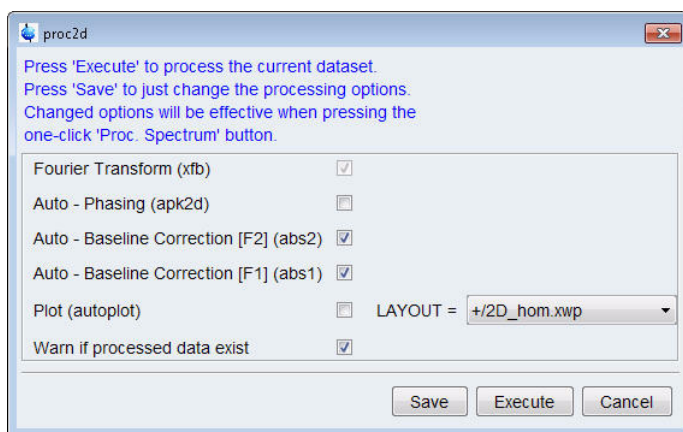
3. Click on the down arrow inside the  button

Figure 4.13



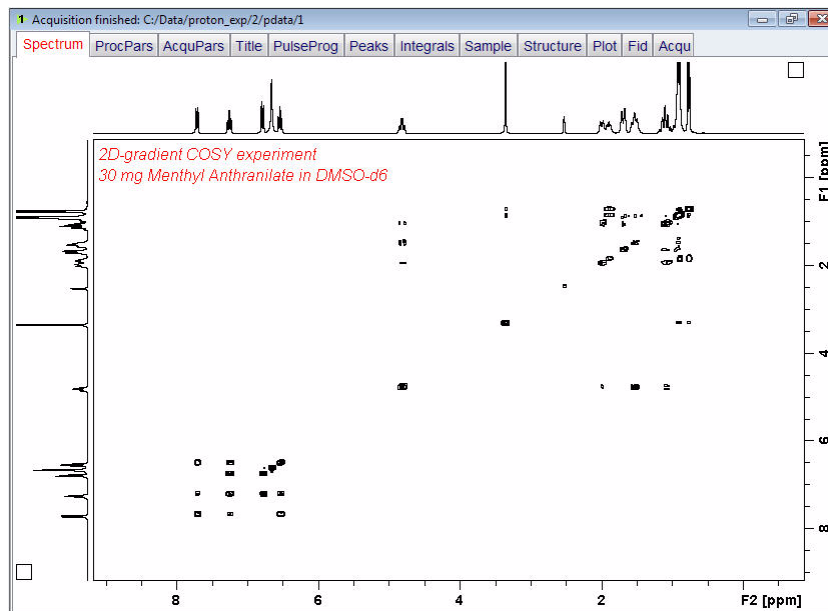
4. Select '**Configure Standard Processing**' by clicking on it

Figure 4.14



NOTE: To avoid the message shown in Figure 4.12 the option '**Auto-Phasing (apk2d)**' may be disabled for magnitude like 2D experiment.

Figure 4.15



## 4.2.6 Plotting


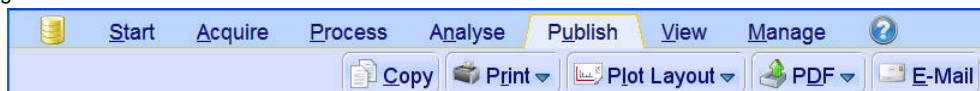
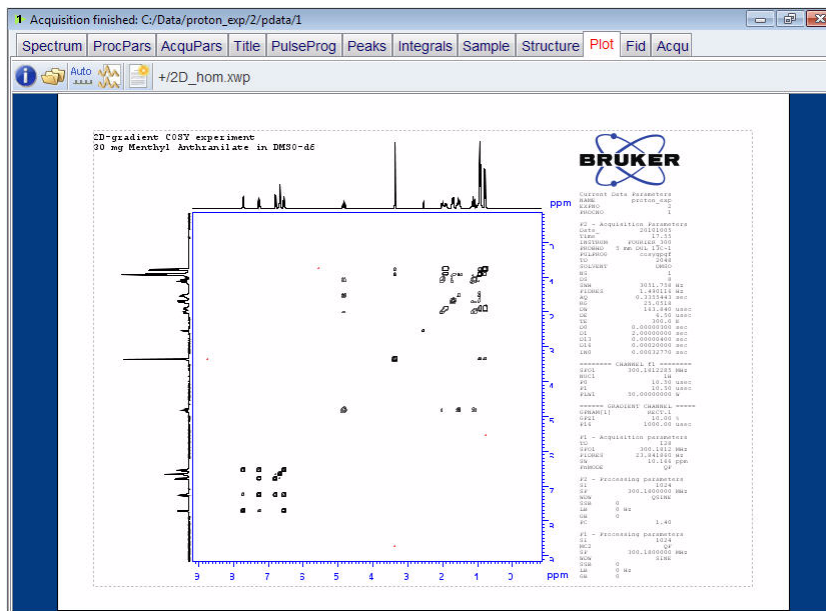
1. Use the  buttons to adjust for a suitable contour level
2. Click on the **'Publish'** tab in the TopSpin Menu bar


Figure 4.16



3. Click on 

Figure 4.17



NOTE: If desired, any changes can be administered by clicking on the  icon to open the Plot Editor.

4. Click on the  to plot the spectrum

## 4.2.7 Observations

---

## 4.3 2-D gradient NOESY experiment

### 4.3.1 Introduction

NOESY (Nuclear Overhauser Effect Spectroscopy) is a 2D spectroscopy method used to identify spins undergoing cross-relaxation and to measure the cross-relaxation rates. Most commonly, NOESY is used as a homonuclear  $^1\text{H}$  technique. In NOESY, direct dipolar couplings provide the primary means of cross-relaxation, and so spins undergoing cross-relaxation are those which are close to one another in space. Thus, the cross peaks of a NOESY spectrum indicate which protons are close to each other in space. This can be distinguished from COSY, for example, which relies on J-coupling to provide spin-spin correlation, and whose cross peaks indicate which  $^1\text{H}$ 's are close to which other  $^1\text{H}$ 's through the bonds of the molecule.

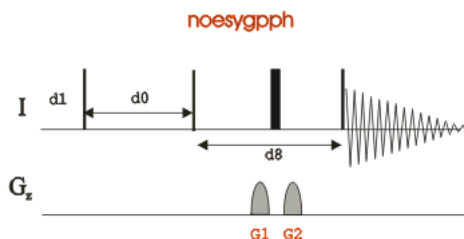
The basic NOESY sequence consists of three  $p/2$  pulses. The first pulse creates transverse spin magnetization. This precesses during the evolution time  $t_1$ , which is incremented during the course of the 2D experiment. The second pulse produces longitudinal magnetization equal to the transverse magnetization component orthogonal to the pulse direction. Thus, the basic idea is to produce an initial situation for the mixing period  $d_8$ . Note that, for the basic NOESY experiment,  $d_8$  is kept constant throughout the 2D experiment. The third pulse creates transverse magnetization from the remaining longitudinal magnetization. Acquisition begins immediately following the third pulse, and the transverse magnetization is observed as a function of the time  $t_2$ . The NOESY spectrum is generated by a 2D Fourier transform with respect to  $t_1$  and  $t_2$ .

Axial peaks, which originate from magnetization that has relaxed during  $t_m$ , can be suppressed using the appropriate phase cycling.

NOESY spectra can be obtained in 2D absorption mode. Occasionally, COSY-type artifacts appear in the NOESY spectrum; however, these are easy to identify by their anti-phase multiplet structure.

Section 4.3.3 describes the acquisition and processing of a two-dimensional  $^1\text{H}$  phase sensitive NOESY. The standard Bruker parameter set is **NOESYGPPHSW** and includes the pulse sequence **noesygpqh** shown in Figure 4.18. It consists of the relaxation delay, three radio-frequency (RF) pulses, separated by the increment delay  $D_0$  between the first and second pulse, a mixing time  $D_8$  between the second and third pulse and the acquisition time during which the signal is recorded. All three pulses are of 90 degree.

Figure 4.18

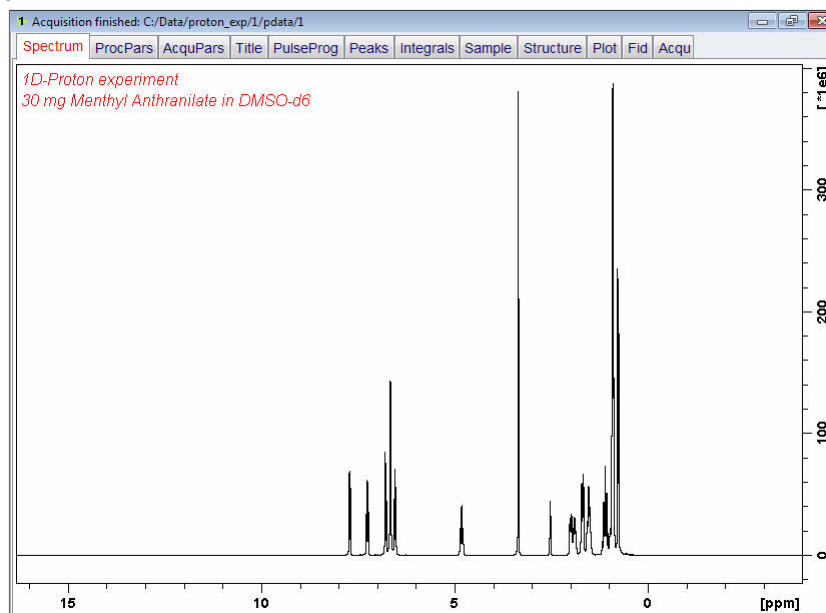


The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example,  $d_1$  is typically a few seconds while  $p_1$  is typically a few microseconds in length.

### 4.3.2 Preparation experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 3, 1-D Proton experiment, Paragraph 3.2.2 Experiment setup** through **3.2.4 Processing**.

Figure 4.19



### 4.3.3 Setting up the NOESY experiment

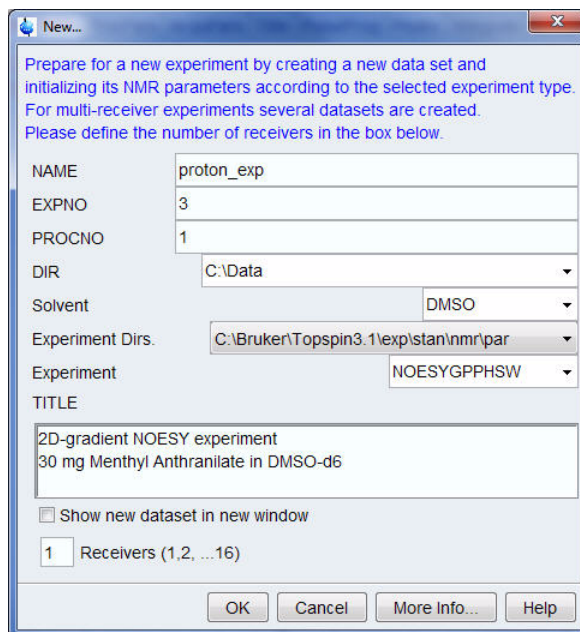
1. Click on the **'Start'** tab in the TopSpin Menu bar

Figure 4.20



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 4.21



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 4.5 above. Click on the down arrow button to browse for a specific directory.


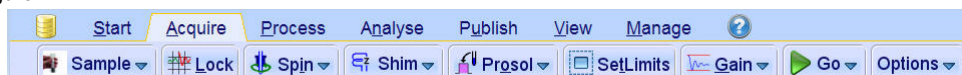
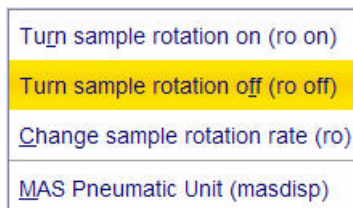
4. Click on 
5. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 4.22



6. Select  by clicking on it

Figure 4.23



7. Select '**ro off**' by clicking on it

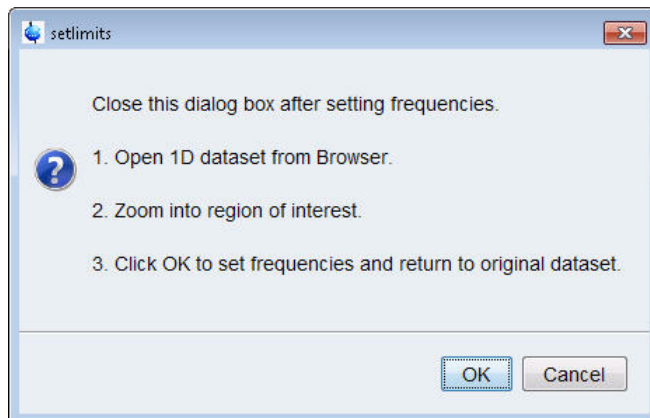
NOTE: 2-D experiments should always be run without rotation.

8. Select  by clicking on it

NOTE: This will load the pulse width and power levels in to the parameter set.

9. Select  by clicking on it

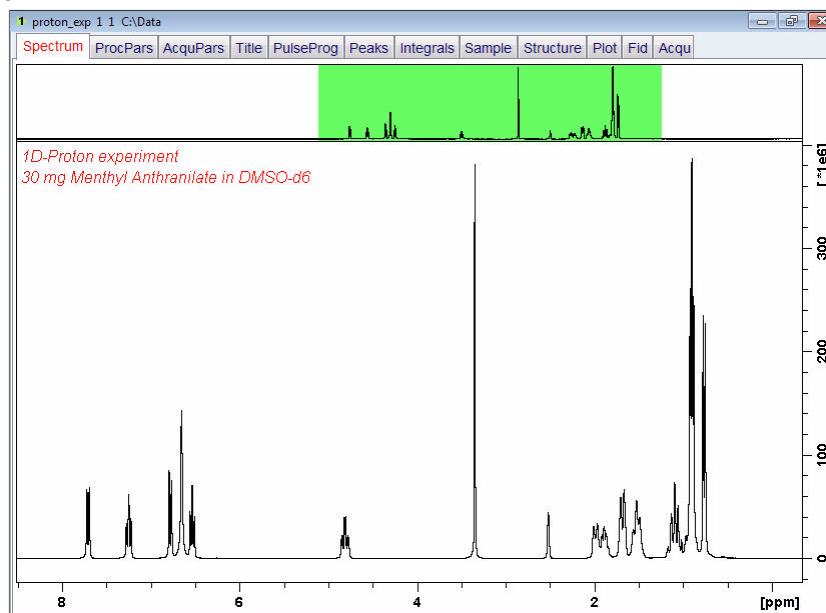
Figure 4.24



10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton\_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window

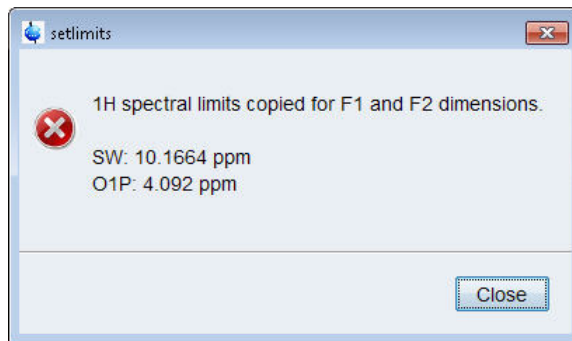
11. Expand the spectrum to display all peaks, leaving ca. 0.2 ppm of baseline on either side of the spectrum

Figure 4.25



12. Click on  to assign the new limit

Figure 4.26



13. Click on 

NOTE: The display changes back to the 2D data set.


14. Select the **'AcquPars'** tab by clicking on it
15. Click on  to display the pulse program parameters
16. Make the following change

Figure 4.27



17. Select the **'Spectrum'** tab by clicking on it

## 4.3.4 Acquisition

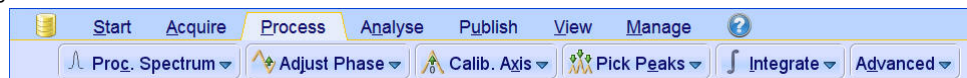
1. Select  by clicking on it
2. Select  by clicking on it

NOTE: The experiment will take ~50 minutes, using the default number of 4 scans and 256 increments.

## 4.3.5 Processing

1. Click on the **'Process'** tab in the TopSpin Menu bar

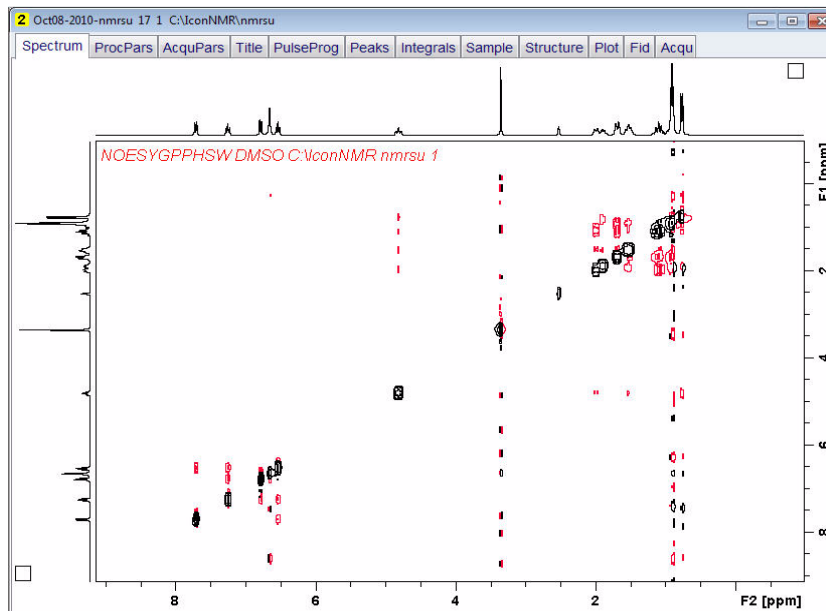
Figure 4.28



2. Select  by clicking on it

NOTE: This executes a standard processing program **proc2**. To configure this program or select other options, click on the down arrow inside the 'Proc. Spectrum' button.

Figure 4.29



### 4.3.6 Plotting


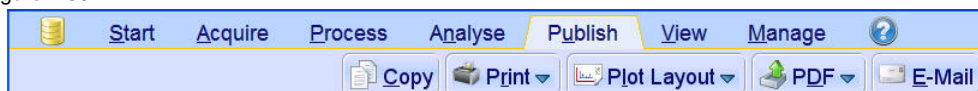
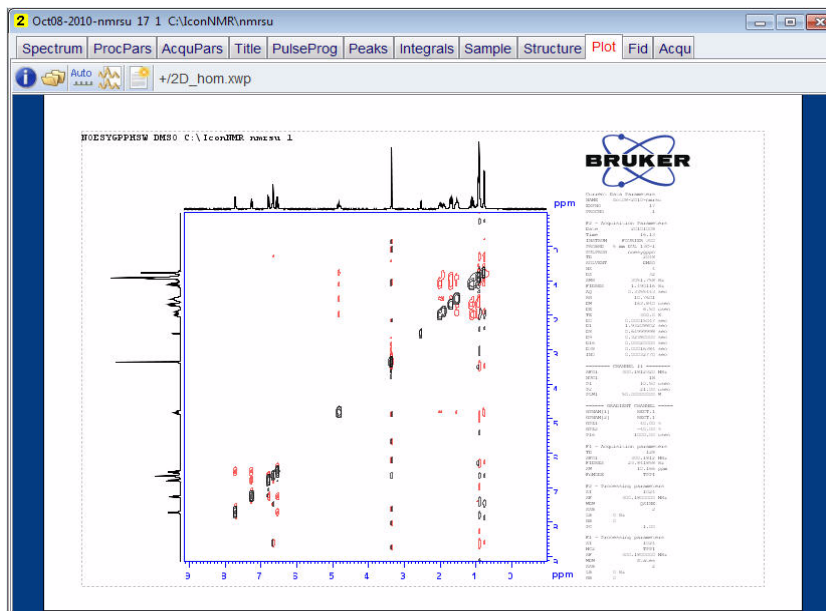
1. Use the  buttons to adjust for a suitable contour level
2. Click on the 'Publish' tab in the TopSpin Menu bar


Figure 4.30




3. Click on 

Figure 4.31



NOTE: If desired, any changes can be administered by clicking on the  icon to open the Plot Editor.

4. Click on the  to plot the spectrum

### 4.3.7 Observations

---

## 4.4 2-D phase sensitive TOCSY experiment

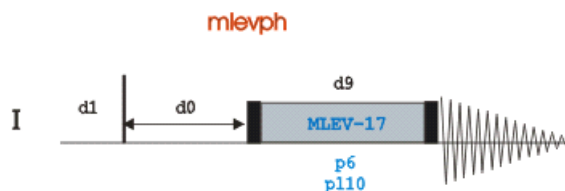
### 4.4.1 Introduction

TOCSY (TOtal Correlation SpectroscopY) provides a different mechanism of coherence transfer than COSY for 2D correlation spectroscopy in liquids. In TOCSY, cross peaks are generated between all members of a coupled spin network. An advantage is that pure absorption mode spectra with positive intensity peaks are created. In traditional COSY, cross peaks have zero integrated intensity and the coherence transfer is restricted to directly spin-coupled nuclei. In TOCSY, oscillatory exchange is established which proceeds through the entire coupling network so that there can be net magnetization transfer from one spin to another even without direct coupling. The isotropic mixing which occurs during the spin-lock period of the TOCSY sequence exchanges all in-phase as well as antiphase coherence.

The coherence transfer period of the TOCSY sequence occurs during a multiple-pulse spin-lock period. The multiple-pulse spin-lock sequence most commonly used is MLEV-17. The length of the spin-lock period determines how far the spin coupling network will be probed. A general rule of thumb is that  $1/(10 J_{HH})$  should be allowed for each transfer step, and five transfer steps are typically desired for the TOCSY spectrum.

Section 4.4.3 describes the acquisition and processing of a two-dimensional  $^1H$  phase sensitive TOCSY. The standard Bruker parameter set is **MLEVPHSW** and includes the pulse sequence **mlevph** shown in Figure 4.32. It consists of the recycling delay, two radio-frequency (RF) pulses, separated by the increment delay  $D_0$  and the acquisition time during which the signal is recorded. The first RF pulse is a 90 degree pulse, the second pulse is the mlev spinlock pulse.

Figure 4.32

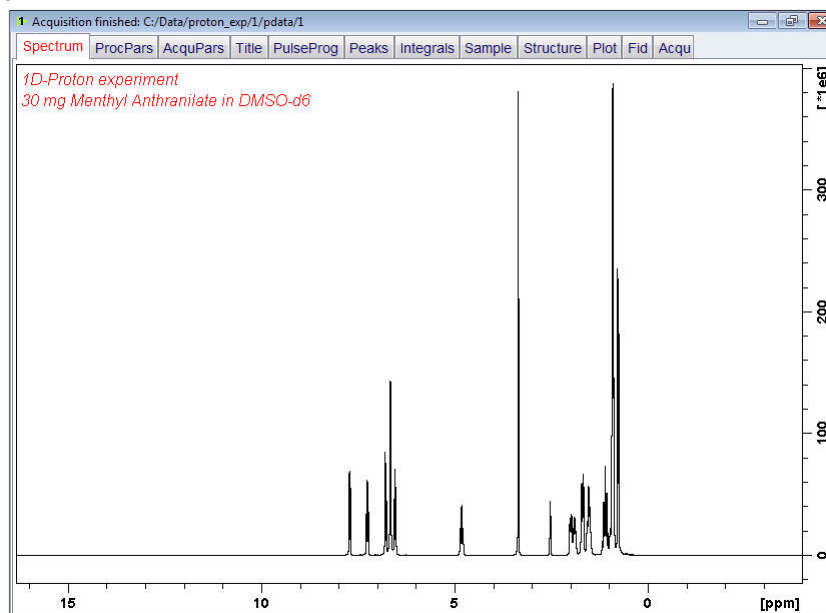


The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example,  $d_1$  is typically a few seconds while  $p_1$  is typically a few microseconds in length.

### 4.4.2 Preparation experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 3, 1-D Proton experiment, Paragraph 3.2.2 Experiment setup** through **3.2.4 Processing**.

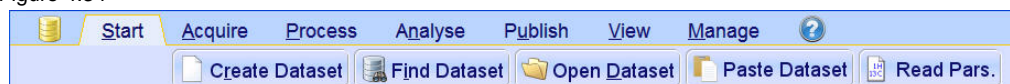
Figure 4.33



### 4.4.3 Setting up the TOCSY experiment

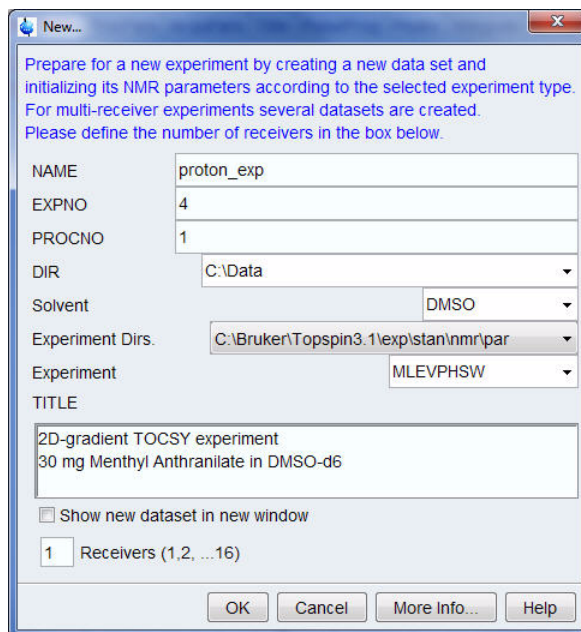
1. Click on the **'Start'** tab in the TopSpin Menu bar

Figure 4.34



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 4.35



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 4.35 above. Click on the down arrow button to browse for a specific directory.


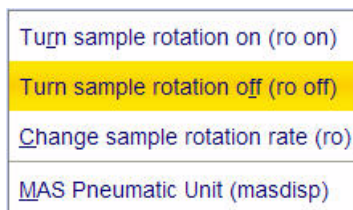
4. Click on 
5. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 4.36



6. Select  by clicking on it

Figure 4.37



7. Select '**ro off**' by clicking on it

NOTE: 2-D experiments should always be run without rotation.

8. Select  by clicking on it

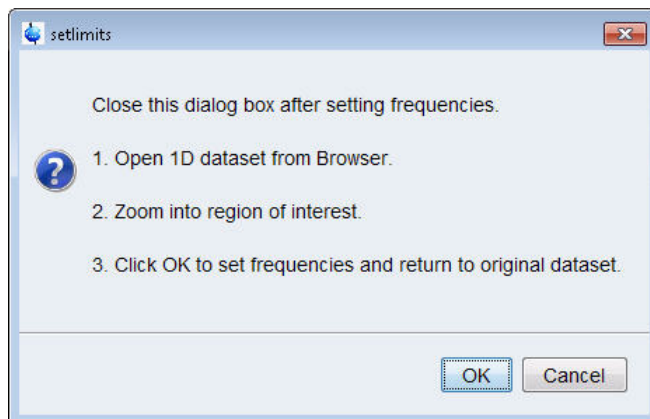
---

NOTE: This will load the pulse width and power levels in to the parameter set.

---

9. Select  by clicking on it

Figure 4.38



10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton\_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window

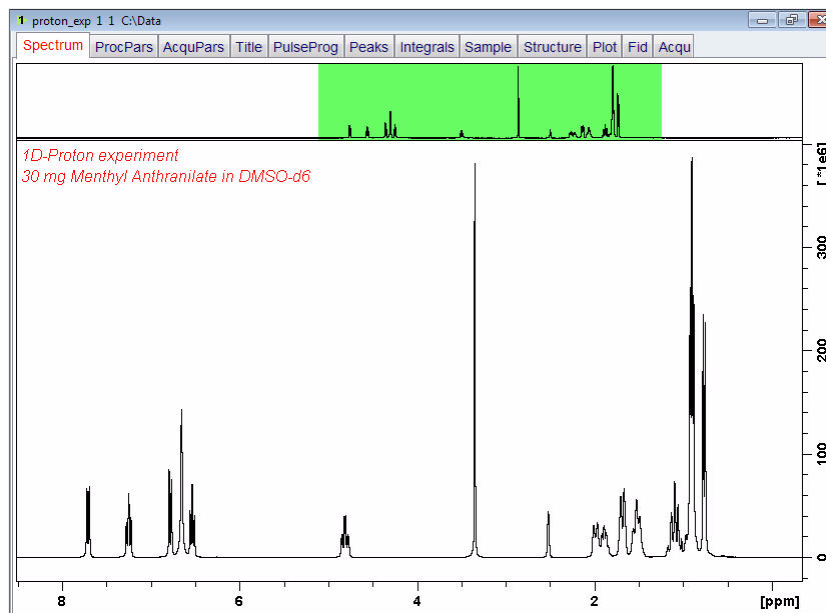
11. Expand the spectrum to display all peaks, leaving ca. 0.2 ppm of baseline on either side of the spectrum

---

NOTE: The solvent peak may be excluded if it falls outside of the region of interest. Digital filtering however is only applied in F2 and the solvent peak is folding in F1.

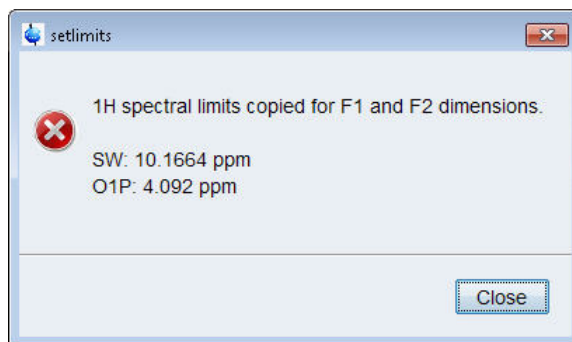
---

Figure 4.39



12. Click on  to assign the new limit

Figure 4.40



13. Click on 

---

NOTE: The display changes back to the 2D data set.

---

14. Select the '**AcquPars**' tab by clicking on it

15. Make the following change:

NS = 8

TD (F1) = 128

16. Select the '**Spectrum**' tab by clicking on it

## 4.4.4 Acquisition

---

1. Select  by clicking on it

2. Select  by clicking on it

---

NOTE: The experiment will take ~42 minutes, using 8 scans and 128 increments.

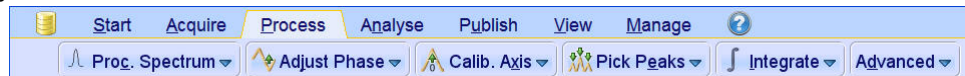
---

## 4.4.5 Processing

---

1. Click on the '**Process**' tab in the TopSpin Menu bar

Figure 4.41



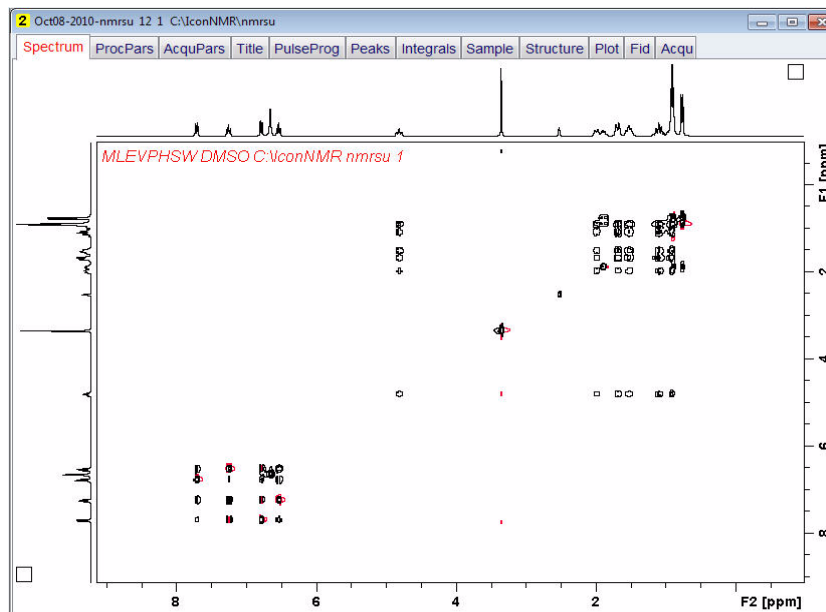
2. Select  by clicking on it

---

NOTE: This executes a standard processing program **proc2**. To configure this program

or select other options, click on the down arrow inside the 'Proc. Spectrum' button.

Figure 4.42



## 4.4.6 Plotting


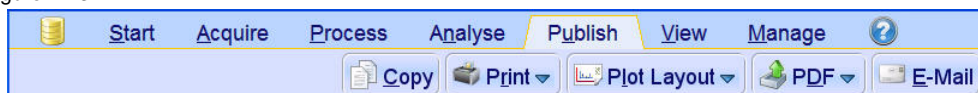
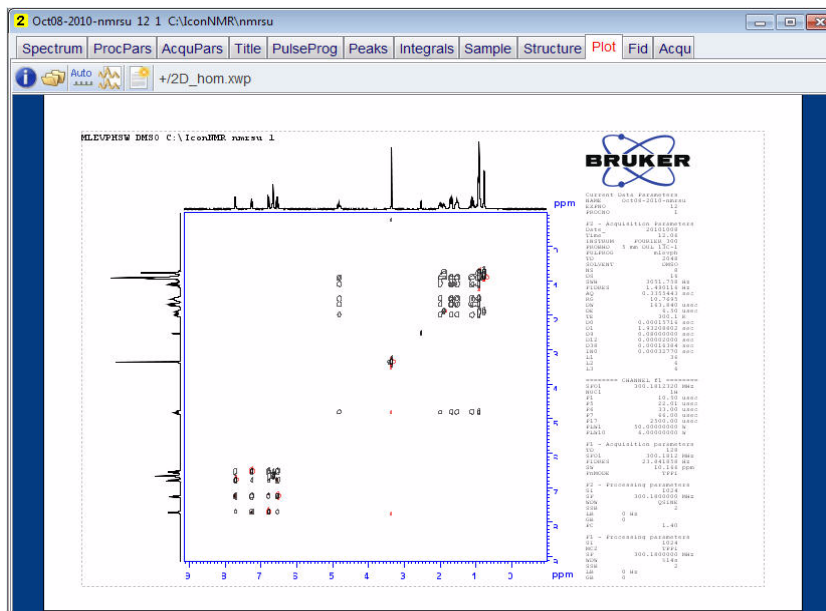
1. Use the  buttons to adjust for a suitable contour level
2. Click on the 'Publish' tab in the TopSpin Menu bar


Figure 4.43



3. Click on 
4. Select the 'Plot' tab by clicking on it

Figure 4.44



NOTE: If desired, any changes can be administered by clicking on the  icon to open the Plot Editor.

5. Click on the  to plot the spectrum

### 4.4.7 Observations

---

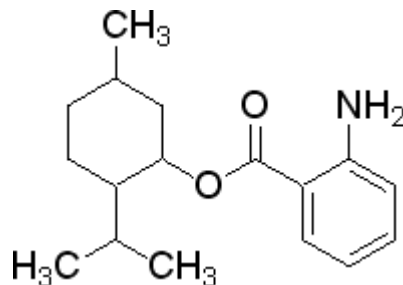


# 5 1-D Carbon experiments

## 5.1 Sample

A sample of **30mg Menthyl Anthranilate in DMSO-d6** is used for the experiments in this chapter

Figure 5.1

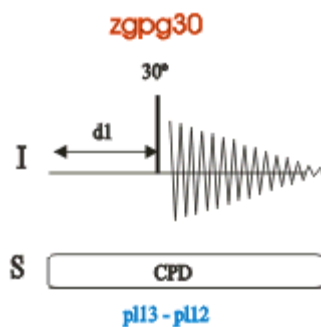


## 5.2 1-D Carbon Experiment

### 5.2.1 Introduction

Section 5.2.2 describes the acquisition and processing of a one-dimensional  $^{13}\text{C}$  NMR spectrum. The standard Bruker parameter set **C13CPD**, includes the pulse sequence **zgpg30**, shown in Figure 5.2. The  $^{13}\text{C}$  channel consists of the relaxation delay, an RF pulse, and the acquisition time during which the signal is recorded. The pulse angle is shown to be 30 degrees. The two parameters, D1 and P1, correspond to the length of the relaxation delay, and the length of the 90 degree RF pulse, respectively. The  $^1\text{H}$  channel consists of two decoupling pulses which can be power gated. The first pulse, an NOE build up pulse during the relaxation delay may be of lower power than the second pulse during the acquisition which is the true decoupling pulse. This can be useful to avoid RF heating on salty samples or probes where a higher decoupling power can be problematic.

Figure 5.2

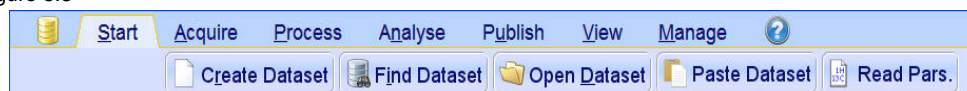


The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

## 5.2.2 Experiment set up

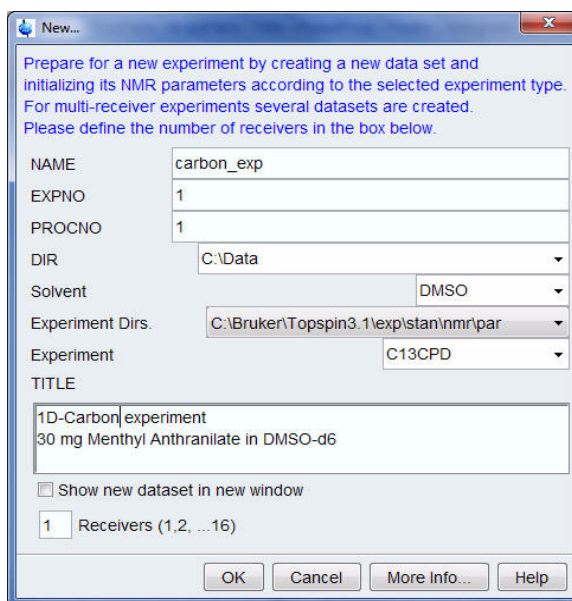
1. Click on the '**Start**' tab in the TopSpin Menu bar

Figure 5.3



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 5.4

The image shows the 'New...' dialog box in TopSpin. It contains the following fields and values:

- NAME: carbon\_exp
- EXPNO: 1
- PROCNO: 1
- DIR: C:\Data
- Solvent: DMSO
- Experiment Dirs.: C:\Bruker\Topspin3.1\exp\stan\nmr\par
- Experiment: C13CPD
- TITLE: 1D-Carbon experiment  
30 mg Menthyl Anthranilate in DMSO-d6
- Show new dataset in new window
- 1 Receivers (1,2, ...16)

Buttons at the bottom include OK, Cancel, More Info..., and Help.

NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 5.4 above. Click on the down arrow button to browse for a specific directory.

4. Click on **OK**
5. Select the '**AcquPars**' tab by clicking on it
6. Make the following change  
NS = 128
7. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 5.5



8. Select  **Sample** by clicking on it

Figure 5.6



9. Select **'ej'** by clicking on it

---

NOTE: Wait till the sample lift air is turned on and remove any sample which may have been in the magnet.

---

10. Place the sample on to the top of the bore tube

11. Select  **Sample** by clicking on it

Figure 5.7



12. Select **'ij'** by clicking on it

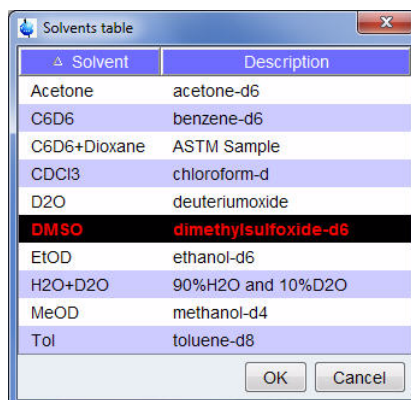
---

NOTE: Wait till the sample is lowered down in to the probe and the lift air is turned off. A clicking sound may be heard.

---

13. Select  **Lock** by clicking on it

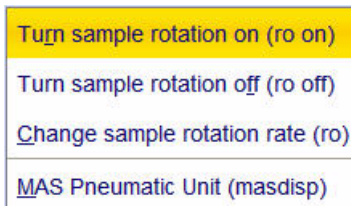
Figure 5.8



14. Select **'DMSO'** by clicking on it

15. Select  **Spin** by clicking on it

Figure 5.9



16. Select '**ro on**' by clicking on it
17. Select  Shim by clicking on it
18. Select  Prosol by clicking on it

---

NOTE: This will load the pulse width and power levels in to the parameter set.

---

## 5.2.3 Acquisition

1. Select  Gain by clicking on it
2. Select  Go by clicking on it

---

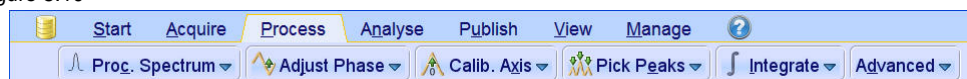
NOTE: The experiment will take ~7 minutes, using 128 scans.

---

## 5.2.4 Processing

1. Click on the '**Process**' tab in the TopSpin Menu bar

Figure 5.10



2. Select  Proc. Spectrum by clicking on it

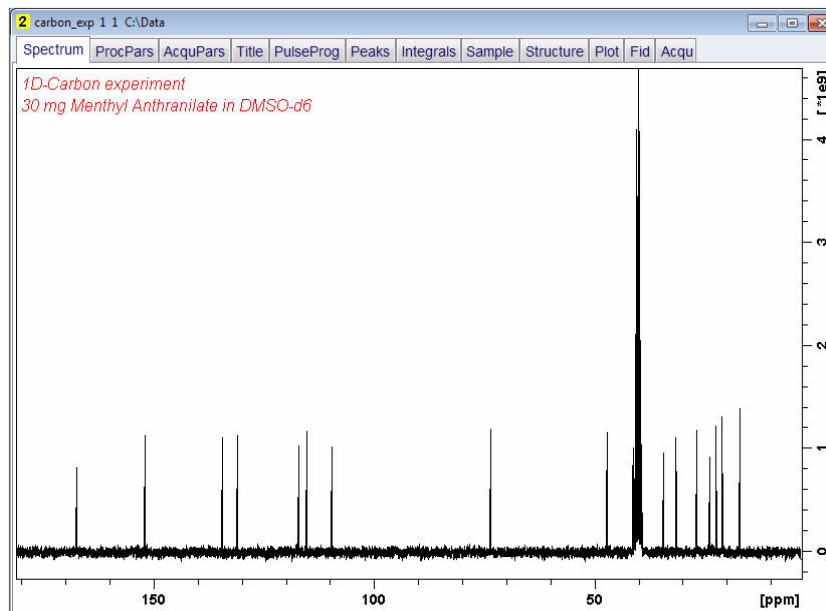
---

NOTE: This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**' button.

---

3. Expand the spectrum to include all peaks

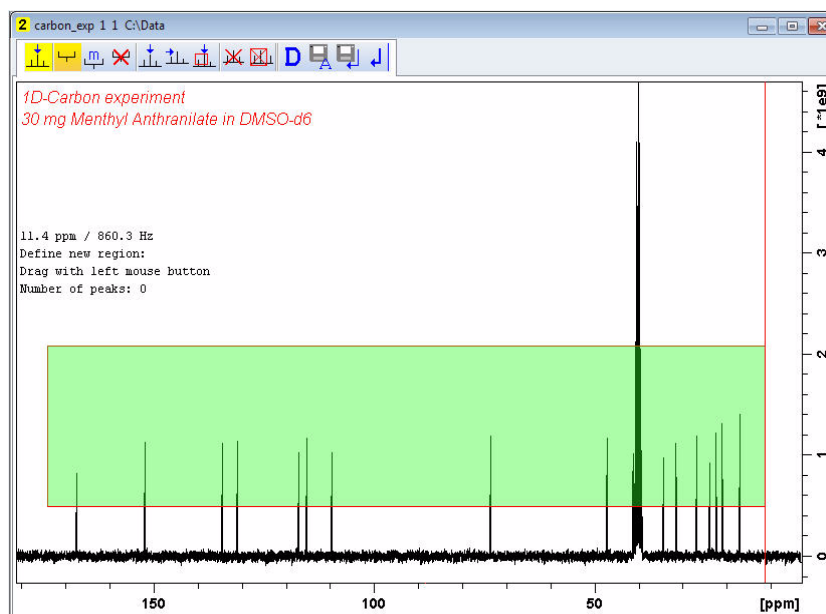
Figure 5.11




4. Select  **Pick Peaks** by clicking on it

5. Click the left mouse button and drag the cursor line from left to the right side of the spectrum

Figure 5.12



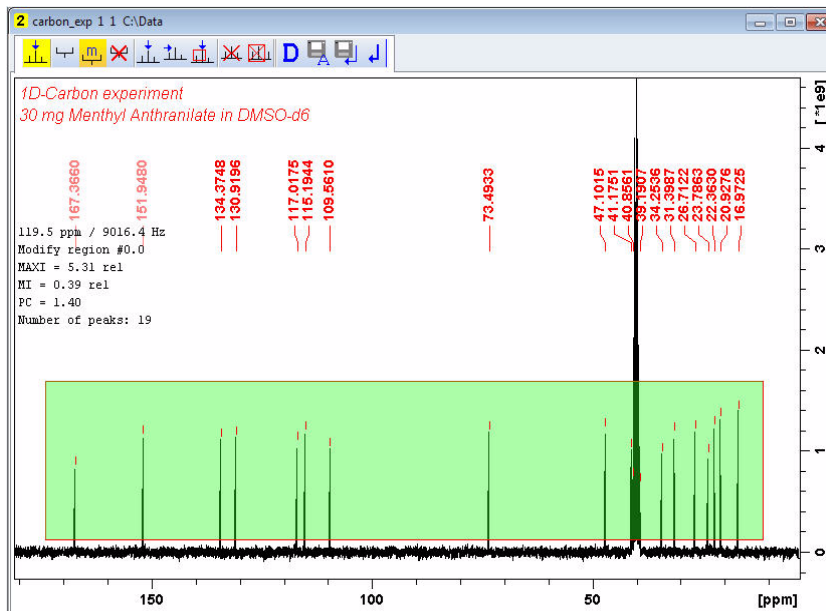
6. Click on  to manually adjust the minimum and maximum intensity levels

7. Click on the bottom line of the region box with the left mouse button and drag the line above the noise level, to set the minimum peak picking level

8. Click on the top line of the region box with the left mouse button and drag the line

below unwanted peaks e.g. solvent peaks, to set the maximum peak picking level

Figure 5.13




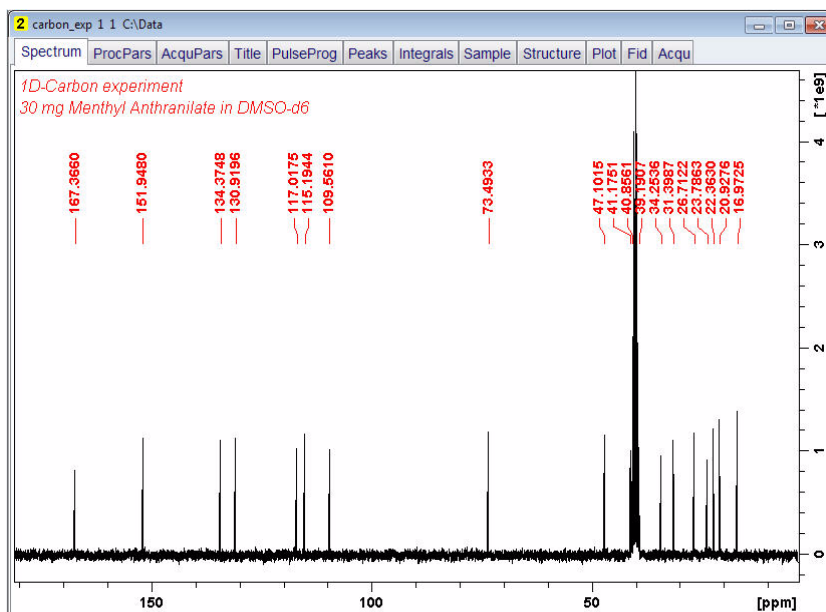
9. Click on  to store the peak picking values

Figure 5.14



NOTE: To display the peak picking labels, right click inside the spectrum window and select 'Spectra Display Preferences' by clicking on it. In the 'Spectrum components' enable 'Peak labels' and 'Peak annotations'. Click 'Apply' and click on 'Close'

## 5.2.5 Plotting the 1D Carbon spectrum


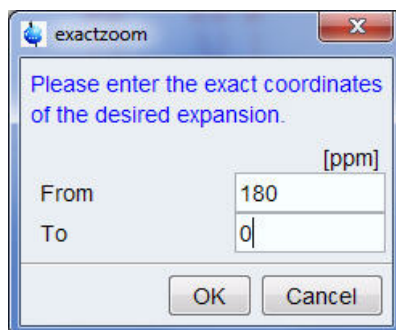
1. Expand the spectrum (all peaks in display)
2. Click on 
3. Type the following F1 [ppm] values:  
From = 180  
To = 0

Figure 5.15




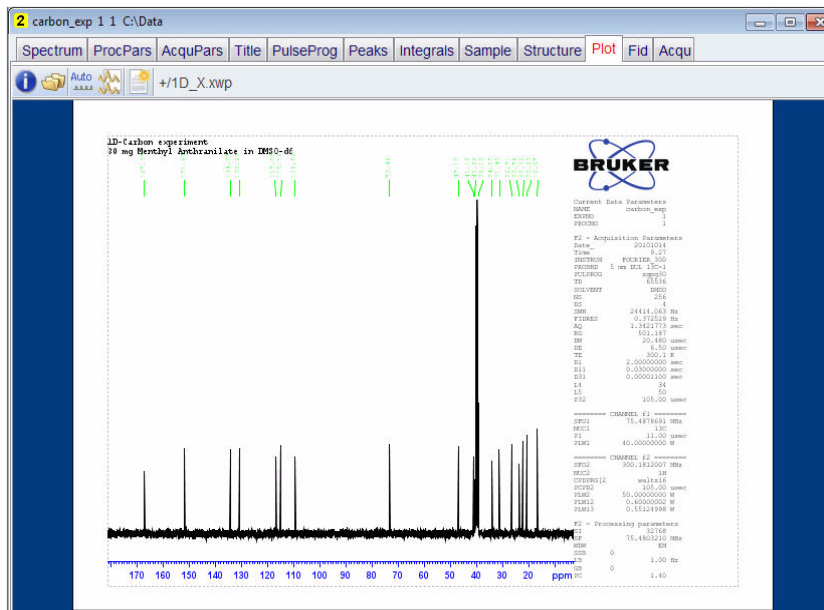
4. Click on 
5. Click on the 'Publish' tab in the TopSpin Menu bar

Figure 5.16




6. Click on 

Figure 5.17



---

NOTE: If desired, any changes can be administered by clicking on the  icon to open the Plot Editor.

---

7. Click on the  to plot the spectrum

## 5.2.6 Observations

---

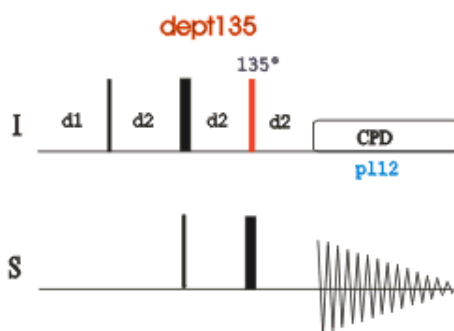
## 5.3 DEPT-135 Experiment

### 5.3.1 Introduction

DEPT (Distortion less Enhancement by Polarization Transfer) is a polarization transfer technique used for the observation of nuclei with a small gyro magnetic ratio, which are J-coupled to  $^1\text{H}$  (most commonly  $^{13}\text{C}$ ). DEPT is a spectral editing sequence, that is, it can be used to generate separate  $^{13}\text{C}$  sub spectra for methyl ( $\text{CH}_3$ ), methylene ( $\text{CH}_2$ ), and methine ( $\text{CH}$ ) signals. DEPT makes use of the generation and manipulation of multiple quantum coherence to differentiate between the different types of  $^{13}\text{C}$  signals. Quaternary carbons are missing a direct bond proton, and as a result are absent from all DEPT spectra.

Section 5.3.2 describes the acquisition and processing of a one-dimensional  $^{13}\text{C}$ -DEPT135 NMR spectrum. The standard Bruker parameter set **C13DEPT135**, includes the pulse sequence **dept135**, shown in Figure 6.17. The  $^{13}\text{C}$  channel consists of the relaxation delay, a 90 degree RF pulse, an editing delay D2 followed by an 180 degree RF pulse and the acquisition time during which the signal is recorded. The editing delay D2 is  $1/2 \cdot J(\text{XH})$ . The  $^1\text{H}$  channel consists of three pulses, a 90 degree, a 180 degree, followed by a 135 degree RF pulse and are separated by the editing delay D2. The final 135 degree  $^1\text{H}$  pulse selects the  $\text{CH}_3$ ,  $\text{CH}_2$  or  $\text{CH}$  signals. The protons are decoupled during the acquisition period.

Figure 5.18



The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

### 5.3.2 Experiment set up

NOTE: This experiment usually follows a regular  $^1\text{H}$  decoupled  $^{13}\text{C}$  experiment. The result of a DEPT-135 experiment shows only the protonated carbons with the  $\text{CH}$  and  $\text{CH}_3$  as positive and the  $\text{CH}_2$  as negative signals.

1. Click on the '**Start**' tab in the TopSpin Menu bar

Figure 5.19



2. Select  by clicking on it
3. Enter the following information in to the 'New' window

Figure 5.20

Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. For multi-receiver experiments several datasets are created. Please define the number of receivers in the box below.

NAME	carbon_exp
EXPNO	2
PROCNO	1
DIR	C:\Data
Solvent	DMSO
Experiment Dirs.	C:\Bruker\Topspin3.1\exp\stan\nmr\par
Experiment	C13DEPT135
TITLE	1D-C13 DEPT135 experiment 30 mg Menthyl Anthranilate in DMSO-d6
<input type="checkbox"/> Show new dataset in new window	
<input type="text" value="1"/> Receivers (1,2, ...,16)	

OK Cancel More Info... Help

---

NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 6.4 above. Click on the down arrow button to browse for a specific directory.

---


4. Click on 
5. Select the '**AcquPars**' tab by clicking on it
6. Make the following change  
NS = 64
7. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 5.21



8. Select  by clicking on it

---

NOTE: This will load the pulse width and power levels in to the parameter set.

---

## 5.3.3 Acquisition

1. Select  **Gain** by clicking on it
2. Select  **Go** by clicking on it

NOTE: The experiment will take ~4 minutes, using 64 scans.

## 5.3.4 Processing

1. Click on the '**Process**' tab in the TopSpin Menu bar

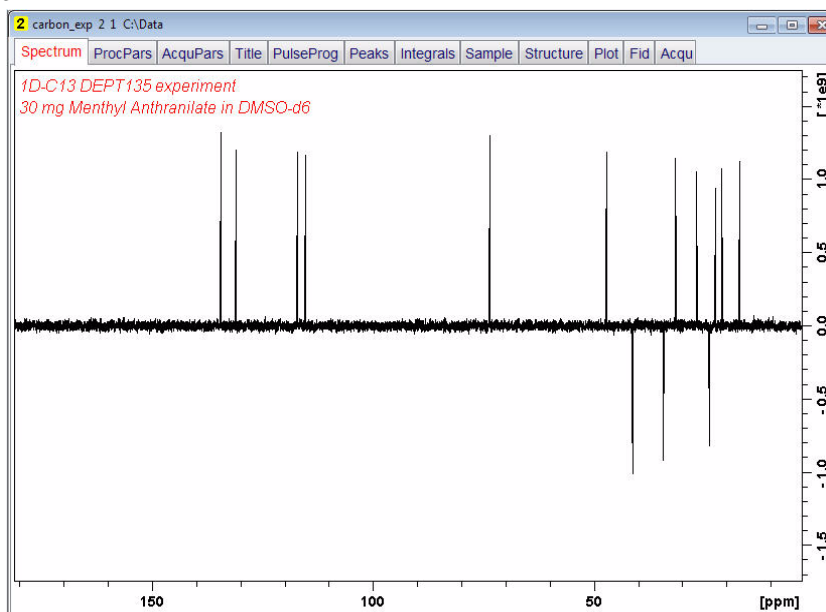
Figure 5.22



2. Select  **Proc. Spectrum** by clicking on it

NOTE: This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**' button. Due to the fact that a DEPT135 spectrum contains negative and positive peaks, there is the possibility of getting phase results that are 180 degrees off. In this case, click on the '**Adjust Phase**' button to enter the manual phase routine and reverse the spectrum by clicking on the '**180**' icon.

Figure 5.23



### 5.3.5 Plotting the Carbon DEPT135 spectrum


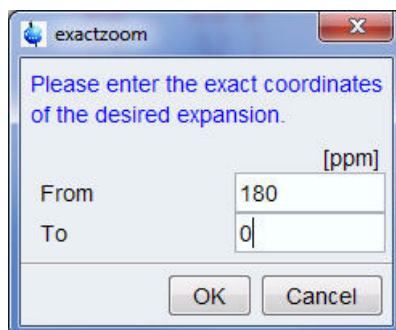
1. Expand the spectrum (all peaks in display)
2. Click on 
3. Type the following F1 [ppm] values:  
From = **180**  
To = **0**

Figure 5.24




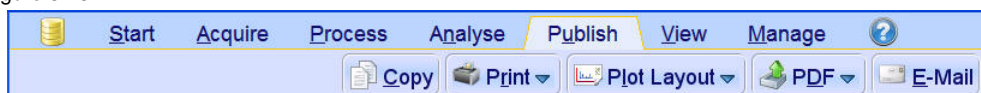
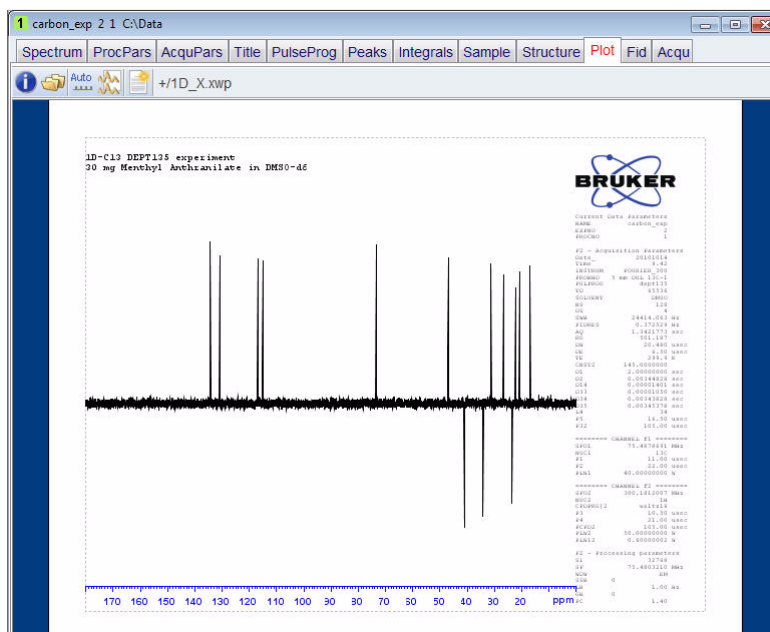
4. Click on 
5. Click on the **'Publish'** tab in the TopSpin Menu bar

Figure 5.25




6. Click on 

Figure 5.26



---

NOTE: If desired, any changes can be administered by clicking on the  icon to open the Plot Editor.

---

7. Click on the  to plot the spectrum

## 5.3.6 Observations

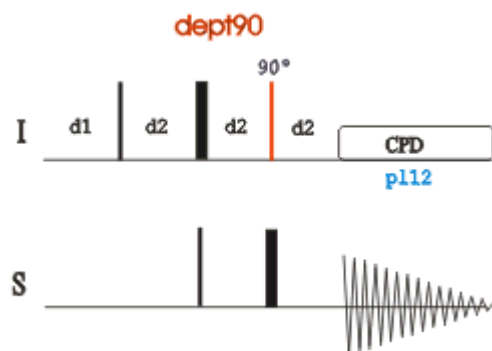
---

## 5.4 DEPT-90 Experiment

### 5.4.1 Introduction

Section 6.4.2 describes the acquisition and processing of a one-dimensional  $^{13}\text{C}$ -DEPT90 NMR spectrum. The standard Bruker parameter set **C13DEPT90**, includes the pulse sequence **dept90**, shown in Figure 5.23. The  $^{13}\text{C}$  channel consists of the relaxation delay, a 90 degree RF pulse, an editing delay D2 followed by a 180 degree RF pulse and the acquisition time during which the signal is recorded. The editing delay D2 is  $1/2 \cdot J(\text{XH})$ . The  $^1\text{H}$  channel consists of three pulses, a 90 degree, a 180 degree, followed by a 90 degree RF pulse and are separated by the editing delay D2. The final 90 degree  $^1\text{H}$  pulse selects the CH signals only. The protons are decoupled during the acquisition period.

Figure 5.27



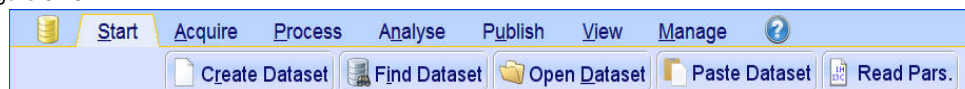
The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

### 5.4.2 Experiment set up

NOTE: The DEPT90 experiment usually follows a regular  $^1\text{H}$  decoupled  $^{13}\text{C}$  experiment and a DEPT-135 experiment. It is used to assign the methine (CH) signals.

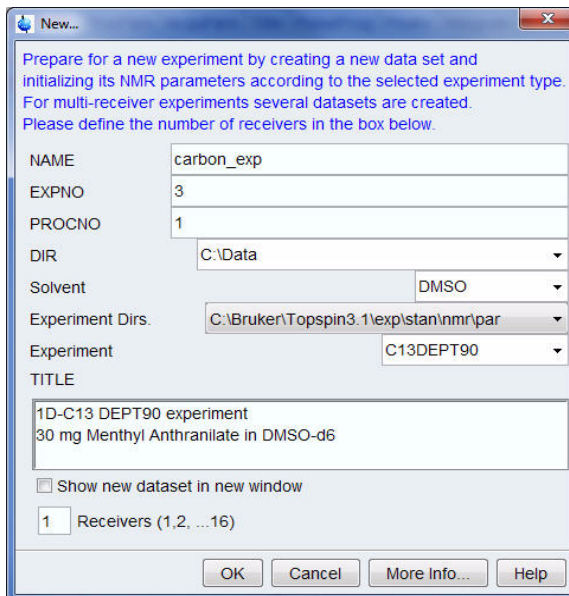
1. Click on the **'Start'** tab in the TopSpin Menu bar

Figure 5.28



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 5.29



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 5.25 above. Click on the down arrow button to browse for a specific directory.


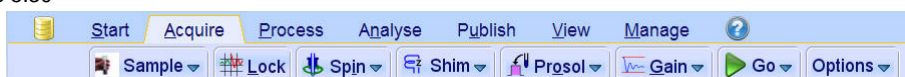
4. Click on 
5. Select the '**AcquPars**' tab by clicking on it
6. Make the following change  
NS = 64
7. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 5.30



8. Select  by clicking on it

NOTE: This will load the pulse width and power levels in to the parameter set.

### 5.4.3 Acquisition

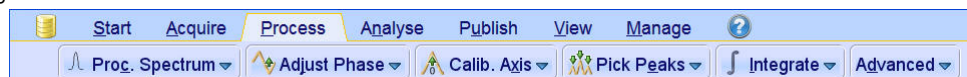
1. Select  by clicking on it
2. Select  by clicking on it

NOTE: The experiment will take ~4 minutes, using 64 scans.

## 5.4.4 Processing

1. Click on the **'Process'** tab in the TopSpin Menu bar

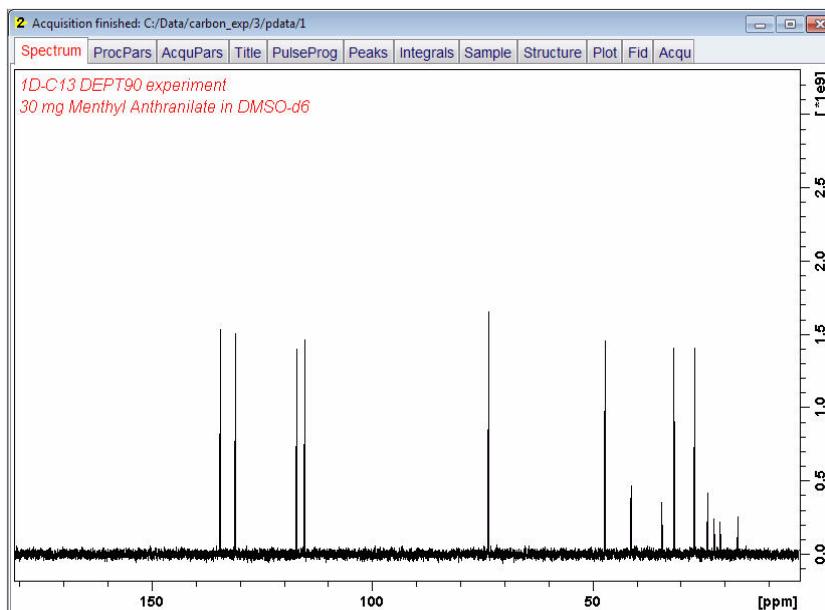
Figure 5.31




2. Select  **Proc. Spectrum** by clicking on it

NOTE: This executes a processing program including commands such as an exponential window function **'em'**, Fourier transformation **'ft'**, an automatic phase correction **'apk'** and a baseline correction **'abs'**. Other options are available by clicking on the down arrow inside the **'Proc. Spectrum'** button.

Figure 5.32



## 5.4.5 Plotting the 1D Carbon spectrum

1. Expand the spectrum (all peaks in display)
2. Click on 
3. Type the following F1 [ppm] values:

From = **180**

To = **0**



## 5.4.6 Observations

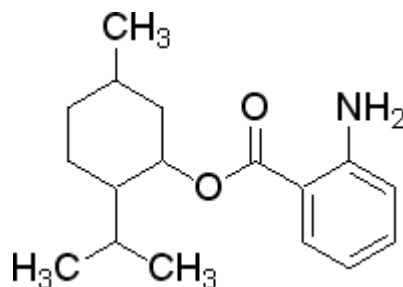
---

# 6 2-D Heteronuclear experiments

## 6.1 Sample

A sample of 30mg Menthyl Anthranilate in DMSO-d6 is used for all experiments in this chapter

Figure 6.1

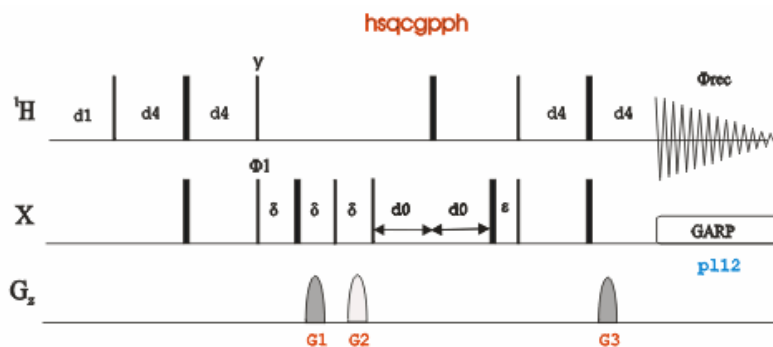


## 6.2 2D HSQC

### 6.2.1 Introduction

The **ge-2D HSQC experiment** is the gradient-enhanced version of the conventional HSQC experiment in which coherence selection is achieved by means of PFG. Thus, clean 2D HSQC spectra can be recorded in a single scan per  $t_1$  increment without need for phase cycle when sample concentration is high. Other advantages are the optimal dynamic range, improved water and artefact suppression, and reduced  $t_1$  noise in the minimally required experiment time. The HSQC experiment allows to trace out directly bonded  $^1\text{H-X}$  pairs via the large  $^1J_{\text{HX}}$  coupling constant. The sequence is shown in Figure 6.2.

Figure 6.2

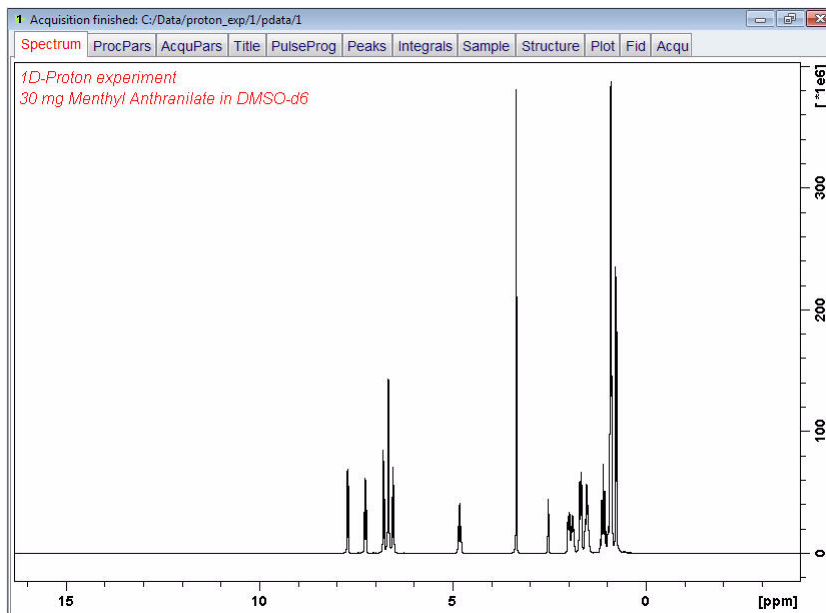


The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example,  $d_1$  is typically a few seconds while  $p_1$  is typically a few microseconds in length.

## 6.2.2 Preparation experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 3, 1-D Proton experiment, Paragraph 3.2.2 Experiment setup** through **3.2.4 Processing**.

Figure 6.3



## 6.2.3 Setting up the HSQC experiment

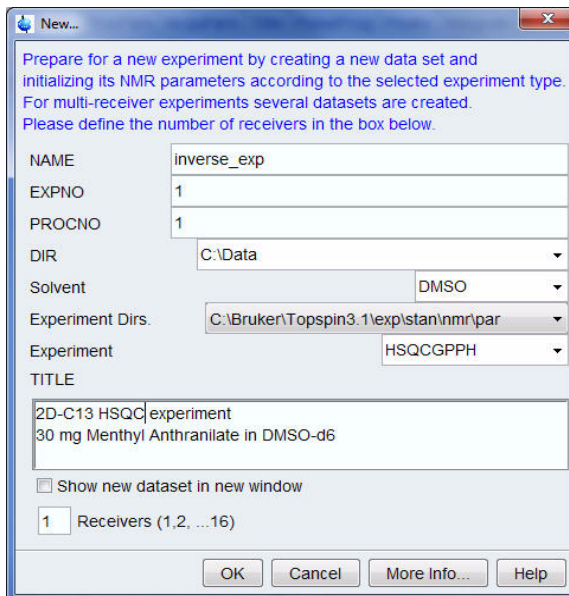
1. Click on the **'Start'** tab in the TopSpin Menu bar

Figure 6.4



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 6.5



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 6.5 above. Click on the down arrow button to browse for a specific directory.


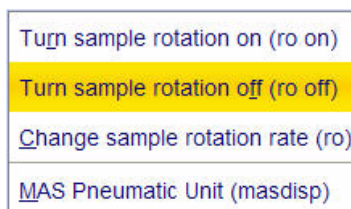
4. Click on 
5. Click on the 'Acquire' tab in the TopSpin menu bar

Figure 6.6



6. Select  by clicking on it

Figure 6.7



7. Select 'ro off' by clicking on it

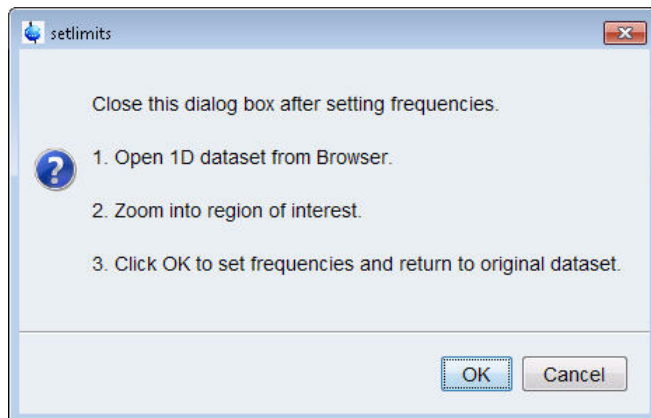
NOTE: 2-D experiments should always be run without rotation.

8. Select  by clicking on it

NOTE: This will load the pulse width and power levels in to the parameter set.

9. Select  by clicking on it

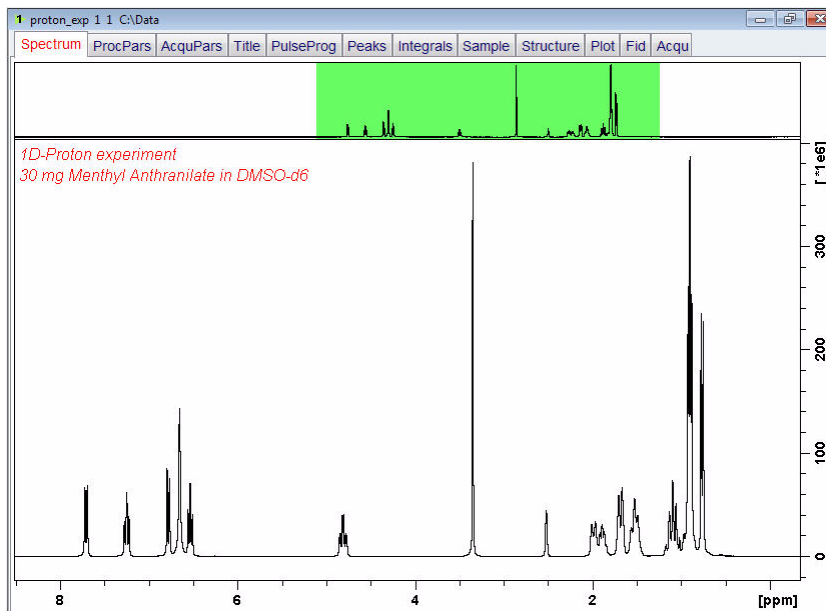
Figure 6.8



10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. **Inverse\_exp 1**) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window

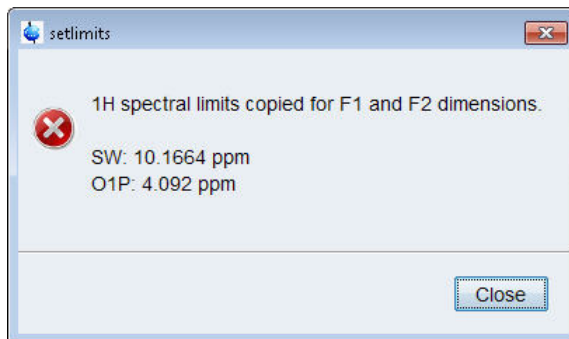
11. Expand the spectrum to display all peaks, leaving ca. 0.2 ppm of baseline on either side of the spectrum

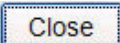
Figure 6.9



12. Click on  to assign the new limit

Figure 6.10

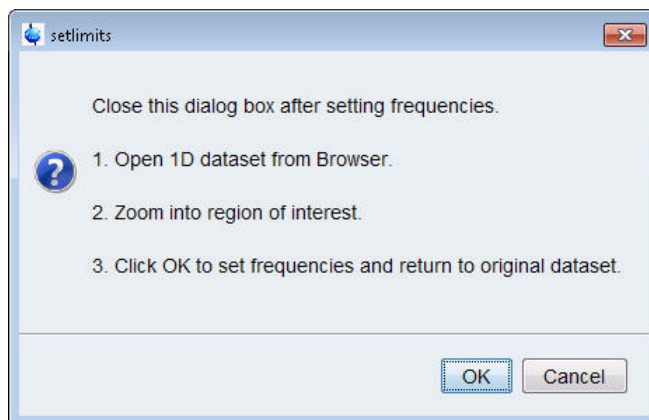


13. Click on 

NOTE: The display changes back to the 2D data set. The parameter set **HSQCGPPH** has a fixed F1 sweep width of 165 ppm and it is big enough to cover the protonated resonances for a broad range of samples. If desired, changes to the F1 sweep width can be done by using the '**Set\_limits**' button for a second time. In this case a 1-D **C13DEPT45** or **C13DEPT135** experiment on the same sample has to be observed. As an example to set the F1 limit, follow the steps below.

14. Select  by clicking on it

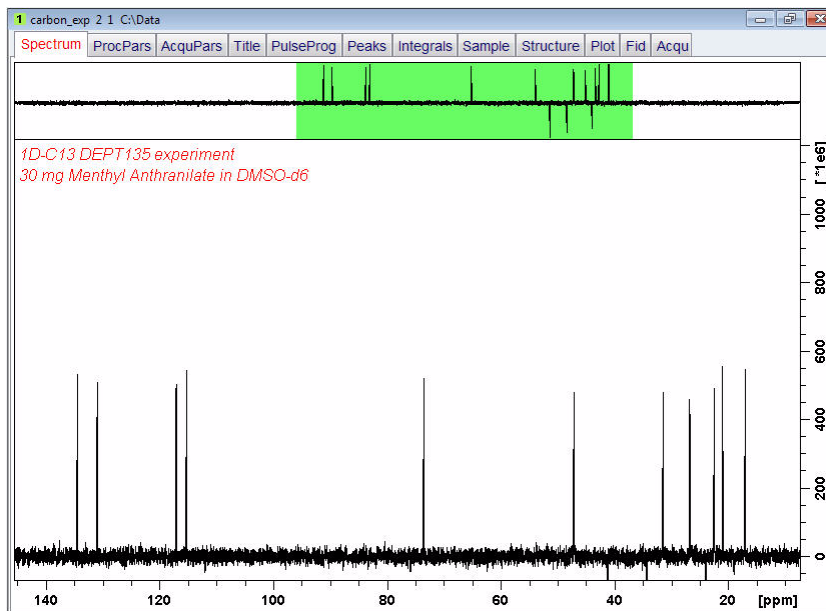
Figure 6.11



15. To open the 1D C13DEPT spectrum, right click on the dataset name in the browser window (e.g. **Carbon\_exp 2**) and select 'Display' or click and hold the left mouse button for dragging the 1D C13DEPT dataset in to the spectrum window

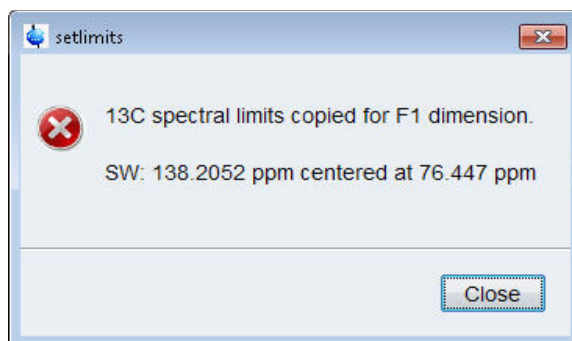
16. Expand the spectrum to display all peaks, leaving ca. 2 ppm of baseline on either side of the spectrum

Figure 6.12N



17. Click on  to assign the new limit

Figure 6.13



18. Click on

## 6.2.4 Acquisition

1. Select  by clicking on it

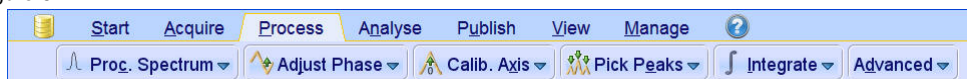
2. Select  by clicking on it

NOTE: The experiment will take ~15 minutes, using the default number of 2 scans and 256 increments.

## 6.2.5 Processing

1. Click on the **'Process'** tab in the TopSpin Menu bar

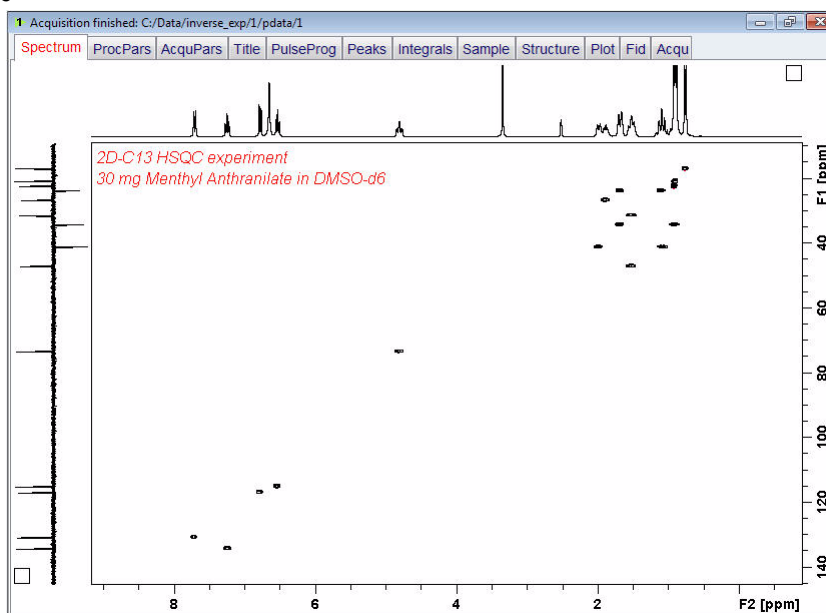
Figure 6.14



2. Select  by clicking on it

NOTE: This executes a standard processing program **proc2**. To configure this program or select the right options, click on the down arrow inside the **'Proc. Spectrum'** button. Since this is a phase sensitive experiment the phase correction **apk2d** has to be enabled.

Figure 6.15



5. Click on the **'Publish'** tab in the TopSpin Menu bar

Figure 6.16



6. Click on  to print the screen view

NOTE: The F1 projection by default does not appear if the plot editor (XWINPLOT) is used to plot the spectrum. To add the F1 projection one has to create a new template.

### 6.2.6 Observations

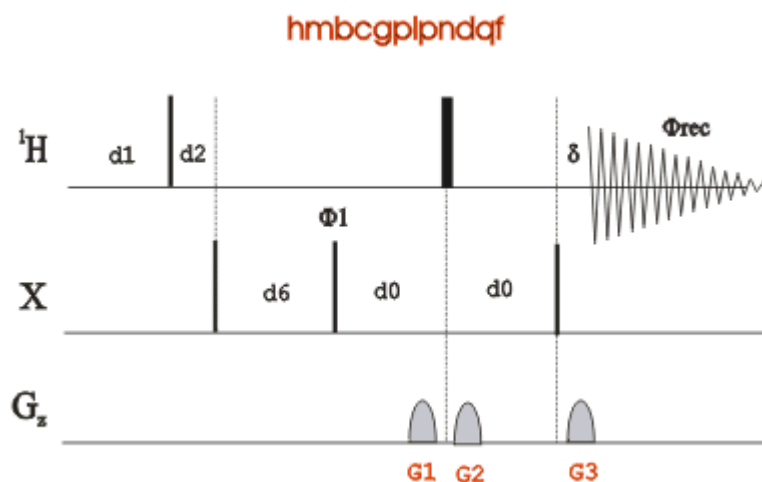
---

## 6.3 2D HMBC experiment

### 6.3.1 Introduction

HMBC (Heteronuclear Multiple Bond Correlation) spectroscopy is a modified version of HMQC suitable for determining long-range  $^1\text{H}$ - $^{13}\text{C}$  connectivity. Since it is a long-range chemical shift correlation experiment the pulse program contains a low pass filter to suppress the one bond correlation and is a gradient-selected version which is not phase-sensitive. The experiment is performed without  $^{13}\text{C}$  decoupling to distinguish signals coming from the one bond coupling. The standard Bruker parameter set **HMBCGP** is used. The graphical display of the pulse program **hmbcgp1pndqf** is shown in Figure 6.16

Figure 6.17

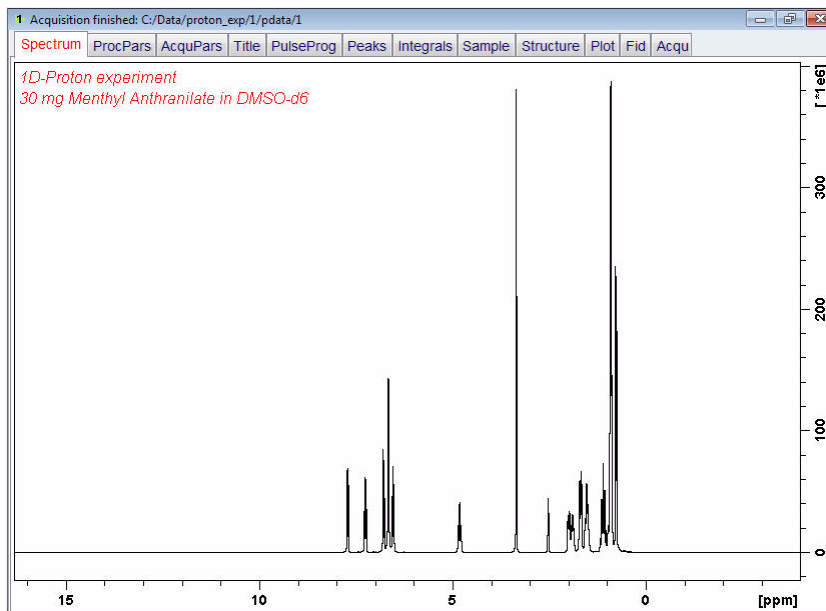


The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example,  $d1$  is typically a few seconds while  $p1$  is typically a few microseconds in length.

### 6.3.2 Preparation experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 3, 1-D Proton experiment, Paragraph 3.2.2 Experiment setup** through **3.2.4 Processing**.

Figure 6.18



## 6.3.3 Setting up the HMBC experiment

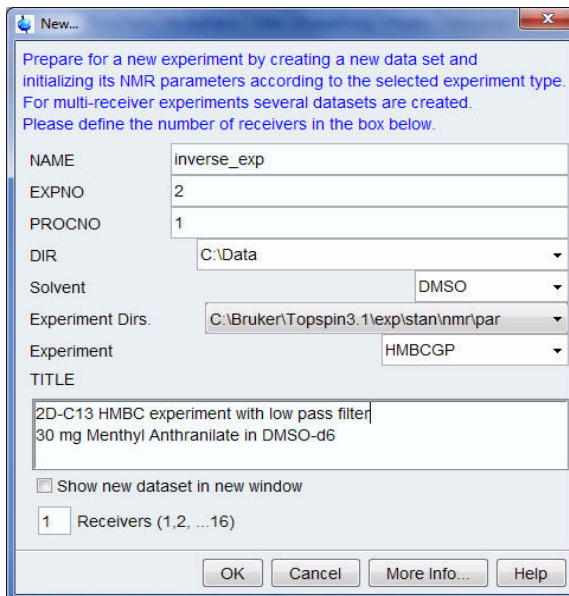
1. Click on the 'Start' tab in the TopSpin Menu bar

Figure 6.19



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 6.20



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 6.19 above. Click on the down arrow button to browse for a specific directory.


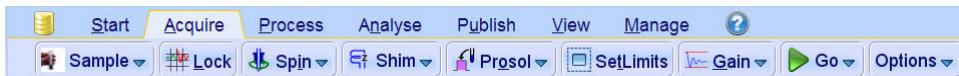
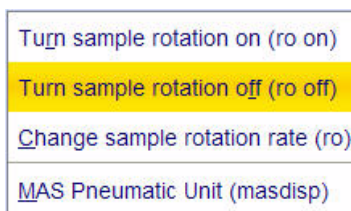
4. Click on 
5. Click on the 'Acquire' tab in the TopSpin menu bar

Figure 6.21



6. Select  by clicking on it

Figure 6.22



7. Select 'ro off' by clicking on it

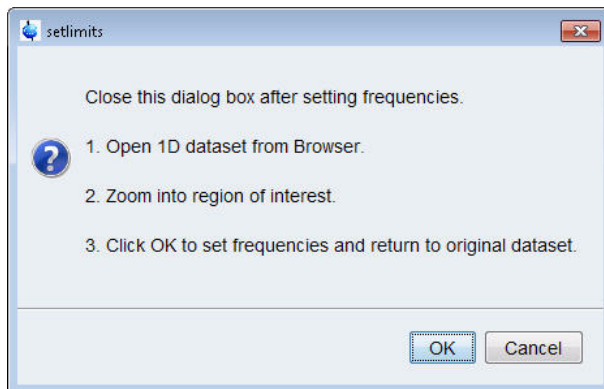
NOTE: 2-D experiments should always be run without rotation.

8. Select  by clicking on it

NOTE: This will load the pulse width and power levels in to the parameter set.

9. Select  by clicking on it

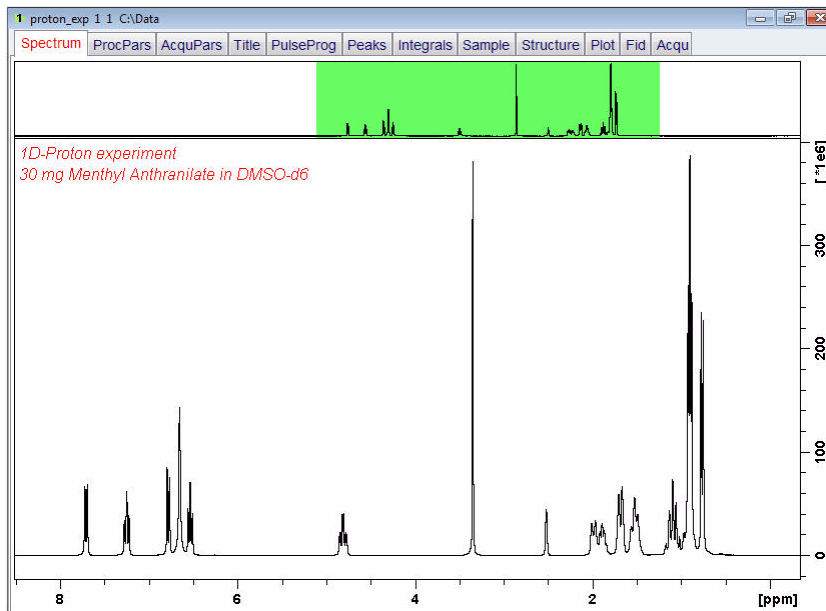
Figure 6.23



10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. **Inverse\_exp 1**) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window

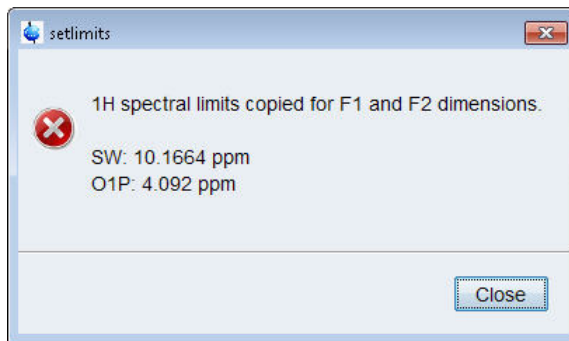
11. Expand the spectrum to display all peaks, leaving ca. 0.2 ppm of baseline on either side of the spectrum

Figure 6.24



12. Click on  to assign the new limit

Figure 6.25

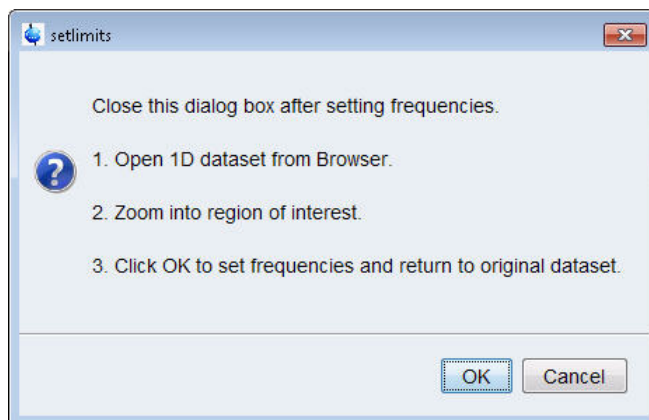


13. Click on 

NOTE: The display changes back to the 2D data set. The parameter set **HMBCGP** has a fixed F1 sweep width of 222 ppm and it is big enough to cover all Carbon resonances for a broad range of samples. If desired, changes to the F1 sweep width can be done by using the 'Set\_limits' button for a second time. In this case a 1-D **C13CPD** experiment on the same sample has to be observed. As an example to set the F1 limit, follow the steps below.

14. Select  by clicking on it

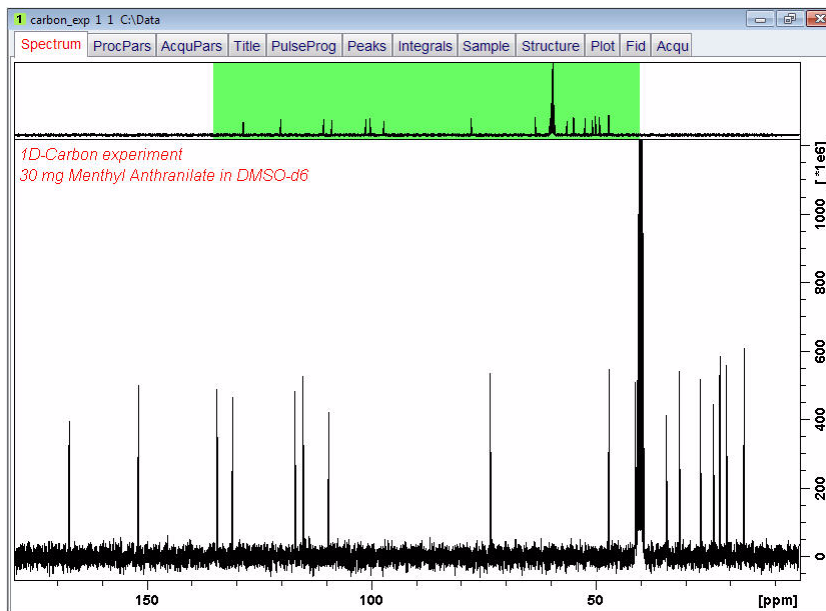
Figure 6.26



15. To open the 1D C13DEPT spectrum, right click on the dataset name in the browser window (e.g. **Carbon\_exp 1**) and select 'Display' or click and hold the left mouse button for dragging the 1D C13DEPT dataset in to the spectrum window

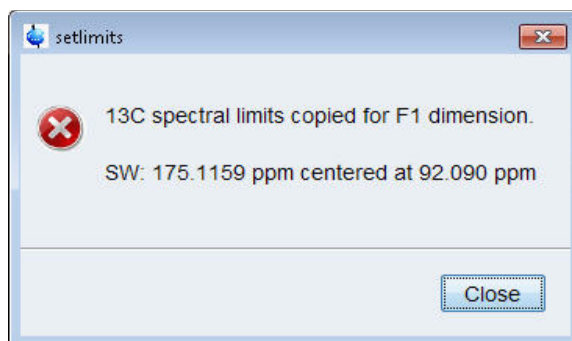
16. Expand the spectrum to display all peaks, leaving ca. 2 ppm of baseline on either side of the spectrum

Figure 6.27



17. Click on  to assign the new limit

Figure 6.28



18. Click on

## 6.3.4 Acquisition

1. Select  by clicking on it
2. Select  by clicking on it

NOTE: The experiment will take ~17 minutes, using the default number of 4 scans and 128 increments.

### 6.3.5 Processing

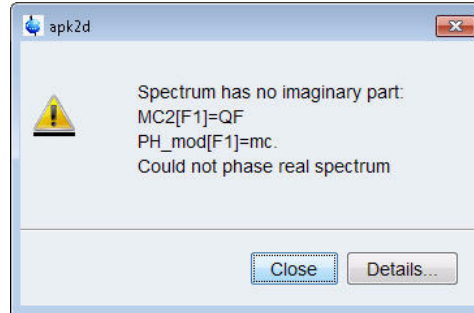
1. Click on the 'Process' tab in the TopSpin Menu bar

Figure 6.29



2. Select  by clicking on it

Figure 6.30



NOTE: This executes a standard processing program **proc2**. The message shown in Figure 6.29 pops up in case of a magnitude 2D experiment and the apk2d option is enabled. To configure the processing program follow the steps below.


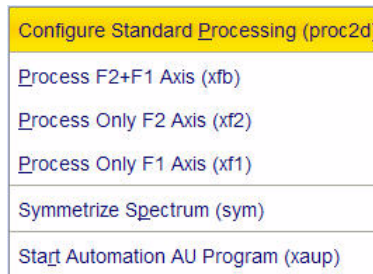
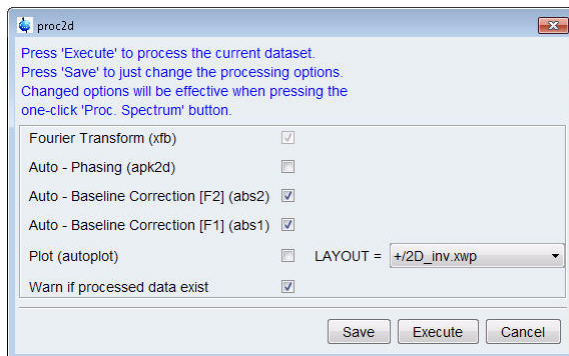
3. Click on the down arrow inside the  button

Figure 6.31



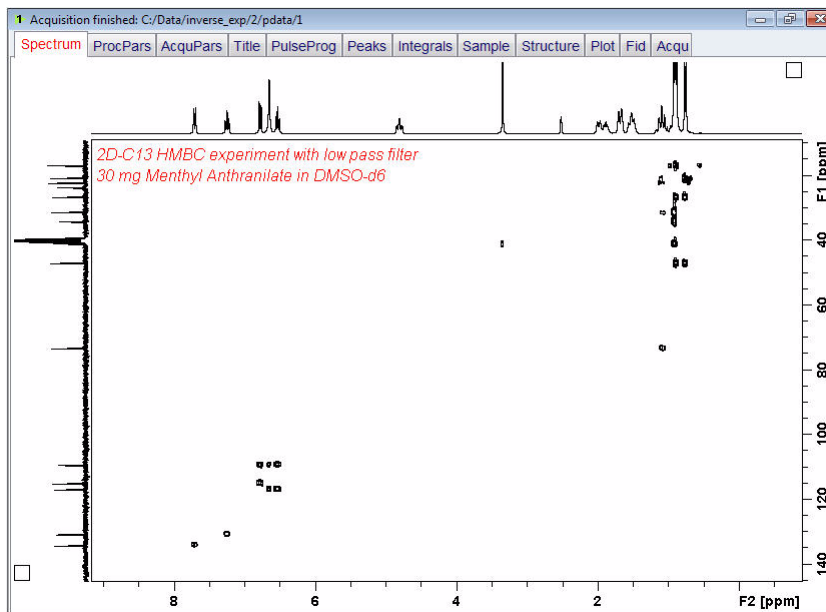
4. Select 'Configure Standard Processing' by clicking on it

Figure 6.32



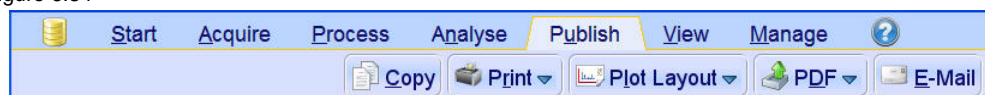
NOTE: To avoid the message shown in Figure 6.29 the option 'Auto-Phasing (apk2d)' may be disabled for magnitude like 2D experiment.

Figure 6.33



5. Click on the 'Publish' tab in the TopSpin Menu bar

Figure 6.34



6. Click on  to print the screen view

NOTE: The F1 projection by default does not appear if the plot editor (XWINPLOT) is used to plot the spectrum. To add the F1 projection one has to create a new template.

## 6.3.6 Observations

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# 7 Determination of 90 degree pulses

## 7.1 Introduction

---

This chapter describes pulse calibration procedures for  $^1\text{H}$  and  $^{13}\text{C}$ . It is assumed that the user is already familiar with acquisition and processing of simple 1D NMR spectra. **Chapter 3 (1-D Proton experiment)** and **Chapter 5 (1-D Carbon experiments)**.

---

NOTE: This chapter is intended as a guide for calibrating the 90 degree pulse of a probe or verifying the values observed by the engineer installing the instrument.

---

## 7.2 Proton 90 degree transmitter pulse

---

Standard Test Sample:

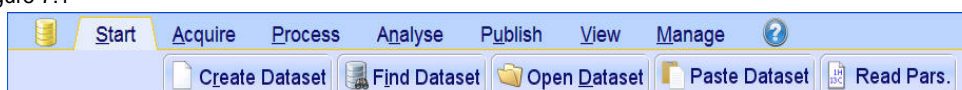
**0.1% Ethylbenzene in  $\text{CDCl}_3$**

### 7.2.1 Parameter setup

---

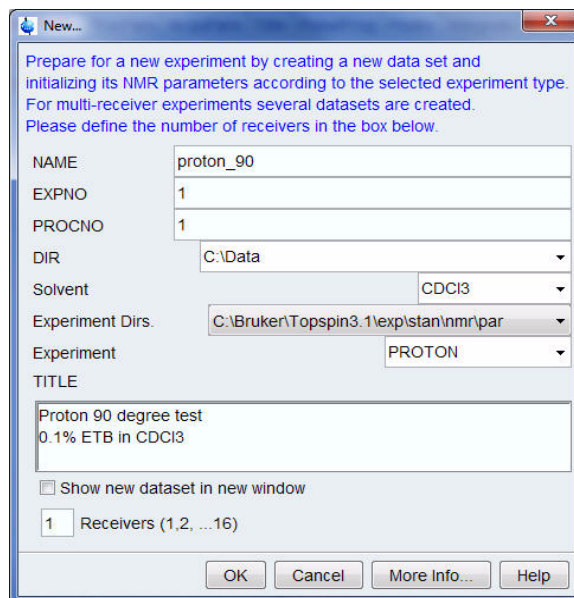
1. Click on the 'Start' tab in the TopSpin Menu bar

Figure 7.1




2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 7.2

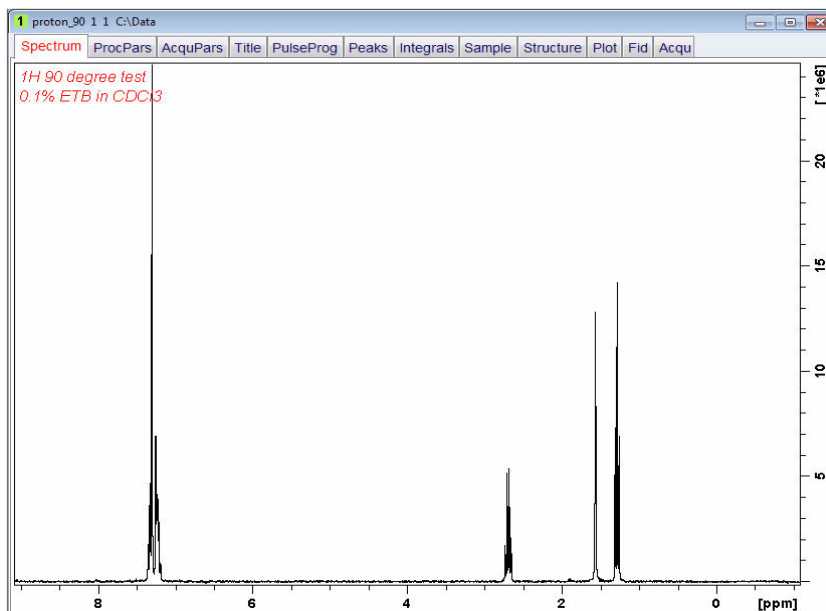


NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 7.2 above. Click on the down arrow button to browse for a specific directory.

4. Click on 

5. Run a **1D Proton** spectrum, following the instructions in **Chapter 3, 1-D Proton experiment, Paragraph 3.2.2 Experiment setup, step 5** through **3.2.4 Processing** using **CDCl<sub>3</sub>** as a lock solvent.

Figure 7.3




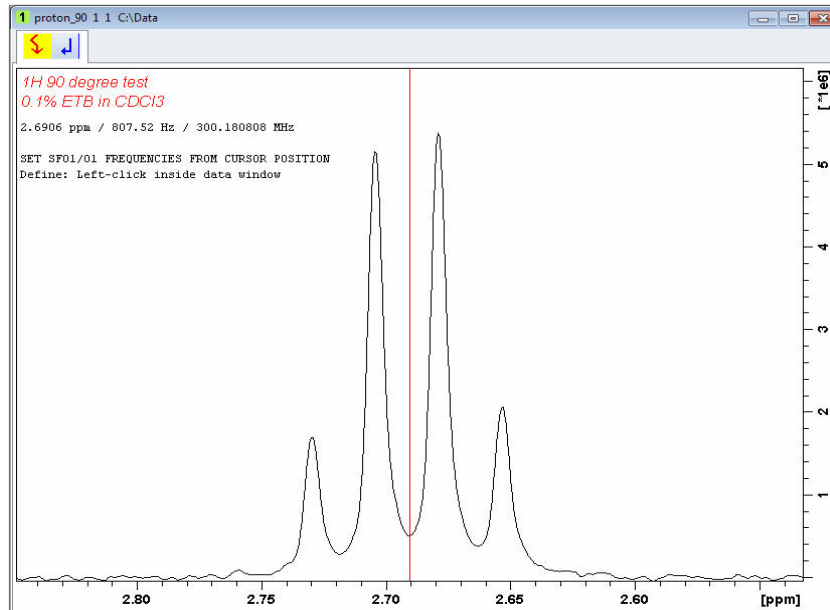
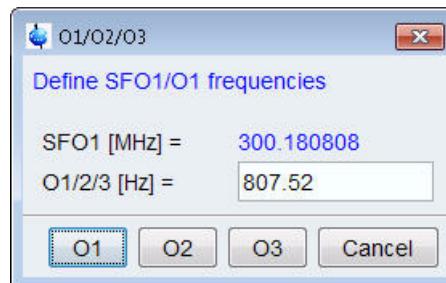
6. Expand peak at 2.7 ppm
7. Click on  to set the RF from cursor


Figure 7.4



8. Move the cursor line in to the center of the multiplet
9. Click the left mouse button to set the frequency

Figure 7.5



10. Click on 
11. Select the '**AcquPars**' tab by clicking on it
12. Make the following changes:
  - PULPROG = **zg**
  - TD = **16k**
  - SW [ppm] = **10**
  - D1 [sec] = **30**
  - DS = **0**
  - NS = **1**
13. Select the '**ProcPars**' tab by clicking on it

14 Make the following changes:

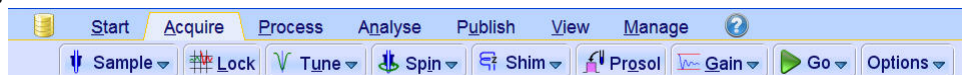
SI = **8k**

LB [Hz] = **1**

PH\_mod = select '**pk**'

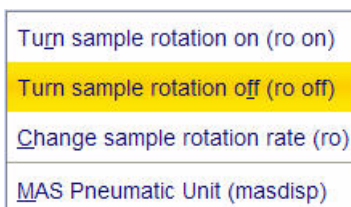
15. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 7.6



16. Select  by clicking on it

Figure 7.7



17. Select '**ro off**' by clicking on it

---

NOTE: This test should be run without rotation

---

## 7.2.2 Acquisition

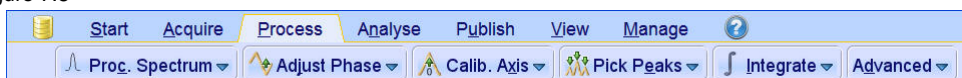
1. Select  by clicking on it

2. Select  by clicking on it

## 7.2.3 Processing

1. Click on the '**Process**' tab in the TopSpin Menu bar

Figure 7.8




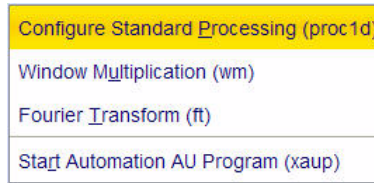
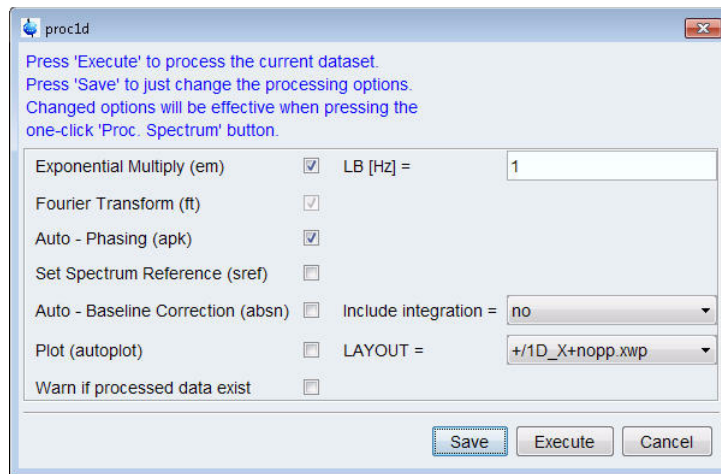
2. Click on the down arrow inside the  button

Figure 7.9



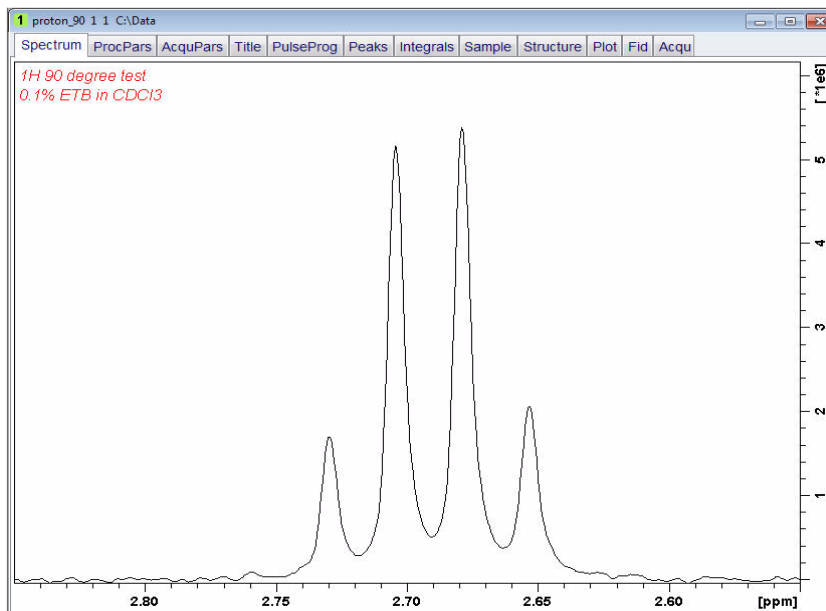
3. Select **'Configure Standard Processing'** by clicking on it
4. Deselect the following options:
  - 'Set Spectrum Reference (sref)'**
  - 'Auto-Baseline correction (absn)'**
  - 'Warn if Processed data exist'**

Figure 7.10



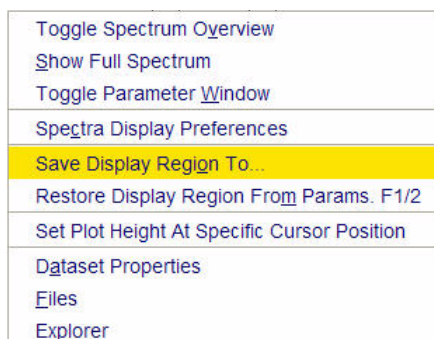
5. Click on **Execute**
6. Expand the spectrum from 2.85 ppm to 2.55 ppm

Figure 7.11



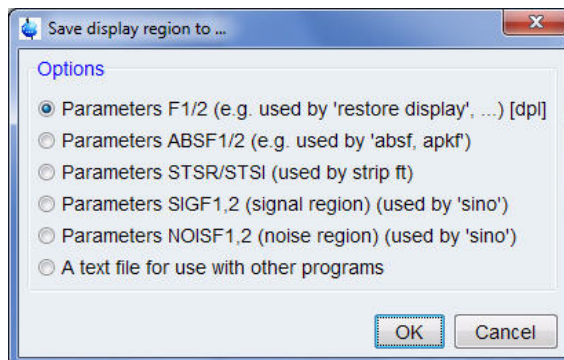
7. Click on the right mouse button inside the spectral window

Figure 7.12




8. Select 'Save Display Region to...' by clicking on it

Figure 7.13

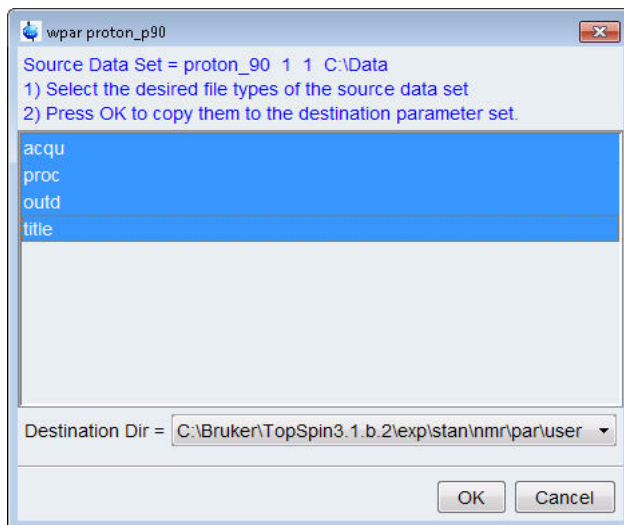


9. Enable 'Parameters F1/2'


10. Click on 

11. Type **wpar proton\_p90** to store the parameter for future use

Figure 7.14



12. Select all parameter options

13. Click on 

## 7.2.4 Determine the 90<sup>0</sup> pulse

1. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 7.15




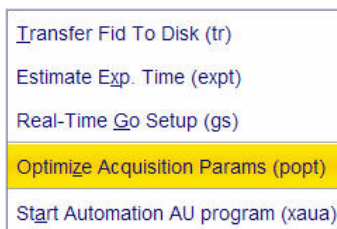
2. Click on the down arrow inside the  button

Figure 7.16



3. Select '**Optimize Acquisition Params (popt)**' by clicking on it

4. Make the following changes:

OPTIMIZE = **Step by step**

PARAMETER = **p1**

OPTIMUM = POSMAX

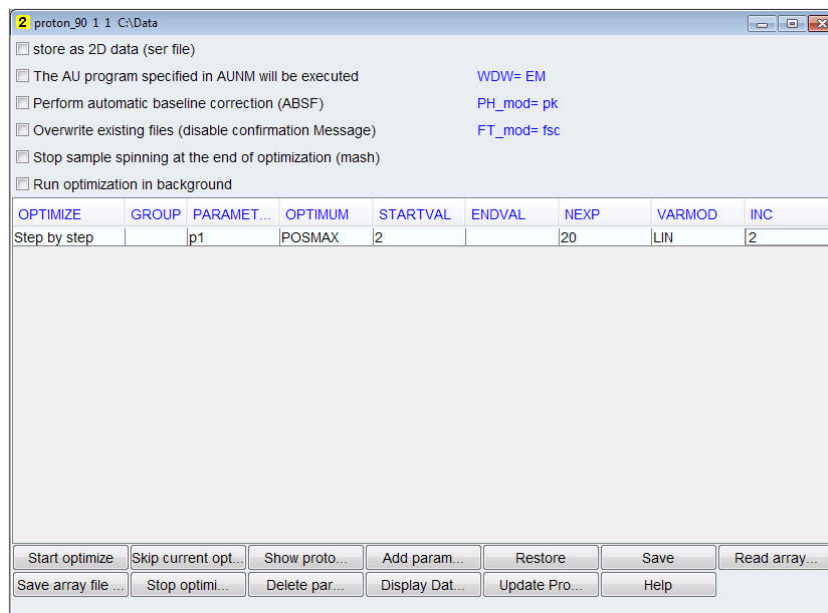
STARTVAL = 2

NEXP = 20

VARMOD = LIN

INC = 2

Figure 7.17



5. Click on 

NOTE: The ENDVAL parameter has been updated.

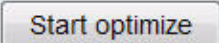
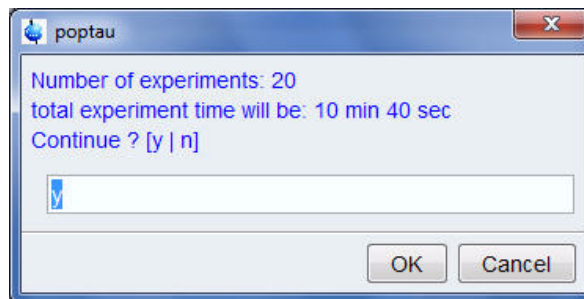

6. Click on 

Figure 7.18

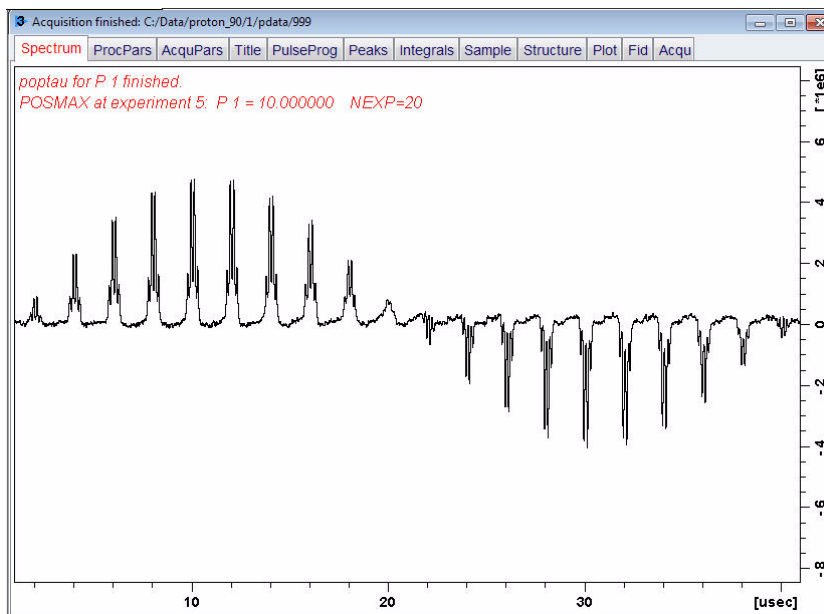


7. Enter **y** in to the poptau window

8. Click on 

NOTE: The parameter optimization starts. The spectrometer acquires and processes 20 spectra with incrementing the parameter p1 from 2 usec by 2 usec to a final value of 40 usec. For each of the 20 spectra, only the spectral region defined above is plotted, and all the spectra are plotted side-by-side in the file proton\_90/1/999 as shown in Figure 7.19.

Figure 7.19



NOTE: The POSMAX value of **p1** is displayed in the title window which is the 90 degree pulse, along with the experiment number and the NEXP value. Write this value down. To obtain a more accurate 90 degree pulse measurement, follow the steps below.

9. Close the popt setup window
10. Type **rep 1**
11. Type **p1**
12. Enter the value which corresponds to a 360 degree pulse (four times the POSMAX value)
13. Type **zg**
14. Type **efp**
15. Change p1 slightly and repeat steps 13 and 14, until the quartet undergoes a zero crossing as expected for an exact 360 degree pulse.

NOTE: The quartet signal is negative for a pulse angle slightly less than 360 degree and positive when the pulse angle is slightly more than 360 degree.

16. Simply divide the determined 360 degree pulse value by 4. This will be the exact 90 degree pulse length for the proton transmitter on the current probe

## 7.2.5 Observations

---

## 7.3 Carbon 90 degree transmitter pulse

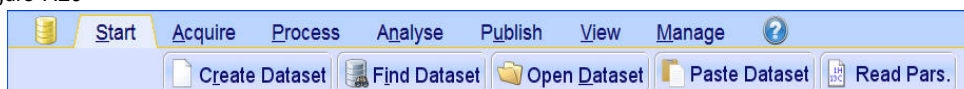
Standard Test Sample:

ASTM (60% C6D6 / 40% p-Dioxane)

### 7.3.1 Parameter setup

1. Click on the 'Start' tab in the TopSpin Menu bar

Figure 7.20



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 7.21

NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 7.21 above. Click on the down arrow button to browse for a specific directory.

4. Click on **OK**
5. Run a **1D Carbon** spectrum, following the instructions in **Chapter 5, 1-D Carbon experiments, Paragraph 5.2.2 Experiment setup, step 5** using **C6D6** as a lock solvent and making the following acquisition parameter changes:

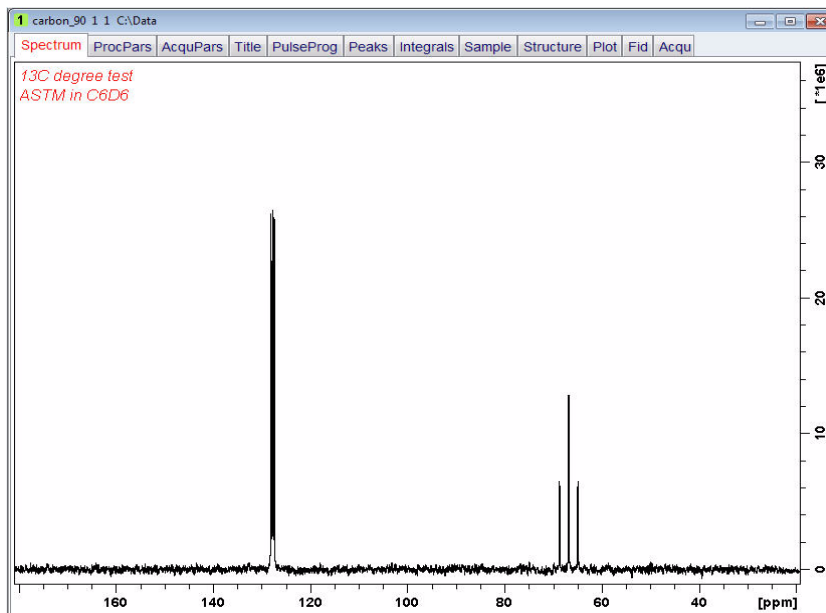
PULPROG = **zg**

DS = 0

NS = 1

## 6. Continue with 5.2.4 Processing.

Figure 7.22



## 7. Expand peak at 67 ppm


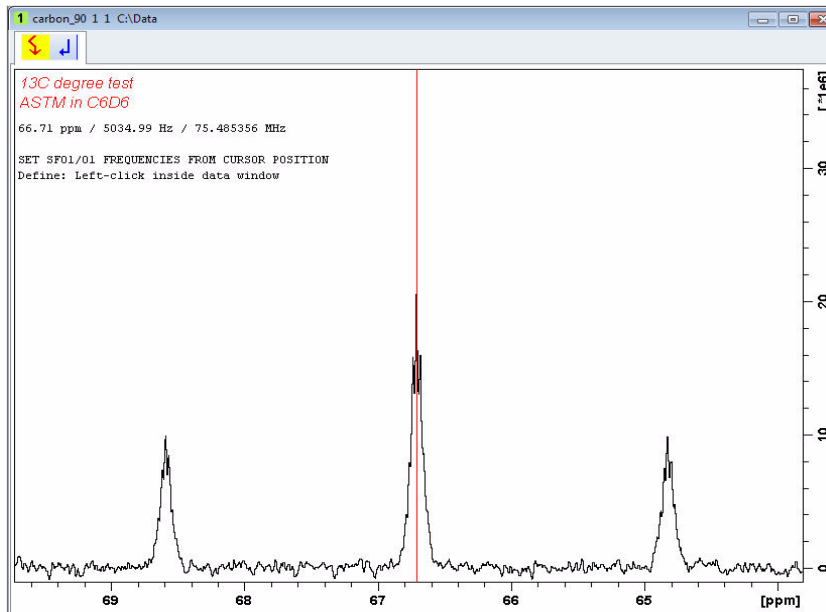
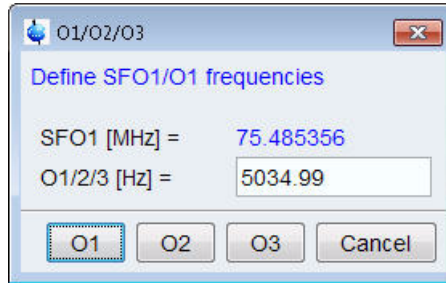
8. Click on  to set the RF from cursor

Figure 7.23



9. Click the left mouse button to set the frequency

Figure 7.24




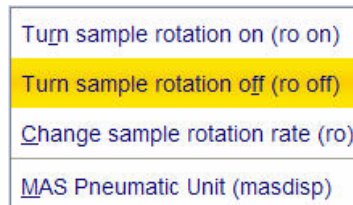
10. Click on 
11. Select the '**AcquPars**' tab by clicking on it
12. Make the following changes:
  - TD = **8k**
  - SW [Hz] = **40**
  - D1 [sec] = **60**
  - DS = **0**
  - NS = **1**
13. Select the '**ProcPars**' tab by clicking on it
- 14 Make the following changes:
  - SI = **4k**
  - LB [Hz] = **3.5**
  - PH\_mod = select '**pk**'
15. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 7.25



- Select  by clicking on it

Figure 7.26



16. Select '**ro off**' by clicking on it

---

NOTE: This test should be run without rotation

---

## 7.3.2 Acquisition

1. Select  by clicking on it
2. Select  by clicking on it

## 7.3.3 Processing

1. Click on the 'Process' tab in the TopSpin Menu bar

Figure 7.27




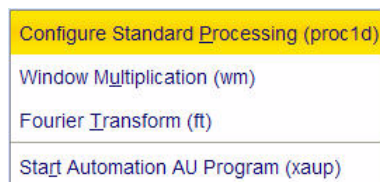
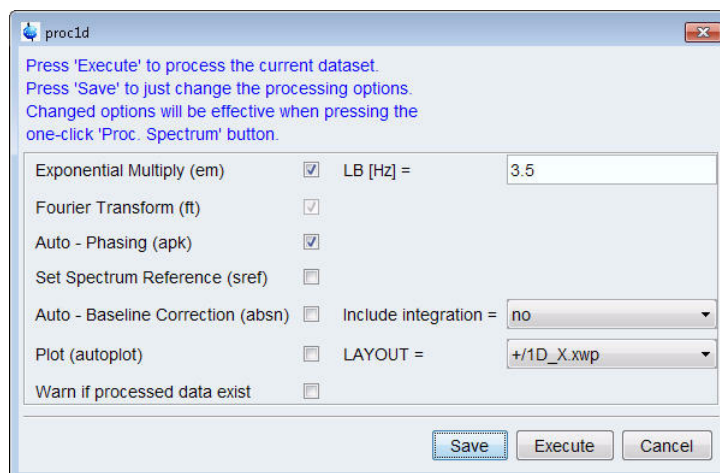
2. Click on the down arrow inside the  button

Figure 7.28



3. Select 'Configure Standard Processing' by clicking on it
4. Deselect the following options:
  - 'Set Spectrum Reference (sref)'
  - 'Auto-Baseline correction (abs)'
  - 'Warn if Processed data exist'

Figure 7.29



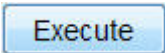
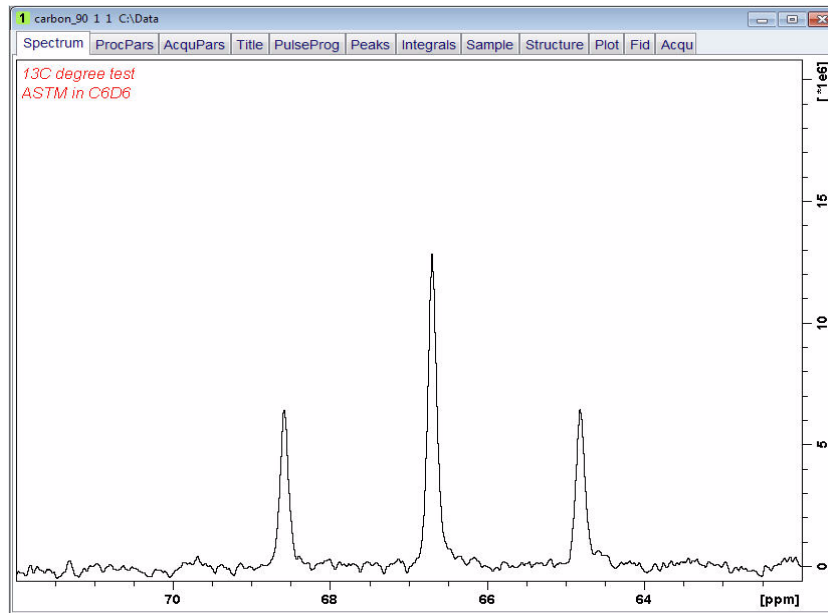
5. Click on 
6. Expand the spectrum from 72 ppm to 62 ppm

Figure 7.30



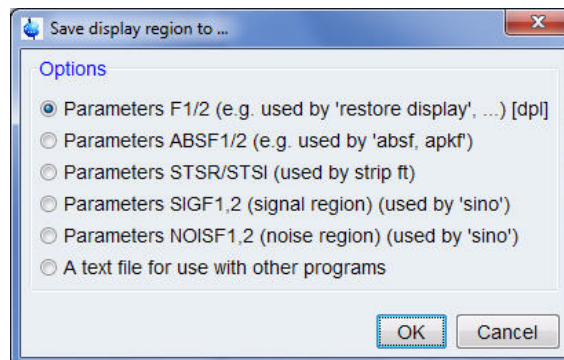
7. Click on the right mouse button inside the spectral window

Figure 7.31



8. Select 'Save Display Region to...' by clicking on it

Figure 7.32



9. Enable 'Parameters F1/2'


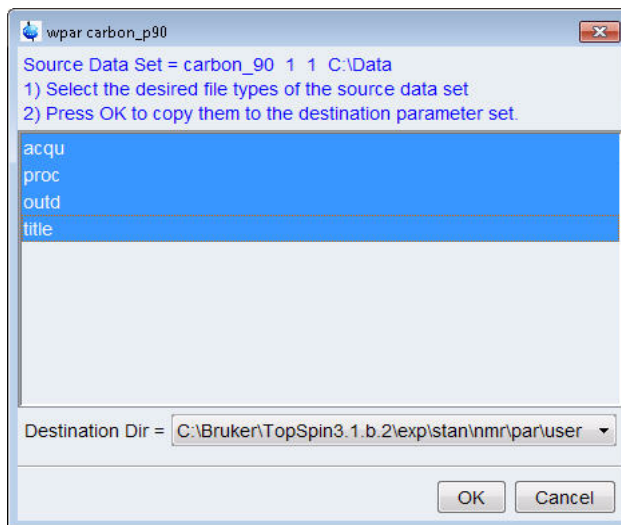

10. Click on 
11. Type **wpar carbon\_p90** to store the parameter for future use

Figure 7.33



12. Select all parameter options
13. Click on 

## 7.3.4 Determine the 90<sup>0</sup> pulse

1. Click on the '**Acquire**' tab in the TopSpin menu bar

Figure 7.34




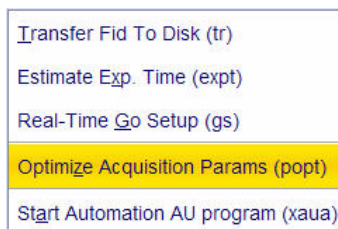
2. Click on the down arrow inside the  button

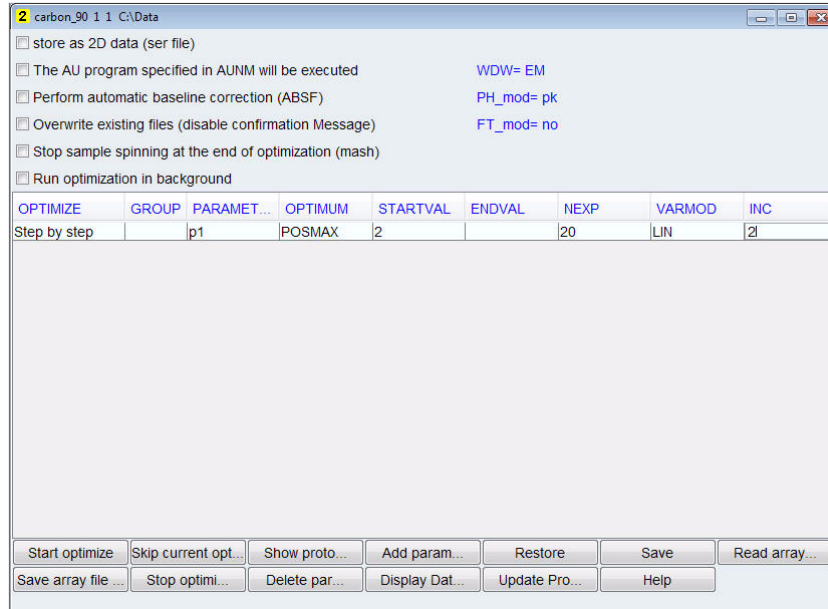
Figure 7.35

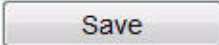


3. Select '**Optimize Acquisition Params (popt)**' by clicking on it
4. Make the following changes:
  - OPTIMIZE = **Step by step**
  - PARAMETER = **p1**

OPTIMUM = POSMAX  
 STARTVAL = 2  
 NEXP = 20  
 VARMOD = LIN  
 INC = 2

Figure 7.36



5. Click on 

NOTE: The ENDVAL parameter has been updated.

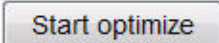
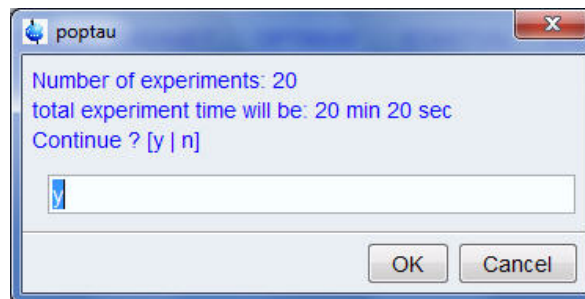

6. Click on 

Figure 7.37

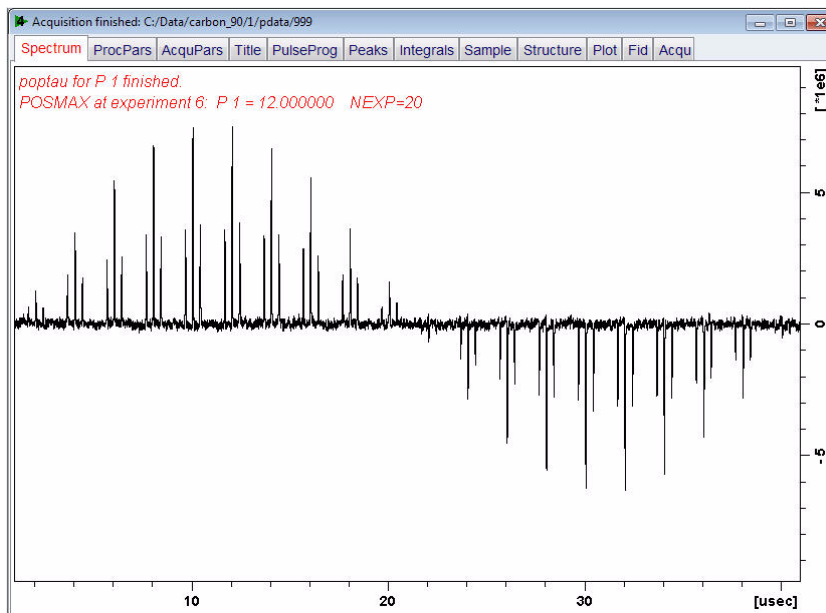


7. Enter **y** in to the poptau window

8. Click on 

NOTE: The parameter optimization starts. The spectrometer acquires and processes 20 spectra with incrementing the parameter p1 from 2 usec by 2 usec to a final value of 40 usec. For each of the 20 spectra, only the spectral region defined above is plotted, and 7.38.

Figure 7.38



NOTE: The POSMAX value of p1 is displayed in the title which is the 90 degree pulse, along with the experiment number and the NEXP value. Write this value down. To obtain a more accurate 90 degree pulse measurement, follow the steps below.

9. Close the popt setup window
10. Type **rep 1**
11. Type **p1**
12. Enter the value which corresponds to a 360 degree pulse (four times the POSMAX value)
13. Type **zg**
14. Type **efp**
15. Change **p1** slightly and repeat steps 13 and 14, until the signal undergoes a zero crossing as expected for an exact 360 degree pulse.

NOTE: The signal is negative for a pulse angle slightly less than 360 degree and positive when the pulse angle is slightly more than 360 degree.

16. Simply divide the determined 360 degree pulse value by 4. This will be the exact 90 degree pulse length for the proton transmitter on the current probe

## 7.3.5 Observations

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# 8 Sensitivity tests

## 8.1 Introduction

---

This chapter describes the sensitivity test procedures for  $^1\text{H}$  and  $^{13}\text{C}$ . It is assumed that the user is already familiar with acquisition and processing of simple 1D NMR spectra. Chapter 3 (1-D Proton experiment) and chapter 5 (1-D Carbon experiments). Also the 90 degree pulses have to be properly calibrated, Chapter 7 (Determination of the 90 degree pulses)

---

NOTE: This chapter is intended as a guide for running the  $^1\text{H}$  and  $^{13}\text{C}$  Signal to Noise tests on a probe or verifying the values observed by the engineer installing the instrument.

---

## 8.2 $^1\text{H}$ Sensitivity test

---

Standard Test Sample:

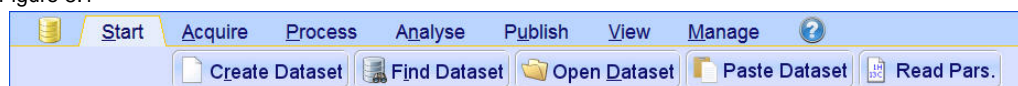
0.1% Ethylbenzene in  $\text{CDCl}_3$

### 8.2.1 Experiment setup

---

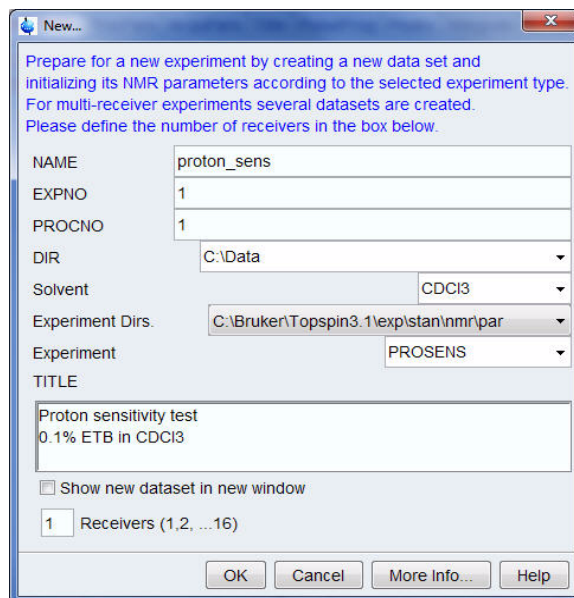
1. Click on the 'Start' tab in the TopSpin Menu bar

Figure 8.1



2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 8.2



NOTE: The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in Figure 8.2 above. Click on the down arrow button to browse for a specific directory.


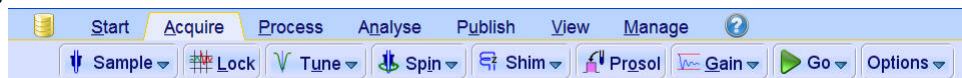
4. Click on 
5. Click on the 'Acquire' tab in the TopSpin menu bar

Figure 8.3



6. Select  **Sample** by clicking on it

Figure 8.4



7. Select 'ej' by clicking on it

NOTE: Wait till the sample lift air is turned on and remove any sample which may have been in the magnet.


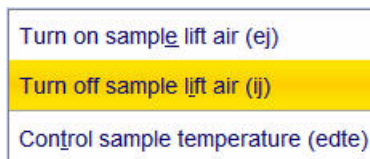
8. Place the sample on top of the magnet
9. Select  **Sample** by clicking on it

Figure 8.5

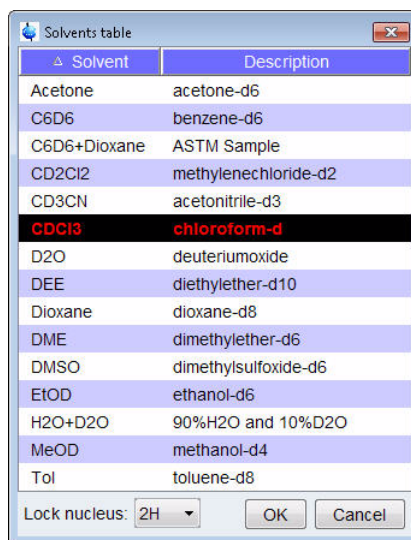


10. Select 'ij' by clicking on it

NOTE: Wait till the sample is lowered down in to the probe and the lift air is turned off. A licking sound may be heard.

11. Select  Lock by clicking on it

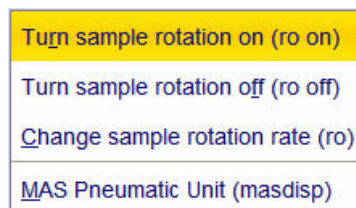
Figure 8.6



12. Select '**CDCl3**' by clicking on it

13. Select  Spin by clicking on it

Figure 8.7



14. Select 'ro on' by clicking on it

NOTE: Rotation may be turned off for probes such as BBI, TXI, TBI and for small sample probes.

15. Select  by clicking on it

---

NOTE: This executes the command 'gradshim'. To select other options, click on the down arrow inside the 'Shim' button.

---

16. Select  by clicking on it

---

NOTE: This will load the pulse width and power levels in to the parameter set.

---

## 8.2.2 Acquisition

1. Select  by clicking on it

---

NOTE: The relaxation time D1 is by default in this parameter set 60 seconds and therefore the adjustment of the receiver gain will take some time.

---

2. Select  by clicking on it

## 8.2.3 Processing

1. Click on the 'Process' tab in the TopSpin Menu bar

Figure 8.8



2. Click on 

---

NOTE: This executes a processing program including commands such as an exponential window function 'em', Fourier transformation 'ft', an automatic phase correction 'apk' and a baseline correction 'abs'. Other options are available by clicking on the down arrow inside the 'Proc. Spectrum' button.

---

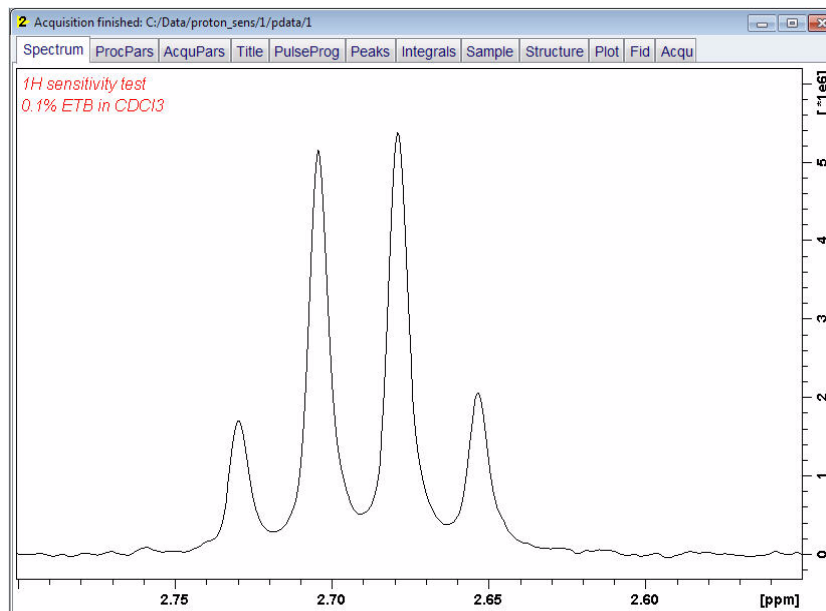
## 8.2.4 Calculating the Signal to Noise ratio

---

The signal to noise ratio is determined on the intensity of the quartet lines between 2ppm and 3ppm. It is calculated by AU-program **sinocal** over a range of 2ppm between 2.8ppm and 7ppm. The s/n ratio is strongly dependant on good resolution and line-shape. The splitting between the two central lines of the methylquartet should go lower than 15% (with LB=1Hz), see Figure 8.9.

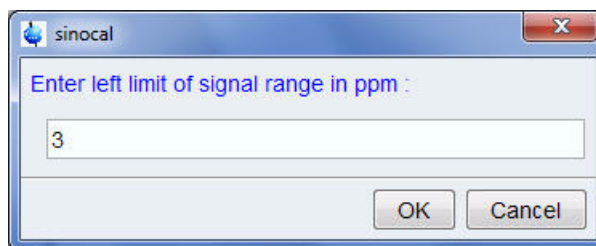
---

Figure 8.9



1. Type **sinocal** on the command line

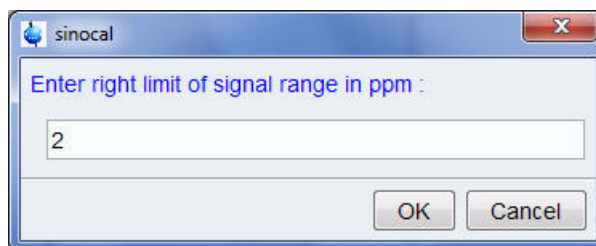
Figure 8.10



2. Enter **3** for the left limit of the signal range

3. Click on

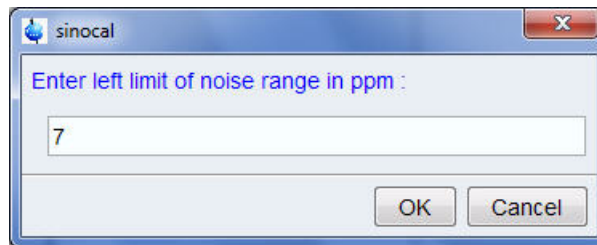
Figure 8.11



4. Enter **2** for the right limit of the signal range

5. Click on

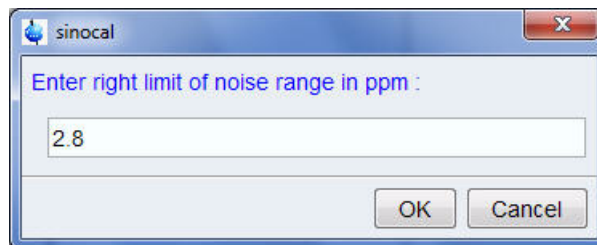
Figure 8.12



6. Enter **7** for the left limit of the noise range

7. Click on

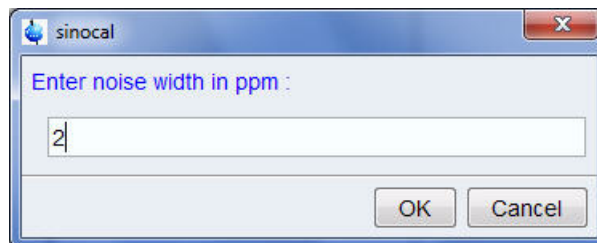
Figure 8.13



8. Enter **2.8** for the right limit of the noise range

9. Click on

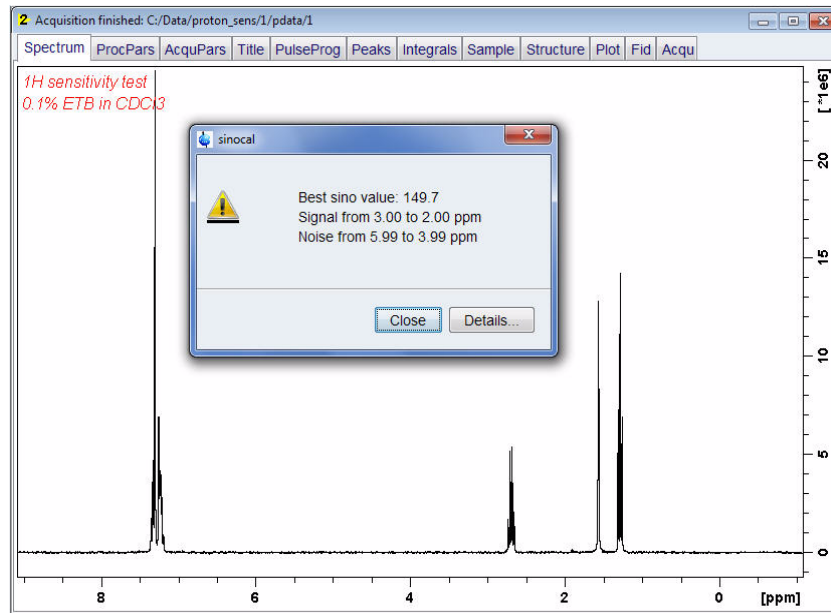
Figure 8.14



10. Enter **2** for the noise width

11. Click on

Figure 8.15



## 8.2.5 Observations

---

## 8.3 <sup>13</sup>C Sensitivity test without <sup>1</sup>H decoupling

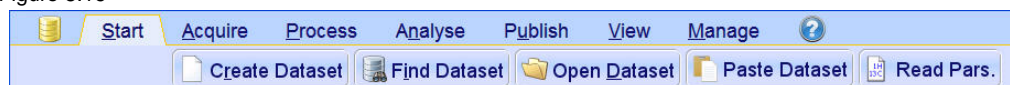
Standard Test Sample:

ASTM (60% C<sub>6</sub>D<sub>6</sub> / 40% p-Dioxane)

### 8.3.1 Experiment setup

1. Click on the 'Start' tab in the TopSpin Menu bar

Figure 8.16



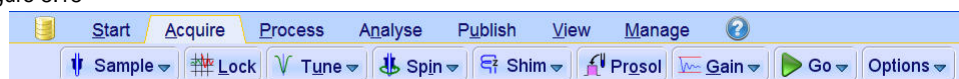
2. Select **Create Dataset** by clicking on it
3. Enter the following information in to the 'New' window

Figure 8.17

NOTE: The directory (DIR) is specific to 8.17 above. Click on the down arrow button to browse for a specific directory.

4. Click on **OK**
5. Click on the 'Acquire' tab in the TopSpin menu bar

Figure 8.18



6. Select  **Sample** by clicking on it

Figure 8.19



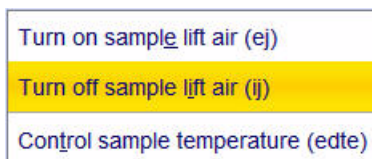
7. Select 'ej' by clicking on it

NOTE: Wait till the sample lift air is turned on and remove any sample which may have been in the magnet.

8. Place the sample on top of the magnet

9. Select  **Sample** by clicking on it

Figure 8.20

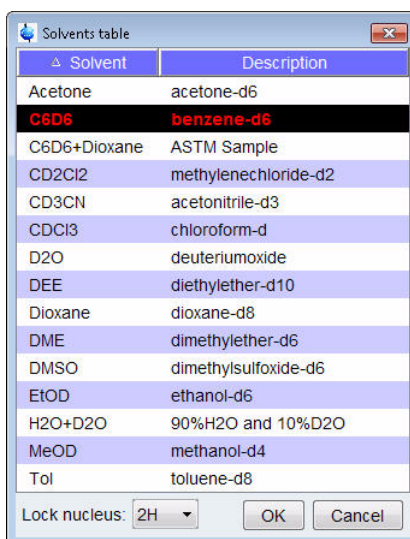


10. Select 'ij' by clicking on it

NOTE: Wait till the sample is lowered down in to the probe and the lift air is turned off. A licking sound may be heard.

11. Select  **Lock** by clicking on it

Figure 8.21




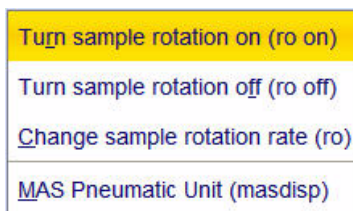
12. Select '**C6D6**' by clicking on it
13. Select  by clicking on it

Figure 8.22



14. Select '**ro on**' by clicking on it

---

NOTE: Rotation may be turned off for probes such as BBI, TXI, TBI and for small sample probes.

---

15. Select  by clicking on it

---

NOTE: This executes the command '**gradshim**'. To select other options, click on the down arrow inside the '**Shim**' button.

---

16. Select  by clicking on it

---

NOTE: This will load the pulse width and power levels in to the parameter set.

---

17. Select the '**AcquPars**' tab by clicking on it

18. Make the following changes:

PULPROG = **zg**

TD = **64k**

SW [ppm] = **160**

D1 = **300**

DS = **0**

NS = **1**

O1 [ppm] = **100**

19. Select the '**ProcPars**' tab by clicking on it

20. Make the following changes:

SI = **32k**

LB [Hz] = **3.5**

21. Click on the '**Acquire**' tab in the TopSpin menu bar

### 8.3.2 Acquisition

---

1. Select  by clicking on it

---

NOTE: The relaxation time D1 is by default in this parameter set 300 seconds and therefore the adjustment of the receiver gain will take some time.

---

2. Select  by clicking on it

## 8.3.3 Processing

---

1. Click on the '**Process**' tab in the TopSpin Menu bar

Figure 8.23



2. Click on 

---

NOTE: This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**' button.

---

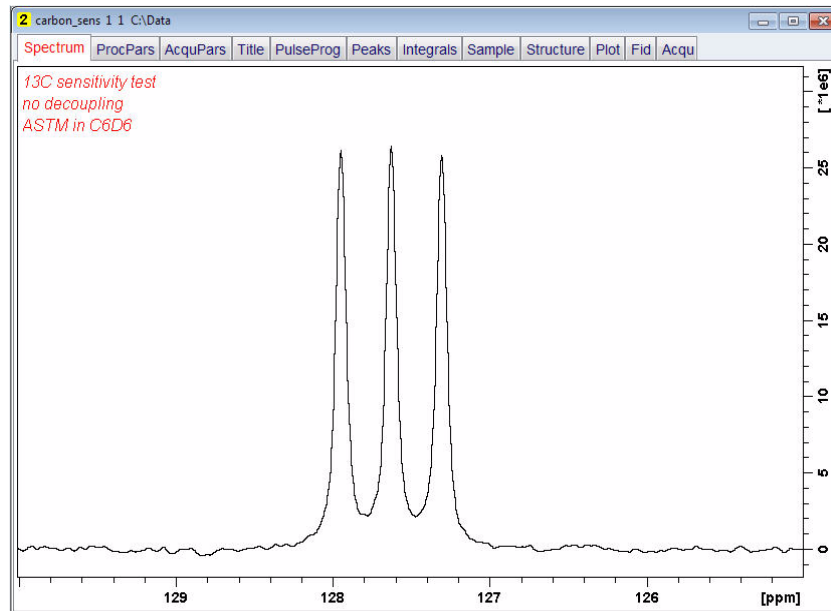
## 8.3.4 Calculating the Signal to Noise ratio

---

The signal to noise ratio is determined on the triplet of the deuterated benzene between 127ppm and 129ppm. It is calculated by AU-program **sinocal** over a range of 40ppm between 70ppm and 125ppm. The s/n ratio is strongly dependant on good resolution and line shape. The splitting of the 1:1:1 triplet should go lower than 9% (5mm) see Figure 8.24. 10% (10mm) and 12% (20mm).

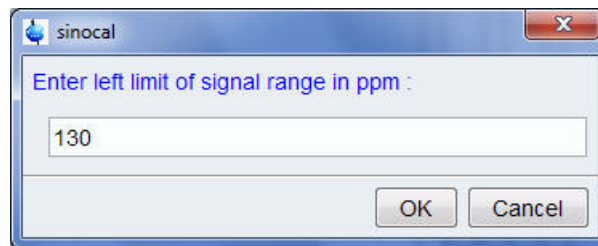
---

Figure 8.24



1. Type **sinocal** on the command line

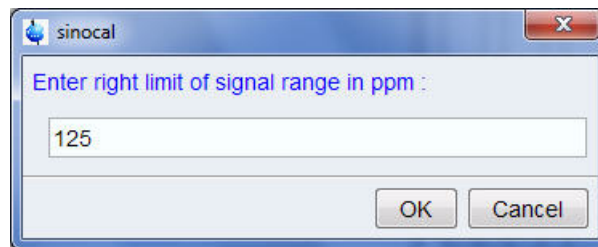
Figure 8.25



2. Enter **128** for the left limit of the signal range

3. Click on

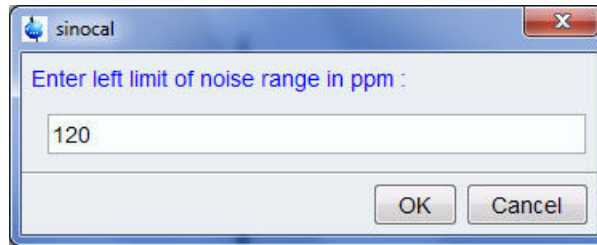
Figure 8.26



4. Enter **127** for the right limit of the signal range

5. Click on

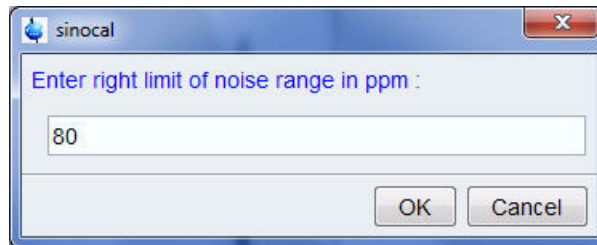
Figure 8.27



6. Enter **125** for the left limit of the noise range

7. Click on

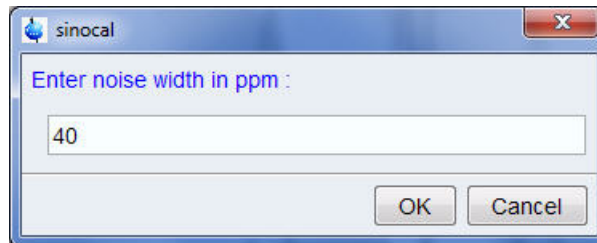
Figure 8.28



8. Enter **30** for the right limit of the noise range

9. Click on

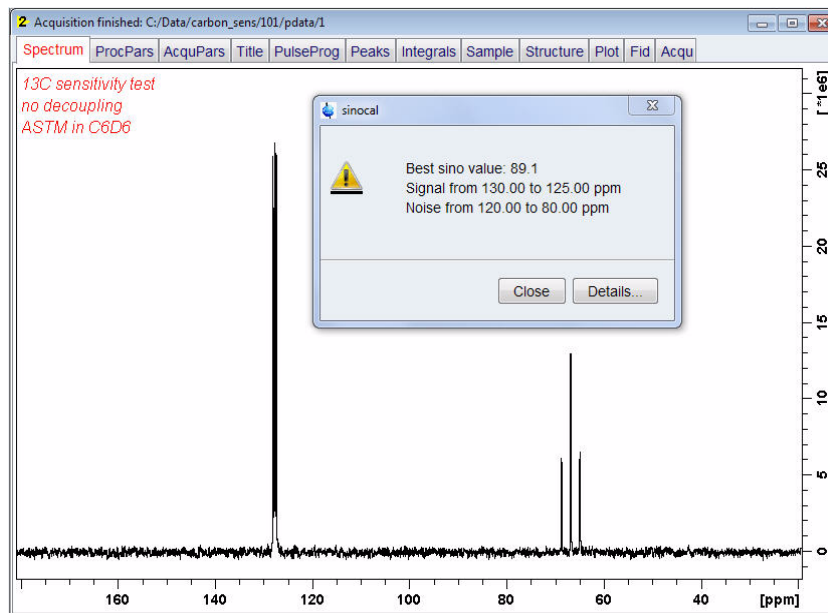
Figure 8.29



10. Enter **40** for the noise width

11. Click on

Figure 8.30



## 8.3.5 Observations

---

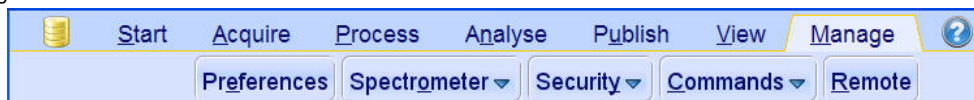
# 9 Spectrometer configuration

## 9.1 Hardware Configuration

---

1. Click on the **'Manage'** tab in the TopSpin Menu bar

Figure 9.1



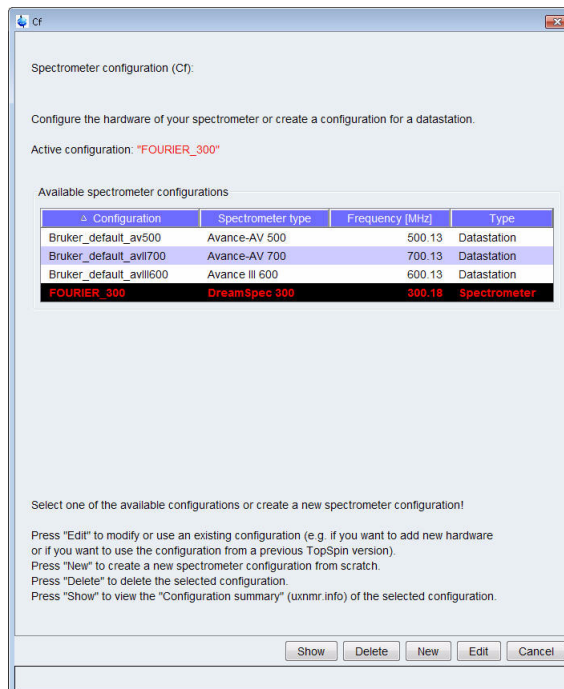
2. Click on the down arrow inside the **Spectrometer** button

Figure 9.2

Hardware Detection	▶	Configure Hardware (cf)
Experiments/Parameters	▶	Initialize Spectrometer Interface (ii)
Save/Restore Installation	▶	Edit the Probehead Table (edhead)
Spectrometer Usage (account)		Setup Linearization Correction Tables (cortab)
		Find Ethernet Addresses (ha)

3. Select **'Hardware Detection'**
4. Select **'Configure Hardware (cf)'** by clicking on it
5. Enter the NMR administration password
6. Click on

Figure 9.3



4. Select Configuration for 'Spect' by clicking on it


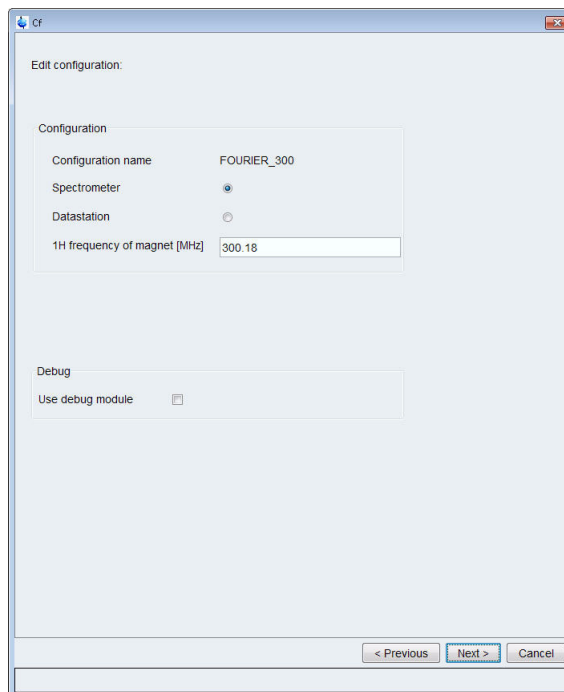
5. Click on 

Figure 9.4



6. Click on 

Figure 9.5

Specify the channel to which external devices are connected.

Communication channels for external devices

HPPR Preamplifier	no
ACB Amplifier Control Board	no
Minispec Lock	149.236.99.254
VTU Variable Temperature Unit	149.236.99.253
LC-NMR Software HyStar	no
Gradient Temperature Unit (BCU-20)	no
MAS Pneumatic Control Unit	no
Bruker Automatic Changer	no
Barcode Printer	no
Cryo Controller	no
HPCU High Power Control Unit	no
Preemphasis/Gradient Unit	no
Fast Gradient Supervisor	no
Gradient Power Supply Control Unit	no
Radio Frequency Supervisor	no

Lockswitch

2H Lockswitch connected to Amplifier Number	1
19F Lockswitch connected to Amplifier Number	no

< Previous   Next >   Cancel

hconfserver: check list of installed devices

7. Enter the ports for the external devices as shown in Figure 9.5

8. Click on **Next >**

Figure 9.6

Additional configuration:

Security configuration

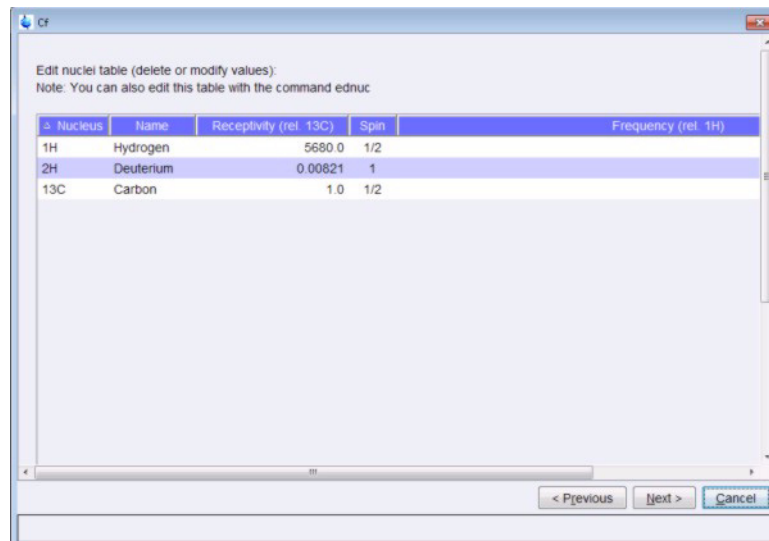
Enable peak power check (POWCHK):

< Previous   Next >   Cancel

hconfserver: check security parameters

9. Click on **Next >**

Figure 9.7




10. Click on 

Figure 9.8

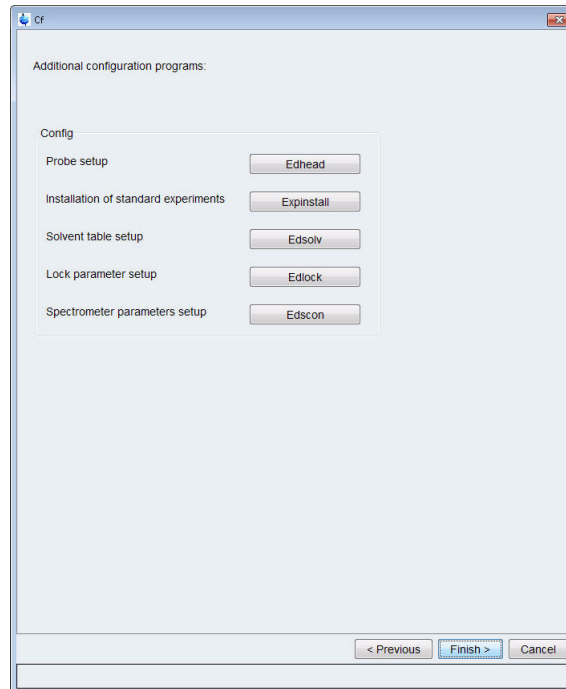


NOTE: The configuration information is displayed on the screen. Store the print out of the configuration information with the installation data

11. Click on 

12. Click on 

Figure 9.9



## 9.2 Expinstall

---

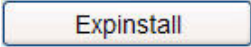

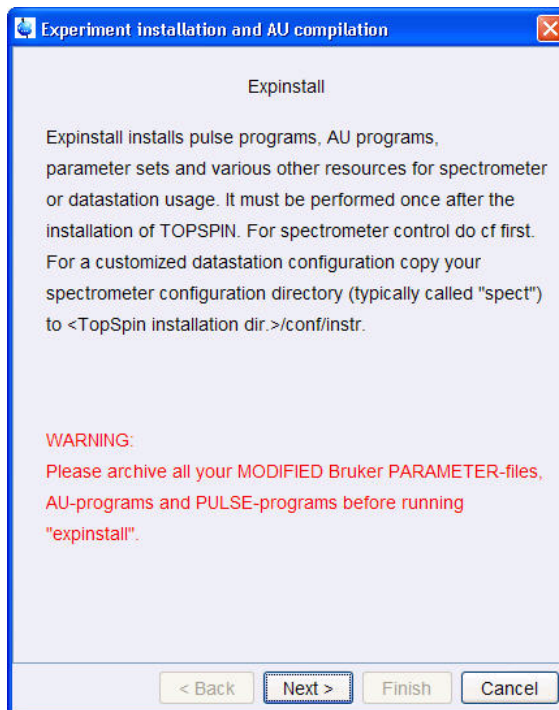
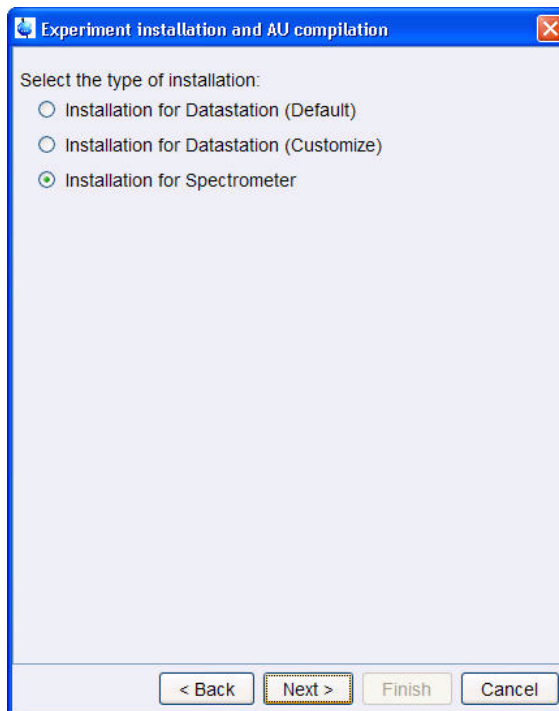
1. Click on 
2. Enter the NMR administration password
3. Click on 

Figure 9.10



4. Click on 

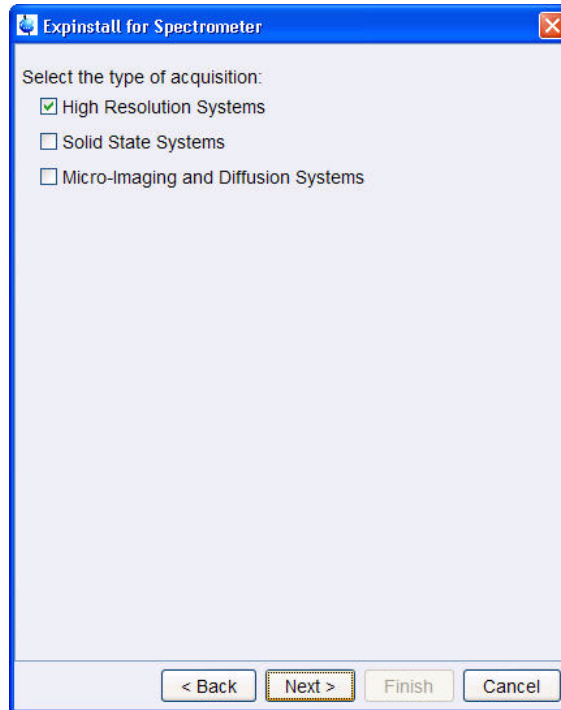
Figure 9.11



5. Select **'Installation for Spectrometer'**

6. Click on 

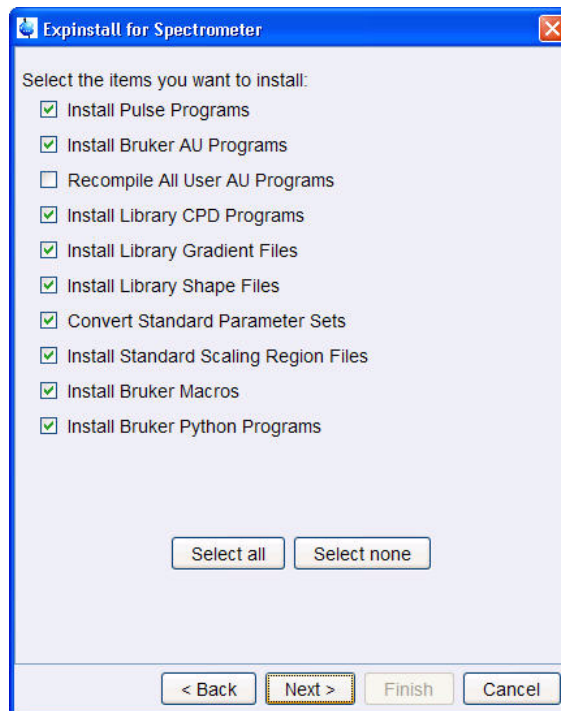
Figure 9.12



7. Select **'High Resolution System'**

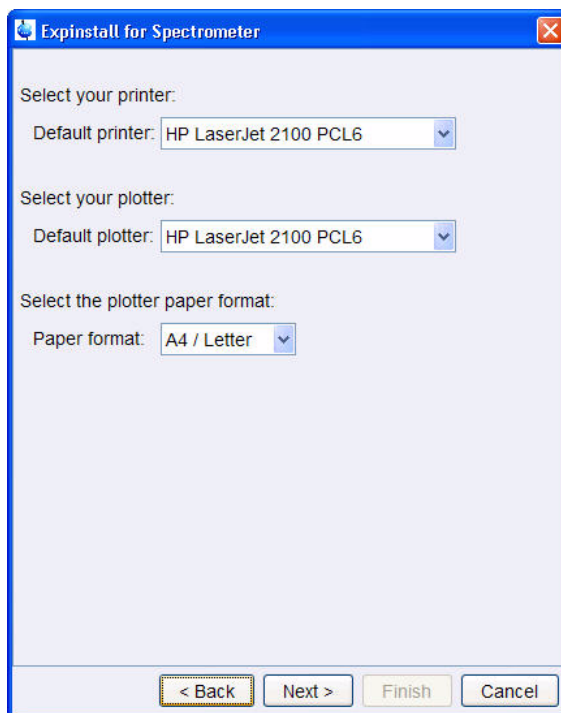
8. Click on 

Figure 9.13



9. Click on 

Figure 9.14

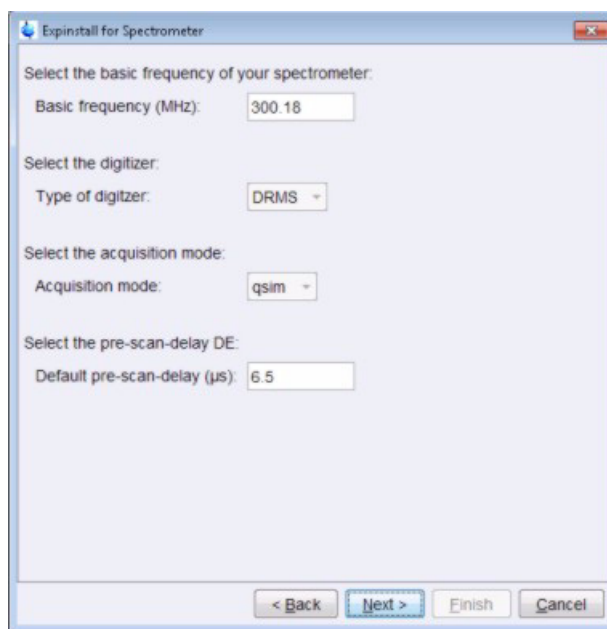


10. Select Default printer and plotter

11. Select Paper format

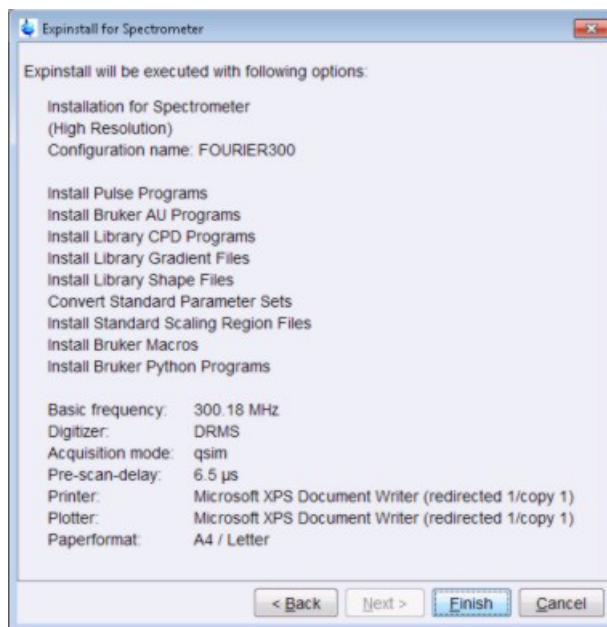
12. Click on 

Figure 9.15



13. Click on 

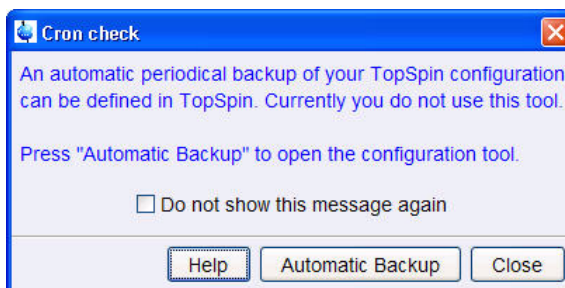
Figure 9.16



14. Click on 

NOTE: expinstall starts now. This process will take approximately 30 seconds. On finish the message below appears (Figure 9.17.). To set up a time schedule to perform an NMR\_save periodically (recommended) follow the instructions in **9.3 Set up the cron job for NMR\_save.**

Figure 9.17



## 9.3 Set up the cron job for NMR\_save

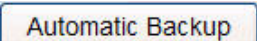
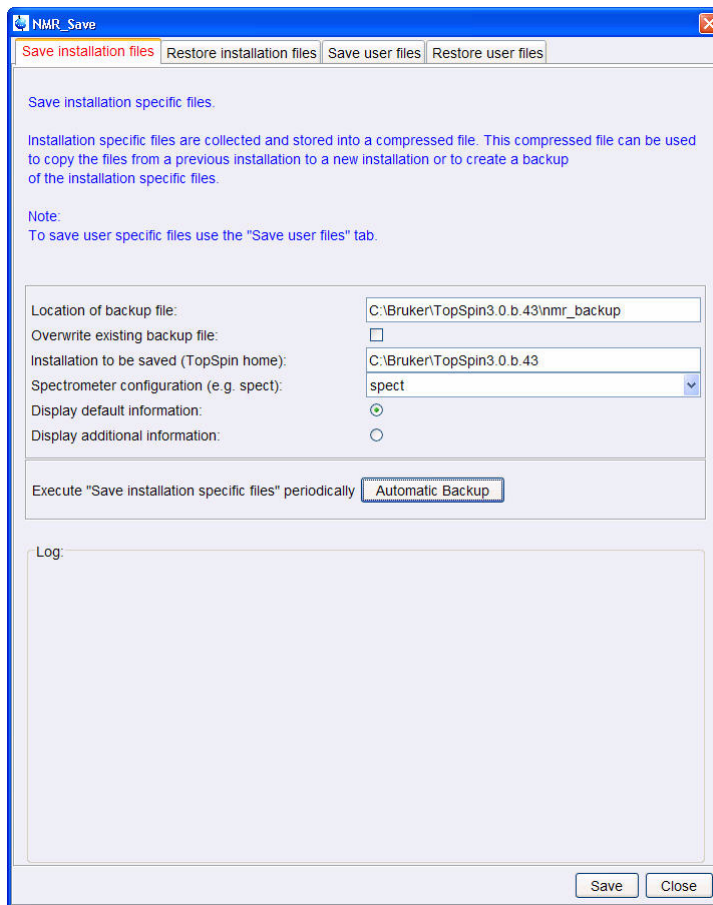
1. Click on 

Figure 9.18



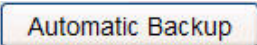
2. Click on 

Figure 9.19

**New periodical**

**Job**

Command: `.nmrsave -date -path "C:\Bruker\TopSpin3.0.b.43\nmr_backup" -source "C:\`

Description: Execute NMR\_SAVE

Execution scope: TopSpin (requires authentication)

**Options**

Off-schedule execution

Direct execution

**Rules**

Minute of the hour: from: 12 to: --- Ignore ---

Hour of the day: from: 14 to: --- Ignore ---

Day of the month: from: 22 to: --- Ignore ---

Month of the year: from: \* to: --- Ignore ---

Day of the week: from: \* to: --- Ignore ---

OK Cancel

---

NOTE: In this example an NMR\_save is performed from January to December on the 1st day of the month at 2 o'clock in the morning.

---


3. Click on 

Figure 9.20

**cf**

Additional configuration programs:

**Config**

Installation of standard experiments:

Solvent table setup:

Probe table setup:

Lock parameter setup:

Spectrometer parameters setup:

< Previous Z31979\_00\_01



# 10 Safety

## 10.1 Introduction

---

In terms of safety the presence of a relatively strong magnet is what differentiates NMR spectrometers from most other laboratory equipment. When designing an NMR laboratory, or training personnel who will work in or around the laboratory, no other feature is of greater significance. As long as correct procedures are adhered to, working in the vicinity of superconductive magnets is completely safe and has no known harmful medical side effects. Negligence however can result in serious accidents. It is important that people working in the vicinity of the magnet fully understand the potential hazards. Of critical importance is that people fitted with cardiac pacemakers or ferromagnetic implants should never be allowed near the magnet.

The magnet is potentially hazardous due to:

- The large attractive force it exerts on ferromagnetic objects.
- The large content of liquid Nitrogen and Helium.

## 10.2 Magnetic Safety

---

A Magnetic Field surrounds the magnet in all directions. This field (known as the stray field) is invisible, hence the need to post warning signs at appropriate locations. Objects made of ferromagnetic materials, e.g. iron, steel etc. will be attracted to the magnet. If a ferromagnetic object is brought too close, it may suddenly be drawn into the magnet with surprising force. This may damage the magnet, or cause personal injury to anybody in the way!

The Fourier 300 super conducting magnet is actively shielded. The following must be understood when working with such a shielded magnet.

- The active shielding of the super conducting coil reduces the stray magnetic field and therefore its effect. The **5 Gauss** line in the horizontal direction extends **26.5 cm** around the outside of the magnet. In the vertical direction it extends about **24 cm** out of the can at the middle but it does not go above the helium stacks or below the floor.
- In spite of the active shielding, the stray magnetic field immediately adjacent to the bore of the magnet is very high and the attractive forces on ferromagnetic objects are very strong!

## 10.3 Cryogenic Safety

---

The magnet contains relatively large quantities of liquid helium and nitrogen. These liquids, referred to as cryogens, serve to keep the magnet core at a very low temperature.

Because of the very low temperatures involved, **gloves, a long sleeved shirt or lab coat** and **safety goggles** should always be worn when handling cryogens. Direct

contact with these liquids can cause frostbite. The system manager should regularly check and make sure that evaporating gases are free to escape from the magnet, i.e. the release valves must not be blocked. Do not attempt to refill the magnet with helium or nitrogen unless you have been trained in the correct procedure.

### 10.3.1 What is a Quench?

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A magnet **quench** is the spontaneous breakdown of superconductivity in a partially or fully energized magnet. The stored field energy is transformed into heat, leading to a fast evaporation of liquid helium. During a quench, an extremely large quantity,  $\sim 40 \text{ m}^3$  ( $1,400 \text{ ft}^3$ ) of helium gas is produced within a short time.

Helium and nitrogen are non-toxic gases. However, because of a possible **magnet quench**, where upon the room may suddenly fill with evaporated gases causing potential danger of suffocation. Adequate ventilation must always be provided and it is recommended that any person should leave the room.

## 10.4 Emergency Planning

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Due to the strong magnetic fields and presence of cryogenics when using NMR systems, it is important to define and communicate what to do in case of problems or an emergency. An **Emergency Plan** can be defined as a documented set of instructions on what to do if something goes wrong. Emergency Plans are often defined as part of the Standard Operating Procedures (SOP), or as a stand-alone document. In any case every NMR laboratory should have an Emergency Plan in effect in case of problems or emergencies.

As every organization has its own policies and procedures, as well as varying laboratory layouts, an Emergency Plan should be individually defined for each laboratory as appropriate. Upon request Bruker can provide useful information on emergency planning.

## 10.5 Fire Department Notification

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**It is recommended that the magnet operator introduce the fire department and/or local authorities to the magnet site. It is important that these organizations be informed of the potential risks of the magnet system, i.e. that much of the magnetic rescue equipment (oxygen-cylinders, fire extinguishers, axes etc.) can be hazardous close to the magnet system. On the other side, their expertise and experience can be invaluable in creating an Emergency plan.**

- Within a NMR laboratory CO<sub>2</sub> magnetic fire extinguishers must NOT be used. Breathing equipment which uses oxygen tanks made out of magnetic material can be life threatening when used close to a magnet system which still has a magnetic field present.
- Helium gas escaping from the system must not be mistaken for smoke. Instruct the fire department and technical service not to extinguish the magnet system with water. The outlet valves could freeze over and generate excess pressure within the system.

- NMR laboratory windows which are accessible during an emergency must be clearly marked with warning signs, visible from the outside.

## 10.6 Earthquake Safety

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In regions where there is a potential risk of earthquakes, additional measures must be taken to reduce the chance of personal or property damage through movement or tipping of the magnet.

Many countries or regions have documented regulations, including building codes, regarding earthquakes. Before installing a magnet system, it is highly advisable that you check with local authorities on whether your area is prone to earthquakes and if there are any regulations in effect.

If your area is regarded as an earthquake area there are several shock absorbing measures or riggings available to reduce the likelihood of damage during an earthquake. Please contact Bruker for more information on earthquake securing equipment.

## 10.7 Country-specific Safety Regulations

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In addition to the above safety precautions, any country-specific safety regulations for operating NMR systems must be fulfilled. These may include, for example, regulations on:

- Facilities of a controlled access area around the magnet.
- Working conditions at computer stations.



# Appendix

# A

## A.1 Standard Parameter set list

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### 1-D Experiments

C13APT - Attached Proton Test using jmod pulse program  
C13CPD - C13 exp. comp. pulse dec. 1024 scans  
C13CPD32 - C13 exp. comp. pulse dec. 32 scans  
C13CPDSN - C13 exp. comp. pulse dec. with signal-to-noise calc.  
C13DE45SN - C13 dept all positive with signal-to-noise calc.  
C13DEPT45 - C13 dept all positive  
C13DEPT90 - C13 dept CH-only  
C13DEPT135 - C13 dept CH,CH3 pos. CH2 neg.  
C13DEPT135p - dept135 with phase of previous C13  
C13GD - C13 exp. gated decoupling  
C13IG - C13 exp. inverse gated decoupling  
C13HUMP - 13C hump (lineshape) test  
C13RESOL - 13C resolution (half width) test  
C13SENS - 13C sensitivity (SINO) test  
NOEDIFF - 1H noe difference  
PROTON128 - 1H experiment 128 scans  
PROTONCONLF - 1H exp. with conditional low field plot  
PROTONEXP - 1H experiment + expansions  
PROTONLF - 1H experiment + low field plot  
PROTONLFEXP - 1H experiment + low field plot + expansions  
PROTONNR - 1H exp. non spinning  
PROTONNREXP - 1H exp. non spinning + expansions  
PROTONNRLF - 1H exp. non spinning + low field plot  
PRONRLFEXP - 1H exp. non spinning + low field plot + expansions  
PROTONT1 - 1H T1 Relaxation measurement  
PROHUMP - 1H hump (lineshape) test  
PRORESOL - 1H resolution (half width) test  
PROSENS - 1H sensitivity (SINO) test  
WATER - water supression

## **2-D Experiments**

COSY45SW - sw opt. COSY45 (magn. mode)

COSY90SW - sw opt. COSY90 (magn. mode)

COSYGPSW - sw opt. COSY with gradients (magn. mode)

COSYDQFPHSW - sw opt. COSY with dq filter (TPPI)

COSYGPDFPHSW - sw opt. COSY with gradients and dq filter (TPPI)

COSYGPMFSW - sw opt. COSY with gradients and mq filter (magn. mode)

MLEVPHSW - sw opt. TOCSY (TPPI)

NOESYPHSW - sw opt. NOESY (TPPI)

NOESYGPPHSW - sw opt. NOESY with gradients (TPPI)

ROESYPHSW - sw opt. ROESY (TPPI)

HMQCGP - sw opt. HMQC with gradients (magn. mode)

HMBCGP - sw opt. HMBC with gradients, low pass J-filter, no decoupling

HSQCGPPH - sw opt. HSQC sens. improved with gradients (e/a TPPI)

HMBCGPND - sw opt. HMBC with gradients

Above are the current list of all parameter sets which are specific to work on a Fourier system. The list is being created during expinstall and stored in the "<TopSpin-home>/exp/stan/nmr/par" directory.

## B Contacts

For further technical assistance, please do not hesitate to contact your nearest BRUKER dealer or contact us directly at:

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### **NMR Hotlines**

Contact our NMR service centers.

Bruker BioSpin NMR provide dedicated hotlines and service centers, so that our specialists can respond as quickly as possible to all your service requests, applications questions, software or technical needs.

Please select the NMR service center or hotline you wish to contact from our list available at:

[http://www.bruker-biospin.com/hotlines\\_nmr.html](http://www.bruker-biospin.com/hotlines_nmr.html)



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