Fourier 300

- NMR Experiments

User Manual

Version 004

Innovation with Integrity

NMR

This manual was written by

Peter Ziegler

© June 26, 2013: Bruker Corporation

Billerica, Massachusetts, USA

P/N: Z31979

DWG-Nr.: Z4D11519C

For further technical assistance on the Fourier 300 unit, please do not hesitate to contact your nearest BRUKER dealer or contact us directly at:

BRUKER BioSpin Corporation
15 Fortune Drive
Billerica, MA 01821
USA

Phone: (978) 667-9580 ext. 5444
FAX: (978) 667-2955
E-mail: applab@bruker-biospin.com
Internet: www.bruker.com

Table of Contents

Table of Contents

Table of Contents

1 About

1.1 This Manual

This manual is intended to be a reference guide for operators and service technicians. It provides detailed information about the user level maintenance and service and overall use of the Bruker device.

The figures shown in this manual are designed to be general and informative and may not represent the specific Bruker model, component or software/firmware version you are working with. Options and accessories may or may not be illustrated in each figure.

Carefully read all relevant chapters before working on the device!

This manual describes parts and procedures relevant to the device version it is delivered with. For older hardware, please refer to the manual supplied at the time.

1.2 Policy Statement

It is the policy of Bruker to improve products as new techniques and components become available. Bruker reserves the right to change specifications at any time.

Every effort has been made to avoid errors in text and figure presentation in this publication. In order to produce useful and appropriate documentation, we welcome your comments on this publication. Support engineers are advised to regularly check with Bruker for updated information.

Bruker is committed to providing customers with inventive, high quality products and services that are environmentally sound.

1.3 Symbols and Conventions

Safety instructions in this manual are marked with symbols. The safety instructions are introduced using indicative words which express the extent of the harzard.

In order to avoid accidents, personal injury or damage to property, always observe safety instructions and proceed with care.

DANGER

This combination of symbol and signal word indicates an immediately hazardous situation which could result in death or serious injury unless avoided.

⚠ WARNING

This combination of symbol and signal word indicates a potentially hazardous situation which could result in death or serious injury unless avoided.

⚠ CAUTION

This combination of symbol and signal word indicates a possibly hazardous situation which could result in minor or slight injury unless avoided.

SAFETY INSTRUCTIONS

This combination of color and signal words are used for control flow and shutdowns in the event of an error or emergency.

NOTICE

This combination of color and signal word indicates a possibly hazardous situation which could result in damage to property or the environment unless avoided.

This symbol highlights useful tips and recommendations as well as information designed to ensure efficient and smooth operation.

2 Introduction

2.1 General

This manual was written for Fourier 300 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.

2.2 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.

Introduction

3 Safety

3.1 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.

3.2 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 die 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!

3.3 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.

3.3.1 What Is A Quench?

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 ,**~40 m^3** (**1,400 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.

3.4 Emergency Planning

Due to the strong magnetic fields and presence of cryogens 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 de- fined 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.

3.5 Fire Department Notification

⚠ CAUTION

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_2 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.

3.6 Earthquake Safety

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.

3.7 Country-Specific Safety Regulations

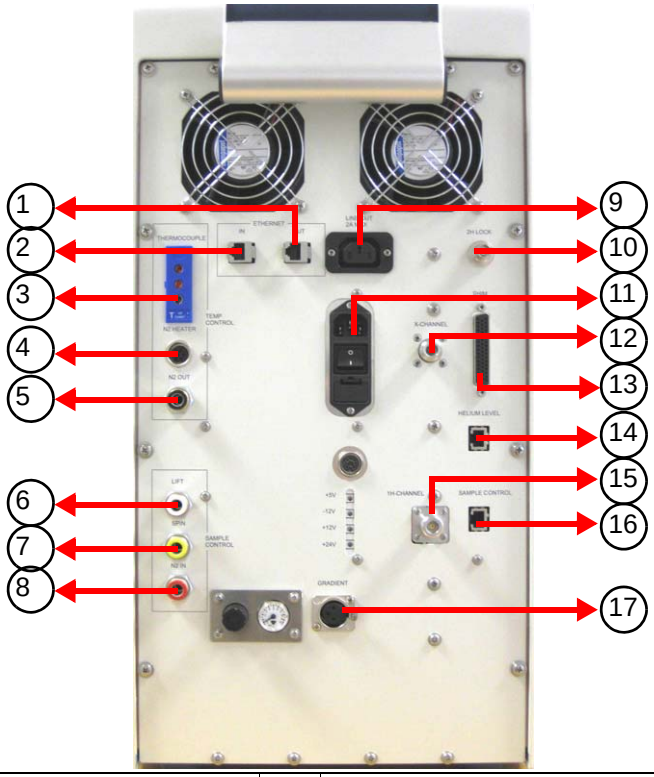
In addition to the above safety precautions, any country-specific safety regulations for operating NMR systems must be fulfilled. These may include, for example, regulations on:

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

3.8 Observations

4 Spectrometer Basics

4.1 Fourier Connection Overview



1	PC/Ethernet (83025) to Shim Coil	9	H15200 BACS2LT CPL (HZ16796) to Sample Changer
2	H15200 BACS2LT CPL (HZ16796) to Sample Changer	10	Z111650 (H2) PH DUL 300S1 C-H-D-05 Z ES (HZ03617) to Probe
3	Z111650 (Thermo) PH DUL 300S1 C-H-D-05 Z ES (W110125) to Probe	11	230 V/AC (3000) to Mains Supply
4	Z111650 (Heater) PH DUL 300S1 C-H-D-05 Z ES (W110117) to Probe	12	Z111650 (13C) PH DUL 300S1 C-H-D-05 Z ES (Z100371) to Probe with Filter (E1451430)
5	Z111650 (Air) PH DUL 300S1 C-H-D-05 Z ES (45964) to Probe	13	Z49734.1 Shim Coil BOSS1 S1 Plug (Z101089) to Shim Coil
6	Z100601 (Lift) Magnet System 300/54 US LH (HZ12223) to Magnet System	14	Z100601 (Helium) Magnet System 300/54 US LH (HZ12223) to Magnet System
7	Z100601 (Spin) Magnet System 300/54 US LH (HZ12223) to Magnet System	15	Z100601 (Sample) Magnet System 300/54 US LH (HZ12223) to Magnet System
8	H15200 BACS2LT CPL (HZ16796) to Sample Changer and Nitrogen (45901)	16	Z111650 (1H) PH DUL 300S1 C-H-D-05 Z ES (Z10057) to Probe with Filter (E1451420)
		17	Z111650 (Gradient) PH DUL 300S1 C-H-D-05 Z ES (W1209903) to Probe

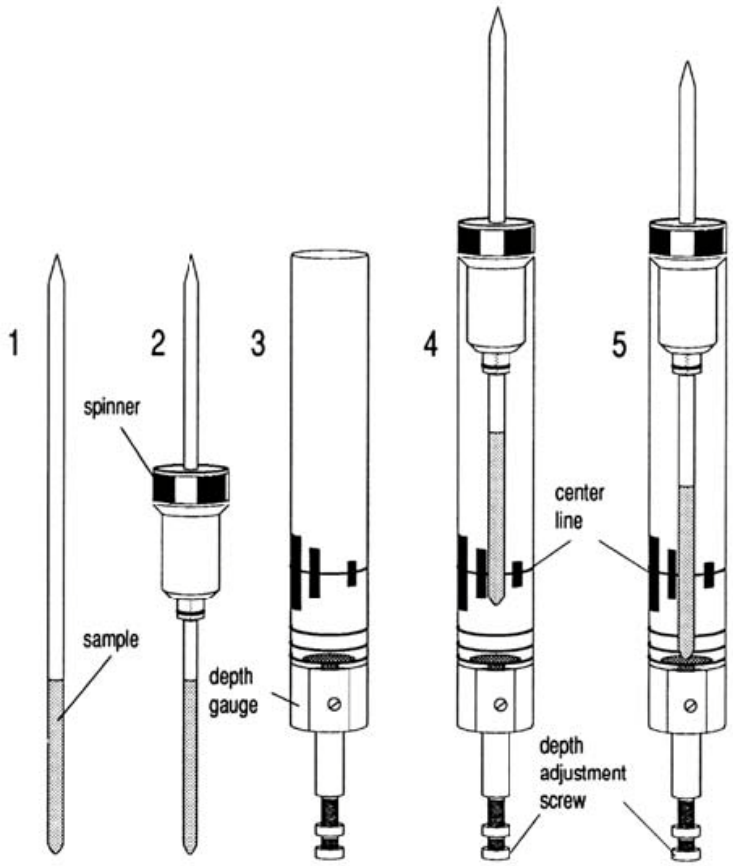
Figure 4.1 Fourier Connection Overview

4.2 Sample Preparation

- Use medium to high quality, clean and dry 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

4.3 Inserting the Sample with Spinner into the Magnet

The raising and lowering of the sample is controlled by a stream of pressurized air. The BST is 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.



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.

4.4 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 20 Hz is used to spin the sample. The spinner is turned off for experiments such as T1 and all 2D’s, to reduce unwanted artifacts from spinning the sample.

4.5 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 the sweep is turned on) 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 to maximize the level. Note that the lock phase for the probe is stored in the edlock table. If the signal oscillates due to saturation then the lock power can be reduced manually after lockin.

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.

4.6 Shimming the Sample

The following is intended to be a practical guide for adjusting the room temperature shim system. The purpose of shimming is to maximize the magnetic field homogneity, which depends somewhat on probehead and sample geometry. In general, it is necessary to shim the magnetic field to correct for any magnet drifts, especially after the magnet has been energized. This may requires to shim the **on-axis** and possible the **off-axis** shims. After a sample change, usually only the **on-axis** shims have to be adjusted.

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 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 (H0) as completely as possible.

The Fourier shim system has **20 room temperature** shims:
6 on-axis shims (Z, Z^2, Z^3, Z^4, Z^5, Z^6)
14 off-axis shims (X, XZ, XZ^2, Y, YZ, YZ^2, XY, XYZ, X^2-Y^2, (X^2-Y^2)z, X^3, XZ^3, Y^3, YZ^3)

4.6.1 Shimming using Gradshim

In gradient shimming (gradshim) pulse field gradients are used to automatically shim the **on-axis** shims (preferably with sample rotation). This feature can be used in automation as well as in manual mode within Topspin (Acquire -> Shim) and takes only a few minutes. It can be done with any deuterated solvent as long as a single strong signal is present in the sample and as long as the sample is locked. The gradient shimming feature will be set up with the initial setup of the spectrometer.

4.6.2 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.

4.6.3 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.

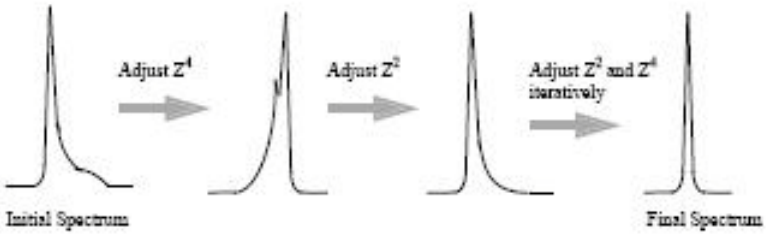
4.7 Optimizing Resolution and Lineshape

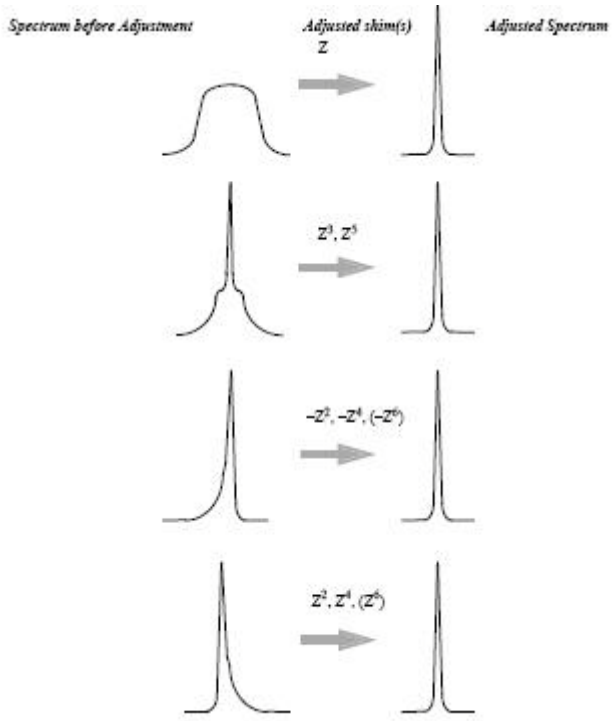
The standard sample for measuring the proton lineshape and resolution specifications is, 3% CHCL3 in Acetone-d6.

For measuring the 13C resolution and lineshape test the standard sample ASTM (60% Dioxane in 40% C6D6) 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.

The figures below are illustrating the influence of the On-axis shims on the lineshape.



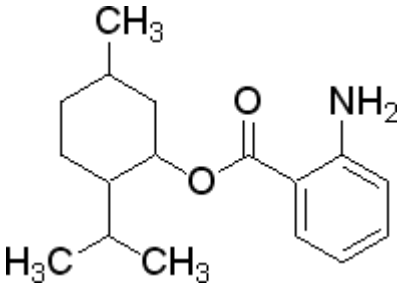


4.8 Observations

5 1-D Proton Experiment

5.1 Sample

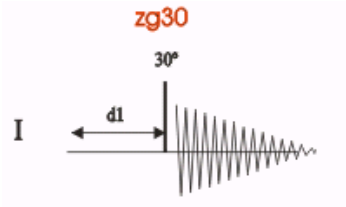
30mg Menthyl Anthranilate in DMSO-d6



5.2 1-D Proton Experiment

5.2.1 Introduction

Section 5.2 describes the acquisition and processing of a one-dimensional 1H NMR spectrum using the standard Bruker parameter set **PROTON**. The pulse sequence **zg30**, showing in the figure below, 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 30^0. The two parameters, D1 and P1, correspond to the length of the relaxation delay, and the length of the 30^0 RF pulse, respectively.



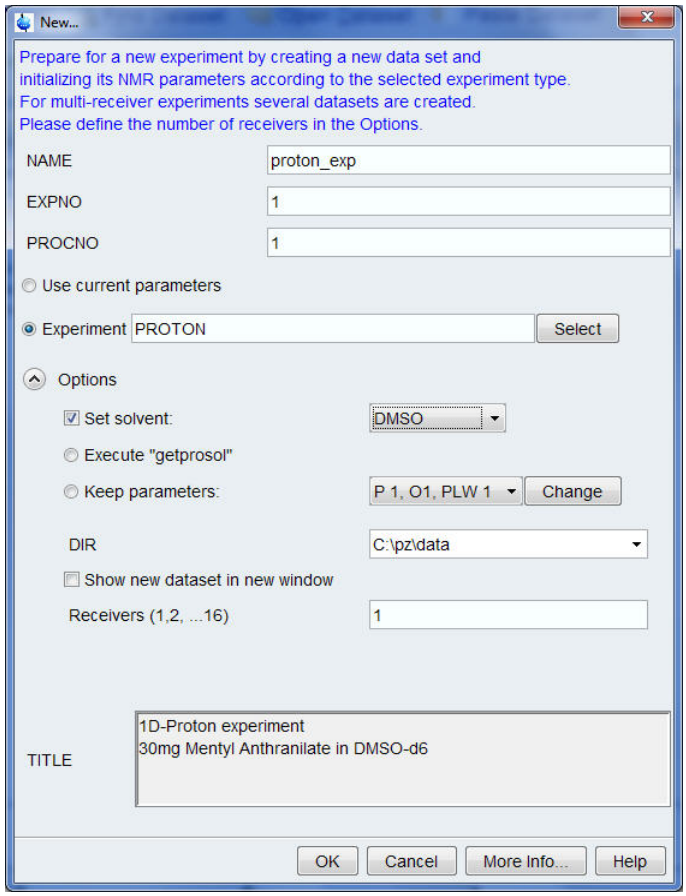
The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

5.2.2 Experiment Setup

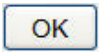
1. Click on the '**Start**' tab in the TopSpin Menu bar.

2. Select  by clicking on it.

3. Enter the following information in to the 'New' window.

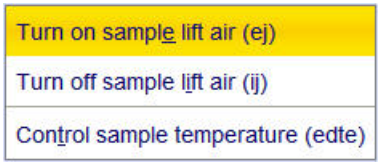


The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure above. Click on the down arrow button to browse for a specific directory.

4. Click on  .

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

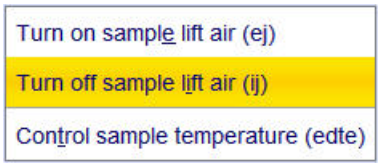
6. Select  by clicking on it.



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

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

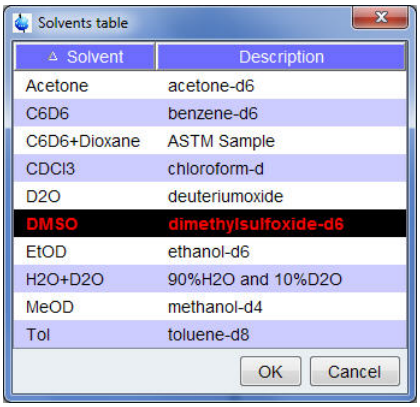
8. Place the sample on to the top of the magnet.
9. Select  by clicking on it.



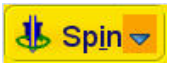
10. Select '**ij**' by clicking on it.

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

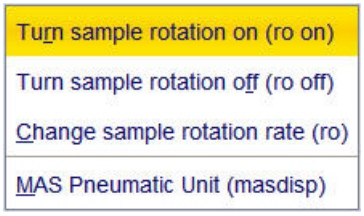
11. Select  by clicking on it.



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

13. Select 

by clicking on it.



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.

5.2.3 Acquisition

1. Select 

by clicking on it.

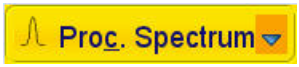
2. Select 

by clicking on it.

The experiment will take ~2 Minutes, using the default number of 16 scans.

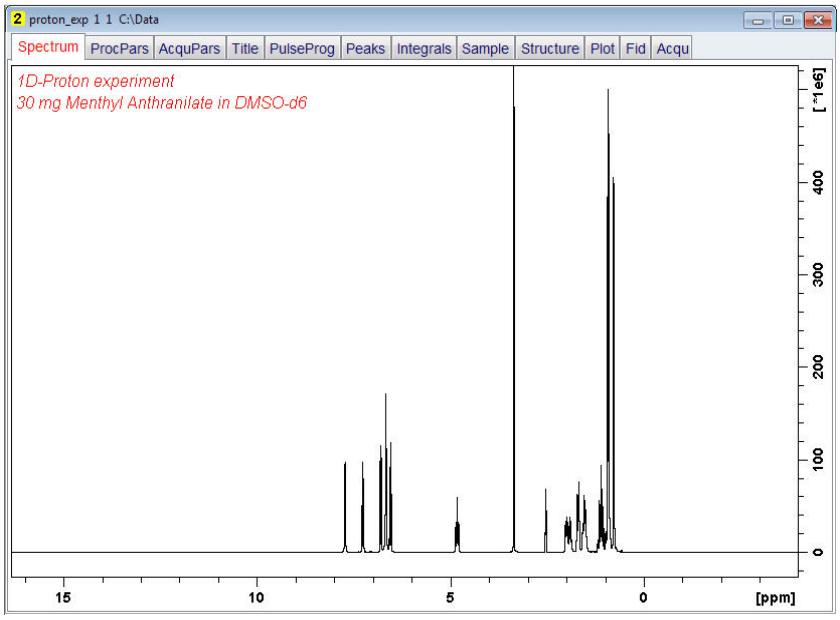
5.2.4 Processing

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

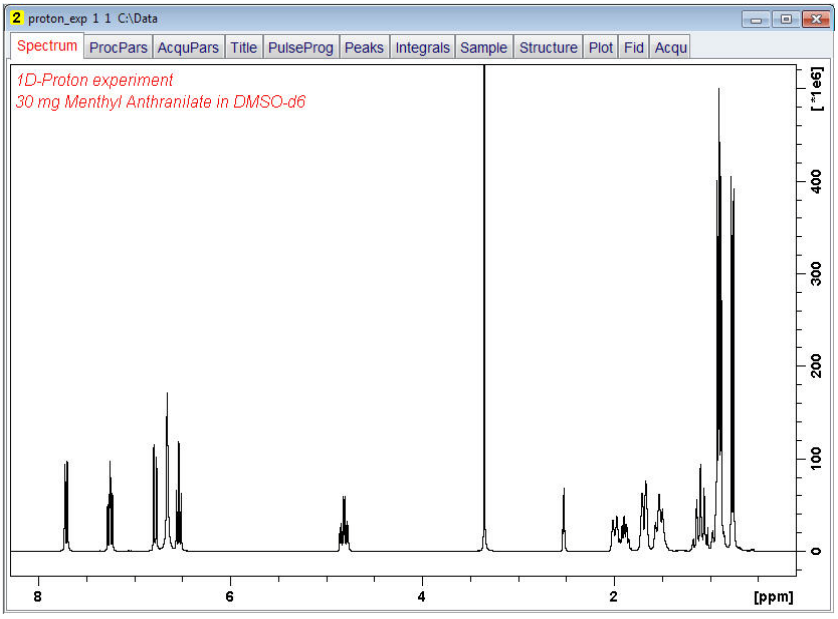
2. Click on 

.

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.

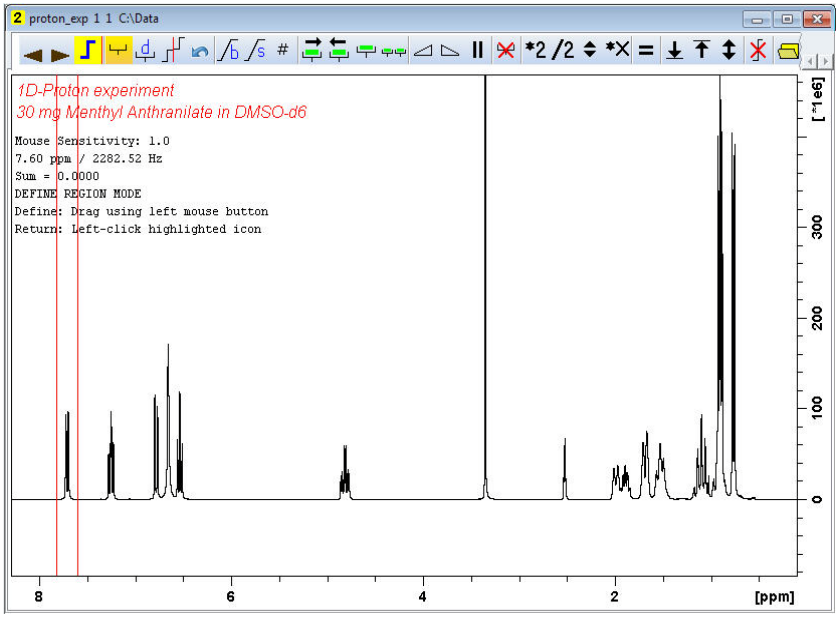


4. Click on 

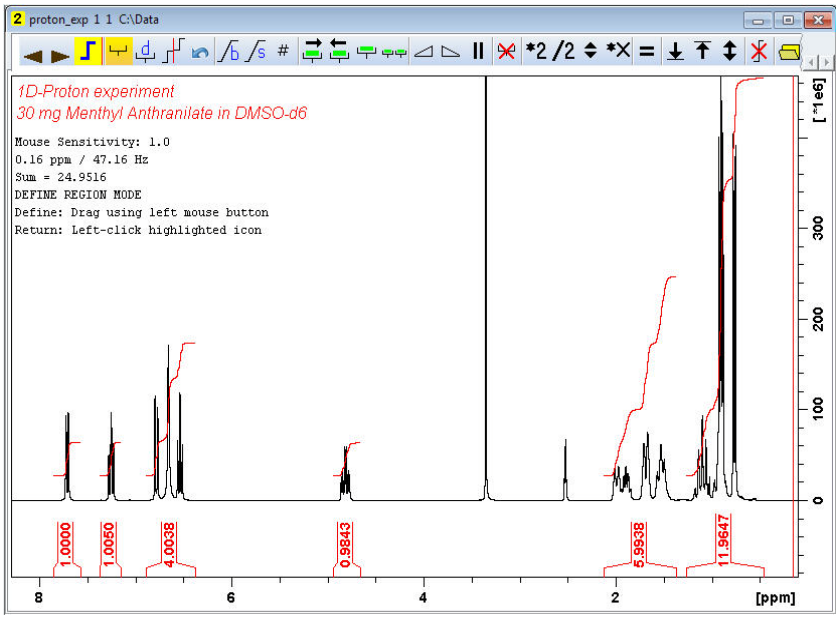
.

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.



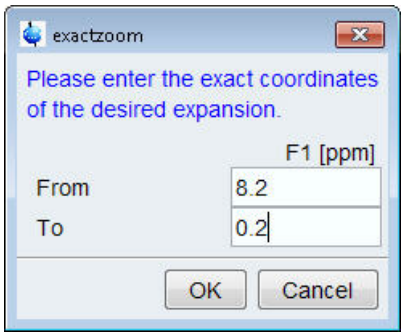
6. Repeat step 5 for the remainder of the peaks.

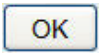


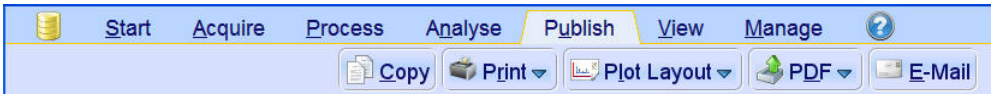
7. Click on  to save the integration regions.

5.2.5 Plotting

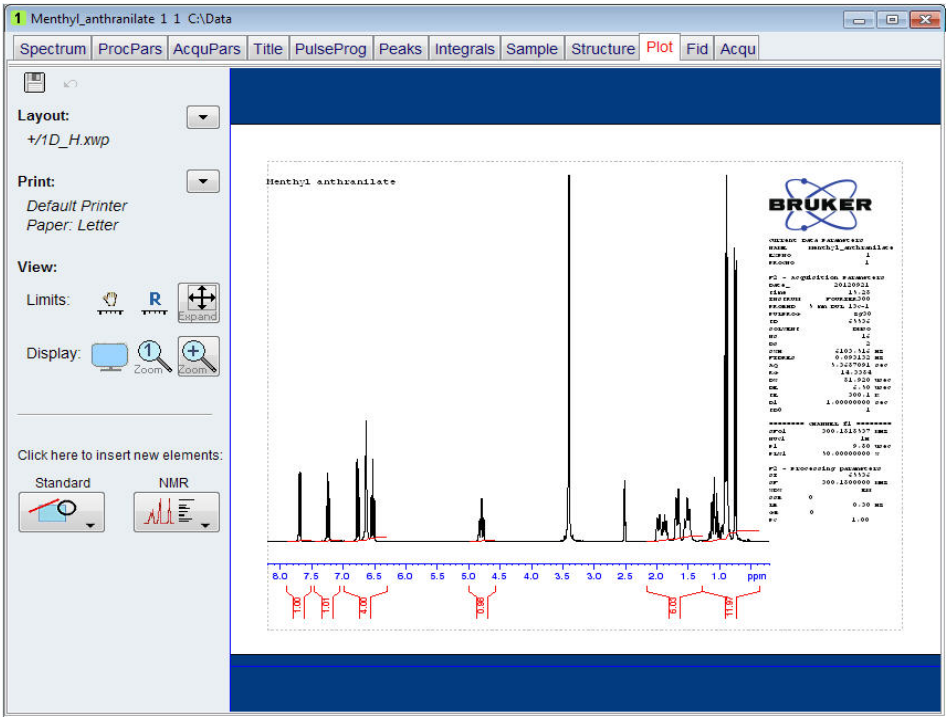
1. Expand the spectrum (all peaks in display).
2. Click on  .
3. Type the following F1 [ppm] values:
   From = **8.2**
   To = **0.2**



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

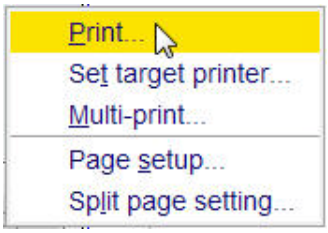


6. Click on  .



If desired, any changes can be administered by using the tools on the left side of the display.

7. Click on  in the Print section on the left side of the display.



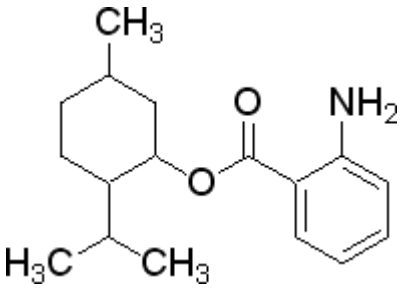
8. Select '**Print**' by clicking in it.

5.3 Observations

6 2-D Homonuclear Experiments

6.1 Sample

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



6.2 2-D Gradient COSY Experiment

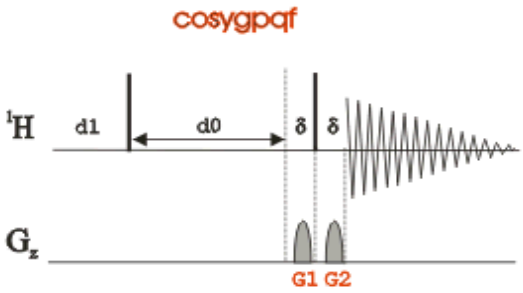
6.2.1 Introduction

The COSY experiment relies on the J-coupling to provide spin-spin correlation, and whose cross peaks indicate which 1H's are close to which other 1H'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.

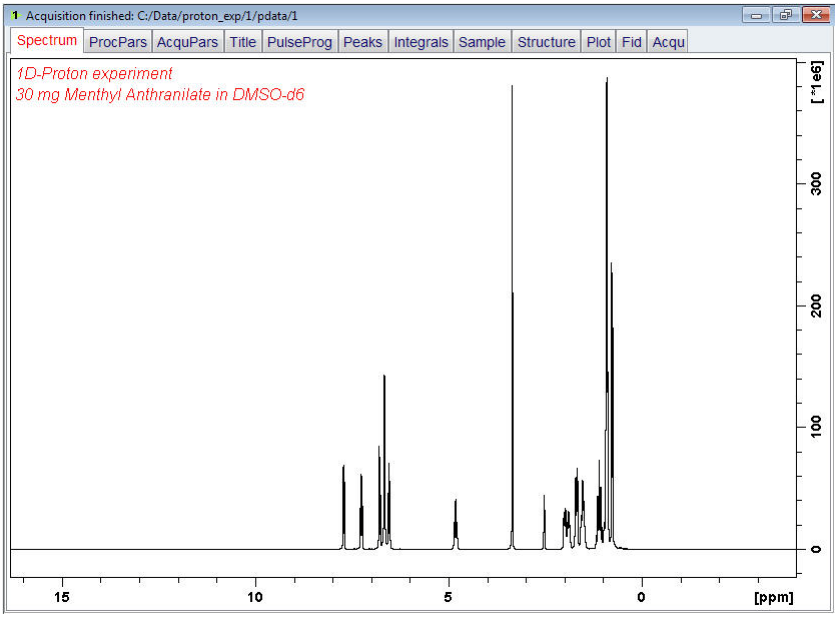
Section 6.2.3 describes the acquisition and processing of a two-dimensional 1H gradient COSY. The standard Bruker parameter set is **COSYGPSW** and includes the pulse sequence **cosygpqf** shown in the figure below. It consists of the relaxation delay, two radio frequency (RF) pulses, separated by the increment delay D0 and the acquisition time during which the signal is recorded. Both pulses have a 90^0 angle. Two gradient pulses are applied before and after the second pulse in the sequence. Purge pulses are applied before d1.



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.

6.2.2 Preparation Experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 5, 1-D Proton experiment, Paragraph 5.2.2 Experiment setup** through **5.2.4 Processing**.

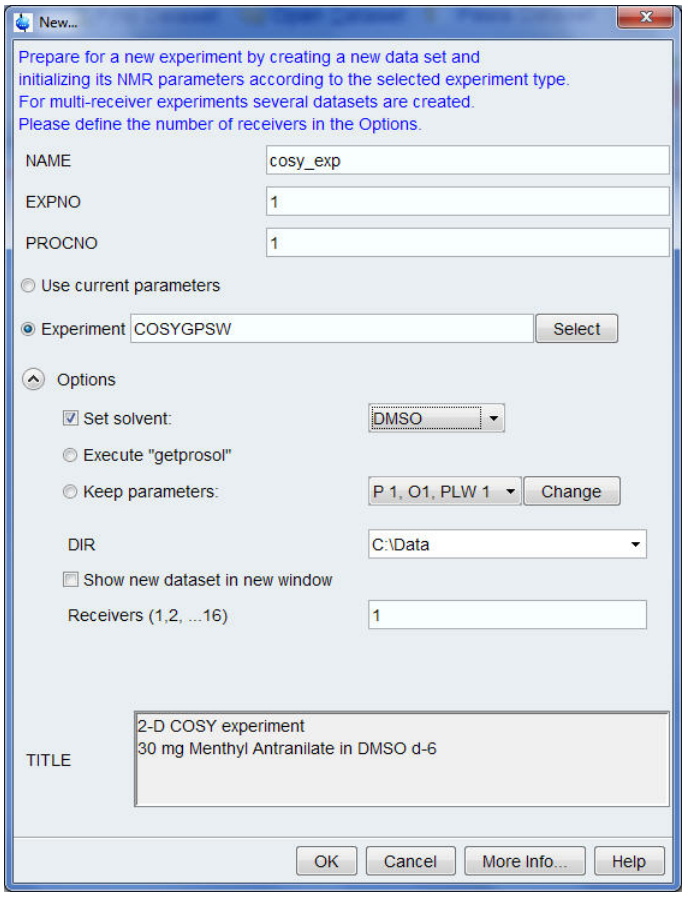


6.2.3 Setting Up The 2-D COSY Experiment

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

2. Select  by clicking on it

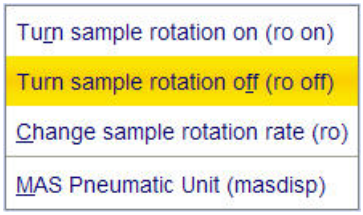
3. Enter the following information in to the 'New' window



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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.

6. Select Spin by clicking on it.



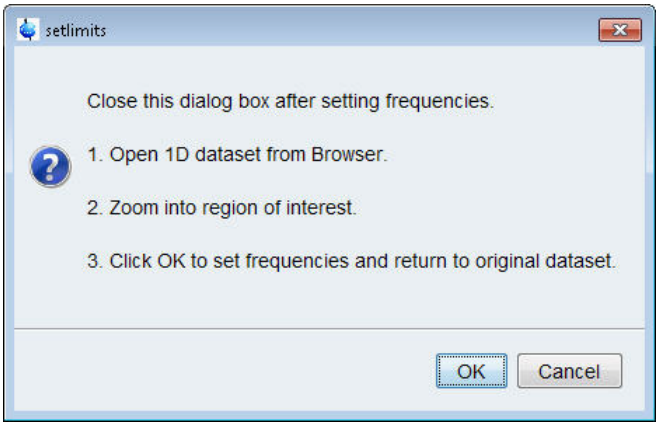
7. Select '**ro off**' by clicking on it.

2-D experiments should always be run without rotation.

8. Select Prosol by clicking on it.

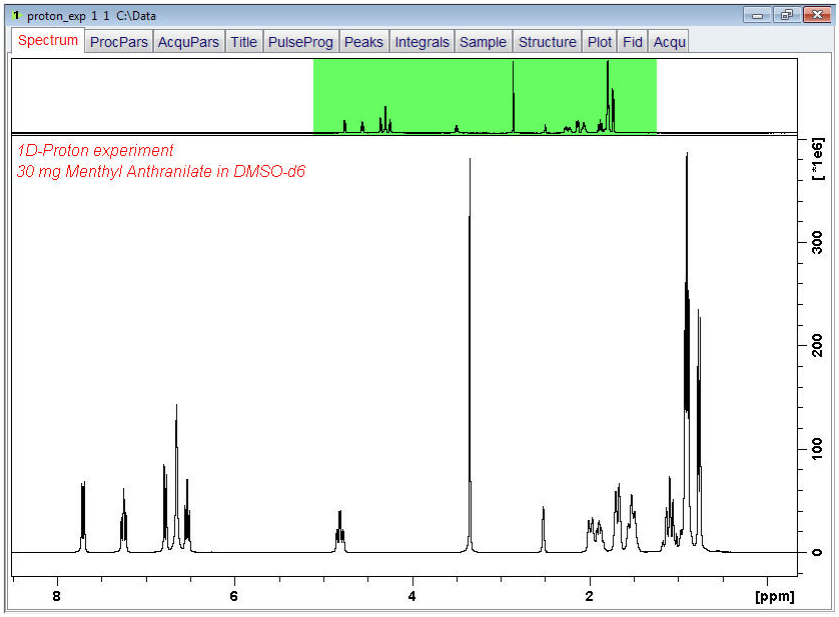
This will load the pulse width and power levels in to the parameter set.

9. Select SetLimits by clicking on it.

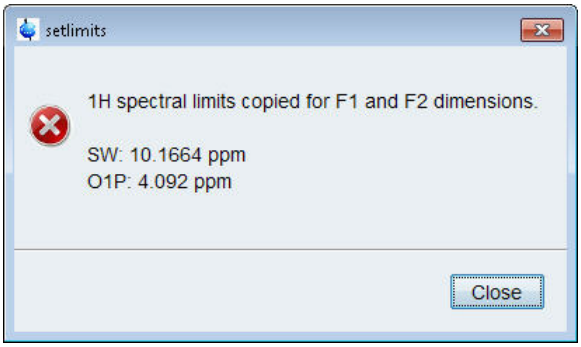


10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window.

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



12. Click on OK to assign the new limit.



13. Click on Close.

The display changes back to the 2-D data set.

6.2.4 Acquisition

1. Select 

by clicking on it.

2. Select 

by clicking on it.

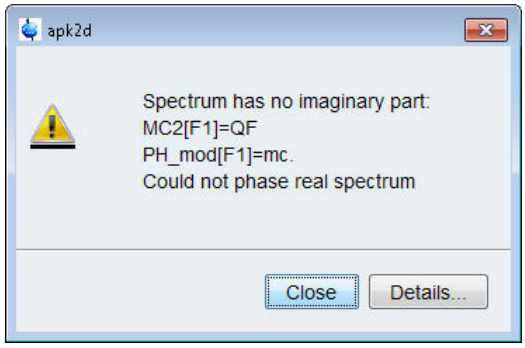
The experiment will take ~5 Minutes, using the default number of 1 scan and 128 increments.

6.2.5 Processing

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

2. Select 

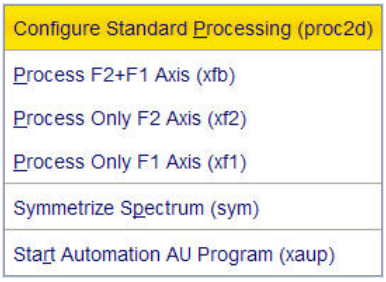
by clicking on it.



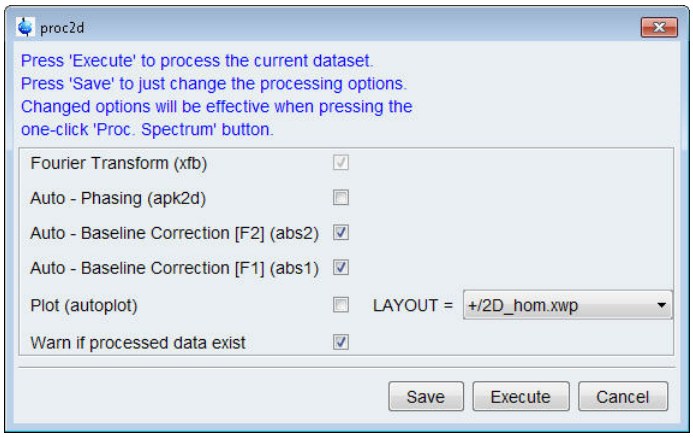
This executes a standard processing program **proc2**. The message shown in the figure above, pops up in case of a magnitude 2D experiment and the apk2d option is enabled. To configure the processing program follow the steps below.

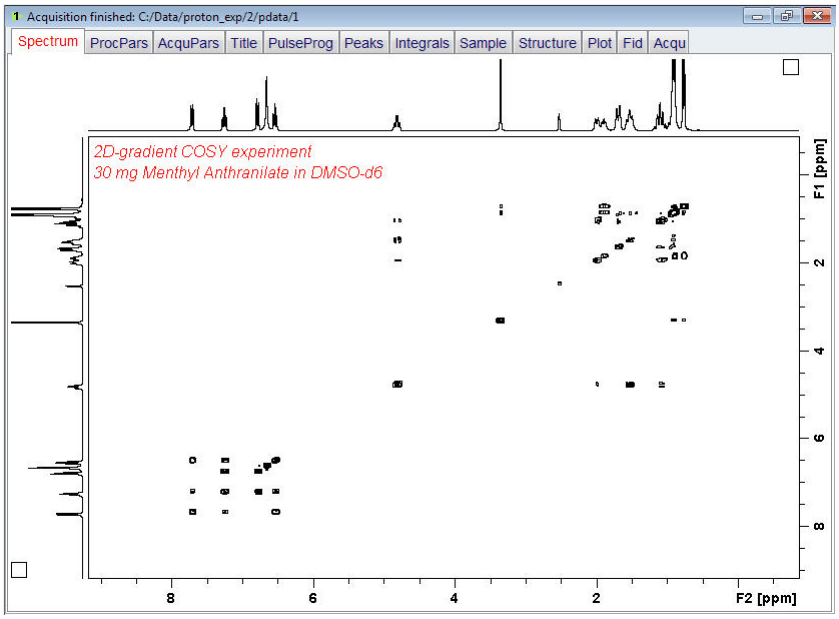
3. Click on the down arrow inside the 

button.



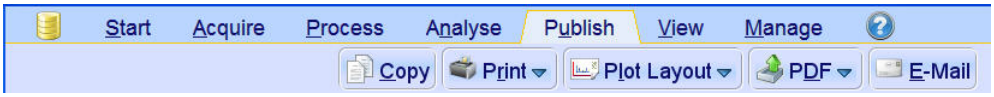
4. Select '**Configure Standard Processing**' by clicking on it.



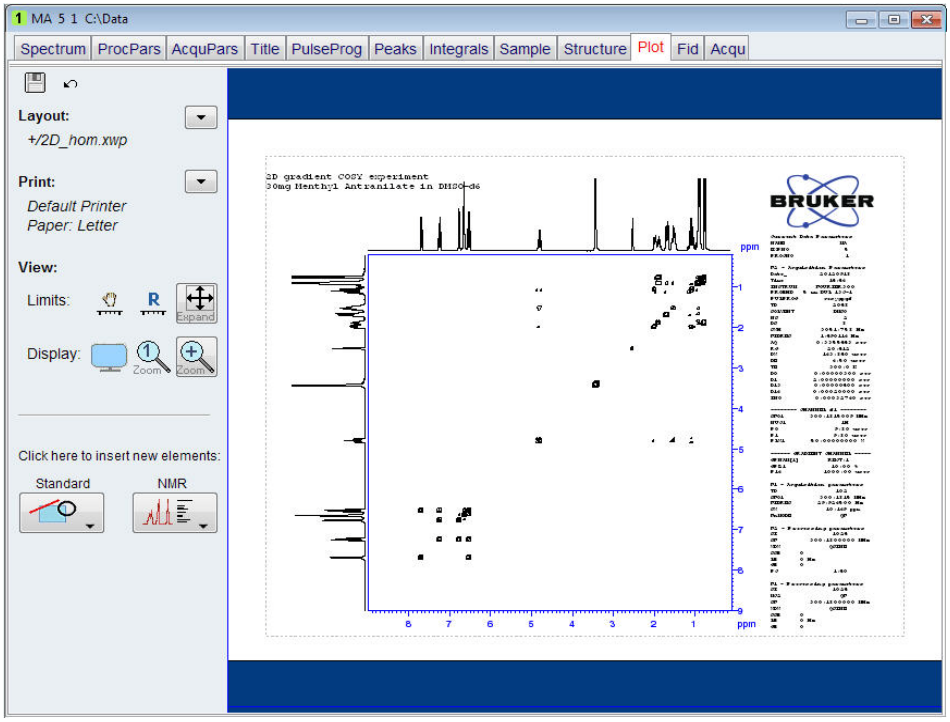


6.2.6 Plotting

1. Use the  buttons to adjust for a suitable contour level.
2. Type **.ls** or click on  to save the contour levels to disk.
3. Click on the '**Publish**' tab in the TopSpin Menu bar.

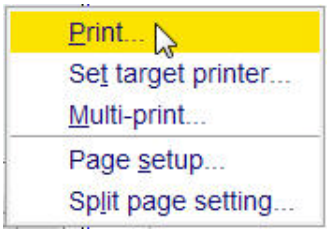


4. Click on 



If desired, any changes can be administered by using the tools on the left side of the display.

5. Click on  in the Print section on the left side of the display.



6. Select '**Print**' by clicking in it.

6.3 Observations

6.4 2-D Gradient NOESY Experiment

6.4.1 Introduction

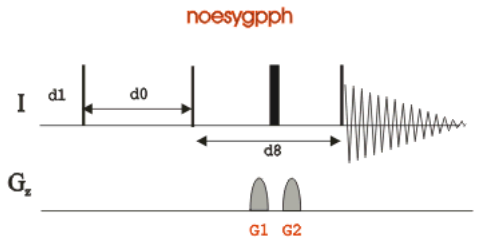
NOESY (Nuclear Overhauser Effect SpectroscopY) is a 2D spectroscopy method used to identify spins undergoing cross-relaxation and to measure the cross-relaxation rates. Most commonly, NOESY is used as a homonuclear 1H technique. In NOESY, direct dipolar couplings provide the primary means of cross-relaxation, 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 1H's are close to which other 1H'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 t1, 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 d8. Note that, for the basic NOESY experiment, d8 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 t2. The NOESY spectrum is generated by a 2D Fourier transform with respect to t1 and t2.

Axial peaks, which originate from magnetization that has relaxed during tm, 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.

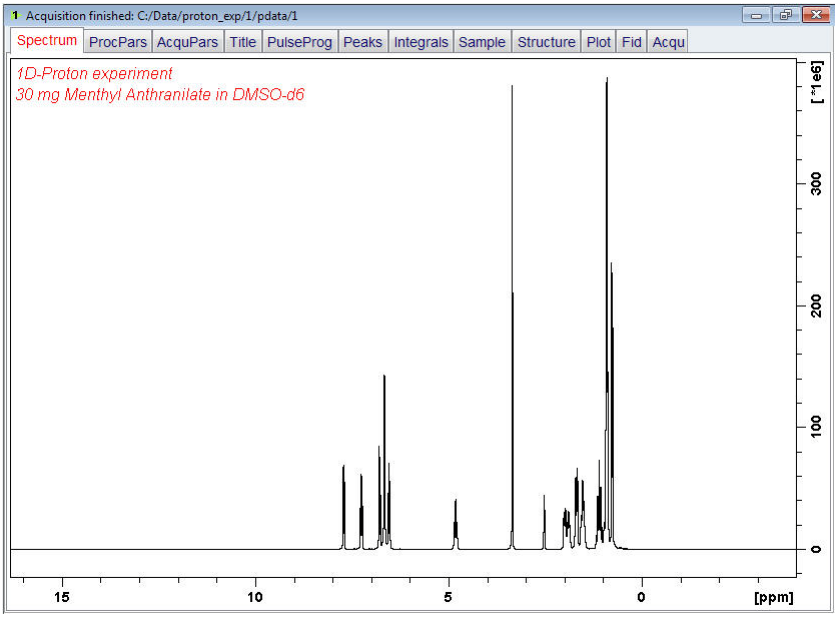
Section 6.4.3 describes the acquisition and processing of a two-dimensional 1H phase sensitive NOESY. The standard Bruker parameter set is **NOESYGPPHSW** and includes the pulse sequence **noesygpph** shown in the figure below. It consists of the relaxation delay, three radio-frequency (RF) pulses, separated by the increment delay D0 between the first and second pulse, a mixing time D8 between the second and third pulse and the acquisition time during which the signal is recorded. All three pulses are of 90^0.



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.

6.4.2 Preparation Experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 5, 1-D Proton experiment, Paragraph 5.2.2 Experiment setup** through **5.2.4 Processing**.

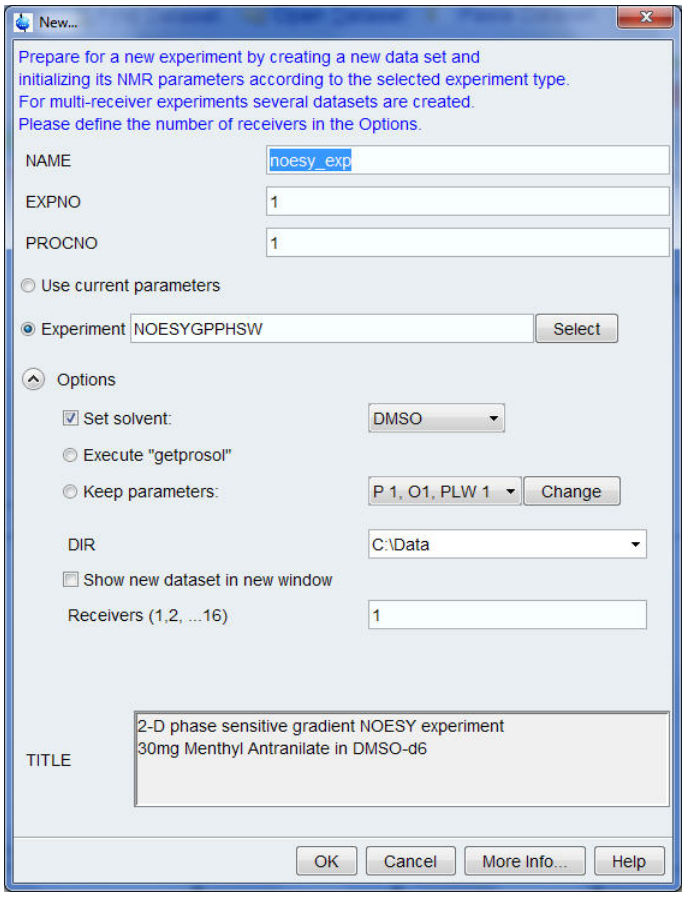


6.4.3 Setting Up The 2-D NOESY Experiment

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

2. Select  by clicking on it.

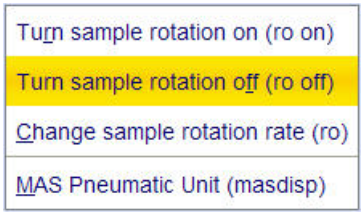
3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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.

6. Select Spin by clicking on it.



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

2-D experiments should always be run without rotation.

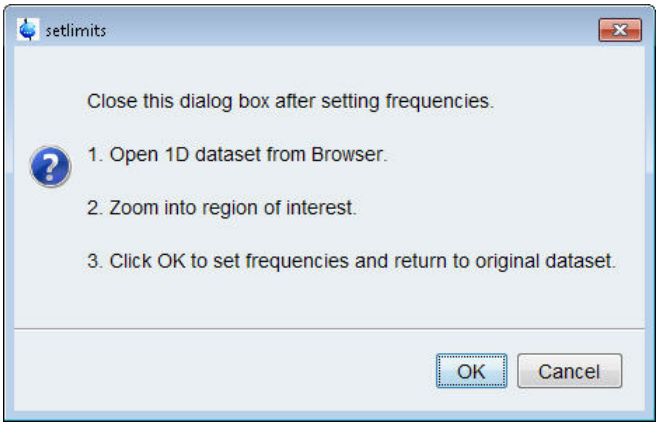
8. Select 

by clicking on it.

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

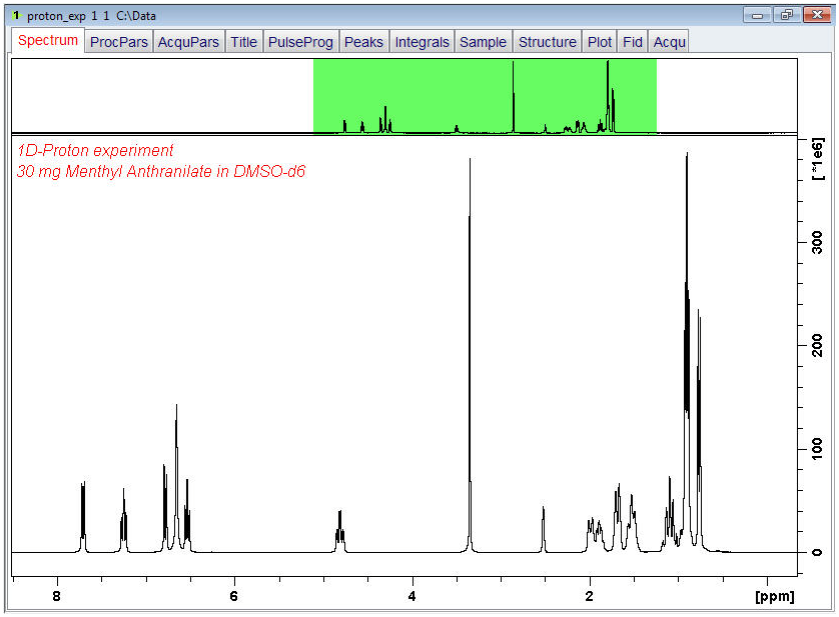
9. Select 

by clicking on it.

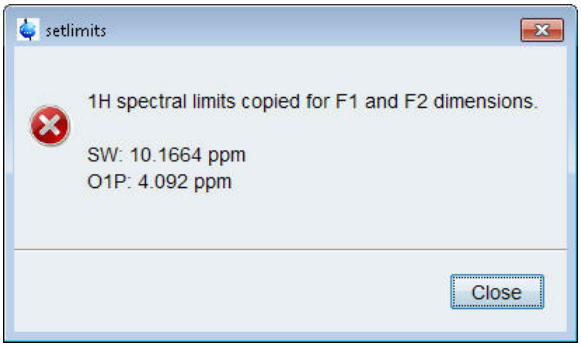


10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window.

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



12. Click on OK to assign the new limit.



13. Click on Close.

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

N14. Select the '**AcquPars**' tab by clicking on it.

15. Click on to display the pulse program parameters.

16. Make the following change.

D8 [sec]	0.450	Mixing time

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

6.4.4 Acquisition

1. Select 

by clicking on it.

2. Select 

by clicking on it.

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

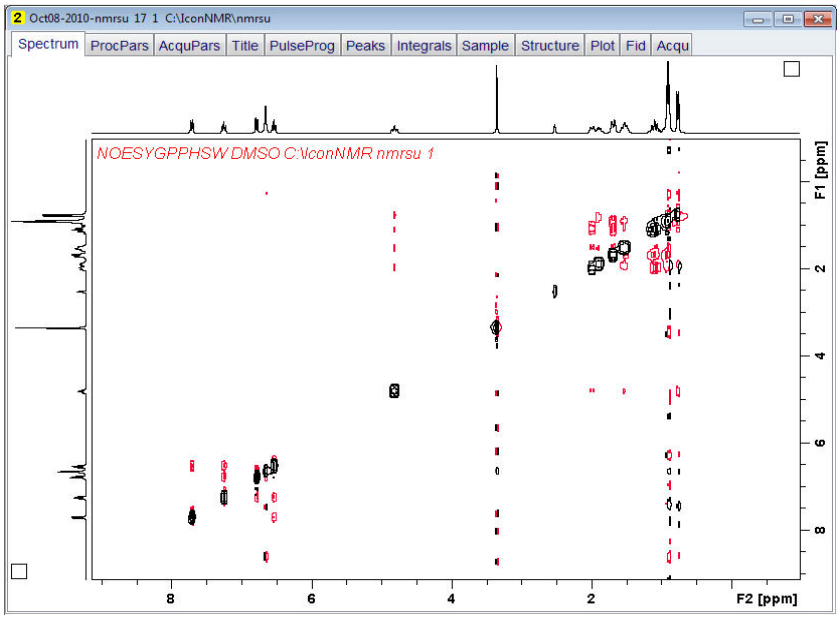
6.4.5 Processing

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

2. Select 

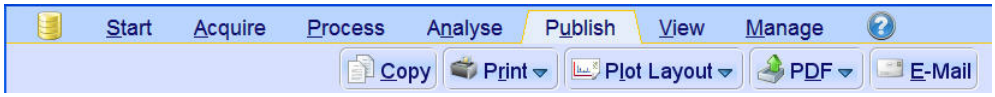
by clicking on it.

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.

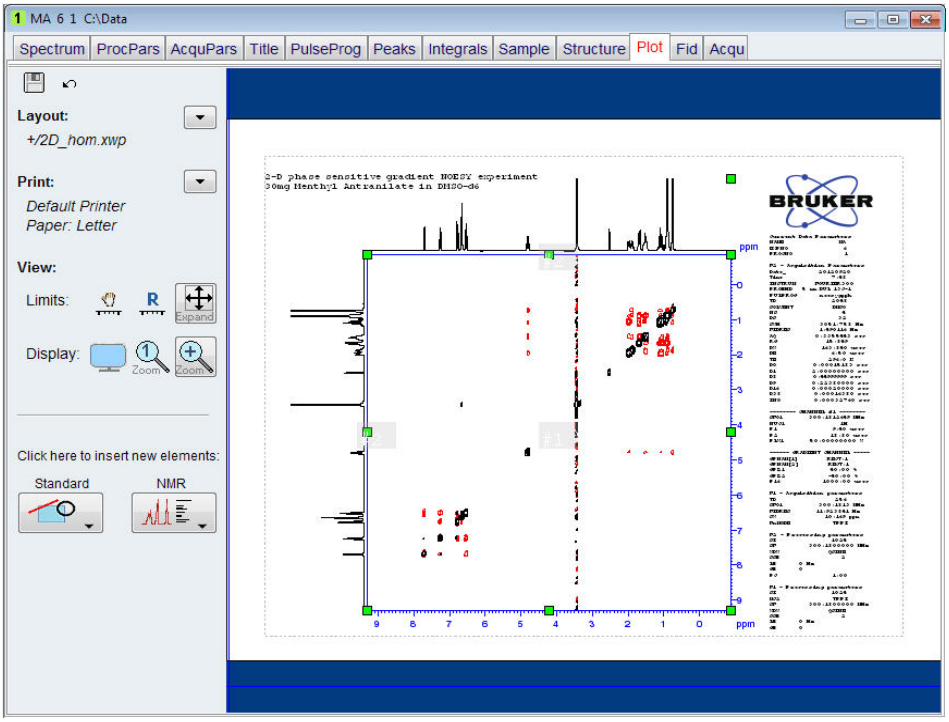


6.4.6 Plotting

1. Use the  buttons to adjust for a suitable contour level.
2. Type **.ls** or click on  to save the contour levels to disk.
3. Click on the '**Publish**' tab in the TopSpin Menu bar.

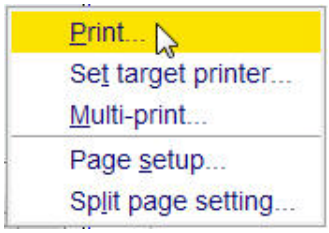


4. Click on 



If desired, any changes can be administered by using the tools on the left side of the display.

5. Click on  in the Print section on the left side of the display.



6. Select '**Print**' by clicking in it.

6.5 Observations

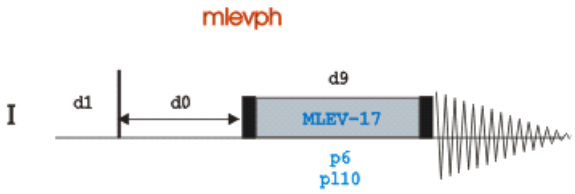
6.6 2-D Phase Sensitive TOCSY Experiment

6.6.1 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 spincoupled 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 JHH) should be allowed for each transfer step, and five transfer steps are typically desired for the TOCSY spectrum.

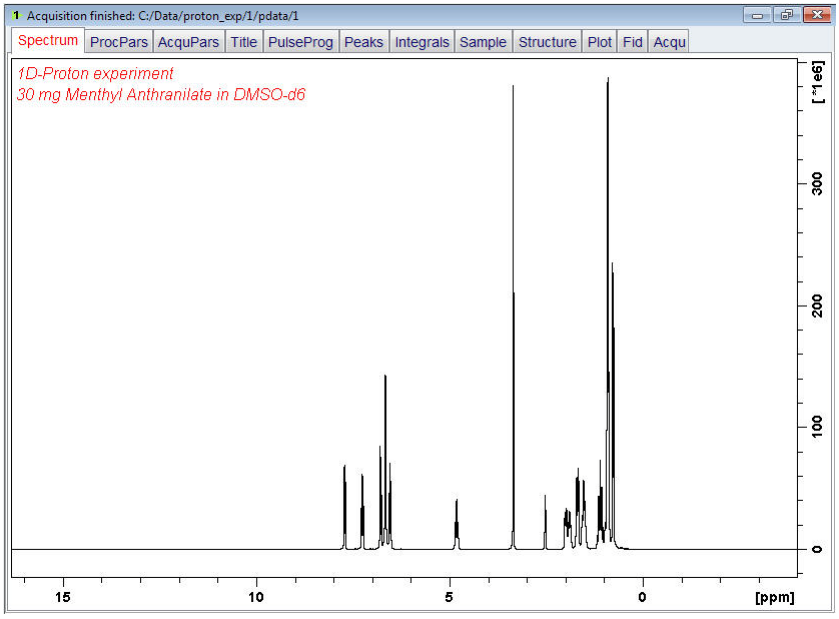
Section 6.6.3 describes the acquisition and processing of a two-dimensional 1H phase sensitive TOCSY. The standard Bruker parameter set is **MLEVPHSW** and includes the pulse sequence **mlevph** shown in the figure below. It consists of the recycling delay, two radio-frequency (RF) pulses, separated by the increment delay D0 and the acquisition time during which the signal is recorded. The first RF pulse is a 90^0 pulse, the second pulse is the mlev spinlock pulse.



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.

6.6.2 Preparation Experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 5, 1-D Proton experiment, Paragraph 5.2.2 Experiment setup** through **5.2.4 Processing**.

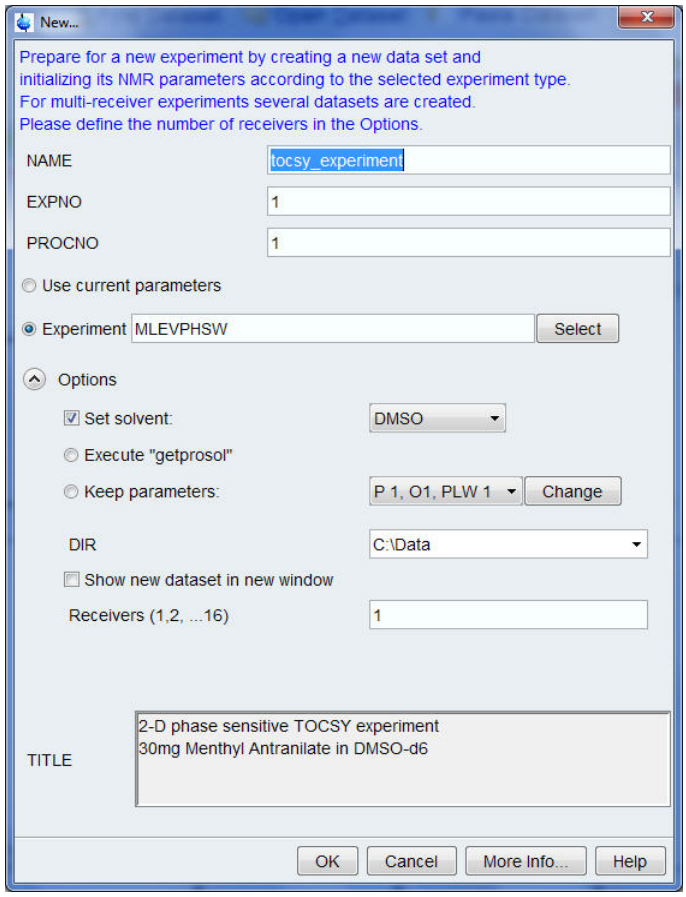


6.6.3 Setting Up The 2-D TOCSY Experiment

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

2. Select  by clicking on it.

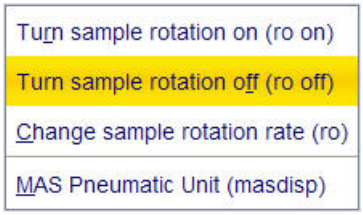
3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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.

6. Select Spin by clicking on it.



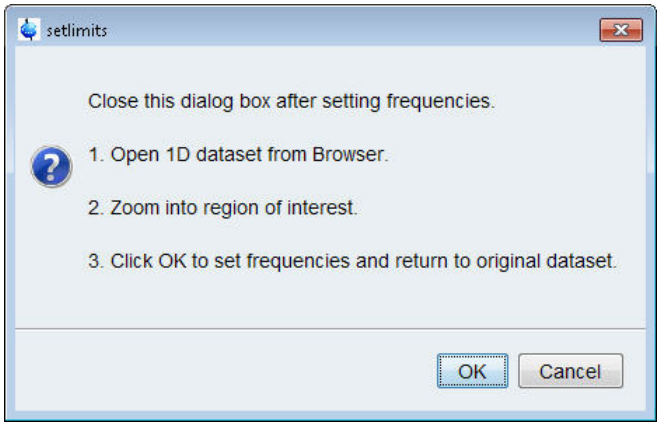
7. Select '**ro off**' by clicking on it.

2-D experiments should always be run without rotation.

8. Select Prosol by clicking on it.

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

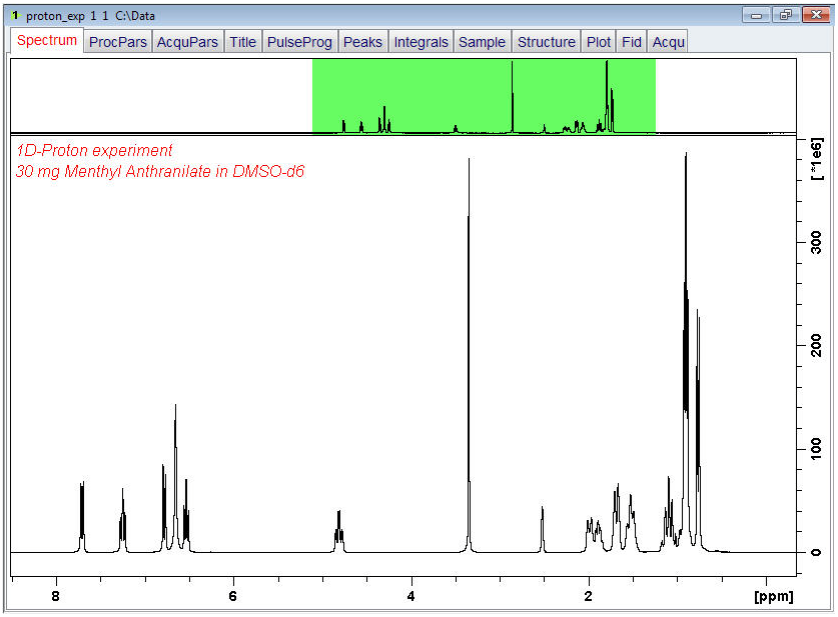
9. Select SetLimits by clicking on it.



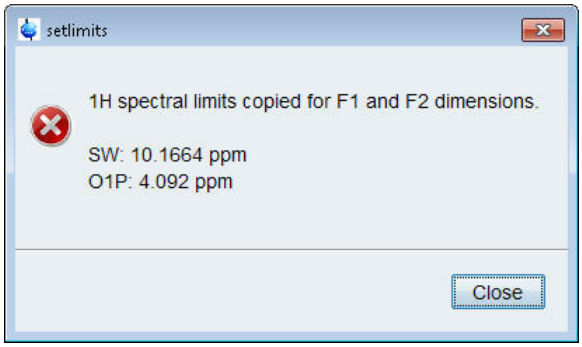
10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window.

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

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.



12. Click on OK to assign the new limit.



13. Click on Close.

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.

6.6.4 Acquisition

1. Select 

by clicking on it.

2. Select 

by clicking on it.

The experiment will take ~42 Minutes, using 8 scans and 128 increments.

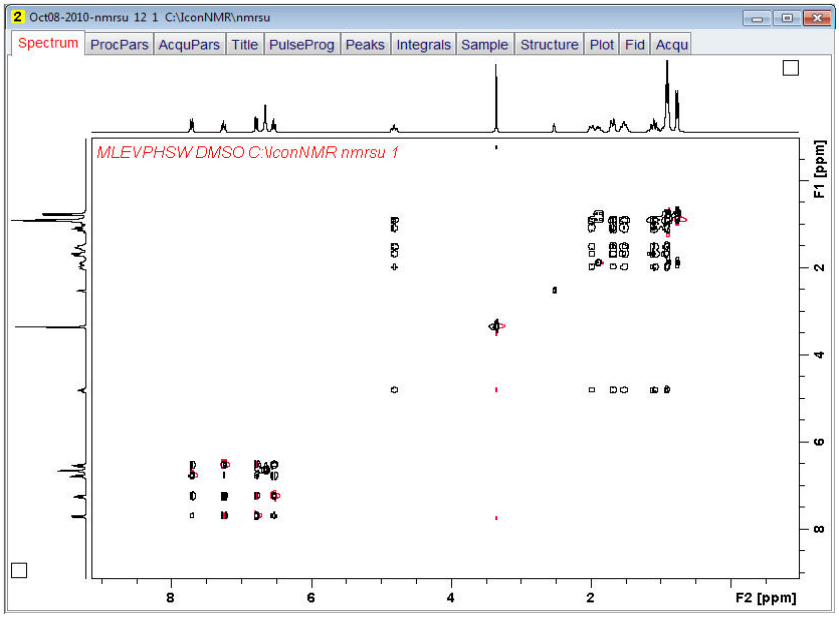
6.6.5 Processing

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

2. Select 

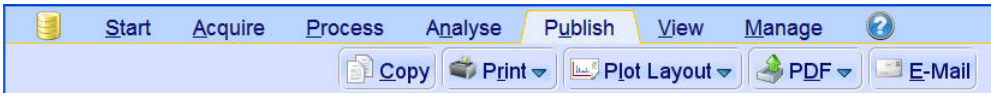
by clicking on it.

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.

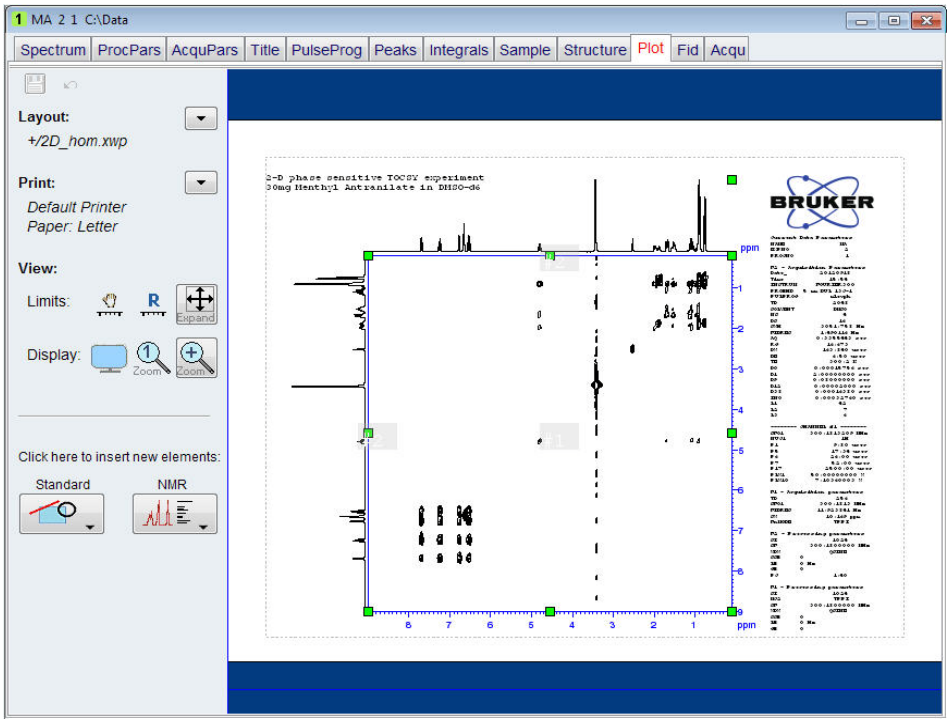


6.6.6 Plotting

1. Use the  buttons to adjust for a suitable contour level.
2. Type **.ls** or click on  to save the contour levels to disk.
3. Click on the '**Publish**' tab in the TopSpin Menu bar.

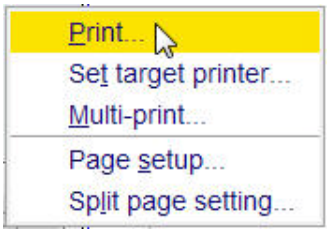


4. Click on  .
5. Select the '**Plot**' tab by clicking on it.



If desired, any changes can be administered by using the tools on the left side of the display.

6. Click on  in the Print section on the left side of the display.



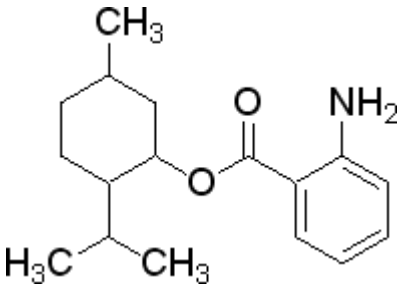
7. Select '**Print**' by clicking in it.

6.7 Observations

7 1-D Carbon Experiments

7.1 Sample

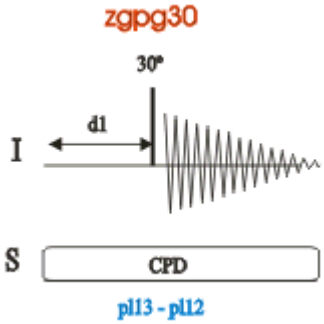
A sample of **30mg Menthyl Anthranilate in DMSO-d6** is used for the experiments in this chapter



7.2 1-D Carbon Experiment

7.2.1 Introduction

Section 7.2.2 describes the acquisition and processing of a one-dimensional 13C NMR spectrum. The standard Bruker parameter set **C13CPD**, includes the pulse sequence **zgpg30**, shown in the figure below. The 13C 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 30^0. The two parameters, D1 and P1, correspond to the length of the relaxation delay, and the length of the 90^0 RF pulse, respectively. The 1H 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 then 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.



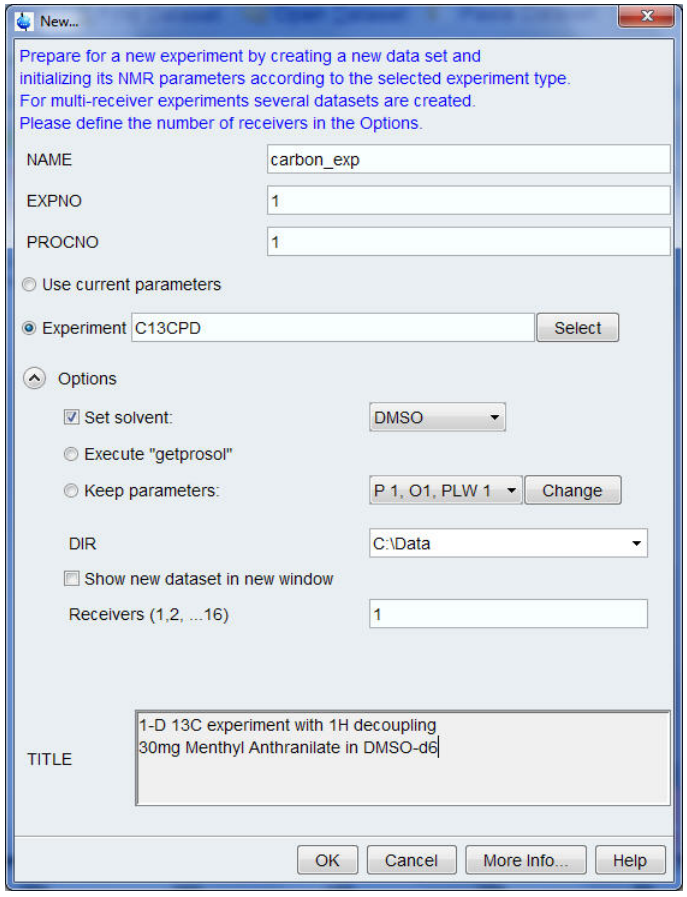
The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

7.2.2 Experiment Set Up

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

2. Select  by clicking on it.

3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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 = 128

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

8. Select  by clicking on it.

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

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.

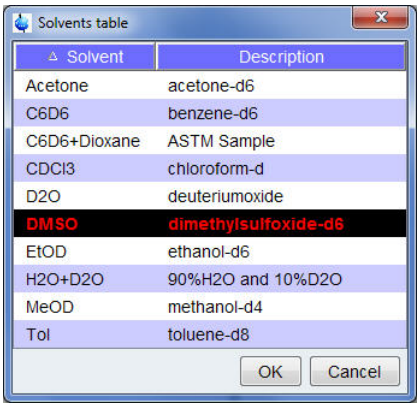
11. Select  by clicking on it.

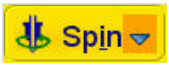
12. Select '**ij**' by clicking on it.

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

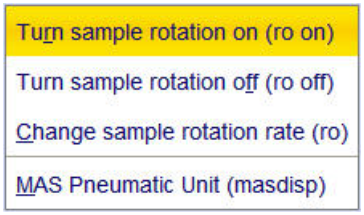
---

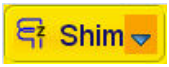
13. Select  by clicking on it.



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

by clicking on it.



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

by clicking on it.
18. Select 

by clicking on it.

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

7.2.3 Acquisition

1. Select 

by clicking on it.
2. Select 

by clicking on it.

The experiment will take ~7 Minutes, using 128 scans.

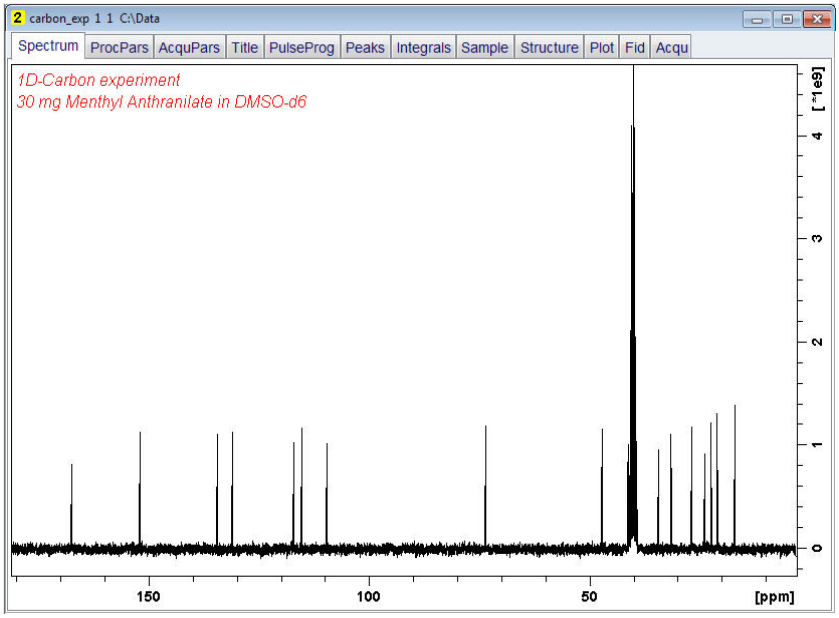
7.2.4 Processing

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

2. Select  by clicking on it.

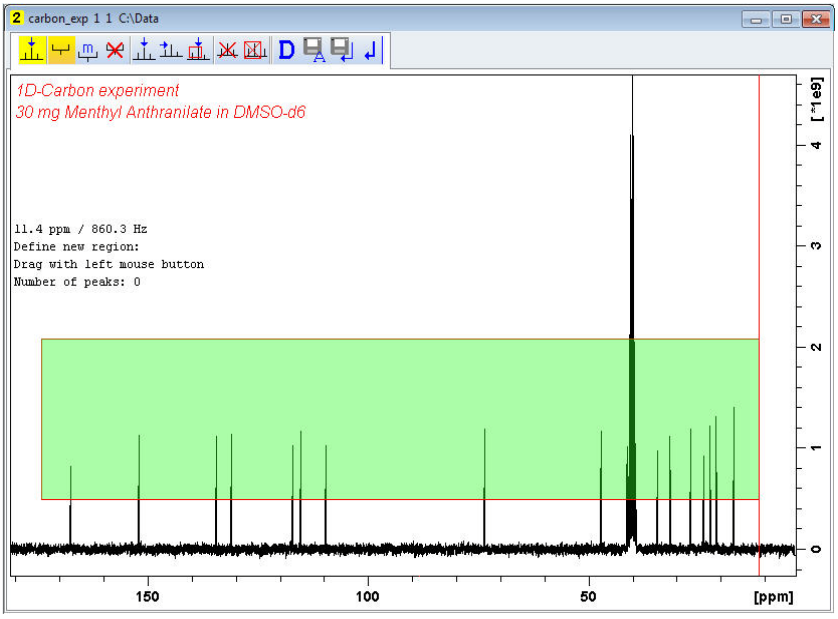
This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**'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

3. Expand the spectrum to include all peaks.



4. Select  by clicking on it.

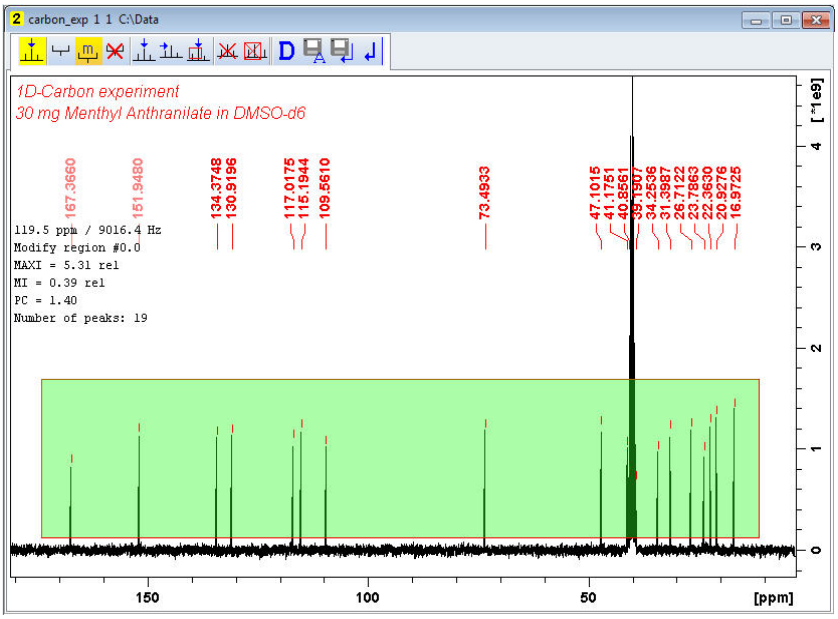
5. Click the left mouse button and drag the cursor line from left to the right side of the spectrum.



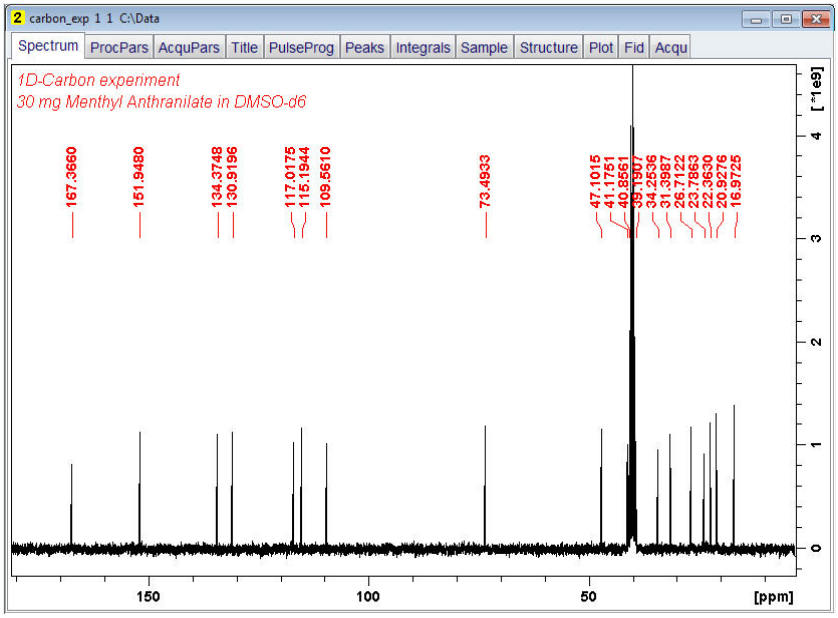
6. Click on  to manually adjust the minimum and maximum intensity levels.

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.



9. Click on  to store the peak picking values.



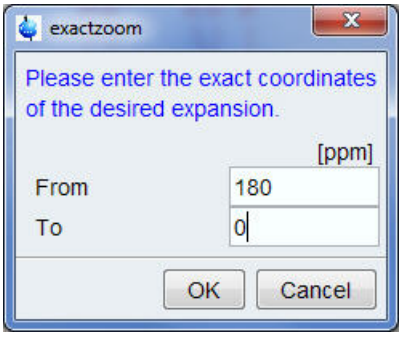
To display the peak picking labels, right click inside the spectrum window and select '**Spectra Display Preferences**' by clicking on it. In the '**Spectrum components**' enable '**Peak labels**' and '**Peak annotations**'. Click '**Apply**' and click on '**Close**'.

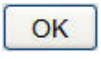
7.2.5 Plotting

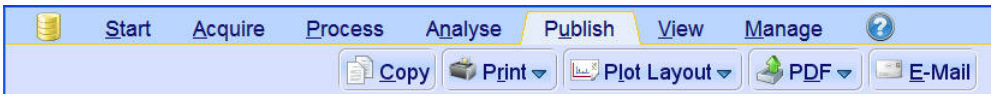
1. Expand the spectrum (all peaks in display).
2. Click on  .
3. Type the following F1 [ppm] values:

   From = **180**

   To = **0**

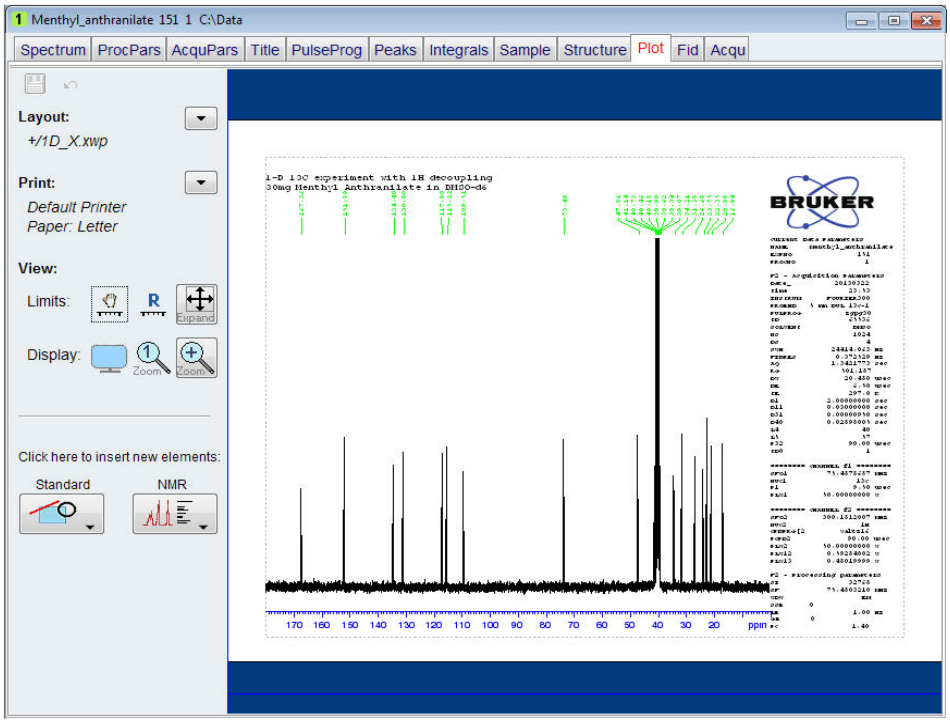


4. Click on  .
5. Click on the '**Publish**' tab in the TopSpin Menu bar.



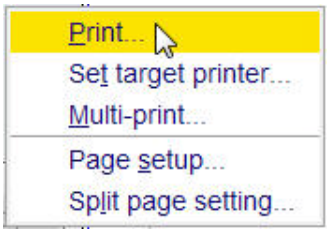
6. Click on 

.



If desired, any changes can be administered by using the tools on the left side of the display.

7. Click on  in the Print section on the left side of the display.



8. Select '**Print**' by clicking in it.

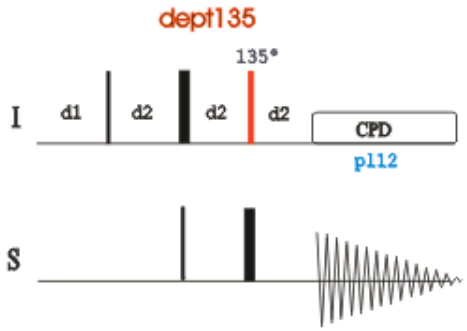
7.3 Observations

7.4 DEPT-135 Experiment

7.4.1 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 1H (most commonly 13C). DEPT is a spectral editing sequence, that is, it can be used to generate separate 13C sub spectra for methyl (CH3), methylene (CH2), and methine (CH) signals. DEPT makes use of the generation and manipulation of multiple quantum coherence to differentiate between the different types of 13C signals. Quaternary carbons are missing a direct bond proton, and as a result are absent from all DEPT spectra.

Section 7.4.2 describes the acquisition and processing of a one-dimensional 13C-DEPT135 NMR spectrum. The standard Bruker parameter set **C13DEPT135**, includes the pulse sequence **dept135**, shown in the figure below. The 13C 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*J(XH). The 1H channel consists of three pulses, a 90^0, a 180^0, followed by a 135^0 RF pulse and are separated by the editing delay D2. The final 135^0 1H pulse selects the CH3, CH2 or CH signals. The protons are decoupled during the acquisition period.



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.

7.4.2 Experiment Set Up

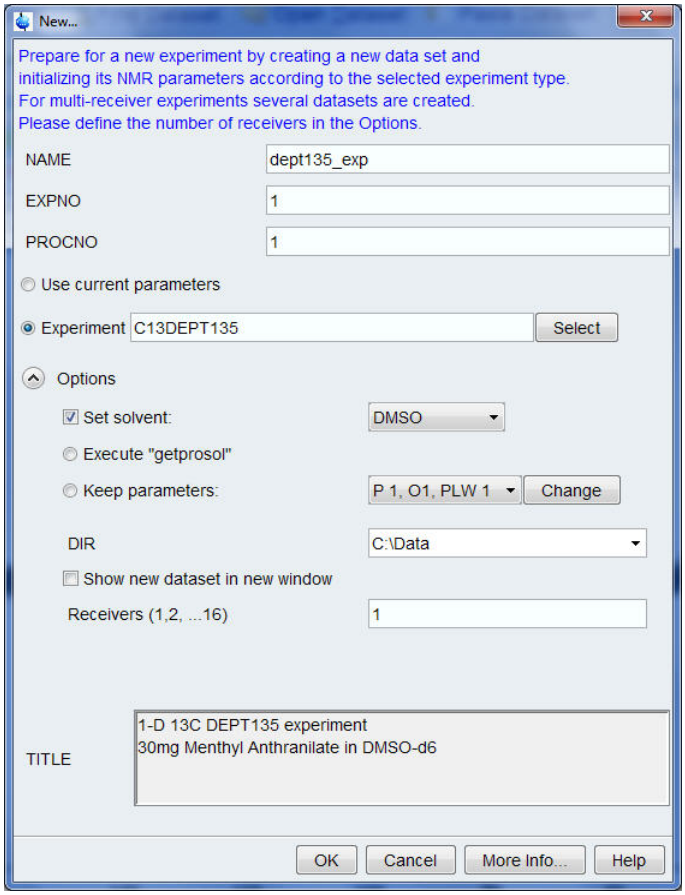
This experiment usually follows a regular Proton decoupled Carbon experiment. The result of a DEPT-135 experiment shows only the protonated carbons with the CH and CH3 as positive and the CH2 as negative signals.

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

2. Select 

by clicking on it.

3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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 = **64**

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

8. Select Prosol by clicking on it.

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

7.4.3 Acquisition

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

The experiment will take ~4 Minutes, using 64 scans.

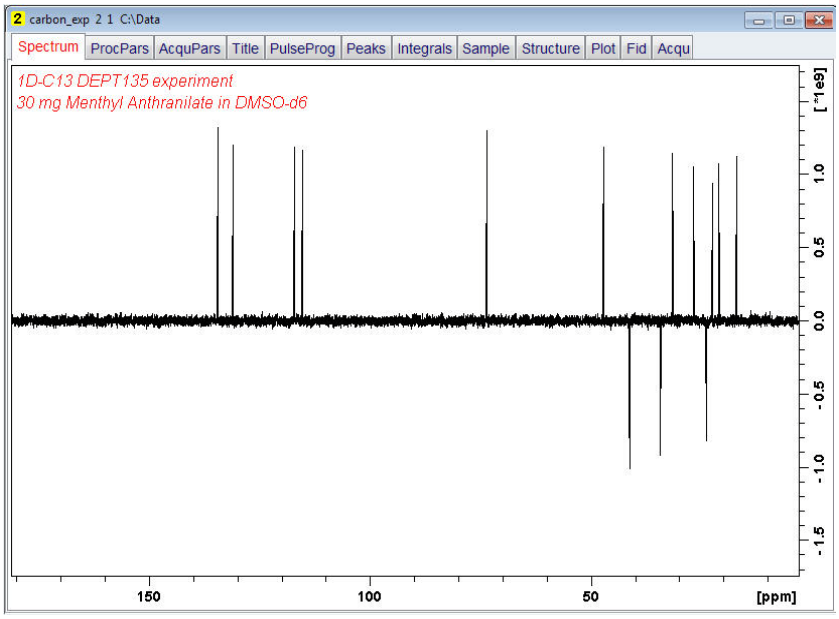
7.4.4 Processing

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

2. Select 

by clicking on it.

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.

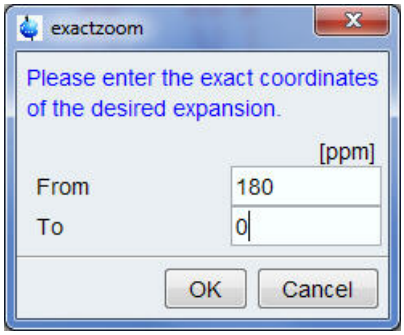


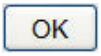
7.4.5 Plotting

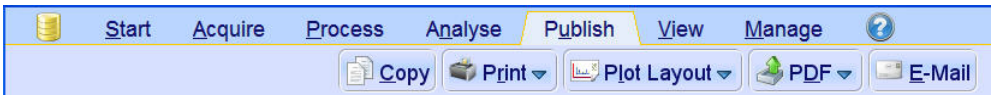
1. Expand the spectrum (all peaks in display).
2. Click on  .
3. Type the following F1 [ppm] values:

   From = **180**

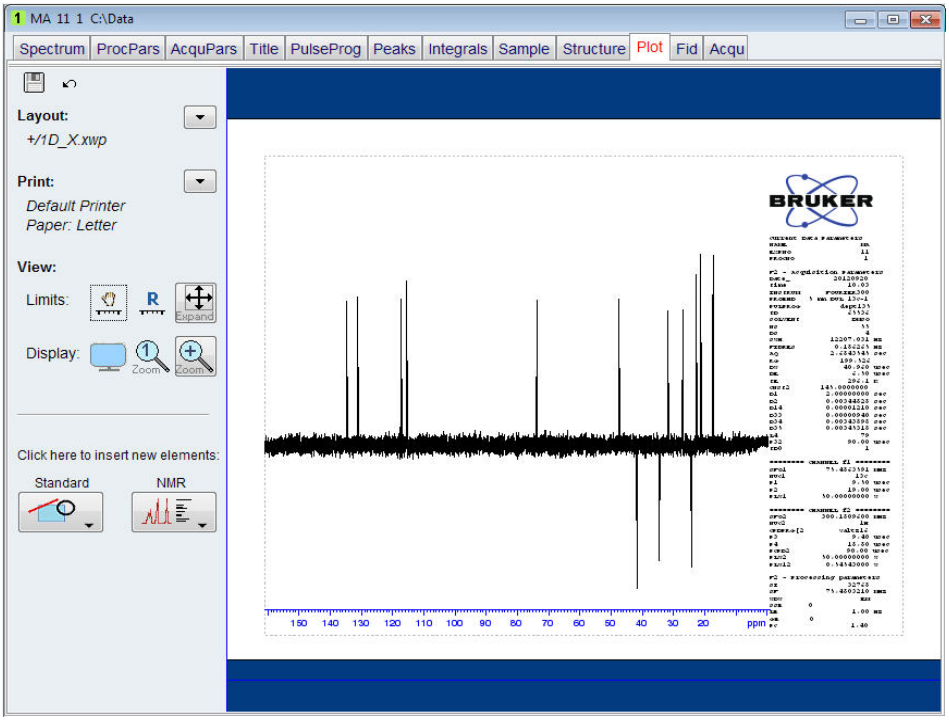
   To = **0**



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

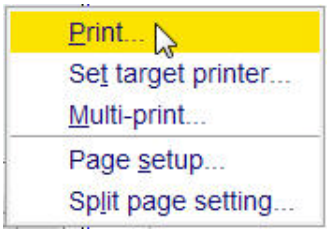


6. Click on  .



If desired, any changes can be administered by using the tools on the left side of the display.

7. Click on  in the Print section on the left side of the display.



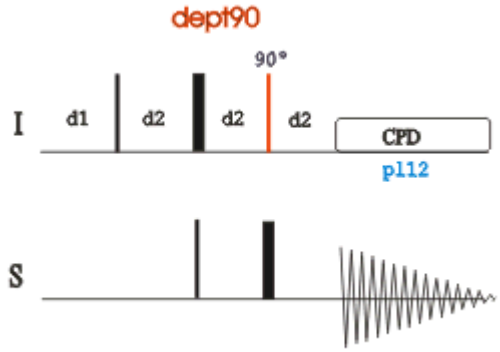
8. Select '**Print**' by clicking in it.

7.5 Observations

7.6 DEPT-90 Experiment

7.6.1 Introduction

Section 7.6.2 describes the acquisition and processing of a one-dimensional 13C-DEPT90 NMR spectrum. The standard Bruker parameter set **C13DEPT90**, includes the pulse sequence **dept90**, shown in the figure 7below. The 13C channel consists of the relaxation delay, a 90^0 RF pulse, an editing delay D2 followed by a 180^0 RF pulse and the acquisition time during which the signal is recorded. The editing delay D2 is 1/2*J(XH). The 1H channel consists of three pulses, a 90 degree, a 180 degree, followed by a 90^0 RF pulse and are separated by the editing delay D2. The final 90^0 1H pulse selects the CH signals only. The protons are decoupled during the acquisition period.

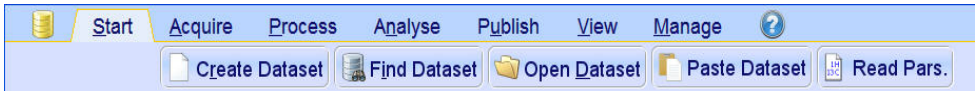


The time intervals depicted in the pulse sequence diagrams are not drawn to scale. For example, d1 is typically a few seconds while p1 is typically a few microseconds in length.

7.6.2 Experiment Set Up

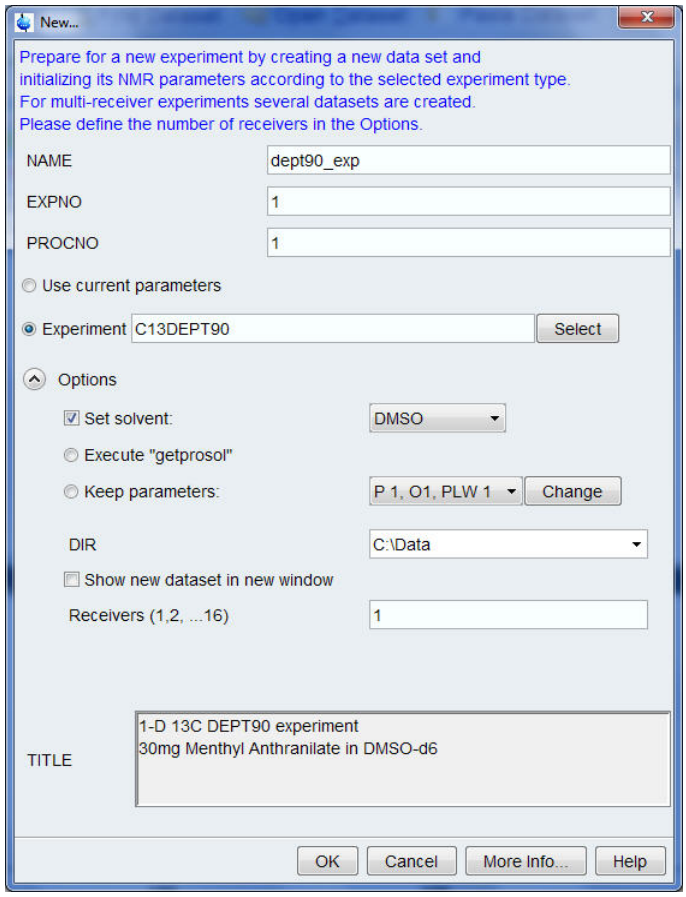
The DEPT90 experiment usually follows a regular 1H decoupled 13C experiment and a DEPT-135 experiment. It is used to assign the methine (CH) signals.

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



2. Select  by clicking on it.

3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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 = **64**

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

8. Select Prosol by clicking on it.

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

7.6.3 Acquisition

1. Select 

by clicking on it.

2. Select 

by clicking on it.

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

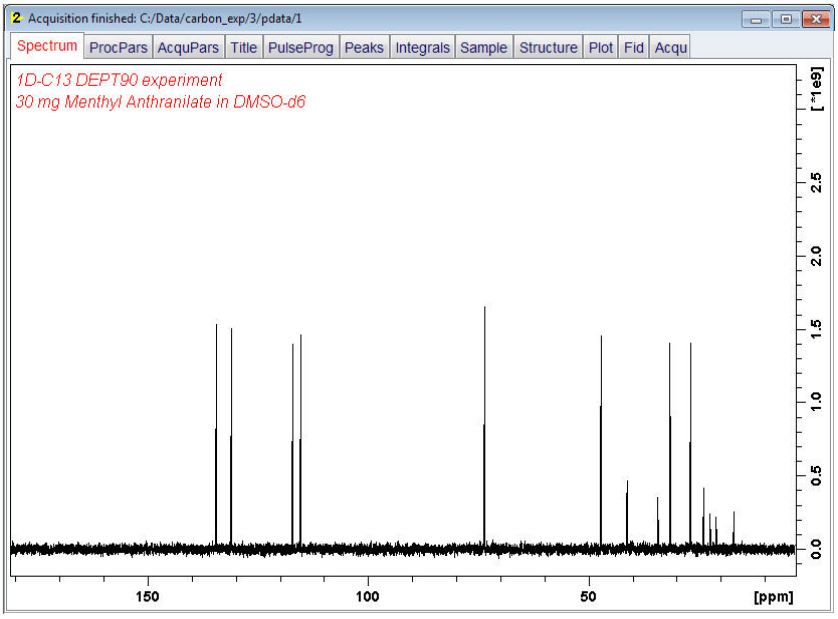
7.6.4 Processing

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

2. Select 

by clicking on it.

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.

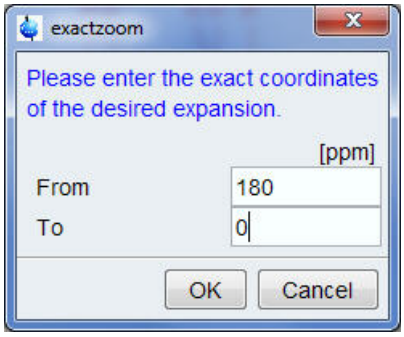


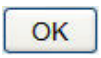
7.6.5 Plotting

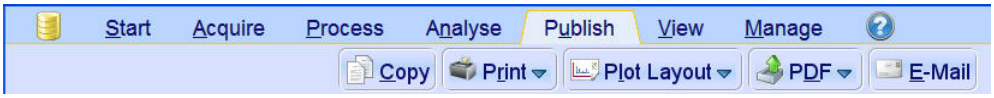
1. Expand the spectrum (all peaks in display).
2. Click on  .
3. Type the following F1 [ppm] values:

   From = **180**

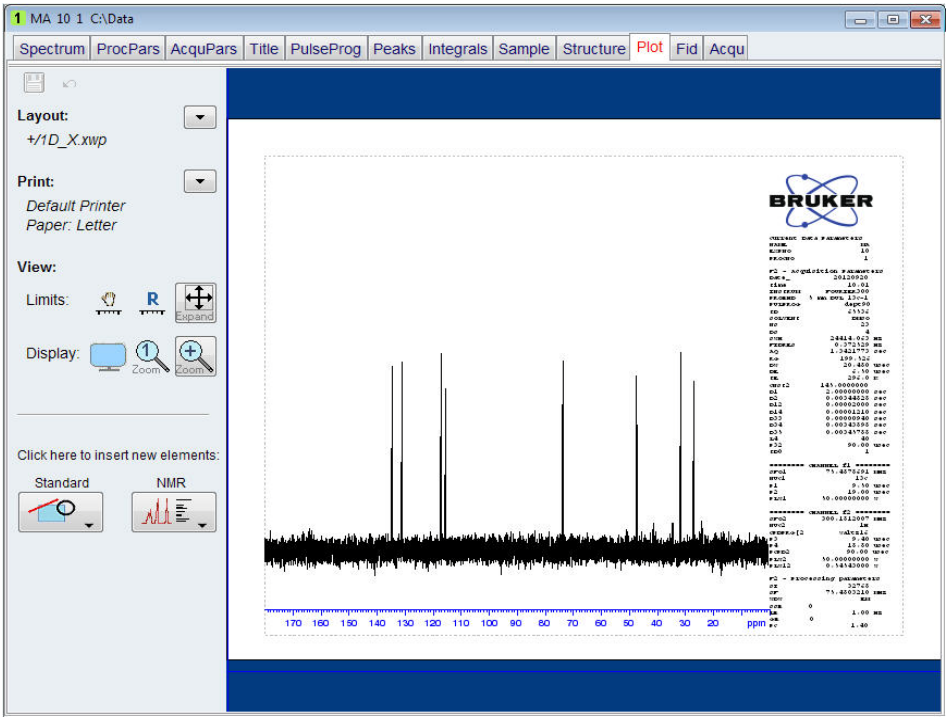
   To = **0**



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

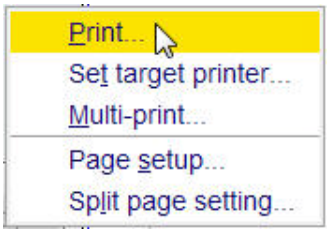


6. Click on  .



If desired, any changes can be administered by using the tools on the left side of the display.

7. Click on  in the Print section on the left side of the display.



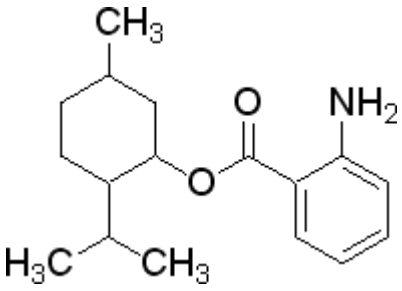
8. Select '**Print**' by clicking in it.

7.7 Observations

8 2-D Heteronuclear Experiments

8.1 Sample

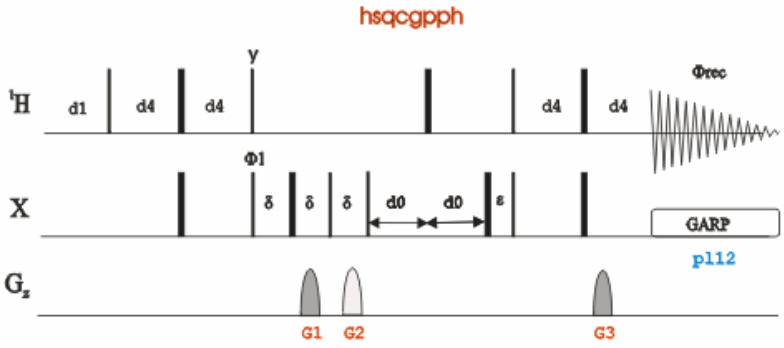
A sample of **30mg Menthyl Anthranilate in DMSO-d6** is used for all experiments in this chapter.



8.2 2-D HSQC Experiment

8.2.1 Introduction

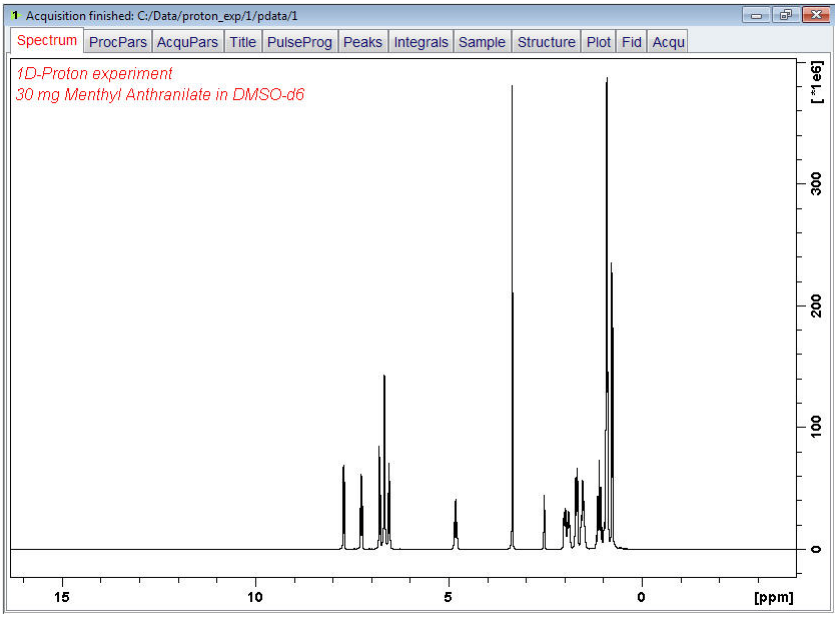
The **ge-2D HSQC experiment** is the gradient-enhanced version of the conventional HSQC experiment in which coherence selection is achieved by means of PFG. Thus, clean 2D HSQC spectra can be recorded in a single scan per t_1 increment without need for phase cycle when sample concentration is high. Other 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}H-X pairs via the large $^1J_{HX}$ coupling constant.The sequence is shown in the figure below.



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.

8.2.2 Preparation Experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 5, 1-D Proton experiment, Paragraph 5.2.2 Experiment setup** through **5.2.4 Processing**.

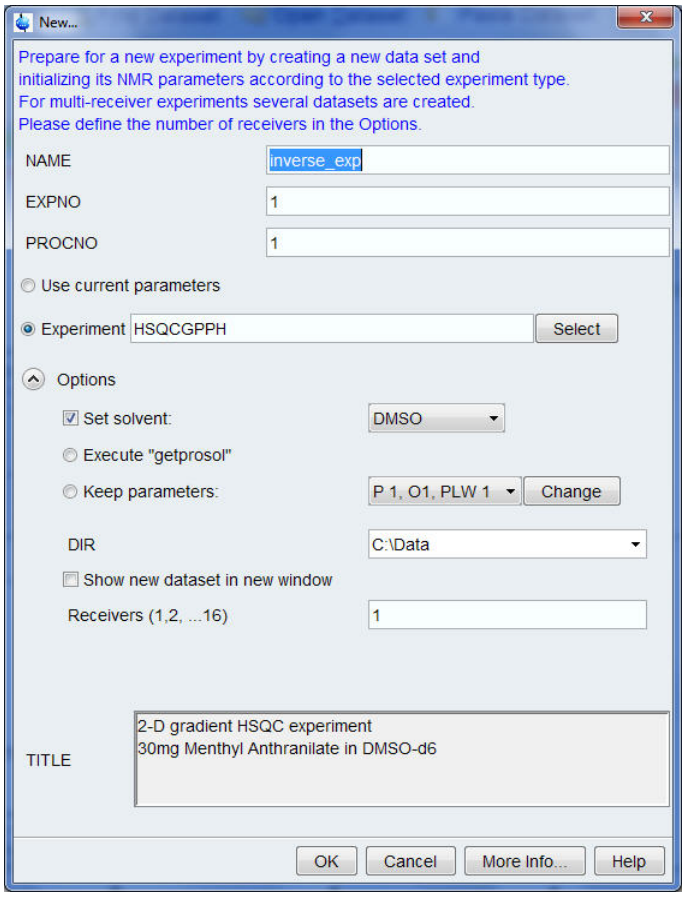


8.2.3 Setting Up The 2-D HSQC Experiment

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

2. Select  by clicking on it.

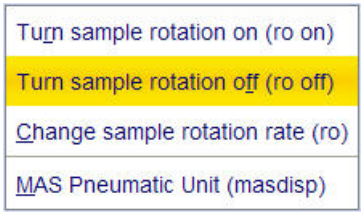
3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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.

6. Select Spin by clicking on it.



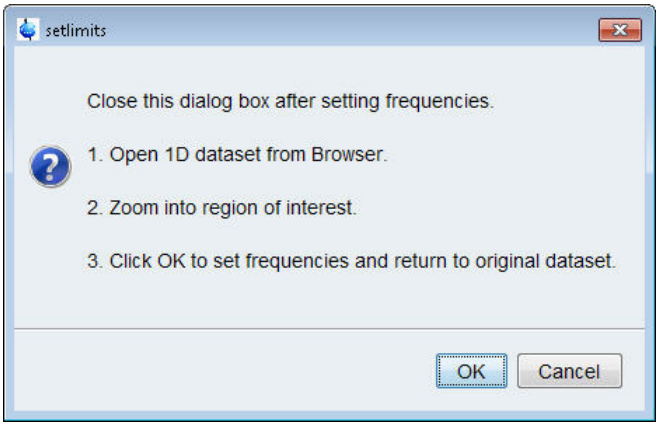
7. Select '**ro off**' by clicking on it.

2-D experiments should always be run without rotation.

8. Select Prosol by clicking on it.

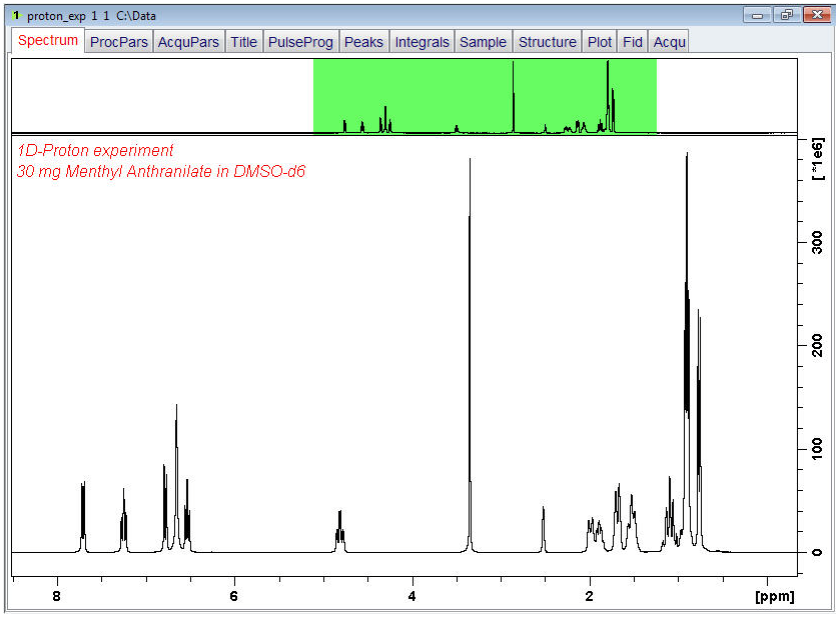
This will load the pulse width and power levels in to the parameter set.

9. Select SetLimits by clicking on it.

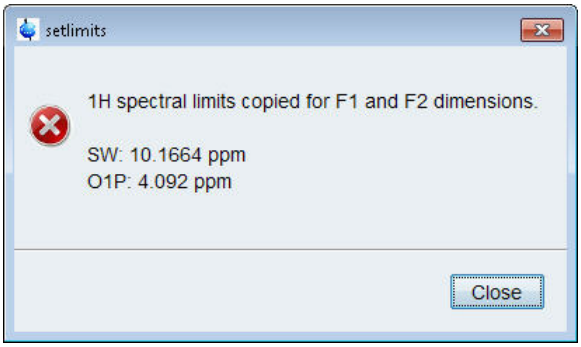


10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. **Inverse_exp 1**) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window.

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



12. Click on OK to assign the new limit.

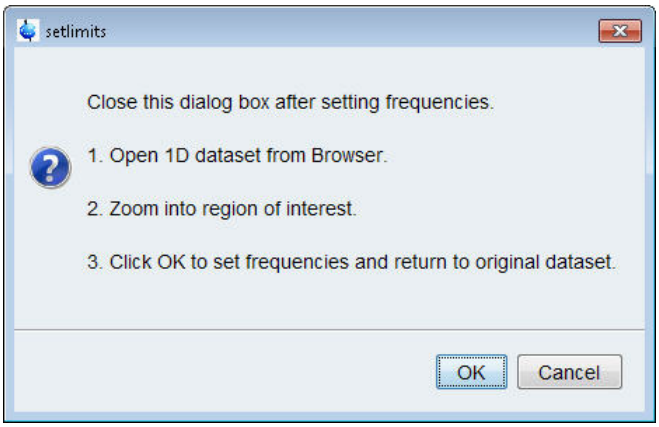


13. Click on Close.

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.

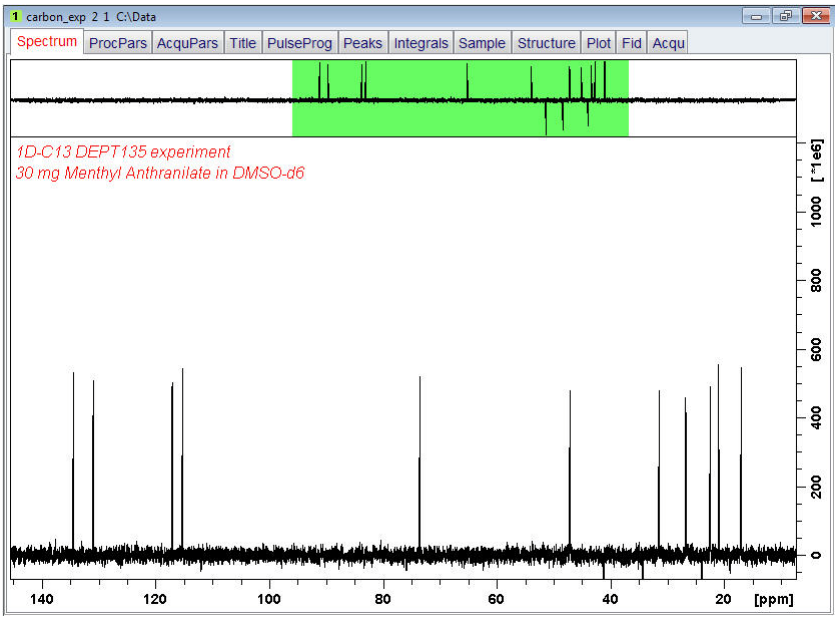
---

14. Select SetLimits by clicking on it.

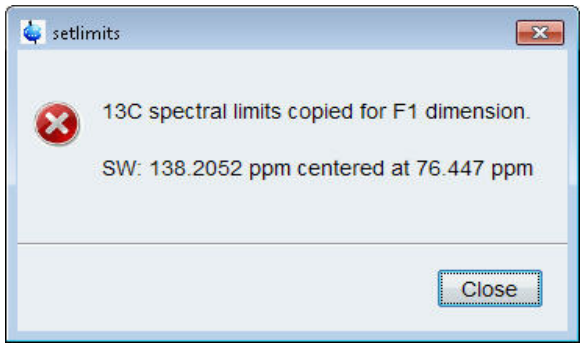


15. To open the 1D C13DEPT spectrum, right click on the dataset name in the browser window (e.g. **Carbon_exp 2**) and select 'Display' or click and hold the left mouse button for dragging the 1D C13DEPT dataset in to the spectrum window.

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



17. Click on OK to assign the new limit.



18. Click on Close.

8.2.4 Acquisition

1. Select Gain by clicking on it.

2. Select Go by clicking on it.

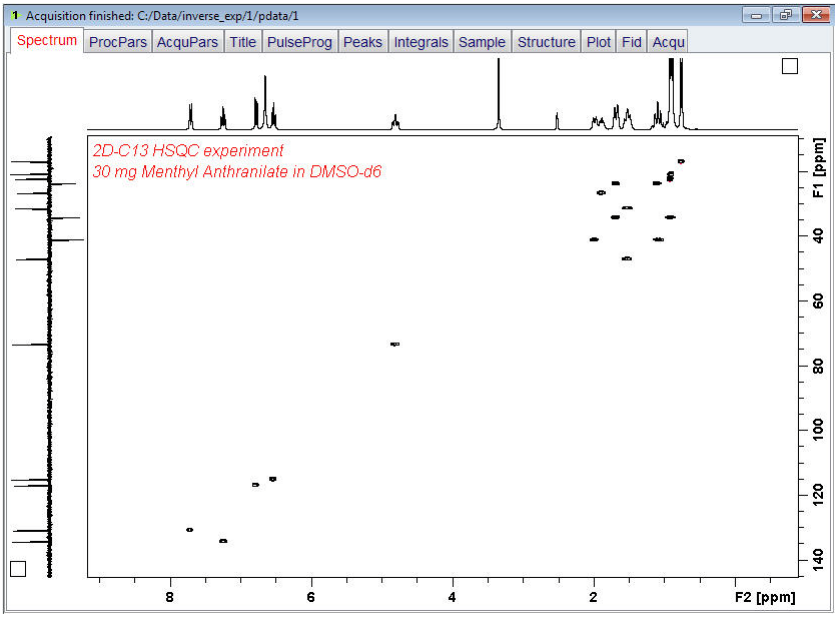
The experiment will take ~15 Minutes, using the default number of 2 scans and 256 increments.

8.2.5 Processing

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

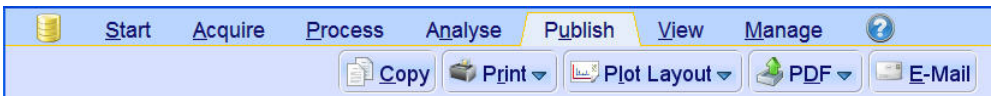
2. Select Proc. Spectrum by clicking on it.

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.



8.2.6 Plotting

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



2. Click on  to print the screen view.

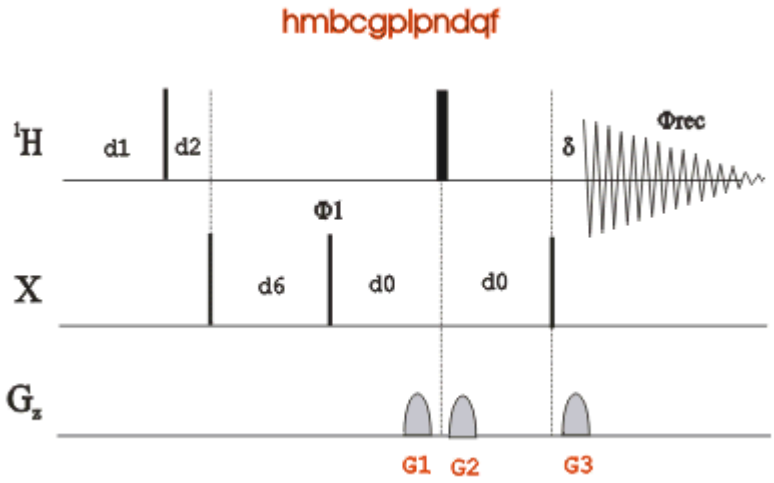
The F1 projection by default does not appear if the plot editor (XWINPLOT) is used to plot the spectrum. To add the F1 projection one has to create a new template.

8.3 Observations

8.4 2-D HMBC Experiment

8.4.1 Introduction

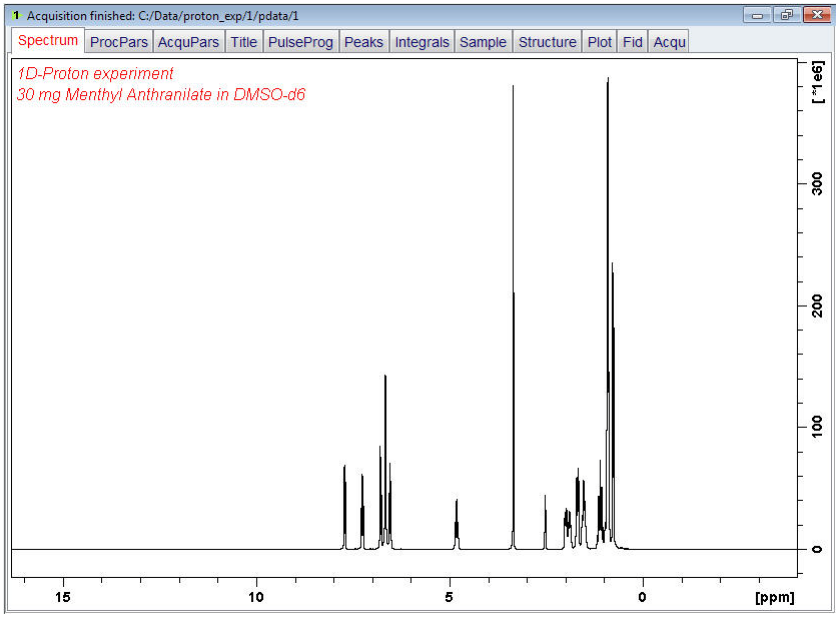
HMBC (Heteronuclear Multiple Bond Correlation) spectroscopy is a modified version of HMQC suitable for determining long-range 1H-13C connectivity. Since it is a long-range chemical shift correlation experiment the pulse program contains a low pass filter to suppress the one bond correlation and is a gradient-selected version which is not phase-sensitive. The experiment is performed without 13C 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 **hmbcgplpndqf** is shown in the figure below.



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.

8.4.2 Preparation Experiment

1. Run a **1D Proton** spectrum, following the instructions in **Chapter 5, 1-D Proton experiment, Paragraph 5.2.2 Experiment setup** through **5.2.4 Processing**.

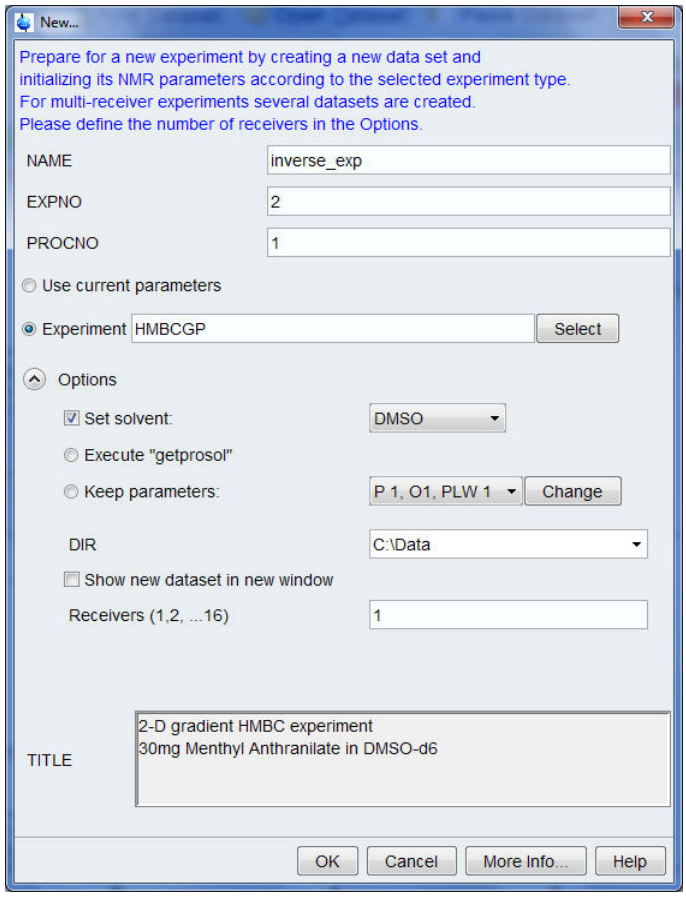


8.4.3 Setting Up The 2-D HMBC Experiment

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

2. Select  by clicking on it.

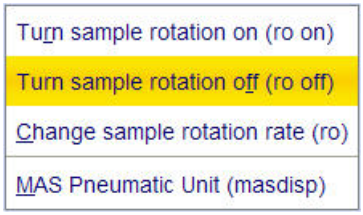
3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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.

6. Select Spin by clicking on it.



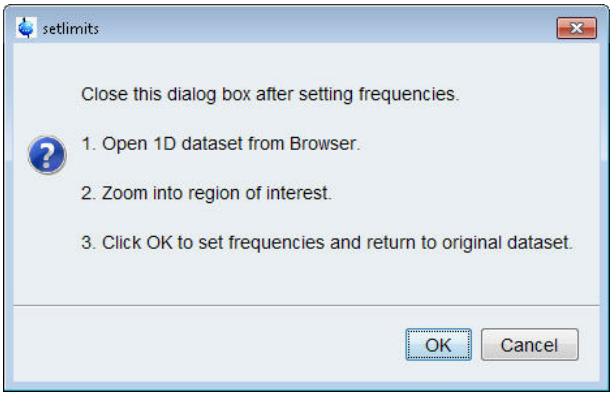
7. Select '**ro off**' by clicking on it.

2-D experiments should always be run without rotation.

8. Select  by clicking on it.

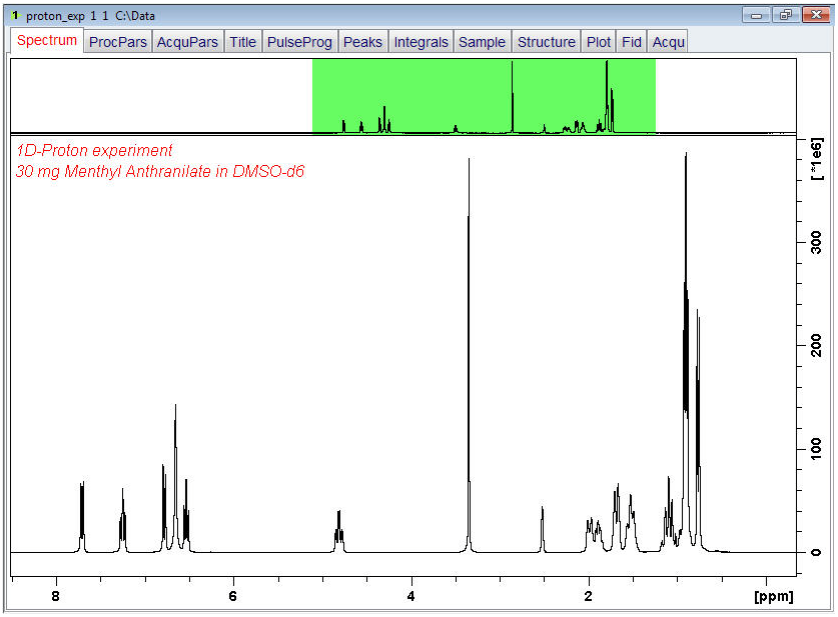
This will load the pulse width and power levels in to the parameter set.

9. Select  by clicking on it.

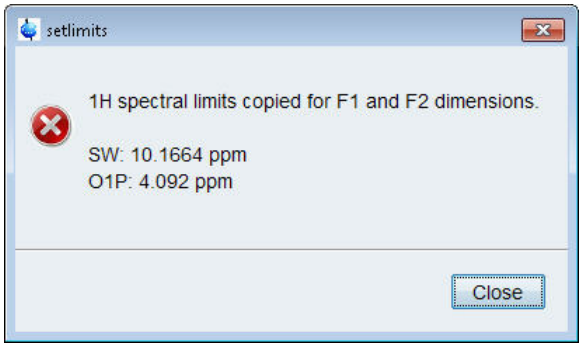


10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. **Inverse_exp 1**) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window.

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



12. Click on OK to assign the new limit.

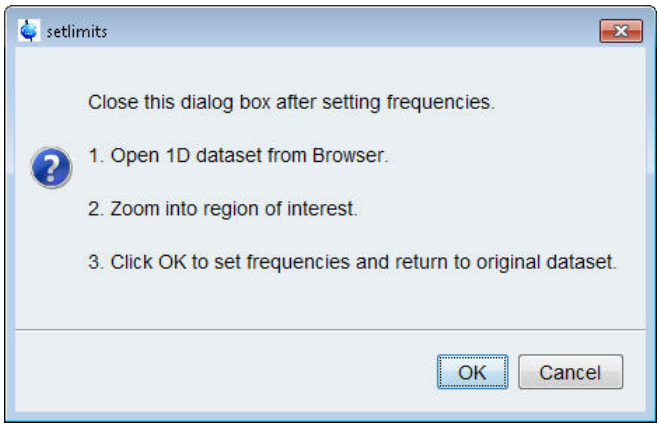


13. Click on Close.

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.

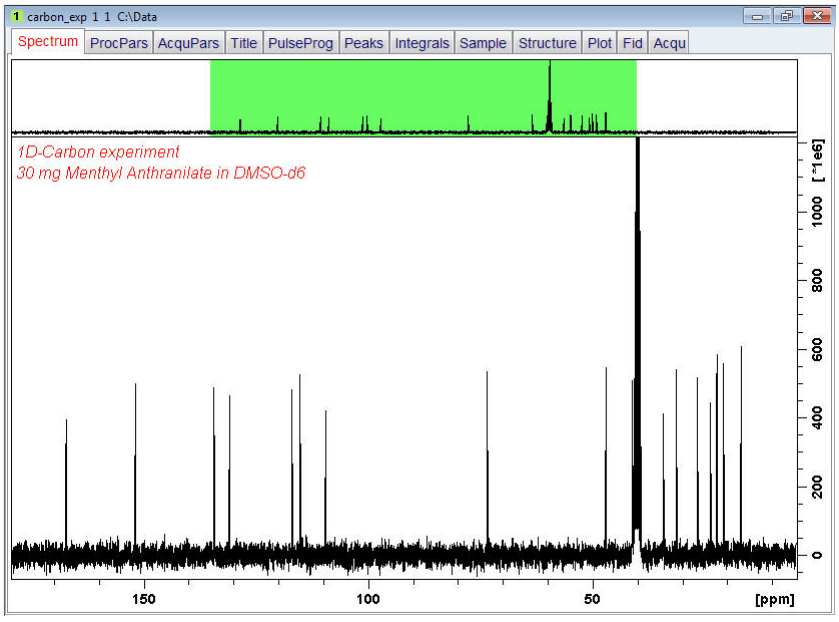
---

14. Select SetLimits by clicking on it.

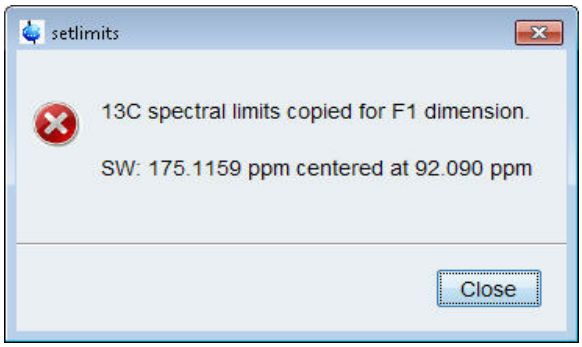


15. To open the 1D C13DEPT spectrum, right click on the dataset name in the browser window (e.g. **Carbon_exp 1**) and select 'Display' or click and hold the left mouse button for dragging the 1D C13DEPT dataset in to the spectrum window.

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



17. Click on OK to assign the new limit.



18. Click on Close

8.4.4 Acquisition

1. Select Gain by clicking on it.

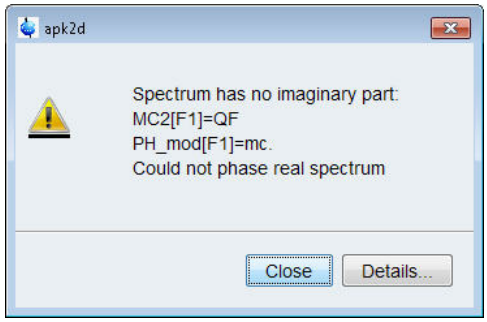
2. Select Go by clicking on it.

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

8.4.5 Processing

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

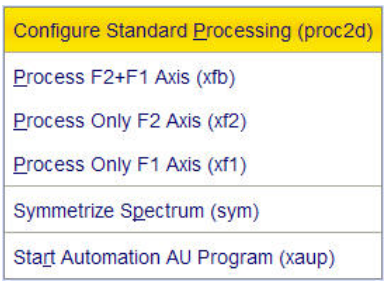
2. Select Proc. Spectrum by clicking on it.



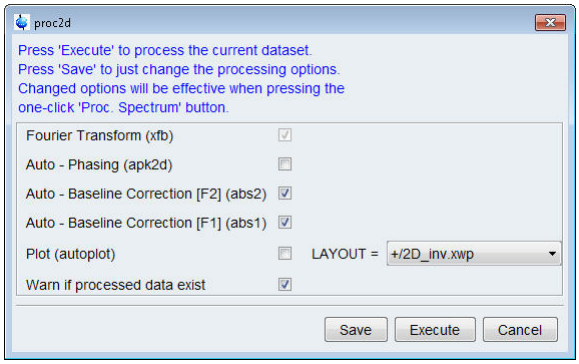
This executes a standard processing program **proc2**. The message shown in the figure above, pops up in case of a magnitude 2D experiment and the apk2d option is enabled. To configure the processing program follow the steps below.

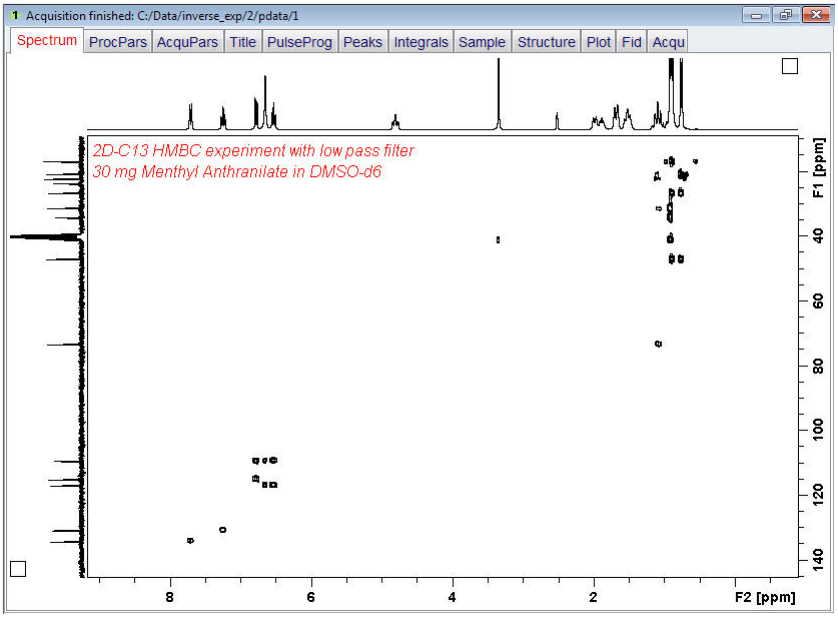
3. Click on the down arrow inside the 

button.



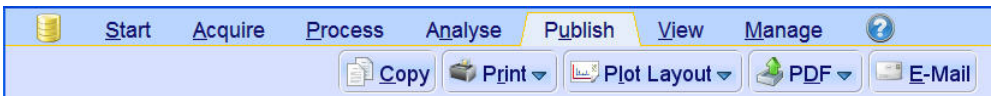
4. Select '**Configure Standard Processing**' by clicking on it.





8.4.6 Plotting

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



2. Click on  to print the screen view.

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.

8.5 Observations

9 Determination Of 90 Degree Pulses

9.1 Introduction

This chapter describes pulse calibration procedures for 1H and 13C. It is assumed that the user is already familiar with acquisition and processing of simple 1D NMR spectra. **Chapter 5, 1-D Proton experiment** and **Chapter 7, 1-D Carbon experiments**.

This chapter is intended as a guide for calibrating the 90^0 pulse of a probe or verifying the values observed by the engineer installing the instrument.

9.2 Proton 90 Degree Transmitter Pulse

Standard Test Sample:

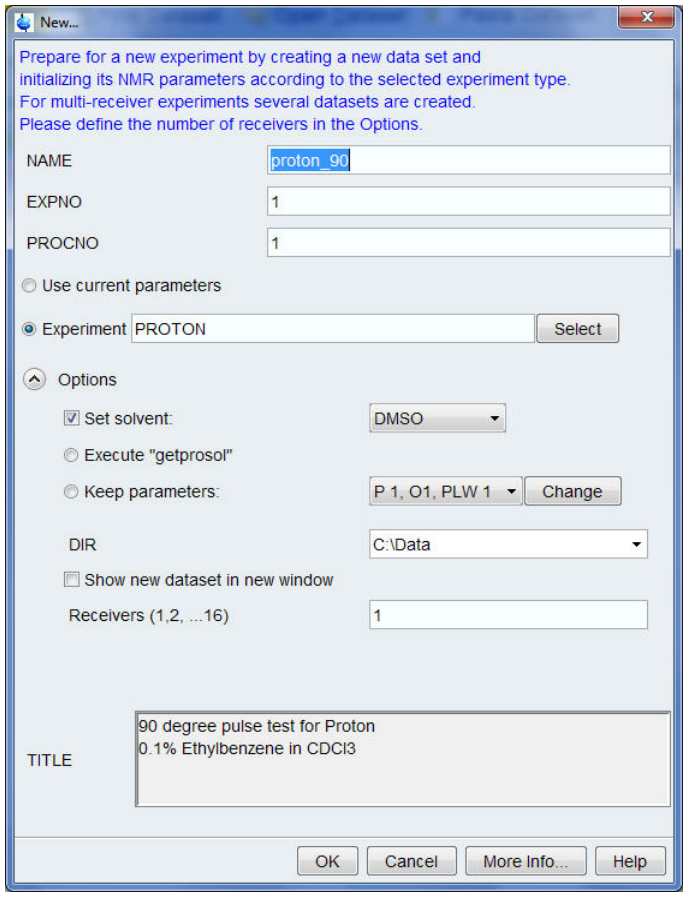
0.1% Ethylbenzene in CDCl3

9.2.1 Parameter Setup

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

2. Select 

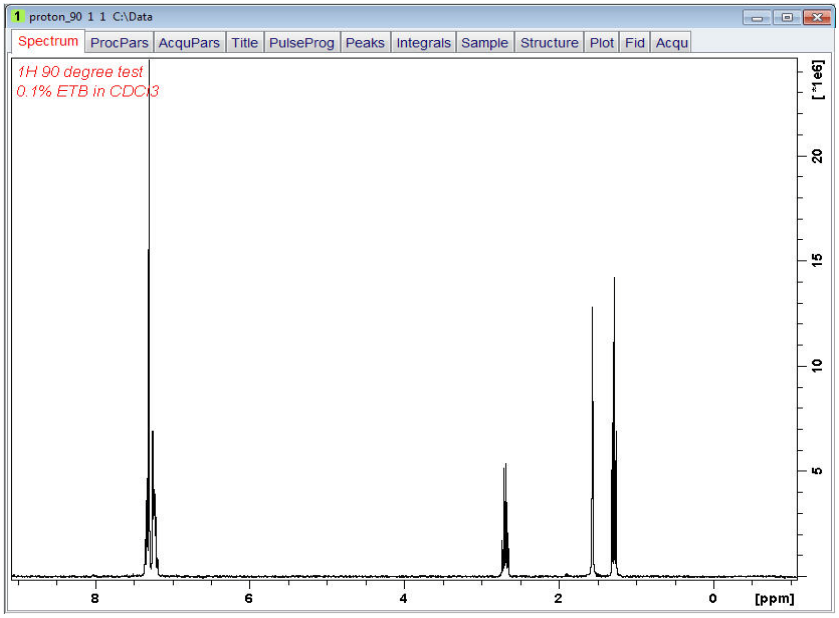
by clicking on it.
3. Enter the following information in to the 'New' window.



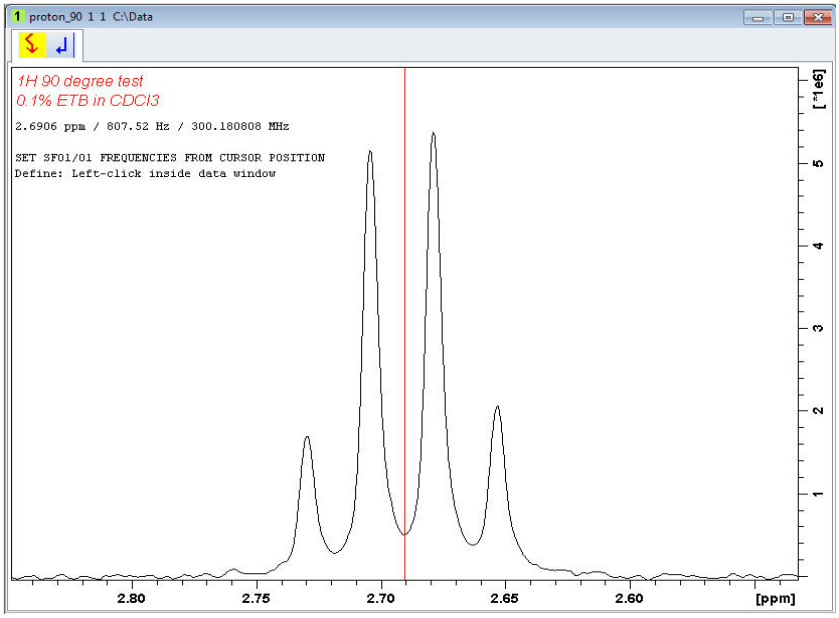
The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure above. Click on the down arrow button to browse for a specific directory.

4. Click on OK.

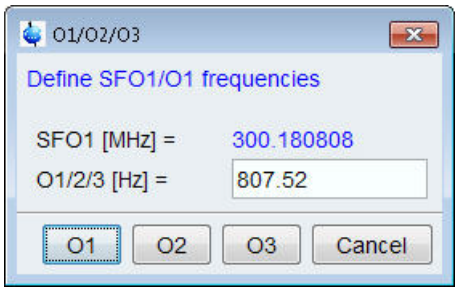
5. Run a **1D Proton** spectrum, following the instructions in **Chapter 5, 1-D Proton experiment, Paragraph 5.2.2 Experiment setup, step 5** through **5.2.4 Processing** using **CDCl3** as a lock solvent.



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



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



10. Click on 

.

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

12. Make the following changes:

    PULPROG = **zg**

    TD = **16384**

    SW [ppm] =**10**

    D1 [sec] = **30**

    DS = **0**

    NS = **1**

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

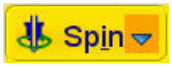
14 Make the following changes:

    SI = **8192**

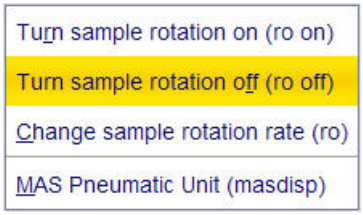
    LB [Hz] = **1**

    PH_mod = select **'pk**'

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

1
6. Select 

by clicking on it.



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

This test should be run without rotation.

9.2.2 Acquisition

1. Select 

by clicking on it

2. Select 

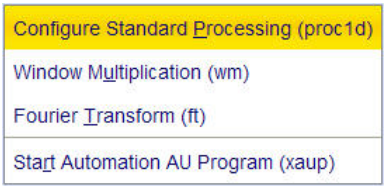
by clicking on it

9.2.3 Processing

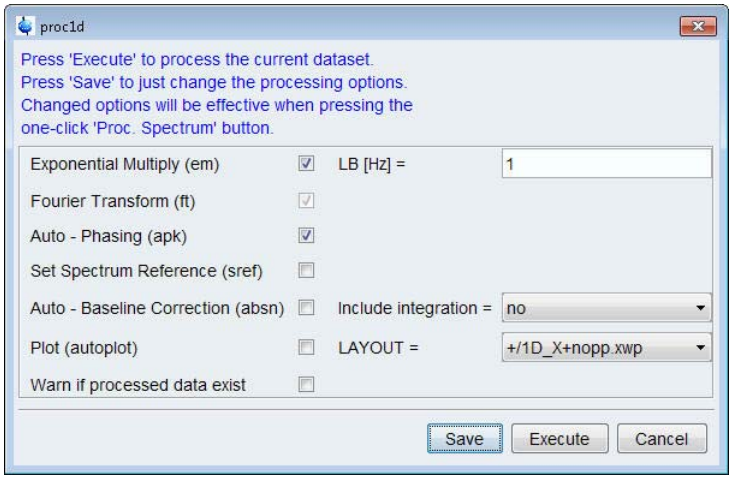
1. Click on the '**Process**' tab in the TopSpin Menu bar.

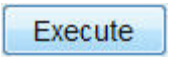
2. Click on the down arrow inside the 

button.

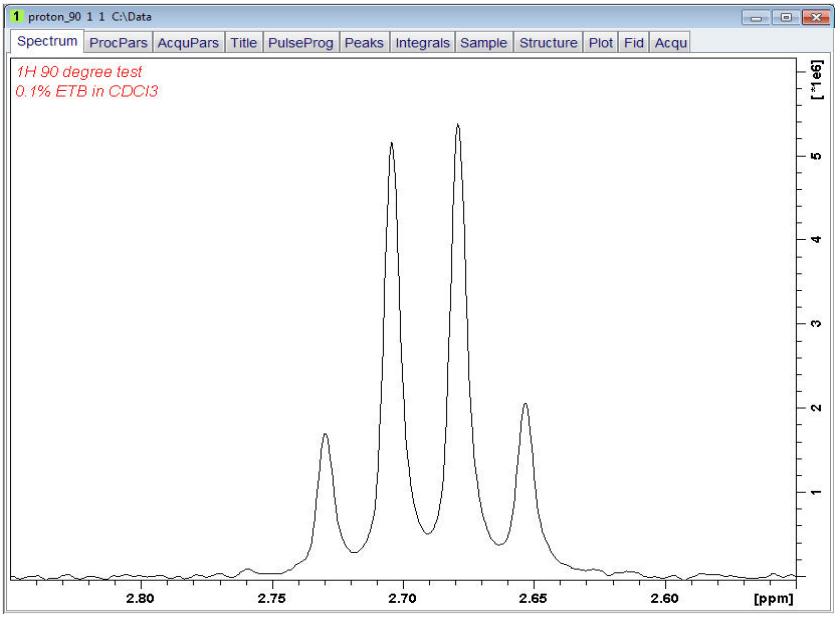


3. Select '**Configure Standard Processing**' by clicking on it.
4. Deselect the following options:
   '**Set Spectrum Reference (sref)**'
   '**Auto-Baseline correction (absn)**'
   '**Warn if Processed data exist**'



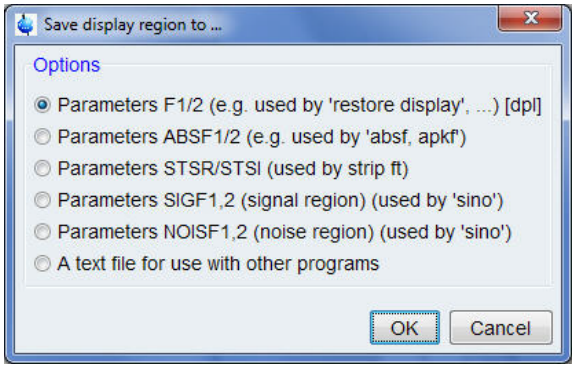
5. Click on 

.
6. Expand the spectrum form **2.85 ppm** to **2.55 ppm.**

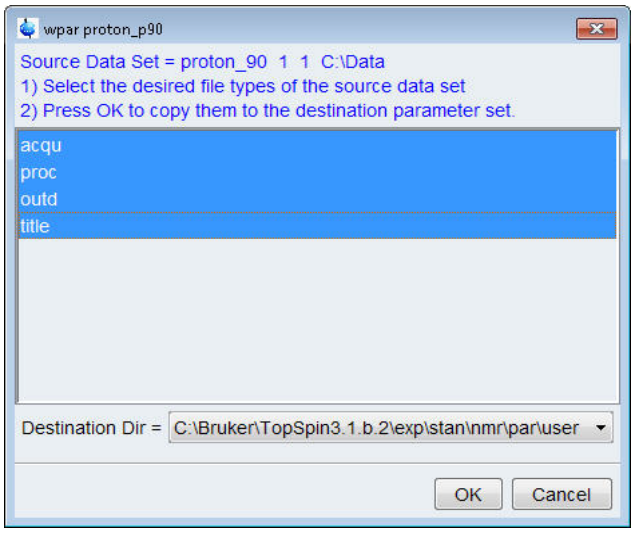


7. Click on the right mouse button inside the spectral window.

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



9. Enable '**Parameters F1/2**'.
10. Click on OK.
11. Type **wpar proton_p90** to store the parameter for future use.

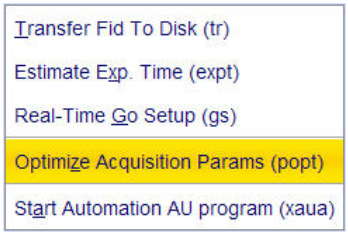


12. Select all parameter options.
13. Click on OK.

9.2.4 Determine The 90 Degree Pulse

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

2. Click on the down arrow inside the Go button.

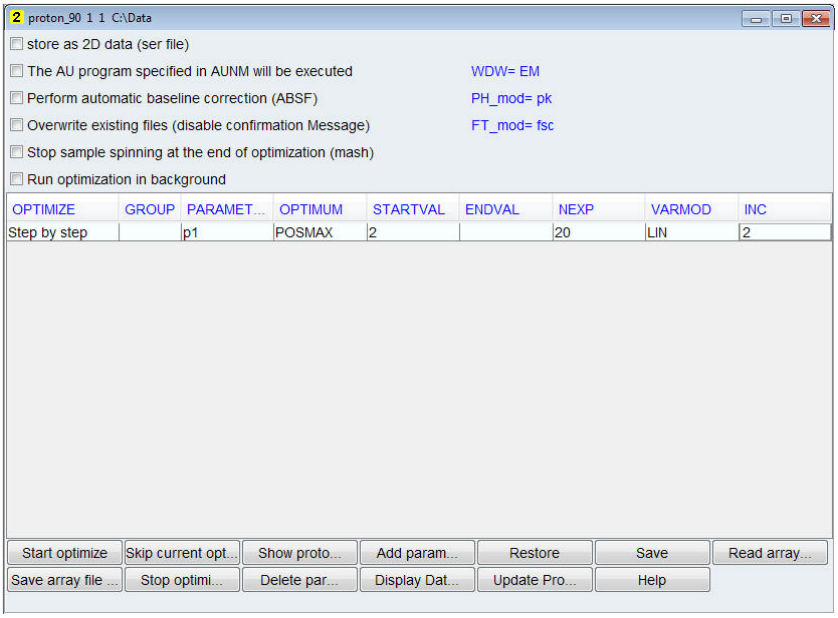


3. Select '**Optimize Acquisition Params (popt)**' by clicking on it.

4. Make the following changes:

   OPTIMIZE = **Step by step**
   PARAMETER = **p1**
   OPTIMUM = **POSMAX**
   STARTVAL = **2**
   NEXP = **20**

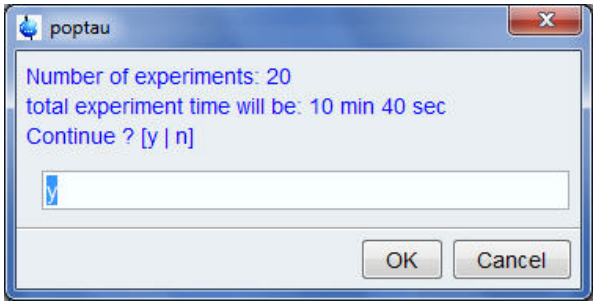
VARMOD = **LIN**
INC = **2**



5. Click on Save.

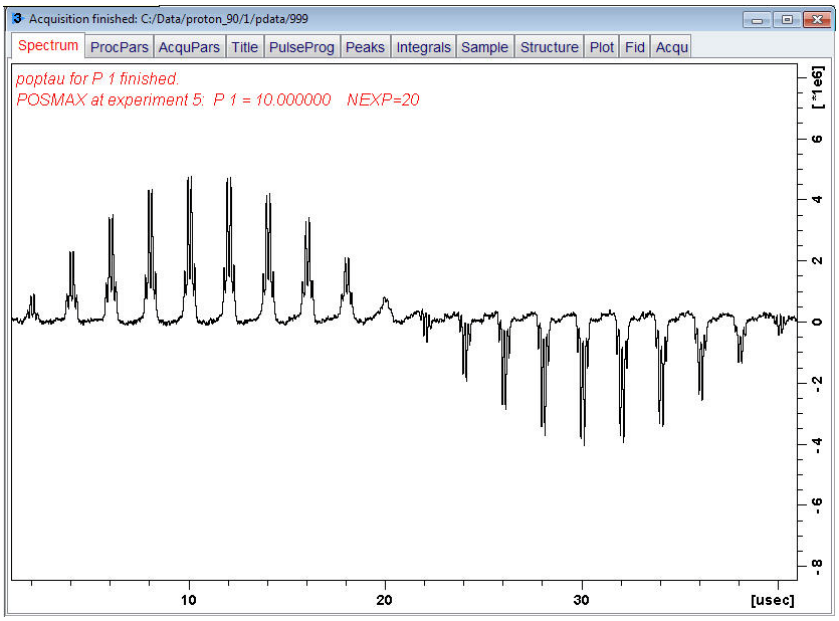
The ENDVAL parameter has been updated.

6. Click on Start optimize.



7. Enter **y** in to the poptau window.
8. Click on OK.

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 the figure below.



The POSMAX value of **p1** is displayed in the title window which is the 90^0 pulse, along with the experiment number and the NEXP value. Write this value down. To obtain a more accurate 90^0 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 360^0 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 360^0 pulse.

The quartet signal is negative for a pulse angle slightly less then 360^0 and positive when the pulse angle is slightly more then 360^0.

16. Simply divide the determine 360^0 pulse value by 4. This will be the exact 90^0 pulse length for the proton transmitter on the current probe.

9.3 Observations

9.4 Carbon 90 Degree Transmitter Pulse

Standard Test Sample:

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

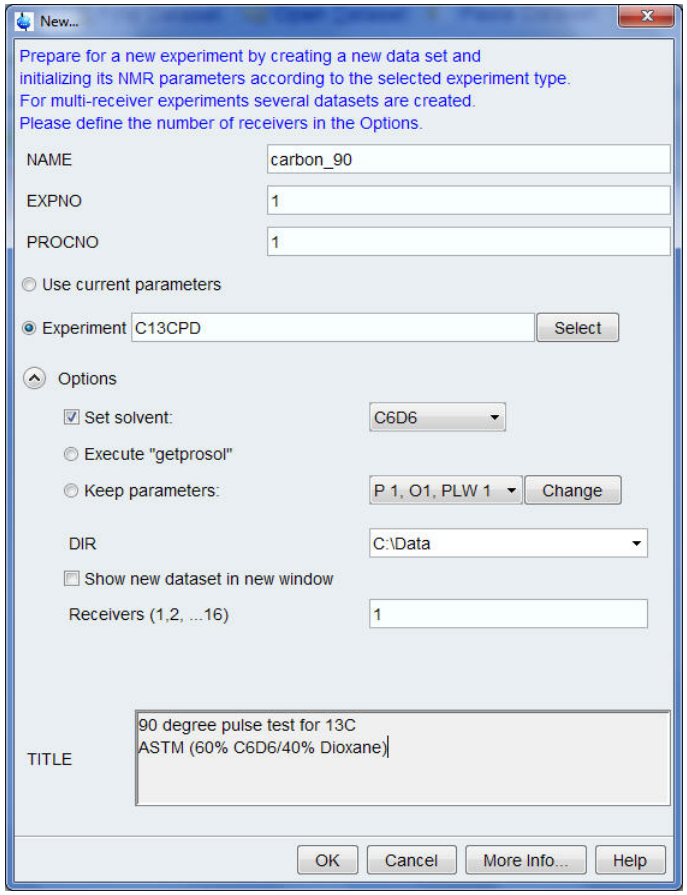
9.4.1 Parameter Setup

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

2. Select 

by clicking on it.

3. Enter the following information in to the 'New' window.



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

4. Click on OK.

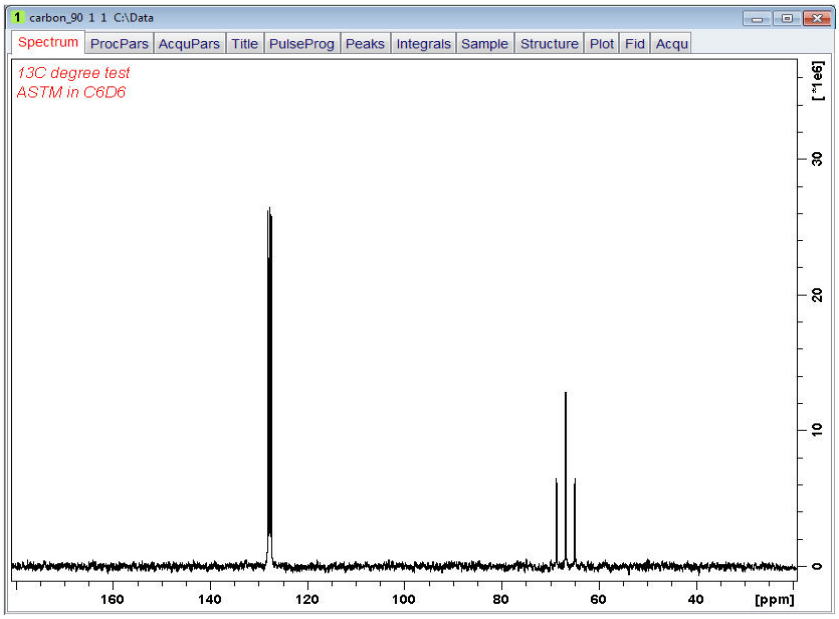
5. Run a **1D Carbon** spectrum, following the instructions in **Chapter 7**, **1-D Carbon experiments, Paragraph 7.2.2 Experiment setup, step 5** using **C6D6** as a lock solvent and making the following acquisition parameter changes:

PULPROG = **zg**

DS = **0**

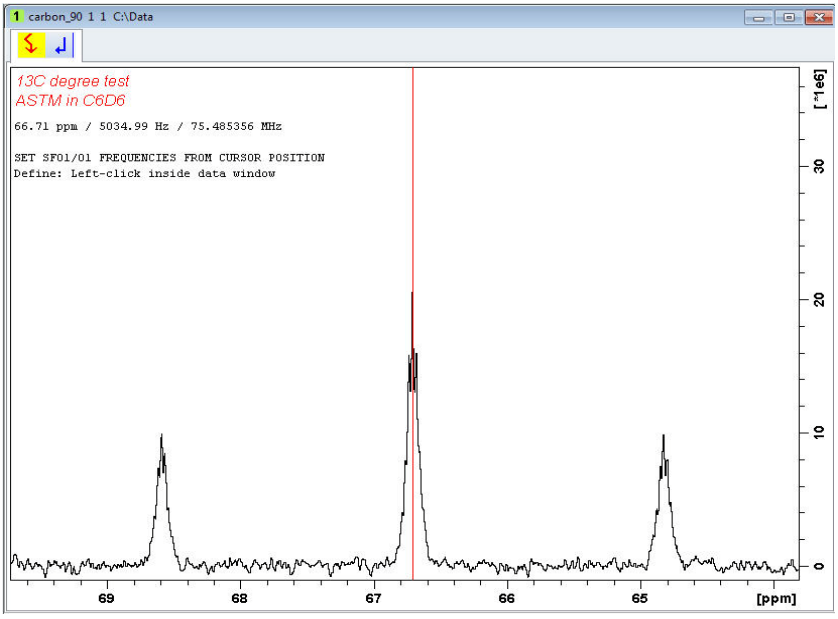
NS = **1**

6. Continue with **7.2.4 Processing**.

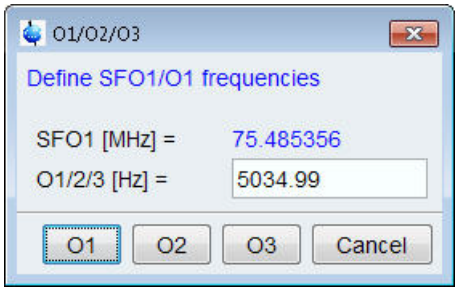


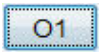
7. Expand peak at **67 ppm**.

8. Click on to set the RF from cursor.



9. Click the left mouse button to set the frequency.



10. Click on 

.

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

12. Make the following changes:

    TD = **8912**

    SW [Hz] =**40**

    D1 [sec] = **60**

    DS = **0**

    NS = **1**

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

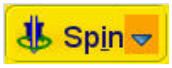
14 Make the following changes:

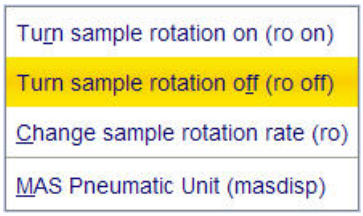
    SI = **4096**

    LB [Hz] = **3.5**

    PH_mod = select **'pk**'

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

16. Select  by clicking on it.



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

This test should be run without rotation.

9.4.2 Acquisition

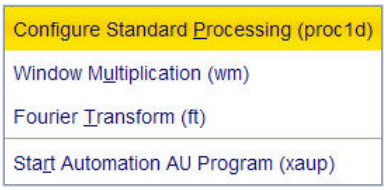
1. Select  by clicking on it.

2. Select  by clicking on it.

9.4.3 Processing

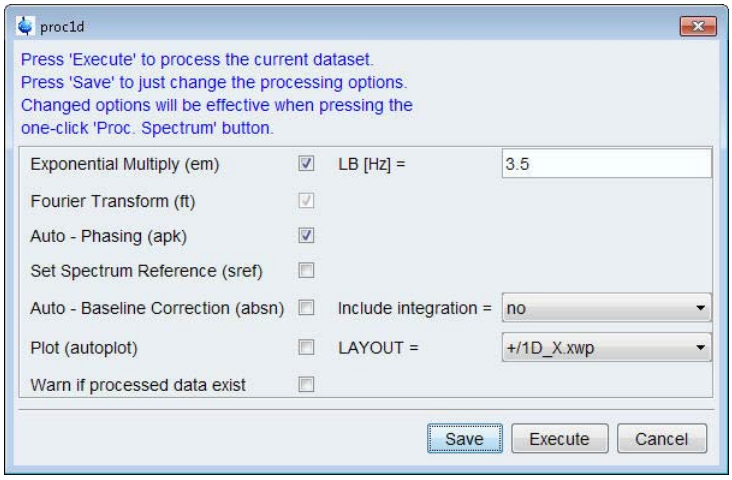
1. Click on the '**Process**' tab in the TopSpin Menu bar.

2. Click on the down arrow inside the  button.

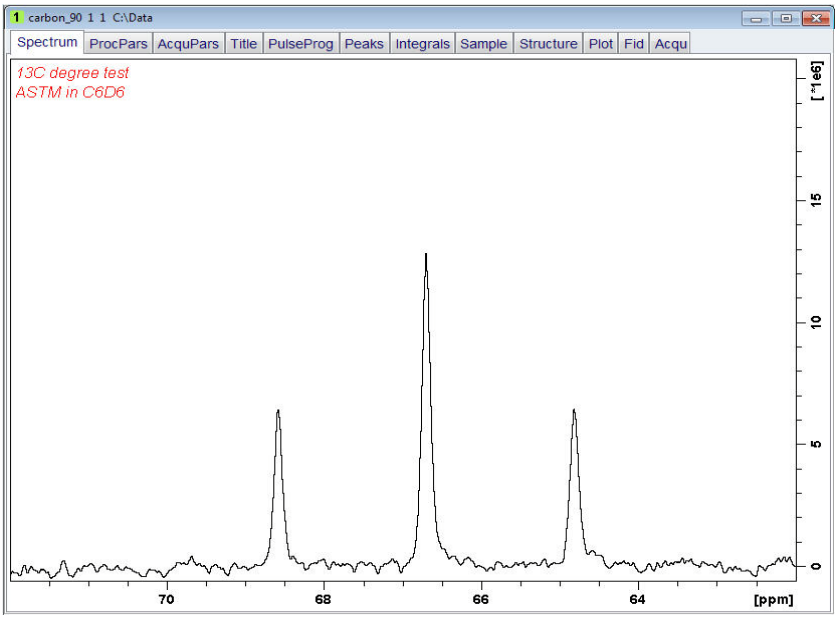


3. Select '**Configure Standard Processing**' by clicking on it.
4. Deselect the following options:
   '**Set Spectrum Reference (sref)**'
   '**Auto-Baseline correction (abs)**'

'**Warn if Processed data exist**'

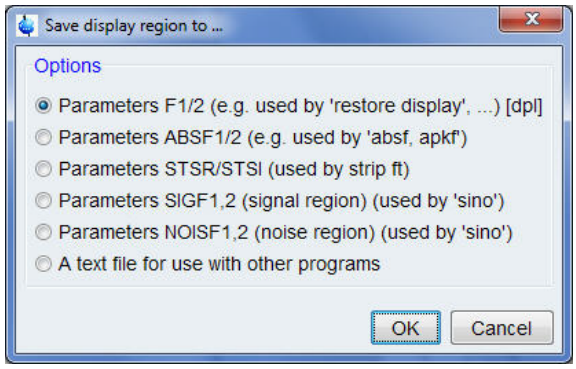


5. Click on Execute.
6. Expand the spectrum from **72 ppm** to **62 ppm**.

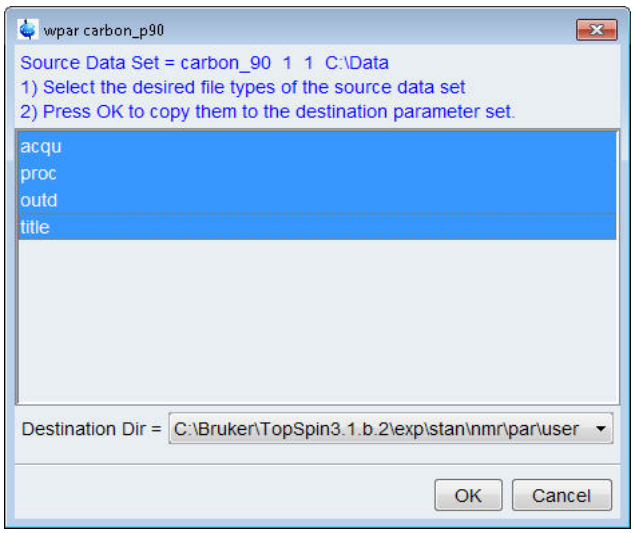


7. Click on the right mouse button inside the spectral window.

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



9. Enable '**Parameters F1/2**'.
10. Click on OK.
11. Type **wpar carbon_p90** to store the parameter for future use.

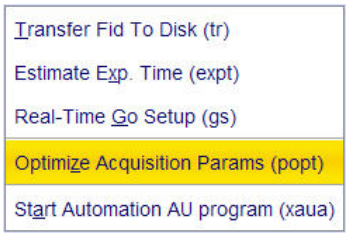


12. Select all parameter options.
13. Click on OK.

9.4.4 Determine The 90 Degree Pulse

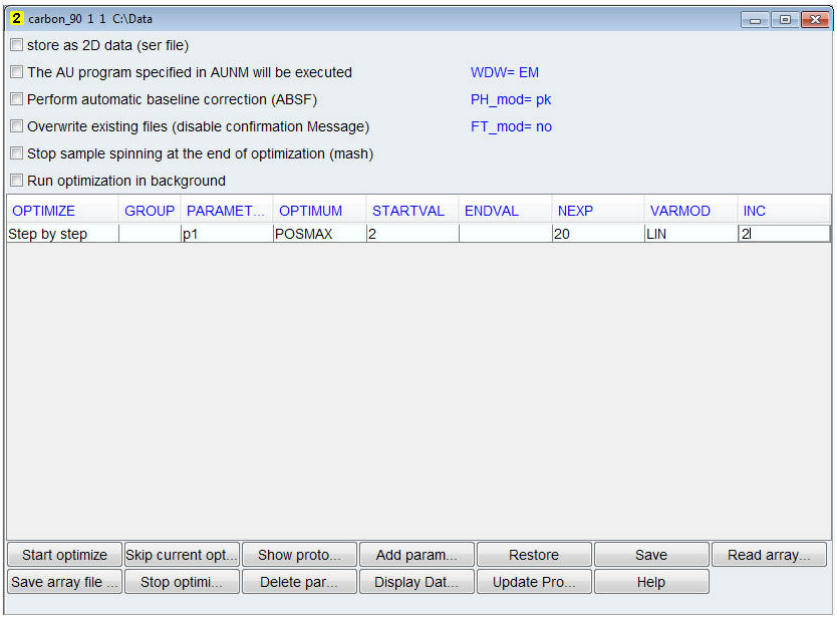
1. Click on the '**Acquire**' tab in the TopSpin menu bar.

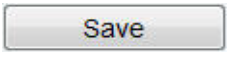
2. Click on the down arrow inside the  button.



3. Select '**Optimize Acquisition Params (popt)**' by clicking on it.
4. Make the following changes:

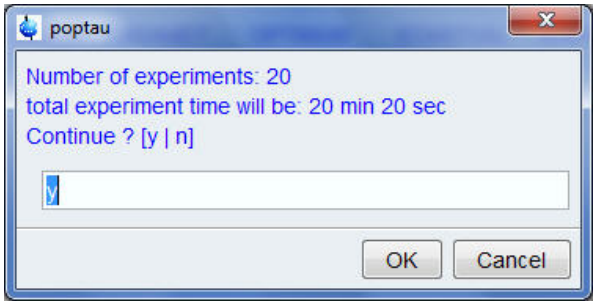
   OPTIMIZE = **Step by step**
   PARAMETER = **p1**
   OPTIMUM = **POSMAX**
   STARTVAL = **2**
   NEXP = **20**
   VARMOD = **LIN**
   INC = **2**



5. Click on  .

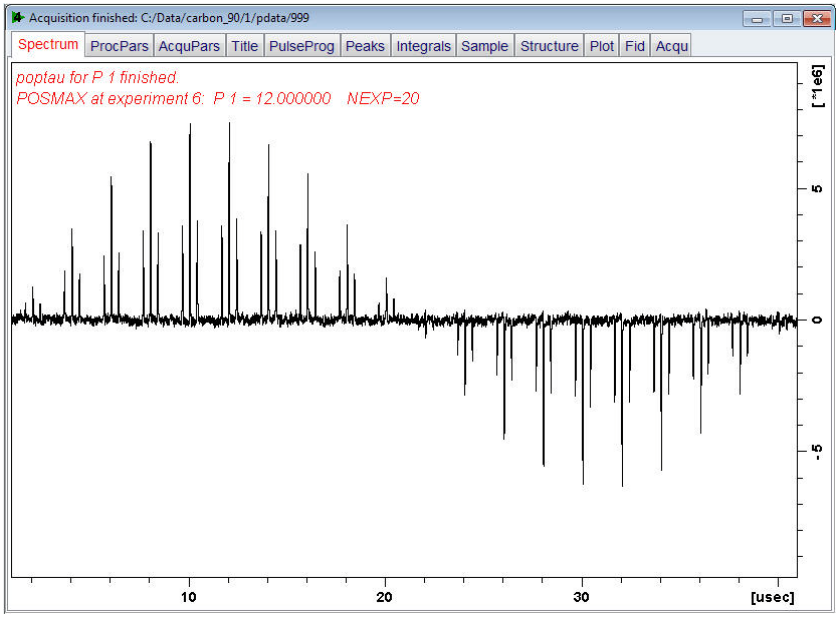
The ENDVAL parameter has been updated.

6. Click on Start optimize.



7. Enter **y** in to the poptau window.
8. Click on OK.

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 carbon_90/1/999 as shown in the figure below.



The POSMAX value of p1 is displayed in the title which is the 90^0 pulse, along with the experiment number and the NEXP value. Write this value down. To obtain a more accurate 90^0 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 360^0 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 360^0 pulse.

The signal is negative for a pulse angle slightly less then 360^0 and positive when the pulse angle is slightly more then 360^0.

16. Simply divide the determine 360^0 pulse value by 4. This will be the exact 90^0 pulse length for the proton transmitter on the current probe.

9.5 Observations

Determination Of 90 Degree Pulses

10 Sensitivity Tests

10.1 Introduction

This chapter describes the sensitivity test procedures for 1H and 13C. It is assumed that the user is already familiar with acquisition and processing of simple 1D NMR spectra. **Chapter 5, 1-D Proton Experiment** and **Chapter 7, 1-D Carbon Experiments**. Also the 90^0 pulses have to be properly calibrated, **Chapter 9, Determination Of The 90 degree Pulses**.

This chapter is intended as a guide for running the 1H and 13C Signal to Noise tests on a probe or verifying the values observed by the engineer installing the instrument.

10.2 Proton Sensitivity Test

Standard Test Sample:

0.1% Ethylbenzene in CDCl3

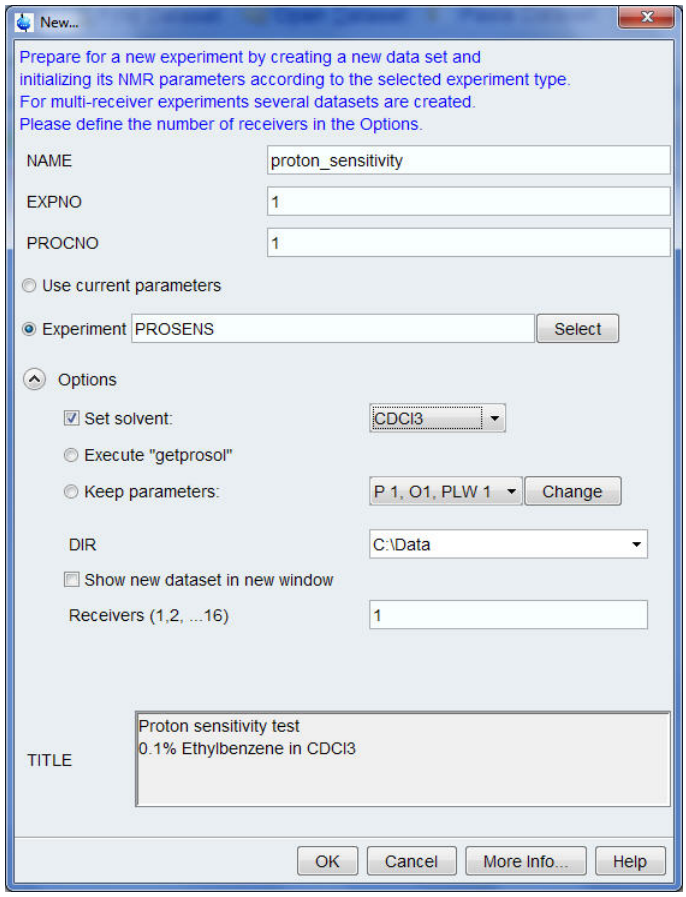
10.2.1 Experiment Setup

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

2. Select 

by clicking on it.

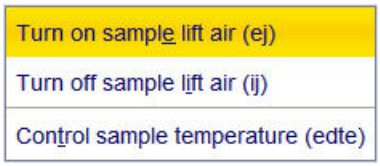
3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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.

6. Select Sample by clicking on it.



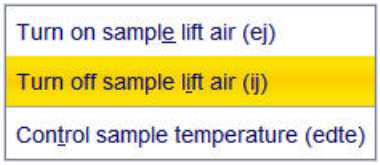
7. Select '**ej**' by clicking on it.

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 too the top of the magnet.

9. Select 

by clicking on it.

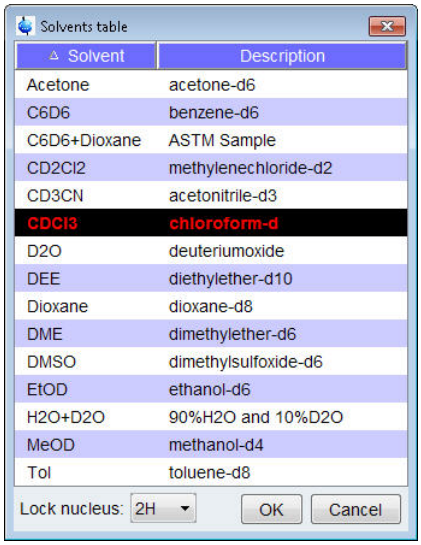


10. Select '**ij**' by clicking on it.

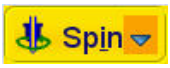
Wait till the sample is lowered down in to the probe and the lift air is turned off. A clicking sound may be heard.

11. Select 

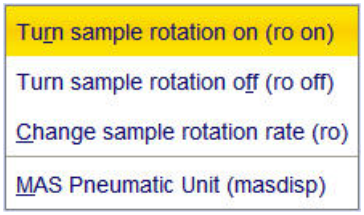
by clicking on it.



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

13. Select 

by clicking on it.



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

15. Select 

by clicking on it.

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

16. Select 

by clicking on it.

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

10.2.2 Acquisition

1. Select 

by clicking on it.

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.

10.2.3 Processing

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

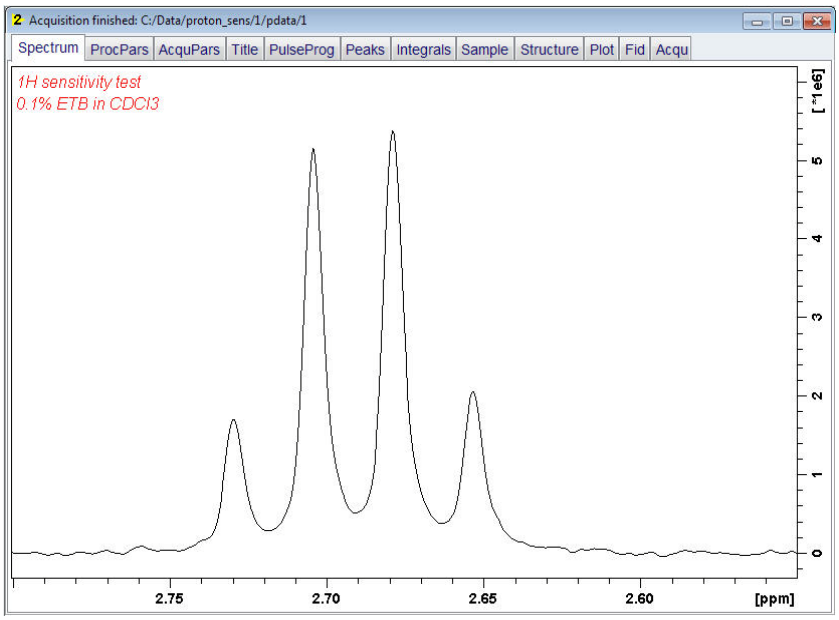
2. Click on 

.

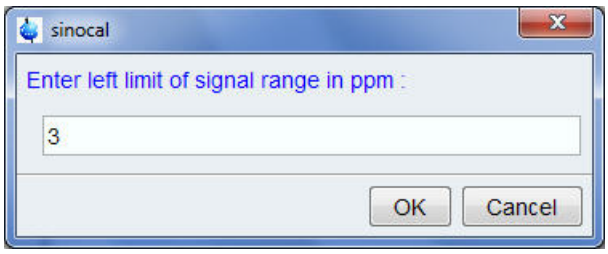
This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**'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

10.2.4 Calculating The Signal To Noise Ratio

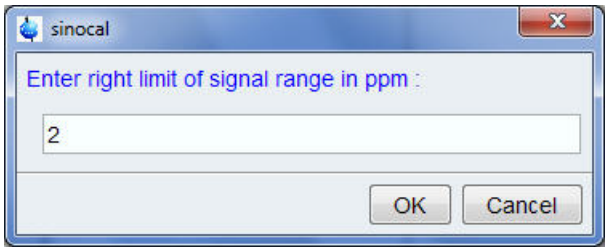
The signal to noise ratio is determined on the intensity of the quartet lines between 2 ppm and 3 ppm. It is calculated by AU-program **sinocal** over a range of 2 ppm between 2.8 ppm and 7 ppm. The s/n ratio is strongly dependant on good resolution and lineshape. The splitting between the two central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



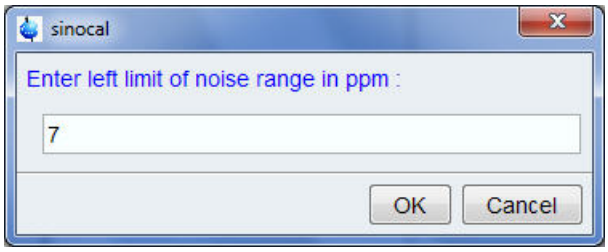
1. Type **sinocal** on the command line.



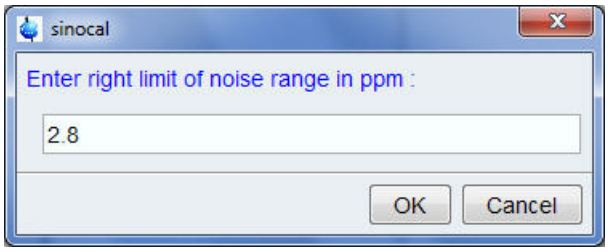
2. Enter **3** for the left limit of the signal range.
3. Click on OK.



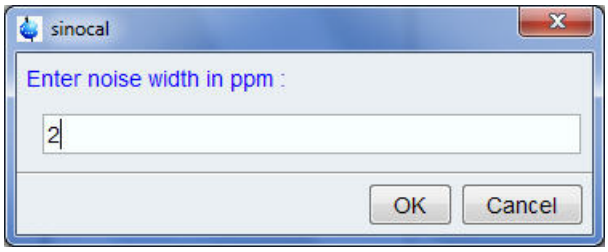
4. Enter **2** for the right limit of the signal range.
5. Click on OK.



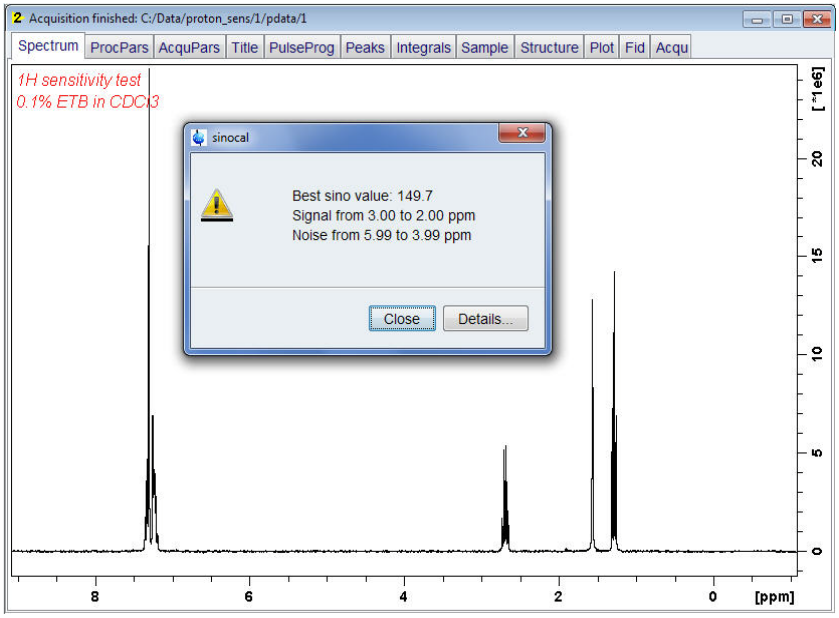
6. Enter **7** for the left limit of the noise range.
7. Click on OK.



8. Enter **2.8** for the right limit of the noise range.
9. Click on OK.



10. Enter **2** for the noise width.
11. Click on OK.


2 Acquisition finished: C:/Data/proton_sens/1/pdata/1
Spectrum
ProcPars
AcquPars
Title
PulseProg
Peaks
Integrals
Sample
Structure
Plot
Fid
Acqu
1H sensitivity test
0.1% ETB in CDCl3
sinocal
Best sino value: 149.7
Signal from 3.00 to 2.00 ppm
Noise from 5.99 to 3.99 ppm
Close
Details...
[*1e6]
20
15
10
5
0
8
6
4
2
0
[ppm]

10.3 Observations

10.4 Carbon Sensitivity Test Without Proton Decoupling

Standard Test Sample:

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

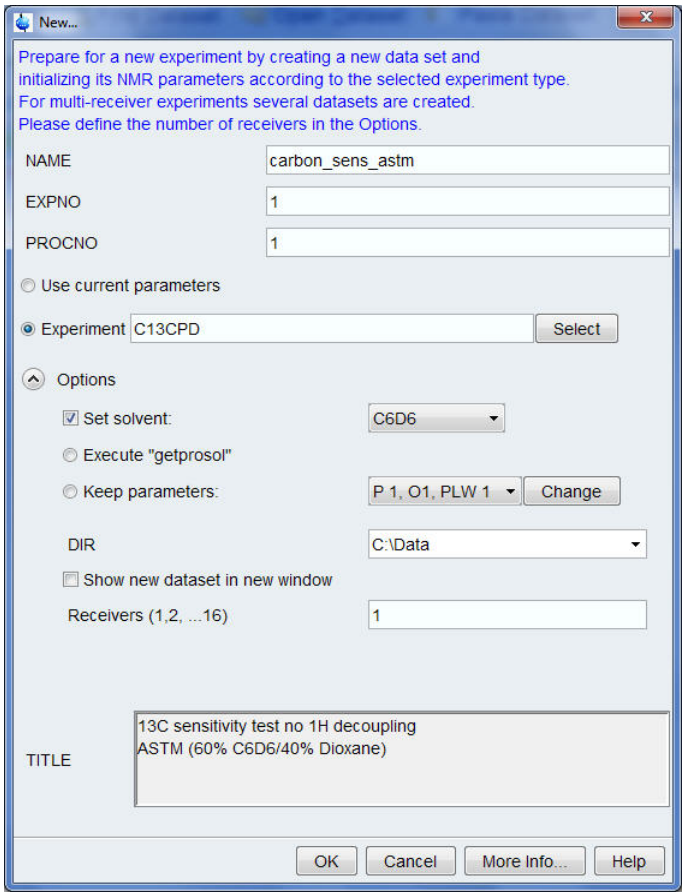
10.4.1 Experiment Setup

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

2. Select 

by clicking on it.

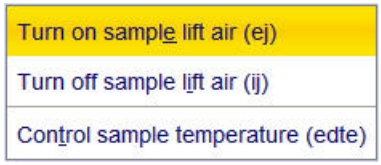
3. Enter the following information in to the 'New' window.



The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure 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.

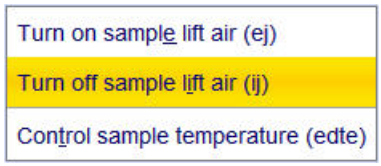
6. Select Sample by clicking on it.



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

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

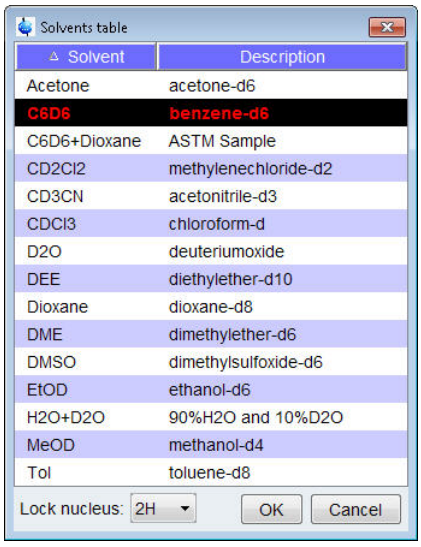
8. Place the sample on too the top of the magnet.
9. Select Sample by clicking on it.

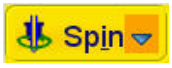


10. Select '**ij**' by clicking on it.

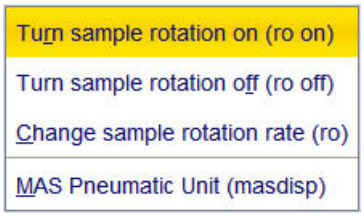
Wait till the sample is lowered down in to the probe and the lift air is turned off. A clicking sound may be heard.

11. Select Lock by clicking on it.



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

by clicking on it.



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

by clicking on it.

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

16. Select 

by clicking on it.

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 = **65536**

    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 = **32768**

    LB [Hz] = **3.5**

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

10.4.2 Acquisition

1. Select 

by clicking on it.

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

2. Select 

by clicking on it.

10.4.3 Processing

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

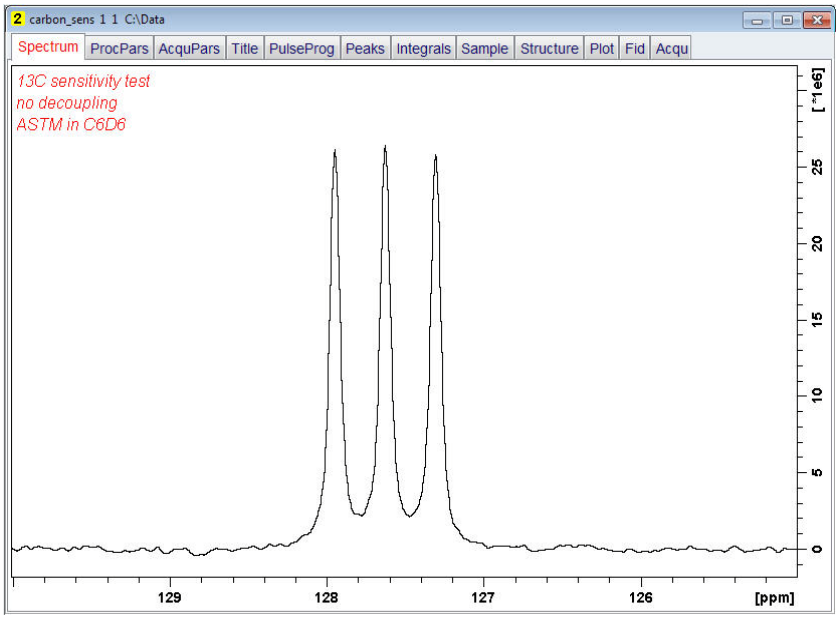
2. Click on 

.

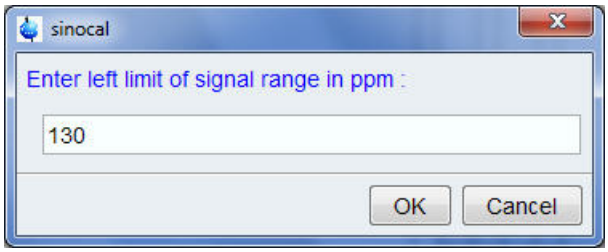
This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**'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

10.4.4 Calculating The Signal To Noise Ratio

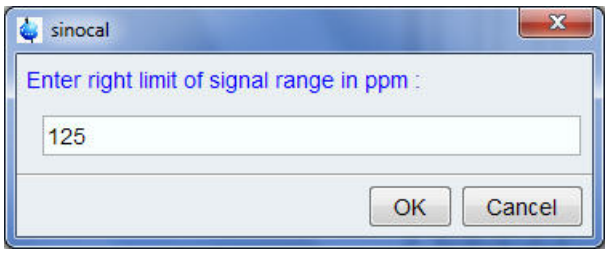
The signal to noise ratio is determined on the triplet of the deuterated benzene between 127 ppm and 129 ppm. It is calculated by AU-program **sinocal** over a range of 4 ppm between 70 ppm and 125 ppm. The s/n ratio is strongly dependant on good resolution and line shape. The splitting of the 1:1:1 triplet should go lower than 9% (5mm) see Figure 10.24. 10% (10 mm) and 12% (20 mm).



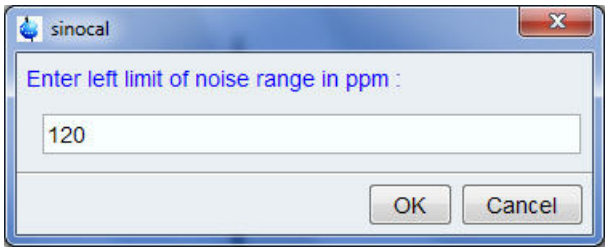
1. Type **sinocal** on the command line.



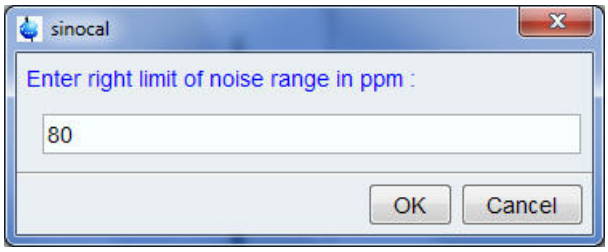
2. Enter **128** for the left limit of the signal range.
3. Click on OK.



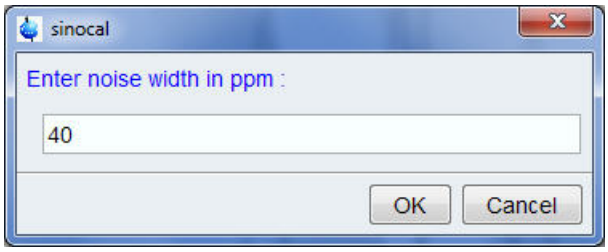
4. Enter **127** for the right limit of the signal range.
5. Click on OK.



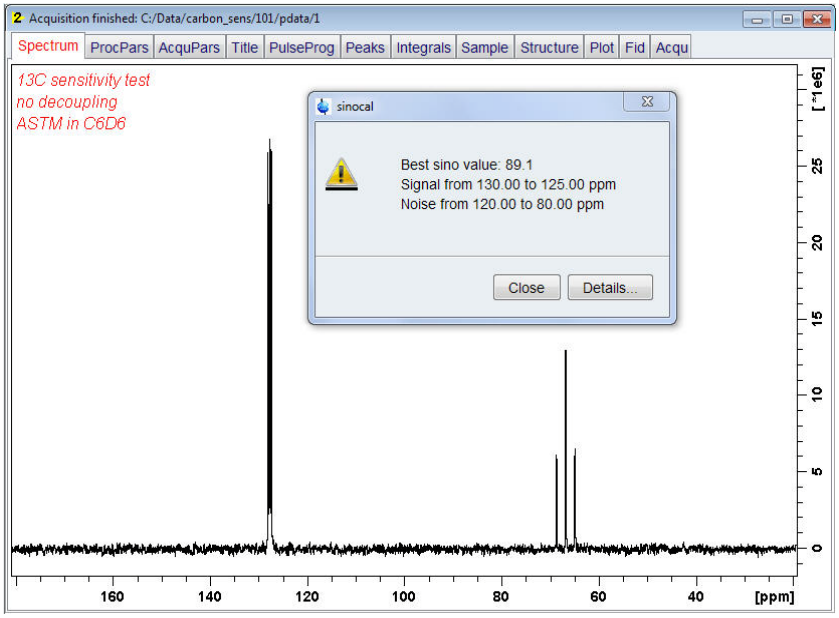
6. Enter **125** for the left limit of the noise range.
7. Click on OK.



8. Enter **30** for the right limit of the noise range.
9. Click on OK.



10. Enter **40** for the noise width.
11. Click on OK.


2 Acquisition finished: C:/Data/carbon_sens/101/pdata/1
Spectrum
ProcPars
AcquPars
Title
PulseProg
Peaks
Integrals
Sample
Structure
Plot
Fid
Acqu
13C sensitivity test
no decoupling
ASTM in C6D6
sinocal
Best sino value: 89.1
Signal from 130.00 to 125.00 ppm
Noise from 120.00 to 80.00 ppm
Close
Details...
[*1e6]
25
20
15
10
5
0
160
140
120
100
80
60
40
[ppm]

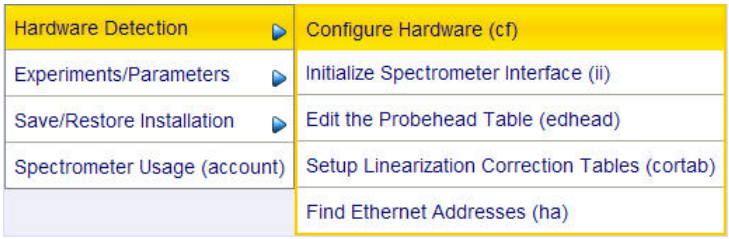
10.5 Observations

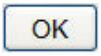
11 Spectrometer Configuration

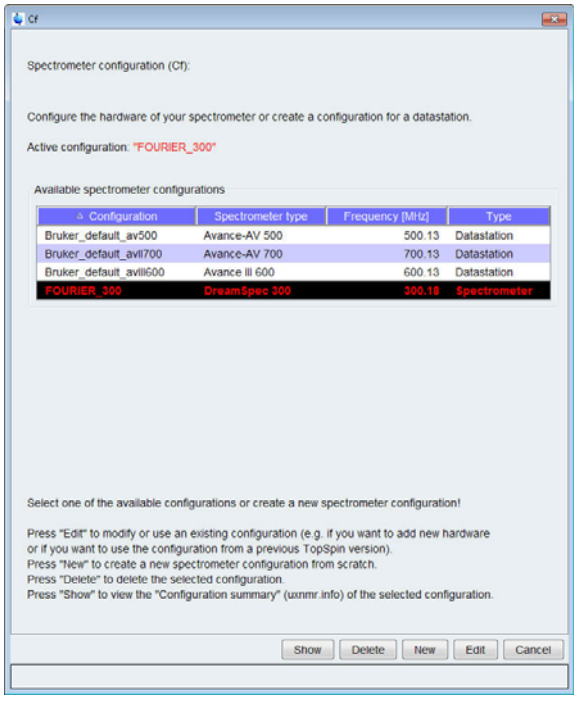
11.1 Hardware Configuration

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

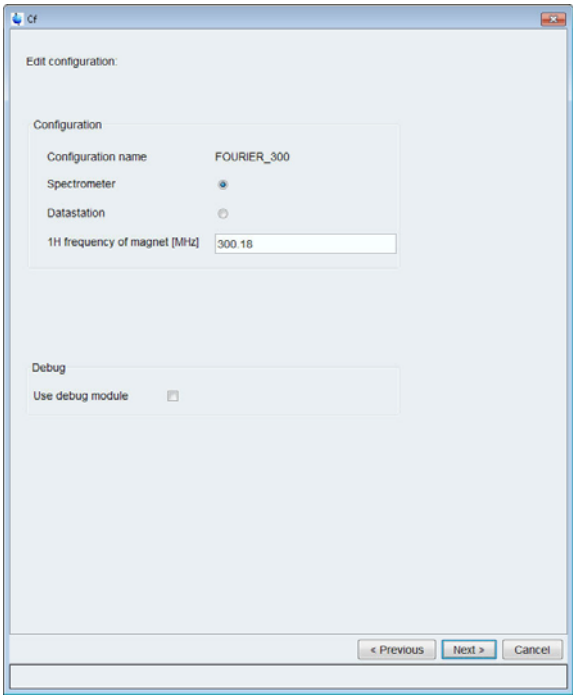
2. Click on the down arrow inside the  button.



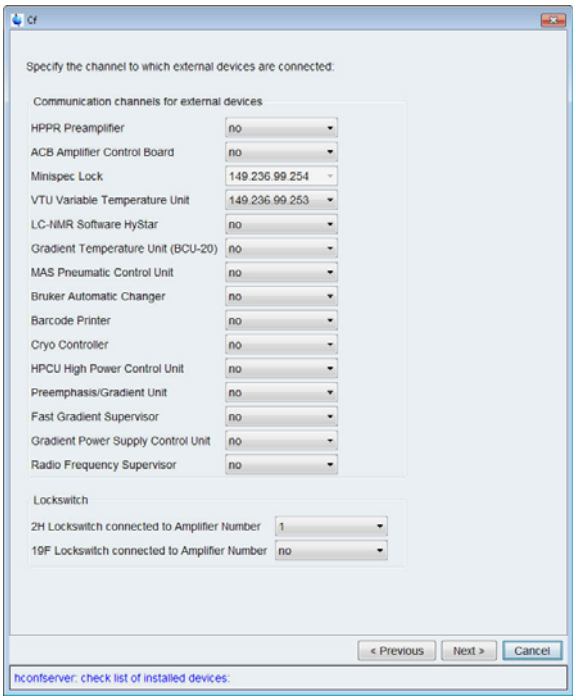
3. Select '**Hardware Detection**'.
4. Select '**Configure Hardware (cf)**' by clicking on it.
5. Enter the NMR administration password.
6. Click on  .



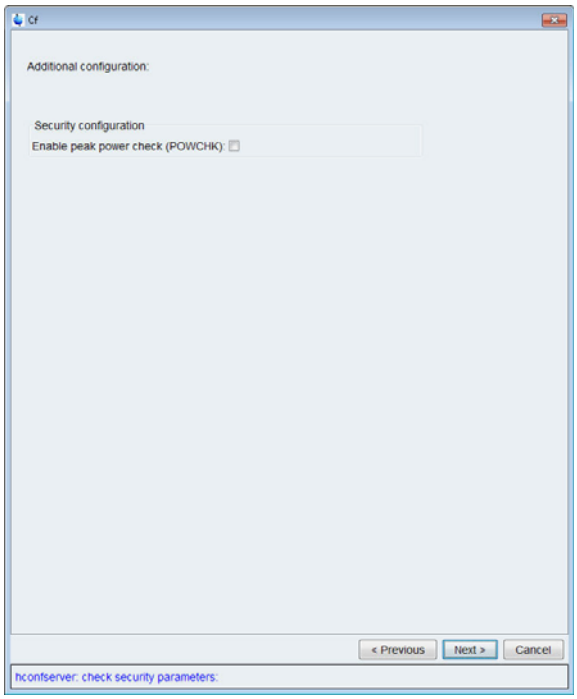
6. Select Configuration for **'Spect'** by clicking on it.
7. Click on Edit.



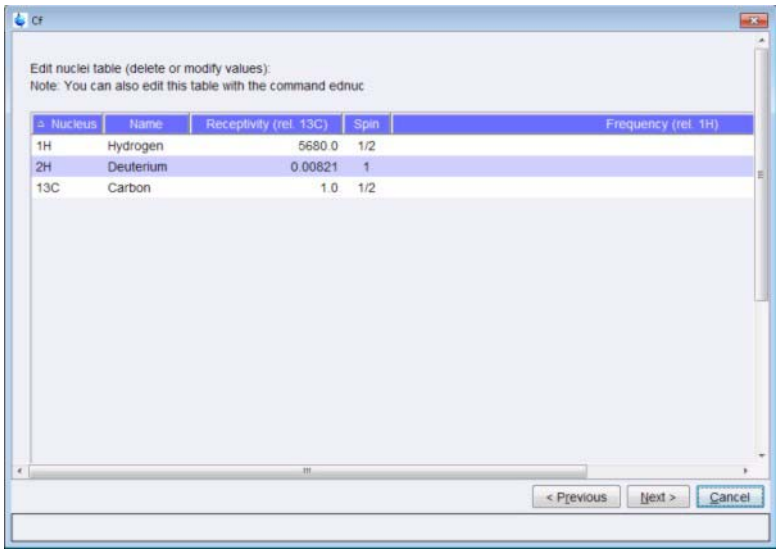
8. Click on Next >.



9. Enter the ports for the external devices as shown in the figure above.
10. Click on Next >.



11. Click on Next >.

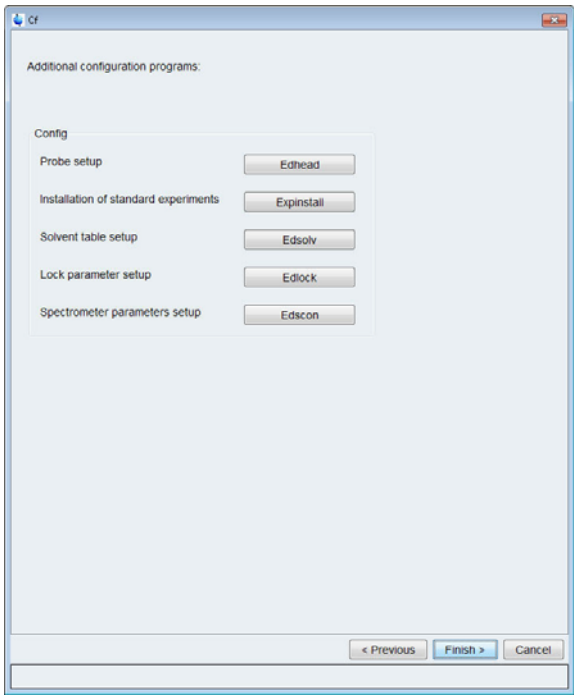


12. Click on Next >.

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

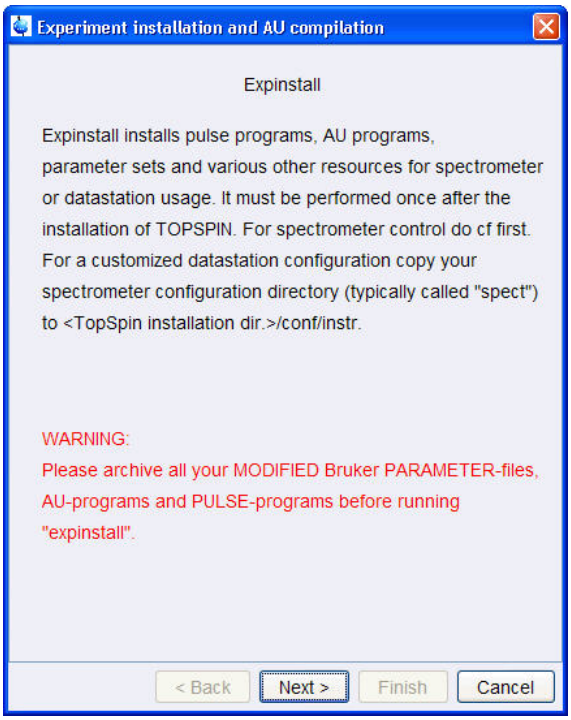
13. Click on Print.

14. Click on Next >.

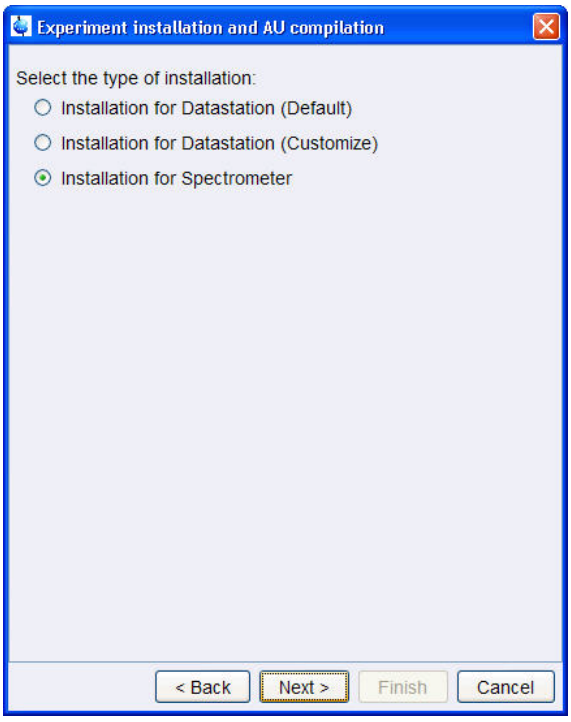


11.2 Expinstall

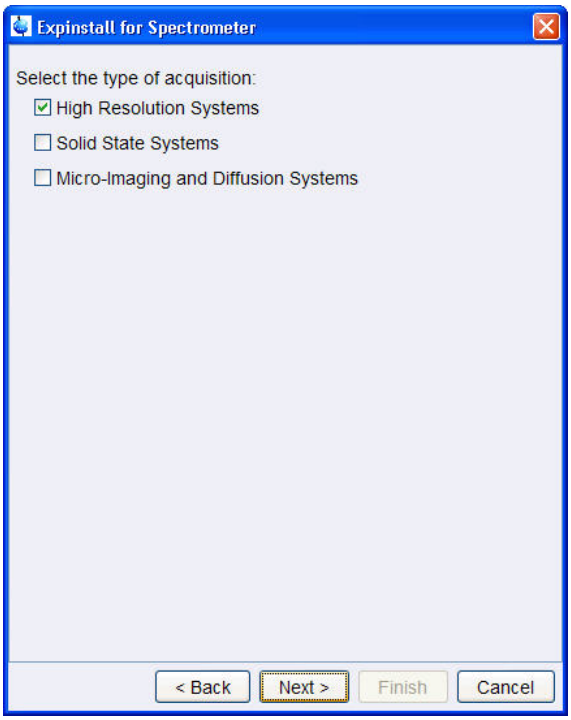
1. Click on Expinstall.
2. Enter the NMR administration password.
3. Click on OK.



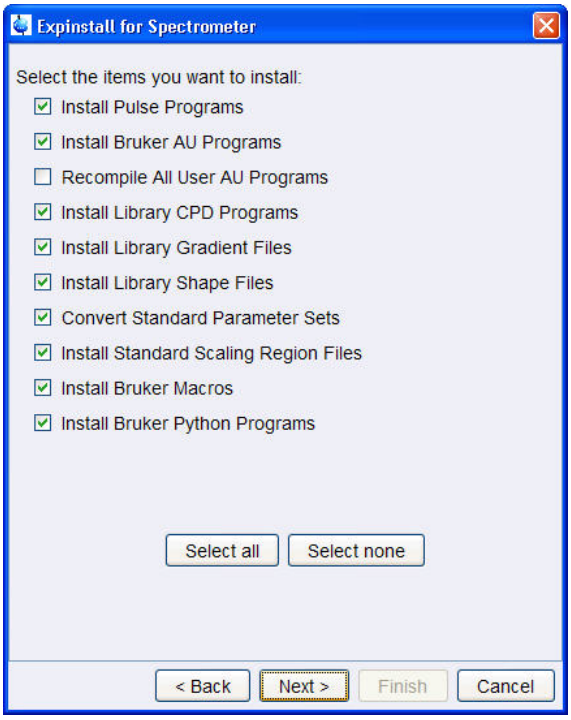
4. Click on Next > .



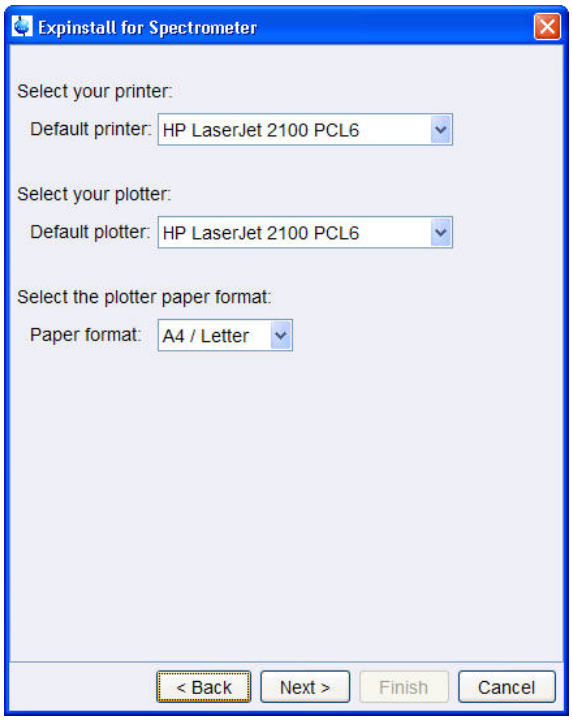
5. Select **'Installation for Spectrometer'.**
6. Click on Next > .



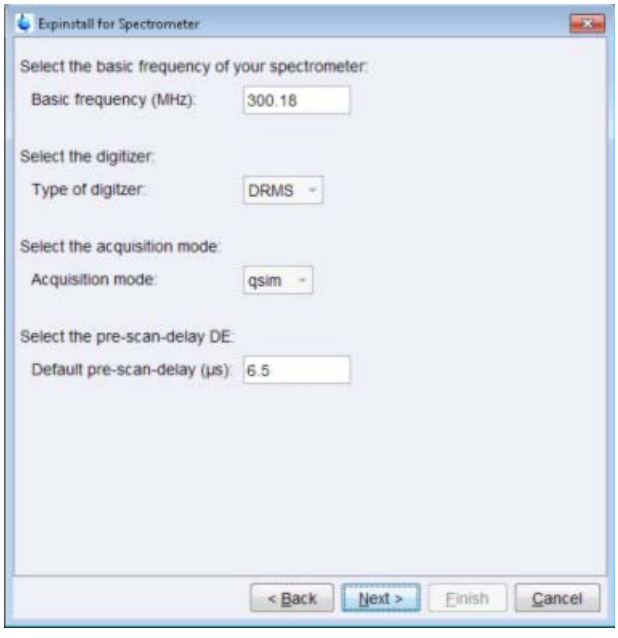
7. Select **'High Resolution System'.**
8. Click on Next > .



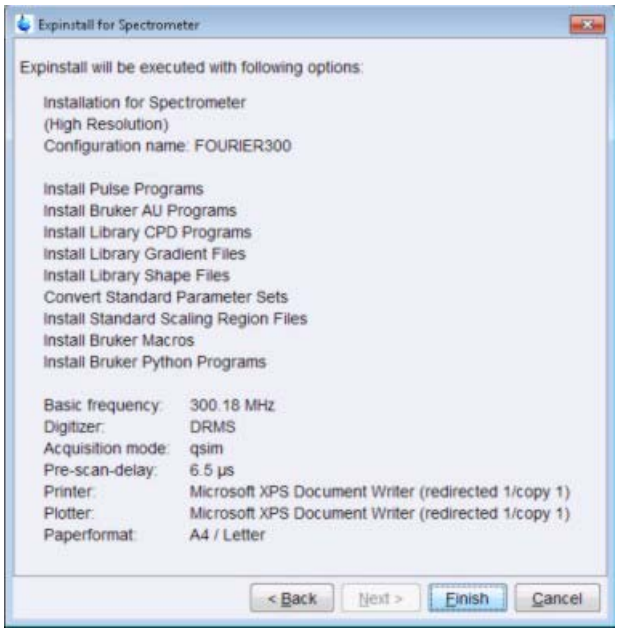
9. Click on Next > .



10. Select Default printer and plotter.
11. Select Paper format.
12. Click on OK.

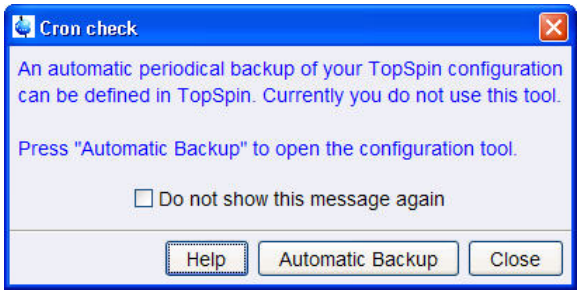


13. Click on Next >.



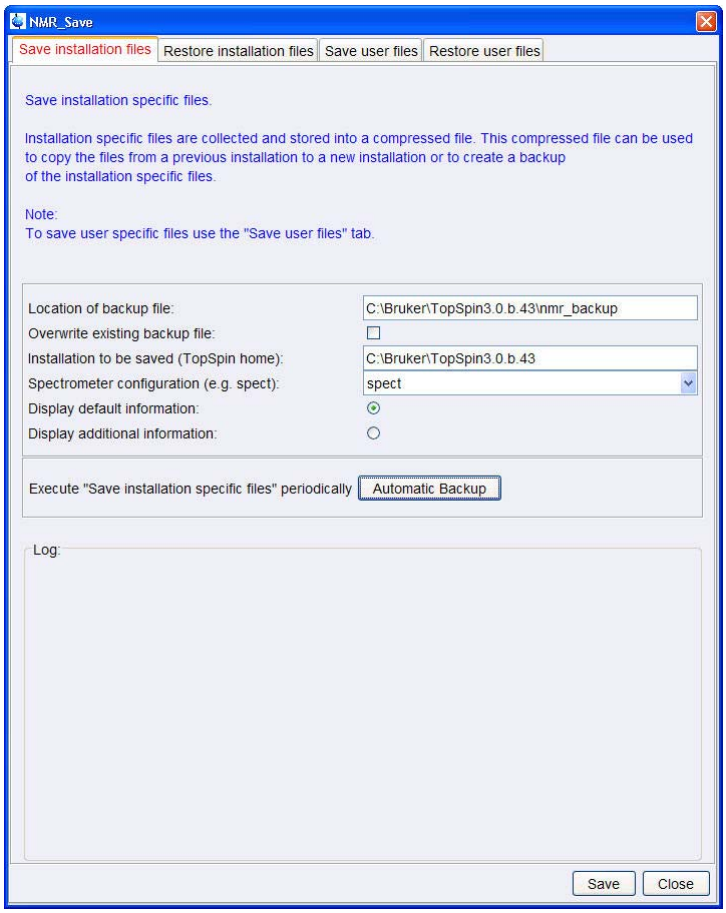
14. Click on Finish.

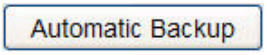
expinstall starts now. This process will take approximately. 30 seconds. On finish the message below appears. To set up a time schedule to perform an NMR_save periodically (recommended) follow the instructions in **10.3 Set up the cron job for NMR_save.**

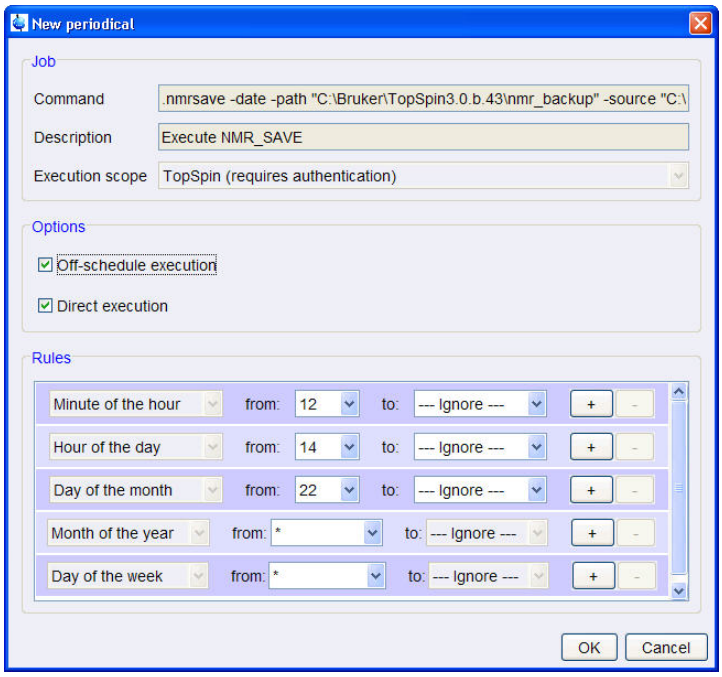


11.3 Set Up The Cron Job For NMR_save

1. Click on Automatic Backup.

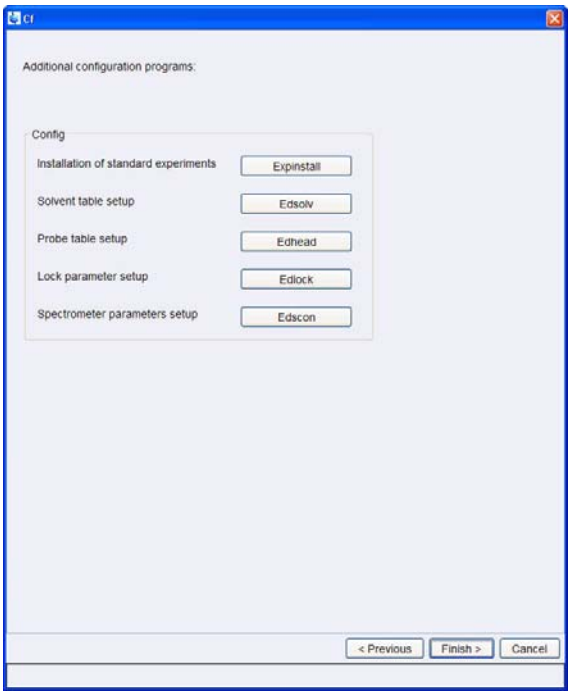

NMR_Save
Save installation files
Restore installation files
Save user files
Restore user files
Save installation specific files.
Installation specific files are collected and stored into a compressed file. This compressed file can be used to copy the files from a previous installation to a new installation or to create a backup of the installation specific files.
Note:
To save user specific files use the "Save user files" tab.
Location of backup file:
C:\Bruker\TopSpin3.0.b.43\nmr_backup
Overwrite existing backup file:
Installation to be saved (TopSpin home):
C:\Bruker\TopSpin3.0.b.43
Spectrometer configuration (e.g. spect):
spect
Display default information:
Display additional information:
Execute "Save installation specific files" periodically
Automatic Backup
Log:
Save
Close

2. Click on 
Automatic Backup
.


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

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 OK.



11.4 Observations

12 Standard Parameter Set List

12.1 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

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

12.2 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

HSQCEDGPPH - sw opt. edited HSQC with gradients (TPPI)

HSQCGPPH - sw opt. HSQC sens. improved with gradients (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.

13 Contact

Manufacturer:

Bruker BioSpin NMR

Silberstreifen

D-76287 Rheinstetten

Germany

Phone: +49 721-5171-02
http://www.bruker.com

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.com/service/information-communication/helpdesk.html

Bruker Corporation

info@bruker.com
www.bruker.com

Order No: Z31979