

# Fourier 300

NMR Experiments
 User Manual
 Version 004

Innovation with Integrity

NMR

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# 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.



### 

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



# **A** WARNING

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



# **A**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.

İ

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.

# 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<sup>3</sup> (1,400 ft<sup>3</sup>) 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

# 

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

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

#### 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

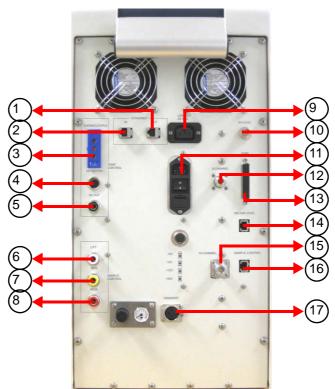
In addition to the above safety precautions, any country-specific safety regulations for operating NMR systems must be fulfilled. These may include, for example, regulations on:

- 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



		100	
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

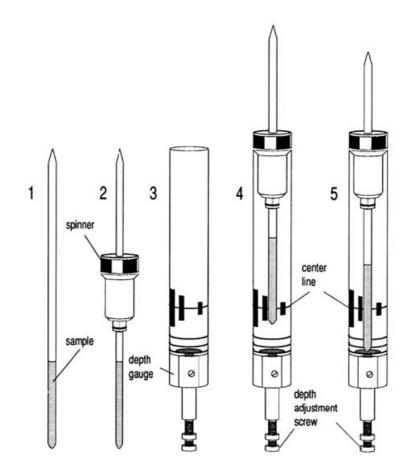
Figure 4.1	Fourier	Connection	Overview
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#### 4.2 Sample Preparation

- · 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.

# j

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<sup>2</sup>, Z<sup>3</sup>, Z<sup>4</sup>, Z<sup>5</sup>, Z<sup>6</sup>) **14 off-axis** shims (X, XZ, XZ<sup>2</sup>, Y, YZ, YZ<sup>2</sup>, XY, XYZ, X<sup>2</sup>-Y<sup>2</sup>, (X<sup>2</sup>-Y<sup>2</sup>)z, X<sup>3</sup>, XZ<sup>3</sup>, Y<sup>3</sup>, YZ<sup>3</sup>)

#### 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.

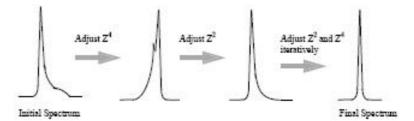
#### 4.7 Optimizing Resolution and Lineshape

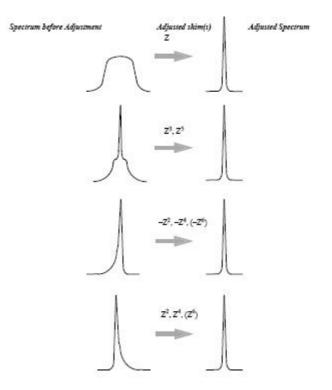
The standard sample for measuring the proton lineshape and resolution specifications is, 3% CHCL3 in Acetone-d6.

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.



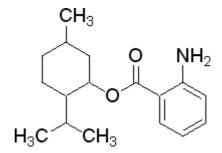


### 4.8 Observations

# 5 1-D Proton Experiment

#### 5.1 Sample

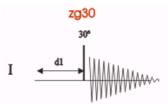
30mg Menthyl Anthranilate in DMSO-d6



#### 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.

<u>S</u> tart	<u>A</u> cquire	<u>P</u> rocess	A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage 🕜	
	C <u>r</u> eate	Dataset	📕 F <u>i</u> nd Dataset	🛛 🕥 Ope	n <u>D</u> ataset	Paste Dataset	Read Pars.

2. Select Create Dataset by clicking on it.

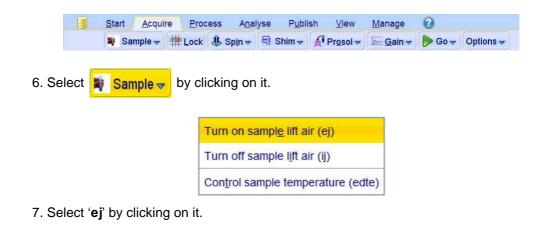
3. Enter the following information in to the 'New' window.

🤹 New	-	×							
Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. For multi-receiver experiments several datasets are created. Please define the number of receivers in the Options.									
NAME proton_exp									
EXPNO	1								
PROCNO	1								
O Use current parameters									
Experiment PROTON		Select							
<ul> <li>Options</li> </ul>									
Set solvent:	Set solvent: DMSO -								
Execute "getprosol	"								
Keep parameters:	© Keep parameters: P 1, O1, PLW 1 ▼ Change								
DIR		C:\pz\data 👻							
🕅 Show new dataset	in new window								
Receivers (1,2,16)	).	1							
1D-Proton of 30mg Ment	experiment yl Anthranilate in	DMSO-d6							
	ОК	Cancel More Info Help							

j

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.

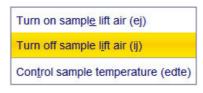
- 4. Click on OK
- 5. Click on the 'Acquire' tab in the TopSpin menu bar.



j

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

- 8. Place the sample on to the top of the magnet.
- 9. Select 🙀 Sample 🗸 by clicking on it.



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.



△ Solvent	Description		
Acetone	acetone-d6		
C6D6	benzene-d6		
C6D6+Dioxane	ASTM Sample		
CDCI3	chloroform-d		
D2O	deuteriumoxide		
DMSO	dimethylsulfoxide-d6		
EtOD	ethanol-d6		
H2O+D2O	90%H2O and 10%D2O		
MeOD	methanol-d4		
Tol	toluene-d8		
	OK Cancel		

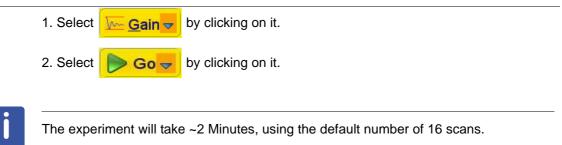
12. Select 'DMSO' by clicking on it.

13. Select <b>Brin</b> by clicking on it.							
	Turn sample rotation on (ro on)						
	Turn sample rotation off (ro off)						
	Change sample rotation rate (ro)						
	MAS Pneumatic Unit (masdisp)						
14. Select ' <b>ro on</b> ' by clicking	on it.						
15. Select <mark>दि Shim </mark> by	clicking on it.						
16. Select <mark>ff Pr<u>o</u>sol</mark> by	clicking on it.						

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

#### 5.2.3 Acquisition

İ



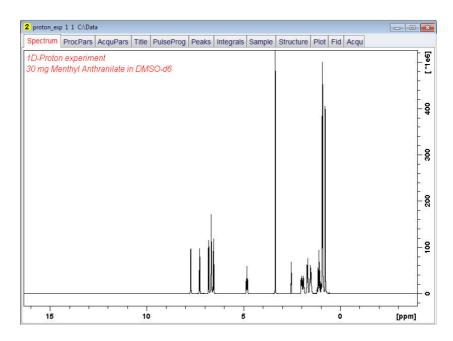
#### 5.2.4 Processing

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

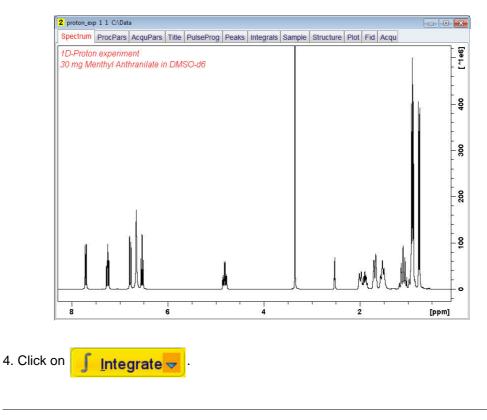




This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**'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



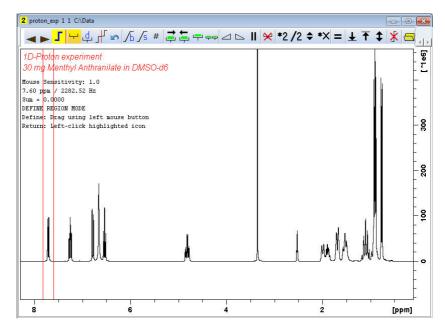
3. Expand the spectrum to include all peaks.



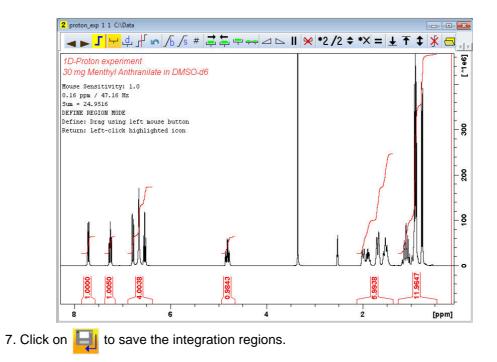
j

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.



#### 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

🖕 exactzoom	
Please enter of the desired	the exact coordinates I expansion.
	F1 [ppm]
From	8.2
То	0.2
	OK Cancel

4. Click on OK

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

<u>S</u> tart	<u>A</u> cquire	<u>P</u> rocess	A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	2
		<u> </u>	opy 🗳 P <u>r</u> int		t Layout <del>-</del>		<u>E</u> -Mail

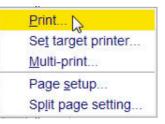
6. Click on Brief Plot Layout

1 Menthyl_anthranilate 1 1 C:\Dat	a		20		5			
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Layout:								
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Limits: 🕂 R Expand								r2         - arguidtion samerter           bate         -           bate         -           tive         13.25           Environ         Promato           sconstr         19001200           sconstr         19001200           sconstr         19001200           sconstr         19001200           sconstr         1970           ro         25352           sconstr         1980
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								per 81.920 us≉e 5.00 us≉e FE 300.1 m DL 1.00000000 s∗e πE0 L
Click here to insert new elements:								GRADNEL E1           arei         200.151537 mm           mvcl         1m           si         5.00 usec           suri         5.00 usec
Standard NMR	1 1 1 1						. 1	P2 - Processing parameters of 23522 of 300.1300000 mm 1000 mm
		8					h h	002. 0 328. 0.30 921 920 1.00
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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.



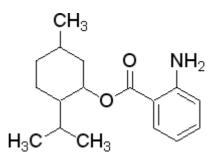
8. Select 'Print' by clicking in it.

## 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

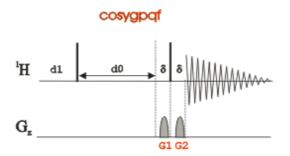
#### 6.2.1 Introduction

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.

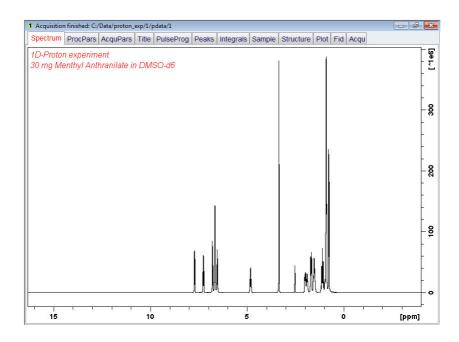
Section 6.2.3 describes the acquisition and processing of a two-dimensional 1H gradient COSY. The standard Bruker parameter set is **COSYGPSW** and includes the pulse sequence **cosygpqf** shown in the figure below. It consists of the relaxation delay, two radio frequency (RF) pulses, separated by the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



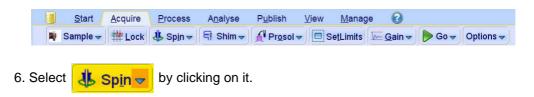
3. Enter the following information in to the 'New' window

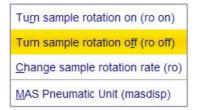
🖕 New				
Prepare for a new experime initializing its NMR paramet For multi-receiver experime Please define the number of	ers according to ents several data	the selected experiment type. sets are created.		
NAME	cosy_exp			
EXPNO	1			
PROCNO	1			
O Use current parameters				
Experiment COSYGPS	V	Select		
<ul> <li>Options</li> </ul>				
Set solvent:		DMSO 🔻		
Execute "getproso	la.			
Keep parameters:		P 1, O1, PLW 1 Change		
DIR		C:\Data		
🖾 Show new dataset	in new window			
Receivers (1,2,16	)	1		
2-D COSY 30 mg Men	experiment thyl Antranilate ir	n DMSO d-6		
	ОК	Cancel More Info Help		



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.



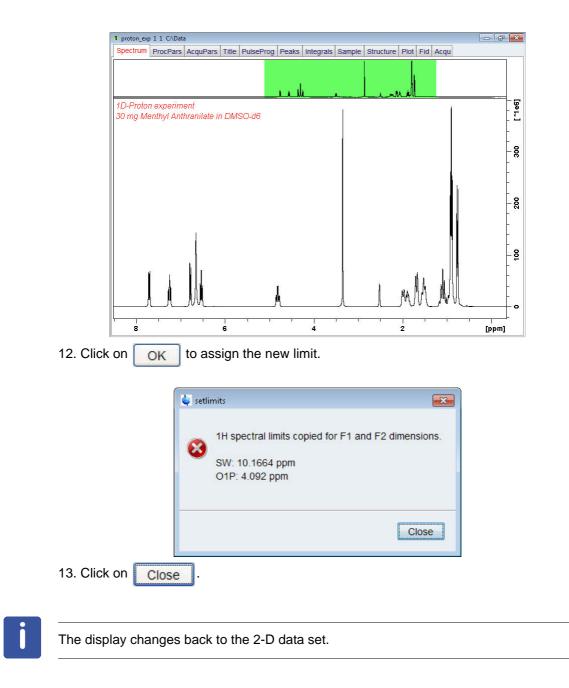


7. Select 'ro off' by clicking on it.

2-D exper	riments should always be run without rotation.	
8. Select	<b>Prosol</b> by clicking on it.	
This will lo	oad the pulse width and power levels in to the parameter set.	
9. Select	SetLimits by clicking on it.	
	🖨 setlimits	
	Close this dialog box after setting frequencies.  1. Open 1D dataset from Browser.  2. Zoom into region of interest.	
	3. Click OK to set frequencies and return to original dataset.	

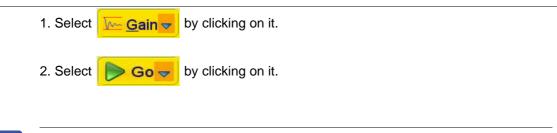
10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton\_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window.

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



# **2-D Homonuclear Experiments**

#### 6.2.4 Acquisition

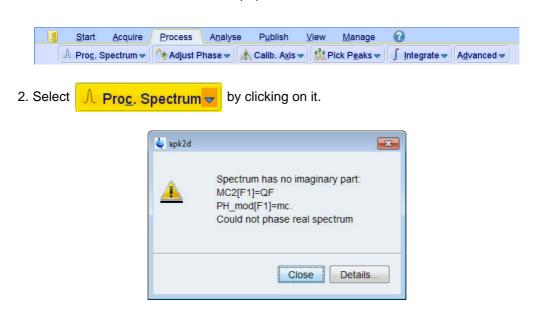


The experiment will take ~5 Minutes, using the default number of 1 scan and 128

# 6.2.5 Processing

increments.

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



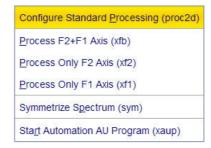


This executes a standard processing program **proc2**. The message shown in the figure above, pops up in case of a magnitude 2D experiment and the apk2d option is enabled. To configure the processing program follow the steps below.

button.

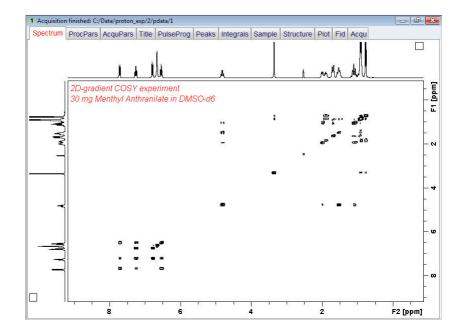
3. Click on the down arrow inside the  $\Lambda$  Proc. Spectrum

Z31979\_00\_004



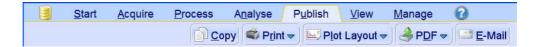
4. Select 'Configure Standard Processing' by clicking on it.

rress 'Execute' to process the current d rress 'Save' to just change the processi Changed options will be effective when p rne-click 'Proc. Spectrum' button.	ng o	ptions.		
Fourier Transform (xfb)				
Auto - Phasing (apk2d)				
Auto - Baseline Correction [F2] (abs2)	<b>v</b>			
Auto - Baseline Correction [F1] (abs1)	V			
Plot (autoplot)		LAYOUT =	+/2D_hom.xwp	÷
Warn if processed data exist	V			

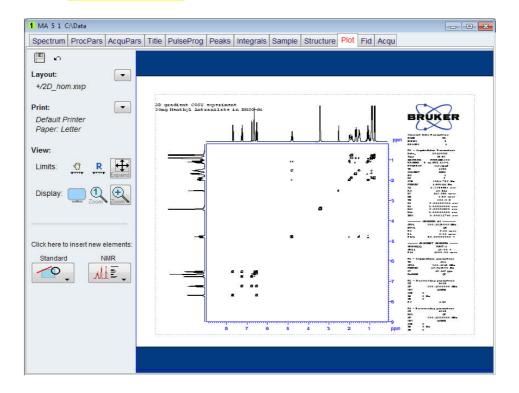


#### 6.2.6 Plotting

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



## 4. Click on Plot Layout





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.

	Print
	Set target printer
	Multi-print
	Page setup
	Split page setting
_	

6. Select 'Print' by clicking in it.

# 6.3 Observations

# 6.4 2-D Gradient NOESY Experiment

### 6.4.1 Introduction

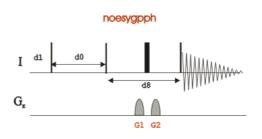
NOESY (Nuclear Overhauser Effect SpectroscopY) is a 2D spectroscopy method used to identify spins undergoing cross-relaxation and to measure the cross-relaxation rates. Most commonly, NOESY is used as a homonuclear 1H technique. In NOESY, direct dipolar couplings provide the primary means of cross-relaxation, 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.

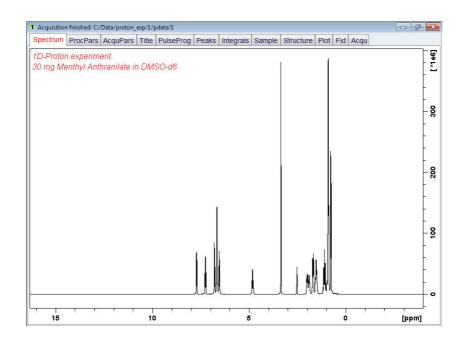
Section 6.4.3 describes the acquisition and processing of a two-dimensional 1H phase sensitive NOESY. The standard Bruker parameter set is **NOESYGPPHSW** and includes the pulse sequence **noesygpph** shown in the figure below. It consists of the relaxation delay, three radio-frequency (RF) pulses, separated 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<sup>0</sup>.



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.



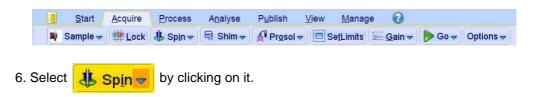
3. Enter the following information in to the 'New' window.

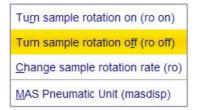
🖕 New	
Prepare for a new experiment by initializing its NMR parameters a For multi-receiver experiments s Please define the number of rec	ccording to the selected experiment type. several datasets are created.
NAME	noesy_exp
EXPNO	1
PROCNO	1
O Use current parameters	
Experiment NOESYGPPHSW	/ Select
<ul> <li>Options</li> </ul>	
Set solvent:	DMSO -
Execute "getprosol"	1977 - E1976
Keep parameters:	P 1, O1, PLW 1 - Change
DIR	C:\Data 👻
🔲 Show new dataset in ne	w window
Receivers (1,2,16)	1
	itive gradient NOESY experiment ntranilate in DMSO-d6
	OK Cancel More Info Help



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.





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

2-D expe	riments should always be run without rotation.
8. Select	Prosol by clicking on it.
This will le	oad the pulse width and power levels in to the parameter set.
9. Select	SetLimits by clicking on it.
	🐳 setlimits
	<ul> <li>setlimits</li> <li>Close this dialog box after setting frequencies.</li> <li>1. Open 1D dataset from Browser.</li> <li>2. Zoom into region of interest.</li> </ul>

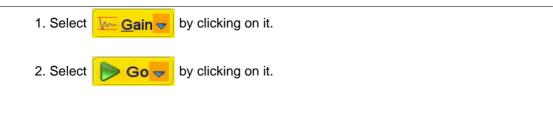
10. To open the 1D Proton spectrum, right click on the dataset name in the browser window (e.g. proton\_exp 1) and select 'Display' or click and hold the left mouse button for dragging the 1D Proton dataset in to the spectrum window.

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

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3. Click c	on Close						
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his will lo 14. Sele 5. Click o	bad the puls	se width and <b>uPars</b> ' tab b display the p	by clicking o	n it.	-	eter set.	
his will Ic 114. Sele 5. Click c	oad the puls ct the ' <b>Acq</b> on <u>, to c</u>	se width and <b>uPars</b> ' tab b display the p	by clicking o	n it.	-	eter set.	
his will lo 14. Sele 5. Click o	oad the puls ct the ' <b>Acq</b> on <u> </u>	se width and <b>uPars</b> ' tab b display the p	by clicking o	n it.	-	eter set.	Mixing

# **2-D Homonuclear Experiments**

### 6.4.4 Acquisition





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.

	<u>S</u> tart Pro <u>c</u> . S	<u>A</u> cquire Spectrum <del>→</del>	<u>P</u> rocess ∕∳ Adjust F	A <u>n</u> alyse Phase <del>v</del> A	P <u>u</u> blish ∖ Calib. A <u>x</u> is <del>⊲</del>	<u>V</u> iew ▼	<u>M</u> anage ick P <u>e</u> aks <del>⊽</del>	Ø ∫ Integrate ⇒	A <u>d</u> vanced <del>マ</del>
2. Select	A	Pro <u>c</u> . S	pectrum	🚽 by d	clicking o	n 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.

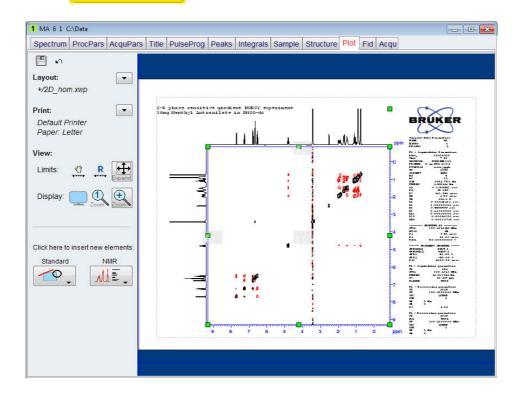
ProcPars AcquPars	ete	A				
NOESYGPPHSW		~ ~	L L	. d.		
NOESYGPPHSW	DMSO Cilloon		1	_ mm	_wul	
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### 6.4.6 Plotting

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



## 4. Click on Plot Layout -



# i

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

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

Print
Set target printer
Multi-print
Page setup
Split page setting

6. Select 'Print' by clicking in it.

# 6.5 Observations

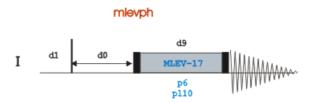
# 6.6 2-D Phase Sensitive TOCSY Experiment

### 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.

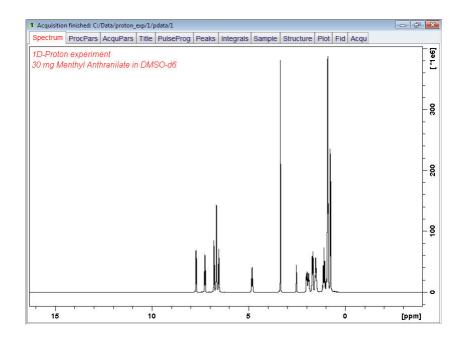
Section 6.6.3 describes the acquisition and processing of a two-dimensional 1H phase sensitive TOCSY. The standard Bruker parameter set is **MLEVPHSW** and includes the pulse sequence **mlevph** shown in the figure below. It consists of the recycling delay, two radio-frequency (RF) pulses, separated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

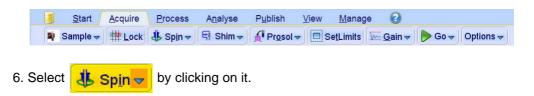
	Ctart	Assuirs	Drasses	Analyza	Dublish	View	Managa	0	
	<u>S</u> tart	<u>A</u> cquire	Process	A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	U	
		C <u>r</u> eate	Dataset	Find Datase	🛛 🌀 Open	n <u>D</u> ataset	🚺 Paste I	Dataset	Read Pars.
2. Selec	ct 🚺	C <u>r</u> eate [	Dataset	by clicking	on it.				
3. Enter	the fol	lowing in	formation	n in to the '	New' wii	ndow.			

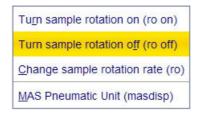
🎍 New	
initializing its NMR parameters	by creating a new data set and according to the selected experiment type. a several datasets are created. eceivers in the Options.
NAME	tocsy_experiment
EXPNO	1
PROCNO	1
O Use current parameters	
Experiment MLEVPHSW	Select
<ul> <li>Options</li> </ul>	
Set solvent:	DMSO -
Execute "getprosol"	- 107 <del>2</del>
Keep parameters:	P 1, O1, PLW 1  Change
DIR	C:\Data
🖾 Show new dataset in r	new window
Receivers (1,2,16)	1
	nsitive TOCSY experiment Antranilate in DMSO-d6
	OK Cancel More Info Help



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.





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

i	-D experiments should always be run without rotation.	
	. Select <b>Prosol</b> by clicking on it.	
i	his will load the pulse width and power levels in to the parameter set.	
	. Select SetLimits by clicking on it.	
	🧔 setlimits	
	<ul> <li>Close this dialog box after setting frequencies.</li> <li>1. Open 1D dataset from Browser.</li> <li>2. Zoom into region of interest.</li> <li>3. Click OK to set frequencies and return to original dataset.</li> </ul>	
	OK Cancel	

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.



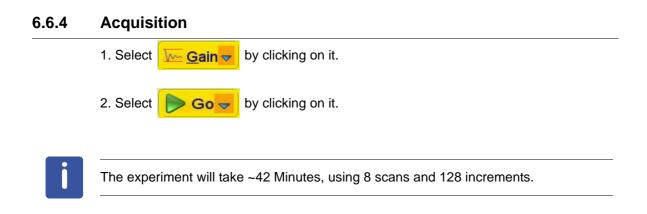
The solvent peak may be excluded if it falls outside of the region of interest. Digital filtering however is only applied in F2 and the solvent peak is folding in F1.

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		experiment hthyl Anthranila	ite in DMSO-di	6		ſ		Ĩ	
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12. Click o	on 🦲	K to a	ssign the	e new li	mit.				
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			-					×	
		seti	-				ĺ	×	
			limits		-	F1 and F2	2 dimensions		
			limits 1H spectr	ral limits co	-	F1 and F2	-		
			limits	ral limits co 664 ppm	-	F1 and F2	-		
			Imits 1H spectr SW: 10.1	ral limits co 664 ppm	-	F1 and F2	-		
			Imits 1H spectr SW: 10.1	ral limits co 664 ppm	-	F1 and F2	2 dimensions		
			Imits 1H spectr SW: 10.1	ral limits co 664 ppm	-	F1 and F2	-		
		Seti	Imits 1H spectr SW: 10.1	ral limits co 664 ppm	-	F1 and F2	2 dimensions		
13. Click o			Imits 1H spectr SW: 10.1	ral limits co 664 ppm	-	F1 and F2	2 dimensions		
		Seti	Imits 1H spectr SW: 10.1	ral limits co 664 ppm	-	F1 and F2	2 dimensions		
13. Click o	on C	Silose .	Imits 1H spectr SW: 10.1 O1P: 4.09	ral limits co 664 ppm 92 ppm	opied for I	F1 and F2	2 dimensions		
	on C	Silose .	Imits 1H spectr SW: 10.1 O1P: 4.09	ral limits co 664 ppm 92 ppm	opied for I	F1 and F2	2 dimensions		
13. Click o	on C	Silose .	Imits 1H spectr SW: 10.1 O1P: 4.09	ral limits co 664 ppm 92 ppm	opied for I	F1 and F2	2 dimensions		

TD (F1) = **128** 

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

j

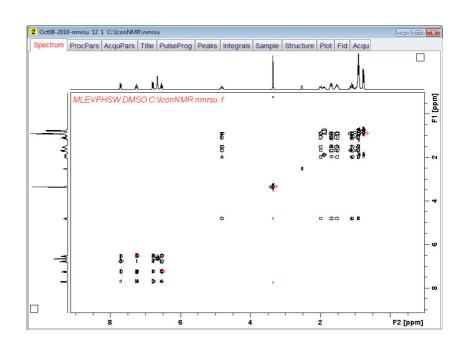


### 6.6.5 Processing

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



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.

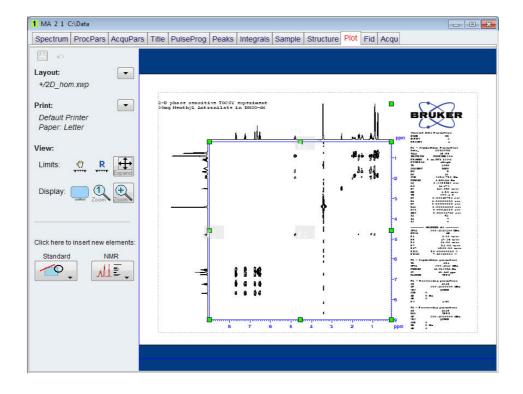


#### 6.6.6 Plotting

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

	<u>S</u> tart	<u>A</u> cquire	<u>P</u> rocess	A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	0
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- 4. Click on Plot Layout
- 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 **w** in the Print section on the left side of the display.

	Print
	Set target printer
	Multi-print
	Page setup
	Split page setting
_	

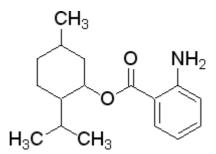
7. Select 'Print' by clicking in it.

# 6.7 Observations

# 7 1-D Carbon Experiments

## 7.1 Sample

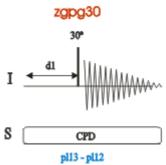
A sample of **30mg Menthyl Anthranilate in DMSO-d6** is used for the experiments in this chapter



### 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.

<u>S</u> ta	art 🔪	<u>A</u> cquire	Process	A <u>n</u> alyse I	P <u>u</u> blish	<u>V</u> iew	<u>Manage</u>	2	
		C <u>r</u> eate	Dataset	📕 F <u>i</u> nd Dataset	🕥 Open !	<u>D</u> ataset	Paste I	Dataset	Read Pars.

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

🖕 New	a come too					
Prepare for a new experiment initializing its NMR parameters For multi-receiver experiments Please define the number of re	according to the several datase	e selected experiment type. ts are created.				
NAME	carbon_exp					
EXPNO	1					
PROCNO	1					
O Use current parameters	O Use current parameters					
Experiment C13CPD	Experiment C13CPD     Select					
<ul> <li>Options</li> </ul>	<ul> <li>Options</li> </ul>					
☑ Set solvent: DMSO ▼						
© Execute "getprosol"						
Keep parameters:		P 1, O1, PLW 1  Change				
DIR		C:\Data 👻				
🖾 Show new dataset in r	iew window					
Receivers (1,2,16)		1				
	iment with 1H d Anthranilate in I					
	ОК	Cancel More Info Help				

j

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 = <b>12</b>	B
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7. Click on the 'Acquire' tab in the TopSpin menu bar.

	<u>S</u> tart <u>A</u> Samp	Acquire Process le → ₩Lock 4	A <u>n</u> alyse	P <u>u</u> blish Shim –		<u>M</u> anage <u>⊡</u> Gain <del>▼</del>	2 Co →	Options <del>~</del>
	Samp		ohīn ▲ u		• FI <u>0</u> 501 ♥		00	Options
8. Select	🛊 Samp	le 🚽 by clicki	ng on it.					
			Insert	Sample	(ii)			
				Sample (				
9. Select '	ei' by clic	kina on it.						
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Wait till the the magne		lift air is turne	d on and	l remove	e any sa	ample wł	nich ma	y have t
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		le on to the to	op of the	bore tub	e.			
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10. Place	the samp		king on it.	Sample	(ij)			



clicking sound may be heard.

Π

A Solvent	Description
Acetone	acetone-d6
C6D6	benzene-d6
C6D6+Dioxane	ASTM Sample
CDCI3	chloroform-d
D2O	deuteriumoxide
DMSO	dimethylsulfoxide-d6
EtOD	ethanol-d6
H2O+D2O	90%H2O and 10%D2O
MeOD	methanol-d4
Tol	toluene-d8

14. Select 'DMSO' by clicking on it.

15. Select	<mark>∛</mark> Sp <u>i</u> n <del>▼</del>	by clicking on it.
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Turn sample rotation on (ro on)
Turn sample rotation off (ro off)
Change sample rotation rate (ro)
MAS Pneumatic Unit (masdisp)

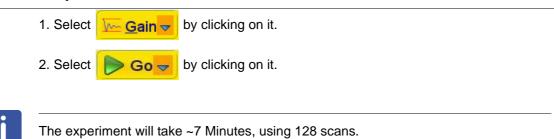
16. Select 'ro on' by clicking on it.

17. Select	덕감 Shim マ	by clicking on it.
18. Select	Frosol	by clicking on it.



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

#### 7.2.3 Acquisition



### 7.2.4 Processing

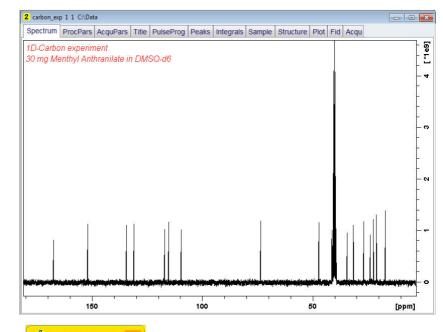
1. Click on the 'Process' tab in the TopSpin Menu bar.





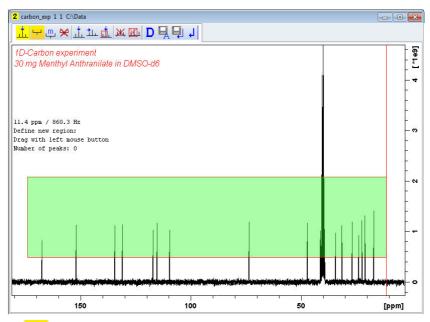
This executes a processing program including commands such as an exponential window function 'em', Fourier transformation 'ft', an automatic phase correction 'apk' and a baseline correction 'abs'. Other options are available by clicking on the down arrow inside the 'Proc. Spectrum' button.

3. Expand the spectrum to include all peaks.



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

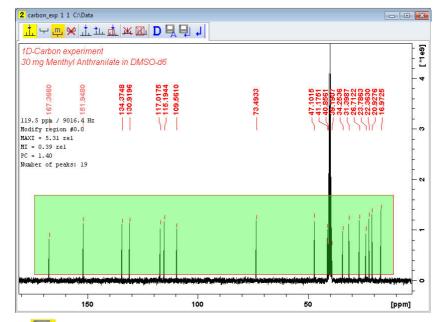
5. Click the left mouse button and drag the cursor line from left to the right side of the spectrum.

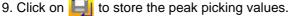


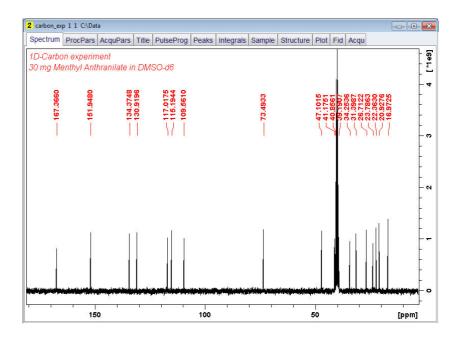
6. Click on manually adjust the minimum and maximum intensity levels.

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.







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 per .
- 3. Type the following F1 [ppm] values:

From = **180** To = **0** 

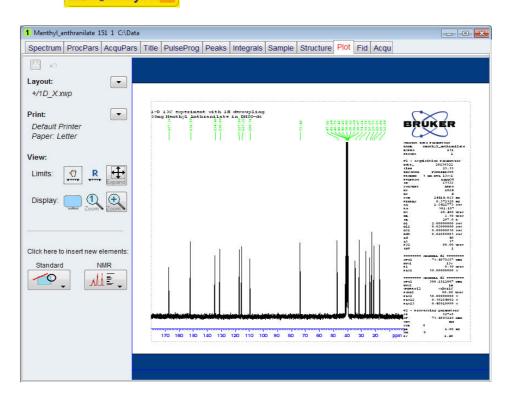
	the exact coordinate
of the desired	expansion.
	[ppr
From	180
То	o

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

# **1-D Carbon Experiments**



6. Click on E Plot Layout



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.

Print
Set target printer
Multi-print
Page setup
Split page setting

8. Select 'Print' by clicking in it.

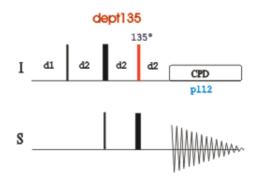
# 7.3 Observations

## 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 Create Dataset by clicking on it.
- 3. Enter the following information in to the 'New' window.

🧅 New	a Casar Sa			
Prepare for a new experiment initializing its NMR parameters For multi-receiver experiments Please define the number of re	according to the several datase	ne selected experiment type. ets are created.		
NAME	dept135_exp			
EXPNO	1			
PROCNO	1			
O Use current parameters				
Experiment C13DEPT135		Select		
<ul> <li>Options</li> </ul>				
Set solvent:		DMSO -		
Execute "getprosol"				
Keep parameters:		P 1, O1, PLW 1 Change		
DIR		C:\Data 👻		
🖾 Show new dataset in n	iew window			
Receivers (1,2,16)		1		
1-D 13C DEPT 30mg Menthyl				
	ОК	Cancel More Info Help		



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.

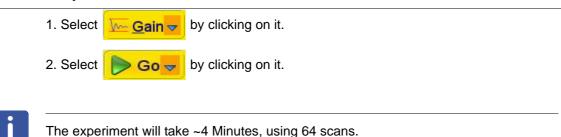


8. Select **Frosol** by clicking on it.



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

### 7.4.3 Acquisition



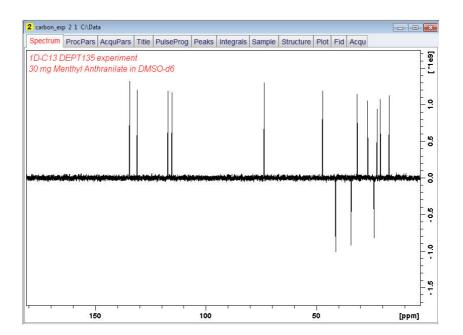
### 7.4.4 Processing

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





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

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📄 Copy 🗳 Print 🗢 🔛 Plot Layout 🗢 🦂 PD	<u>)</u> F <del>▼</del> <u></u> <b>E</b> -Mail	ıt	Plot	opy 🗳 P <u>r</u> int				

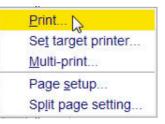
6. Click on Plot Layout -

<b>1</b> MA 11 1 C:\Data						98	<i>1</i> 2				
Spectrum ProcPars	AcquPars	Title	PulseProg	Peaks	Integrals	Sample	Structu	re Plot	Fid	Acqu	
<u>ب</u> 🖪											
Layout:	<b>•</b>										
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	<i>π</i> = -										arcer.e.[2 ualcold s3 9.40 usec s4 15.50 usec
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											0X 32748 0F 75.4303210 mmn 1007 888
			150 140 130		0 100 90			D 40	30 2		pm ** 0 1.40



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.



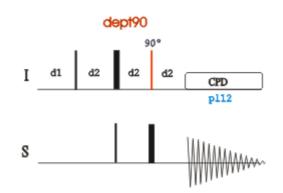
8. Select 'Print' by clicking in it.

## 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<sup>0</sup> RF pulse, an editing delay D2 followed by a 180<sup>0</sup> 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<sup>0</sup> RF pulse and are separated by the editing delay D2. The final 90<sup>0</sup> 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.

	<u>S</u> tart	Acquire	<u>P</u> rocess Dataset		P <u>u</u> blish	<u>∨</u> iew n <u>D</u> ataset	<u>M</u> anage	Oataset	Read Pars.
2. Select	C	<u>r</u> eate D	ataset	by clicking	on it.				

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

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Prepare for a new experimen initializing its NMR parameter For multi-receiver experiment Please define the number of	s according to t ts several datas	the selected experiment type. sets are created.				
NAME	dept90_ex	p				
EXPNO	1					
PROCNO	1					
O Use current parameters						
Experiment C13DEPT90		Select				
Options						
Set solvent:		DMSO				
Execute "getprosol"		1922 - 22.0				
Keep parameters:		P 1, O1, PLW 1  Change				
DIR		C:\Data				
🖾 Show new dataset in	new window					
Receivers (1,2,16)		1				
	1790 experimen 1 Anthranilate ii					
	ОК	Cancel More Info Help				

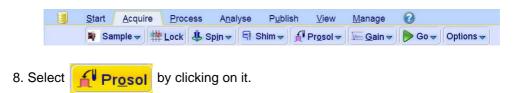


The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure above. Click on the down arrow button to browse for a specific directory.

- 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:

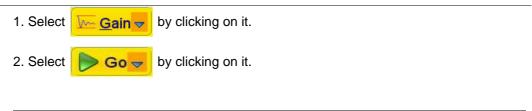


## **1-D Carbon Experiments**



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

#### 7.6.3 Acquisition



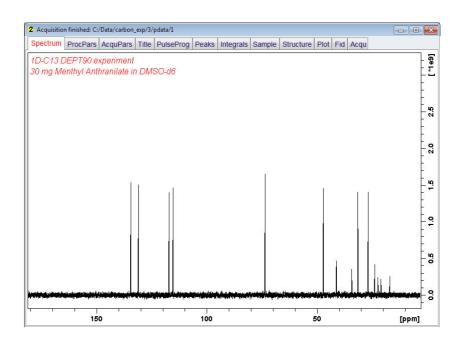
The experiment will take ~4 minutes, using 64 scans.

#### 7.6.4 Processing

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



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 ipp.
- 3. Type the following F1 [ppm] values:

From = **180** To = **0** 

	the exact coordinate
of the desired	expansion.
	[ppm
From	180
То	o

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

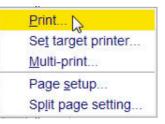


1 MA 10 1 C:\Data				v		
Spectrum ProcPars AcquPars	Title PulseProg	Peaks Integrals	Sample	Structure Plo	t Fid Acqu	
<b>е</b> ю						
Layout:						
+/1D_X.xwp						
Print:						
Default Printer Paper: Letter						BRUKER
View:						ондуна: Бигл Раглантоно илан. на влано 10 релоно 1 релоно 1 релоно 1 релоно 1 релоно 1
Limits: 🖑 🖳 🙀						ране
Display: Display:				Î î	Ĩ,	DO         4           ottm         3441.40.62 мл           #70000000         0.375352 мл           No         1.195.132 мл           Kor         1.295.132           Dr         20.420           Dr         20.420           Dr         20.420           Dr         20.420           Dr         20.420           Dr         20.000000           Dr         2.00000000
Click here to insert new elements:						141.000000           11           11           12           13           14           14           14           15           14           14           14           14           14           14           141           142           143           144           144           145           145           146           147           148           149           141           141           141           141           141           141           141           141           141           141           141           141           141           141           141           141           142           141           141
Standard NMR						eroi 71,4575/01 mm eroi 73,4575/01 mm eroi 9,10 user 91 9,10 user 92 19,00 user 911 10.0000000 m
						Prei 10.00000000 0 prei 200.1312007 mm
	1000 s.0 3 3 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	ng panji kulipang salah kana matang Manji kani <sup>kulan</sup> pang pani na matanin.	un de la fastela de la des num de la fastela de la deservición de la fastela de la fastela de la fastela de la f	a san an  and date in particular and date in the particular	*********************************	
						07 32745 07 73,4303210 1861 1007 888
		140 130 120 110 1		70 60 50 4		pm 2.00 mm pm 2.00 mm pm 2.00 1.40



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.



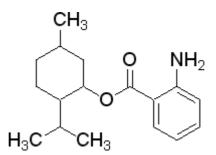
8. Select 'Print' by clicking in it.

## 7.7 Observations

# 8 2-D Heteronuclear Experiments

#### 8.1 Sample

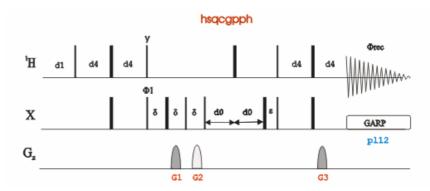
A sample of **30mg Menthyl Anthranilate in DMSO-d6** is used for all experiments in this chapter.



### 8.2 2-D HSQC Experiment

#### 8.2.1 Introduction

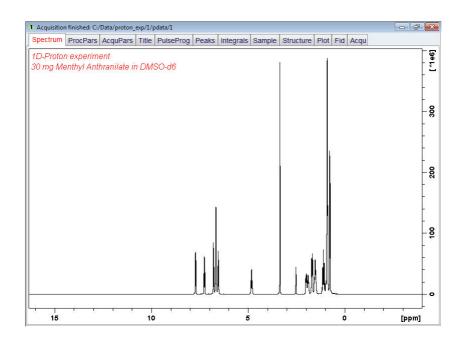
The **ge-2D HSQC experiment** is the gradient-enhanced version of the conventional HSQC experiment in which coherence selection is achieved by means of PFG. Thus, clean 2D HSQC spectra can be recorded in a single scan per  $t_1$  increment without need for phase cycle when sample concentration is high. 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 <sup>1</sup>H-X pairs via the large <sup>1</sup>J<sub>HX</sub> 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.



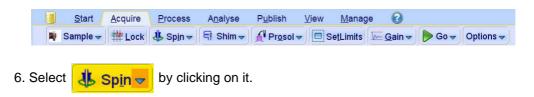
3. Enter the following information in to the 'New' window.

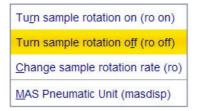
🖕 New	
Prepare for a new experiment b initializing its NMR parameters a For multi-receiver experiments Please define the number of re	according to the selected experiment type. several datasets are created.
NAME	inverse_exp
EXPNO	1
PROCNO	1
O Use current parameters	
Experiment HSQCGPPH	Select
<ul> <li>Options</li> </ul>	
Set solvent:	DMSO -
Execute "getprosol"	
Keep parameters:	P 1, O1, PLW 1 - Change
DIR	C:\Data 👻
🖾 Show new dataset in n	ew window
Receivers (1,2,16)	1
	SQC experiment Anthranilate in DMSO-d6
	OK Cancel More Info Help



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.



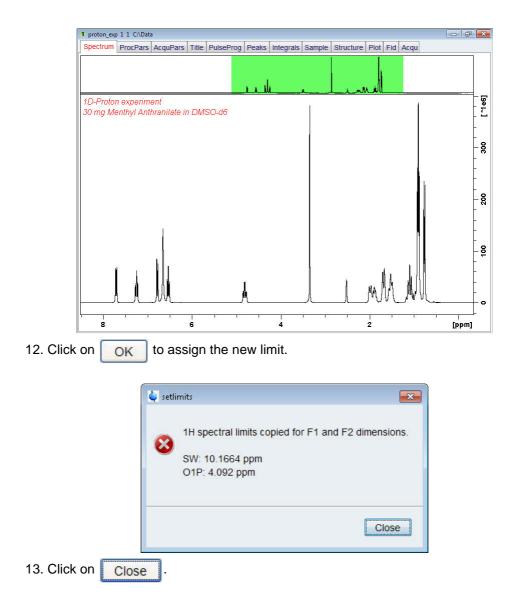


7. Select 'ro off' by clicking on it.

i	2-D experiments should always be run wit	hout rotation.
	8. Select <b>I Prosol</b> by clicking on it.	
i	This will load the pulse width and power le	evels in to the parameter set.
	9. Select <b>C SetLimits</b> by clicking on it	
	setlimits	
	Close this dialog box after 1. Open 1D dataset from 2. Zoom into region of in 3. Click OK to set freque	n Browser.
		OK Cancel

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.

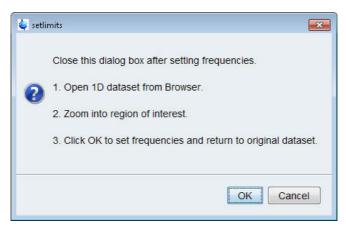
11. Expand the spectrum to display all peaks, leaving ca. **0.2 ppm** of baseline on either side of the spectrum.



j

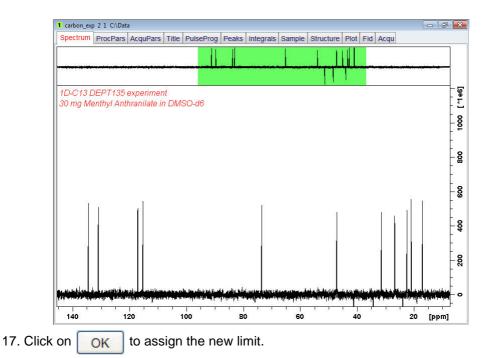
The display changes back to the 2D data set. The parameter set **HSQCGPPH** has a fixed F1 sweep width of 165 ppm and it is big enough to cover the protonated resonances for a broad range of samples. If desired, changes to the F1 sweep width can be done by using the '**Set\_limits**' button for a second 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.

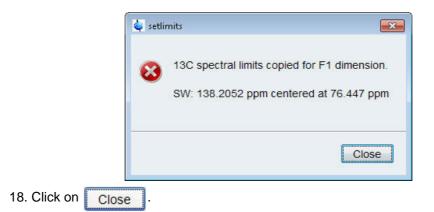
14. Select SetLimits by clicking on it.



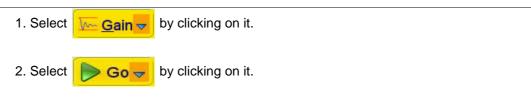
15. To open the 1D C13DEPT spectrum, right click on the dataset name in the browser window (e.g. **Carbon\_exp 2**) and select 'Display' or click and hold the left mouse button for dragging the 1D C13DEPT dataset in to the spectrum window.

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





#### 8.2.4 Acquisition





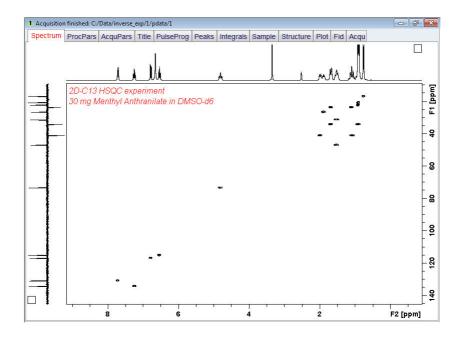
The experiment will take ~15 Minutes, using the default number of 2 scans and 256 increments.

#### 8.2.5 Processing

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



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.



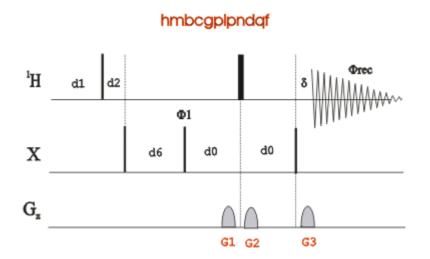
The F1 projection by default does not appear if the plot editor (XWINPLOT) is used to plot the spectrum. To add the F1 projection one has to create a new template.

## 8.3 Observations

#### 8.4 2-D HMBC Experiment

#### 8.4.1 Introduction

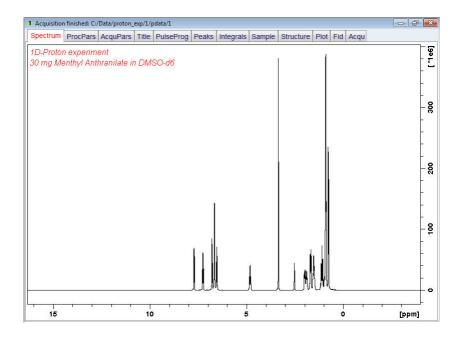
HMBC (Heteronuclear Multiple Bond Correlation) spectroscopy is a modified version of HMQC suitable for determining long-range 1H-13C connectivity. Since it is a long-range chemical shift correlation experiment the pulse program contains a low pass filter to suppress the one bond correlation and is a 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.

	<u>S</u> tart	<u>A</u> cquire	Process	/	P <u>u</u> blish	<u>V</u> iew	<u>M</u> anage	0	
		C <u>r</u> eate	e Dataset	📕 F <u>i</u> nd Datase	t 🖄 Ope	n <u>D</u> ataset	Paste	Dataset	Read Pars.
2. Selec	ct 🚺	Create	Dataset	by clicking	g on it.				

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

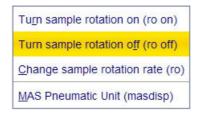
🖕 New	
initializing its NMR parameter	t by creating a new data set and s according to the selected experiment type. ts several datasets are created. receivers in the Options.
NAME	inverse_exp
EXPNO	2
PROCNO	1
O Use current parameters	
Experiment HMBCGP	Select
<ul> <li>Options</li> </ul>	
Set solvent:	DMSO -
Execute "getprosol"	
Keep parameters:	P 1, O1, PLW 1 - Change
DIR	C:\Data
🔲 Show new dataset in	new window
Receivers (1,2,16)	1
	HMBC experiment I Anthranilate in DMSO-d6
	OK Cancel More Info Help



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.





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

i	2-D experiments should always be run without rotation.
	8. Select <b>Prosol</b> by clicking on it.
i	This will load the pulse width and power levels in to the parameter set.
	9. Select SetLimits by clicking on it.
	🧔 setlimits
	<ul> <li>Close this dialog box after setting frequencies.</li> <li>1. Open 1D dataset from Browser.</li> <li>2. Zoom into region of interest.</li> <li>3. Click OK to set frequencies and return to original dataset.</li> </ul>
	OK Cancel

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.

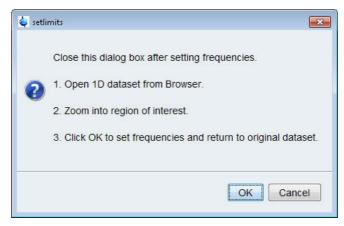
11. Expand the spectrum to display all peaks, leaving ca. **0.2 ppm** of baseline on either side of the spectrum.

1 proton_exp 1 1 C:\Data	- 6 🔀
Spectrum ProcPars AcquPars Title PulseProg Peaks Integrals Sample Structure Plot Fid Acqu	
the second second second second second second second second second second second second second second second se	
1D-Proton experiment 30 mg Menthyl Anthranilate in DMSO-d6	[ *1e6]
	- 300 - -
	- - - - -
	- - - - - - -
	[ppm]
12. Click on <b>OK</b> to assign the new limit.	
🧔 setlimits	
1H spectral limits copied for F1 and F2 dimensions. SW: 10.1664 ppm O1P: 4.092 ppm	
Close	
13. Click on Close .	

i

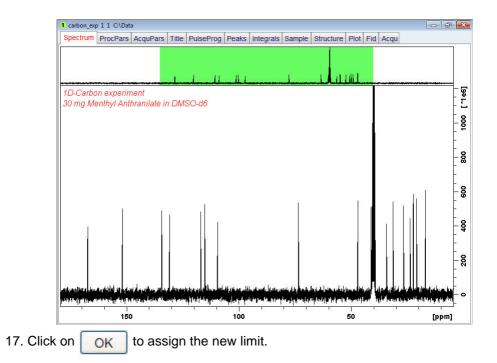
The display changes back to the 2D data set. The parameter set **HMBCGP** has a fixed F1 sweep width of 222 ppm and it is big enough to cover all Carbon resonances for a broad range of samples. If desired, changes to the F1 sweep width can be done by using the '**Set\_limits**' button for a second time. 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.

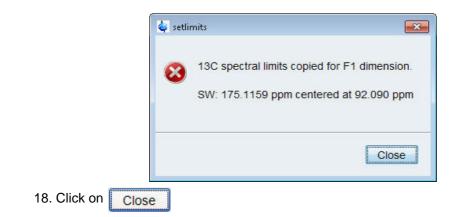
14. Select **SetLimits** by clicking on it.



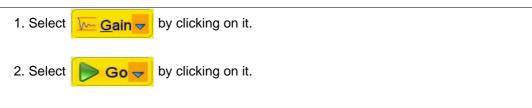
15. To open the 1D C13DEPT spectrum, right click on the dataset name in the browser window (e.g. **Carbon\_exp 1**) and select 'Display' or click and hold the left mouse button for dragging the 1D C13DEPT dataset in to the spectrum window.

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





#### 8.4.4 Acquisition





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

#### 8.4.5 Processing

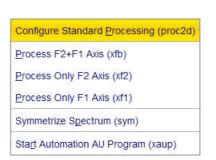
1. Click on the 'Process' tab in the TopSpin Menu bar.





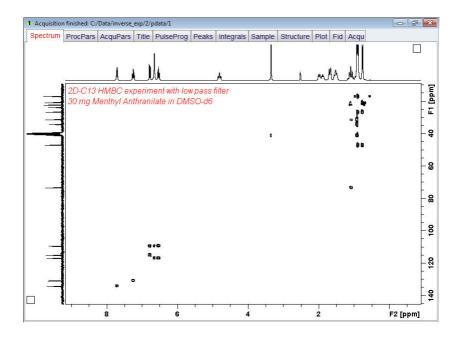
This executes a standard processing program **proc2**. The message shown in the figure above, pops up in case of a magnitude 2D experiment and the apk2d option is enabled. To configure the processing program follow the steps below.

3. Click on the down arrow inside the  $\frac{1}{\sqrt{1-1}}$  Pro<u>c</u>. Spectrum  $rac{1}{\sqrt{1-1}}$  button.



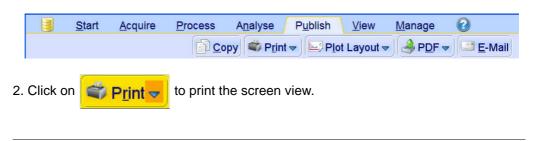
4. Select 'Configure Standard Processing' by clicking on it.

ress 'Execute' to process the current da ress 'Save' to just change the processi hanged options will be effective when p ne-click 'Proc. Spectrum' button.	ng oj	otions.	
Fourier Transform (xfb)			
Auto - Phasing (apk2d)			
Auto - Baseline Correction [F2] (abs2)	<b>V</b>		
Auto - Baseline Correction [F1] (abs1)			
Plot (autoplot)		LAYOUT = +/:	2D_inv.xwp
Warn if processed data exist			



#### 8.4.6 Plotting

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



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<sup>0</sup> 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 CDCI3

#### 9.2.1 Parameter Setup

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



2. Select Create Dataset by clicking on it.

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

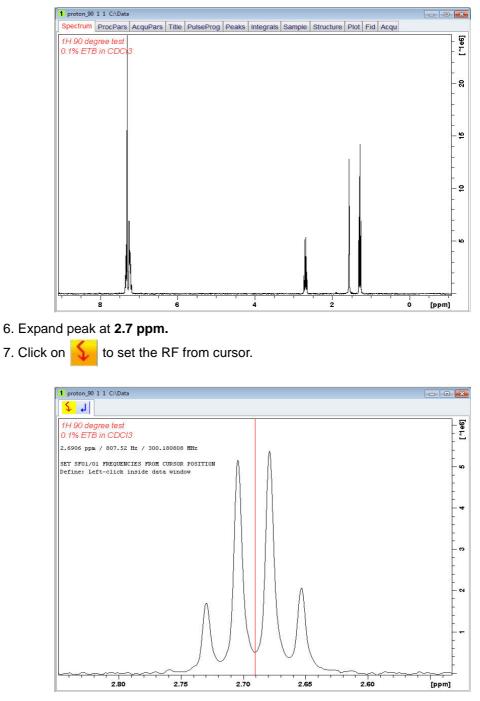
e New	of Chinese Par	Carden St. Parce 14	×
Prepare for a new experiment initializing its NMR parameters For multi-receiver experiments Please define the number of r	according to t	he selected experiment t sets are created.	ype.
NAME	proton_90		
EXPNO	1		
PROCNO	1		
O Use current parameters			
Experiment PROTON			Select
<ul> <li>Options</li> </ul>			
Set solvent:		DMSO -	
Execute "getprosol"			
⊘ Keep parameters:		P 1, 01, PLW 1 👻	Change
DIR		C:\Data	-
Show new dataset in	new window		
Receivers (1,2,16)		1	
	se test for Prof Izene in CDCl3		
	ОК	Cancel More In	fo Help



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 **CDCI3** as a lock solvent.



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

## **Determination Of 90 Degree Pulses**

01/02/03 Define SFO1/O1 1	frequencies
SFO1 [MHz] =	300.180808
O1/2/3 [Hz] =	807.52

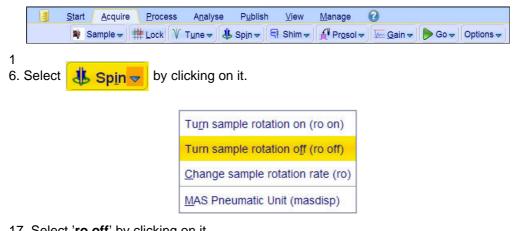
- 10. Click on O1
- 11. Select the 'AcquPars' tab by clicking on it.
- 12. Make the following changes:

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.



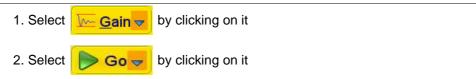
17. Select 'ro off' by clicking on it.



This test should be run without rotation.

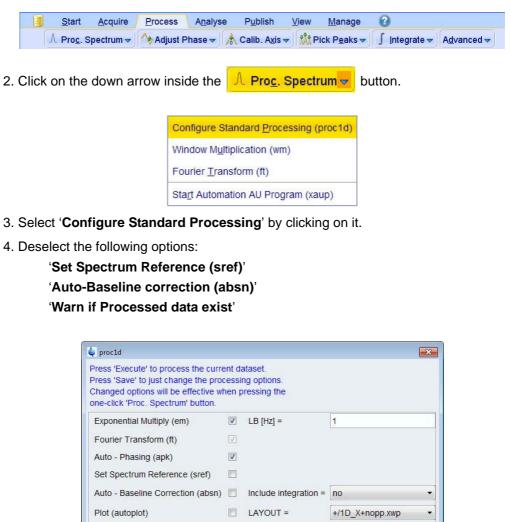
## **Determination Of 90 Degree Pulses**

#### 9.2.2 Acquisition



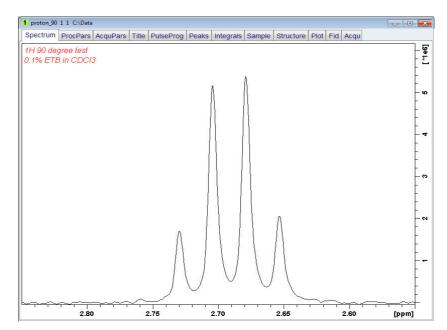
#### 9.2.3 Processing

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



Exponential Multiply (em)	V	LB [Hz] =	1
Fourier Transform (ft)	$\checkmark$		
Auto - Phasing (apk)	V		
Set Spectrum Reference (sref)			
Auto - Baseline Correction (absn	) 🔳	Include integration =	no
Plot (autoplot)		LAYOUT =	+/1D_X+nopp.xw
Warn if processed data exist			

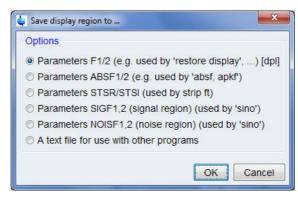
- 5. Click on Execute .
- 6. Expand the spectrum form 2.85 ppm to 2.55 ppm.



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

то	ggle Spectrum O <u>v</u> erview
<u>S</u> h	ow Full Spectrum
То	ggle Parameter <u>W</u> indow
Sp	e <u>c</u> tra Display Preferences
Sa	ve Display Regi <u>o</u> n To
Re	store Display Region From Params. F1/2
Se	t Plot Height At Specific Cursor Position
Da	taset Properties
<u>E</u> ile	es
Exp	blorer

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.

## **Determination Of 90 Degree Pulses**

	= proton_90 1 1 C:\Data
	ired file types of the source data set
<ol><li>Press OK to co</li></ol>	opy them to the destination parameter set.
acqu	
proc	
outd	
title	
anc	
Destination Dir =	C:\Bruker\TopSpin3.1.b.2\exp\stan\nmr\par\user
Destination Dir =	C:\Bruker\TopSpin3.1.b.2\exp\stan\nmr\par\user
Destination Dir =	C:\Bruker\TopSpin3.1.b.2\exp\stan\nmr\par\user

12. Select all parameter options.

13. Click on OK

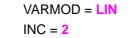
#### 9.2.4 Determine The 90 Degree Pulse

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

<u>Start</u> <u>A</u> cquire <u>Process</u>	A <u>n</u> alyse P <u>u</u> blish <u>V</u> iew	<u>M</u> anage	0		
💐 Sample 🗢 🗰 Lock V	T <u>u</u> ne ▼	¶ Pr <u>o</u> sol <del>▼</del>	<u> </u>	Þ Go 🗢	Options 🗢
2. Click on the down arrow in	side the 📄 Go 🚽	button.			
	Č				
	Transfer Fid To Disk (tr)				
	Estimate Exp. Time (expt)				
	Real-Time <u>G</u> o Setup (gs)				
	Optimize Acquisition Params	(popt)			
	Start Automation AU program	n (xaua)			

- 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

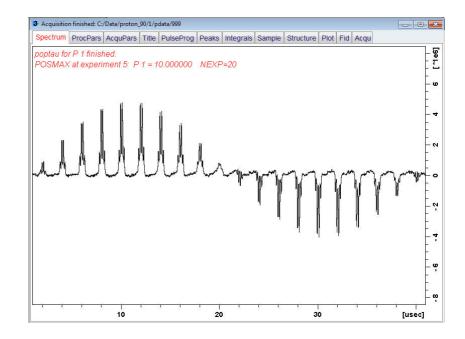


In the AU program specified in AUNM will be executed WDW= EM Perform automatic baseline correction (ABSF) PH_mod= pk Overwrite existing files (disable confirmation Message) FT_mod= fsc Otop sample spinning at the end of optimization (mash) Run optimization in background OPTIMIZE GROUP PARAMET. OPTIMUM STARTVAL ENDVAL NEXP VARMOD INC Step by step p1 POSMAX 2 20 LIN 2 Start optimize Skip current opt. Show proto Add param Restore Save Read array. Save array file Stop optimil Delete par Display Dat. Update Pro Help Actick on Save . Click on Start optimizer has been updated Click on Start optimize .		🗐 store as 2D d	C:\Data lata (ser file	e)						
Perform automatic baseline correction (ABSF) PH_mod=pk Overwrite existing files (disable confirmation Message) FT_mod=fsc Stop sample spinning at the end of optimization (mash) Run optimization in background OPTIMIZE GROUP PARAMET. OPTIMUM STARTVAL ENDVAL NEXP VARMOD NC Step by step pt POSMAX 2 20 LIN 2 LIN 2 Step by step pt POSMAX 2 20 LIN 2 Read array. Save array file Stop optimi Delete par Display Dat Update Pro Help he ENDVAL parameter has been updated.					vill be executed		WDW= EM			
Overwrite existing files (disable confirmation Message)       FT_mod=fsc         Stop sample spinning at the end of optimization (mash)         Run optimization in background         OPTIMIZE       GROUP PARAMET OPTIMUM         Step by step       p1         POSMAX       2         Start optimize       Skip current opt         Add param       Restore         Save array file       Stop optimi         Delete par       Display Dat         Update Pro       Heip										
Stop sample spinning at the end of optimization (mash)         Run optimization in background         OPTIMIZE       GROUP PARAMET. OPTIMUM STARTVAL ENDVAL NEXP VARMOD NC         Step by step       p1         POSMAX       2         Start optimize       Skip current opt.         Start optimize       Skip current opt.         Start optimize       Skip current opt.         Start optimize       Stop optimi.         Delete par.       Display Dat         Update Pro       Heip						je)				
OPTIMIZE       GROUP       PARAMET       OPTIMUM       STARTVAL       ENDVAL       NEXP       VARMOD       NC         Step by step       p1       POSMAX       2       20       LN       2         Step by step       p1       POSMAX       2       20       LN       2         Step by step       p1       POSMAX       2       20       LN       2         Start optimize       Skip current opt       Show proto       Add param       Restore       Save       Read array.         Save array file       Stop optimi       Delete par       Display Dat       Update Pro       Help										
Step by step       p1       POSMAX       2       20       LN       2         Start optimize       Skip current opt       Show proto       Add param       Restore       Save       Read array.         Start optimize       Skip current opt       Display Dat       Update Pro       Help         Click on       Save       .         he       ENDVAL parameter has been updated.										
Start optimize_Skip current optShow proto Add param Restore Save Read array. Save array file Stop optimi Delete par Display Dat Update Pro Help Click on Save		OPTIMIZE	GROUP	PARAMET.	OPTIMUM		ENDVAL		VARMOD	
Save array fileStop optimiDelete parDisplay DatUpdate ProHelp Click on Save . ne ENDVAL parameter has been updated.		Step by step		p1	POSMAX	2		20	LIN	2
Save array file Stop optimi Delete par Display Dat Update Pro Help										
Click on Save . e ENDVAL parameter has been updated.						1				Read array.
ne ENDVAL parameter has been updated.		Save array file .	Stop c	optimi	Delete par	Display Dat.	. Update	Pro	Help	
	. Click c	on 🦳	Save		•					
	he END	VAL para	amete	r has b	een upd	lated.				
	ne END	VAL para	amete	r has b	een upd	lated.				
🧅 poptau	e END	VAL para	amete rt optir	r has b mize	een upd	lated.			x	
	e END	VAL para	amete rt optin	r has b mize						
Number of experiments: 20	e END	VAL para	amete rt optin	r has b mize	experimen	its: 20			×.	
Number of experiments: 20 total experiment time will be: 10 min 40 sec	e END	VAL para	amete	r has b mize poptau mber of c al experin	experimen ment time	its: 20	min 40 s	sec		
Number of experiments: 20	e END	VAL para	amete	r has b mize poptau mber of c al experin	experimen ment time	its: 20	min 40 s	sec		
Number of experiments: 20 total experiment time will be: 10 min 40 sec Continue ? [y   n]	e END	VAL para	amete t optin	r has b mize poptau mber of c al experin	experimen ment time	its: 20	min 40 s	sec	×	
Number of experiments: 20 total experiment time will be: 10 min 40 sec	e END	VAL para	amete t optin	r has b mize poptau mber of c al experin	experimen ment time	its: 20	min 40 s	sec		
Number of experiments: 20 total experiment time will be: 10 min 40 sec Continue ? [y   n]	ne END	VAL para	amete t optin	r has b mize poptau mber of c al experin	experimen ment time	its: 20	min 40 s	sec		
Number of experiments: 20 total experiment time will be: 10 min 40 sec Continue ? [y   n]	ne END	VAL para	amete t optin	r has b mize poptau mber of c al experin	experimen ment time	its: 20				

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.



i

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<sup>0</sup> 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 Create Dataset by clicking on it.
- 3. Enter the following information in to the 'New' window.

🖕 New	States of the	a come to	case 6 Pace	×
initializing its For multi-red	a new experiment l NMR parameters ceiver experiments ne the number of re	according to t several datas	he selected experiment ets are created.	t type.
NAME		carbon_90	p	
EXPNO		1		
PROCNO		1		
O Use curre	ent parameters			
Experime	nt C13CPD			Select
Option:	s			
☑ Set	solvent:		C6D6 •	
© Exe	cute "getprosol"			
© Kee	ep parameters:		P 1, 01, PLW 1 👻	Change
DIR			C:\Data	-
🗖 Sho	ow new dataset in r	new window		
Recei	ivers (1,2,16)		1	
	90 degree puls	e test for 130		
TITLE	ASTM (60% C6			
		ОК	Cancel More	Info Help



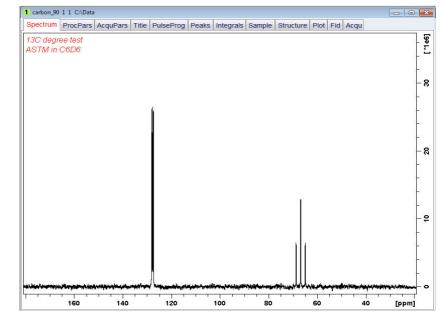
The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure above. Click on the down arrow button to browse for a specific directory.

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:

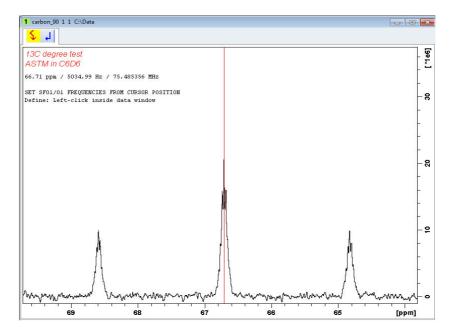
PULPROG = **zg** DS = **0** NS = **1** 

6. Continue with **7.2.4 Processing**.



7. Expand peak at 67 ppm.

8. Click on **5** to set the RF from cursor.



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

🧅 01/02/03	
Define SF01/01 f	requencies
SFO1 [MHz] =	75.485356
O1/2/3 [Hz] =	5034.99
01 02	O3 Cancel

- 10. Click on O1
- 11. Select the 'AcquPars' tab by clicking on it.
- 12. Make the following changes:

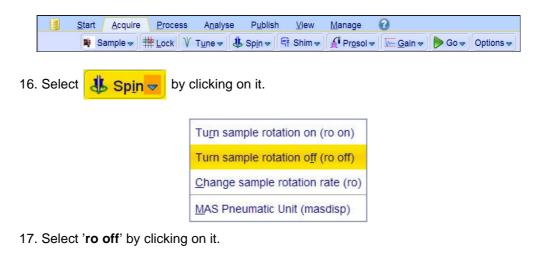
TD = 8912 SW [Hz] =40 D1 [sec] = 60 DS = 0 NS = 1

- 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.

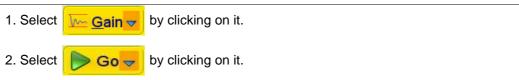
### **Determination Of 90 Degree Pulses**





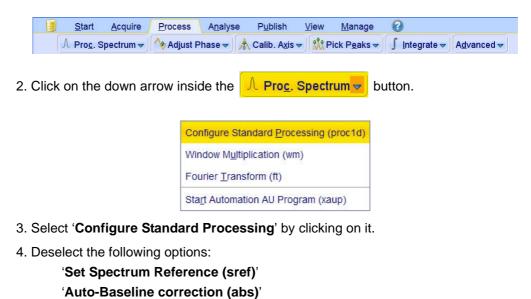
This test should be run without rotation.

#### 9.4.2 Acquisition



#### 9.4.3 Processing

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

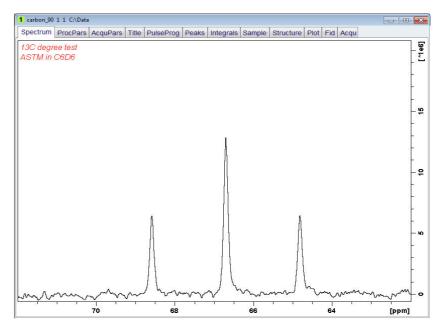


Auto-Baseline correction (a

'Warn if Processed data exist'

Press 'Execute' to process the curre Press 'Save' to just change the prod Changed options will be effective will one-click 'Proc. Spectrum' button.	essi	ng options.	
Exponential Multiply (em)	V	LB [Hz] =	3.5
Fourier Transform (ft)	<b>V</b>		
Auto - Phasing (apk)			
Set Spectrum Reference (sref)			
Auto - Baseline Correction (absn)		Include integration =	no
Plot (autoplot)		LAYOUT =	+/1D_X.xwp
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.

Тод	gle Spectrum O <u>v</u> erview
<u>S</u> ho	w Full Spectrum
Тод	gle Parameter <u>W</u> indow
Spe	ctra Display Preferences
Sav	e Display Regi <u>o</u> n To
Res	tore Display Region From Params. F1/2
Set	Plot Height At Specific Cursor Position
D <u>a</u> ta	aset Properties
<u>File</u>	5
Expl	orer

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

(	Options
0	Parameters F1/2 (e.g. used by 'restore display',) [dpl
0	Parameters ABSF1/2 (e.g. used by 'absf, apkf')
0	Parameters STSR/STSI (used by strip ft)
0	Parameters SIGF1,2 (signal region) (used by 'sino')
0	Parameters NOISF1,2 (noise region) (used by 'sino')
0	A text file for use with other programs

9. Enable 'Parameters F1/2'.

10. Click on OK

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

	= carbon_90 1 1 C:\Data
	ired file types of the source data set
2) Press OK to co	opy them to the destination parameter set.
acqu	
proc	
outd	
title	
Destination Dir =	C:\Bruker\TopSpin3.1.b.2\exp\stan\nmr\par\us
Destination Dir =	C:\Bruker\TopSpin3.1.b.2\exp\stan\nmr\par\us
Destination Dir =	C:\Bruker\TopSpin3.1.b.2\exp\stan\nmr\par\us

- 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.

```
      Start
      Acquire
      Process
      Analyse
      Publish
      View
      Manage
      Manage

      Image: Sample =
      Image: Sample =
```

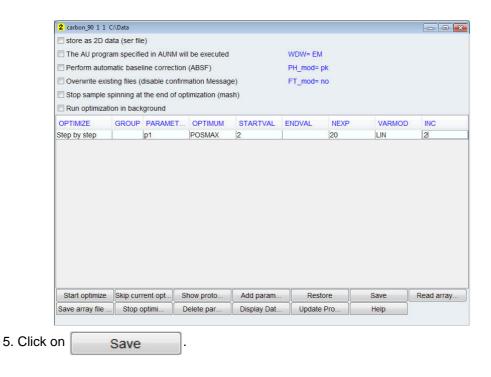
button.

2. Click on the down arrow inside the So-



- 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

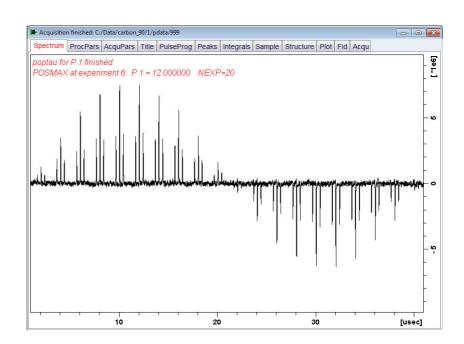


# **Determination Of 90 Degree Pulses**

i	The ENDVAL parameter has been updated.			
	6. Click on Start optimize			
	🖕 poptau			
	Number of experiments: 20 total experiment time will be: 20 min 20 sec Continue ? [y   n]			
	7. Enter <b>y</b> 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<sup>0</sup> 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

# **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<sup>0</sup> 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 CDCI3

#### 10.2.1 Experiment Setup

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



2. Select Create Dataset by clicking on it.

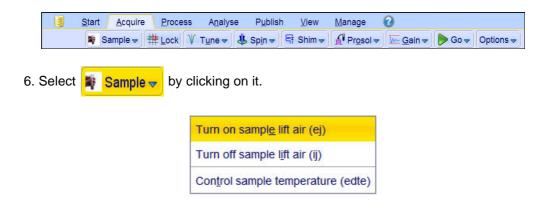
3. Enter the following information in to the 'New' window.

👹 New	
Prepare for a new experiment b initializing its NMR parameters a For multi-receiver experiments Please define the number of rec	according to the selected experiment type. several datasets are created.
NAME	proton_sensitivity
EXPNO	1
PROCNO	1
O Use current parameters	
Experiment PROSENS	Select
<ul> <li>Options</li> </ul>	
Set solvent:	CDCI3 -
Execute "getprosol"	··*=
Keep parameters:	P 1, O1, PLW 1  Change
DIR	C:\Data 👻
🖾 Show new dataset in ne	ew window
Receivers (1,2,16)	1
Proton sensitivit 0.1% Ethylbenz	
	OK Cancel More Info Help



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.

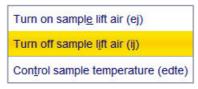


7. Select 'ej' by clicking on it.

•
ň.

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.



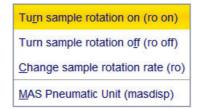
Wait till the sample is lowered down in to the probe and the lift air is turned off. A clicking sound may be heard.

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

Solvent	Description
Acetone	acetone-d6
C6D6	benzene-d6
C6D6+Dioxane	ASTM Sample
CD2Cl2	methylenechloride-d2
CD3CN	acetonitrile-d3
CDCI3	chloroform-d
D2O	deuteriumoxide
DEE	diethylether-d10
Dioxane	dioxane-d8
DME	dimethylether-d6
DMSO	dimethylsulfoxide-d6
EtOD	ethanol-d6
H2O+D2O	90%H2O and 10%D2O
MeOD	methanol-d4
Tol	toluene-d8

12. Select 'CDCI3' by clicking on it.



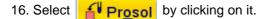


14. Select 'ro on' by clicking on it.



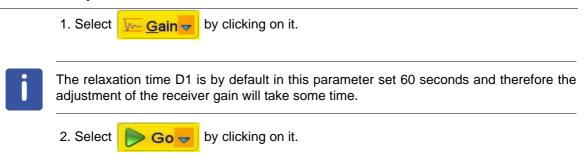
i

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



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

#### 10.2.2 Acquisition



#### 10.2.3 Processing

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

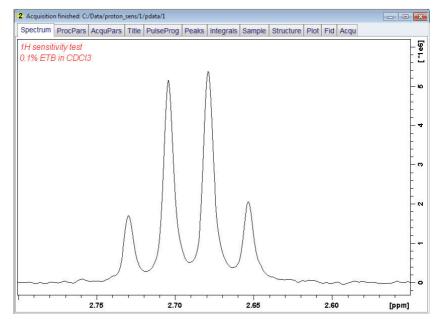




This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**'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

🤹 sinocal	×
Enter left limit of sign	al range in ppm :
3	
	OK Cancel

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

🧅 sinocal	X
Enter right limit of signal range in ppm :	
2	
	OK Cancel
	OK

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

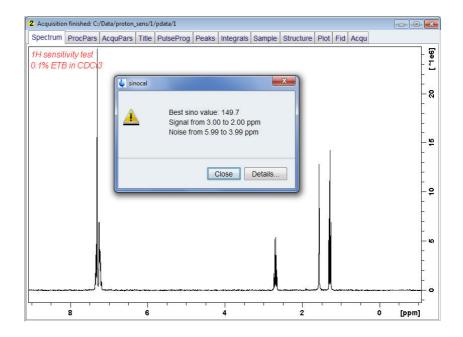
5. Click on OK	].	Ū	
	🤹 sinocal		
	Enter left limit of no	bise range in pp	.m.:
			OK Cancel
6. Enter 7 for the le	eft limit of the nois	e range.	
7. Click on OK	].		

iinocal	
Enter right limit o	f noise range in ppm :
2.8	
	OK Canc

Enter 2.8 for the right limit of the noise range.
. Click on OK.
🤹 sinocal
Enter noise width in ppm :
2
OK Cancel
0 Enter 2 for the noise width

e wiath.





## 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.



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

🤹 New		Canal & Parce 1		X
Prepare for a new experim initializing its NMR parame For multi-receiver experim Please define the number	ters according to ents several data	the selected experiment sets are created.	type.	
NAME	carbon_se	ns_astm		
EXPNO	1			_
PROCNO	1			
O Use current parameters	i			
Experiment C13CPD	~		Select	
<ul> <li>Options</li> </ul>				
Set solvent:		C6D6 •		
Execute "getprose	ol"			
Keep parameters:		P 1, 01, PLW 1 👻	Change	
DIR C:\Data				-
Show new datase	t in new window			
Receivers (1,2,16	5)	1		
	ivity test no 1H de % C6D6/40% Diox			
	ОК	Cancel More	Info Hel	p

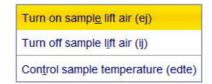


The directory (DIR) is specific to how the data are stored and therefore may show different entries as the one in the figure above. Click on the down arrow button to browse for a specific directory.

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



6. Select 🙀 Sample 🗸 by clicking on it.

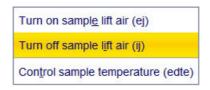


7. Select 'ej' by clicking on it.



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.

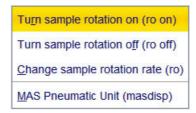


Wait till the sample is lowered down in to the probe and the lift air is turned off. A clicking sound may be heard.

11. Select <u>Lock</u> by clicking on it.

A Solvent	Description		
Acetone	acetone-d6		
C6D6	benzene-d6		
C6D6+Dioxane	ASTM Sample		
CD2Cl2	methylenechloride-d2		
CD3CN	acetonitrile-d3		
CDCI3	chloroform-d		
D2O	deuteriumoxide		
DEE	diethylether-d10		
Dioxane	dioxane-d8		
DME	dimethylether-d6		
DMSO	dimethylsulfoxide-d6		
EtOD	ethanol-d6		
H2O+D2O	90%H2O and 10%D2O		
MeOD	methanol-d4		
Tol	toluene-d8		

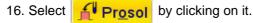
- 12. Select 'C6D6' by clicking on it.
- 13. Select **U** Spin → by clicking on it.



- 14. Select 'ro on' by clicking on it.
- 15. Select 🤤 Shim 🚽 by clicking on it.



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



Thi

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

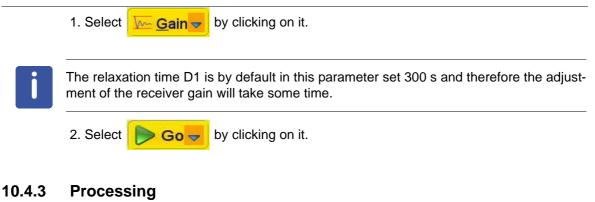
- DS = 0 NS = 1 O1 [ppm] = 100
- 19. Select the 'ProcPars' tab by clicking on it.
- 20. Make the following changes:

SI = 32768

LB [Hz] = 3.5

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

#### 10.4.2 Acquisition



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

	<u>S</u> tart	<u>A</u> cquire	Process	A <u>n</u> alyse	P <u>u</u> blish	<u>V</u> iew	Manage	0	
Λ	Pro <u>c</u> . S	pectrum <del>v</del>	Adjust P	hase 🗢	Å Calib. A <u>x</u> is <del>⊽</del>	N P	ick P <u>e</u> aks <del>√</del>	∫ Integrate <del>→</del>	A <u>d</u> vanced <del>▼</del>

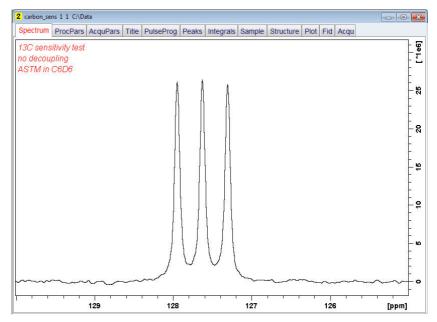




This executes a processing program including commands such as an exponential window function '**em**', Fourier transformation '**ft**', an automatic phase correction '**apk**' and a baseline correction '**abs**'. Other options are available by clicking on the down arrow inside the '**Proc. Spectrum**'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

Enter left limit of sigr	nal range in ppm :
130	
	OK Cancel

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

3. Click on O	< .
	🤹 sinocal
	Enter right limit of signal range in ppm : 125
	OK Cancel
4. Enter 127 for	the right limit of the signal range.

5. Click on OK

🖕 sinocal	×
Enter left limit of nois	e range in ppm :
120	
	OK Cancel

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

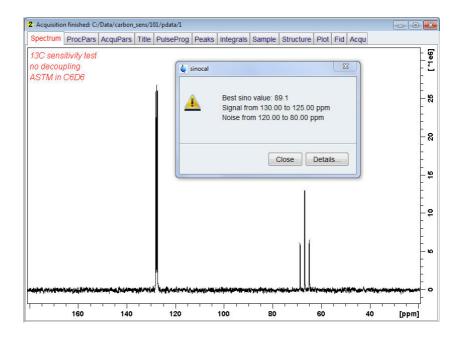
7. Click on OK	].
ĺ	🤹 sinocal
	Enter right limit of noise range in ppm :
	80
	OK Cancel

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

9. Click on OK	].	
	🤹 sinocal	X
	Enter noise width in ppm :	
		OK Cancel

10. Enter 40 for the noise width.

11. Click on OK



# 10.5 Observations

# **11** Spectrometer Configuration

### 11.1 Hardware Configuration

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

🧾 <u>S</u> tart	<u>A</u> cquire	<u>P</u> rocess A <u>n</u> aly	se P <u>u</u> blis	h <u>∨</u> iew ∫	Manage	0
	Pr <u>e</u> ferences	Spectr <u>o</u> meter <del>▼</del>	Securit <u>y</u> <del>⊸</del>	Commands 🗟	Remote	

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

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

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

	Ŋ:		
onfigure the hardware of you	r spectrometer or create a cor	nfiguration for a datast	ation.
ctive configuration: "FOURIER	R_300"		
Available spectrometer config	purations		
Configuration	Spectrometer type	Frequency [MHz]	Туре
Bruker_default_av500	Avance-AV 500	500.13	Datastation
Bruker_default_avII700	Avance-AV 700	700.13	Datastation
Bruker_default_aviii600	Avance III 600	600.13 300.18	Datastation Spectromete
FOURIER 300			
	DreamSpec 300		
	ifgurations or create a new sp	ectrometer configuratio	
slect one of the available cor	ifigurations or create a new sp n existing configuration (e.g. if	you want to add new h	on!
elect one of the available con ress "Edit" to modify or use a if you want to use the config	ifigurations or create a new sp n existing configuration (e.g. it uration from a previous TogS)	you want to add new h	on!
elect one of the available cor ress "Edit" to modify or use a if you want to use the config	ifigurations or create a new sp n existing configuration (e.g. it uration from a previous TopS) sectrometer configuration from	you want to add new h	on!
elect one of the available con ress "Edit" to modify or use a if you want to use the config ress "Never" to create a new sy ress "Detect" to detect the se	ifigurations or create a new sp n existing configuration (e.g. it uration from a previous TopS) sectrometer configuration from	you want to add new h pin version). I scratch.	on! tardware
elect one of the available con ress "Edit" to modify or use a if you want to use the config ress "Never" to create a new sy ress "Detect" to detect the se	Ifgurations or create a new sp n existing configuration (e.g. if uration from a previous TopS) sectrometer configuration from sected configuration.	you want to add new h pin version). I scratch.	on! tardware

# **Spectrometer Configuration**

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

7. Click or	Edit	.
-------------	------	---

( T			
CF CF			
Edit configuration:			
Configuration			
Configuration name	FOURIER_300		
Spectrometer	٠		
Datastation	0		
1H frequency of magnet [MHz]	300.18		
in nequency of magnet (mile)	300.18		
Debug			
Use debug module			
L		< Previ	ious Next > Cancel
· · · · · · · · · · · · · · · · · · ·			indus   indus   connect
<u>_</u>			
<u>4</u> a			
Cf Specify the channel to which extern	al devices are connecte	d:	
Specify the channel to which extern		d:	
Specify the channel to which extern Communication channels for extern	nal devices		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier	nal devices	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board	nal devices no no	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock	nal devices no no 149 236.99 254	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit	nal devices no 149 236 99 254 149 236 99 253	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar	nal devices no 149 236 99 254 149 236 99 253 no	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit	nal devices no 149 236 99 254 149 236 99 253 no	•	
Specify the channel to which extern Communication channels for exter HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2	nal devices no 149.236.99.254 149.236.99.253 no 00 no	•	
Specify the channel to which extern Communication channels for exter HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit	nal devices no 149.236.99.254 149.236.99.253 no no no no no		
Specify the channel to which extern Communication channels for exter HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer	nal devices no 149.236.99.254 149.236.99.253 no 0) no no no no		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer	nal devices no 149 236 99 254 149 236 99 253 no no no no no no no		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller	nal devices no 149 236 99 254 149 236 99 253 no no no no no no no no no no	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit Bruker Automatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Unit	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit Bruker Automatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Unit	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Uni Radio Frequency Supervisor	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no		
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Uni Radio Frequency Supervisor Lockswitch	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Unit Radio Frequency Supervisor Lockswitch	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Unit Radio Frequency Supervisor Lockswitch	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Unit Radio Frequency Supervisor Lockswitch	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no	•	
Specify the channel to which extern Communication channels for extern HPPR Preamplifier ACB Amplifier Control Board Minispec Lock VTU Variable Temperature Unit LC-NMR Software HyStar Gradient Temperature Unit (BCU-2 MAS Pneumatic Control Unit Bruker Automatic Changer Barcode Printer Cryo Controller HPCU High Power Control Unit Preemphasis/Gradient Unit Fast Gradient Supervisor Gradient Power Supply Control Unit Radio Frequency Supervisor Lockswitch	nal devices no 149.236.99.254 149.236.99.253 no no no no no no no no no no	•	

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

Additional configuration         Enable peak power check (POVVCHK);	Security configuration   Enable peak power check (POWCHK):     Enable peak power check (POWCHK): <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> <pre> </pre> Interver: check security parameters:      ext > 1  cancel   table> </th <th></th> <th>🤹 Cf</th> <th></th> <th></th>		🤹 Cf		
Enable peak power check (POWCHK):	Enable peak power check (POWCHK):         Enable peak power check (POWCHK):		Additional configu	ration:	
Enable peak power check (POWCHK):	Enable peak power check (POWCHK):         Enable peak power check (POWCHK):				
<pre>     revious rext &gt; Cancel.     rcontserver: check security parameters: </pre>	wile table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:         viciei table (delete or modify values):         You can also edit this table with the command edituic:				
hcontserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1		Enable peak pow	er check (POWCHK):	
hcontserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hcontserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hcontserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hconfserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hcontserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hcontserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hcontserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hconfserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hoonfserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hconfserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hconfserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hoonfserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hconfserver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
hconfeerver: check security parameters:	Incontserver: check security parameters:         ext >         ext >         •         uclei table (delete or modify values):         You can also edit this table with the command ednuc         acteus       Name       Receptivity (ref. 13C)       Spin       Frequency (ref. 142)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
	ext >       .         uclei table (delete or modify values):         You can also edit this table with the command ednuc         icteus       Name       Receptivity (rel. 13C)       Spin       Frequency (rel. 13C)         Hydrogen       6580.0       1/2       Deuterium       0.00621       1				
x on Next > .	uclei table (delete or modify values): You can also edit this table with the command ednuc ucleus Name Receptivity (rel. 13C) Spin Frequency (rel Hydrogen 6580.0 1/2 Deuterium 0.00621 1				< Previous Next > Cancel
<b>ξ</b> α	You can also edit this table with the command ednuc icleus Name Receptivity (rel. 13C) Spin Frequency (rel Hydrogen 5680.0 1/2 Deuterium 0.00821 1	Ne		security parameters:	< Previous Next > Cancel
Edit nuclei table (delete or modify values): Note: You can also edit this table with the command ednuc	Name         Receptivity (rel. 13C)         Spin         Frequency (rel           Hydrogen         5680.0         1/2           Deuterium         0.00821         1			security parameters:	< Previous Next > Cancel
	Hydrogen         5680.0         1/2           Deuterium         0.00821         1	CF Edit nuc	xt > .	y values):	< Previous Next > Cancel
1H Hydrogen 5680.0 1/2		Edit nucl	xt > .	y values): with the command ednuc	
	Carlon 1.0 1/2	Edit nuci Note: Yo	xt > . lei table (delete or modify vu can also edit this table cus Name Re	y values): with the command ednuc eceptivity (rel. 13C) Spin	
10 Carbon 1.0 1/2		Cf Edit nucl Note: Yo 1H 2H	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
		Edit nucl Note: Yo	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
		Cf Edit nucl Note: Yo 1H 2H	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
		Cf Edit nucl Note: Yo 1H 2H	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
		Cf Edit nucl Note: Yo 1H 2H	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
		Cf Edit nucl Note: Yo 1H 2H	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
		Cf Edit nucl Note: Yo 1H 2H	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
		Cr Edit nuc Note: Yo 1H 2H 13C	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	
< III III III III III IIII IIII IIII I	"	Cr Edit nuc Note: Yo 1H 2H 13C	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	Frequency
	" < Previous Next >	Cr Edit nuc Note: Yo 1H 2H 13C	iel table (delete or modif) su can also edit this table eus Name Re Hydrogen Deuterium	y values): with the command ednuc ecceptivity (rel. 13C) Spin 5680.0 1/2 0.00821 1	Frequency

	N INFORMATION
Path Date	
Date	: C:/Bruker/TopSpin3.1.b.2/conf/instr/FOURIER 300/uxnmr.info
Release	: Tue Oct 19 12:10:19 2010
	: TopSpin Acquisition Version alpha-pl0
Installed in	a : C:/Bruker/TopSpin3.1.b.2
Host	: APPS1
03	: Windows 7 (Vs 6.1)
CPU	: Intel(R) Xeon(R) CFU W3505 @ 2.53GHz (2 cores at 2533 MHz)
User	: nmrsu
Description	1
Location	1
System	: unknown
Order Number	
Configured i	<pre>in: C:/Bruker/TopSpin3.1.b.2/conf/instr/FOURIER_300</pre>
AcqControlSe	rver: DRMS DRU
- TCP/IP add	iress = 127.0.0.1
- Firmware V	fersion = 29259
Router: none	installed
Transmitters	at the spectrometer subnet:
Minianac Ber	olifier HMG01/215 ECL 200:
	iress = 149.236.99.252
	= DrmsTrmX: HMG01/215 ECL 200
	blifier HMG02/215 ECL 200:
	ireas = 149.236.99.252
- Amplifier	= DrmsTrmH: HMG02/215 ECL 200
BSMS: DreamS	Spec Magnet Controller connected to ethernet
	< Previous Next > Cancel



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 >

Edhead	
Expinstall	
Edsolv	
Edlock	
Edscon	
	Finish > Cano
	Expinstall Edsolv Edlock

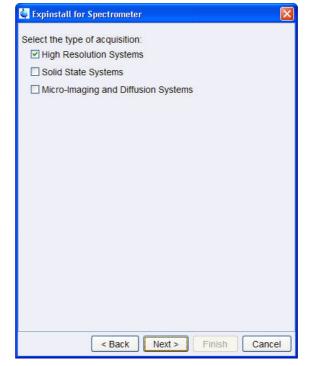
# 11.2 Expinstall

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

<b>a</b>	Experiment installation and AU compilation
	Expinstall
	Expinstall installs pulse programs, AU programs, parameter sets and various other resources for spectrometer or datastation usage. It must be performed once after the installation of TOPSPIN. For spectrometer control do cf first. For a customized datastation configuration copy your spectrometer configuration directory (typically called "spect") to <topspin dir.="" installation="">/conf/instr.</topspin>
	WARNING: Please archive all your MODIFIED Bruker PARAMETER-files, AU-programs and PULSE-programs before running "expinstall".
4. Click on Next >	< Back Next > Finish Cancel
	Experiment installation and AU compilation
S	elect the type of installation: Installation for Datastation (Default) Installation for Datastation (Customize) Installation for Spectrometer
	< Back Next > Finish Cancel

6. Click on Next >

.



7. Select 'High Resolution System'.

8. Click on	Next >
-------------	--------

l l	Expinstall for Spectrometer
	<ul> <li>Select the items you want to install:</li> <li>Install Pulse Programs</li> <li>Install Bruker AU Programs</li> <li>Recompile All User AU Programs</li> <li>Install Library CPD Programs</li> <li>Install Library Gradient Files</li> <li>Install Library Shape Files</li> </ul>
	<ul> <li>Convert Standard Parameter Sets</li> <li>Install Standard Scaling Region Files</li> <li>Install Bruker Macros</li> <li>Install Bruker Python Programs</li> </ul>
	Select all Select none
9. Click on Next >	< Back Next > Finish Cancel

💐 Expinstall for S	Spectrometer		
Select your printe	ar.		
	HP LaserJet 2100 PCL6	~	
Deladit printer.	The Lasenset 2100 PGE0		
Select your plotte	er:		
Default plotter:	HP LaserJet 2100 PCL6	~	
Select the plotter			
Paper format:	A4 / Letter 👻		
	< Back Next >	Finish	Cancel

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

Select the basic frequency of y	our spectrometer:	
Basic frequency (MHz):	300.18	
Select the digitizer:		
Type of digitzer:	DRMS ~	
Select the acquisition mode:		
Acquisition mode:	qsim -	
Select the pre-scan-delay DE:		
Default pre-scan-delay (µs)	6.5	
	Back Nexts	Einie
	< Back Next >	Einis

ectrometer
ne: FOURIER300
rams
Programs
) Programs
dient Files
pe Files
Parameter Sets
caling Region Files
ros
ion Programs
300.18 MHz
DRMS
qsim
6.5 µs
Microsoft XPS Document Writer (redirected 1/copy 1)
Microsoft XPS Document Writer (redirected 1/copy 1)
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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**.

🤹 Cron check	
An automatic periodical backup of your To can be defined in TopSpin. Currently you	
Press "Automatic Backup" to open the con	figuration tool.
Do not show this message	again
Help Automatic Ba	ackup Close

### 11.3 Set Up The Cron Job For NMR\_save

1. Click on Automatic Backup

save installation files	Restore installation files	Save user files	Restore user files
to copy the files from of the installation spe Note:	es are collected and store a previous installation to	a new installatior	ssed file. This compressed file car I or to create a backup
Location of backup fi Overwrite existing ba	le:		1\TopSpin3.0.b.43\nmr_backup
Installation to be save			TopSpin3.0.b.43
Spectrometer configu		spect	
Display default inform		() ()	
Display additional inf		0	
-Log:			

2. Click on Automatic Backup

Command	.nmrsave	e -date -pa	ath "C:	Bruker\T	opSpin3.0.b.43\r	nmr_ba	ckup" -source "C:)	
Description	Execute	Execute NMR_SAVE						
Execution scope	TopSpin	TopSpin (requires authentication)						
Options Off-schedule Direct execution								
Rules			16.		-			
Minute of the h	nour 🕑	from:	12	✓ to:	Ignore	*	+ -	
Hour of the da	y 🖌	from:	14	🖌 to:	Ignore	*	+ -	
Day of the mor	nth 🛃	from:	22	✓ to:	Ignore	~	+ -	
Month of the ye	ear 🖌	from: *		*	to: Ignore		+ -	
		from: *		~	to: Ignore			

## **Spectrometer Configuration**

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<u> </u>	

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.

Additional configuration programs:		
Config		
Installation of standard experiments	Expinstall	
Solvent table setup	Edsolv	
Probe table setup	Edhead	
Lock parameter setup	Edlock	
Spectrometer parameters setup	Edscon	

## 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. NOESY (TPPI) HMQCGP - sw opt. HMQC with gradients (magn. mode) HMBCGP - sw opt. HMBC with gradients, low pass J-filter, no decoupling HSQCEDGPPH - 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

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### **NMR Hotlines**

Contact our NMR service centers.

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

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

http://www.bruker.com/service/information-communication/helpdesk.html

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