



Advanced Experiments for Automation

ICON-NMR User Guide

Version 001



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Introduction

1

What is ICON-NMR

1.1

Welcome to Icon-nmr, a comprehensive user interface, tailor made for all your NMR laboratory management and control needs. Icon-nmr was designed to make the execution of routine NMR experiments easy and straightforward by providing a state of the art icon based user interface. The steps necessary to acquire, process, and plot a spectrum are reduced to inserting a new sample, defining the data file name, the solvent, and the experiment. No other knowledge about the instrument, the parameters, or the software is necessary. Although running on top and under the control of the computer's Linux/Windows operating system and Bruker's Topspin program, providing the acquisition and processing functions, Icon-nmr shields the user entirely from both. Accessing Topspin commands is only possible if permission has been granted by the laboratory manager. As such, Icon-nmr is particularly suited for open access spectrometers with a large number of users.

Warnings and Notes

1.2

There are two types of information notices used in this manual. These notices highlight important information or warn the user of a potentially dangerous situation. The following notices will have the same level of importance throughout this manual.



Note: Indicates important information or helpful hints



WARNING: Indicates the possibility of severe personal injury, loss of life or equipment damage if the instructions are not followed.

Contact for Additional Assistance

1.3

For further assistance on ICON-NMR, please do not hesitate to contact Bruker Application. Have your BH number available.

BRUKER BioSpin Corporation
15 Fortune Drive, Manning Park
Billerica, MA 01821
USA

Phone: (978) 667-5444
FAX:
Email: <applab@bruker-biospin.com>
Internet: www.bruker-biospin.com

1-D NOE Difference Experiment

2

Introduction

2.1



NOTE: The NOEDIFF experiment for Automation has one frequency list and one pre saturation power level. To set up this experiment, a 1-D Proton experiment has to be acquired and processed first to assign a frequency list. To prevent the automation to advance to the next sample before the NOEDIFF experiment receives the frequency list, a other experiment on same sample should be submitted before the NOEDIFF experiment. The creation of the frequency list can take some time, depending on the skill of the operator, therefor choose a experiment which covers the time of the set up. COSYGPSW takes ca. 4 Minutes if longer time is required then use a HSQC experiment.

Sample:

30 mg Pamoic acid in DMSOd6

Experiment set up

2.2


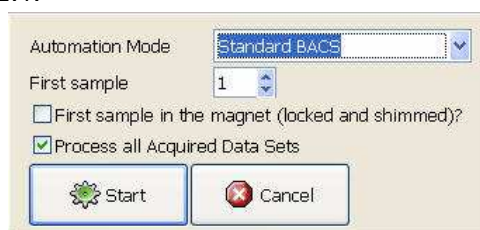
1. Log in to ICON-NMR Automation
- 2 Click on  to initialize the run

Figure 2.1.



3. Select the appropriate Automation Mode



NOTE: ICON-NMR gives you the option to lock and shim the sample prior to running the automation or, if you have stopped or halted the previous automation run, to continue on the sample already in the magnet. For that purpose click on the small button next to **First sample in the magnet (locked and shimmed)?** and a check mark will appear. The system will skip the inject/eject, locking and shimming for that sample. In addition the user can choose to process all acquired data sets or do it later. This option can be set in the ICON configuration set up.

4. Click on



5. Double click on '**Holder 1**'

6. Click inside the Name window and choose a name e.g. **Sample1**



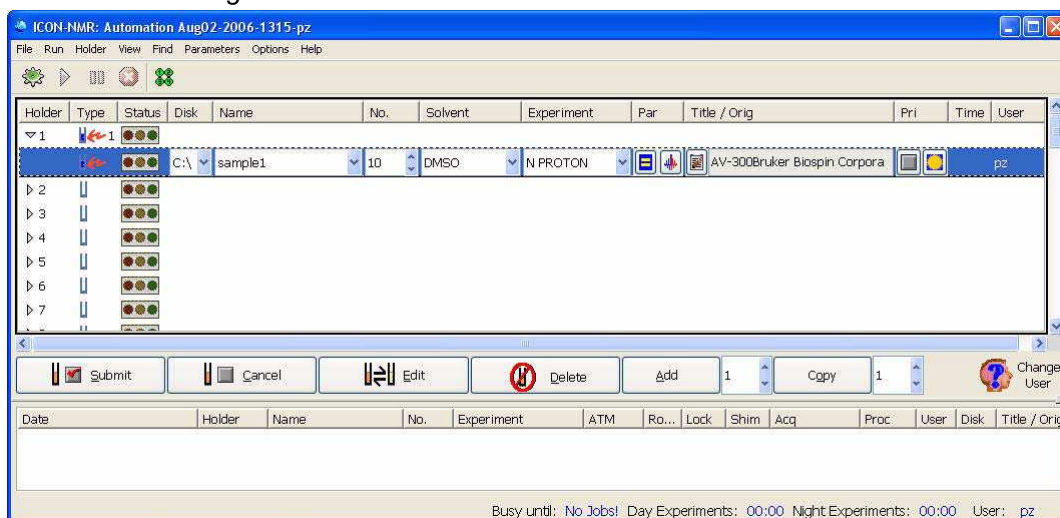
NOTE: The experiment number automatically increments to the next available number if a data set already exists with that name and experiment number.

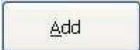
7. Click on the arrow next to the Solvent window and select '**DMSO**' by clicking on it.

8. Click on the arrow next to the Experiment window and select '**PROTON**' by clicking on it.

9. Enter a title e.g. **Pamoic acid**

Figure 2.2.



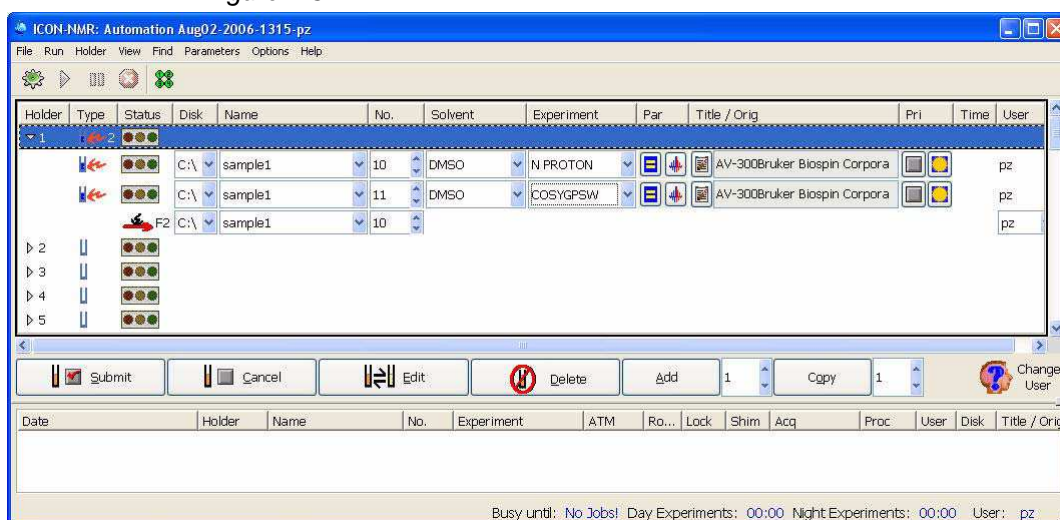
9. Click on  and select **1**

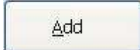
10. Click on the arrow next to the Experiment window and select '**COSYGPSW**' by clicking on it.



NOTE: If desired, any other experiments can be chosen, since this experiment is only to give enough time to create the frequency list.

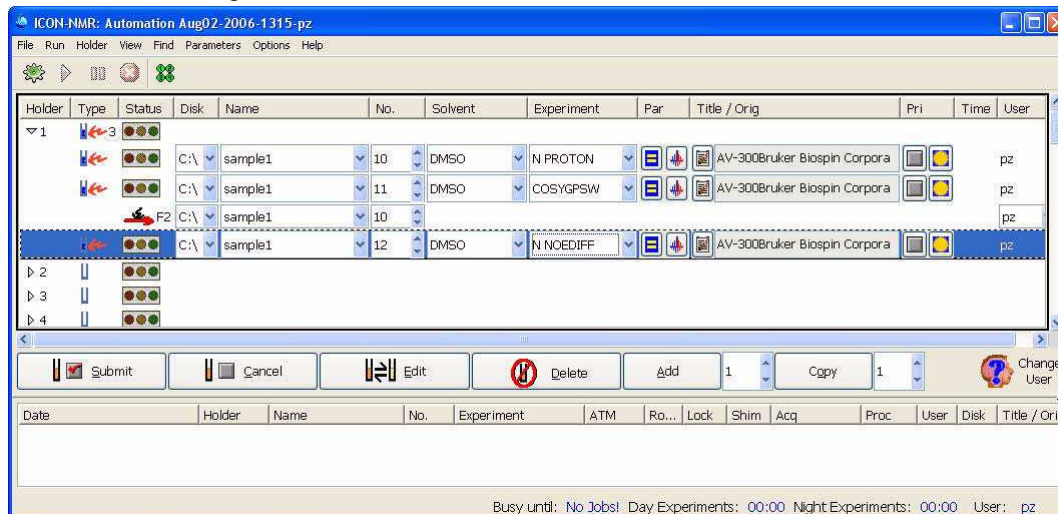
Figure 2.3.



11. Click on  and select **1**

12. Click on the arrow next to the Experiment window and select '**NOEDIFF**' by clicking on it.

Figure 2.4.



13. Click on the first line of '**Holder 1**'

14. Click on 



NOTE: At this point, the acquisition status icons appear and the run has started on the first experiment.

15. In the history window, double click on the completed Proton experiment

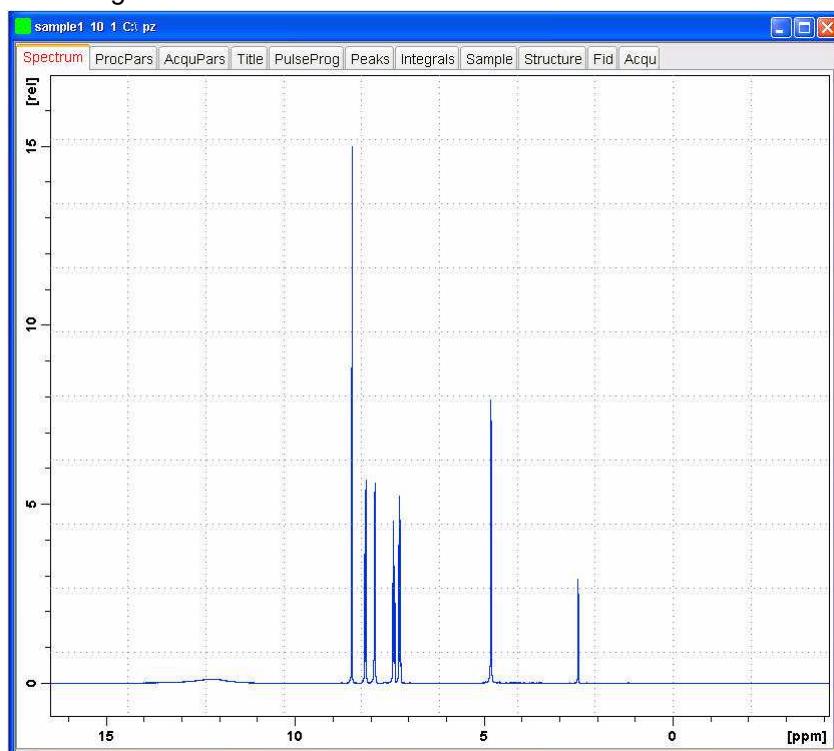
Figure 2.5.

Date	Holder	Name	No.	Experiment	ATM	Ro...	Lock	Shim	Acq	Proc	User	Disk	Title / Orig
2006-08-02 14:08:35	1.	sample1.	11	COSYGPSW		✓					pz	C:\	Instrument Customer (Biospin Co)
2006-08-02 14:03:01	1.	sample1.	10	PROTON	✓	✓	✓	✓	✓		pz	C:\	Instrument Customer (Biospin Co)



NOTE: The TOPSPIN window appears in the foreground and displays the Proton spectrum.

Figure 2.6.



16. Click on



Figure 2.7.



17. Select '**FQLIST**'

18. Type **noeau** for the frequency list name



NOTE: The parameter file NOEDIFF contains '**noeau**' as a default frequency list name and it is being overwritten every time a new list is created. If a different name is been chosen, then this name has to be changed in the acquisition parameters during the set up of the NOEDIFF experiment.

19. Click on

Figure 2.8.



20. Click on

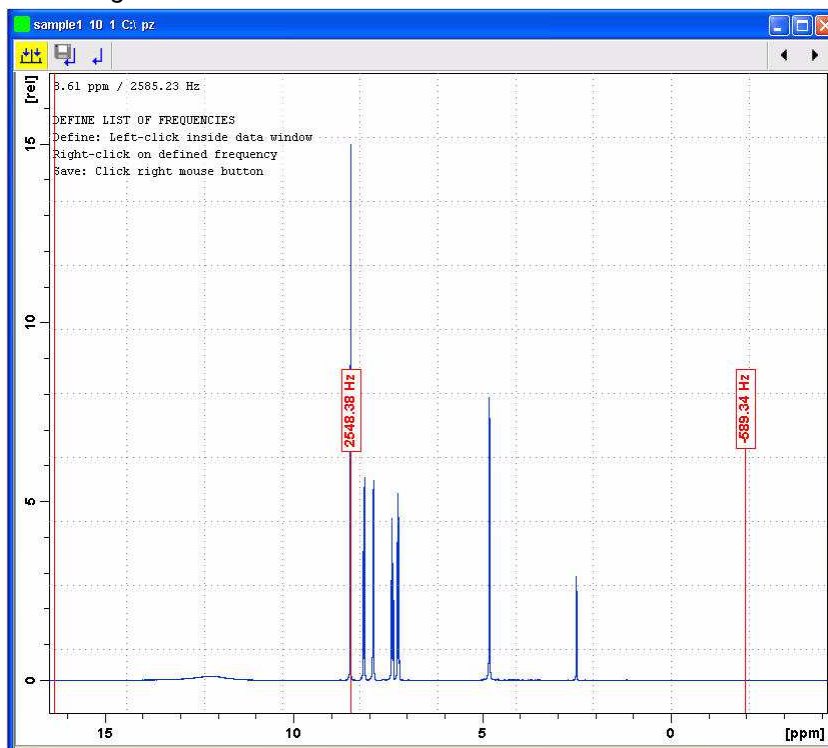
21. Move the cursor line to -2 ppm and click the left mouse button to assign the off resonance frequency

22. Using the  tools to expand the peak at 8.6 ppm



NOTE: To assign additional peaks, repeat steps 22 and 23.

Figure 2.9.




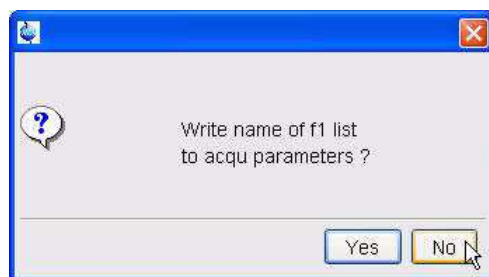

24. click on 

Figure 2.10.

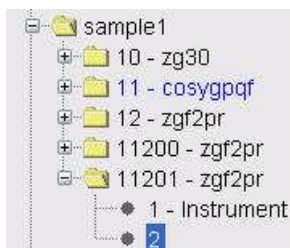


25. Click on 



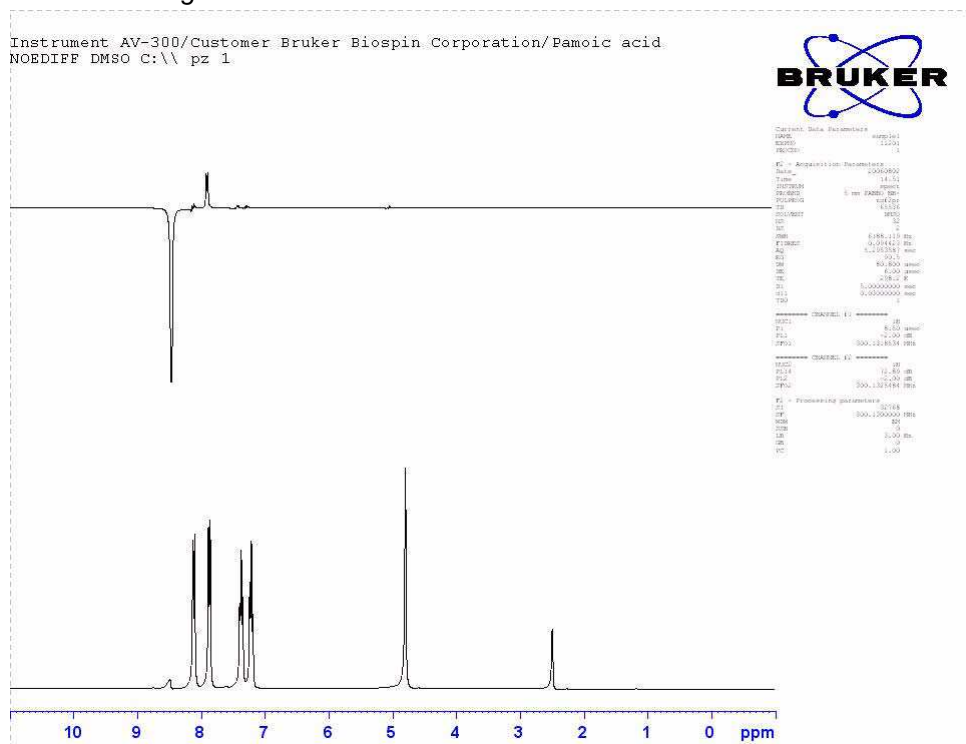
NOTE: At this point the NOEDIFF experiment is ready to run. By default it will do 4 cycles of 8 scans each for all the frequencies in the list, which amounts to total of 32 scans. The spectra are stored starting with a experiment number 11200 which is the off resonance frequency data and increment for each frequency in the list. It will perform a automatic spectra subtraction and the results are stored in the processing number 2 (see picture 2.11 below)

Figure 2.11.



NOTE: The plot layout may be have to be adjusted for a suitable plot (see picture 2.12 below)

Figure 2.12.



1-D Selective NOESY Experiment

3

Introduction

3.1



NOTE: ICON-NMR has various selective experiments listed in the default experiment table. All of them are none gradient experiments. In most cases adding pulse field gradients to the experiment helps eliminating artifacts in the spectrum. The first part of this chapter discibes set up of a selective NOESY parameter set to be used in Automation. The second part is how to run the new created experiment in Automation

Sample:

30 mg Pamoic acid in DMSOd6

Creating the selective NOESY parameter set

3.2


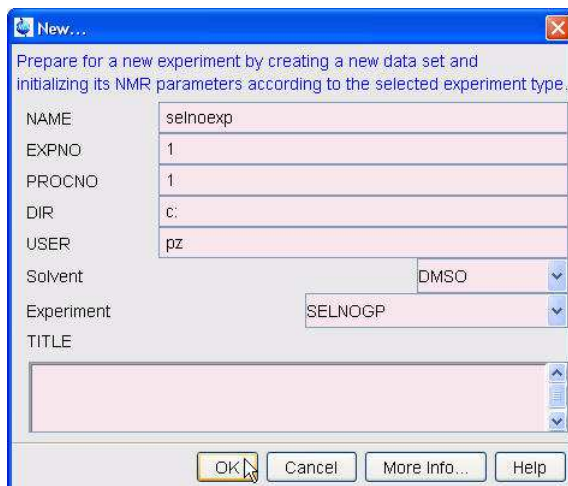
1. Click on  or type **new** in the command line
2. Change the following parameters:


Figure 3.1.



The 'New...' dialog box is shown with the following parameters:

Parameter	Value
NAME	selnoexp
EXPNO	1
PROCNO	1
DIR	c:
USER	pz
Solvent	DMSO
Experiment	SELNOGP
TITLE	

Buttons at the bottom: OK, Cancel, More Info..., Help.

3. Click on 



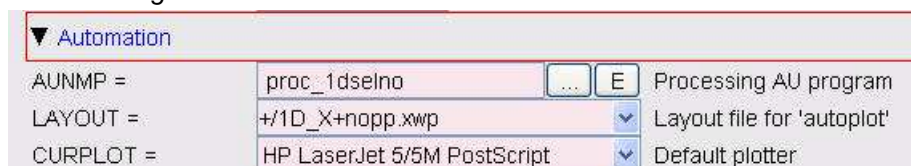
NOTE: Some parameters have to be changed before the experiment is added to ICON-NMR. In addition to the parameter change below, any other parameters such as **ns**, **td**, **d1** etc. can be administered at this point.

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

5. In the Automation section of the processing parameters, make the following changes:

AUNMP = **proc_1dselno**
LAYOUT = **+1D_X+nopp.xwp**

Figure 3.2.



Automation		
AUNMP =	proc_1dselno	Processing AU program
LAYOUT =	+1D_X+nopp.xwp	Layout file for 'autoplots'
CURPLOT =	HP LaserJet 5/5M PostScript	Default plotter



NOTE: The AU-program **proc_1dselno** is not part of the Bruker AU-program library and therefore has to be created. As part of the data processing in Automation, the **apk** command is applied to phase the spectrum. This results in the tallest peak to be phased positive. In a selective NOESY experiment, the tallest peak will be the one which the selective pulse is applied to and therefore will be automatically phased positive. The NOESY peaks can be positive or negative, depending on the molecular size and or the magnetic strength. It is necessary to phase the peak where the selective pulse is applied to negative, to assure the right phasing for the NOESY peaks. The **nm** (negate memory) command flips the phase 180 degree and therefore should be applied after **apk** (see 3.3 Creating the **proc_1dselno** AU-program).

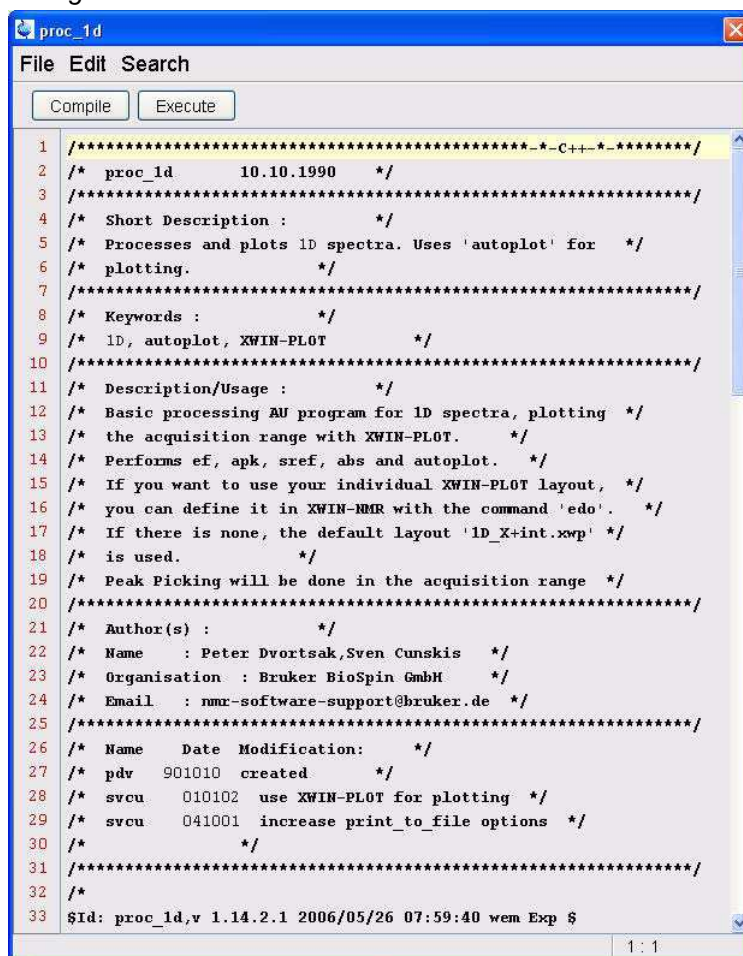
6. Type **wpar SELNOGP.mod all**

Creating the proc_1dselno Au-program

3.3

1. Type **edau proc_1d** in the command line

Figure 3.3.



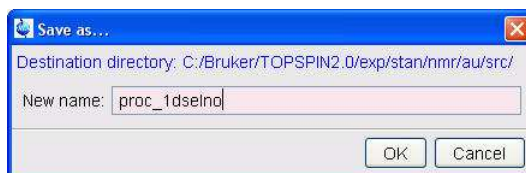
```

1  /*****--C++--*****/
2  /* proc_1d      10.10.1990  */
3  /*****/
4  /* Short Description :      */
5  /* Processes and plots 1D spectra. Uses 'autoplot' for */
6  /* plotting.              */
7  /*****/
8  /* Keywords :              */
9  /* 1D, autoplot, XWIN-PL0T */
10 /*****/
11 /* Description/Usage :      */
12 /* Basic processing AU program for 1D spectra, plotting */
13 /* the acquisition range with XWIN-PL0T. */
14 /* Performs ef, apk, sref, abs and autoplot. */
15 /* If you want to use your individual XWIN-PL0T layout, */
16 /* you can define it in XWIN-NMR with the command 'edo'. */
17 /* If there is none, the default layout '1D_X+int.xwp' */
18 /* is used. */
19 /* Peak Picking will be done in the acquisition range */
20 /*****/
21 /* Author(s) :              */
22 /* Name : Peter Dvortsak,Sven Cunsakis */
23 /* Organisation : Bruker BioSpin GmbH */
24 /* Email : nmr-software-support@bruker.de */
25 /*****/
26 /* Name Date Modification: */
27 /* pdv 901010 created */
28 /* svcu 010102 use XWIN-PL0T for plotting */
29 /* svcu 041001 increase print_to_file options */
30 /* */
31 /*****/
32 /*
33 $Id: proc_1d,v 1.14.2.1 2006/05/26 07:59:40 wem Exp $

```

2. Click on 'File' and select 'Save As'
3. Type **proc_1dselno** in to the save as window

Figure 3.4.



4. Click on 
5. Make the following changes:

line 2 **proc_1dselno** and change the date e.g. **08.04.2006**

Figure 3.5.

```

1  /*****_C++_*****/
2  /*  proc_1dse1no      08.04.2006      */
3  /*****/

```

line 14 Perform ef, apk, sref, abs, **nm** and autoplot

Figure 3.6.

```

10 /*****/
11 /* Description/Usage :      */
12 /* Basic processing AU program for 1D spectra, plotting */
13 /* the acquisition range with XWIN-PL0T.      */
14 /* Performs ef, apk, sref, abs and autoplot.  */
15 /* If you want to use your individual XWIN-PL0T layout, */
16 /* you can define it in XWIN-NMR with the command 'edo'. */
17 /* If there is none, the default layout '1D_X+int.xwp' */
18 /* is used.      */
19 /* Peak Picking will be done in the acquisition range */
20 /*****/

```

line 30 **pz** **060804** adding the NM command

Figure 3.7.

```

25 /*****/
26 /* Name    Date    Modification:    */
27 /* pdv    901010    created    */
28 /* svcu    010102    use XWIN-PL0T for plotting */
29 /* svcu    041001    increase print_to_file options */
30 /* pz      060804    adding the NM command    */
31 /*****/

```

line 53 **NM**

Figure 3.8.

```

46 /*****/
47 /* processing */
48 EF
49 ERRORABORT
50 APK
51 SREF
52 ABS
53 NM
54 /*****/

```

6. Click on 'File' and select 'Save'

7. Click on 



NOTE: Any illegal commands or characters entered in to the AU-program would result in a compilation error.

8. Click on 

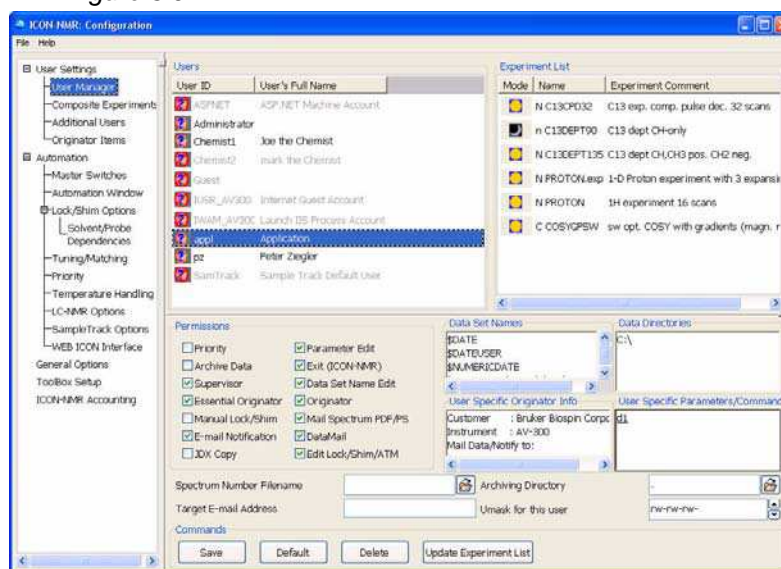
9. Click on **'File'** and select **'Close'**

Adding the experiment SELNOGP.mod to ICON-NMR

3.4

1. Click on **'Spectrometer'** in the TOPSPIN menu bar
2. Select **'ICON-NMR'**
3. Select **'Configuration'**
4. Enter the NMR Administrative password
5. Select **'User Manager'**
6. Select a user e.g. **'appl'**

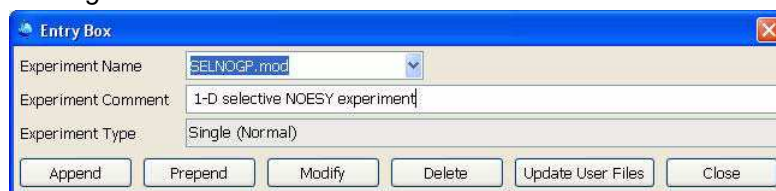
Figure 3.9.



7. Click inside the Experiment List window
8. Make the following changes in to the Entry Box window:

Experiment Name = **SELNOGP.mod**
 Experiment Comment = **1-D selective NOESY with gradients**

Figure 3.10.



9. Click on

10. Click on

Figure 3.11.

Mode	Name	Experiment Comment
	N C13CPD32	C13 exp. comp. pulse dec. 32 scans
	n C13DEPT90	C13 dept CH-only
	N C13DEPT135	C13 dept CH,CH3 pos. CH2 neg.
	N SELNOGP.mod	1-D selective NOESY experiment
	N PROTON.exp	1-D Proton experiment with 3 expans
	N PROTON	1H experiment 16 scans
	C COSYGPSW	sw opt. COSY with gradients (magn.

11. Click inside the User Specific Parameters / Commands window

12. Make the following changes in the Entry Box window:

User Specific Parameter / Command = **o1p**

Figure 3.12.



13. Click on

14. Click on

Figure 3.13.



15. Click on in the User Manager window

16. Click on **'File'** and select **'Save'** by clicking on it

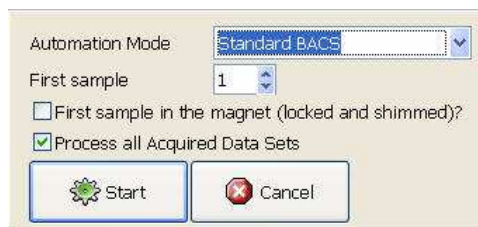
17. Click on **'File'** and select **'Close'** by clicking on it

Running the SELNOGP.mod experiment

3.5

1. Log in to ICON-NMR Automation
2. Click on  to initialize the run

Figure 3.14.



3. Select the appropriate Automation Mode



NOTE: ICON-NMR gives you the option to lock and shim the sample prior to running the automation or, if you have stopped or halted the previous automation run to continue on the sample already in the magnet. For that purpose click on the small button next to **First sample in the magnet (locked and shimmed)?** and a check mark will appear. The system will skip the inject/eject, locking and shimming for that sample. In addition the user can choose to process all acquired data sets or do it later. This option can be set in the ICON configuration set up.

4. Click on 

5. Double click on '**Holder 1**'

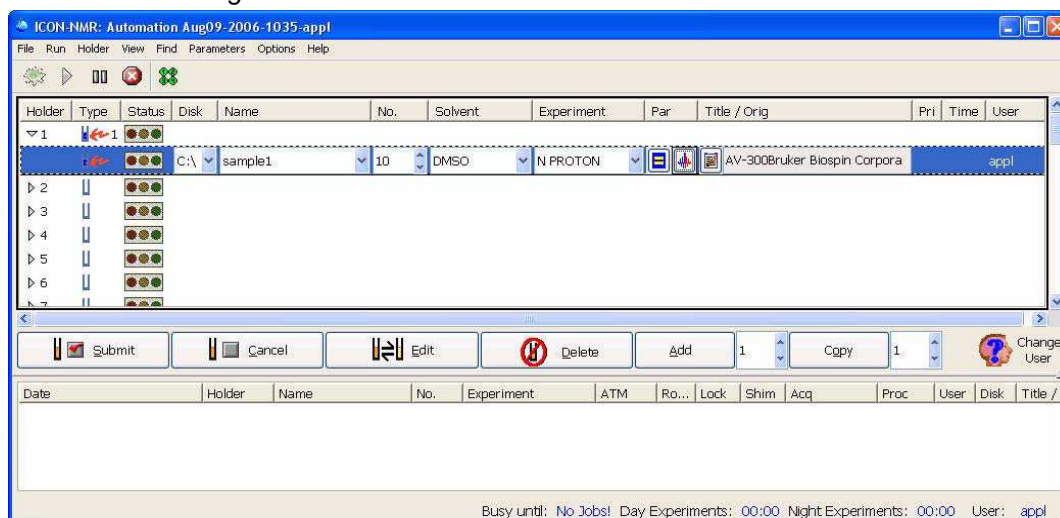
6. Click inside the Name window and choose a name e.g. **Sample1**

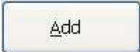


NOTE: The experiment number automatically increments to the next available number if the data set already exists.

7. Click on the arrow next to the Solvent window and select '**DMSO**' by clicking on it.
8. Click on the arrow next to the Experiment window and select '**PROTON**' by clicking on it.
9. Enter a title e.g. **Pamoic acid**

Figure 3.15.



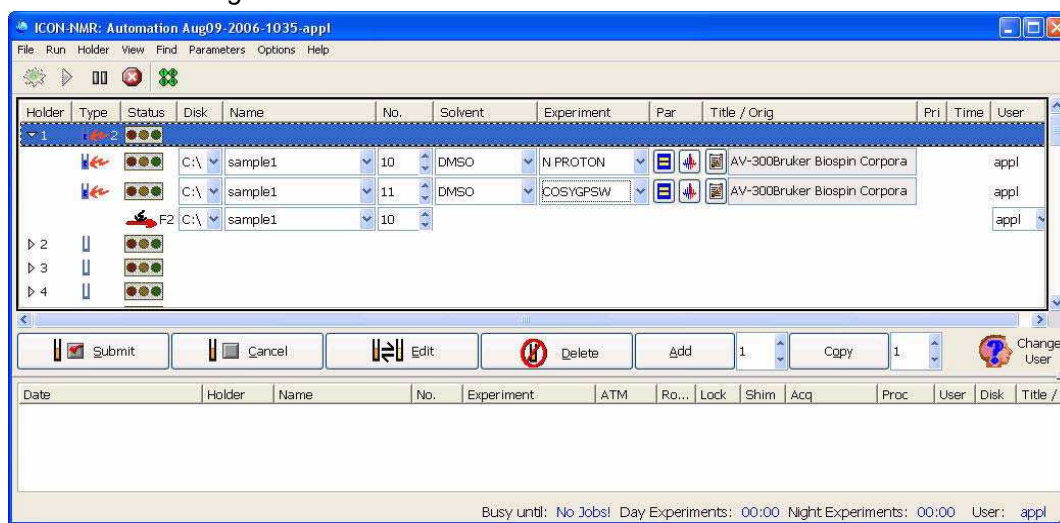
9. Click on  and select **1**

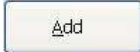
10. Click on the arrow next to the Experiment window and select '**COSYGPSW**' by clicking on it.



NOTE: If desired, any other experiments can be chosen, since this experiment is only to give enough time to get the O1 frequency from the completed Proton spectrum and assign it to the selective NOESY experiment.

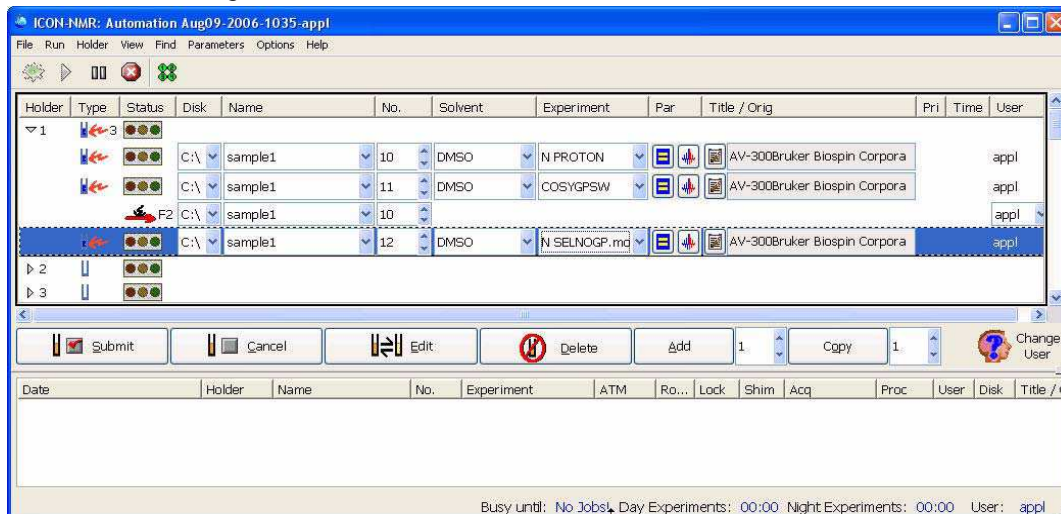
Figure 3.16.



11. Click on  and select **1**

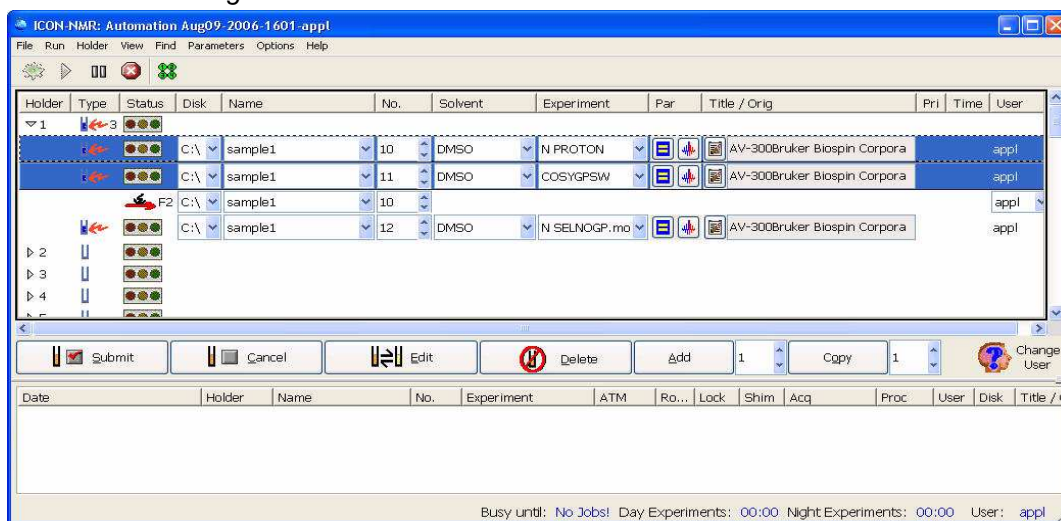
12. Click on the arrow next to the Experiment window and select 'SELNOGP.mod' by clicking on it.

Figure 3.17.



13. Select the PROTON and the COSY experiment using the 'Shift' or 'Ctrl' key

Figure 3.18.



14. Click on



NOTE: Do not submit the SELNOGP.mod experiment at this point. Get the O1P value for the applied selective pulse from the completed Proton spectrum. After

the O1P value has been entered in to the selective NOESY experiment, then submit it.

15. Double click on the completed PROTON experiment in the history window

Figure 3.19.

Date	Holder	Name	No.	Experiment	ATM	Ro...	Lock	Shim	Acq	Proc	User	Disk	Title
2006-08-09 10:56:12	1	sample1	11	COSYGPSW		✓					appl	C:\	Instr Cust Bios
2006-08-09 10:50:28	1	sample1	10	PROTON	✓	✓	✓	✓	✓	✓	appl	C:\	Instr Cust Bios



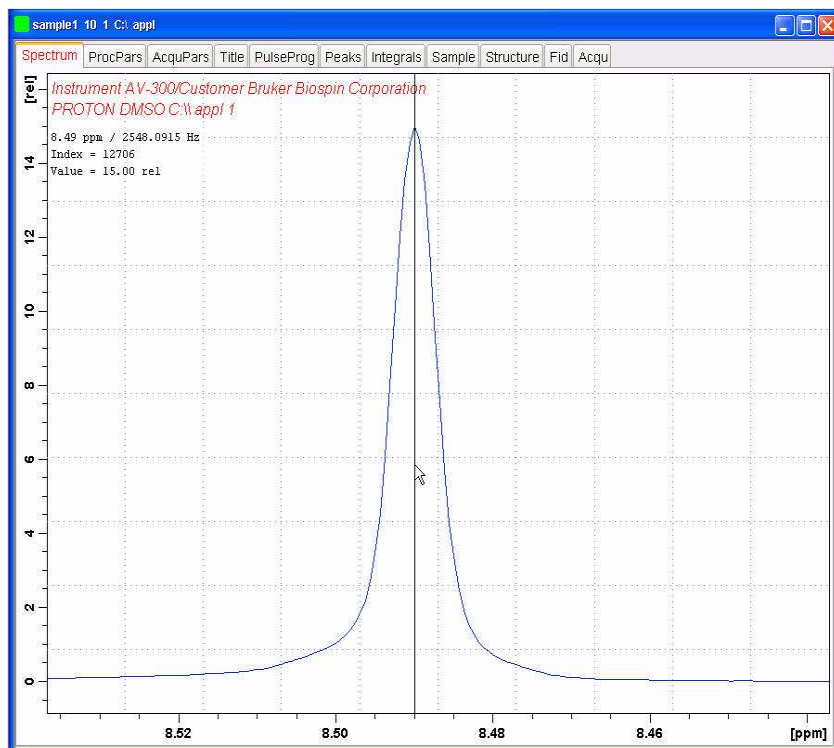
NOTE: The TOPSPIN window appears in the foreground and displays the Proton spectrum.

16. Using the  tools to expand the peak at 8.6 ppm

17. Move the cursor line in to the center of the peak

18. Write down the ppm value (e.g. 8.49) which is part of the cursor information displayed at the upper left of the spectrum window

Figure 3.20.



NOTE: To display the cursor information in the spectrum window, right click inside the spectrum window, select '**Display Properties**' and enable '**Cursor information**'.


19. In the ICON-NMR Automation window highlight the selective NOESY experiment by clicking on the traffic light of the experiment
20. Click on  of the selective NOESY experiment
21. Type the value (e.g. **8.49**) from step 18 for O1P in the user specific parameter window

Figure 3.21.

22 Click on



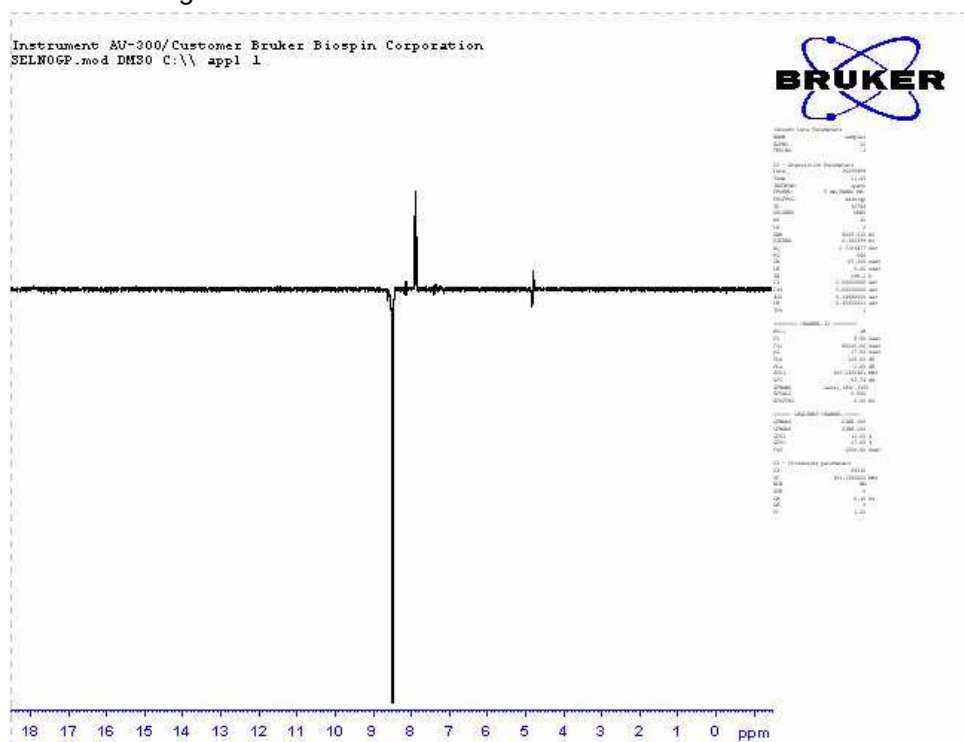
NOTE: The user specific parameter icon changes  to indicate changes to parameters

23. Click on



NOTE: The plot layout may be have to be adjusted for a suitable plot (see picture 3.24 below)

Figure 3.22.



Proton experiment with additional plot expansions

4

Introduction

4.1



NOTE: This chapter will guide you through the set up of adding to ICON-NMR a normal 1-D Proton experiment with 3 different plot regions using the PLOTEDITOR. The sample Gramicidin in DMSO used in this chapter is just an example and any other sample can be used to set up this experiment.

Sample:

30 mg Pamoic acid in DMSOd6

Creating the PROTON.exp parameter set

4.2


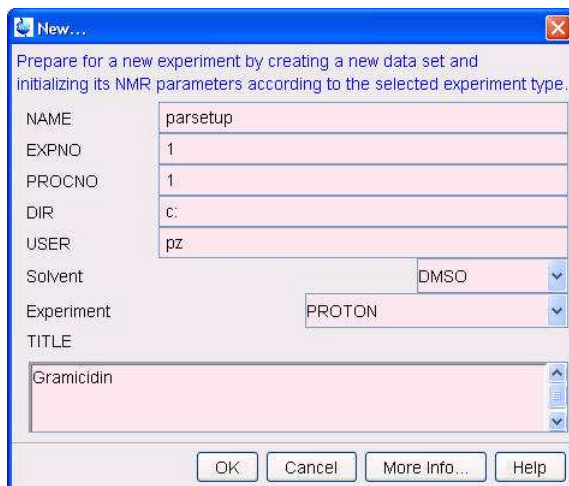
1. Click on  or type **new** in the command line
2. Change the following parameters:


Figure 4.1.



Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type.

NAME	parsetup		
EXPNO	1		
PROCNO	1		
DIR	c:\		
USER	pz		
Solvent		DMSO	▼
Experiment		PROTON	▼
TITLE	Gramicidin		

OK Cancel More Info... Help

3. Click on 



NOTE: Some parameters have to be changed before the experiment is added to ICON-NMR. In addition to the parameter change below, any other parameters such as **ns**, **td**, **d1** etc. can be administered at this point.

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

5. In the Peak Picking section of the processing parameters, make the following changes:

F1[ppm] = 10

F2[ppm] = -1

Figure 4.2.

▼ Peak picking		
MI [rel] =	0.00	Minimum intensity for pp
MAXI [rel] =	10000.00	Maximum intensity for pp
PC =	1.00	Peak detection sensitivity for pp
PSIGN =	pos.	Peak sign for pp
PSCAL =	sreg	Defines ref. peak for vertical scaling
SREGLST =	1H.CDCI3	Region file for PSCAL = sreg/psreg
ASSFAC =	0	Assign highest or second highest peak for scaling
ASSFACX =	0	Same as ASSFAC but for automatic expansion of plots
ASSWID =	0	Region excluded from second highest peak search
F1P [ppm] =	10	Left limit for pp
F2P [ppm] =	-1	Right limit for pp
CY [rel] =	15	Intensity of reference peak



NOTE: The default sweep width is 20 ppm and covers a range from -4 ppm to 16 ppm. The changes above are for the first plot expansion which covers 11 ppm from 10 ppm to -1 ppm. The other two regions are created using the processing AU-program proc_1dexp.

6. In the Automation section of the processing parameters, make the following changes:

AUNMP = proc_1dexp

Figure 4.3.

▼ Automation		
AUNMP =	proc_1dexp	Processing AU program
LAYOUT =	+1D_H.xwp	Layout file for 'autoplots'
CURPLOT =	HP LaserJet 5/5M PostScript	Default plotter



NOTE: The AU-program proc_1dexp is not part of the Bruker AU-program library and therefore has to be created (4.4 Creating the proc_1dexp AU-program).

7. Type wpar PROTON.exp all

Running the reference spectrum

4.3

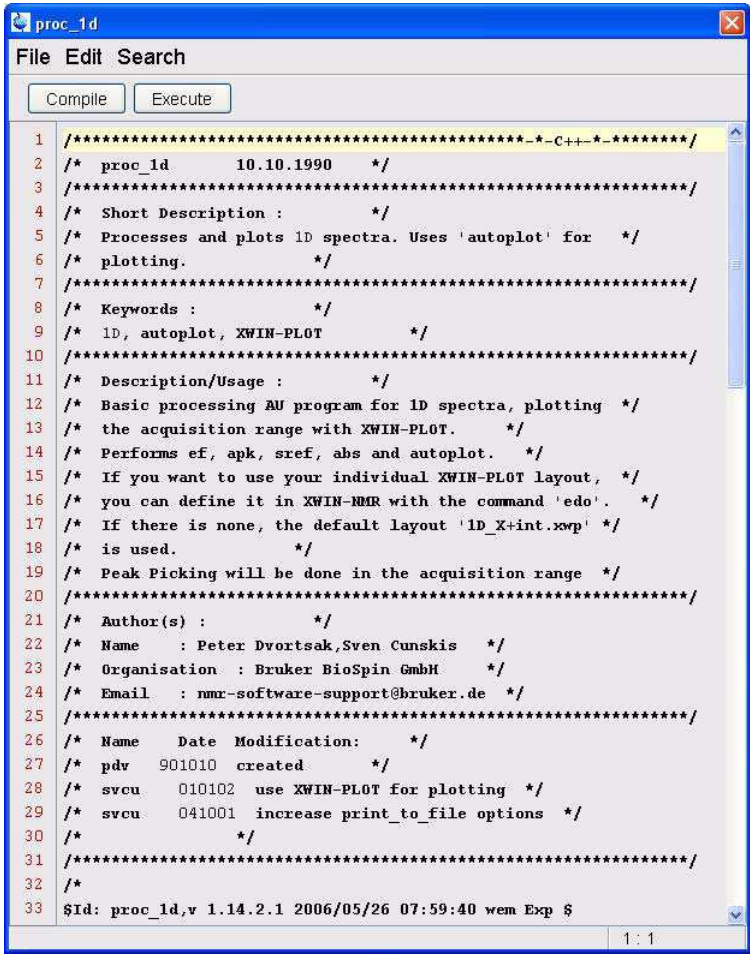
1. Type **lock** and select 'DMSO'
2. Shim for best homogeneity
3. Type **getprosol**
4. Type **rga**
5. Type **zg**
6. Type **ef**
7. Type **apk**
8. Type **abs**

Creating the proc_1dexp AU-program

4.4

1. Type **edau proc_1d** in the command line

Figure 4.4.



```

1  /*****_C+*_*****/
2  /* proc_1d      10.10.1990      */
3  /*****/
4  /* Short Description :          */
5  /* Processes and plots 1D spectra. Uses 'autoplot' for      */
6  /* plotting.          */
7  /*****/
8  /* Keywords :          */
9  /* 1D, autoplot, XWIN-PL0T      */
10 /*****/
11 /* Description/Usage :          */
12 /* Basic processing AU program for 1D spectra, plotting */
13 /* the acquisition range with XWIN-PL0T.          */
14 /* Performs ef, apk, sref, abs and autoplot.      */
15 /* If you want to use your individual XWIN-PL0T layout, */
16 /* you can define it in XWIN-NMR with the command 'edo'. */
17 /* If there is none, the default layout '1D_X+int.xwp' */
18 /* is used.          */
19 /* Peak Picking will be done in the acquisition range */
20 /*****/
21 /* Author(s) :          */
22 /* Name       : Peter Dvortsak,Sven Cunsakis */
23 /* Organisation : Bruker BioSpin GmbH      */
24 /* Email      : nmr-software-support@bruker.de */
25 /*****/
26 /* Name      Date      Modification: */
27 /* pdv       901010    created      */
28 /* svcu      010102    use XWIN-PL0T for plotting */
29 /* svcu      041001    increase print_to_file options */
30 /*          */
31 /*****/
32 /*
33 $Id: proc_1d,v 1.14.2.1 2006/05/26 07:59:40 wem Exp $

```

2. Click on 'File' and select 'Save As'
3. Type **proc_1dexp** in to the save as window

Figure 4.5.



4. Click on

5. Make the following changes:

line 2 **proc_1dexp** and change the date e.g. **08.07.2006**

Figure 4.6.

```

1  /*****-*-C++-*****/
2  /* proc_1dexp      08.07.2006  */
3  /*****/

```

line 12 Basic processing AU program for **three** 1D spectra, plotting

line 13 **expansions of** the acquisition range with XWIN-PLOT

Figure 4.7.

```

10 /*****/
11 /* Description/Usage :      */
12 /* Basic processing AU program for three 1D spectra, plotting */
13 /* expansions of the acquisition range with XWIN-PLOT.      */
14 /* Performs ef, apk, sref, abs and autoplot.      */
15 /* If you want to use your individual XWIN-PLOT layout, */
16 /* you can define it in XWIN-NMR with the command 'edo'. */
17 /* If there is none, the default layout '1D_X+int.xwp' */
18 /* is used.      */
19 /* Peak Picking will be done in the acquisition range */
20 /*****/

```

line 30 **pz 060807 adding two more plot expansions**

Figure 4.8.

```

25 /*****/
26 /* Name      Date      Modification:      */
27 /* pdv      901010      created      */
28 /* svcu      010102      use XWIN-PLOT for plotting */
29 /* svcu      041001      increase print_to_file options */
30 /* pz        060807      adding to more plot expansions */
31 /*****/

```

line 91 **AUTO PLOT**

line 92 **STOREPAR("LAYOUT","+/exp1.xwp")**

line 93 **AUTO PLOT**

line 94 **STOREPAR("LAYOUT","+/exp2.xwp")**

line 95 **AUTO PLOT**

line 96 **STOREPAR("LAYOUT",&xwlay)**

Figure 4.9.

```

87  /*****
88  /* restore the original plotregion */
89  STOREPAR("F1P",f1porig)
90  STOREPAR("F2P",f2porig)
91  AUTOPLLOT
92  STOREPAR("LAYOUT", "+/exp1.xwp")
93  AUTOPLLOT
94  STOREPAR("LAYOUT", "+/exp2.xwp")
95  AUTOPLLOT
96  STOREPAR("LAYOUT",&xwlay)
97
98  *****/

```

6. Click on **'File'** and select **'Save'**

7. Click on



NOTE: Any illegal commands or characters entered in to the AU-program would result in a compilation error.

8. Click on

9. Click on **'File'** and select **'Save'** by clicking on it

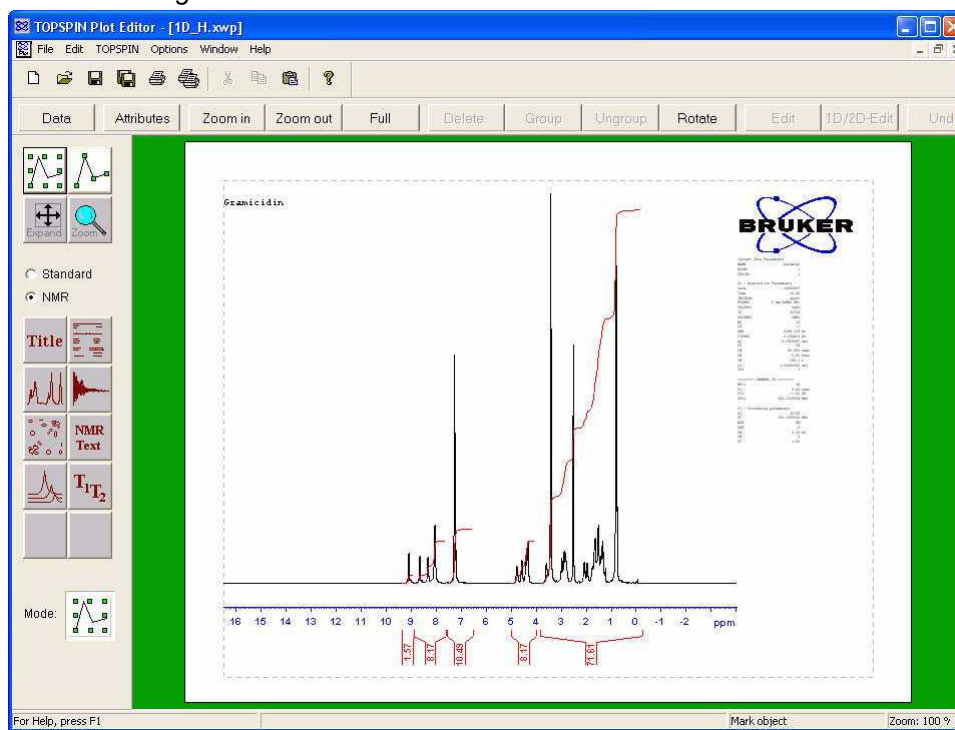
10. Click on **'File'** and select **'Close'** by clicking on it

Setting up the plotting expansions

4.5

1. In the TOPSPIN window display the spectrum observed in 4.3 Running the reference spectrum
2. Type **xwp** in the command line

Figure 4.10.



3. Click inside the spectrum window to display the green handles

4. Click on **Edit**

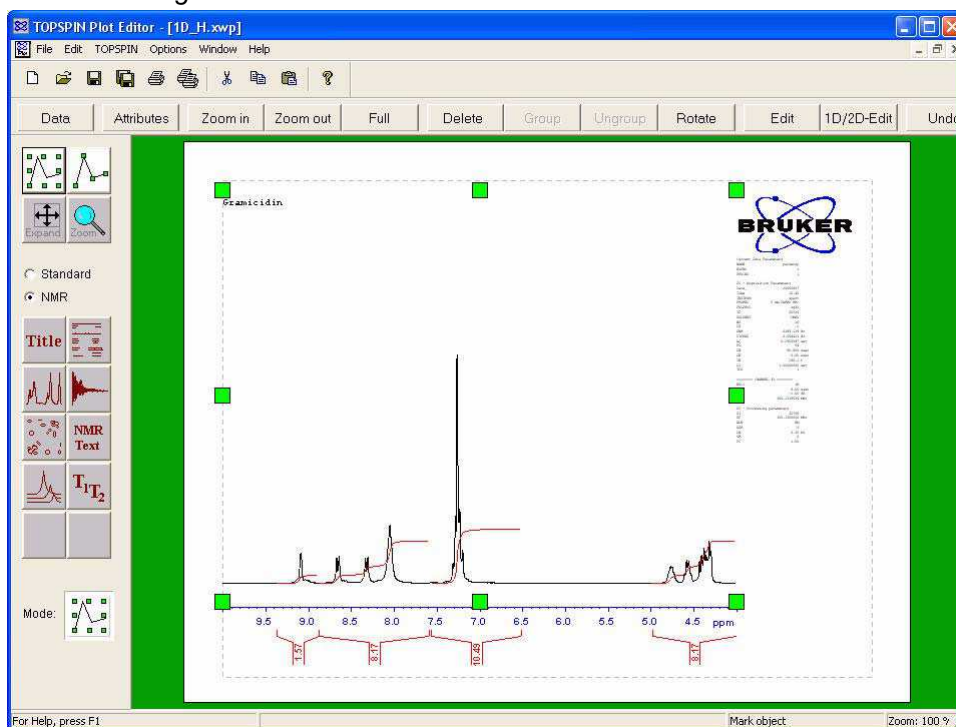
5. Make the following changes:

Xmin/Xmax = 10 / 4

Figure 4.11.

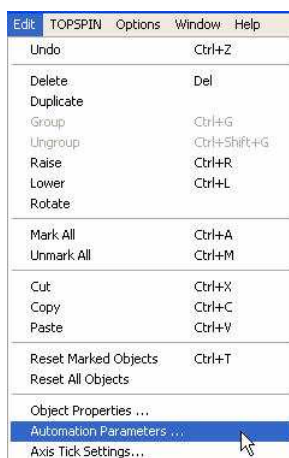
6. Click on **OK**

Figure 4.12.



7. Click on **'Edit'** in the main menu bar and select **'Automation Parameters'** by clicking on it

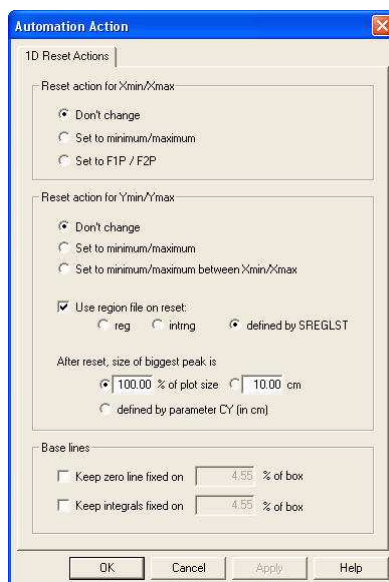
Figure 4.13.



8. Make the following changes:

Reset action for Xmin/Xmax	=	Don't change
Reset action Ymin/Ymax	=	Don't change

Figure 4.14.

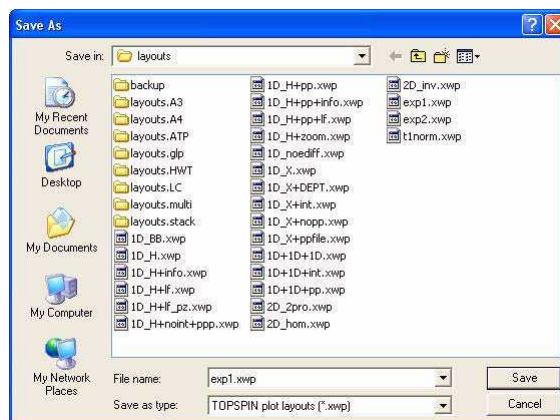


9. Click on 

10. Click on **'File'** in the main menu bar and select **'Save as'**

11. Change the File name to: **exp1.xwp**

Figure 4.15.



12. Click on 

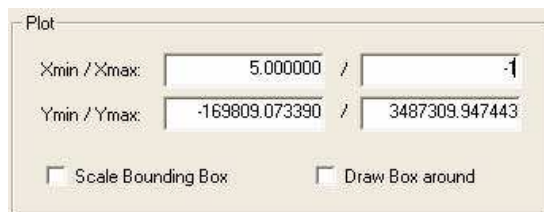
13. Click inside the spectrum window to display the green handles

14. Click on 

15. Make the following changes:

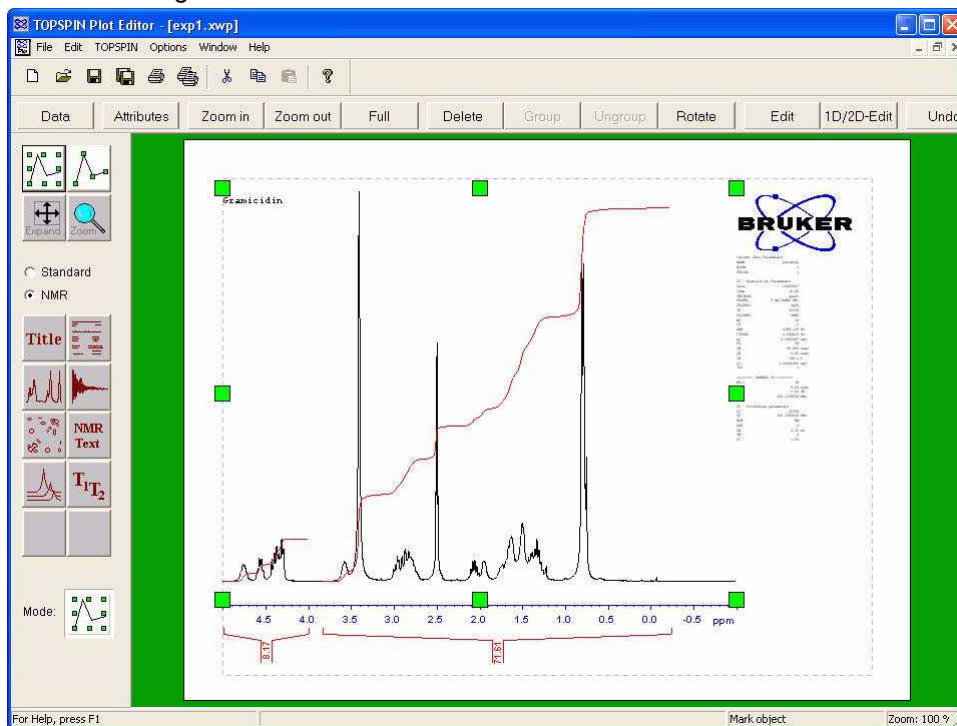
$$Xmin/Xmax = 5 / -1$$

Figure 4.16.



16. Click on **OK**

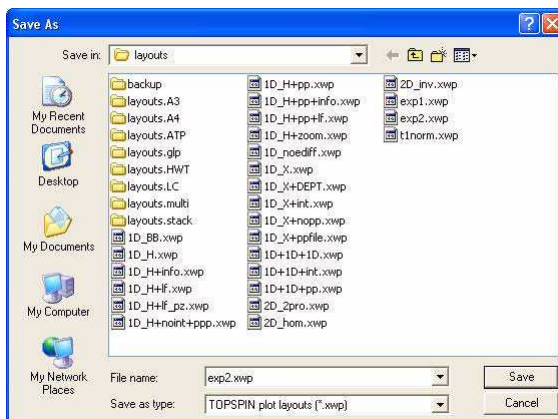
Figure 4.17.




17. repeat steps 7 through 10 above

18. Change the File name to: **exp2.xwp**

Figure 4.18.



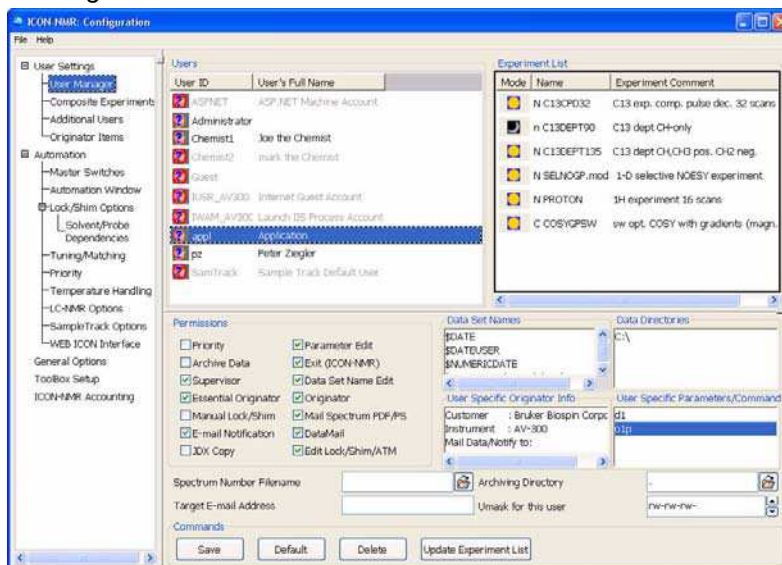
19. Click on 

Adding the experiment PROTON.exp to ICON-NMR

4.6

1. Click on 'Spectrometer' in the TOPSPIN menu bar
2. Select 'ICON-NMR'
3. Select 'Configuration'
4. Enter the NMR Administrative password
5. Select 'User Manager'
6. Select a user e.g. 'appl'

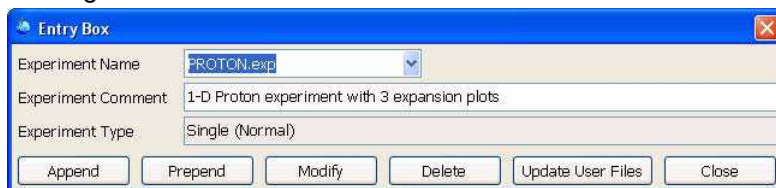
Figure 4.19.



7. Click inside the Experiment List window
8. Make the following changes in to the Entry Box window:

Experiment Name	=	PROTON.exp
Experiment Comment	=	1-D Proton experiment with 3 expansion plots

Figure 4.20.












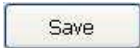
9. Click on 
10. Click on 

Figure 4.21.

Experiment List		
Mode	Name	Experiment Comment
	N C13CPD32	C13 exp. comp. pulse dec. 32 scans
	n C13DEPT90	C13 dept CH-only
	N C13DEPT135	C13 dept CH,CH3 pos. CH2 neg.
	N SELNOGP.mod	1-D selective NOESY experiment
	N PROTON.exp	1-D Proton experiment with 3 expansions
	N PROTON	1H experiment 16 scans
	C COSYGPSW	sw opt. COSY with gradients (magn.

11. Click on  in the User Manager window
12. Click on **'File'** and select **'Close'** by clicking on it



NOTE: To save time to test the new created experiment, insert the sample in to the magnet, lock and shim for best homogeneity. Open up a new data set window and load the PROTON.exp experiment. Type **getprosoli** and type **xaui**. After the acquisition is completed, type **xaup**. Check if the plots come out to your satisfaction.

1-D Kinetic Experiment

5

Introduction

5.1



NOTE: A Kinetic experiment consist of a series of 1-D spectra. Intensity, line width and shift changes of peaks in the spectrum are measured as a function of time, temperature or ph of a sample. The instructions in the chapter do not show the results of a particular Kinetic experiment. It will guide you through the creation of a ICONNMR experiment which can be used in a Kinetic study.

Sample:

30 mg Pamoic acid in DMSOd6

Creating the Kinetic experiment parameter set

5.2


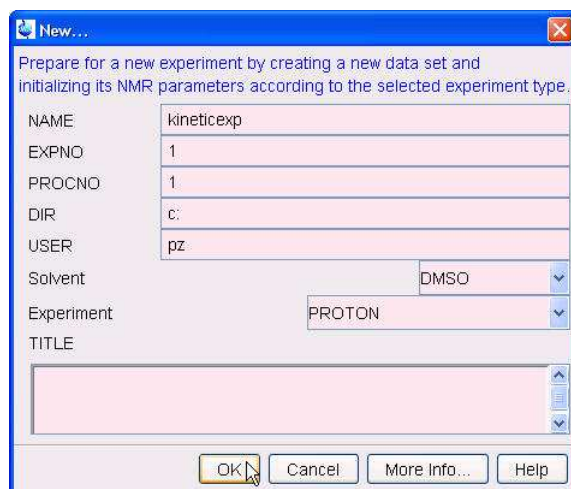
1. Click on  or type **new** in the command line
2. Change the following parameters:

Figure 5.1.




New...

Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type.

NAME	kineticexp
EXPNO	1
PROCNO	1
DIR	c:\
USER	pz
Solvent	DMSO
Experiment	PROTON
TITLE	

OK Cancel More Info... Help

3. Click on 



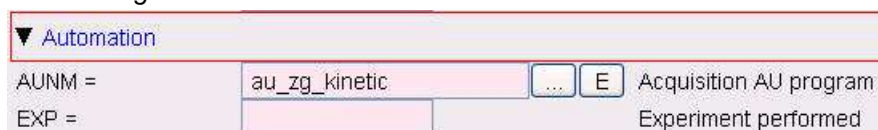
NOTE: Some parameters have to be changed before the experiment is added to ICON-NMR. In addition to the parameter change below, any other parameters such as **ns**, **td**, **d1** etc. can be administered at this point.

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

5. In the Automation section of the processing parameters, make the following changes:

AUNM = au_zg_kinetic

Figure 5.2.




NOTE: The AU-program au_zg_kinetic is not part of the Bruker AU-program library and therefore has to be created. As part of the Kinetic experiment in Automation, a series of 1-D files are collected. The first experiment will include a receiver gain adjustment and is observed with a minimal delay. For all the other experiments, the acquisition starts with a fixed delay and the receiver gain is been carried over from the first experiment. The delay and number of experiments are changed within the AU-program. The data are stored with a experiment number = (EXPNO) * 100 + 10'000 and is incremented by 1.(see 5.3 Creating the au_zg_kinetic AU-program).

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

7. In the Automation section of the processing parameters, make the following changes:

AUNMP = proc_1d_kinetic

Figure 5.3.

Automation			
AUNMP =	proc_1d_kinetic	...	E Processing AU program
LAYOUT =	+1D_H.xwp	▼	Layout file for 'autoplot'
CURPLOT =	HP LaserJet 5/5M PostScript	▼	Default plotter



NOTE: The AU-program 'proc_1d_kinetic' is not part of the Bruker AU-program library and therefore has to be created. This processing program is used in ICON-NMR to process a series of 1-D spectra collected using the acquisition AU-program 'au_zg_kinetic'. (see 5.4 Creating the proc_1d_kinetic AU-program).

8. Type **wpar PROTON.kinetic all**

Creating the au_zg_kinetic AU program

5.3

1. Type **edau au_zg** in the command line

Figure 5.4.

```

1  /*** ^^A -*-C++-*- *****/
2  /* au_zg 10.10.1990 */
3  /*****/
4  /* Short Description : */
5  /* General AU program for data acquisition. */
6  /*****/
7  /* Keywords : */
8  /* zg */
9  /*****/
10 /* Description/Usage : */
11 /* General AU program for data acquisition. */
12 /* First an rga is done, then the acquisition is started. */
13 /*****/
14 /* Author(s) : */
15 /* Name : Peter Dvortsak */
16 /* Organisation : Bruker Analytik */
17 /* Email : peter.dvortsak@bruker.de */
18 /*****/
19 /* Name Date Modification: */
20 /* pdv 901010 created */
21 /*****/
22 /*
23 $Id: au_zg,v 1.5 2000/07/12 11:39:39 gsc Exp $
24 */
25
26 GETCURDATA
27 RGA
28 ZG
29 QUIT

```

2. Click on 'File' and select 'Save As'

3. Type **au_zg_kinetic** in to the save as window

Figure 5.5.



4. Click on **OK**

5. Make the following changes:

line 2 **au_zg_kinetic** and change the date e.g. **09.05.2006**

Figure 5.6.

```

1  /*** ^^A -*-C++-*- *****/
2  /* au_zg_kinetic 09.05.2006 */
3  /*****/

```

line 5 **Kinetic experiment**

Figure 5.7.

```

3  /****/
4  /* Short Description :          */
5  /* Kinetic experiment.        */
6  /****/

```

line 8 **Kinetic**

Figure 5.8.

```

6  /****/
7  /* Keywords :                  */
8  /* kinetic                     */
9  /****/

```

line 11 **Kinetic experiment to be used in ICONNMR**

line 12 **rga is done on the first experiment**

Figure 5.9.

```

9  /****/
10 /* Description/Usage :          */
11 /* Kinetic experiment to be used in ICONNMR.        */
12 /* rga is done on the first experiment.              */
13 /****/

```

line 15 Change name

line 16 Change Organisation

line 17 Change E-mail address

Figure 5.10.

```

13 /****/
14 /* Author(s) :                  */
15 /* Name : Peter Ziegler          */
16 /* Organisation : Bruker Biospin INC.        */
17 /* Email : peter.ziegler@bruker-biospin.com */
18 /****/

```

line 20 Change initials and date created

Figure 5.11.

```

18 /****/
19 /* Name     Date   Modification:   */
20 /* PZ 08.31.2006 created          */
21 /****/

```

line 26, through 29 Delete

Figure 5.12.

```

26 GETCURDATA
27 RGA
28 ZG
29 QUIT

```

line 25 through 44

Add the text below

Figure 5.13.

```

26 int startexpno, expnosave;
27 GETCURDATA
28 expnosave = expno;
29 startexpno = expno * 100 + 10000;
30 expno = startexpno;
31 SETCURDATA
32 for (i1=1;i1<=10;i1++)
33 {
34   if (i1==1)
35   {
36     RGA
37   }
38   ZG
39   IEXPNO
40   sleep(5);
41 }
42 expno = expnosave;
43 SETCURDATA
44 QUIT

```



NOTE: The number of experiments are set in line 32: for (i1=1;i1<=10;i1++). In this example it is set to 10 experiments. The delay between the experiments are set in line 40: sleep(5) where the time in brackets is entered in Seconds. In this example it is set to 5 seconds.

6. Click on **'File'** and select **'Save'**

7. Click on



NOTE: Any illegal commands or characters entered in to the AU-program would result in a compilation error.

8. Click on

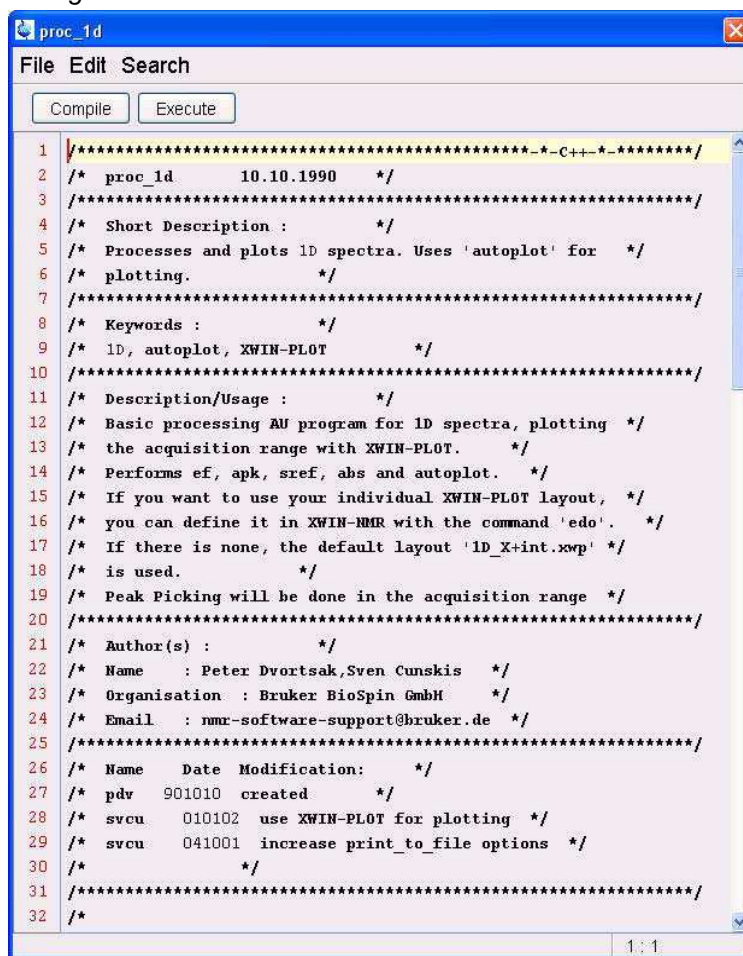
9. Click on **'File'** and select **'Close'**

Creating the proc_1d_kinetic AU program

5.4

1. Type **edau proc_1d** in the command line

Figure 5.14.



```

1  /*****_C++_*****/
2  /* proc_1d      10.10.1990      */
3  /*****/
4  /* Short Description :          */
5  /* Processes and plots 1D spectra. Uses 'autoplot' for      */
6  /* plotting.          */
7  /*****/
8  /* Keywords :          */
9  /* 1D, autoplot, XWIN-PL0T      */
10 /*****/
11 /* Description/Usage :          */
12 /* Basic processing AU program for 1D spectra, plotting */
13 /* the acquisition range with XWIN-PL0T.          */
14 /* Performs ef, apk, sref, abs and autoplot.      */
15 /* If you want to use your individual XWIN-PL0T layout, */
16 /* you can define it in XWIN-NMR with the command 'edo'. */
17 /* If there is none, the default layout '1D_X+int.xrp' */
18 /* is used.          */
19 /* Peak Picking will be done in the acquisition range */
20 /*****/
21 /* Author(s) :          */
22 /* Name       : Peter Dvortsak,Sven Cunsakis      */
23 /* Organisation : Bruker BioSpin GmbH          */
24 /* Email      : nmr-software-support@bruker.de    */
25 /*****/
26 /* Name      Date      Modification:          */
27 /* pdv      901010     created          */
28 /* svcu     010102     use XWIN-PL0T for plotting */
29 /* svcu     041001     increase print_to_file options */
30 /*          */
31 /*****/
32 /*

```

2. Click on 'File' and select 'Save As'
3. Type **proc_1d_kinetic** in to the save as window

Figure 5.15.



4. Click on
5. Make the following changes:

line 2 **proc_1d_kinetic** and change the date e.g. **09.05.2006**

Figure 5.16.

```

1  /*****_C+--+_*****/
2  /*  proc_1d_kinetic      09.05.2006  */
3  /*****/

```

line 5 Processes and plots series of 1D spectra uses 'autoplot' for

Figure 5.17.

```

3  /*****/
4  /*  Short Description :      */
5  /*  Processes and plots series of 1D spectra. Uses 'autoplot' for */
6  /*  plotting.                */
7  /*****/

```

line 12 Basic processing Au program for a series of 1D spectra

line 13 observed using the au_zg_kinetic acqu AU program

line 14 Plotting the acquisition range with XWIN-PLOT

Figure 5.18.

```

10 /*****/
11 /*  Description/Usage :      */
12 /*  Basic processing AU program for a series of 1D spectra */
13 /*  observed using the au_zg_kinetic acqu AU program. */
14 /*  Plotting the acquisition range with XWIN-PLOT.      */
15 /*  Performs ef, apk, sref, abs and autoplot.          */
16 /*  If you want to use your individual XWIN-PLOT layout, */
17 /*  you can define it in XWIN-NMR with the command 'edo'. */
18 /*  If there is none, the default layout '1D_X+int.xwp' */
19 /*  is used.                */
20 /*  Peak Picking will be done in the acquisition range */
21 /*****/

```

line 31 Change initials and date and modification

Figure 5.19.

```

26 /*****/
27 /*  Name    Date  Modification:  */
28 /*  pdv     901010  created      */
29 /*  svcu     010102  use XWIN-PLOT for plotting */
30 /*  svcu     041001  increase print_to_file options */
31 /*  pz       090506  plotting a series of 1-D spectra */
32 /*****/

```

line 49 through 56 Add the text below

Figure 5.20.

```

47  /*****
48  /* processing */
49  /* get initial dataset and set startexpno according to 'au_zg_kinetic' */
50  int expno_orig, startexpno, expnosave;
51  GETCURDATA
52  expno_orig=expno;
53  startexpno=expno*100+10000;
54  expno=startexpno;
55  i1=10;
56  TIMES(i1) |
57  EF
58  ERRORABORT
59  APK
60  SREF
61  ABS
62
63  *****/

```



NOTE: The number of experiments are set in line 55: i1=10 and should be set to the same number as in the acquisition AU program au_zg_kinetic.

line 102 IEXPNO

line 104 DEXPNO

Figure 5.21.

```

96  /*****
97  /* restore the original plotregion */
98  STOREPAR("F1P",f1porig)
99  STOREPAR("F2P",f2porig)
100
101  AUTOPLT;
102  IEXPNO;
103  END
104  DEXPNO;
105
106
107  *****/

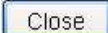
```

6. Click on 'File' and select 'Save'

7. Click on 



NOTE: Any illegal commands or characters entered in to the AU-program would result in a compilation error.

8. Click on 

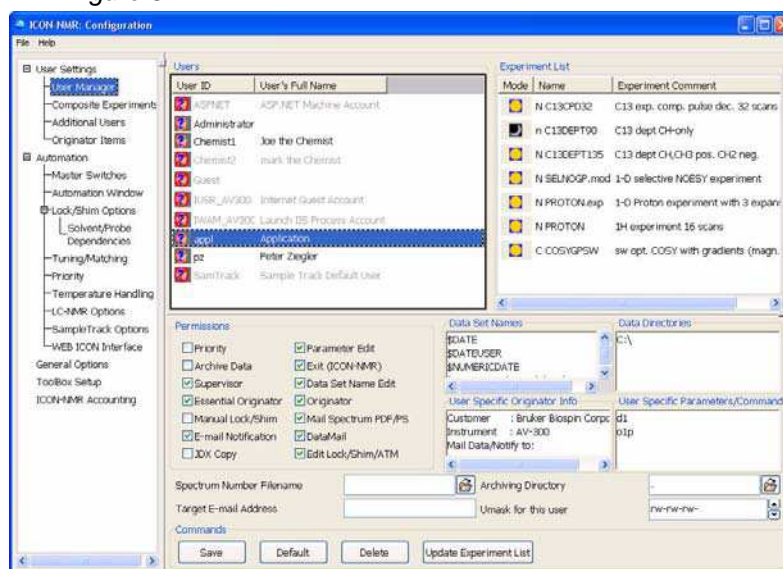
9. Click on **'File'** and select **'Close'**

Adding the experiment *PROTON.kinetic* to *ICON-NMR*

5.5

1. Click on **'Spectrometer'** in the TOPSPIN menu bar
2. Select **'ICON-NMR'**
3. Select **'Configuration'**
4. Enter the NMR Administrative password
5. Select **'User Manager'**
6. Select a user e.g. **'appl'**

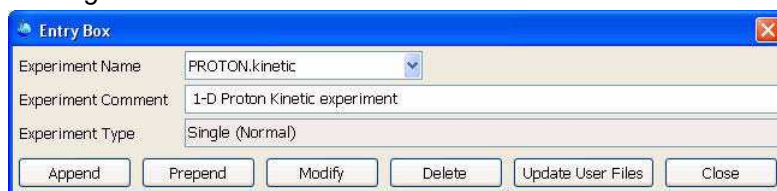
Figure 5.22.



7. Click inside the Experiment List window
8. Make the following changes in to the Entry Box window:

Experiment Name = **PROTON.kinetic**
 Experiment Comment = **1-D Proton Kinetic experiment**

Figure 5.23.



Entry Box

Experiment Name: PROTON.kinetic

Experiment Comment: 1-D Proton Kinetic experiment

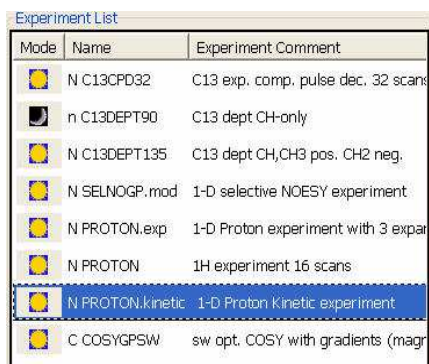
Experiment Type: Single (Normal)









Buttons: Append, Prepend, Modify, Delete, Update User Files, Close

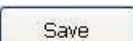
9. Click on 

10. Click on 

Figure 5.24.



Mode	Name	Experiment Comment
	N C13CPD32	C13 exp. comp. pulse dec. 32 scans
	n C13DEPT90	C13 dept CH-only
	N C13DEPT135	C13 dept CH,CH3 pos. CH2 neg.
	N SELNOGP.mod	1-D selective NOESY experiment
	N PROTON.exp	1-D Proton experiment with 3 expt
	N PROTON	1H experiment 16 scans
	N PROTON.kinetic	1-D Proton Kinetic experiment
	C COSYGPSW	sw opt. COSY with gradients (magr

11. Click on  in the User Manager window

12. Click on **'File'** and select **'Save'** by clicking on it

13. Click on **'File'** and select **'Close'** by clicking on it

Running the PROTON.kinetic experiment

5.6

1. Log in to ICON-NMR Automation


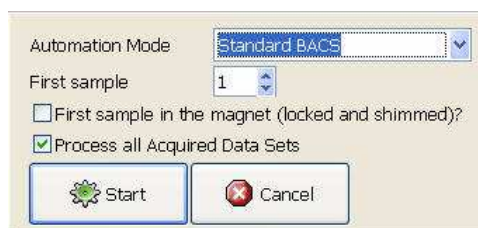
2 Click on  to initialize the run

Figure 5.25.





Automation Mode: Standard BACS

First sample: 1

☐ First sample in the magnet (locked and shimmed)?

☒ Process all Acquired Data Sets

Buttons:  Start,  Cancel

3. Select the appropriate Automation Mode



NOTE: ICON-NMR gives you the option to lock and shim the sample prior to running the automation or, if you have stopped or halted the previous automation run to continue on the sample already in the magnet. For that purpose click on the small button next to **First sample in the magnet (locked and shimmed)?** and a check mark will appear. The system will skip the inject/eject, locking and shimming for that sample. In addition the user can choose to process all acquired data sets or do it later. This option can be set in the ICON configuration set up.

4. Click on



5. Double click on '**Holder 1**'

6. Click inside the Name window and choose a name e.g. **Sample1**



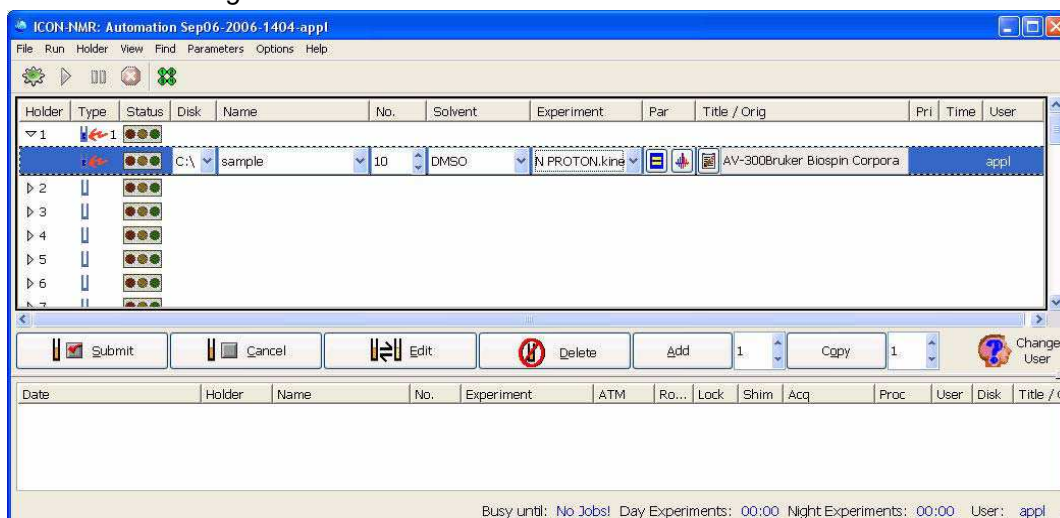
NOTE: The experiment number automatically increments to the next available number if the data set already exists.

7. Click on the arrow next to the Solvent window and select the solvent (e.g. '**DMSO**') by clicking on it.

8. Click on the arrow next to the Experiment window and select '**PROTON.kinetic**' by clicking on it.

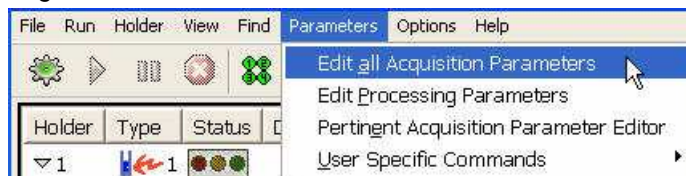
9. Enter a title e.g. **Kinetic experiment**

Figure 5.26.



NOTE: If the delay between the experiments and the number of experiments have been set as default in the AU-programs 'au_zg_kinetic' and 'proc_1d_kinetic', then the experiment can be submitted at this time. If desired, the delay and number of experiments can be changed if the 'Parameter Edit' permission is been granted to the user. For the parameter changes follow the steps below.

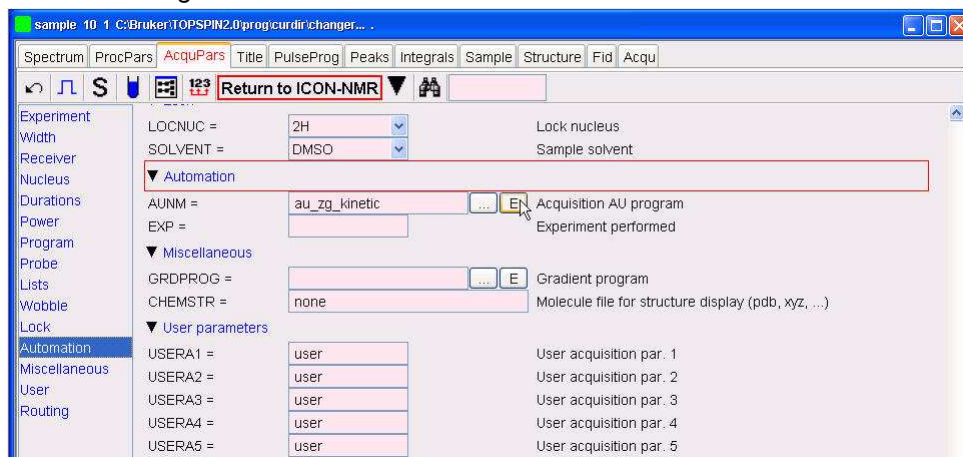
Figure 5.27.



10. Click on '**Parameters**' in the main menu

11. Select '**Edit all Acquisition Parameters**' by clicking on it

Figure 5.28.



Chapter Template

6

Introduction

6.1



Notes: