IconNMR

Software Manual

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Please make use of the report form at the end of the manual for comments, error reports and suggestions.

Special thanks to:	Peter Ziegler
	Patricia Stone Wilkinson and Kimberly Colson for the
	Journey through ICON-NMR Setup Manual
and to	Michael Ackermann and
	Joyce James for the
	BEST-NMR Software Manual

Contents

Chapter 1

Introduction

1.1 What is ICON-NMR?

Welcome to ICON-NMR, a comprehensive user interface, tailormade 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 Unix/Windows NT operating system and Bruker's XWIN-NMR program, providing the acquisition and processing functions, ICON-NMR shields the user entirely from both. Accessing XWIN-NMR 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.

1.2 Hardware/software requirements

Computer and Operating System:
Silicon Graphics: Operating System IRIX 5.3, 6.2, 6.3 or 6.5

Personal Computers: Operating System *Windows NT* Version 4.0 Service Pack 3

Spectrometer: AMX/ARX or Avance

Software:
XWIN-NMR 3.0 must have been installed prior to ICON-NMR 3.0

Release Media:

ICON-NMR 3.0 is available on the NMR-Suite 3.0 CD.

This manual applies to ICON-NMR version 3.0. ICON-NMR contains this manual as on-line documentation accessible from the *Help* menu. ICON-NMR 3.0 can only work if XWIN-NMR 3.0 has been installed prior to ICON-NMR.

ICON-NMR 3.0 provides the *Routine Spectroscopy Desktop* for experiments *without* a sample changer plus the *Automation Desktop* for both sample changer and manual usage.

1.3 Support

Software support is available from your local Bruker office or via e-mail from the following address:

nmr-software-support@bruker.de

The Bruker ftp servers *ftp.bruker.de* and *ftp.bruker.com* provide additional information such as known bugs and their workarounds in the directory

/pub/nmr/ICONNMR or /pub/nmr/mirror.bruker.de/ICONNMR respectively.

1.4 Installation of ICON-NMR

Please proceed as described in the XWIN-NMR 3.0 release letter. ICON-NMR can be separately selected as an installation item from the XWIN-NMR installation dialog

window.

1.5 Starting up ICON-NMR

1.5.1 XWIN-NMR configuration

Before you can start ICON-NMR and begin your work, the laboratory manager must have installed XWIN-NMR 3.0 and ICON-NMR from the release media. In addition, the config XWIN-NMR configuration suite (see the XWIN-NMR manual) must have been executed successfully.

Notes on XWINNMRHOME:

While pre XWIN-NMR 2.0 versions could only be installed in the directory */u*, XWIN-NMR 3.0 can be installed in any directory. When you install XWIN-NMR from the distribution CD on Unix systems, you may either choose *Standard Installation* or *User-Defined Installation*. The standard directory is */u* , the user-defined installation allows you to specify any directory. In this manual the actual installation directory of XWIN-NMR with XWINNMRHOME. All program modules of XWIN-NMR as well as some utility programs such as the Service Tools are located in *XWINNMRHOME/prog* and its subdirectories. All parameter sets, pulse programs, AU programs, etc. are located in *XWINNMRHOME/exp* and its subdirectories. The spectrometer configuration files are located in *XWINNMRHOME/conf* and its subdirectories. The plotter and printer configurations files are located in *XWINNMRHOME/plot* and *XWINNMRHOME/print/* and their subdirectories, respectively.

XWINNMRHOME and /u will be used interchangeably in this manual.

1.5.2 Unix desktop configuration

An ICON-NMR user should have configured his Unix desktop so as to display a Windows overview box in order to avoid X Window Icons to appearing on his ICON-NMR desktop.

1.5.2.1 SGI computers

To enable the Windows overview box on SGI systems, please proceed as follows:
Subsequently click on the menu items *Desktop -> Customize -> Windows*
in the IRIX *Toolchest* and check the button *Display windows overview* in the

upcoming dialog window. Close the window, thereby restarting the window manager.

The SGI 4D window manager allows a user to enter the key sequence ‘ALT + spacebar’ to open a new Unix window even if the Unix desktop is completely covered by ICON-NMR. If an ICON-NMR user should be prevented from accessing any other programs including Unix, this feature must be disabled using the following procedure: Open the file *.4Dwmrc* in the user’s home directory with a text editor, search for the definition of the menu *4DwmRootMenu*, and insert a ‘!’ character in front of the line containing the item *new window*. The exclamation sign will disable the new window feature. In addition, insert a ‘!’ character in front of the line containing the item *<Btn1Down>*, contained in the section *Buttons 4DwmButtonBindings*. If you do not have a *.4Dwmrc* file, you’ll find a sample file in */usr/lib/X11/system.4Dwmrc*

1.5.2.2 Windows NT

Here the Start Menu bar will contain the icons for the respective programs. No changes to the default configuration are necessary.

1.5.3 Start-up methods

Select one of the following methods to start ICON-NMR.

1. **Starting ICON-NMR from Unix/Windows NT level**
   Open a terminal window and enter the command iconnmr.
2. **Starting ICON-NMR from XWIN-NMR level**
   If XWIN-NMR is active, enter the command iconnmr in its command line, or call it from the *Windows* menu using the mouse.
3. **Auto-Start ICON-NMR when logging into the workstation**
   For SGI systems, set up a text file with the name *.sgisession* in your home directory. Insert the command *iconnmr* as the last line of this file. When logging in, the system will execute the file and start ICON-NMR.
   For Bruker AspectStations, the file name must be *.Xsession* rather than *.sgisession*.

1.6 System and user management

The first time any user uses ICON-NMR a default user file will automatically be created allowing full access to the program. It is recommended, however, that the Laboratory Manager create userfiles for all users who will be using the program. All aspects of the software may be configured from the *System Manager* button of the ICON-NMR main window and is described in detail in the *System manager* chapter of this manual.

System and user management comprises the following items:

- Defining the legal spectrometer users
- Defining the experiments a particular user may execute
- Defining a number of permissions for a user, e.g.
  - whether he is allowed to change the parameters of an experiment
  - whether he may exit from *Routine Spectroscopy/Automation* mode to XWIN-NMR or Unix
  - whether he may access the desktop while ICON-NMR is active
  - whether he is allowed to lock and shim a sample manually
  - file name restrictions
- Defining user specific information, such as e-mail address or a costing group
- Defining the disk partitions to store the acquired data
- Defining new *composite experiments*, i.e. several experiments under one name
- Configuring ICON-NMR's Automation module and Default Shim file loader

1.7 Using the mouse or the keyboard

Within ICON-NMR, you can use the mouse or the keyboard to select items and execute commands, and to position the text cursor.

- In order to execute a function represented by an icon or a button or a menu item, either click on it using the left mouse button, or hit the *Enter* key once it is *selected.*
- In order to select the *next* or *previous* icon or a button in a window for execution with *Enter*, use the *Tab* or *Shift +Tab* keys.

- In order to select the *next* or *previous* menu item or list box item, use the *Down Arrow* or *Up Arrow* keys, or the left mouse button.
- In order to move the text cursor within text entry fields, use the *Left Arrow* or *Right Arrow* keys, or the left mouse button.

For Automation use only:

- In *Automation* mode scrolling up and down in the experiment display area may be done with the *Alt + z* and *Alt + x* key combinations respectively.
- Use *Alt + Tab* in the Title window in *Automation* mode to select the next button/ entry. (*Tab* on its own is taken as being a normal *Tab* indent function)

Using Function Keys:

- ICON-NMR does not per default recognize function Key definitions as defined on the workstation. If ICON is to recognize Function Key input you have to create a resource file called _iconnmrrc in the home directory of the user who normally starts XWIN-NMR for ICON-NMR use.

Here is an example of one such resource file which defines F1 for ICON-NMR use. Put both of the following lines in your _iconnmrrc file to be able to use the F1 key in your Title and Originator entries:

bind Entry <F1> {%W insert insert “This is F1”}

bind Text <F1> {%W insert insert “This is F1”}

Chapter 2

The Main Desktop

2.1 Overview

After starting ICON-NMR using one of the procedures described in the *Introduction* chapter of the manual, the ICON-NMR main desktop will appear, showing the icons in Figure 2.1 as command buttons. The following sections present a short description of the assigned functions. Every function will be discussed in detail in the following chapters of this manual.

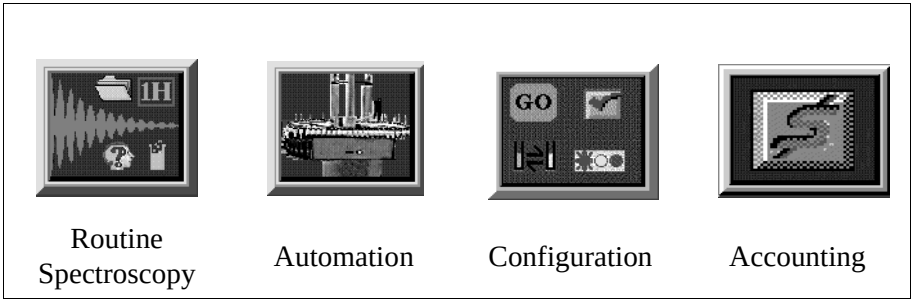


Figure 2.1 Icons of the main desktop

The main desktop may cover the entire monitor screen, shielding the user from the other windows active on the workstation. The desktop has a window manager

frame, which may be removed via the Supervisor permission, making it is impossible to move it aside and access other programs. Should this default setting be too restrictive for a particular user, the laboratory manager can add a window manager frame to the desktop by activating the Supervisor permission in ICON-NMR's *User Manager.*

You can, of course, reduce the size of the initial window so that it only contains the 4 main start buttons as shown in Figure 2.1. ICON-NMR 3.0 retains the size information for future use.

2.1.1 Routine spectroscopy

This ICON-NMR mode was designed to help the user perform standard experiments (provided by Bruker or set up by the laboratory manager) by simply following the steps:

- Insertion of the sample into the magnet (manually, no sample changer)
- Defining a file name
- Defining the solvent
- Defining the experiment
- Starting execution

If not configured differently by the laboratory manager, experiment execution consists of data acquisition, processing and plotting.

Any experiment will generate an entry in a protocol file, which can be for user accounting in the *System Manager* Accounting program.

2.1.2 Automation

This is the extension of Routine Spectroscopy for spectrometers equipped with/ without a sample changer. It may be used in a manual mode without a sample changer as an alternative to the simple *Routine Spectroscopy* interface.

2.1.3 Configuration

Here you may define experiments and user specific properties of the program interface. The Configuration interface consists of a browser style interface with logical pages of configuration information pertaining to the program

2.1.4 Accounting

This utility allows the laboratory manager to to generate accounting reports based on the instrument time used by individual users or groups. It can also be used to generate a list of experiments which were performed on a particular sample. The Accounting prog makes use of accounting files stored in the XWINNMRHOME/conf/instr/<Instrument Name>/inmrusers directory.

2.1.5 Terminating ICON-NMR

The *File* menu provides several methods to terminate ICON-NMR:

1. **Logout** (only available on SGI systems)
   Terminates ICON-NMR, XWIN-NMR, and the current Unix session.
2. **Exit to Unix**
   Terminates ICON-NMR and XWIN-NMR and allows you to continue work in the current Unix session.
3. **Exit to XWIN-NMR**
   Terminates ICON-NMR and leaves XWIN-NMR active and ready for command input. You may exit any ICON-NMR window using the key sequence (ALT-F, E).
4. **Clearing an error situation**
   Should it happen that for any reason your keyboard and mouse seem to be dead and you can't continue work, you should try the following key combination to restart if you are working with an SGI computer: CTRL SHIFT ALT F12 / (press all 5 keys simultaneously). On WINDOWS NT, press CTRL-Alt-Delete and use the task manager instead. If this does not help, but you are connected to a network, you can log into your system from another terminal (using the same id) and execute the command shmrm from the keyboard. If this is not possible or does not help, you must turn off the computer and restart it.

2.1.6 Help

The *Help* menu allows you to access this ICON-NMR manual on line.

Chapter 3

Routine Spectroscopy

3.1 Entering *Routine Spectroscopy* mode

You enter *Routine Spectroscopy* mode by clicking on the respective icon of the ICON-NMR main desktop. A flashing icon will invite you to identify yourself. To exit click on the *Exit* button of the *File* menu.

3.2 User identification

3.2.1 Identify yourself

When you click on the *identify user* icon, a dialog box (Figure 3.1) is opened which displays a list of possible spectrometer users. Select your user id from the list either with a single mouse click, followed by a click on the OK button, or double click on your ID. The program will then request your password. If your ID does not appear in the list, use the User Manager, accessible form the Configuration Menu to create a userfile for yourself.

User ID’s and passwords

The user identification dialog box similar to that shown in Figure 3.1, will contain

the ID's of all users which have both a Unix account on this workstation and a user file defined using ICON-NMR's User Manager window. In addition, the box includes the ID's of so-called *NMR users*. NMR users need not have their own workstation account. Instead, they are defined by the laboratory manager in the *System Manager* mode of ICON-NMR where they are assigned an ID and, if desired, their own password (Unix only).

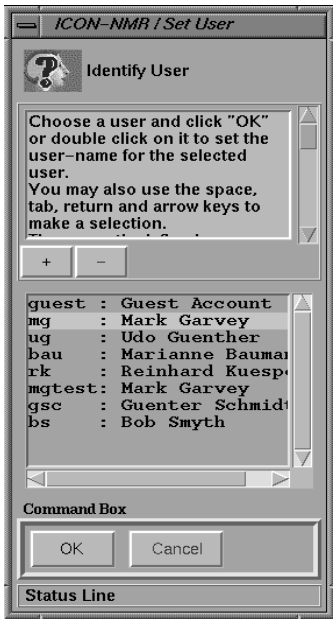


Figure 3.1 User identification

In other words, legal workstation users may enter *Routine Spectroscopy* mode, as well as other people to whom the administrator wishes to grant access to the spectrometer by adding them to the NMR users list.

An NMR user who does not have a password must use the *effective NMR user's* password. This is a legal workstation user on the workstation who was defined to be the effective NMR user by the laboratory manager in the Nmr *User Manager*

mode of ICON-NMR.

The user ID selected to get into *Routine Spectroscopy* mode has a direct influence on the ownership of the acquired data. If your ID is a legal Unix login ID, the data will get read/write access permissions for yourself and all members of the Unix group you belong to. You may alter this using the Umask setting in the User Manager. If you are an “NMR user”, the data access permissions are those of the effective NMR user.

3.3 The *Routine Spectroscopy* desktop

3.3.1 The flow chart

If your identification was successful, the window of Figure 3.2 (left side) will appear on the ICON-NMR desktop containing icons that are arranged as a flow chart in order to guide you in the appropriate way through the set up steps required before the experiment can be started. As you can see from the flow chart, there are only a few operations to perform: Inserting the sample into the magnet, defining a file name, defining the solvent, and defining the experiment. Then you are ready to click on the *start* button to execute the experiment. Before that, you may at any time repeat a set up step to apply any corrections.

The flow chart can be displayed in two modes by clicking the *Status* toggle button. The second mode (Figure 3.2, right side) shows a status panel appended to the chart which allows you to view the parameters defining the current experiment. If anyone of them remains *undefined* (except for the title parameter), you cannot start the experiment. Please note that within the status panel no parameters modifications are possible.

3.3.2 The *File* menu

This menu contains the *Exit* command to terminate the *Routine Spectroscopy* desktop. After successful log in the Exit to Iconnmr button may be used to leave the routine mode without returning to the identify user stage.

3.3.3 The *Options* menu

This menu contains the following commands:

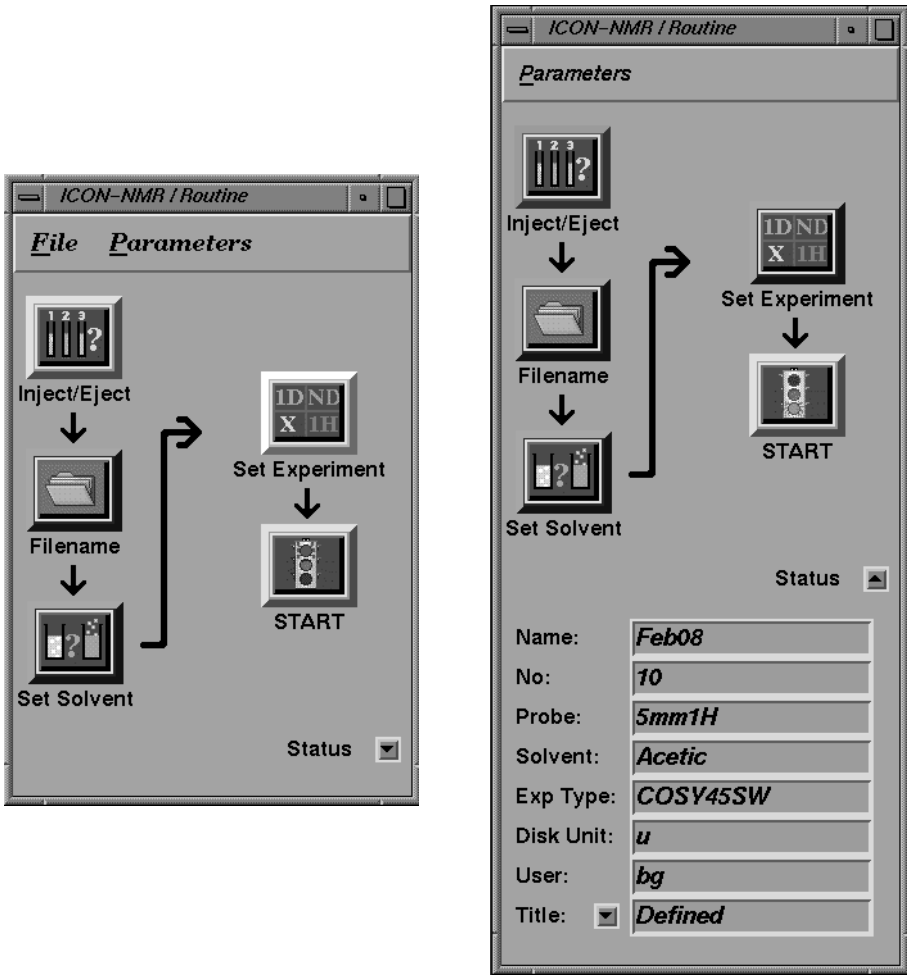


Figure 3.2 *Routine spectroscopy flow charts* without/with status panel

- **Manual Locking and Shimming**
  If the check button *Manual Lock/Shim* is activated, the acquisition software will neither lock the field nor shim automatically. These tasks are left to the user: As soon as the procedures are required after starting the experiment, a dialog box

will invite the user to execute them manually. Manual locking and shimming require a permission to be set in *User Manager* mode.

- **Iconbook** (SGI systems only)
  This command places a number of utilities onto ICON-NMR's desktop. They are presented in form of icons, contained in their own box, e.g. a text editor, a game or anything else available on the workstation. The contents of the box must be configured by the system manager at Unix level. The iconbook is only provided if the laboratory manager enables it in *User Manager* mode.
- **Change password (only available for NMR users)**
  This command allows the current ICON-NMR user to change his password.

3.3.4 The *Configuration* Menu

This menu contains the following commands:

- User Manager
  Direct access to all the User manager features. Note that you may need to Identify yourself a second time before all changes will take effect. See "The User Manager" on page 45..
- Composite Manager
  Access to composite experiment definition for ICON-NMR. See "The Composite Manager" on page 57..
- ICON-NMR CONFIGURATION
  Use this to define global features of ICON-NMR, like the shimming method to be used before Acquisition, or the default increment to use for the experiment numbers. See "Configuration Suite" on page 49. for more details.

3.4 The *Inject/Eject* icon

Figure 3.3 (left side) shows the window which appears when you click on the *Inject/Eject Sample* icon.

The window has 2 different modes of operation *Manual* and *Sample Changer operation*. To use the sample changer mode the unit must have been configured using the cfbacs XWIN-NMR command.

The manual mode consists of a *command sequence* panel, and a command panel with a flashing frame, inviting you to select a command. Choose either *Exit* to

close the window, or *Insert Sample* to turn on the sample lift. A sample residing in the magnet will be lifted, and a dialog box will ask you to insert a new sample. The command sequence panel monitors the actions of the spectrometer during this procedure.

After the experiment has been successfully completed the *Inject/Eject* window will also contain the buttons *Eject + Terminate*, *Use Same/Continue* and *Insert New Sample*. The *Eject + Terminate* button ejects the sample from the magnet, and subsequently terminates the current ICON-NMR routine session. *Use Same/Continue* increments the experiment number by one leaving the sample in the magnet, while the *Insert New Sample* starts the change sample procedure.

3.5 The *Filename* icon

3.5.1 Selecting the data set name

When you have inserted a sample in the magnet, you must define a data set name for the fid and spectrum to be acquired. The dialog box of Figure 3.3 (right side) will be opened as soon as you click on the *Filename* icon. The data set filename follows the conventions of XWIN-NMR, where an fid is stored in the directory

/DU/data/USER/nmr/NAME/EXPNO/

The corresponding spectrum is stored in

/DU/data/USER/nmr/NAME/EXPNO/pdata/PROCNO/

DU is the disk unit or partition where the data will be stored. If for a particular user only a single partition is provided in the *User Manager* mode, DU will not appear in the *Filename* selection box. Otherwise a user may select the desired DU from a list.

USER is the identification code a user typed in to enter *Routine Spectroscopy* mode. It not only defines the location of the data on disk, but also the access rights for the data (cf. section for details).

NAME is a string to identify the data files of a particular sample. A user enters the desired name into the respective text entry field, or selects the name from a given list by clicking on the *Names* button right of the entry field. The list is defined for each user in the *User Manager* mode. In this mode, the *Data Set Edit* checkbutton

can be set, so that a user can only use the names on the list, but not enter his own particular name. Furthermore, a *Spectrum Number* file may be set up which automatically assigns a system wide, unique name for each sample used (see Section 4.8).

EXPNO is a number with the default value 10. The purpose of EXPNO is that a user may keep the same NAME for several experiments, but differentiate the data by another EXPNO. We recommend you increment EXPNO by 10 to prevent a conflict with the EXPNO counting used in *composite experiments*. A composite experiment actually performs several experiments, e.g. a 1D preparation experiment followed by a 2D. ICON-NMR will increment EXPNO by 1 in these cases.

PROCNO is a number with the default value 1. It need not be changed in *Routine Spectroscopy* mode.

3.5.2 The originator information

Several other items may appear in the Filename selection box, such as the Department or the Email address in Figure 3.3. This so-called originator information can be configured in *User Manager* mode by invoking the *Originator Items Editor.* The originator information, if present, will automatically be appended to the plot title. See “The Originator Items Editor” on page 54..

3.5.3 Command buttons

The *Filename* box provides the following command buttons:

- **OK**
  Accept all changes and return to the *Routine Spectroscopy* flow chart.
- **Cancel**
  Discard all changes and return to the *Routine Spectroscopy* flow chart.
- **Continue**
  Accept all changes and carry on with the next item of the *Routine Spectroscopy* flow chart, i.e. open the *Solvent* selection box.

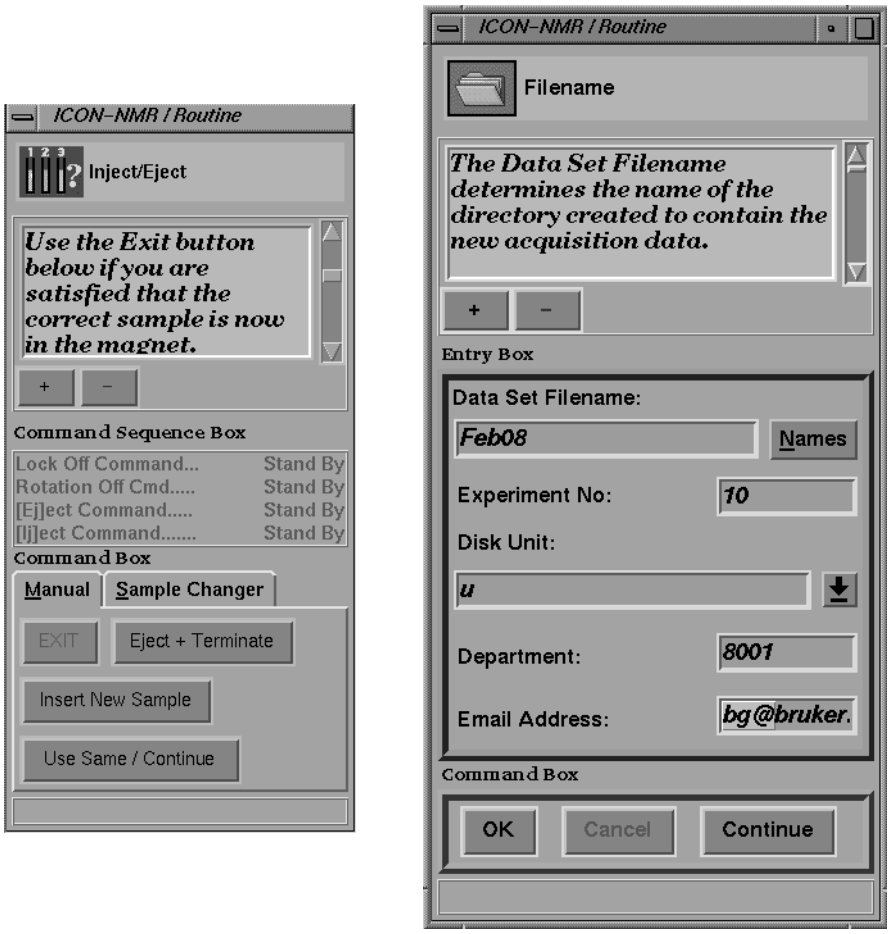


Figure 3.3 *Inject/Eject* and *Filename* dialog boxes

3.6 The *Solvent* icon

3.6.1 Selecting the solvent

The *Solvent* selection box (Figure 3.4, left side) is shown when you click on the *Solvent* icon or on the *Continue* button in the *Filename* selection box. Choose the

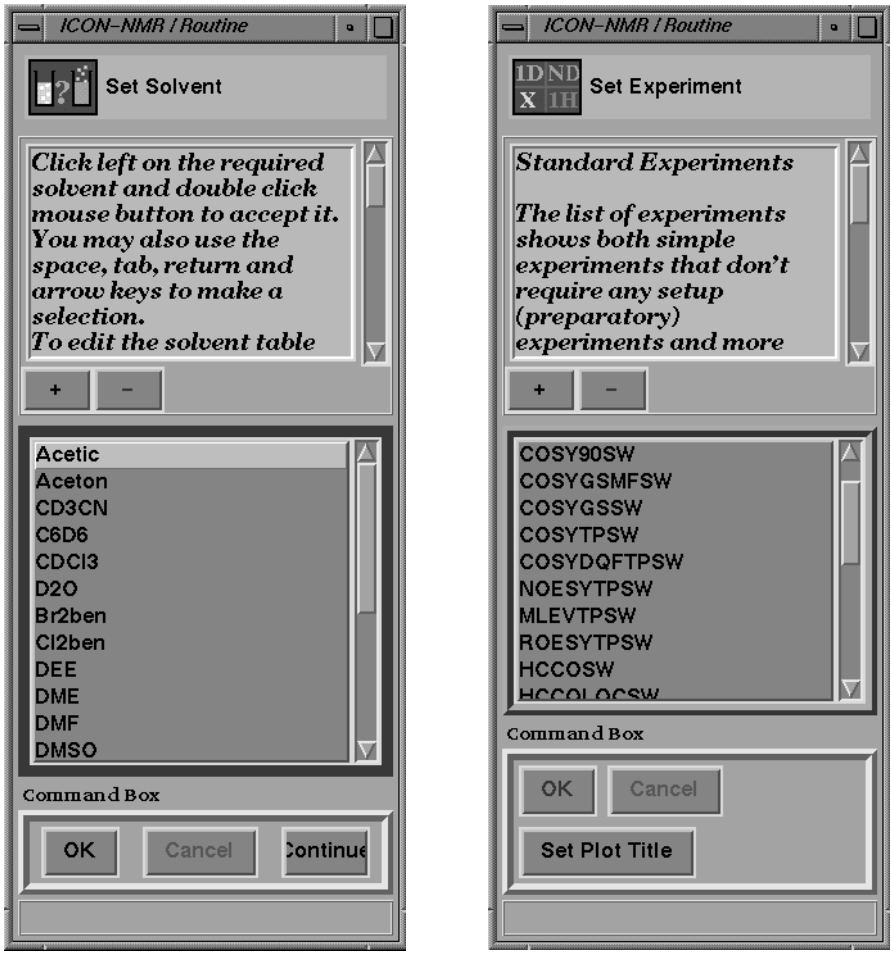


Figure 3.4 *Solvent* and *Experiment* selection windows

solvent for your sample.

The solvent list displayed is the list setup by the laboratory manager with the command edsolv during XWIN-NMR configuration.

3.6.2 Command buttons

The *Solvent* box provides the following command buttons:

- **OK**
  Accept the selected solvent and return to the *Routine Spectroscopy* flow chart.
- **Cancel**
  Discard the selected solvent and return to the *Routine Spectroscopy* flow chart.
- **Continue**
  Accept the selected solvent and carry on with the next item of the *Routine Spectroscopy* flow chart, i.e. open the *Experiment* selection box.

3.7 The *Experiment* icon

3.7.1 Selecting the experiment

The *Experiment* selection box (Figure 3.4, right side) is shown when you click on the *Experiment* icon or on the *Continue* button in the *Solvent* selection box. Choose the desired experiment for your sample.

The experiment list displayed is user dependent and setup by the laboratory manager in *User Manager* mode. See “The Experiment List box” on page 47..

3.7.2 What is an experiment?

Each experiment name in the list is associated with a set of acquisition, processing, and plotting parameters stored in the directory

XWINNMRHOME/exp/stan/nmr/par/<experiment name>/

Bruker provides a pool of the most important experiments with its software, installed at XWIN-NMR configuration time with the command expinstall. The laboratory manager may define additional experiments using the XWIN-NMR eda, edp, edg, rpar, wpar commands.

Some experiments are denoted as *composite experiments* defined in the *Composite Experiment Setup* mode of ICON-NMR. A composite experiment is a sequence of normal experiments which you define in *Set Composite Experiment* mode. The member experiments of a sequence may depend on each other. For example, the

first experiment of a sequence could be a preparation experiment for a subsequent 2D experiment. In such cases, the composite experiment manager allows the laboratory manager to specify the reference experiment(s).

When an experiment is started, a sequence of preparation steps for data acquisition are executed, followed by the acquisition of the fid(s), followed by data processing and plotting. The preparation steps select the preamplifier, adjust the lock, the shims, and the receiver gain, controlled by the TCL script

XWINNMRHOME/prog/tcl/xwish3_scripts/inmr_scripts/qnmr_sx_tcl

The actual data acquisition is performed by the AU program AUNM which an acquisition parameter defined in the experiment. AUNM may be modified by using the eda parameter editor before experiment start.

For processing (Fourier transform, phasing, integration) and plotting, the tcl script calls the AU program AUNMP, which is a processing parameter of the experiment. The AUNMP parameter may be modified with the edp parameter editor.

3.7.3 Command buttons

The *Experiment* box provides the following command buttons:

- **OK**
  Accept the selected experiment and return to the *Routine Spectroscopy* flow chart.
- **Cancel**
  Discard the selected experiment and return to the *Routine Spectroscopy* flow chart.
- **Set Plot Title**
  A text entry window is opened where you can type in one or several lines which will become the title of the spectrum plot. The title window will already contain the originator information if defined. When you terminate the window by clicking on its OK button, the title text will be saved in the following file where it is looked up by the plot software:

/DU/data/USER/nmr/NAME/EXPNO/pdata/PROCNO/title

3.7.4 Modifying parameters

After an experiment has been defined, its acquisition, processing, and plotting

parameters are initialized from the standard parameters for this experiment. Using the commands of the *Parameters* menu, the parameters can be changed before the *Start* icon is activated. The standard parameters sets will not be affected. Parameter modification requires the governing permission checkbutton to be set in *User Manager* mode.

3.8 The *Start* icon

Once everything is setup correctly, i.e. the sample is in the magnet, file name, solvent, experiment, and plot title are defined, the traffic light in the START icon will turn green and you may the activate the *Start* icon to launch the experiment.

3.8.1 The *acquisition in progress* window

After starting the experiment, the flow chart will be overlayed by the acquisition in progress window (Figure 3.5) containing the file name and EXPNO, the remaining time the experiment needs to finish, the experiment name and its comment line. The figure shows EXPNO=31, which in this case indicates, that a composite experiment is in progress: The 1D reference experiment had EXPNO=30.

The window provides the following command buttons:

- **FID**
  Displays the fid being acquired in real time.
- **Spectrum**
  Displays the spectrum being acquired in real time.
- **Lock**
  Displays the lock signal in real time.
- **Halt**
  Halts data acquisition when the current fid is complete and stores it on disk for further processing using the *Plot* button.
- **Plot**
  Processes the fid stored on disk when *Halt* was pressed and plots the spectrum. In fact, the AUNMP AU program is called for this purpose. If processing has already been carried out, this button merely plots the spectrum afresh.

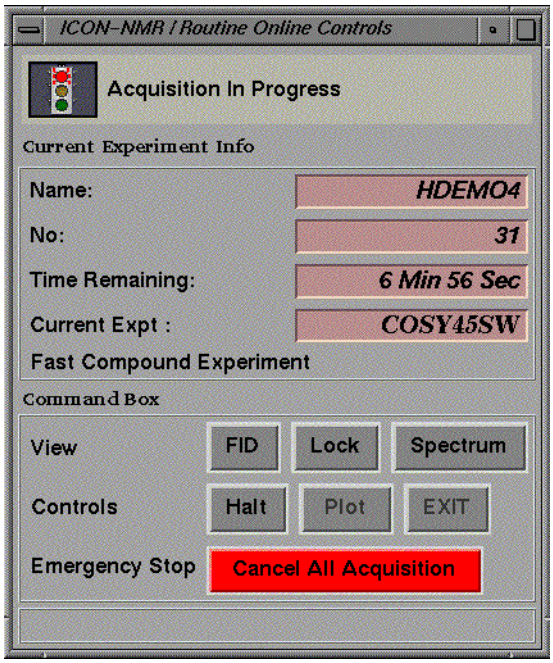


Figure 3.5 Acquisition in progress window

- **Exit**
  Returns to the flow chart. This button is not active while the experiment is in progress.
- **Stop**
  Stops all spectrometer activity immediately. No valid data will be available afterwards.
- **Search**
  After an experiment has been completed this button may be used to look at previously acquired spectra.

3.8.2 The acquisition status icons

These icons allow you to observe the current experiment status. Whenever the program begins to execute a particular procedure, the corresponding status icon will

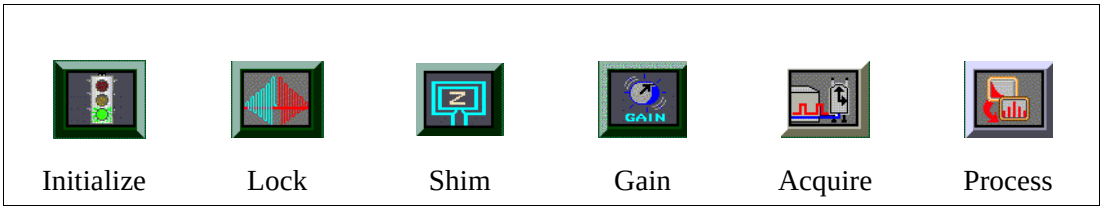


Figure 3.6 Acquisition status icons

blink until the task has been completed. When processing is finished, the exit button will be enabled. Using this button will return control to the flow chart for setting up the next sample.

3.8.3 The accounting file

Every experiment started in ICON-NMR will generate an entry in the accounting file

/XWINNMRHOME/conf/instr/<spectrometer>/inmrusers/Inmracct.brief and

/XWINNMRHOME/conf/instr/<spectrometer>/inmrusers/Inmracct.full

The .brief files contain only essential information and are shorter.

Table 3.1 shows the initial section of such a .full file which reports the spectrometer users and the experiments they performed, including the time and disk space requirements. The file will grow with time, and it is the responsibility of the laboratory manager to archive and delete it periodically. Such accounting files are the basis for the *accounting manager* provided by ICON-NMR which generates an invoice for any user, user group etc. based on unit prices per hour of spectrometer time and Megabyte of disk usage.

```
-------------------------------------------
----  IconNmr Accounting File
----
----  Created Tue Jan 23 12:31:50 MEZ 1996
-------------------------------------------
#Login
name: mg1
host: bigi
group: user 20
date: 01/23/96 12:32 822396721
-------------------------------------------
#Spectrometer: DPX250
-------------------------------------------
#Experiment
nameOfExperiment: PROTONNREXP
solvent: Acetic
fileName: Mark2076 40 1 u mg1
fileSizeAcq: 32768
fileSizeProc: 16384
NUCLEUS: 1H
INSTRUM: DPX250
timeOfStart: 01/23/96 12:32 822396744
timeOfTermination: 01/23/96 12:33 822396824
#User Specific Info
Email Address: mg@bruker.de
-------------------------------------------
#Logout
date: 01/23/96 12:33 822396838
-------------------------------------------
```

Table 3.1 Section of an accounting file

Chapter 4

Automation

4.1 Entering *Automation* mode

Enter *Automation* mode by clicking on the respective icon of the ICON-NMR main desktop. A identification window will invite you to identify yourself. To return to the main ICON-NMR desktop click on the *Exit* button of the *File* menu.

4.1.1 User identification

See “User identification” on page 11 for more details on identification requirements.

4.2 The Automation Setup Window layout

After successful user identification, the user interface shown in Figure 4.1 will appear on the ICON-NMR desktop showing an array of sample holders. The size of the array will depend on what type of sample changing mechanism you have installed on your instrument see ’Default Number of Sample Holders’ on page 64. The setup window shows all samples and their respective status in the system, Available, Queued, running completed or error. You’ll find control buttons to add/change/delete experiments from the setup window experiment list, a history list

giving information on what the instrument actually did and various other control buttons.

The setup window is the Automation control center. It is the master scheduler for the instrument. It consists of the following items:

- **The window manager bar showing the directory where the automation information is to be temporarily stored**.
  This is allocated afresh each time the Automation program is started. A directory is formed based on the time/date the program was started on and the username of the operator who started the program. This directory is used for temporary purposes only and, if the setup is not explicitly saved, it will be deleted when the program exits. The directory resides in XWINNMRHOME/prog/curdir/changer/inmrchanger.
- **The main menu bar with pull down menus for Automation operation.**
  Most of the commands in the commands in this menu may be accessed quickly using keyboard accelerators. For example <*Alt-f, c*> closes the automation setup window. This is the same as clicking with the mouse on the *File->Close* menubutton. The setup window is designed to be suitable for keyboard or mouse interactions at all times. That way you can speedily set up new experiments without having to jump back and forth between keyboard and mouse operation. See “Using the mouse or the keyboard” on page 5.
- **The setup Icon-Bar.**
  These are buttons exclusively for mouse use to quickly access basic setup window features, e.g. Start/Stop Automation, show the sample holder overview, show more detailed experiment information etc.
- **The experiment display area consisting of a hierarchical listbox of experiment entries**
  Hierarchical simply describes the way in which the three levels of information shown in the listbox are stacked above each other. At the top are the sample holders, then come the experiments followed by any reference experiment information, Figure 4.1 Holder 11 is at the top or the hierarchy. It has 2 experiments (shown by the type icon). These 2 experiments are lower down in the hierarchy, they belong to holder 11, the COSY experiment having a reference spectrum information line which is at the bottom of the hierarchy, it belongs to the COSY experiment. Double clicking on a list entry folds it up or down to reveal information already entered or entry boxes for addition of further experiments.

- **The experiment queue control buttons**
  The buttons in the center of the setup window always apply to selected entries (marked in blue) in the experiment listbox area. Use them to submit experiments to the queue, remove experiments from the queue, edit previously cancelled experiments, delete experiments or for addition/copying of experiment information. Their operation will be described in detail later.

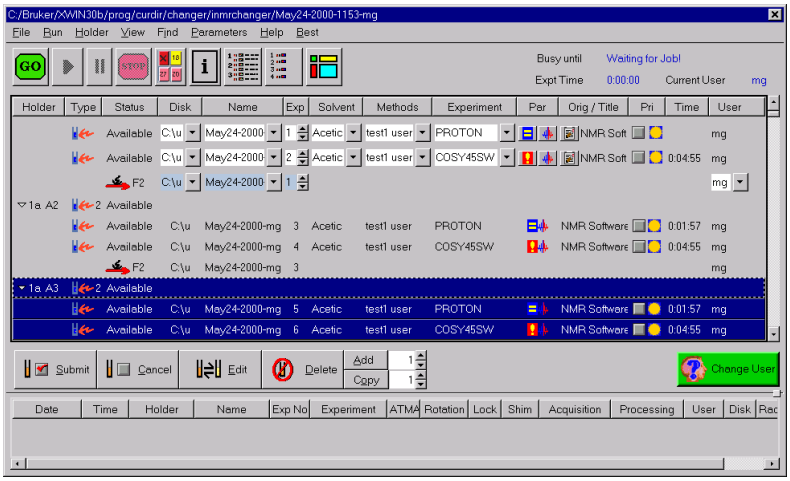


Figure 4.1 Automation Setup Window

- A history box showing details of all experiments performed

Here you'll find detailed information on completed experiments no longer in the queue. The 400 most recently performed experiments will appear here. The various stages of acquisition are shown; tuning/matching, rotation, lock, shim, acquisition and processing. Successfully completed stages are shown with a tick whereas stages where any problems were encountered are marked in red with a cross. The sample change is currently not shown, however, if no ticks or x's appear this implies that the sample change was unsucessful.

Double clicking on any entry in the history area of ICON-NMR's setup window,

will cause XWIN-NMR to appear showing the data set which was clicked on.

Note:

More detailed information on what the Instrument actually did may be attained by using the XWIN-NMR history feature. Ascii files containing all automation commands are then stored in XWINNMRHOME/prog/curdir/<user who started XWIN-NMR>/history. You may activate this feature via the Display->Status & history->On for all data sets menu button in XWIN-NMR.

Once you've come to grips with the overall layout of the setup window the next step is to actually perform some experiments.

4.3 Automation Quick Start Guide

Here you'll find all you need to know to get the automation up and running a.s.a.p.

4.3.1 First steps in ICON-NMR Automation

As is the case for Routine Spectroscopy Mode the XWIN-NMR software must be installed and configured correctly before commencing automation. See "XWIN-NMR configuration" on page 3 for more details.

To get your first automation run underway proceed as follows:

1. Start ICON-NMR from the Windows->ICON-NMR pull down in XWIN-NMR.
1. Click on the Automation Icon.
2. Identify yourself to the system. If you do not see your user id in the identify user window, you or the laboratory manager should install a user file using the *User Manager* form the *Configuration* Menu. See "The User Manager" on page 45 for more details.
3. Once you've successfully logged in, the Automation setup window should appear containing the appropriate number of sample holders. If you see the incorrect number of holders in the setup window in the Holders column then either:

a) You have not configured your sample changer correctly, e.g. for BACS do cfbacs in XWIN-NMR or

b) You must set the number of default holders using the Configuration Manager. This will be necessary in *Manual* and *Sixpack* or *Mas* automation modes.

See “Default Number of Sample Holders” on page 64 for more details.

4. Choose the particular holder you’re interested in and

a) Double click with the left mouse button on the holder *or*

b) Click on the holder and use the *Add* button <Alt - a>.

5. Once you’ve set the experiment details, go ahead and *Submit* it.
After an experiment has been submitted you should no longer make modifications to it. Should you wish to change something then you should first *Cancel* the experiment, then *Edit* it and finally *Submit* it again. The safe way to change parameters on an experiment is first to *Cancel* the experiment, use the particular parameter editor and *Submit* the experiment again.

6. You may add further experiments on that sample with the *Add* button as required.

7. Click on the Go icon to start the run. The program may also be configured to start the run automatically. See “Configuration Options” on page 49.

4.4 Component Descriptions

4.4.1 The experiment entries

Each sample holder line in the setup window consists of 12 columns of information classified as follows:

1. Holder
A little triangular symbol and number indicate the current holder. The triangle shows whether individual experiments are displayed or not.

2. Type
Shows whether a holder line has any information and if so displays the number of experiments defined for that holder. An empty tube icon depicts an empty holder. To add an experiment to an empty holder either

a) Double click on it or

b) Hit Return on the anchored[1] entry.

3. **Status**
   This may be configured to show traffic lights in two different modes or text. The mode can be changed by using the Configuration user interface. See “Configuration Options” on page 49. The following experiment states are currently supported:
   - •Inactive
   - •Submitted to Queue
   - •Running
   - •Completed
   - •Error
4. **Disk**
   Disk directory where experiment is to be stored. User selectable or definable form the *User Manager See “The Partition Names box” on page 52*. The system may thus be configured to allocate the disk partition automatically.
5. **Name**
   Name of experiment. This may be entered by the operator or defined to be set automatically from the *User Manager. See “The Sample Names box” on page 51*. The system may thus be configured to allocate the name automatically.
6. **Exp No**
   Experiment number to be used for this entry. This will automatically be allocated to the next free experiment number based on the current user and the disk and name the user has chosen. If the operator has the Data Set Edit permission he may allocate the experiment number himself.
7. **Solvent**
   The solvent may be set up here as defined at XWIN-NMR configuration time by the edsolv command. Changing the solvent for any component experiment of a holder changes all other solvents for non-submitted experiments automatically.
8. **Experiment**
   Here the operator may choose the experiment from his pre-defined list. This list

1. This is the entry with a dotted line border. May be moved by mouse clicking on another entry or by using the keyboard accelerators <*Alt* + *z*> for up and <*Alt* + *x*> for down.

is defined with the *User Manager*. See “The Experiment List box” on page 47. The experiment type, name and comment are given here. There are presently two types of experiment normal (single) N or n and composite C or c. Lower case characters imply that the experiment will be forced to run in the low priority “night-time” mode. If a composite experiment is chosen then new experiment entries will be added as required for all components of the composite experiment.

9. **Method**
   This column is only available if the program has been configured to run in BEST mode. Depending on the chosen solvent the corresponding methods as defined in bestadm Methods will appear in the pulldown menu. This information used not be sample dependent, but now that the BEST utility has the capability of changing solvents the introduction of a solvent specific method, makes the system even more flexible.

10. **Par**
    This column currently consists of 2 buttons controlling the User Specific Parameters and the Lock/Shim/ATM settings for the individual probe/experiment respectively.
    User Specific parameters may be accessed directly from this column, provided they have been defined for the respective user, see Figure 5.2.1.10. Changes in this window will be copied to all copied entries. If the Modify Lock/Shim/ATM permission has been set in the User Manager, then the current settings for Lock, Shim as well as the experiment dependent ATM mode may be altered as necessary.
    The Lock options is treated on a holder basis and not on an experiment basis. The Shim option may be set up such that it is possible to shim on every experiment. For this option set the “Shim the sample” option in the configuration window’s Shimming Controls page to “on every experiment”.

11. **Title**
    The title icon when activated pop’s up a text entry box which may be used to enter a specific title for later plotting. Originator information items (if activated) are also to be entered here. See “The Originator Items Editor” on page 54. The title information will automatically be copied to all other experiments added on a particular sample. The title icon changes after text has been entered. Use the Set & Copy Title button to force all other title entries (those on-edit for the current sample) to set their entries to the same values as the current title box.

12. **Priority**
    Users who have this option enabled in their user permissions file may decide to prioritize certain experiments. Such experiments will then be run before non-prioritized experiments. The priority will be based on the time of their submission. Such experiments will either be run after the current experiment is completed or when all experiments scheduled on the current holder have been completed. For more details see 'Priority Options' on page 72. ICON-NMR also has a Day/Night mode which may be used to ensure that time consuming experiments are always run at night. Click on the Sun icon and it will toggle to a moon icon, this will classify the experiment as lower (night-time) priority.
    Note the day night mode only applies to each experiment. If you want the sample to be measured at night you must mark all its experiments as being "night" experiments. If you'd like to run one experiment during the day and another at night then simply mark one as day and one as night.
13. **Time**
    The experiment time (estimated acquisition time) is calculated each time an experiment is submitted and also after any parameter editor has been used. This feature may be deactivated to save some processor overhead.
14. **User**
    The user who entered the experiment on the experiment list is denoted in column 12. Note: Experiments belonging to other users may not normally be copied, deleted or altered in any way. If you want to have complete access to other users experiments, change user to the nmr super user.

4.4.2 Queue Control buttons

You'll find an array of queue control buttons in the centre of the Setup Window:

All of the following buttons apply to the selected holder/experiments. Selected experiments have a light blue background to distinguish them. If only the holder is selected (no experiment information selected) then the button's function will be carried out on all of the experiments in that holder. Of course you can also mark particular experiments.

- **Submit <*Alt* + *s*>**
  Submit generates all data necessary for the experiment, calculates the experiment time and sets the status of the experiment to ready. If the run is active the experiment will be found and executed. Submitting an experiment removes the editable boxes and substitutes them with fixed text elements which may no

longer be edited. You may only submit an experiment if all the required information required to start the experiment is available.

- **Cancel <*Alt* + *c*>**
  All of the selected experiments will be effectively removed from the queue. This button is useful together with the edit button if you want to make changes to the experiment before run-time. Note you can always re-submit a cancelled experiment, the information content will not be removed. The experiment is merely ‘deactivated’.

- **Edit <*Alt* + *e*>**
  This button re-inserts the editable boxes on an experiment entry. These may then be edited in the usual manner. Use in conjunction with the cancel button to change previously submitted experiments.

- **Delete <*Alt* + *d*>**
  Removes all experiment information from the display and from the queue. Does not remove any spectral information from the target disk. As mentioned above to remove all experiments on any sample holder mark the sample holder the entry where the holder number is indicated. To remove a single experiment select that experiment only.

- **Add <Alt + a>**
  To add an experiment to a holder with none (empty tube icon) double click on the holder or use the Enter key (once the holder has the anchor. See “Hit Return on the anchored entry.” on page 32). If the holder already has at least one experiment use the *Add* button to create more experiments on that holder. The spin box to the right of the *Add* button determines the number of experiment lines to be added. These lines will automatically receive a valid disk, name, experiment number and solvent entry. The only information you have to enter is the experiment type.

- **Copy**
  There are two methods of copying a holders experiments to another holder:

a) **Sequentially**
All experiments on a particular holder are copied to the next (empty) N holders. This is the default method. Proceed by selecting the holder you wish to copy. Use the spin widget to the right of the *Copy* button to set the required number of copies. All experiments will be copied to the next N holders in the display area. The expno is incremented to the next free expno which is a multiple of 10. Should you wish to keep the sane experiment numbers and increment the Name of the experiment; e.g. Jan-19.001, Jan-19.002 set the *Holder->Increment Name* menu entry before copying. The experiment number will, in this case remain constant.

b) **Targeted**
The anchored holder's experiments are copied to every other selected holder. For more information on how to identify the anchored holder see page 32. This method is automatically selected if more than one holder has been selected (blue background). The anchored holder's experiments are copied to all other selected holders. The same numbering strategy is applied as in the sequential case.

When the copy routine gets to the end of the setup window the copy is wrapped around to the beginning of the table.

- **Change User**

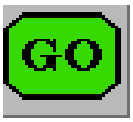
Click on this icon should you wish to login/logoff the system. The login user determines what experiments and environment the operator will have. The border color of this icon turns red when no user is currently logged in. You may also use this Icon to lock ICON-NMR. When ICON-NMR is in the locked mode no changes may be made to the current setup. It is also impossible to leave the program. If you want to alter Experiments entered by other users login as the NMR super user.

4.4.3 Setup Window Icon Bar

The following icons appear under the menu bar and may be used to access particu-

lar features with the mouse.

The Automation ‘Go’ button



This button is responsible for starting the run. It has the same effect as the *Run->New Run* menu command. Once activated a popup dialog will appear as shown in Figure 4.2.

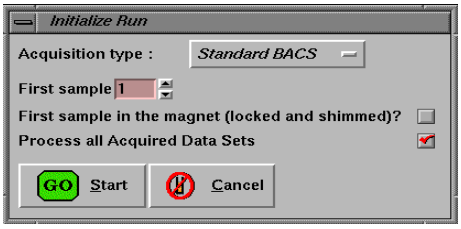


Figure 4.2 Initialize Run Dialog

The Initialize Run Dialog is displayed each time the run is started, unless the Laboratory manager has configured the system to start the run automatically at start up time. See “Automation Driver Engine” on page 65.

The *Acquisition Type* field refers to the type of sample changer Driver which is to be used in the system, BACS, Sixpack, MAS or Manual mode (no sample changer). These drivers are TCL/TK scripts and may be adapted to suit your own environment. You’ll find them in:

XWINNMRHOME/prog/tcl/libtix/iconnmr/inmrlib/Automation/driver_lib

Drivers

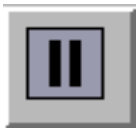
Driver files have the extension .drv, there’s also a file *drivers* which is used to tie a comment to a particular driver file. This comments appear in the Initialize Run window.

The *First Sample* entry tells the system where to start, i.e. which holder will be run first. With the ‘*First sample in the magnet (locked and shimmed)*’ it’s possible to

tell the system to skip the locking and shimming routines for the first sample.

As a further option you may instruct ICON-NMR to skip the processing on all the experiments it performs. This question may be deactivated from the ICON-NMR Configuration window.

The Automation ‘Pause’ button



Use this button to pause the acquisition. This action will take effect after the current running experiment has been completed.

The Automation ‘Play’ button

As you might expect, the play button sets the Run in motion again.

The Automation ‘Stop’ button

This cancels the current run. It will also stop the currently running experiment. If you wish to stop the run without interfering with the currently running experiment use the pause button instead. All experiments which have not yet been completed remain in the queue and are still available for running at a later date. The Stop permission is user specific, only the user who started the run or the NMR Super user (password required) may stop it.

The Sample Holder Overview

Use this button to call up the window shown in Figure 4.3. This window lets you see at a glance what the sample changer has been up to during the automation. It shows all sample holders in a grid array. As in the history list if an error occurs on any sample a cross appears in that particular holder.

The sample holder overview may also be used to position the setup display at a particular entry. Just click on the holder you're interested in. You can even open an entry on a previously unused holder by double clicking on it. You'll also notice that holders in the overview status may be selected in the same way as the holders in the setup window. Click on the first holder you wish to mark and drag the mouse. All following sample holders will be selected (marked in blue) in both the overview and setup window. If you wish to select holders, which are not marked sequentially (e.g. for a targeted copy), hold down the Ctrl button before clicking on the particular sample holders. Don't forget to select the 'source' holder for the targeted copy as the last element to make sure it's the anchored entry.

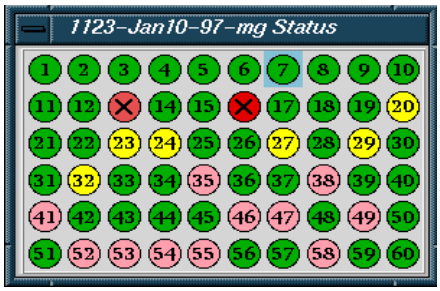
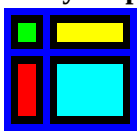


Figure 4.3 Sample Holder Overview

Tidy Display



ICON-NMR makes full use of multiple windows to display as much or as little information as is required. This can leave your desktop looking rather cluttered. Use this button to move and resize all windows to a standard setting. This is the setting which was saved last time the program was terminated.

Acquisition Controls Window

This button displays the Automation Control window, provided it has been enabled in the view menu. The button acts as a toggle. If the window is not viewable it will appear. If the controls window is on the display, pushing this will remove it.

4.5 Menu Bar commands

4.5.1 The File Menu

The file menu contains the following items:

- New

You may start a second experiment setup window and setup further experiments to be run at a later date.

- Open

Open reads in a previously saved experiment setup into the current setup window. The history information is also loaded.

- Save

If you’re going to perform all the experiments again it might be a good idea to save a copy of all the experiment information stored in the setup. The ‘Save’ button will do this for you. It saves all the data in an ASCII format which can easily be retrieved or modified using any text editor. The save button will save this file in the directory XWINNMRHOME/prog/curdir/changer/<setup directory name>.set

ICON-NMR saves this file automatically for you. See “Automatic Setup File Save” on page 64.

- Print (List Setup)

This prints a copy of the Setup file in an easy to read format on the default printer. On Windows NT the notepad program is used to print the file. Use the Edit->Set Font switch to change the font (if necessary).

- Print History File

Prints the history information at the bottom of the setup window to the default printer. On NT you may have to set the default font for the notepad program to obtain reasonable results. Should you have problems you can always print the file directly from the prog/curdir/changer/inmrchanger directory.

- Close

Closes the setup window and returns to the Identify User mode.

- Close All

Use the close all when you wish to close several setup windows in one sweep.

4.5.2 The Run Menu

These menu items may be used in the same manner as those already described in section 4.4.3.

4.5.3 The Holder Menu

Selection Menu buttons

Items in the upper half of the holder menu may be used to quickly select several items at once. For example you may wish to cancel all successfully completed experiments in one swoop and then delete them. In this case you'd use the *Holder->Select Completed* menu option, followed by the *Cancel* button and *Delete* button.

Copy Mode

Here you can decide how ICON-NMR Automation is to copy experiments. *Increment Expno* is the default setting and should be optimal for most purposes. Here any number of experiments will be copied. The system checks and automatically allocates sensible experiment numbers, while keeping the current Name setup.

The '*Increment Name*' mode keeps the same current experiment numbers while

adding numbered extensions to the name to make new unique data names.

4.5.4 View and Find menus

These contain various menu buttons which control which elements of the setup window are to be shown. You may use them to improve the clarity of all display information. The find pull down helps you quickly find particular types of entries. This may be useful if in certain circumstances you have so many entries that you can't see the wood for the trees.

4.5.5 Parameter Menu

As in Routine Spectroscopy mode you can use the parameter menu buttons to access any of the parameter editors which XWIN-NMR has to offer. If the laboratory manager has configured the operator so that he/she can execute specific commands, e.g. lockdisp then these will also appear in the *User Specific Commands* pull-down. See "User Specific Parameters/Commands" on page 53. These buttons apply to the entries in the setup window which have been selected.

Note: Should you accidently hit Edit Parameters when multiple experiment entries have been selected which you do not wish to edit/view, you may cancel the operation by hitting the Esc key while the keyboard input focus is in the setup window.

4.5.6 Best Menu

This is only present if ICON is running in BEST mode.

Show BEST Monitor causes the BEST driver to display its debug window showing all commands sent to the Gilson Liquid Handler, replies and other information.

Show provided solvents displays the BEST solvent editor's provided solvents page, normally accessible from the bestadm XWIN-NMR command. On start-up BEST may require that you use this window to set the correct port for a specific solvent or set the solvent for a particular wash rack.

4.6 Automation Window Tips and Tricks

Here are some features of the Automation window which might not otherwise be apparent.

- To raise XWIN-NMR in any previously measured data set, double click on the corresponding entry in the history (lower) part of the setup window
- Use Ctrl-x and Ctrl-z to move the selection anchor up and down in the setup list
- Use Ctrl-Tab to move the focus out of the title window
- You can change the size of individual columns in the setup window by dragging the mouse with the left mouse button depressed on any column header
- The Delete Key deletes marked experiment setup entries
- The Insert key adds an entry at the anchor position
- The Esc (Escape) Key breaks out of any loop action; e.g. Copying, Submitting, Deleting multiple entries

4.7 External Setup

The Automation module of ICON-NMR 3.0 may be used in external setup mode. This means that all the submitted entries are inserted into the setup window for processing without the need for any direct keyboard input to the setup window.

The entries are created via an ASCII file which must be copied to the XWINNMRHOME/prog/tmp directory. ICON-NMR *Automation* scans this directory periodically (once a minute) for new “setup” files. All you need to do is logon to the ICON-NMR *Automation* software as any valid user.

This directory should be made writable for all those XWIN-NMR users who will be starting the run/copying the files, if external setup mode is to be used. The format of this file is exactly the same as it was for the set/run Automation routines.

Here is an excerpt from the XWIN-NMR manual on external setup:

4.7.1 Acquisition parameter setup with extset

Laboratories often employ a centralized sample management, which requires that the experiments are not defined locally on the NMR spectrometer with ICON-NMR, but rather on PCs or a central laboratory computer, from where they must be transferred into the spectrometer via the network or via magnetic storage media. ICON-NMR offers the following means for accomplishing such tasks:

The experiments for each sample may be defined in a text file in ASCII format, e.g. on a remote computer. If ICON-NMR should execute such an experiment, the file must be copied into the directory XWINNMRHOME/prog/tmp/ of the spectrometer computer (e.g. using rcp or ftp in an Ethernet network).

ICON-NMR periodically (every 60 seconds) checks the directory XWINNMRHOME/prog/tmp/ for new ASCII experiment files. Any new file will automatically be inserted into the desired setup window. The result is the same as if the user had entered the information directly on the spectrometer.

Once entered into the setup file, the ASCII file is deleted from the temporary directory XWINNMRHOME/prog/tmp/. It is therefore the user's responsibility to keep a backup copy of this file, if at all. If several ASCII experiment files are found by extset, they are processed in the order of their file name extensions NNN.

An experiment is defined by the keywords HOLDER, NAME, EXPNO, SOLVENT, EXPERIMENT, TITLE.

They correspond exactly to the fields of the ICON-NMR setup window. NAME, EXPNO, and TITLE are optional, i.e. they may be omitted. In that case ICON-NMR uses standard default values: EXPNO=10; no title; NAME is formed from the name of the experiment file, the extension being replaced by the HOLDER number. Only experiments of type N (no preparation experiments required) or C (composite experiments) are allowed.

Figure 4.4 illustrates the main Keywords used in ASCII experiment files. Lines starting with the character # are comment lines, which are ignored. The file consists of a series of keywords (one per line) combined with information.

Features
1. No limit to the number of experiments per holder.
2. The first experiment on a holder can be a composite experiment.
3. Each experiment can have an individual title. Titles can have more than one line. The line separator is the \n sequence. If several experiments are defined for one holder, each experiment can have an individual name (NAME) and experiment number (EXPNO).

When ICON-NMR sees a new extset file it will normally read all the information in it and enter the experiments in the setup and submit them. If you do not wish the created entries to be submitted then you should include the Keyword NO_SUBMIT in your extset file before the EXPERIMENT keyword line. With the

Keyword SUBMIT_HOLDER you can submit all experiments for a particular holder at a later stage. The SUBMIT_HOLDER keyword requires a preceding HOLDER and EXPERIMENT keyword to be recognized.

There are also new keywords DELETE and PRIORITY which may be used to delete all experiments for a given holder and to set the Priority flag for a given experiment respectively.

#	Comment line
USER	data set USER parameter
HOLDER	Holder number to be used
NAME	data set NAME parameter (optional)
EXPNO	data set EXPNO parameter (optional)
SOLVENT	solvent name
EXPERIMENT	experiment name (of type N or C)
METHOD	method name e.g. methodname user (for BEST)
TITLE	rest of line is the plot title (optional)
DELETE	deletes all experiments for a holder
PRIORITY	enables the priority flag for this experiment
NO_SUBMIT	the entry will appear in the setup list but will not be submitted
SUBMIT_HOLDER	all entries for the defined holder will be submitted, no new entries are created
END	end of holder definitions section

Figure 4.4 Keywords used in Ext Set files

The following example ASCII text file summarizes the ext set file features:

```
# in 1. column means: comment
USER eng
HOLDER 8
#
NAME June15
EXPNO 10
SOLVENT CDCl3
EXPERIMENT sw_cosy45
TITLE This is a title\nwith two lines
TITLE This is a second title for the first exp. of the comp.
TITLE This is a third title for the second exp. of the comp.
NAME June16
EXPERIMENT PROTON
TITLE This is a title\nwith two lines
EXPERIMENT C13CPD
TITLE This is a title\nwith three lines\nThird line
#
HOLDER 9
NAME June17
PRIORITY
SOLVENT CDCl3
EXPERIMENT PROTON
TITLE This is a default title
EXPERIMENT C13CPD
TITLE This is a default title
EXPERIMENT C13DEPT45
TITLE This is a default title
#The next two lines delete all experiments for holder 10
HOLDER 10
DELETE
# Only one END statement per complete file !
END
```

Chapter 5

Configuration Suite

5.1 Overview

The Configuration Suite (Figure 5.1) is started from the respective icon in the main mode of ICON-NMR. You may also use the *Configuration Suite* menu in *Routine Spectroscopy* or *Automation* modes. This window may only be activated if you know the NMR Super User password or if password checking has been disabled.

Configuration Options

The configuration suite is used to configure all aspects of ICON-NMR. This user interface is intended to grow with the ICON-NMR program. At the moment the Configuration Manager is divided into 14 pages: User Settings, Additional ICON-NMR Users, Originator Items, Composite Experiments, Automation Window, Driver Engine, Automatic Tuning/Matching, Priority, Shimming and General, Temperature Handling, LC-NMR Options, LIMS Options, Barcode Printing Routines and General Options. Not all of the configuration options apply to all Automation modes. For example the Temperature Control options are of no value if you only have a sixpack sample changer.

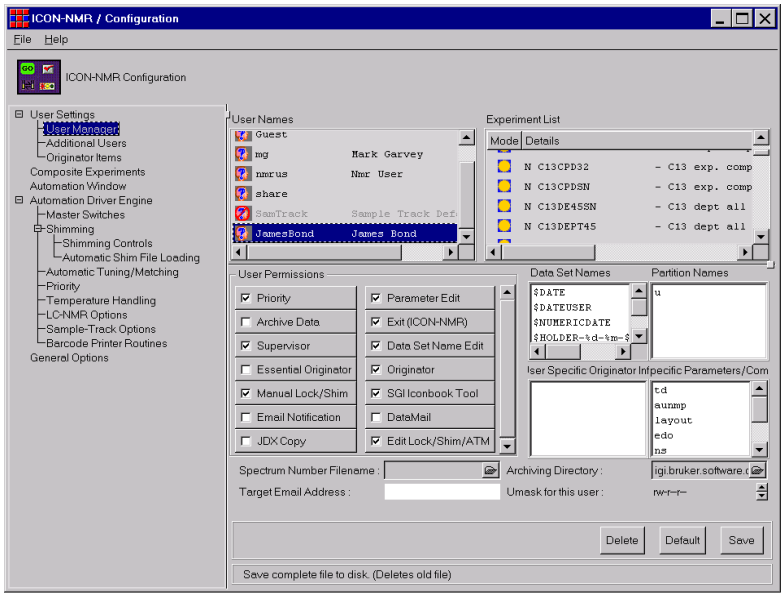


Figure 5.1 ICON-NMR Configuration Suite

5.2 User Settings

5.2.1 User Manager

The User Manager lets you comfortably control just how much freedom the users of the instrument are going to get. These features apply for the most part to both the *Automation* and *Routine Spectroscopy* parts of the program. Setup one template for your users and save it for all the users on the system. Update the experiments which have been cleared for particular users in one sweep. Save user specific information which can later be used to group particular users with similar information together. Custom tailor (for your lab) exactly what information the user must enter before any experiment may be started (E.g. An Originator ID describing the probe being measured.)

Now you can restrict the input to suit your particular file handling needs.

5.2.1.1 The *User Names* box

When first started the *User Manager* shows all the workstation user names and *Additional user* names which are currently defined for ICON-NMR in the *User Names* box (Figure 5.1, upper left). *Additional users* do not normally need to be defined. Those users who already have a userfile are shown with a bold font and have an “enabled” indicator icon.

All items displayed in the various boxes of the User Manager window are valid for the user whose ID is highlighted in the User Names box. Click on the name whose user information you wish to view or change. The respective information is then loaded from a file and displayed. When you're happy with the settings for a particular user, save the file using the save button.

5.2.1.2 The *Experiment List* box

This listbox is used to define the experiments a user may perform. The list shows all experiments currently enabled for the user selected in the User Names box. The mode and details of the experiment are shown. Mode defines whether the experiment may be run anytime (sun) or only at night (moon). Details comprises three columns show experiment *type*, *name* and *comment*.

The type is encoded into a single character: N denotes a *normal (single)* experiment and C a *composite* experiment. A composite experiment is a sequence of normal experiments which can be defined in the *Composite Experiment Manager.* The member experiments of a sequence may depend on each other. For example, the first experiment of a sequence could be a preparation experiment for a subsequent 2D experiment. In such cases, the composite experiment manager allows you to specify the reference experiment(s).

5.2.1.3 Setting up the experiment list

To make any changes in this list you must first activate it by clicking on an entry in it or by using standard keyboard traversal (tab key) to activate a particular entry.

The entry box shown in the center of Figure 5.1 will show the type of experiment you have selected in the list, its name and its comment. Use the following buttons to alter the list:

- **Update User Files**
  The contents of the respective listbox (Experiment List, Partition Names, etc.) may be saved directly to several user files, leaving all other information in the respective user files intact. Choose the user files you wish to update from the list shown in Figure 5.2.

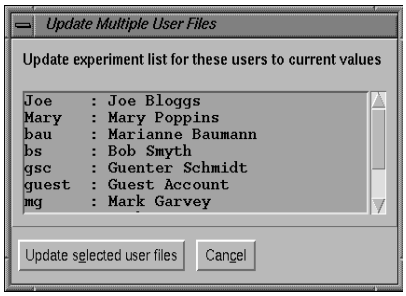


Figure 5.2 Update Multiple User Files

- **Delete**
  Remove the marked (=highlighted) experiment(s) from the list. In order to remove several experiments at once, mark them in the following way: Click on one of the experiments using the left mouse button. Keep the button depressed and move on to the next line, and so on, until all desired experiments are marked. You can achieve the same effect by marking the first experiment with a mouse click followed by a mouse click on the last experiment while the Shift key is held down. If you want to delete several experiments which are not displayed on subsequent lines, hold down the Ctrl key while you click on the relevant experiments.

- **Prepend**
  Use this button to add a new experiment above the currently selected experiment. Select the name of the new experiment from the button at the right of the *Experiment Name* entry. The displayed list is constructed from the parameter sets present in the directory *XWINNMRHOME/exp/stan/nmr/par/*, and the defined composite experiments. This means that you can only add an experi-

ment to a user's experiment list if the respective set of acquisition, processing, and plotting parameters already exists. If you did not set up your own experiments, Bruker's standard experiments and standard composite experiments are available, provided XWIN-NMR's expinstall command was run successfully.
Before you add the experiment to the user's list, you may change the comment. The experiment type is a view-only parameter which can take on the values *Single (Normal*) (N) or *Composite* (C). When you add a new parameter set to the directory *XWINNMRHOME/exp/stan/nmr/par/*, it will be assigned type N by default. If the new parameter set designates an experiment requiring preparation experiments, you *must* define a composite experiment in *Composite Experiment Setup* mode, which includes the preparation experiments. You can give the composite experiment the same name as the parameter set: If a composite experiment and a parameter set use the same name, the name will only appear once in the user's experiment list, and the executed experiment will be the composite experiment.

- **Append**
  As the name suggests this button adds the information to the list after the currently highlighted entry. Apart from that, everything said about *Prepend entry* is valid.
- **Modify**
  This button allows the information shown in the entry field to be transferred directly to the currently highlighted line in the listbox.
- **EXIT**
  This button removes the entry box from the top of the window. The entry box disappears.

You can now change the order in which the experiments will appear in the user specific experiment list by using the right-mouse button to move experiment entries around in the list. First set the focus to the Experiment List (by using the tab key), that way you can then grab an entry from the bottom of the list, use the Pg Up key to move the listbox to the top and then release the mouse button to drop the entry. You do not have to set the focus first if you only wish to move an entry around in the currently visible part of the list.

5.2.1.4 The User Permissions panel

This panel is located below the User Names box of Figure 5.1 and allows you to enable or disable certain ICON-NMR features for a user.

Priority

A user may declare a sample to be treated as a priority sample. This option is only meaningful for automation with a sample changer. Use the Configuration window in the system manager to define how the priority switch is to be handled.

Archive Data
Use this option to copy all the current experiments data to the *Archiving Directory* (also entered from the user manager) after processing has been completed on the current experiment.

Supervisor
If enabled for a user, the next time this user starts ICON-NMR all ICON-NMR windows will get borders and a menu bar, allowing them to be moved or iconified on the desktop, thus allowing access to other programs without having to terminate ICON-NMR. This permission will also gives such users full control over XWIN-NMR to start/stop other processes at will.

Essential Originator
If enabled, the user *must* fill in the originator entry which appears in the filename entry box before he can carry on with the setup.

Manual Lock/shim
If enabled, the user may either select manual or automatic locking and shimming. If disabled, the automatic procedures will be enforced.

Parameter Edit
If enabled, the user may invoke the parameter editing commands from the *Parameter* menu in *Routine Spectroscopy* or *Automation* modes and alter the acquisition, processing, or plotting parameters of an experiment before he starts it.

Exit (Icon-Nmr)
If enabled, the user may terminate ICON-NMR.

Data Set Edit
If enabled, the user may define his own data set filenames. If disabled, he may only choose from the names provided in the *Sample Names* box (see below).

Originator
If enabled, the so-called originator information and the associated user specific information will be included in the filename setup box for *Routine Spectroscopy* or in the title window for *Automation* modes. You set up originator information from

the *Managers->Originator Setup* menu, and user specific information from the respective box in the User Manager window. See “The Originator Items Editor” on page 60.

Iconbook
If enabled, the user may invoke the command *Iconbook* from the *Options* menu of respective ICON-NMR mode. (SGI only)

Email Notification
If enabled, the operator will receive an email notifying him of the completion of his/her experiment provided:

a) Email is operational on the controlling workstation. (NT Windows messaging has been activated)

b) The *Target Email Address* (also available in the User Manager or as an Originator Item) has been setup correctly for the current user.

Data Mail
Data Mail uses the smail XWIN-NMR program to send 1D data on the Internet to the *Target Email Address* (if defined). For SGI systems this option is only available on Workstations where a Zmail (MediaMail or MediaMail Pro) client is available. You do not need a Software license to use Zmail to send mails. Install the Media Mail software from the IRIX CDs accompanying your workstation.

Edit Lock/Shim/ATM (Automation Mode Only)
Use this permission flag to decide whether the User should be allowed to change the default lock/shim/atm settings. When this flag is activated the user will have an additional menu button, (with a lock signal displayed on it) in the Par column of the setup window. If multiple experiments have been submitted for a particular holder position, some of this information may be ignored. For example, if the Shim setting is to be considered for each individual experiment then the configuration option “Shim the sample” in the Shimming Controls window should be set to “on every experiment”

JDX Copy

From ICON-NMR 3.0 onwards it’s possible to have ICON-NMR generate a copy of the data set in JCAMP-DX format and store the data with an unique name in a directory as specified by the fields shown under the General Options Tab of ICON-NMR’s Configuration Suite. This feature is in addition to all the other Archiving/ Data Mail modes.

5.2.1.5 The *Sample Names* box

Use this box to define the data set NAMEs you want to propose or prescribe for a particular spectrometer user. All names defined here will appear in the *names* list of the Filename box where the user defines the data set name during experiment set up. If you enable the *Data Set Edit* checkbutton (see above), the user may also enter his own data set name. Otherwise he will be restricted to those you set up here.

ICON-NMR provides five special names, *$DATE, $DATEUSER, $NUMERICDATE, $USER and $HOLDER.* These macros will be expanded when the user opens the *names* list in *Routine Spectroscopy* and *Automation* modes. You can also make use of date variables such as %M and %Y etc. to form your own standard data set names.

Click in the Sample Names box to open the respective edit box at the upper right corner of the User Manager window. Enter the desired name, $DATE, $NUMERICDATE or $DATEUSER and click on *Prepend entry* or *Append entry* to add the name to the list. Click on *Delete entry* to remove the currently selected name from the list. Click on *Modify entry* to apply changes to the currently selected name. *Update users* may be used to save the listbox settings to multiple user files.

5.2.1.6 The *Spectrum Number Filename* entry

This entry at the bottom of Figure 5.1 provides an additional method of prescribing data filenames.

The contents of this entry field will be considered as the pathname of a text file (Example: */usr/adm/spectrumno.txt*) set up by the laboratory manager. The file must hold a name beginning with letters and ending with a number, e.g. *H1000* or *C1*. This name will appear in the *names* list of the Filename box where the user defines the data set name during experiment set up. This particular type of data set name will automatically be incremented with the next sample. The names in the above example would then change to *H1001* and *C2*. The incremented name will be written back to the text file: Should ICON-NMR be terminated and restarted, the user would still get the next name in sequence for his new experiment, provided he uses the *Eject & Terminate* option in the insert sample window.

For Automation, when this file has been defined, only the name prescribed in the file will be available to the user.

5.2.1.7 Target Email Address, Archiving Directory and Umask

These three entries allow the above items to be defined on a per user basis, even for so-called *NMR* users who do not have a direct login on the workstation. The archiving directory could be used for example, to define an NFS mounted filesystem which is to be used to store a copy of any experiments data after processing. The workstation manager is responsible for the setup of such an archiving directory.

5.2.1.8 The *Partition Names* box

Use this box to define the disk partition(s) where data sets acquired by the current user may be stored (this corresponds to the possible settings of the XWIN-NMR parameter DU). If you allow for several partitions, all names defined here will appear in the Filename box where the user defines the data set name during experiment set up.

Click in the Partition Names box to open the respective edit box at the upper left corner of the User Manager window. Enter the desired partition and click on *Prepend entry* or *Append entry* to add the partition to the list. Click on *Delete entry* to remove the currently selected partition from the list. Click on *Modify entry* to apply changes to the currently selected partition. *Update users* may be used to save the listbox settings to multiple user files.

In Automation mode the parameters are accessed from the Par column button which is displayed on every active experiment entry line. The Commands may be accessed from the Parameters pulldown at the top of the setup window.

5.2.1.9 The *User Specific Information* box

Using “The Originator Items Editor” on page 60, you set up entry items which will appear in the *Filename* box (when using Routine Spectroscopy), or in the Title window (when using Automation) where the user enters the data set name for an experiment. The corresponding entry fields (e.g. *Department, Costing Group,* or whatever you have defined) will remain empty, and the user must enter a suitable item, e.g. a department number. In some cases the item to be filled in is known in advance, and the user should not need to type it in (e.g. his e-mail address).

Click in the User Specific Information box to open the respective edit box at the upper right corner of the User Manager window. Select the desired item (which

you have previously defined via the Originator Item Editor) from the *User Information Type* pull down menu of this box (e.g. the item *Mail Data/Noify to*). Enter the respective user data (e.g. the user's e-mail address) into the *User Information Data* entry. If the data changes from experiment to experiment then you should leave the Originator data field empty. The user can fill it in at experiment set-up time.

Click on *Prepend entry* or *Append entry* to add the information to the list. Click on *Delete entry* to remove the currently selected information from the list. Click on *Modify entry* to apply changes to the currently selected information. *Update users* may be used to save the listbox settings to multiple user files.

5.2.1.10 User Specific Parameters/Commands

Here the laboratory manager may define the names of parameters or commands which the specified user is allowed to change or run. The Parameter Edit permission does not have to be enabled to allow this type of parameter editing.

As an example, if the user should only be allowed to alter the TD parameter then the *Parameter Edit* checkbutton should be de-activated and the entry td added to the user specific parameters/commands list.

Note that XWIN-NMR commands may also be entered in this box allowing users with no "Supervisor" permission to access specific XWIN-NMR commands. In Automation, where possible the command will be run on the appropriate corresponding acquired data set.

You may configure entries from this list in the same manner as described above for the *Partition Names* box.

5.2.1.11 The *Delete, Default,* and *Save* buttons

The respective commands are also accessible via the *File* menu. The Save button saves the current settings on disk, in the file

XWINNNMRHOME/conf/instr/<instrument-name>/inmrusers/<userid>

The *Default* button initializes all boxes with the default information stored in the file

XWINNMRHOME/exp/stan/nmr/lists/sam_users_exam.inmr

You may save everything you have set up so far for several users at once. Hold down the CTLR key and mark all desired users in the *User Names* box by clicking on them. Or keep the Shift key depressed while clicking on a user. This will automatically mark all users from the currently selected one to this position. Then click on the *Save* button.

Due to popular demand a Delete button has been added to remove users from ICON-NMR! Mark the users you wish to delete and click on the delete button. Their user file will be deleted.

Note:

With XWIN-NMR 2.5 you can use the AU program runproc to run batch processing commands on data sets which have been acquired by ICON-NMR *Automation*. The program uses the history files which ICON saves automatically to retrieve the names and paths to the data sets acquired during any given run. You may select all data sets for reprocessing or choose only the ones you are interested in. Use the xaup command to reprocess. If you haven't compiled the runproc program already, you will need to type xau runproc on the XWIN-NMR command line, to use the program.

Tip: You may also re-run the processing in ICON-NMR Automation by submitting the experiments to be re-run and starting the Automation using the "Processing Only" Automation driver.

5.2.2 The Additional Users page (was NMR User Manager)

Additional Users are Spectrometer Users who do not have a Unix user account on the workstation managing the spectrometer. They may, however, still perform experiments and the experiment information fids etc. will be stored using the NMR effective users ID and permission as defined. Additional Users is an extra feature available only in ICON-NMR. These users must not be defined for normal operation.

All Additional Users may have their own password and may initialize or reset it once they have logged into the ICON-NMR system.

Additional User names and the respective abbreviations used for their names are shown in the listbox above the command box. You may modify this list using the Delete, Append, and Modify buttons shown next to it. The Append entry button

acts relative to the highlighted position.

The NMR Super User is the name of the privileged user whose password (provided one exists), must be entered before carrying out potentially sensitive tasks. This information is defined when XWIN-NMR is installed. It should normally not be necessary to change this. Should you wish to change the NMR Super User then please use the installnmr script in XWINNMRHOME/prog/bin.

Each Additional User name entered here also appears in the main User Manager Window *User Names* list box. User specific information may the be set up there.

5.2.3 The Originator Items Editor

The Originator Items Editor lets you edit the list of items which may be entered in the *Routine Spectroscopy* Filename window or the title entry in *Automation* mode, when an experiment is being setup. Each entry in the list box created here will appear in the Filename or Title window along with an entry box where the corresponding information is to be entered (provided the *Originator* checkbutton for the particular user has been activated).

Use the *types* pulldown menu or make new Originator types using the entry box and the append button. The Append entry button acts relative to the highlighted entry's position.

Use the User Manager window to decide on a per user basis whether the additional entry boxes will appear at all (*Originator* Checkbutton) and whether the information must be entered before the experiment may be submitted and started (Essential Originator). Default values on a per user basis may be entered in the user specific information box in the User Manager.

5.2.3.1 Automation Mode Options

ICON-NMR 3.0 has new options to control originator handling more flexibly. With "Generate orig file in Data Set" you can control whether an "orig" file containing the originator information, will be saved in the data set. "Include Originator Item field in orig/title file" determines whether the Item Names will appear in the title file or Orig file. "Insert this text between Originator Items" can be used to control exactly how the originator information will be formatted.

ICON-NMR 3.0 provides a special Originator Item called “Mail Data/Notify to”. This entry may be used in the Automation mode to override the email address for the current user as set up in the user manager. This is useful for installations with a large number of users where only one user configuration is being used. If an email address is entered here then it will override the User Manager email address setting for the user. If there is no email address for the current user and no address has been entered in Automation mode at the respective Title entry field, then no email will be sent. This way you can decide on a per experiment basis when a notification/data e-mail will be sent. Note that the Email Notification and Data Mail notification flags for the particular user determine what type of e-mail will be sent (if any).

5.3 Composite Experiments

Experiments that may be executed by a user from ICON-NMR are stored in form of parameter sets (i.e. acquisition, processing, and plot parameter files) in the directory

XWINNMRHOME/exp/stan/nmr/par/.

Bruker experiments are installed in this directory by the expinstall command in XWIN-NMR. The laboratory manager may add new parameter sets. An experiment name is the name of a parameter set directory, e.g. the COSY experiment would fetch the required parameters from the parameter files from the directory

XWINNMRHOME/exp/stan/nmr/par/COSY/.

A composite experiment is a sequence of several such experiments. In the composite experiment manager you define new composite experiments or alter existing ones. You assign a name to a new composite experiment, and you specify the names of the experiments that should be included, and the sequence of their execution. When a spectrometer user selects a composite experiment, all components are executed automatically. Composite experiments may not include other composite experiments.

In ICON-NMR, any experiment requiring preparation experiments *must* be set up as a composite experiment. For example, a sweep width optimized Cosy would be defined as a composite experiment consisting of a suitable 1D, followed by the actual 2D experiment. A preparation experiment is designated reference experi-

ment within ICON-NMR.

5.3.1 Defining experiments

In order to define a new or alter an existing composite experiment, click on the "Add new" button at the base of the manager window.

Apply your changes in the Composite Experiment Editor window shown in Figure 5.3 or enter a new name and a comment. Click on the *Modify* button to make the modification effective, or click on *Prepend* or *Append* to add the new experiment to the list.

Click on the *Experiment Name* button and select the first experiment of the composite experiment and append it using the *Append* button. Carry on in this way adding other experiments to the composite experiments. If an experiment you want to append requires one or two reference experiments, use the *F2 Reference* and *F1 Reference* buttons to select them. When you click on these buttons, all experiments which are already part of the composite experiments will be displayed along with their sequence label (a, b, c, ...), and you can choose which ones should serve as references.

5.3.2 The '*Save to*' button

After a new composite experiment has been defined you may wish to 'save' it to a particular users user file, thus bypassing the *User Manager* page.

Mark the composite experiment(s) you wish to save and click on the 'Save to' button. A list of the users who currently have a user file is shown. Mark the particular users who are to use the newly defined composite experiment and use the 'Save to selected users' button.

The marked composite experiment(s) will be appended to the start of the respective users experiment list.

5.3.3 The *Default, Revert* and *Save* buttons

The *Save* button saves the current settings on disk, in the file:

XWINNMRHOME/conf/instr/<instrument-name>/inmrusers/.pool_txt.

The *Default* button initializes all boxes with the default information stored in the

file:

XWINNMRHOME/exp/stan/nmr/lists/def_comp_exps.inmr.

The *Revert* button reloads the last saved settings from the *inmrusers/.pool_txt* file.

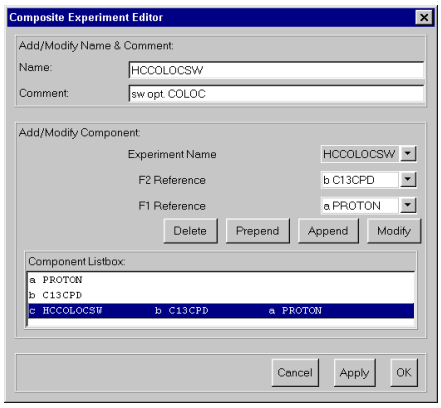


Figure 5.3 Composite Experiment Editor

5.4 Automation Window

The Automation Window page currently contains the following options:

- Holder Status Display Style
  Here you'll find two different status 'traffic light' styles, North American and European, plus a spell-it out text only mode. There is also a new Bruker Standard Style which should increase the conformity between Bruker's various Automation Software programs.
- Touch Screen Compatibility Mode (NT Only)
  This check button puts the ICON-NMR Routine Spectroscopy and Automation modes programs into Touch Screen mode. All ComboBox style input widgets switch to a large icon mode which may be easily be accessed with your finger. Title input/Parameter Input windows get a "virtual keyboard/keypad" eliminat-

ing the need for Keyboard and mouse! This mode is currently only available on Windows NT workstations.

- Default Number of Sample Holders

The number of samples to be shown in the experiment manager window can be set here.

- Automatic Experiment acquisition time calculation

Every time an experiment is submitted the experiment time as calculated by the XWIN-NMR expt command will be shown. This calculation is done once per experiment type and in cases where the parameters have been changed.

- Logout Idle users Automatically
  In an open access environment, operators should be required to log themselves out after they have submitted all their experiments to be run. They may, with this option be logged-out automatically after a certain amount of 'Idle time' has elapsed. After this period of inactivity the Identify User window will re-appear and the experiment manager window will be disabled. It is, however, still possible to observe what the Instrument is doing. Use the Logout Delay to adjust the required idle time.

- Enable Automation Controls Window

This flag determines whether a control panel similar to that shown in Figure 3.5 on page 23 will be shown during an automation run. This feature may be disabled to prevent users from (for example) processing the data.

- Automatic Setup File Save

This flag will cause the contents of the setup window to be automatically saved at regular intervals. Should anything go wrong, this file may be loaded later to restore the setup.

The ASCII file will be saved in

XWINNMRHOME/prog/curdir/changer/<setup dir name>.set and may be read using the File->Open menu in the Setup Window.

- Approx. overhead time for sample change (secs)

ICON-NMR calculates the time required for the acquisition in order to know how long the instrument will be busy. With this window you can define just how much time ICON should allow for the sample changing/locking shimming etc. when calculating the total busy time.

- HTML History File

ICON-NMR 2.0 can save all the information stored in the *history list* in HTML format. This entry is used to define where ICON saves this file. It's a good idea to make sure that everyone can write to this file. If this file is located in a directory which is supported by a HTML server the History may then be observed remotely, over the net. IRIX 6.3 has a directory ~/public_html which makes its contents publicly accessible from the net.

Note the automatic history file is stored with a .hist extension with the same name as the setup directory itself i.e. in XWINNMRHOME/prog/curdir/changer/inmr-changer/<setup directory name>.hist

5.5 Automation Driver Engine

5.5.1 Master Switches

These options apply to the Automation part of ICON-NMR only. The "Driver" is responsible for all spectrometer control commands which ICON-NMR issues to perform experiments. As such it is ICON's engine.

For more information on drivers see 'Drivers' on page 37.

- **Start run at User Login**

Normally the run must be started manually after starting the *Automation* part of ICON-NMR. This means that operators who forget to do this, will submit their experiments for execution and leave the instrument, to do the work which it will

never see. To get around this potential problem the Run may be started using this function automatically. When this mode has been activated you must also define which driver is to be started automatically. Use the *Default Automation Mode* to define this.

- Default Automation Mode

You only need to specify the default automation mode if automatic run has been specified. It informs the run interface which driver to use. For more information on drivers see 'Drivers' on page 37.

- Eject Last Sample in queue

When this check button has been activated the sample changer will also eject the last sample which has been measured, after all other submitted samples have been measured. Some automation modes do this regardless of the setting e.g. MAS, HRMAS.

- Process Data Sets after Acquisition

Possible settings include 'Always', 'Never' or 'Ask on Start-up'. When using Ask on Start-up a checkbutton will be displayed in the window which appears after clicking on the 'Go' button.

- Delete temporary Data Sets after experiment end

With this option activated some disk space will be saved during the run. However, if you wish to retain modified parameter information in it is better to leave this option off. Otherwise modified parameters may be lost should you wish to resubmit a completed experiment.

Temporary data sets will be removed by ICON on exiting if you do not save the setup. Icon uses the temporary data sets to store changed parameters, save time when using the Copy function and for other reasons. The data sets are stored in

XWINNMRHOME/prog/curdir/changer/inmrchanger/MonthDay-Year-Time-user

The date information is obtained from the system when the setup window is first opened.

- Automatic Lock Program

Here you may define exactly what ICON's driver does when trying to lock. With the Skip option you can tell ICON to skip locking altogether e.g. lock externally from the acquisition AU program, or just run a few samples without the lock.

The default value for this setting is:

LOCK #Default

You can enter any XWIN-NMR command here, including AU programs macros or commands you would normally enter on the command line. The CPR_exec_wait macro command tells XWIN-NMR to execute the following and wait for the command to finish. You must always include this to execute your command.

If you want an AU program to be executed and ICON to wait until the AU program is finished then you should use the XAU macro, instead of the CPR_exec_wait macro:

XAU auprogram args

where auprogram is the name of your AU program and args are any arguments you may wish to forward it.

Should you wish the command to be executed in the background (without waiting for completion) use the command CPR_exec

Multiple commands may also be entered, each command should be separated by a ";". The "#" signifies that a comment follows.

Delays may also be included before or after a command if required. To wait 60 seconds before locking include the following text before the CPR_exec_wait macro:

after 60000;

If you're familiar with TCL the tool command language from John Ousterhout, feel free to enter TCL commands here too!

- Lock only after a solvent change

Should ICON only try to lock when the solvent has changed, activate this flag.

This feature is only available in Automation mode.

- Ignore the XWIN-NMR Prosol Parameters

This is a useful feature which tells ICON-NMR not to update the parameters which are usually controlled over the edprosol interface. That way you can be certain, if you save your parameter set with wpar ParameterSetName all, that you will always be able to reproduce the experiments under exactly the same conditions.

- Never Rotate the Sample

This may be interesting for some users who wish to disable sample rotation completely without having to edit the acquisition parameters (RO) of all parameter sets used in Automation.

- BEST Mode Settings

Here you may switch ICON into BEST (Bruker Efficient Sample Transfer) mode. Leave this option off unless you have purchased BEST! BEST uses a flow injection technique to boost sample throughput to the limit. In BEST mode there are various configuration options possible via the bestadm commands. These are accessible from the BEST Administration Tool button.

- Use Barcode Reader for Best

When ICON is running in BEST mode a barcode reader may be added to the Gilson liquid-handler which works in conjunction with SampleTrack, Brukers Laboratory Information Management System to fully automate Sample preparation and experiment submission. In this mode ICON receives all of its input directly from the Bruker's SampleTrack software.

5.6 Shimming

ICON-NMR 3.0 has lots of features to help you optimize your Automation run to the limit.

5.6.1 Shimming Controls

- Automatic Shim Program

Use this to decide which type of shimming you'll be using. ICON-NMR provides 3 default options:

Default

TUNESX

Shim based on the probe/solvent dependent tune file saved when you setup the system using edprosol XWIN-NMR commands.

GRADSHIM

Use the gradient shimming automation program to greatly increase the shimming performance (you need gradients installed on your spectrometer).

Skip Shimming

Use this mode when shimming is not required, or where the shim control is handled externally, (e.g. by the Acquisition AU program see the AUNM parameter).

You may of course enter your own command here. For simplex shimming (with your own tune file) You could enter a command like:

CPR_exec_wait tune mytunefile

The mytunefile can be generated using the edtune XWIN-NMR command. You'll find example tune files in:

XWINNMRHOME/exp/stan/nmr/lists/group

Delays may also be included before or after a command if required. To wait 60 seconds before locking include the following text before the CPR_exec_wait macro:

after 60000;

The argument to the TCL after command is in milliseconds.

If you're familiar with TCL the tool command language from John Ousterhout, feel free to enter TCL commands here too! See "Automatic Lock Program" on page 67.

- Shim the sample
  Specify how intensively the shim routine is to be called.
- Maximum Shim time per iteration
  Here you can indirectly specify just how long the shimming part of the automation will run. This limit applies to each gradient as defined in the tune file. If the time limit is exceeded on any iteration then that gradient is skipped and the next gradient started. If the time limit is exceeded at any stage ICON-NMR will mark the shimming as failed, but the experiment will still be performed.
- Total Time allocated for tune command (minutes)
  To ensure that the length of time taken for shimming remains manageable, regardless of how many iterations are performed, set this field accordingly. If XWIN-NMR is still shimming after this time has expired the automatic shim routine will be halted and the experiment acquisition will commence.

Note:

From ICON-NMR 3.0 onwards, you may control exactly what ICON does when it gets to the lock or shim stage in Automation on a per sample basis. For this feature to work you must first enable the Edit Lock/Shim/ATM permission on a per user basis.

5.6.2 Automatic Shim File Loading

ICON-NMR has facilities to load a particular standard shim file in *Routine Spectroscopy* and *Automation* modes. The shim files are loaded automatically each time a new sample has been inserted into the magnet, before the system reaches the lock stage. That way it is always ensured that the system is in a well defined state for each new operator/sample. The shim files will be linked to the solvent and the probe being used.

This window consists of a list of file entries (one for each solvent). The file entries apply to the probe which is shown in the *Current Probe Type* box. The entries are self explanatory, see Figure 5.4.

- Lock using -noauto flag

This option should be used sparingly! For some solvents with several strong peaks, the lock program may lock on the wrong one. The lock -noauto flag will cause the lock program to lock on that peak nearest to the field value stored in the /conf/instr/<spectrometer>/2Hlock file for the particular solvent. The command 'lock -noauto' is used instead of the standard 'lock -acqu' for these solvents.

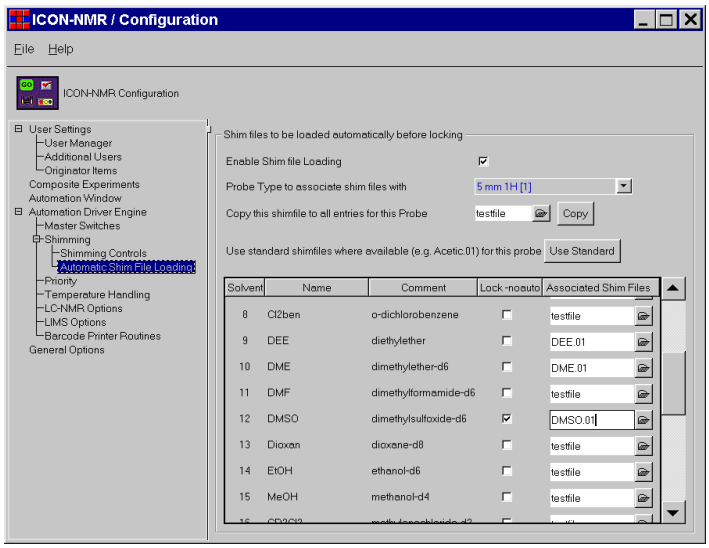


Figure 5.4 Default Shim Files Setup

5.7 Automatic Tuning/Matching

Some of the latest probes are equipped with tiny motors so that their tuning and matching characteristics may be varied under software control. The XWIN-NMR command atma tunes and matches the probe automatically according to the NUC settings in the current data set. ICON can, if required, run this program automatically.

The ATM setup page contains the Enable ATM Optimization flag used to globally enable/disable all ATM activity. Apart from that it also contains a list where

matching and tuning may be enabled and disabled on a per experiment basis. You can either use the copy button at the top right of the page to change elements of the selected experiments, or use the right mouse button when over an entry to get a popup menu whereby the underlying entry may be changed.

Possible settings include “Always”, “Never” and “after a solvent change” for both H and X channels respectively.

Note: Users may be granted permission to edit the ATM setting for any experiment when logged into the Automation set-up mode. The Edit Lock/Shim/ATM permission in the user manager is responsible for this feature.

5.8 Priority Options

These options only apply to the Automation part of ICON-NMR.

- Enable Priority

Here you may globally disable priority handling, including Day/Night checking altogether. This will speed up ICON’s search for the next sample. If you don’t plan on having priority samples then leave this option off.

5.8.1 Strategy

These options determine (in general) how priority requests will be handled.

- Modes accounted for

Determine what levels of Priority you wish to cater for. Options include Night & Day, Priority Only, Priority and Night & Day.

- Priority Sample Handling

Decide whether **ICON** should jump to a priority sample immediately. This is a hardware/time unfriendly setting as the sample changer will move to a priory sample even if more experiments are queued for the current sample. The default is for the sample holder only to be changed to the priority sample after all experiments have been completed on the current holder. This saves unnecessary wear on the sample changer as well as system overhead (i.e. the uncompleted experiments on the currently active holder will have to be locked and shimmed again!).

5.8.2 Night Day Switch-over

Set the times used for night-time experiments. You can also specify whether night time experiments should be allowed to run during idle Day time.

5.9 LC-NMR Options / Sample Track Options

For further information on these settings see the LCNMR and Sample Track Manuals respectively.

5.10 Temperature Handling

ICON-NMR has features to set and check the temperature before and after sample insertion.

This system is currently only available to customers using a BACS sample changer, and those who are changing their samples manually. Provision is made for checking and controlling the temperature both before and after sample insertion. You can set a fixed temperature or use the temperature setting according to the TE parameter set parameter. The TESET macro sets the temperature while the TEREADY macro requires 2 arguments. The first defines the maximum time ICON should wait for the temperature to be reached. The second parameter determines to what degree of accuracy the program should require temperature conformance to the desired value. Once the accuracy has been attained the Automation can proceed.

For VT experiments in Automation it’s a good idea to define TE as a user specific parameter. That way you can access the TE value quickly from the experiment definition line.

5.11 General Options

- Archiving Copy User (Unix only)

If root access on the workstation is allowed, directories will be generated where needed. If you use the normal user action, ICON-NMR may not be able to copy the data if the directory structure does not already exist.

- Include Originator Information in Title

Normally the originator information will be stored in the title file. Use this option to disable this feature. Originator Info will in any case be stored in the data set, in the file *"orig"*. For more information on Originator Entries see 'The Originator Items Editor' on page 54.

- Experiment Number Automatic Increment

Normally a space of 10 experiment numbers will be left for additional experiments on any given sample. Use this dialog to change this

- Flash Entry Zones

This applies to *Routine Spectroscopy* only. The blinking frames red and green frames may be deactivated using this checkbox option.

- ICON Color Palette

Some other programs may not be able to get their default colors when ICON is running. Use this option to reduce the number of colors requested by ICON. This may alleviate the problems in other programs as more colors will be available for them. You may even run ICON in black and white mode, for that great 50's black and white movie feeling!

- Password Checking

For those of you working in a trustworthy environment, use this menu to slacken the password checking mechanism in ICON-NMR 2.0. You may disable checking altogether, or reduce password checking to Manager Items and start-up with the 'Sensitive Tasks Only' option.

- Automation Error Handling

This new feature available only in Automation mode allows for even more Instrument supervision. If one of the systems being used in Automation fails completely, for example the lock, then the complete Automation Run may be stopped automatically. Optionally you may choose to allow the run to continue and should you

enter a valid E-mail address in the space provided, a notification Mail informing of the error will be sent to the given address. Multiple addresses may be separated by semicolons.

This feature is especially useful in BEST mode where invalid results may not necessarily be corrected by running a second time (insufficient sample).

- JDX Copy Mode

From ICON-NMR 3.0 onwards, ICON will generate JCAMP-DX files automatically at the end of the measurement/processing and copy them to the directory which may be entered here. It is also possible to define which filename will be used for the file. Use the File Naming format to decide how the files are to be called. Click on the pulldown menu to see some examples. The Keywords $DATASETNAME and $EXPNO are available in both Routine Spectroscopy and Automation modes.

If processing has been disabled, then only the fid may be converted. All other modes will be ignored.

Chapter 6

The Accounting Manager

6.1 Introduction

The accounting software is started from the *Accounting* icon in *System Manager* mode. It gathers the information required to calculate prices for spectrometer usage from an *accounting file*. The structure of an accounting file has already been discussed in the *Routine Spectroscopy* section of this manual. Each time an experiment is executed under control of ICON-NMR, the standard accounting file:

XWINNMRHOME/conf/instr/<instrument name>/inmrusers/Inmracct.brief

is updated.

At start up time the accounting program collects and sorts all user information stored in this file. Depending on its size (which can grow significantly during e.g. a month's period), you will have to wait some time until the dialog box similar to the one shown in Figure 6.1 appears.

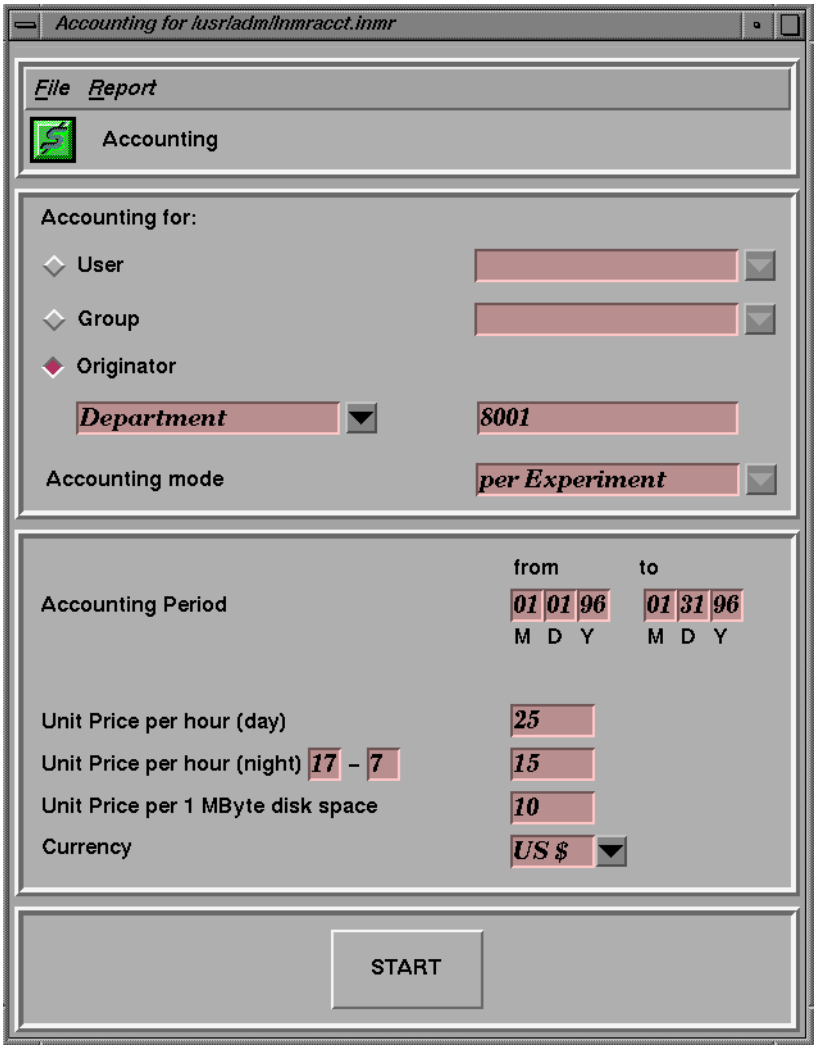


Figure 6.1 Accounting dialog box

6.2 The *File* menu

The following commands are available:

Open accounting file
By default, *Inmracct.brief* is the standard file responsible for all accounting information. However, the accounting information search can also be performed on other files with the extension *.brief*. The *open* command will display all files of this type stored in the directory *XWINNMRHOME/conf/instr/<instrument name>/inmrusers/*. Select the one you wish to use for accounting. Its name will be displayed in the header bar of the dialog box. After you have chosen a new file the accounting information is collected and sorted. This process may take some time, so please be patient.

Exit
Terminate the accounting program.

6.3 The *Report* menu

The following commands are available:

Display on screen
Re-display the report generated via the Start button.

Print
Print the report generated via the Start button on the current printer.

Save as
Store the report generated via the Start button in the specified file. If the file name does not begin with a "/" character, the report will be stored in the login user's home directory.

6.4 The *Accounting for* box

In this box, you enter the users or the department etc. for which you want to generate an accounting report. The accounting mode is also selectable.

- Enabling the *User* button will instruct the accounting software to generate a report for the user which you may select from a list. Click on the down arrow

button to the right of the *User* button to display the list. This list is determined at startup time from the standard accounting file, or from the file you have chosen via the *Open* command in the *File* menu. The special list entry *All* will compile a report for all users present in the list.

- The *Group* button is similar to the previous *User* item, however, accounting is performed for the specified Unix *group*. The special list entry *All* will compile a report for all groups present in the list.
- Enabling the *Originator* button provides the most flexible accounting method in the *per Experiment mode*. Click on the down arrow button to get the list of originator items displayed that you have set up with the *Originator Template Editor*, and select one of them (e.g. *Department*). Enter the suitable accounting criterion into the field to the right of it (e.g. a department identifier).

  Example 1:
  Assume you have selected the accounting type *Department*. The item *Department* is member of the list because you have defined it as originator item with the *Originator Template Editor*. Assume further that in your organization departments are identified by numbers. In order to generate the accounting report for the department 8009, enter this number as the accounting criterion. As a result you will get the total sum of spectrometer usage for all users belonging to this department.

  Example 2:
  Assume you have selected the accounting type *Email Address*. The item *Email Address* is member of the list because you have defined it as originator item with the *Originator Template Editor*. Assume further your email addresses have the general form *user@organisation.country*. In order to generate the accounting report for all users belonging to *organization*, enter the text *@organization* as the accounting criterion. Accounting would then be performed for all users of *organization*, also for different *country* extensions.

  This example indicates that the accounting program applies a slightly different identification method to alphanumeric text as compared to plain numbers: In the first case substring matching is employed, while numbers must be matched exactly.
- Setting the *per Experiment* mode will instruct the accounting software to account the time between the start and end of an experiment.

Setting the mode *per Login* will instruct the accounting software to compute the accounting information based on the time between Login and Logout.

6.5 The *Accounting Period* and *Unit Prices*

Please fill in the date of the first and last day of the accounting period (the sequence is month, day, year). If you want to charge night time rates specify the start and end time. Otherwise set the time of night start and end to 0, in this case the accounting program requires less calculation time. Then fill in unit prices that you intend to charge to the spectrometer users: A unit price per hour of spectrometer usage and a unit price per megabyte disk storage consumed during the accounting period. The total is calculated according to

number of hours of spectrometer usage (day) * unit price per hour (day) +
number of hours of spectrometer usage (night) * unit price per hour (night) +
number of Megabytes consumed * unit price per MByte

If you don't want to charge for the disk space, set the unit price per MByte to 0.

You can also enter a currency. The program will append it to all prices printed in the report, but will not perform any calculations with it. You can select the currency from a list. If you don't find that of your country, you may add it to the file

/u/prog/<version>/curdir/<user>/.currency

Next time you enter the accounting software, your own currency will be included in the list. You may also delete unwanted items from this file.

6.6 The *Start* button

The program will start accounting according to the parameters you have defined during the previous steps, and display the report. Figure 6.2 shows a typical report including the performed experiments. The number close to each experiment name indicates the number of times this type of experiment was executed during the accounting period. The report also lists incomplete experiments which have been started but were interrupted either by the operator or because of an error situation. Failed experiments are not included in the price calculation.

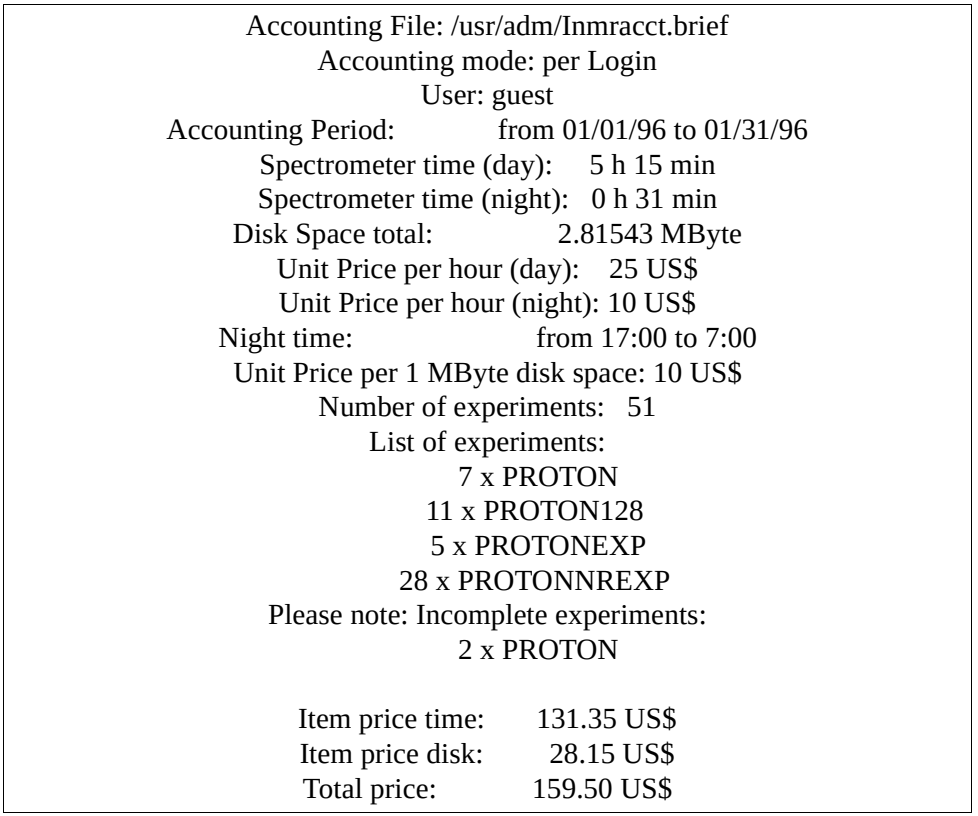


Figure 6.2 Accounting report example

Chapter 7

Journey through ICON-NMR Setup

7.1 Introduction

This chapter is intended to guide you, step by step, through the various setup activities in both XWIN-NMR and ICON-NMR and start you off on your first experiments in ICON.

Unfortunately the information here applies to ICON-NMR 2.0 and the figures shown may bear little resemblance to version 3.0. Also, the prosol handling has been completely revamped for XWIN 3.0 which renders much of the setup description obsolete. See the edprosol manual entry in the XWIN-NMR Acquisition manual. We hope to bring you a new version of this Chapter in the not too distant future. In the mean time this chapter may still be of some use to help users get an overall feel for how ICON-NMR works.

Before any experiments can run under ICON-NMR control, XWIN-NMR must be configured to control your spectrometer correctly. If XWIN-NMR has already been configured proceed to “User Manager” on page 127 for more details on ICON-NMR configuration.

Of course the information given here is system dependent, but it should give relatively new users of Bruker Spectrometers a look behind the scenes at the general software spectrometer configuration.

7.2 Software and Hardware Configuration

7.2.1 SUMMARY OF COMMANDS

Configuration:

cf	spectrometer configuration
cfbsms	BSMS configuration
cfpp	installation of printers and plotters
expinstall	installation of Bruker standard experiments

7.2.2 LOADING XWINNMR

To load the XWIN-NMR program, follow the instructions in the NMR SOFTWARE Release Letters manual.

7.2.3 HARDWARE CONFIGURATION

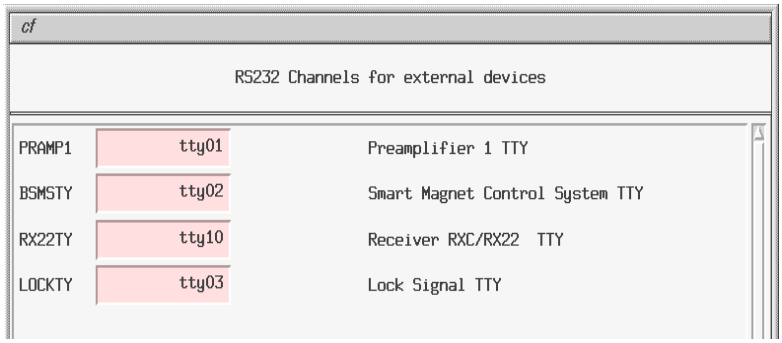
1. Call in any dataset
2. Type cf
3. Enter the root password and answer the following questions:

NOTE: The answers below are an example and are suited for a single rack AVANCE 300.

Configuration for spect. Change that? (y/n): n

What type of spectrometer? (DMX, DRX,DPX): DPX

Basic 1H frequency (with offset O1 = 0 in MHz): 300.13



4. Enter the following RS232 ports for the external devices:

NOTE: Use the default connection listed on the label on the back of the console door.

PREAMP1:	tty01
BSMSTY:	tty02
RX22TY:	tty10
LOCKTY:	tty03

5. Click on ‘SAVE’

NOTE: The nuclei table is displayed.

6. Click on ‘RESTORE’ and then on ‘SAVE’

NOTE: The configuration information is displayed on the screen.

7. Click on ‘List’

NOTE: Store the printout of the configuration information with the installation data.

8. Click on ‘OK’

7.2.4 SHIM UNIT CONFIGURATION

1. Type cfbsms and answer the following question:
   Which device is used for BSMS: tty02

7.2.5 TEMPERATURE CONTROL UNIT CONFIGURATION

1. Type cfte and answer the following question:

Which device is used for VTU: tty05

7.2.6 PLOTTER CONFIGURATION

1. Type cfpp
2. Click on ‘printer-plotter installation’

3. Click on ‘remove existing printer-plotter’ and enter the root password
4. Click on the printer-plotter name which is displayed and confirm the message.
5. Click on ‘install new printer-plotter’ and enter the root password
6. Click on the printer-plotter name to be installed.
7. Answer the following questions:

Enter identification name:	hit the return key
Do you want to specify a class:	n
Please enter output device:	plp
Install (name of printer-plotter) as default printer?	y
Configure (name of printer-plotter) like an existing printer-plotter?	n

8. Click on ‘QUIT’

7.2.7 EXPINSTALL

1. Type expinstall
2. Click on ‘OK’
3. Enter the root password
4. Click on ‘proceed’ for default installation
5. Click on ‘select all’ for user permission file
6. Click on OK and answer the following questions:

NOTE: The entries below are an example if the system is connected to an HP Laserjet 4L using 8.5” x 11” paper.

Enter current printer name:	$hplj4l
Enter current plotter name:	hplj4l
Enter paper format:	A
Enter digitizer type:	(instrument dependent)
Select acquisition mode:	qsim or DQD if available
Enter default pre-scan-delay DE:	6

NOTE: expinstall starts now. This process will take approximately 1/2 hour.

7.2.8 CONFIGURATION OF THE AUTOMATION

7.2.8.1 Manual sample change and SIXPACK sample changer:

1. Type cfbacs and answer the following question:
   Which device is used for Sample Changer? tty20
   (or any unused RS232 port)
2. Click on OK to verify the error message
3. Type 6 for the sample holder capacity

7.2.8.2 Automatic sample changer.

1. Type cfbacs and answer the following question:

Which device is used for Sample Changer?	tty08

NOTE1: The configuration will check the connection between the computer and the sample changer. Therefore be sure the sample changer is connected to the selected RS232 port and the power is turned on.

Using barcode reader:	no
Delay between SX and next command [sec]?	4
Is Airflow controlled by Sample Changer?	yes (to use BACS air)
	no (to use BSMS air)

7.3 How to set up the prosol parameters.

7.3.1 SUMMARY OF COMMANDS.

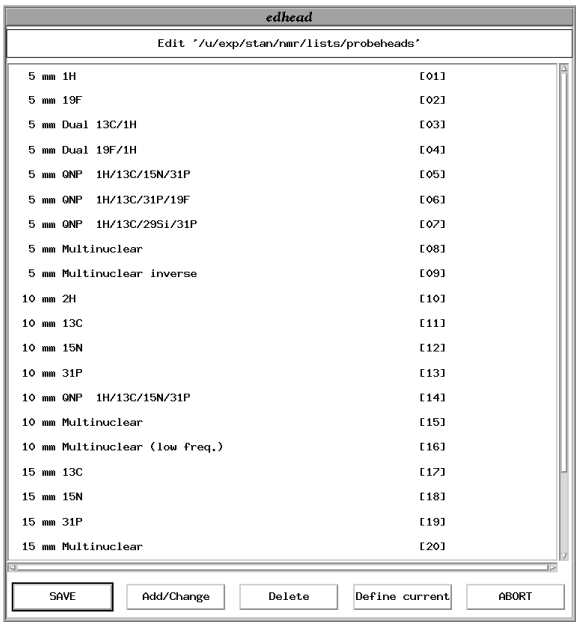
Configuration:

edhead	set up the probehead table
solvloop	setup probehead parameters for all solvents
prosol	setup probehead parameters for one solvent

edlock edit 2HLock file

7.3.2 SELECTION OF CURRENT PROBEHEAD

1. Type edhead

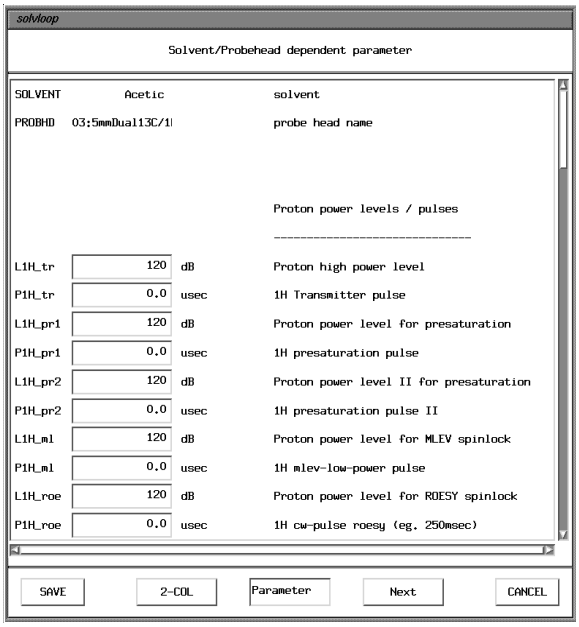


2. Click on 'Define current'
3. Select current probehead
4. Click on 'seen' in the message window
5. Click on 'SAVE'

7.3.3 PROSOL AND TUNE FILE SET UP

1. Type solvloop

2. Select and click on current probehead



3. Edit the following parameters:

NOTE: choose only those nuclei which can be observed on the current probe. The transmitter power levels and pulses below are starting values only which are to be used in Chapter 3 ”OPTIMIZING PARAMETERS”. If the probe is already calibrated, enter the correct values in the appropriate fields.

Proton power levels / pulses

L1H_tr	0 dB	Proton transmitter high power level
P1H_tr	3 usec	1H Transmitter pulse
L1H_pr1	68 dB	Proton power level for presaturation
P1H_pr1	2000000 usec	1H presaturation pulse
L1H_pr2	68 dB	Proton power level II for presaturation
P1H_pr2	2000000 usec	1H presaturation pulse II
P1H_roe	250000 usec	1H cw-pulse roesy (eg.250msec)

Proton decoupler power levels/pulses

L1H_hi	0 dB	Proton decoupler high power level

13C power levels and pulses

L13C_tr	0 dB	13C transmitter high power level
P13C_tr	2 usec	13C transmitter pulse
L13C_hi	0 dB	13C decoupler high power level
P13C_hi	2 usec	13C decoupler high power pulse

15N power level and pulses

L15N_ tr	0 dB	15N high power level	**
P15N_tr	3 usec	15N transmitter pulse	**

19F power levels and pulses

L19F_tr	0 dB	19F high power level	*

P19F_tr	3 usec	19F transmitter pulse	*

29SI power levels and pulses

L29Si_t	0 dB	29Si high power level	**
P29Si_t	3 usec	29Si transmitter pulse	**

31P power levels and pulses

L31P_tr	0 dB	31P high power level
P31P_tr	3 usec	31P transmitter pulse

Miscellaneous power levels and pulses

P_trim	2500 usec	trim pulse (mlev)	
P_hsqc	2000 usec	trim pulse hsqc	**
L1H_noe	75 dB	Proton power level for NOE-difference	
P_grad	1000 usec	gradient pulse	***
D_grad	500 usec	gradient recovery delay	***

* for QNP with 19F only

** for QNP with 15N or 29Si and BBI, BBO only

*** for probes with Gradients only

4. Click on 'SAVE' and enter the password

5. Hit Return to answer y for the question: Edit tune file?

6. Display the line numbers and make the following changes:

insert Z and Z2 twice after line 32 and add a # at the beginning of the lines 37 and

46.

NOTE: below is a printout of the modified section of the tune file

```
1 # Example of tunefile for BSMS Unit           (2.0)95/01/25
2 # ================================================================================
3
4 ###
5 ###   All Gradient names must be written in capital letters !!!!
6 ###
7
8 DELAY             1      # wait 1 second between set_value and lock_read
9
10 MAXLOCK          3500   Show lock always on screen. Reduce LOCKGAIN
11 # if Lockvalue > 3500. (using BSMS-AutoGain)
12
13 # Two new options USE_LOCKLEVEL and USE_FIDAREA allows the modification
14 # of the paramter to be used for optimisation.
15 #     USE_LOCKLEVEL is set as default
16# The new option USE_FIDAREA starts the gs-command and read the FID area
17# after each scan (until the number of scans == LOCKDWELL). The mean value
18# is used for optimisation. With the option
19# LOCK  off
20# or
21 # LOCK on
22
23LOCKDWELL         5      # redefine number of values, used to calculate
24# a mean lock_value (Minimum 3, Maximum 32, default 5). The measure-
25# ment is repeated until the range of values of a straight line
26# fitted through all measured points is less than the total noise.
27# For each gradient which is to be shimmed, a line similar to the
28#  following is needed:
29
30# Shim_name       Maximum_Step_Size           Number_of_Iterations
31    Z                    30                   2
32    Z2                   30                   2
33    Z                    15                   2
34    Z2                   15                   2
```

```
35    Z                   10                  2
36    Z2                  10                  2
37#   Z3                  10                  3
38# The shim method is, to increase one shim value by step-size until the
39# measured lock value decreases again. Then the step-size is reduced
40# according to the measured values. This is repeated number-of-iteration
41# times.
42#   Z                   5                   3
43#   Z2                  5                   3
44#   Z3                  5                   3
45
46#AUTOSHIM Z=5 Z2=2
47# For BSMS all gradients are allowed for AUTOSHIM.
```

7. Store text and exit the editor

8. Hit Return to answer ‘y’ for the question: Store always the same parameters

7.3.4 LOCK FILE SET UP

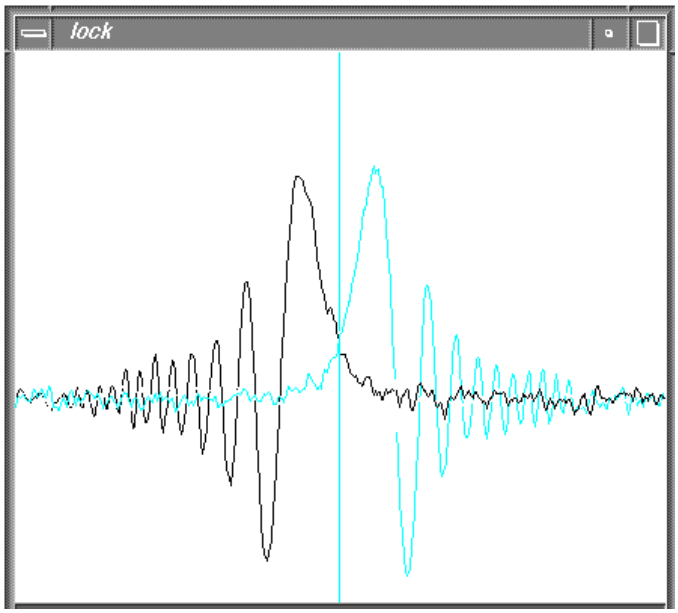
Sample: 0.1% Ethyl benzene in CDCl3 or any sample in CDCl3

1. Insert sample into the magnet

2. Type lock and select CDCl3

3. Press the ‘LOCK ON/OFF’ key on the BSMS panel (light should be off)

4. Center the lock trace within the lock window by changing the field value.



4. Press the Lock ON/OFF key to lock (light should be on)
5. Shim for best resolution
6. Press Phase and adjust the phase, write the value down
7. Type edlock at the XWINNMR command line

8. Click on ‘BSMS_FIELD’

NOTE: This procedure should be performed on a regular basis. (e.g. once a month)

9. Change the Lock Power value of all solvents as shown in the example below:

Solvent	**Lock Power**	**Solvent**	**Lock Power**
Acetic	-30	DMF	-30
Acetone	-36	DMSO	-20
CDCl3	-27	Dioxane	-30
CD2Cl2	-32	ETOH	-30
CDC3N	-30	MeOH	-40

C6D6	-30	THF	-30
D2O	-20	Tol	-30
DEE	-30	Pyr	-30
DME	-30		

10. Change LGain value of Acetic to -2.1 and click on ‘COPY_VALUE’
11. Change LTime value of Acetic to 0.272 and click on ‘COPY_VALUE’
12. Change LFilter value of Acetic to 150 and click on ‘COPY_VALUE’
13. Change LPhase value of Acetic to value found in step 6 and click on ‘COPY_VALUE’
14. Click on ‘SAVE’
15. Type lock CDCl3

NOTE: The system will enter the lockshift value of CDCl3 and automatically lock and adjust the lock gain

7.4 Optimizing Parameters

7.4.1 SUMMARY OF COMMANDS

Acquisition:

asedautomatic setup editor

eda edit acquisition parameters

ii initialize spectrometer interface

p1 - p31 pulse widths

pl1 - pl31 power levels

rga automatic receiver gain adjustment

rpar read parameter set

swh sweep width in Hz

wpar write parameter set

zg start acquisition

Pulse Programs:

decp90decoupler calibration

inv4ndrd1d1D inverse experiment with no decoupling

zg1D experiment with 90 degree pulse

zg301D experiment with 90 degree pulse

zgflqn1D 19F observe experiment

AU Programs:

paropt parameter optimization

Data:

edc create a new data set

Processing:

edp edit processing parameters

ef em + ft

efp em + ft + pk

em exponential multiplication

ft fourier transform

pk phase correction using PHC0 and PHC1

NOTE: For precise measurement of any pulse calibration it is essential to tune the probe to the observed nuclei.

7.4.2 DETERMINE THE 90^{o} PULSE LENGTH FOR 1H

7.4.2.1 High power level for proton observe

Sample: 0.1% Ethylbenzene in CDCl3

1. Type edc and change the following parameters:

NAME	=	test1h
EXPNO	=	1
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar PROTON all

4. Insert the sample into the magnet, type lock and select CDCl3, then shim for best homogeneity

5. Type eda and change the following parameters:

PULPROG	=	zg
TD	=	4 K
NS	=	1
DS	=	0
D1	=	20s
SWH	=	1000 Hz
O1p	=	2.5 ppm
SOLVENT	=	CDCl3
PROSOL	=	TRUE

6. Click on’ SAVE’

7. Type edp and change the following parameters:

SI	=	2 K
LB	=	1 Hz
PSCAL	=	global

8. Click on ‘SAVE’

9. Type ii

10. Type rga

11. Type zg

12. Type ef

13. Correct the phase

14. Expand the spectrum to display the quartet at 2.6 ppm only

15. Click on ‘utilities’

16. Click on ‘O1’

17. Move the cursor into the center of the quartet and click the middle mouse button to assign the O1 frequency

18. Click on ‘return’

19. Type ii

20. Type zg

21. Type efp

22. Click on ‘dp1’ and set the following parameters:

F1 = 2.9 ppm

F2 = 2.5 ppm

Change y-scaling on display according to PSCAL? = **y**

23. Type wpar protonp90 all

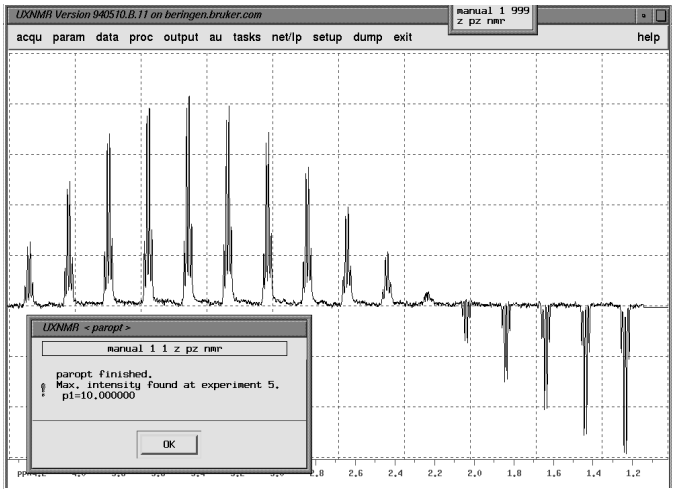
24. Type paropt and answer the following questions:

Enter parameter to modify: p1

Enter initial parameter value: 2

Enter parameter increment: 2

Enter # of experiments: 16



NOTE: At the end of the experiment, a message: "paropt finished" and a value for p1 is displayed. This will be the 90^o pulse length for the 1H transmitter. Write this value down! To obtain a more accurate value, follow steps 25 through 30 below.

25. Type re 1 1

26. Type p1 and change the value to be a 360^o pulse (multiply by 4 the value observed in step 24)

27. Type zg

28. Type efp

29. Change p1 in small increments until the signal goes through a null at 360^o

30. Simply divide the determined 360^o value by 4. This will be the 90^o pulse length for the 1H transmitter using the current probehead

31. Type solvloop and enter the following parameters:

L1H_tr = 0

P1H_tr = value determined in step 30

7.4.2.2 Low power level for MLEV spinlock

NOTE: To determine the power level for the MLEV spinlock, a 90^o pulse length of 30-35 usec is desirable.

1. Type re 1 1
2. Type p1 and change the value to 35 usec
3. Type paropt and answer the following questions:

   Enter parameter to modify: pl1

   Enter initial parameter value: 2

   Enter parameter increment: 1

   Enter # of experiments: 16

NOTE: At the end of the experiment, a message: "paropt finished" and a value for pl1 is displayed. This will be the 1H transmitter power for a 90^o pulse width of 35 usec. Write this value down! To obtain a more accurate value, follow steps 4 through 10 below. (Starting at 0 dB, increasing the attenuation by 6 dB, the pulse width should double).

4. Type re 1 1
5. Type pl1 and change to the value determined in step 3
6. Type pl and change the value to be a 360^o pulse (multiply 35usec by 4)
7. Type zg
8. Type efp
9. Change p1 in small increments until the signals go through a null at 360^o

10. Simply divide the determined 360^o value by 4

11. Type solvloop and enter the following parameters:

L1H_ml	=	(value determined in step 3)
P1H_mll	=	(value determined in step 10)

7.4.2.3 Low power level for ROESY spinlock

NOTE: To determine the power level for the ROESY spinlock a 90^o pulse length of 140 - 160 usec is desirable.

1. Type re 1 1

2. Type p1 and change the value to 150 usec

3. Type paropt and answer the following questions:

Enter parameter to modify:	pl1
Enter initial parameter value: 10	
Enter parameter increment:	1
Enter # of experiments:	20

NOTE: At the end of the experiment, a message: "paropt finished" and a value for pl1 is displayed. This will be the 1H transmitter power for a 90^o value of 150 usec. Write this value down! To obtain a more accurate value, follow steps 4 through 9 below. (Starting at 0 dB, increasing the attenuation by 6 dB, the pulse width should double).

4. Type re 1 1

5. Type p1 and change the value to be a 360^o pulse (multiply 150 usec by 4)

6. Type zg

7. Type efp

8. Change p1 in small increments until the signals go through a null at 360^o

9. Simply divide the determined 360^o value by 4

10. Type solvloop and enter the following parameters:

L1H_roe	=	(value determined in step 9)
P1H_roe	=	250000 usec

7.4.3 DETERMINE THE 90^o PULSE LENGTH FOR 13C

Sample: 80% Benzene in Acetone-d6

1. Type edc and change the following parameters:

NAME	=	test13c
EXPNO	=	1
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar C13CPD all

4. Insert the sample into the magnet, type lock and select Acetone, then shim for best homogeneity

5. Type eda and change the following parameters:

PULPROG	=	zg
TD	=	4 K
NS	=	1
DS	=	0
D1	=	10 s
SWH	=	1000 Hz
O1p	=	127 ppm
SOLVENT	=	Aceton
PROSOL	=	TRUE

6. Type edp and change the following parameters:

SI	=	2 K
PSCAL	=	global

7. Click on ‘SAVE’

8. Type ii

9. Type rga

10. Type zg

11. Type ef

12. Correct the phase (both peaks positive)

13. Expand the spectrum to display the doublet

14. Click on ‘utilities’

15. Click on ‘O1’

16. Move the cursor into the center of the doublet and click the middle mouse button to assign the O1 frequency

17. Click on ‘return’

18. Type ii

19. Type zg

20. Type efp

21. Click on ‘dp1’ and set the following parameters:

F1 = 131 ppm

F2 = 127 ppm

Change y-scaling on display according to PSCAL? = y

22. Type wpar c13p90 all

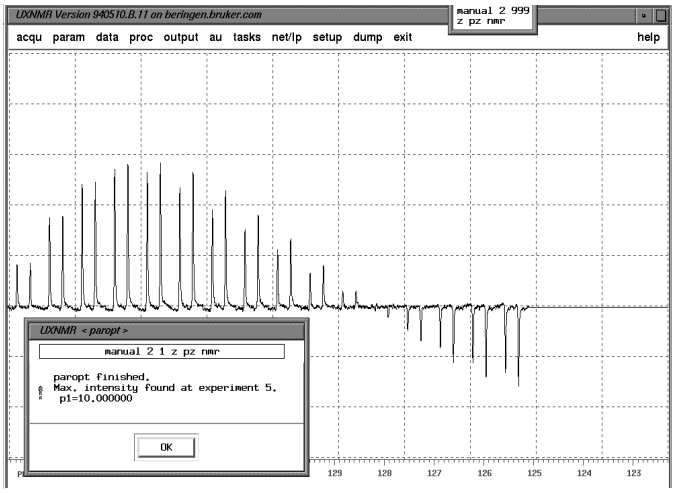
23. Type paropt and answer the following questions:

Enter parameter to modify: p1

Enter initial parameter value: 2

Enter parameter increment: 2

Enter # of experiments: 16



NOTE: An the end of the experiment, a message: ”paropt finished” and a value for p1 is displayed. This will be the 90^{o} pulse length for the 13C transmitter. Write this value down! To obtain a more accurate value, follow the steps 24 through 29 below.

24. Type re 1 1

25. Type pl and change the value to be a 360^{o} pulse (multiply the value observed in step 23 by 4)

26. Type zg

27. Type efp

28. Change p1 in small increments until the signals go through a null at 360^{o}

29. Simply divide the determined 360^{o} value by 4. This will be the 90^{o} pulse length for the 13C transmitter using the current probehead.

30. Type solvloop and enter the following parameters:

L13C_tr	=	0
P13C_tr	=	(value determined in step 29)

NOTE: WARNING! If the 90^o pulse length is less then 5 usec for 5m probes and less then 10 usec for 10mm probes, the probe is at risk of arcing. To prevent arcing, change the transmitter power pl1 to a higher dB value and repeat steps 25 through 29.

7.4.4 DETERMINE THE 90^o PULSE LENGTH FOR THE DECOUPLING NUCLEUS 1H

7.4.4.1 At high power

Sample: 80 % Benzene in Acetone-d6

1. Type edc and change the following parameters:

NAME	=	testdec
EXPNO	=	1
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar c13p90 all

NOTE: if the parameter files c13p90 do not exist, follow the instructions in CHAPT. 3C steps 2 through 22.

4. Insert the sample into the magnet, type lock and select Acetone, then shim best

homogeneity

5. Type eda and set the following parameters:

PULPROG	=	decp90
O2p	=	7.28
D2	=	3.125 ms
CNST[2]	=	160
P3	=	2 us
PL2	=	0 dB
SOLVENT	=	ACETONE
PROSOL	=	TRUE

6. Click on ‘SAVE’

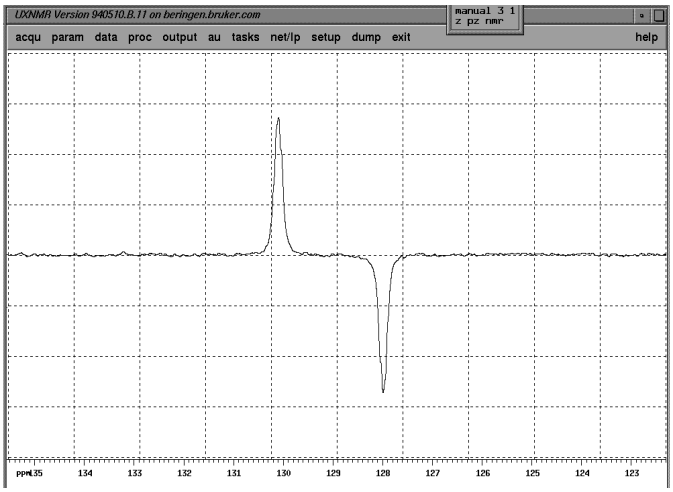
7. Type wpar decp90 all

8. Type rga

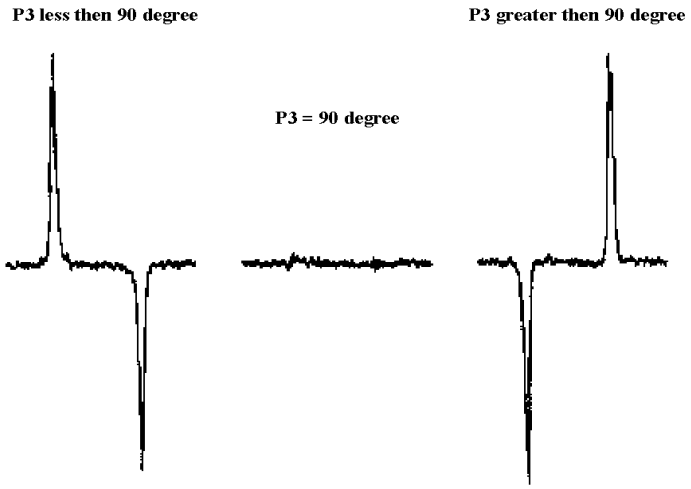
9. Type zg

10. Type ef

11. Correct the phase with the left signal up and the right down



12. Increase p3 in increments of 1 or 2 usec, execute zg and efp until the signals become zero. This is where p3 is 90^{o} at maximum power.



7.4.4.2 At low power for WALTZ decoupling

Sample: 80 % Benzene in Acetone-d6

1. Type edc and change the following parameters:

NAME	=	testdec
EXPNO	=	2
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar decp90 all

NOTE: if the parameter files decp90 do not exist, follow the instructions in “At high power” on page 111.

4. Insert the sample into the magnet, type lock and select Acetone, then shim best homogeneity

5. Increase pl2 and p3 until you find a 90 ^{o} pulse between 100 and 120 usec (starting at 0dB, increase the attenuation by 6 dB, the pulse width should double). This will be the 90^{o} pulse for WALTZ decoupling

6. Type solvloop and enter the following parameters:

L1H_hi	=	0
L1H_lo	=	(pl2 value for WALTZ determined in step 5)
L1H_cpn	=	L1H_lo (for 5mm probes) = L1H_lo
		(for 10mm probes add 6dB)
P1H-hi	=	(p3 value determined in step 12)
P1H_lo	=	(p3 value for WALTZ determined in step 5)

7.4.5 DETERMINE THE 90^{o} PULSE LENGTH FOR INVERSE MODE

7.4.5.1 Determination of the 90^{o} high power pulse for 13C in inverse mode

Sample: 10% CHCl3 in Aceton-d6(for instruments up to 300MHz)

3% CHCl3 in Acetone-d6(for instruments 400MHz and up)

1. Type edc and change the following parameters:

NAME	=	testinv
EXPNO	=	1
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar PROTON all

4. Insert the sample into the magnet, type lock and select Aceton, then shim for best homogeneity

5. Type eda and change the following parameters:

PULPROG	=	decp90
TD	=	8 K
NS	=	1
DS	=	0
D1	=	5 s
D2	=	2.34 ms
SWH	=	1000 Hz

NUCLEI	=	click on edit and switch NUC2 to 13C
O1p	=	8 ppm
O2p	=	79 ppm
CNST[2]	=	210 Hz
SOLVENT	=	Acetone
PROSOL	=	TRUE

6. Click on ‘SAVE’

7. Type edp and change the following parameters:

SI	=	4 K
PSCAL	=	global

8. Type ii

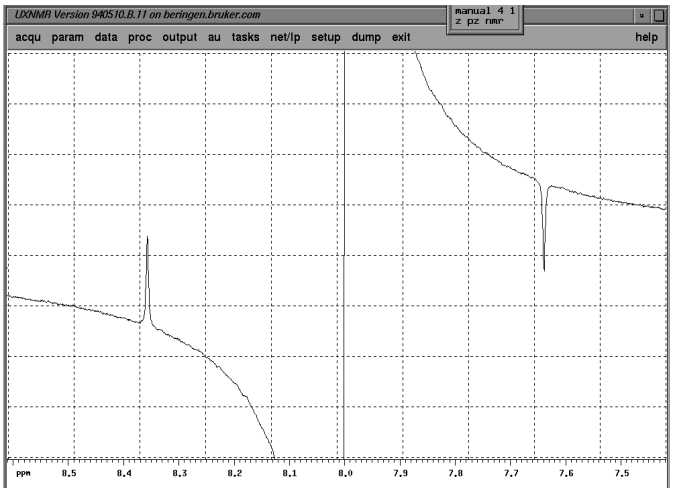
9. Type rga

10. Type zg

11. Type ef

12. Display the Chloroform peak from 8.5 - 7.5 ppm to include the 13C satellites

13. Correct the phase pointing the left satellite up and the right down



14. Type wpar inv90 all

13. Type p3 and increase the value by 2 us

14. Type zg

15. Type efp

16. Repeat steps 13 through 15 until the intensity of the 13C satellites becomes zero. This value of p3 will be the 90^o pulse length for the 13C channel in inverse mode. Write down this value!

17. Type solvloop and enter the following parameters:

P13C_hi = (p3 value from step16)

7.4.5.2 Inverse test experiment without 13C decoupling

Sample: 10% CHCl3 in Aceton-d6(for instruments up to 300MHz)

3% CHCl3 in Acetone-d6(for instruments 400MHz and up)

1. Type edc and change the following parameters:

NAME	=	testinv
EXPNO	=	2
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar inv90 all

NOTE: if the parameter file inv90 do not exist, follow the instructions in “Determination of the 90o high power pulse for 13C in inverse mode” on page 115.

4. Insert the sample into the magnet, type lock and select Aceton, then shim for best homogeneity

5. Type eda and change the following parameter:

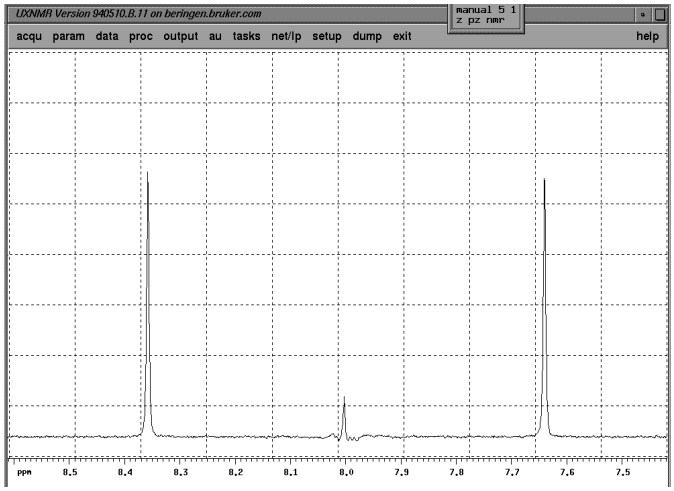
PULPROG	=	inv4ndrd1d
NS	=	16
DS	=	4
D1	=	20 s
D13	=	3 us
SOLVENT	=	Acetone
PROSOL	=	TRUE

6. Click on ‘SAVE’

7. Type zg

8. Type efp

9. Correct the phase pointing both satellites up



7.4.5.3 Determination of the 90^{o} low power pulse for 13C GARP decoupling.

Sample: 10% CHCl3 in Aceton-d6(for instruments up to 300MHz)

3% CHCl3 in Acetone-d6(for instruments 400MHz and up)

NOTE: To determine the power level for 13C GARP decoupling a 90^{o}pulse length of 60 - 65 usec is desirable.

1. Type edc and change the following parameters:

NAME = testinv

EXPNO = 3

PROCNO = 1

2. Click on ‘SAVE’

3. Type rpar inv90 all

NOTE: if the parameter file inv90 do not exist, follow the instructions in “Determination of the 90o high power pulse for 13C in inverse mode” on page 115.

4. Insert the sample into the magnet, type lock and select Aceton, then shim for best homogeneity

5. Type eda and set the following parameters:

SOLVENT = Acetone

PROSOL = TRUE

6. Click on ‘SAVE’

7. Type p3 and change the value to 64 us

8. Type pl2 and increase the value by 2 dB

9. Type zg

10. Type ef

11. Correct the phase pointing the left satellite up and the right down

12. Repeat steps 9 through 11 using the command efp until the intensity of the 13C satellites becomes zero. (starting at 0dB, increase the attenuation by 6 dB, the pulse width should double). To determine an accurate null, the value of p3 may be varied. The final value of p3 will be the 90^{o} pulse length at low power for 13C GARP decoupling. Write down this value!

13. Type solvloop and enter the following parameters:

L13C_lo = (pl2 value determined in step 12)

P13C_lo = (p3 value determined in step 12)

7.4.6 DETERMINE THE 90^{o} PULSE LENGTH FOR 19F

Sample: 0.05% Trfluorotoluene in CDCl3

.

1. Type edc and change the following parameters:

NAME	=	test19f
EXPNO	=	1
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar F19 all

4. Insert the sample into the magnet, type lock and select CDCl3, then shim for best homogeneity

5. Type eda and change the following parameters:

PULPROG	=	zgflqn
TD	=	4 K
NS	=	1
DS	=	0
D1	=	5 s
SWH	=	1000 Hz
O1p	=	-63 ppm
SOLVENT	=	CDCl3
PROSOL	=	TRUE

6. Type edp and change the following parameters:

SI = 2 K

PSCAL = global

7. Click on ‘SAVE’

8. Type ii

9. Type rga

10. Type zg

11. Type ef

12. Correct the phase

13. Expand the spectrum to display the signal at -63ppm only

14. Click on ‘utilities’

15. Click on ‘O1’

16. Move the cursor into the center of the peak and click the middle mouse button to assign the O1 frequency

17. Click on ‘return’

18. Type ii

19. Type zg

20. Type efp

21. Click on ‘dp1’ and set the following parameters:

F1 = -62.9 ppm

F2 = -63.1 ppm

Change y-scaling on display according to PSCAL? = y

22. Type wpar f19p90 all

23. Type paropt and answer the following questions:

Enter parameter to modify:	p1
Enter initial parameter value:	2
Enter parameter increment:	2
Enter # of experiments:	16

NOTE: An the end of the experiment, a message: "paropt finished" appears and a value for p1 is displayed. This will be the 90^o pulse length for the 19F transmitter. Write this value down!
To obtain a more accurate value, follow the steps 24 through 29 below.

24. Type re 1 1

25. Type pl and change the value to be a 360^o pulse (multiply the value observed in step 23 by 4)

26. Type zg

27. Type efp

28. Change p1 in small increments until the signals go through a null at 360^o

29. Simply divide the determined 360^o value by 4. This will be the 90^o pulse length for the 19F transmitter using the current probehead

30. Type solvloop and enter the following parameters:

L19F_tr	=	0
P19F_tr	=	(value determined in step 29)

NOTE: <u>WARNING!</u> If the 90^o pulse length is less then 5 usec for 5m probes and less then 10 usec for 10 mm probes, the probe is at risk of arcing. To prevent this from happening, change the transmitter power pl1 to a higher dB value and repeat steps 25 through 29.

7.4.7 DETERMINE THE 90^{o} PULSE LENGTH FOR 31P

Sample: 0.0485 M Triphenylphophate in Acetone-d6

1. Type edc and change the following parameters:

NAME	=	test31p
EXPNO	=	1
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar P31 all

4. Insert the sample into the magnet, type lock and select Acetone, then shim for best homogeneity

5. Type eda and change the following parameters:

PULPROG	=	zg
TD	=	4 K
NS	=	1
DS	=	0
D1	=	15 s
SWH	=	1000 Hz
O1p	=	-15 ppm
SOLVENT	=	Acetone
PROSOL	=	TRUE

6. Type edp and change the following parameters:

SI	=	2 K
LB	=	1 Hz
PSCAL	=	global

7. Click on ‘SAVE’

8. Type ii

9. Type rga

10. Type zg

11. Type ef

12. Correct the phase

13. Expand the spectrum to display the signal at -16.4 ppm only

14. Click on ‘utilities’

15. Click on ‘O1’

16. Move the cursor into the center of the peak and click the middle mouse button to assign the O1 frequency

17. Click on ‘return’

18. Type ii

19. Type zg

20. Type efp

21. Click on ‘dp1’ and set the following parameters:

F1	=	-16.1 ppm
F2	=	-16.7 ppm

Change y-scaling on display according to PSCAL? = y

22. Type wpar p31p90 all

23. Type paropt and answer the following questions:

Enter parameter to modify:	p1
Enter initial parameter value:	2
Enter parameter increment:	2
Enter # of experiments:	16

NOTE: An the end of the experiment, a message: "paropt finished" appears and a value for p1 is displayed. This will be the 90^o pulse length for the 31P transmitter. Write these value down!
To obtain a more accurate value, follow the steps 24 through 29 below.

24. Type re 1 1

25. Type pl and change the value to be a 360^o pulse (multiply the value observed in step 23 by 4)

26. Type zg

27. Type efp

28. Change p1 in small increments until the signals go through a null at 360^o

29. Simply divide the determined 360^o value by 4. This will be the 90^o pulse length for the 31P transmitter using the current probehead

30. Type solvloop and enter the following parameters:

L31P_tr	=	0
P31P_tr	=	(value determined in step 29)

NOTE: <u>WARNING!</u> If the 90^o pulse length is less then 5 usec for 5m probes and less then 10 usec for 10mm probes, the probe is at risk of arcing. To prevent this from hap-

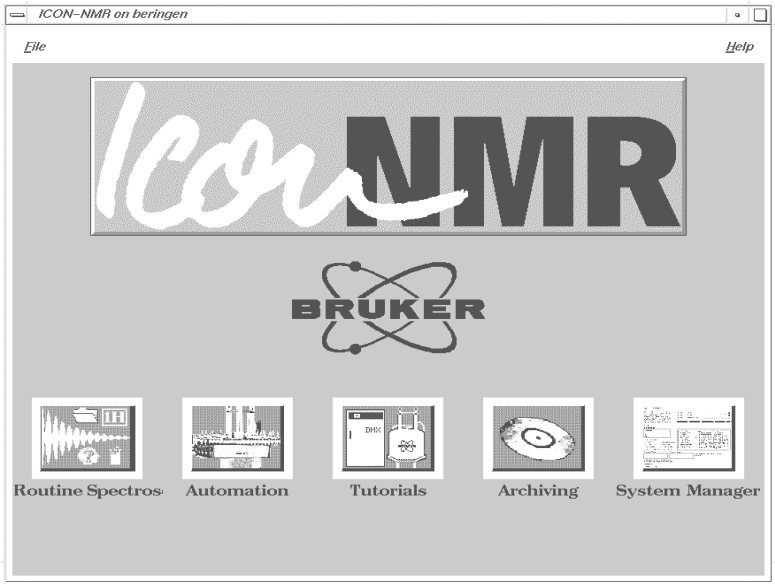
pening, change the transmitter power pl1 to a higher dB value and repeat steps 25 through 29.

7.5 Set up of ICON-NMR

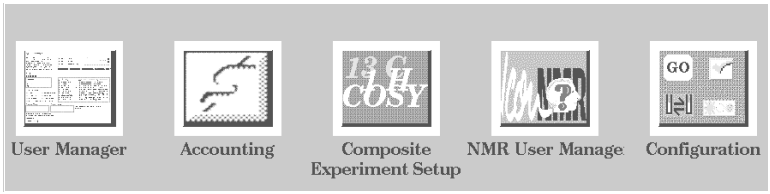
NOTE: It is only necessary to complete the steps in Chapter 4, if ICON-NMR is being set up for the first time. Please refer to the on-line ICON-NMR manual for customization beyond what is shown here.

7.5.1 User Manager

1. Type iconnmr

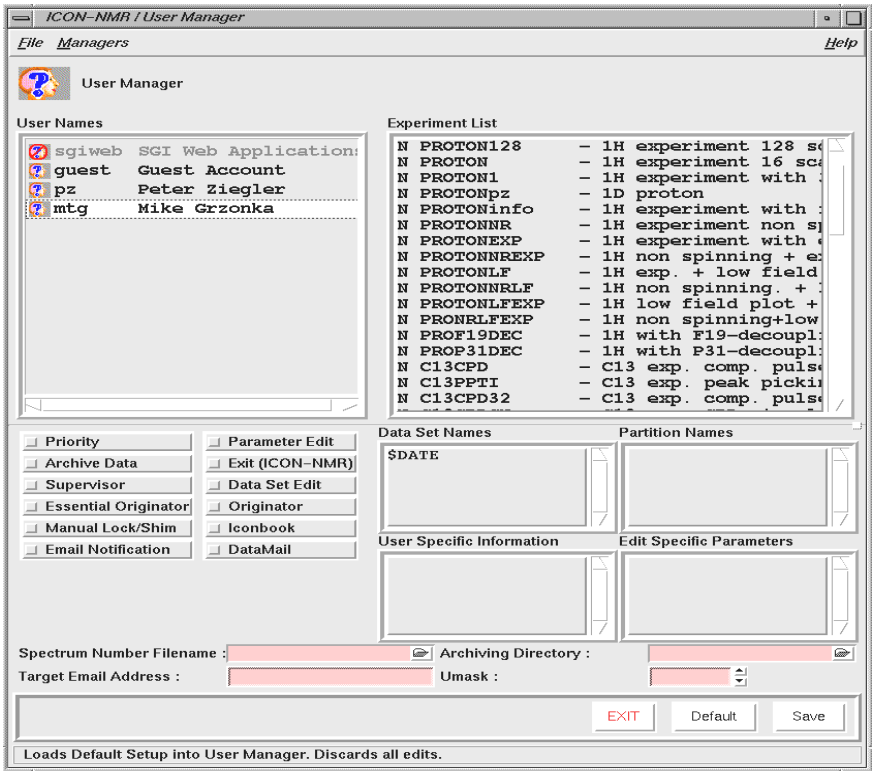


2. Click on System Manager



3. Click on User Manager

4. Enter NMR Super user password



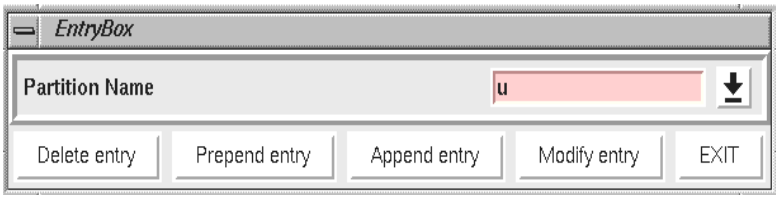
5. Select a user name by clicking on it

NOTE: If the Experiment List is blank click on 'Default' to load the default experiment list.

6. Click on the square button in the field 'Exit (ICON-NMR)' to allow permission to exit ICON-NMR

7. Click on the square button in the field 'Data Set Edit' to allow permission to edit the Data Set Filename

8. Click inside the field 'Partition Names'



9. Type the name of the disk partition where the data will be stored (e.g. u)

10. Click on 'Append entry'

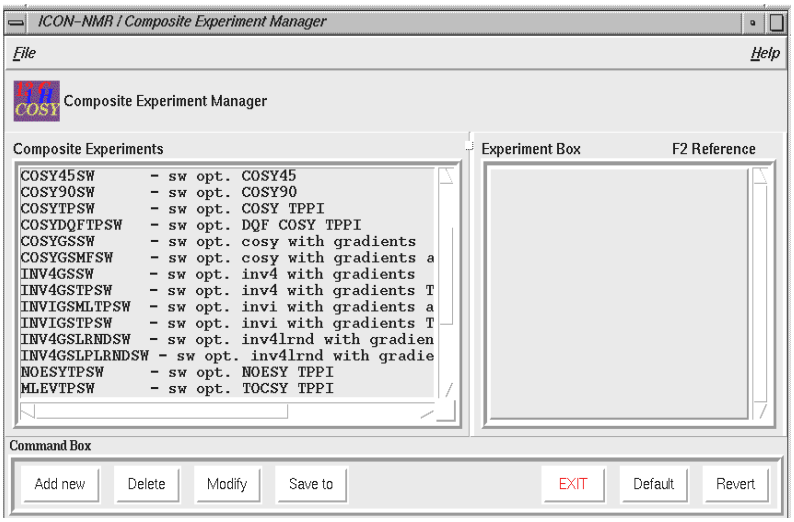
11. Click on 'EXIT'

12. Click on 'Save & EXIT' in the Warning window

7.5.2 Composite Experiment Set up

1. Click on 'Composite Experiment Setup'

2. Enter NMR Super user password

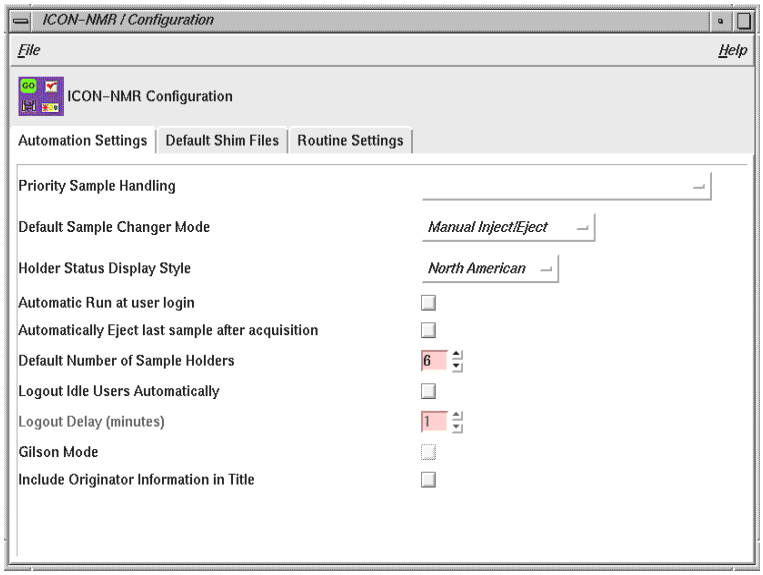


3. Click on ‘Default’ to load the list of composite experiments
4. Click on ‘EXIT’
5. Click on ‘Save & EXIT’ in the Warning window

7.5.3 Configuration

1. Click on ‘Configuration’
2. Enter the NMR Super user password

3. Click on ‘Default Sample Changer Mode’ and select the operation mode that

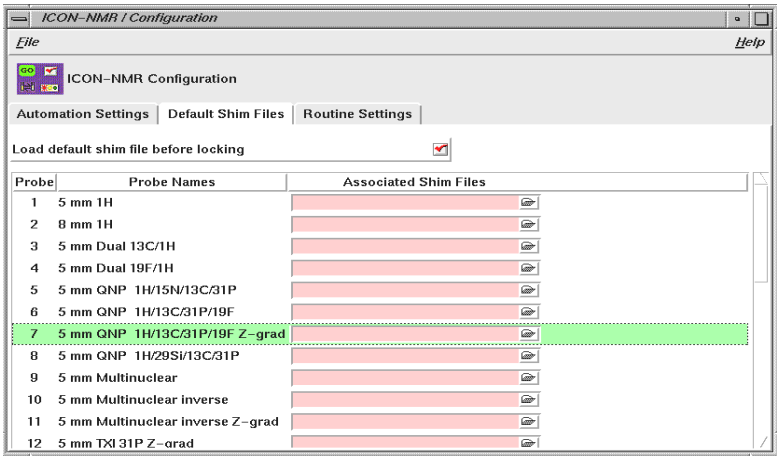


the system should default to.

4. Click on ‘Holder Status Display Style’ and select North American

5. Select the number of sample holders. For Manual and SIXPACK operation select 6 holders and for BACS enter 60 or 120 holders.

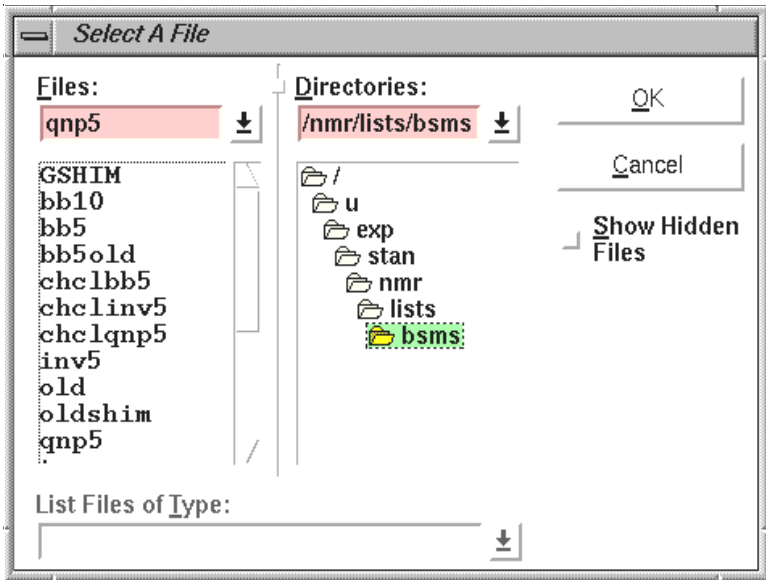
6. Click on ‘Default Shim Files’



7. Click on the small square ‘Load default shim file before locking’

8. Click on the file icon of the current probe head

9. Select a good shim file for the current probe head in the directory: /u/exp/stan/nmr/lists/bsms



NOTE: Select a shim file which can be used for running routine samples and update this file regularly.

10. Click on ‘OK’

11. Click on ‘File’ and click on ‘EXIT’

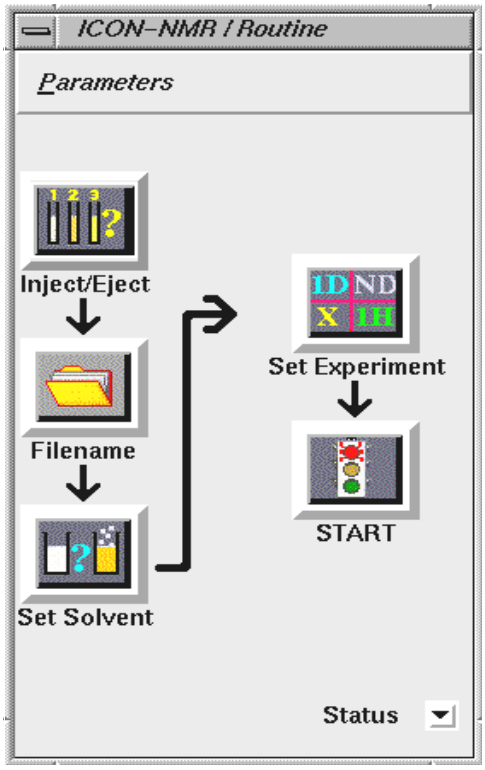
12. Click on ‘Save & EXIT’ in the Warning window

13. Close the ICON-NMR System Manager window by clicking on ‘File’ and then on ‘EXIT’

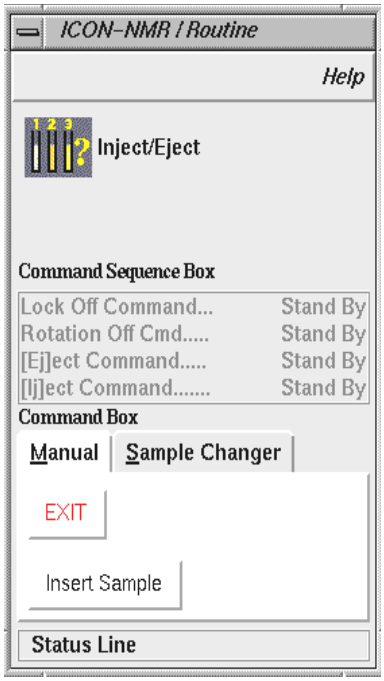
7.6 Routine Spectroscopy

NOTE: Routine Spectroscopy is designed to help users perform NMR experiments by following a step by step menu.

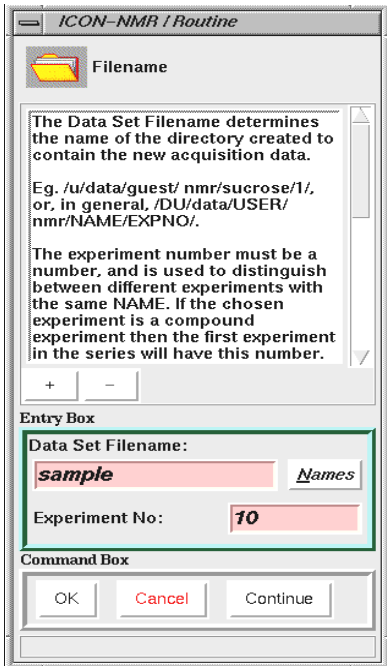
1. Type iconnmr
2. Click on ‘Routine Spectroscopy’
3. Click on ‘Identify User’
4. Select user
5. Click on ‘OK’
6. Enter the password



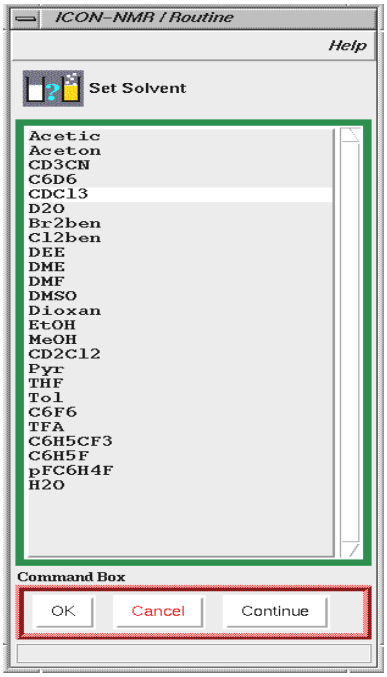
7. Click on ‘Inject/Eject’



8. Click on ‘Insert Sample’ and wait until the ”Insert Sample” window appears
9. Insert your sample
10. Click on ‘OK’

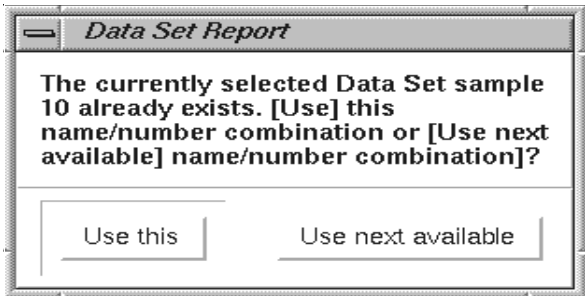


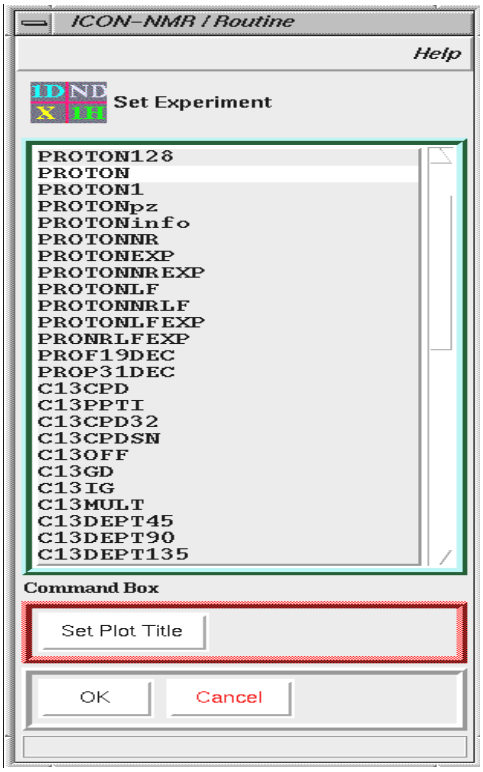
11. Type sample for Data Set Filename
12. Type 10 for Experiment No.
13. Click on ‘OK’
14. Click on ‘Continue’



15. Select solvent e.g. CDCl3
16. Click on ‘Continue’

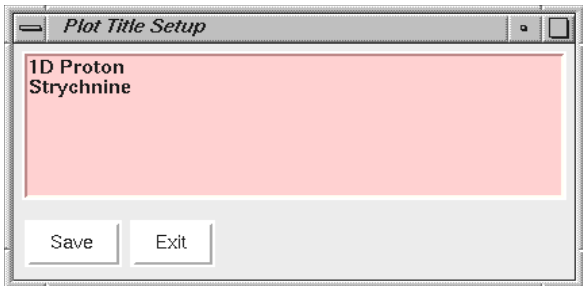
NOTE: If the data set name ”sample 10” already exists, a Data Set Report window appears which gives the option to overwrite the existing data set or to increment the experiment number to the next available one.



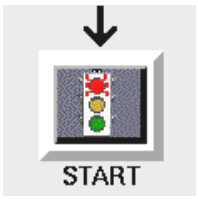


17. Select the experiment PROTON

18. Click on ‘Set Plot Title’

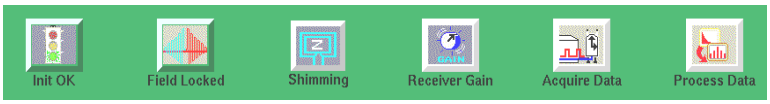


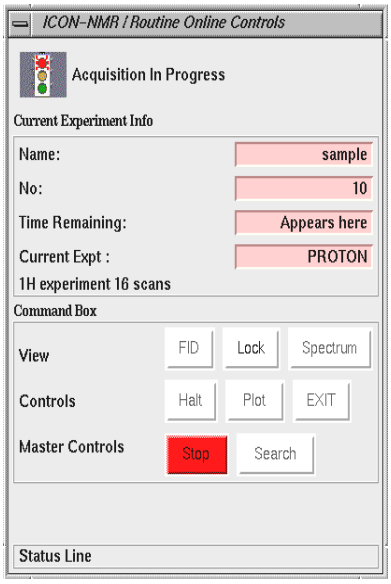
19. Click in the ”Plot Title Setup” window and enter a plot title
20. Click on ‘Save’
21. Click on ‘OK’



22. Click on ‘START’

NOTE: At this point an ”Online Control” window and the acquisition status icons appear on the screen.



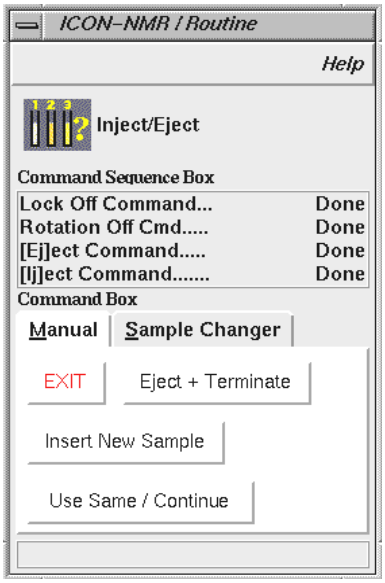


Online Control function keys:

FID	display the acquisition window
	(active after the acquisition has started
Lock	view the lock window
Spectrum	view the spectrum window
Halt	stop the acquisition after the next scan
	is completed then start the processing
Plot	plot data or additional expansions
EXIT	to exit back to ICON-NMR/Routine window
Stop	stop all processes and do not continue

Search open the Portfolio Editor

23. Click on ‘EXIT’ after all processing status icons stop flashing



24. Click on ‘Eject + Terminate’

25. Remove your sample and insert another other sample if desired.

NOTE: It is generally better to keep a sample in the magnet if the instrument will not be used for a long periods of time.

7.7 Automation

NOTE: Automation is designed to run experiments using a variety of sample changers and can also be used in manual sample change operation allowing greater flexibility than “Routine Spectroscopy” on page 133.

7.7.1 SAMPLE SET UP

NOTE: The following setup examples are simple exercises to demonstrate the different acquisition method in ICON-NMR using two samples.

Samples: ~0.3 M in 0.6 ml CDCl3 of a small organic molecule such as Strychnine

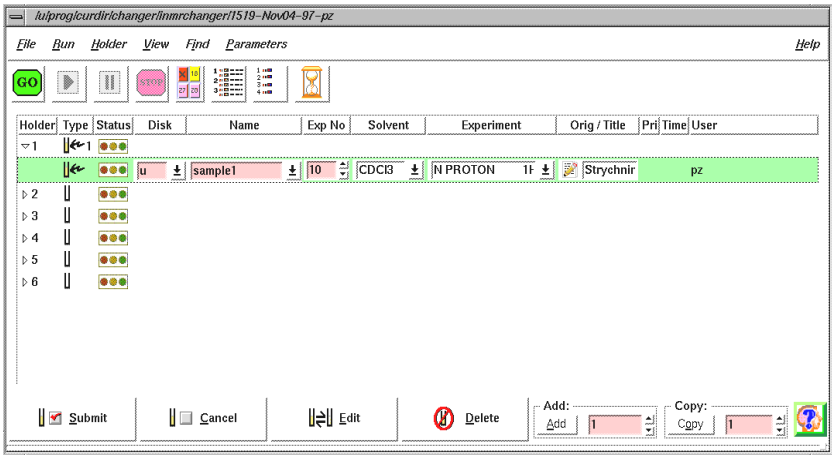
~0.3 M in 0.6 ml DMSO of a small organic molecule such as Carvone

~0.3 M in 0.6 ml CDCl3 of a small organic molecule such as Menthol

1. Type iconnmr
2. Click on ‘Automation’
3. Click on ‘Identify User’
4. Select user
5. Click on ‘OK’
6. Enter the user’s password

7.7.1.1 Set up of one single experiment

7. Double click on Holder 1



NOTE: The data is stored to disk ”u” by default. The current date is the Name and the Exp No is the first free experiment number under that name.

8. Click inside the Name window and change the name to sample1

9. Click on the small button to the right of the solvent window and click on ”CDCl3”

10. Click on the small button to the right of the Experiment window and select ”PROTON” by clicking on it

NOTE: ”PROTON” is a single experiment and is marked with the letter N.

11. Click on the small icon to the left of the Orig/Title window and enter a title

12. Click on the first line of Holder 1

13. Click on ‘Submit’

NOTE: The Status traffic light changes to yellow to indicate the holder is ready to run and the experiment time is calculated. Proceed to “SAMPLE RUN” on page 150” to start the automation.

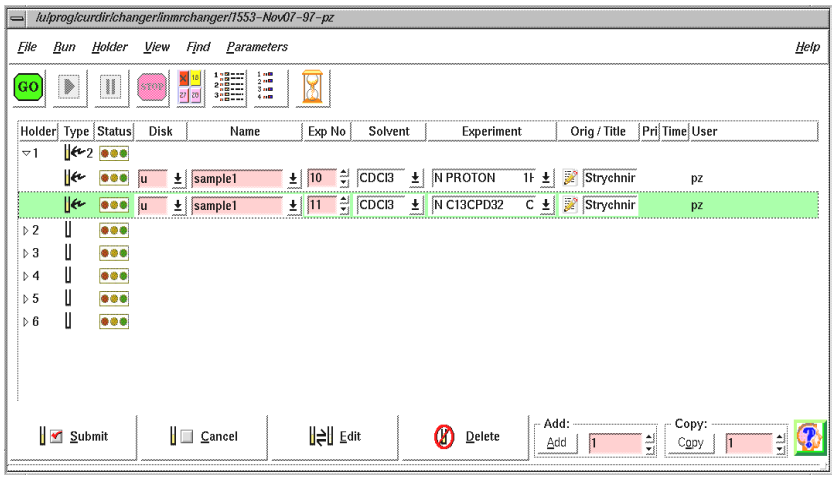
7.7.1.2 Set up of two single experiments

1. Follow steps 7 through 11 of “Set up of one single experiment” on page 142.

2. Click on ‘Add’

NOTE: To add one experiment, regardless if it is a single or composite one, a ”1” should be displayed in the window next to the ‘Add’ button. A new line opens up containing the same information for the Disk, Name, Solvent and Orig/Title. The Exp No is incremented by 1.

3. Click on the small button to the right of the Experiment window and select C13CPD32 by clicking on it



4. Click on the first line of Holder 1

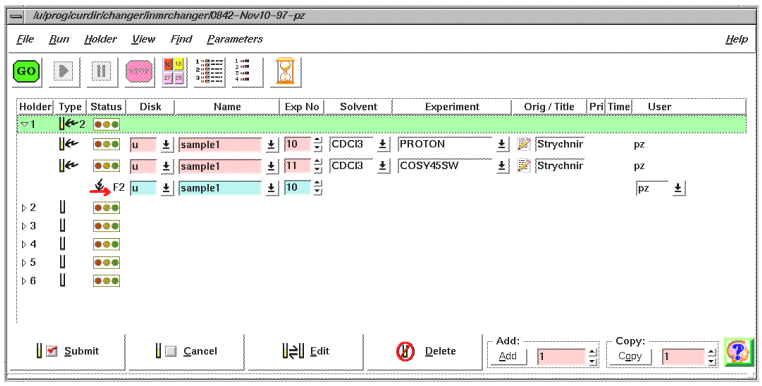
5. Click on ‘Submit’

NOTE: The Status traffic lights of the two experiments change to yellow to indicate the holder is ready to run and the experiment time of each experiment is calculated. Proceed to “SAMPLE RUN” on page 150 to start the automation.

7.7.1.3 Set up of a composite experiment

1. Follow steps 7 through 9 of “Set up of one single experiment” on page 142.

2. Click on the small button to the right of the Experiment window and select COSY45SW by clicking on it



NOTE: ”COSY45SW” is a composite experiment and is marked with the letter C. A composite experiment contains two or more single experiments. In this case the ”COSY45SW” is a 2D Homonuclear Correlation experiment which needs a preparation experiment ”PROTON” to prepare the ”COSY” experiment to optimize the 2D

parameters. The preparation experiment is entered automatically into the setup on the present holder. The system will check if a preparation experiment already exists on the present holder so that it does not run the experiment twice.

4. Click on the small icon to the left of the Orig/Title window and type a title

5. Click on the first line of Holder 1

6. Click on ‘Submit’

NOTE: The Status traffic lights of the two experiments change to yellow to indicate the holder is ready to run and the experiment time of each experiment is calculated. Proceed to “SAMPLE RUN” on page 150 to start the automation.

7.7.1.4 Set up of two composite experiments

1. Follow steps 7 through 9 of “Set up of one single experiment” on page 142.

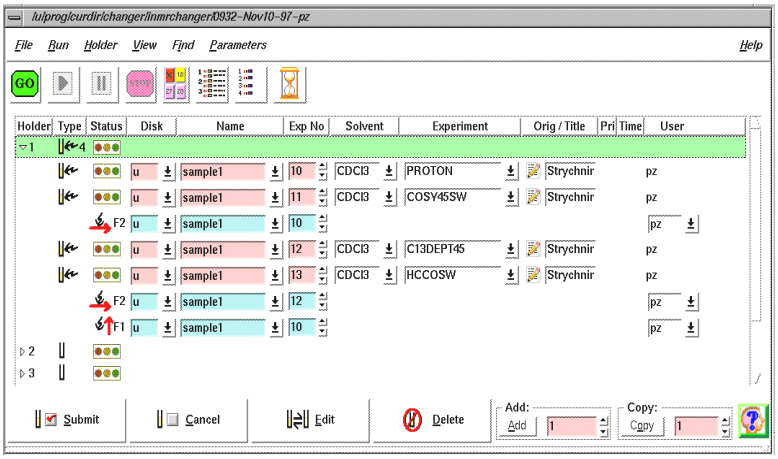
2. Click on the small button to the right of the Experiment window and select COSY45SW by clicking on it

3. Click on the small icon to the left of the Orig/Title window and type a title

4. Click on ‘Add’

NOTE: To add one experiment, regardless if it is a single or composite one a ”1” should be displayed in the window next to the ‘Add’ button. A new line opens up which contains the same information for the Disk, Name, Solvent and Orig/Title. The Exp No is incremented by 1.

5. Click on the small button to the right of the Experiment window and select HCCOSW by clicking on it



NOTE: ”HCCOSW” is a composite experiment like COSY45SW and is marked with the letter C. It is a 2D Hetero Correlation experiment which needs two 1D preparation experiments to optimize the 2D parameters in both dimensions. In this case ”PROTON” and ”C13DEPT45” are used for the optimization. Since the ”PROTON” experiment already exists, only the ”C13DEPT45” is automatically entered into the setup on the present holder.

5. Click on the first line of Holder 1
6. Click on ‘Submit’

NOTE: The Status traffic lights of the four experiments change to yellow to indicate the holder’s ready to run and the experiment time of each experiment is calculated. Proceed to “SAMPLE RUN” on page 150 to start the automation.

7.7.1.5 Set up of two or more holders with different solvents and experiments

1. Follow steps 7 through 13 of “Set up of one single experiment” on page 142.

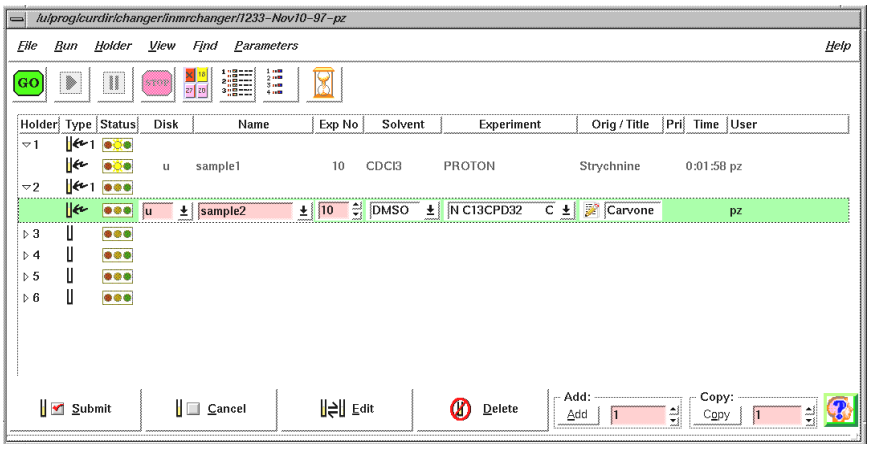
2. Double click on Holder 2

3. Click inside the Name window and change the name to sample2

4. Click on the small button to the right of the solvent window and select ”DMSO” by clicking on it

5. Click on the small button to the right of the Experiment window and select “C13CPD32” by clicking on it

6. Click on the small icon to the left of the Orig/Title window and type a title



7. Click on the first line of Holder 2

8. Click on ‘Submit’

NOTE: The Status traffic light changes to yellow to indicate the holder is ready to run and the experiment time is calculated. Proceed to “SAMPLE RUN” on page 150.

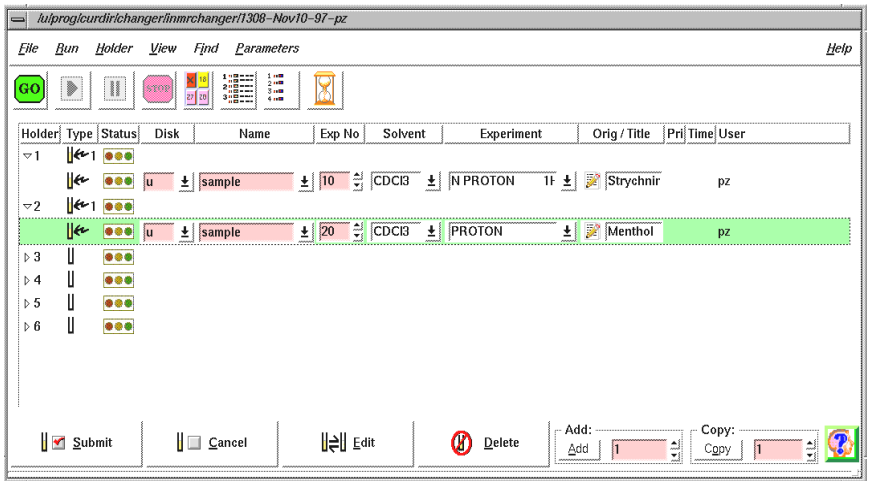
7.7.1.6 Set up of two or more holders with the same solvents and experiments

1. Follow steps 7 through 11 of “Set up of one single experiment” on page 142.
2. Click on ‘Copy’

NOTE: To copy the setup from one holder into another holder click on the ‘copy’ button. The number displayed in the window next to the ‘Copy’ button is to how many holders it will copy to. For this example use ”1”. The next holder will have the same setup as the previous one except that the Exp No is incremented to the next number divisible by 10 (e.g. 20). To edit the new holder, follow the next steps below.

3. Click on the experiment line in holder 2
4. Click on ‘Edit’
5. Click on the small icon to the left of the Orig/Title window and change the title

NOTE: In addition to the title all other entries can be edited in the new holder. DO NOT CHANGE!! the Exp No, unless you are sure the new Exp No does not already exist.



6. Click on the first line of holder 1
7. Press and hold the ”Shift” key
8. Click on the first line of holder 2
9. Release the ”Shift” key

NOTE: Both holders are now highlighted.

10. Click on ‘Submit’

NOTE: The Status traffic lights on both holders change to yellow to indicate the holder’s ready to run and the experiment time for each experiment is calculated. Proceed to “SAMPLE RUN” on page 150 to start the automation.

7.7.2 SAMPLE RUN

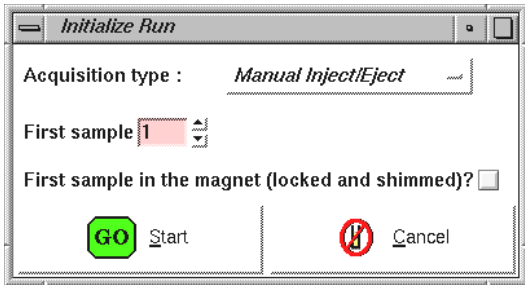
NOTE: Be sure that cfbacs has been done according to the instruction in “CONFIGURATION OF THE AUTOMATION” on page 89.

7.7.2.1 Manual Inject/Eject

NOTE: As an example set up to run under manual Inject/Eject mode, use the sample setup 7.7.1.1, 7.7.1.2, 7.7.1.3 or 7.7.1.4.

1. Click on ‘GO’

NOTE: The ”Initialization Run” window appears. The default ”Acquisition type” displayed is what has been set in the ICON-NMR configuration.



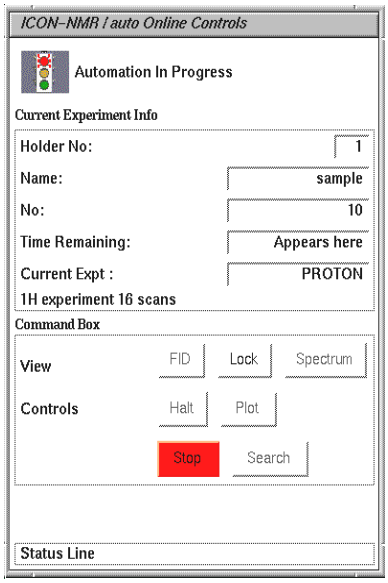
2. Click on Acquisition type button and select ‘Manual Inject/Eject’

3. Select ”1” for First sample

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.

4. Click on ‘Start’

NOTE: At this point the ”ICON-NMR / Auto Online Controls” window and the ”acquisition status icons” appear.

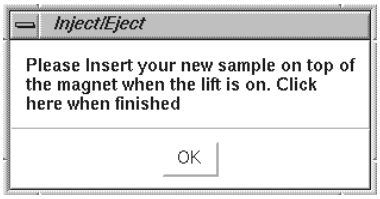


Online Control function keys:

FID	displaytheacquisitionwindow(activeaftertheacquisition has started)
Lock	view the lock window
Spectrum	view the spectrum window
Halt	stop the acquisition after the next scan is completed then start the processing
Plot	plot data or additional expansions
EXIT	exit back to ICON-NMR/Routine window
Stop	stop all processes and do not continue

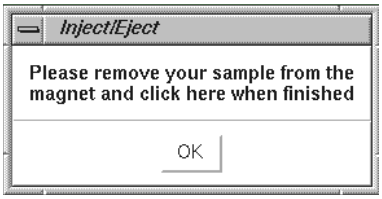
Search open the Portfolio Editor

NOTE: The sample lift air will turn on and the ”Inject/Eject” window appears.



5. Insert sample
6. Click on ‘OK’

NOTE: At this point the Automation starts the tasks of locking, shimming, adjusting of receiver gain, acquisition and processing. A history box on the bottom of the ”Automation Manager” window shows the detailed information of what tasks have been completed or failed for experiment(s) that have been setup to run on the sample.
After all experiments are finished, the sample lift turns on and the ”Inject/Eject” window appears.

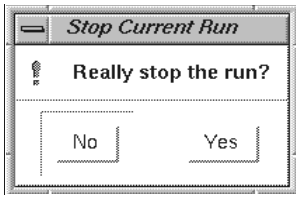


7. Remove the sample

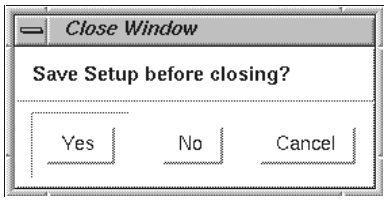
NOTE: It is generally better to keep a sample in the magnet when the instrument is

not in use.

8. Click on ‘OK’
9. Click on ‘STOP’



10. Click on ‘Yes’
11. Click on ‘File’ and select ‘Close’



12. Click on ‘No’

NOTE: The Automation window closes and the Identify User icon appears. At this point the user may exit or a new user may log in to run the automation.

7.7.2.2 Sixpack use

NOTE: As an example set up to run under Sixpack use mode, use the sample setup 7.7.1.5 or 7.7.1.6.

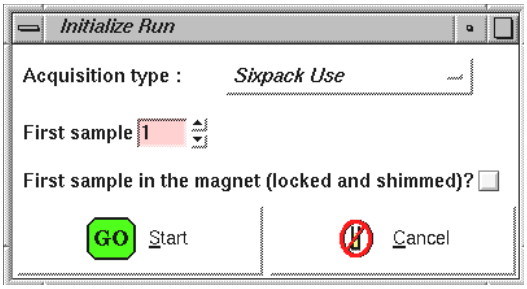
1. Turn the the switch on the sixpack controller box to OFF position
2. Turn on the sample lift (shim key pad) and remove the sample

!!!WARNING!!! Be absolutely sure that no sample is in the magnet and the sample holder is empty before proceeding to the next step.

3. With the sample lift air still on, move the sample holder to the start position (set the small

   hole over the shim stack)
4. Turn off the sample lift (from the key pad)
5. Load the samples into the sample holder
6. Turn the switch on the sixpack controller box to ON position
7. Click on ‘GO’

NOTE: The ”Initialization Run” window appears. The default ”Acquisition type” displayed is what has been set in the ICON-NMR configuration.



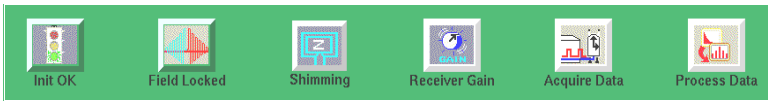
2. Click on Acquisition type button and select ‘Sixpack use’

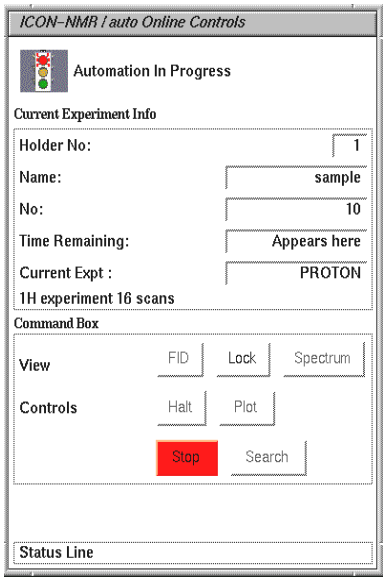
3. Select ”1” for First sample

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.

4. Click on ‘Start’

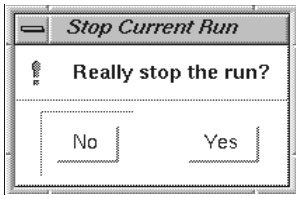
NOTE: At this point the ”ICON-NMR / Auto Online Controls” window and the ”acquisition status icons” appear.





NOTE: The Automation starts the tasks of changing the sample, locking, shimming, adjusting of receiver gain, acquisition and processing. A history box on the bottom of the "Automation Manager" window shows the detailed information of what tasks have been completed or failed for experiment(s) that have been setup to run on the samples. For a definition of the Online Control function keys, see 'Online Control function keys:' on page 152.

5. Wait until the last sample has finished running and has been ejected
6. Click on 'STOP'

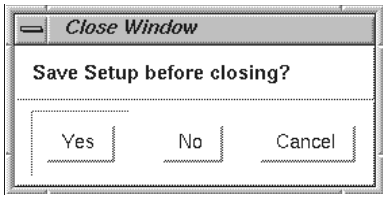


7. Click on ‘Yes’

8. Turn the switch on the sixpack controller box to OFF

9. Remove all samples from the sample holder

10. Click on ‘File’ and select ‘Close’



11. Click on ‘No’

NOTE: The Automation window closes and the Identify User icon appears. At this point the user may exit or a new user may log in to run the automation.

7.7.2.3 Standard BACS

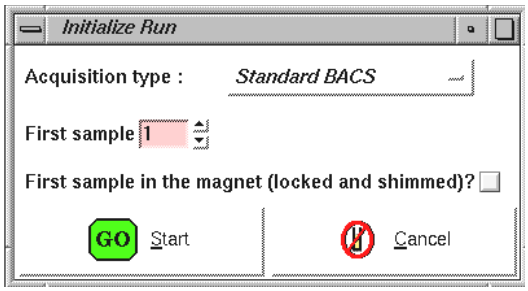
NOTE: As an example set up to run under Standard BACS mode, use the sample setup 7.7.1.5 or 7.7.1.6.

1. Insert the samples into the holders 1 and 2

3. Press RESET on the front of the sample changer

4. Click on ‘GO’

NOTE: The ”Initialization Run” window appears. The default ”Acquisition type” displayed is what has been set in the ICON-NMR configuration.



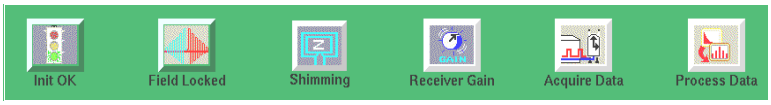
5. Click on Acquisition type button and select ‘Standard BACS’

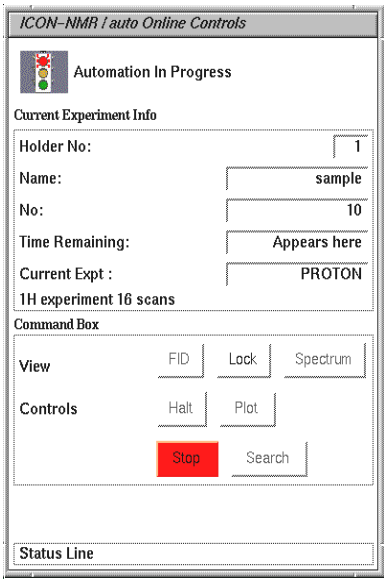
6. Select ”1” for First sample

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.

7. Click on ‘Start’

NOTE: At this point the ”ICON-NMR / Auto Online Controls” window and the ”acquisition status icons” appear.

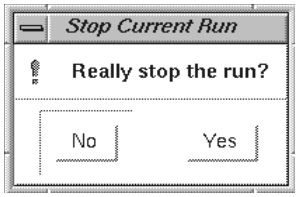




NOTE: The Automation starts the tasks of changing the sample, locking, shimming, adjusting of receiver gain, acquisition and processing. A history box on the bottom of the ”Automation Manager” window shows the detailed information of what tasks have been completed or failed for experiment(s) setup to run on the samples.
For the definition of Online Control function keys see ’Online Control function keys:’ on page 152.

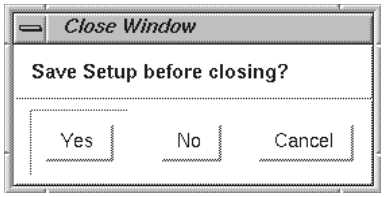
8. Wait until the last sample has finished running and has been ejected and put back into the holder

9. Click on ‘STOP’



7. Click on ‘Yes’

8. Click on ‘File’ and select ‘Close’



9. Click on ‘No’

NOTE: The Automation window closes and the Identify User icon appears. At this point the user may exit or a new user may log in to run the Automation or exit ICON-NMR.

7.8 Adding new experiments

7.8.1 Adding a new single experiment

!!!IMPORTANT!!!: Before creating a new experiment for automation make a layout of how the new experiment should work and how you would like to present the result. Check the parameter library first to see if the new experiment already exists. If not, check in the pulse program library for an existing pulse program and in the au program library for the acquisition and processing programs to satisfy the plans for the new experiment. If you have to create a new pulse program or have to write an au program, be sure to follow to the nomenclature of file names, pulse widths and delay numberings that Bruker has defined. Any deviation may result in a malfunction of the new experiment. Always test out the new experiment before you add it to ICON-NMR.

7.8.1.1 Layout of the new experiment

Experiment:	attached proton experiment (APT)	
Parameter file:	C13DEPT135	
Pulse program:	jmod	
Au programs:	aunm	au_zg
	aunmp	proc_1d

7.8.1.2 Creating the parameter file

NOTE: In this case the APT experiment does not exist in the parameter set library and it has to be created. Since the acquisition and processing of APT is similar to the DEPT135 experiment, the parameter file C13DEPT135 can be used as a template for

creating the new experiment.

1. Type edc and change the following parameters:

   NAME = newexp

   EXPNO = 1

   PROCNO = 1

2. Click on ‘SAVE’

3. Type rpar C13DEPT135 all

4. Type pulprog jmod

NOTE: The only change necessary in the parameters, is the pulse program. If desired, number of scans ‘ns’ or relaxation time ‘d1’ may be changed.

5. Type wpar C13APT all

NOTE: The parameter files for C13APT are now stored in the directory /u/exp/stan/nmr/par and before being added to the experiment list in ICON-NMR it should be tested on a sample.

7.8.1.3 Testing the experiment

Sample: ~0.3 M in 0.6 ml CDCl3 of a small organic molecule such as Cholesteryl-acetate

1. Type edc and change the following parameters:

NAME	=	newexp
EXPNO	=	2
PROCNO	=	1

2. Click on ‘SAVE’

3. Type rpar C13APT all

4. Type lock and select CDCl3 by clicking on it

5. Wait until the locking process is finished, then shim for best homogeneity

6. Type eda and change the following parameters:

PROSOL	TRUE

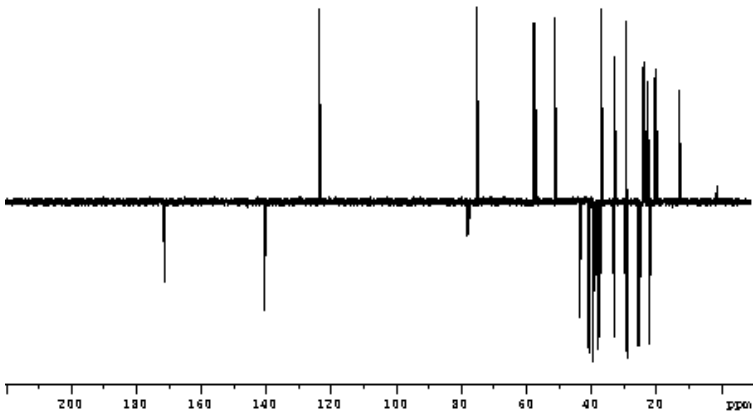
7. Click on ‘SAVE’

8. Type rga

9. Type zg

10. Type ef

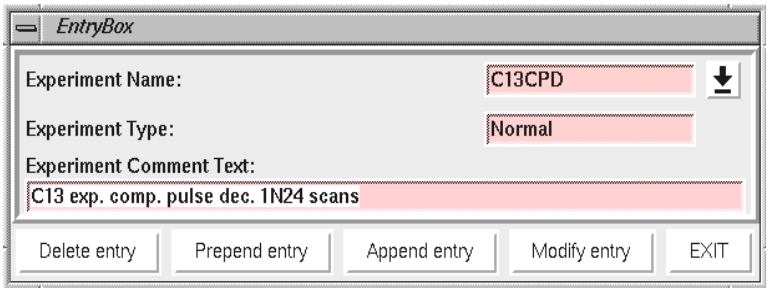
11. Correct the phase by making the CH3 and CH carbons of the same phase and the CH2 and C carbons of opposite phase. The relative phase is arbitrary.



NOTE: Repeat step 8 through 11 if you want to make changes in the parameters. Apply the changes in the parameter file C13APT.

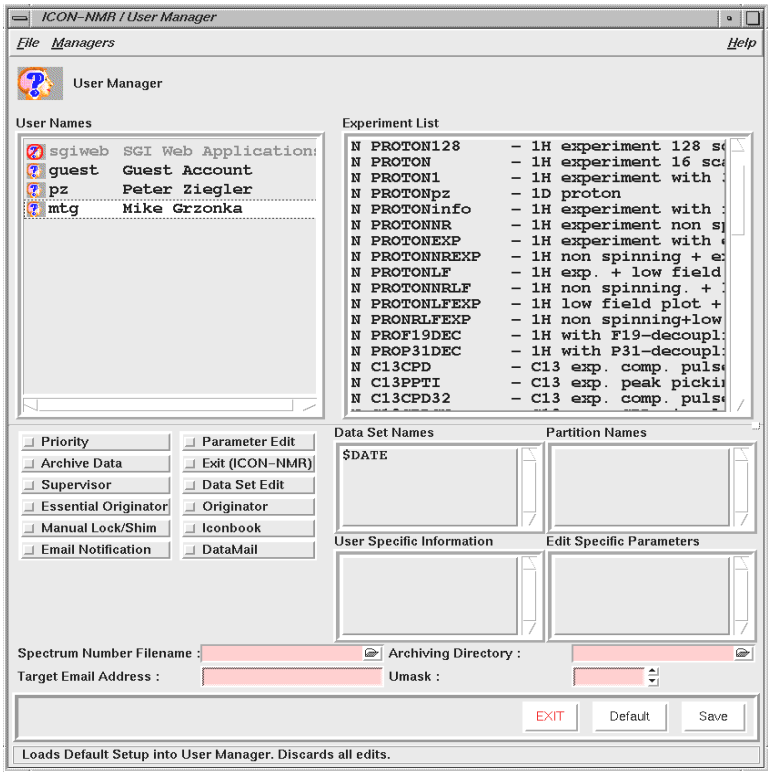
7.8.1.4 Adding the new experiment to ICON-NMR

1. Type iconnmr
2. Click on ‘System Manager’
3. Click on ‘User Manager’
4. Enter the NMR Super user password
5. Select a ”user name” by clicking on it
6. In the Experiment List open an entry box by clicking on ”C13CPD”



7. Click on the ‘box (down arrow)’ next to the Experiment Name

!!!WARNING!!! The list of names shown in the pop up window are parameter files

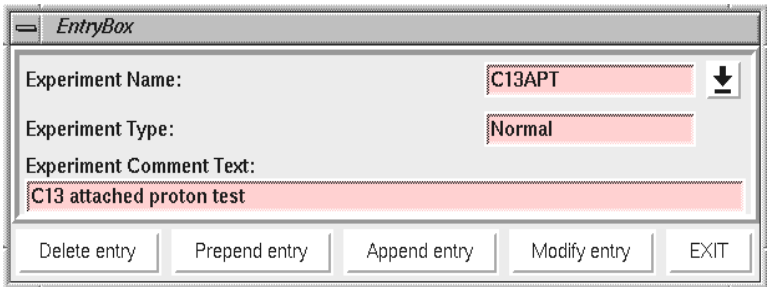


which are stored in the /u/stan/exp/nmr/par directory. Not all of the files are suitable to run in Automation. Some files require additional operator input in order to be used as an experiment (e.g. SELCO1H, NOEDIFF etc.). Other files are not possible to run in ICON-NMR (e.g. plot135, standard1D etc.).

8. Select ”C13APT” by clicking on it
9. Click on Experiment Comment Text window and enter the following text:

   C13 attached Proton test

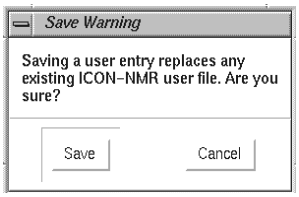
NOTE: The Experiment Type is Normal, indicating a single experiment.



10. Click on ‘Append entry’

NOTE: The new experiment has now been added to the Experiment List. To move the entry to a different place in the list, click and hold the right mouse button on the name and drag it to the desired location.

11. Click on ‘EXIT’ (Entry Box)
12. Click on ‘Save’



13. Click on ‘Save’
14. Click on ‘EXIT’
15. Select ”File” from the menu bar by clicking on it
16. Select ”EXIT” by clicking on it

NOTE: Test the new added experiment with Routine Spectroscopy or Automation for

proper operation.

7.8.2 ADDING A NEW COMPOSITE EXPERIMENT

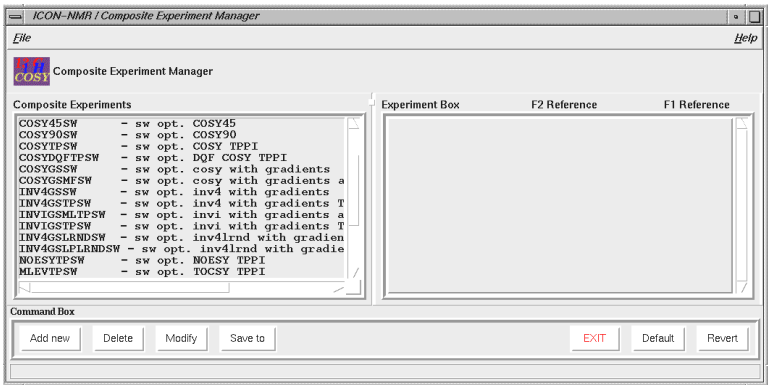
!!!IMPORTANT!!!: Before creating a new Composite experiment, make a layout of how the new experiment should work and how you would like to present the result. Check the parameter library first to see if the experiments of interest for the composite already exist. If they don't, create the experiment by following the steps in section "Adding a new single experiment" on page 162. Always test out the single experiment outside ICON-NMR according "Testing the experiment" on page 163.

7.8.2.1 Layout of the new composite experiment

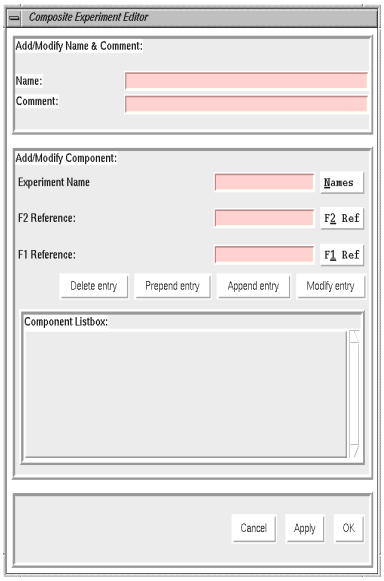
Experiments:	1D-Proton followed by a 2D-COSY45 and a 1D-APT
Parameter files:	PROTON, COSY45SW, C13APT
Name:	PROCOSAPT

7.8.2.2 Creating the new composite experiment

1. Type iconnmr
2. Click on 'System Manager'
3. Click on 'Composite Experiment Setup'
4. Enter NMR Super user password



5. Click on ‘Add new’



6. Type the following text into the ”Add/Modify Name & Comment” window:

Name:	PROCOSAPT
Comment:	Proton, COSY45, APT

7. Click on ‘Names’ in the ”Add/Modify Component” Window

8. Select ”PROTON” from the pop up window by clicking on it. (Click twice on ”next page” until PROTON appears).

9. Click on ‘Append entry’

10. Click on ‘Names’

11. Select ”COSY45SW” from the pop up window by clicking on it.

12. Click on ‘F2 Ref’

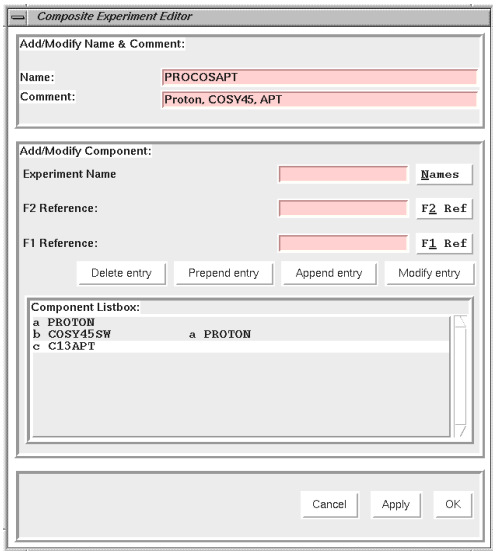
13. Select ” PROTON” by clicking on it

14. Click on ‘Append entry’

15. Click on ‘Names’

16. Select ”C13APT” from the pop up window

17. Click on ‘Append entry’



18.Click on ‘OK’

NOTE: The new composite experiment has now been added to the Composite Experiment List in the Composite Experiment Manager. To add this experiment to the user experiment list, stay in this manager window and follow the next steps.

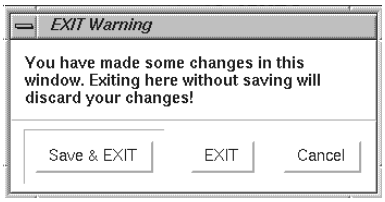
7.8.2.3 Adding the new composite experiment to the user experiment list

1. Click on the new experiment ”PROCOSAPT”
2. Click on ‘Save to’

3. Click on the user that will be allowed to access the new composite experiment

NOTE: To select multiple users, hold down the Shift key when selecting them.

4. Click on ‘Save to selected users’
5. Click on ‘Cancel’
6. Click on ‘EXIT’



7. Click on ‘Save & EXIT’
8. Select ”File” from the menu bar by clicking on it
9. Select ”EXIT” by clicking on it

NOTE: Test the new composite experiment with Routine Spectroscopy or Automation for proper operation.

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ICON-NMR Comment Form

(Please mail to software support group. The address is printed on the second manual page.)

Name / Institution / Company:

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Address:

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Phone /Fax /E-mail:

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Spectrometer / Computer:

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Operating System / ICON-NMR Version:

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Comments / Error report / Suggestions (continue on extra sheet if required)**:**

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