Automation

User Guide

Pub. No. 91000168 Rev. A 01/22/10

VARIAN

Automation

User Guide

Pub. No. 91000168 Rev. A

2700 Mitchell Drive
Walnut Creek, CA 94598 USA

http://www.varianinc.com

Contents

Chapter 1 Introduction

The Varian MR is the system of choice for laboratories requiring a compact and easy-to-use instrument that delivers fast and reliable results day in and day out. The Varian MR system provides unmatched productivity for diverse chemical applications by combining easy-to-use software with outstanding performance. Push-button experiments, along with straightforward processing and data export capabilities, make the MR the best choice for compound detection, quantification, and structure confirmation.

Advanced automation accessories enable users to take full advantage of the MR capabilities. These technologies seamlessly integrate with sample-centric data management in the VnmrJ software.

1.1 About This Guide

This VnmrJ Automation User Guide describes how to gather MR data using VnmrJ 3 and 400 MHz system automation features.

Chapters in this user guide include:

- Chapter 1, Introduction
- Chapter 2, VnmrJ Liquids Interface Description
- Chapter 3, Prepare and Load Samples
- Chapter 4, Studies
- Chapter 5, Reprocessing Data
- Chapter 6, Interpreting Data
- Chapter 7, Printing, Plotting, and Data Output
- Chapter 8, VnmrJ 3 Preferences

1.2 Warnings and Caution Symbols

This user guide helps establish operating conditions that permits safe and efficient use of the equipment. Special considerations and precautions are shown in the form of **NOTES**, **CAUTIONS**, and **WARNINGS** as described below. Operate the equipment in accordance with this user guide and any additional information provided by Varian. Contact Varian, Inc. if there are any questions regarding the safe and proper use of your equipment.

The following warning and caution notices illustrate the styles used in this user guide for safety precaution notices and explain when each type is used.

NOTE: Notes give additional and important information.

Warning Symbol	**Warning Description**
⚠ CAUTION	Alerts you to situations when failure to observe instructions could result in serious damage to equipment or loss of data.

⚠ **WARNING**	Alerts you to potentially hazardous situations that could result in serious injury or death to humans or animals, or significant property damage.
⚠ **WARNING:** **SHOCK HAZARD**	Hazardous voltages are present inside instrument. Disconnect from main power before removing screw-attached panels. No user serviceable parts inside system.
⚠ **WARNING:** **BURN HAZARD**	Very hot or cryogenically cold surfaces may be exposed. Use proper skin protection.
⚠ **WARNING:** **EYE HAZARD**	Eye damage could occur either from flying particles, chemicals, or UV radiation. Use proper eye and face protection.
⚠ **WARNING:** **FIRE HAZARD**	The potential for fire may be present. Follow user guide for safe operation.
⚠ **WARNING:** **EXPLOSION HAZARD**	The potential for explosion may exist because of type of gas or liquid used.
⚠ **WARNING:** **MOVING PARTS**	Keep hands and fingers away.

1.3 General Safety Precautions

Follow these safety procedures to ensure safe equipment operation:

- Perform periodic leak checks on all supply lines and pneumatic plumbing.
- Do not allow gas lines to become kinked or punctured.
- Put lines away from foot traffic and extreme heat or cold.

1.4 Warning Notices

Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these warnings or with specific warnings elsewhere in Varian documentation violates safety standards of design, manufacture, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

⚠ **WARNING**	Persons with implanted or attached medical devices such as pacemakers and prosthetic parts must remain outside the 5-gauss perimeter from the centerline of the magnet.

The superconducting magnet system generates strong magnetic fields that can affect operation of some cardiac pacemakers or harm implanted or attached devices such as prosthetic parts and metal blood vessel clips and clamps

Pacemaker wearers should consult the documentation provided by the pacemaker manufacturer or contact the pacemaker manufacturer to determine the effect on a specific pacemaker. Pacemaker wearers should also always notify their physician and discuss the health risks of being in proximity to magnetic fields. Wearers of metal prosthetics and implants should contact their physician to determine if a danger exists.

Refer to the documentation supplied with the magnet for the size of a typical 5-gauss stray field. Check this gauss level after the magnet is installed.

⚠ **WARNING**	Keep metal objects outside the 10-gauss perimeter from the centerline of the magnet.

The strong magnetic field surrounding the magnet attracts objects containing steel, iron, or other ferromagnetic materials, which includes most ordinary tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Unless restrained, such objects can suddenly fly towards the magnet, causing possible personal injury and extensive damage to the probe, dewar, and superconducting solenoid. The greater the mass of the object, the more the magnet attracts the object.

Use only non ferromagnetic materials, for example, plastics, aluminum, wood, nonmagnetic stainless steel, and so on, in the area around the magnet. If an object is stuck to the magnet surface and cannot easily be removed by hand, contact Varian service for assistance.

Refer to the documentation supplied with the magnet for the size of a typical 10-gauss stray field. Check the gauss level after the magnet is installed.

⚠ **WARNING**	Only qualified maintenance personnel removes equipment covers or makes internal adjustments.

Dangerous high voltages that can kill or injure exist inside the instrument. Before working inside a cabinet, turn off the main system power switch located on the back of the console.

⚠ **WARNING**	Do not substitute parts or modify the instrument.

Any unauthorized modification could injure personnel or damage equipment and potentially terminate the warranty agreements or service contract. Written authorization approved by a Varian, Inc. product manager is required to implement any changes to the hardware of a Varian MR spectrometer. Maintain safety features by referring system service to a Varian service office.

⚠ **WARNING**	Do not operate in the presence of flammable gases or fumes.

Operation, with flammable gases or fumes present, creates the risk of injury or death from toxic fumes, explosion, or fire.

⚠ **WARNING**	Leave area immediately in the event of a magnet quench.

If the dewar should quench (sudden appearance of gasses from the top of the dewar), leave the area immediately. Sudden release of helium or nitrogen gasses can rapidly displace oxygen in an enclosed space creating a possibility of asphyxiation. Do not return until the oxygen level returns to normal.

⚠**WARNING**	Avoid helium or nitrogen contact with any part of the body.

Cold gasses or liquids (helium and nitrogen) contacting the body can cause an injury similar to a burn. Never put your head over the helium and nitrogen exit tubes on top of the magnet. If helium or nitrogen contacts the body, get immediate medical attention, especially if the skin is blistered or the eyes are affected.

⚠**WARNING**	Do not look down the upper barrel.

Unless the probe is removed from the magnet, never look down the upper barrel. You could be injured by the sample tube as it ejects pneumatically from the probe.

⚠**WARNING**	Do not exceed the boiling or freezing point of a sample during variable temperature experiments.

A sample tube subjected to a change in temperature can build up excessive pressure, which can break the sample tube glass and cause injury by flying glass and toxic materials. To avoid this hazard, establish the freezing and boiling point of a sample before doing a variable temperature experiment.

⚠**WARNING**	Support the magnet and prevent it from tipping over.

The magnet dewar has a high center of gravity and could tip over in an earthquake or after being struck by a large object, injuring personnel and causing sudden, dangerous release of nitrogen and helium gasses from the dewar; the magnet must be supported by at least one of two methods: with ropes suspended from the ceiling or with the anti-vibration legs bolted to the floor. Refer to the *Pre-installation Instructions* for details.

⚠**WARNING**	Do not remove the relief valves on the vent tubes.

The relief valves prevent air from entering the nitrogen and helium vent tubes. Air that enters the magnet contains moisture that can freeze, causing blockage of the vent tubes and possibly extensive damage to the magnet. It could also cause a sudden dangerous release of nitrogen and helium gasses from the dewar. Always be sure to secure the relief valves on the vent tubes except when transferring nitrogen or helium.

1.5 Caution Notices

Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these cautions or with specific cautions elsewhere in Varian documentation violates safety standards of design, manufacture, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

⚠**CAUTION**	Keep magnetic media, ATM and credit cards and watches, outside the 5-gauss perimeter from the centerline of the magnet.

The strong magnetic field surrounding a superconducting magnet can erase magnetic media such as floppy disks and tapes. The field can also damage the strip of magnetic media found on credit cards, automatic teller machine (ATM) cards, and similar plastic cards. Many wrist and pocket watches are also susceptible to damage from intense magnetism.

Refer to the documentation supplied with the magnet for the size of a typical 5-gauss stray field. Check if this gauss level is checked after the magnet is installed.

⚠ **CAUTION**	Keep the computers (PC) beyond the 5-gauss perimeter of the magnet.

Avoid equipment damage or data loss by keeping PCs away from the magnet. Generally, keep the PC beyond the 5-gauss perimeter of the magnet. Refer to the *Pre-installation Instructions* for magnet field plots.

⚠ **CAUTION**	Check helium and nitrogen gas flow meters daily.

Record the readings to establish the operating level. The readings will vary somewhat because of changes in barometric pressure from weather fronts. If the readings for either gas should change abruptly, contact qualified maintenance personnel. Failure to correct the cause of abnormal readings could result in extensive equipment damage.

⚠ **CAUTION**	Take electrostatic discharge (ESD) precautions to avoid damage to sensitive electronic components.

Wear grounded antistatic wristband or equivalent before touching any parts inside the doors and covers of the spectrometer system. Also, take ESD precautions when working near the exposed cable connectors on the back of the console.

1.6 Electrical Hazards

- Disconnect the instrument from all power sources before removing protective panels to avoid exposure to potentially dangerous voltages.
- When it is necessary to use a non-original power cord plug, make sure the replacement cord adheres to the color-coding and polarity described in the user guide and all local building safety codes or local electrical legislation.
- Replace blown fuses with fuses of the size and rating stipulated on the fuse panel or in this user guide.
- Replace faulty or frayed power cords immediately with the same type and rating.
- Make sure that voltage sources and line voltage match the value for which the instrument is wired.

1.7 Compressed Gas Cylinders

- Store and handle compressed gases carefully and in strict adherence to safety codes.
- Secure cylinders to an immovable structure or wall.
- Store and move cylinders in an upright, vertical position. Before transport, remove regulators and install cylinder cap.

- Store cylinders in a well-ventilated area away from heat, direct sunshine, freezing temperatures, and ignition sources.
- Mark cylinders clearly so there is no doubt as to their contents.
- Use approved regulators and connections only.

1.8 Safety Hazards of Strong Magnetic Fields

You must understand and plan for the potential safety hazards of strong magnetic fields for devices, such as certain pacemakers. Check for these hazards after a particular magnet has been installed.

⚠ **WARNING**	Cardiac pacemaker wearers must remain outside the 5-gauss perimeter from the magnet until safety is clearly established. An MR superconducting magnet generates strong magnetic and electromagnetic fields that can inhibit operation of some cardiac pacemakers, which could result in death or serious injury. Consult the pacemaker user documentation, contact the manufacturer, or confer with a physician to determine the effect on a specific pacemaker. Varian provides signs with each system to warn pacemaker wearers of this hazard.

1.9 Pacemakers

Cardiac pacemaker wearers must not enter a zone that would subject a cardiac pacemaker to a magnetic intensity that could cause adverse effects. In some instances, this zone might include space on the floors directly above and below the magnet. For assistance in determining the effect of a system on a pacemaker:

- Consult the pacemaker user documentation
- Contact the pacemaker manufacturer
- Confer with a physician to determine the effect on a specific pacemaker

Actual levels vary. Check for safety hazards after a particular magnet has been installed.

1.10 Magnetic Field Exposure

MR workers are often exposed to high levels of static magnetic fields. No conclusive scientific evidence exists indicating adverse health effects at current exposure levels.

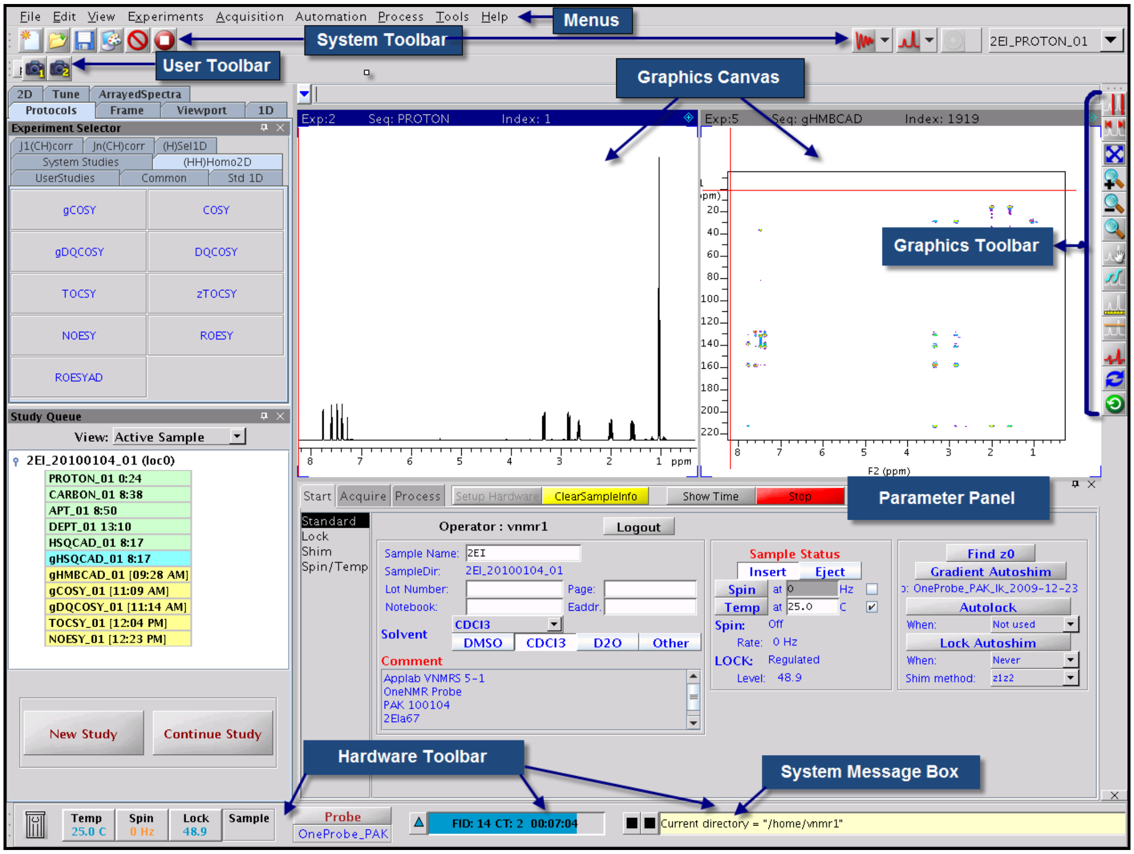
The 400-MR shielded magnet contains the high-level fields within the cryostat body.

An article by the American Conference of Governmental Industrial Hygienists (ACGIH) entitled "*Threshold Limit Values and Biological Exposure Indices, 5th ed*." states the following:

"Routine occupational exposures should not exceed 60 millitesla (mT)-equivalent to 600 gauss-whole body or 600 mT (6000 gauss) to the extremities on a daily 8 hour, time- weighted average basis. A flux density of 2 tesla (20,000 gauss) is recommended as a ceiling value."

Chapter 2 VnmrJ Liquids Interface Description

VnmrJ 3 Liquids NMR has an integrated set of tools designed to acquire a series of one and two-dimensional data sets from a library of pulse sequences for any given sample. Access the sophisticated experiments for routine use in a fully automated environment. Automation can be used with or without a sample changer. Regardless of when or how individual datasets are acquired, the interface tools can view and interact with the data on a sample-to-sample basis. Use the View menu to access or hide these tools.



Operator : vnmr1

Sample Name: 2EI
SampleDir: 2EI_20100104_01
Lot Number:
Page:
Notebook:
Eaddr:
Solvent CDCl3

Comment
Applab VNMRS 5-1
OneNMR Probe
PAK 100104
2EIa67

Spin at 0 Hz ☐
Temp at 25.0 C ☑
Spin: Off
Rate: 0 Hz
LOCK: Regulated
Level: 48.9

): OneProbe_PAK_lk_2009-12-23
When: Not used
When: Never
Shim method: z1z2

Temp 25.0 C
Spin 0 Hz
Lock 48.9
Probe OneProbe_PAK
FID: 14 CT: 2 00:07:04
Current directory = "/home/vnmr1"

Figure 1 VnmrJ Liquids Interface Window

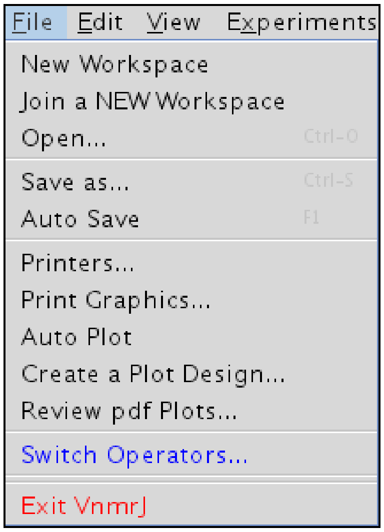
Sections in this chapter include:

- 2.1 Menus
- 2.2 System Toolbar
- 2.3 User Toolbar
- 2.4 Protocols
- 2.5 Graphics Canvas
- 2.6 Graphics Toolbar
- 2.7 Parameter Panel
- 2.8 Hardware Toolbar

2.1 Menus

Standard menu items display at the top of the VnmrJ window. Each of the menu items are described in order of appearance in the interface, left to right.

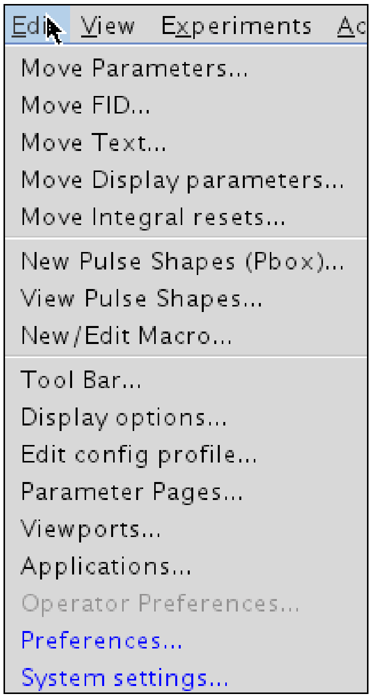
2.1.1 File Menu



Item	Description
New Workspace	Creates a new workspace for use. Workspaces are called exp1, exp2, and so on, up to exp9999. A workspace is simply a directory where data is acquired or processed.
Join a NEW Workspace	Creates a new workspace and then actively join that in the interface. A command line equivalent could be cexp(7) jexp7 to create and join exp7.
Open	Leads to a graphical tool for opening files
Save as	Opens a graphical tool wherein you can explicitly specify the location and name for saving the data in the current workspace.

Auto Save	Saves the data that has been acquired in an experiment workspace using the template set up in User Preferences. See Templates Tab. The location and file name are automatically set based upon the values defined in the Preferences Templates tool.
Printers.	Allows the user to select a valid printer and/or plotter for output
Print Graphics	Prepares a graphics file in any desired format for saving to disk
Auto Plot	Calls the appropriate automatic plotting routine for any type of data in the current workspace
Review pdf Plots	Allows you to review the pdf plot in Adobe Acrobat. You can set the user preference to create a pdf plot automatically for data that has been acquired of a given sample.
Create a Plot Design	Opens the JDesign plot tool for creating plot designs or outputting with same.
Switch Operators	Allows the current operator of the system to logout during automation thereby freeing the system for use by another operator.
Exit VnmrJ	Executes an orderly exit of the VnmrJ program. It is equivalent to typing `exit` in the command line.

2.1.2 Edit Menu



The available commands and options depend on the rights assigned by the VnmrJ Administrator.

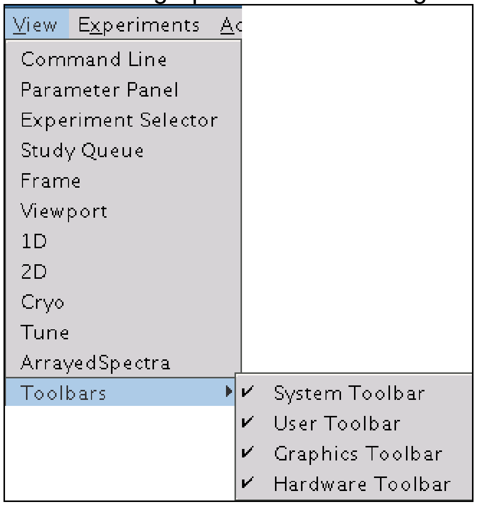
Each of the following menu options opens a dialogue that prompts the user to enter the source and destination workspaces of the items. The command and parameter reference refers to these tools as `mp`, `mf`, `mt`, `md`, and `mz`.

Item	**Description**
Move Parameters…	Opens the Move Parameters window that allows parameters to be moved from one experiment number / workspace to another.
Move FID…	Opens the Move FID window to move an FID from one experiment number / workspace to another.
Move Text…	Opens the Move Text window to move text from one experiment number / workspace to another.
Move Display parameters…	Opens the Move Display Parameters window to move display parameters from one experiment number / workspace to another.
Move Integral resets…	Opens the Move Integral Resets window to move integral resets from one experiment number / workspace to another.
New Pulse Shapes (Pbox)…	Opens the powerful Pbox tool for the creation of pulses and decoupling shapes.
View Pulse Shapes…	Opens the Pulse tool, a Bloch simulator for viewing the effects of any shaped pulse.
New/Edit Macro…	Opens a macro directly in a text editor.
Tool Bar…	Enables the addition of a button to the top bar of the user interface with a user-specific function.
Display options	Opens a graphical interface from which you can modify and save/recall the colors used in every tool used in VnmrJ.
Edit config profile…	Allows modification of what experiments are shown in the Experiment Selector tool. The starting point is based on the profile assigned to them by the VnmrJ administrator
Parameter Pages	Enables you to build/modify Parameter pages.
Viewports…	Enables you to toggle viewports. It is a tool to view multiple workspaces simultaneously, on or off
Applications…	Enables you to define an account with collections of Applications Directories. An Applications Directory is simply a path to a specific directory that could contain macros, parameter templates, and so on. For example, the AutoTest facility can be toggled on or off with this menu item.
Operator Preferences…	Enables the account administrator to allow the individual operators to manage their own preferences for the interface to automatically preset items as email address, preferred solvent, plotter, or notebook. In this example Operator Preferences is not active because in the Preferences menu User Remembrance is not enabled. The list of choices for this list is completely general and is defined by the account administrator.

Preferences…	Enables the account administrator to define items such as data saving template and default behaviors for plotting, automatic creation of pdf plot files, and a number of operator privileges. This is an important menu item and is discussed in detail in “VnmrJ 3 Preferences.”
System Settings...	Opens a graphics tool with which many system options are defined

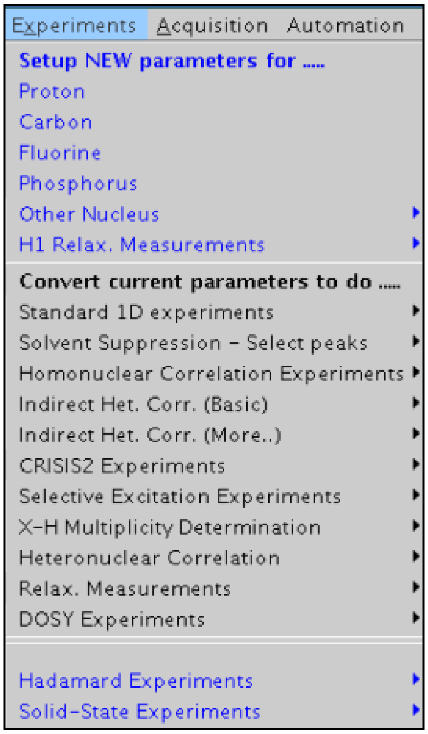
2.1.3 View Menu

Use this menu to access parts of the interface and restore the interface elements. Close the window by clicking the Close button in the upper right hand corner of each window. Access the hardware or graphics toolbars using the Toolbars submenu.



2.1.4 Experiments Menu

Unlike the Experiments Selector which can be configured by both the administrators and individual operators in terms of content, the Experiments menu shows the full selection of experiments accessible for the account. Do not perform automation submissions from this menu, use the Experiment selector tool.



Use the submenus and the menu options to perform the following tasks:

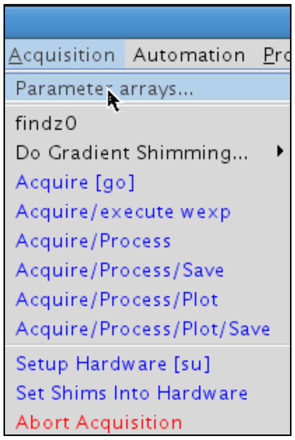
Item	**Description**
Setup NEW parameters for…	Executes a simple retrieval of standard parameters for the selected experiment and also completely clears all sample tags (parameters used to define a sample’s identity).This is a clean slate.
Convert current parameters to do…	Sets up the selected requested experiments but retains all sample tags. If you choose to use the Experiment selector without first requesting New Study by the Study Queue controls the result is the same as this conversion. The conversion of parameters with retention of sample identification parameters is the modality of “more on this sample.” The acquired data is auto saved and added to the pre-existing data acquired the current study
Setup NEW parameters to do..	Allows a simple retrieval of default parameters for all 2D and a few 1D experiments, according to your need.

Hadamard Experiments	Provides access to all of the Hadamard Fast methods 2D experiments.
DOSY Experiments	Allows access to DOSY experiments, if you want to acquire locally in a workspace, provided the license for DOSY is installed. DOSY experiments are also an optional part of the Experiment Selector.
Solid State Experiments	Allows access to all routine Solids NMR experiments

2.1.5 Acquisition Menu

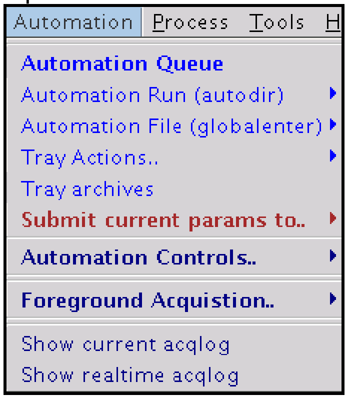
The Acquisition menu provides a convenient non-command line access to a number of core VnmrJ commands such as `go`, `ga`, or `au`.

The menu option, Parameter Arrays, facilitates the creation of parameter arrays.



2.1.6 Automation Menu

Use the Automation menu to access features other than the process of sample submission. Access is not needed for typical submission of samples in sample changer-driven NMR experiments.



Automation Queue

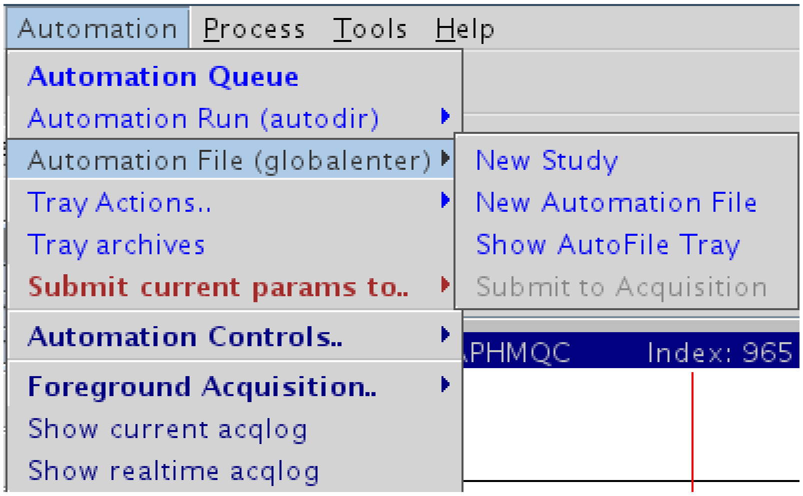
Displays the Automatin queue.

Automation Run (autodir)

Provides graphical tools to create a new automation run or to begin the submission process. Most VnmrJ 3 sample changer access is done on demand by using Study Queue controls or the tools in the Automation Run submenu. The user can also create a list of experiments for a set of samples that will not immediately run and can be saved until the account administrator activates the actual acquisition process.

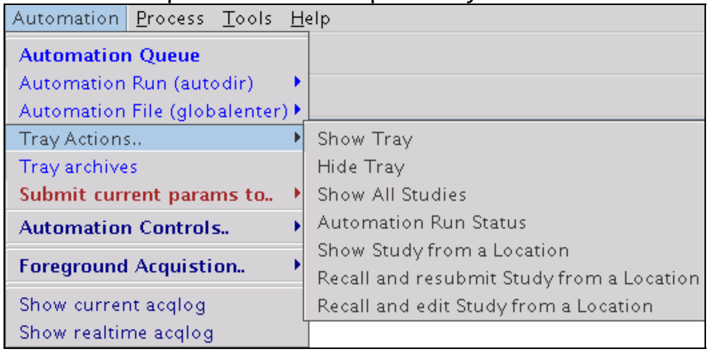
Automation File (global enter)

Use the Automation File submenu (global enter) to achieve the delayed mode of sample changer NMR.



Tray Actions

Tray Actions provides functionality that is also available on the Study Queue controls. The labels of the menu options are self-explanatory.

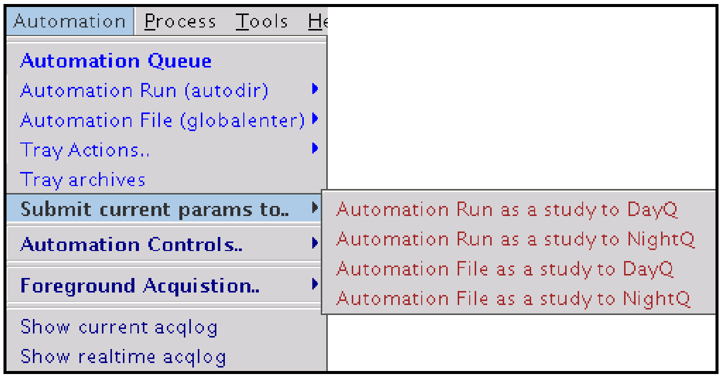


Tray Archives

Tray archives allows the user to browse completed automation runs from other dates. If the sample submission is not accessible in an old completed automation run, Tray archives will not proceed.

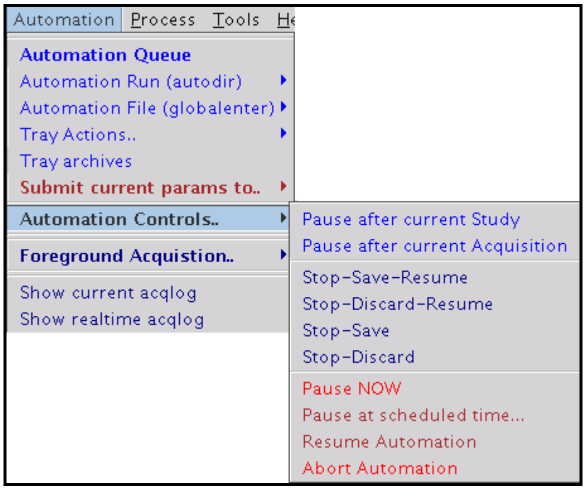
Submit current params to…

Use to manually build any desired experiment in the current workspace and to submit to an automation queue, both day and night.



Automation Controls

Automation Controls contains powerful tools to interact with the running automation queue in real-time.

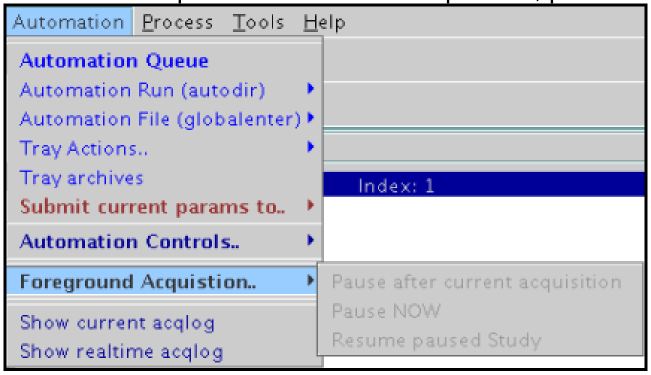


Item	**Function**
Pause after current Study Pause after current Acquisition	To pause the automation run, manually run an emergency sample, resume the automation run, or to pause a run to fill the magnet with cryogens. Double click the active node in the Study Queue window to combine submenus with the ability to process automation data as acquired in real time.
Stop-Save-Resume	Stops the running experiment, process or plot, or save to move on to the next experiment in the chain or queue. For example, if a 4 hour experiment were running in automation and after 30 minutes it was processed and nearly complete, then this action allows you to choose a rational action at that time.
Stop-Discard-Resume	Stops the running experiment and move on to the next item on the list.
Stop-Save and Stop-Discard	Functions exactly as the submenus described above except that the queue is not resumed until you select Resume Automation from this submenu.

Pause NOW	Allows you to pause the experiment immediately.
Pause at scheduled time	Allows the administrator of the account to define in advance an exact time for the automation run to be paused along with a time for automation to resume. During the time of pausing you can use the interface to submit more samples and to acquire NMR data manually. You can also allow time for cryogen fills and the magnet time to recover. The automation run can resume automatically.
Resume Automation	Resumes any paused automation run.

Foreground Acquisition

Allows user to pause after current acquisition, pause immediately, or resume paused study.



Show current acqlog

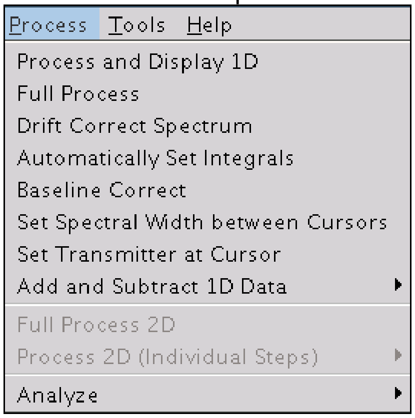
Displays the current acqlog in a popup window.

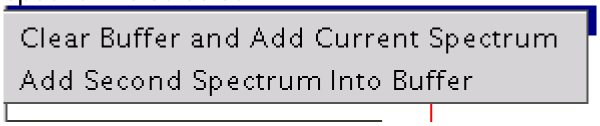
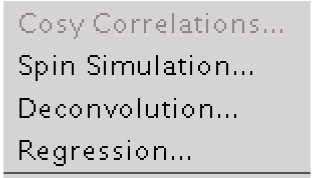
Show realtime acqlog

Displays the realtime acqlog in a popup window.

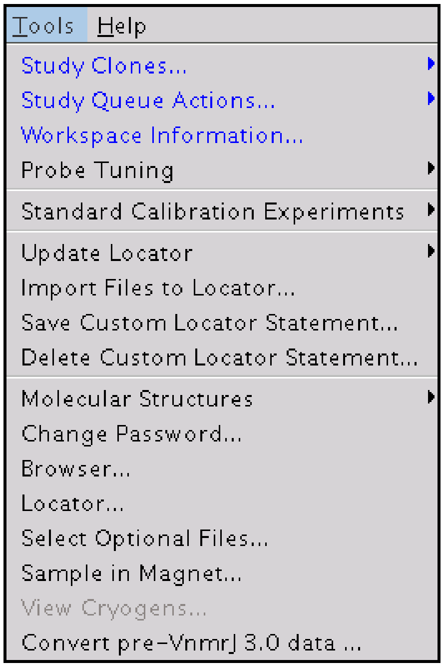
2.1.7 Process Menu

The Process menu provides tools for common tasks as an alternative to the command line.



Item	**Function**
Process and Display 1D	Process and display 1D data.
Full Process	Process and display 1D data using the processing associated with the protocol.
Drift Correct Spectrum	Apply drift correction along both axes of a 2D data set.
Automatically Set Integrals	Automatically find and set integral regions.
Baseline Correct	Apply baseline correction.
Set Spectral Width between Cursors	Mark new spectral width on the graphics screen using the left and right cursors and set the new spectral width.
Set Transmitter at Cursor	Mark new transmitter location on the graphics screen and set the transmitter.
Add and Subtract 1D Data	Results are shown displayed in current when second spectrum is selected. 
Full Process 2D	Process and display 2D data using the processing and display parameters associated with the protocol.
Process 2D (Individual Steps)	Step by step processing of 2D data.
Analyze	

2.1.8 Tools Menu



Item	**Function**
Study Clones…	See below Table 1 Study Clones…
Study Queue Actions…	See below Table 2 Study Queue Actions…
Workspace Information…	See below Table 3 Workspace Information…
Probe Tuning	Displays option to tune probe: Auto tune setup…—Opens the Setup AutoTune window. Tune Probe…—Opens the Tune Probe window. Manual Tune Probe—Loads manual tuning parameters and panels.
Standard Calibration Experiments	Provides the interfaces for probe calibration and gradient shimming setup: Probe Calibration—Opens a window for running a series of experiments to calibrate the probe. Set Up Gradient Shimming—Loads the pulse sequence and panels for making a shim map for gradient shimming. Set Up 3D Gradient Shimming—Selection appears only if this option is installed. Loads the pulse sequence and panels for making a 3D shim map for gradient shimming.
Update Locator	Opens a submenu that provides choices for updating the different parts of the Locator.
Import Files to Locator…	Opens a window for importing files to the Locator.
Delete Custom Locator Statement…	Opens a window for deleting custom locator statements.

Molecular Structures	Display all—Display all molecular structures. Plot all—Plot all molecular structures. JChempaint…—Opens the open source application JChempaint in a separate window. Refer to the online manual provided with Jmol.
Change Password…	Opens a window for changing operator passwords. If the operator has an operating system login account, the password will also be changed.
Browser…	Opens a file browser window.
Locator…	Opens a Locator and File window.
Select Optional Files…	Additional files that can be automatically saved in the .fid directory.
Sample in Magnet…	Tool to command sample changer to change sample and to assign sample position currently in magnet.
View Cryogens…	Tool to monitor cryogens. For more information see the *Cryogen Monitoring* user guide.
Convert pre-VnmrJ 3.0 data…	Tool to convert pre-VnmrJ 3 data for use in VnmrJ 3.

Study Clones Submenu

The Study Clones submenu is a group of convenience utilities.

A Study Clone is a set of experiments built in the Study Queue with any desired parameter customizations. This set of experiments is automatically represented as a new button in the Experiment Selector. After creation, the user can repeat the exact set of experiments simply by selecting that button either in or out of automation and with or without a sample changer. One use of a Study Clone is to set the desired parameters for a very fast PROTON spectrum, such as `nt=1 ss=0 gain=4` and Clone current Exp with a button label Fast_H. It might also be useful to set parameters for a slow quantitative PROTON experiment such as `d1=60 pw(90) ss=2 nt=8` and Clone current Exp with a label as Quant_H. The user can use the Study clones to recall complex sets of multidimensional experiments or to simply establish a convenience button for commonly done tasks. Clones from other clones can also be created by adding more experiments in any desired fashion.

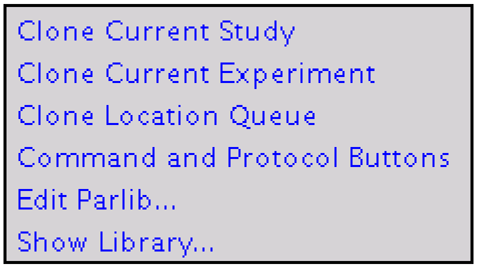
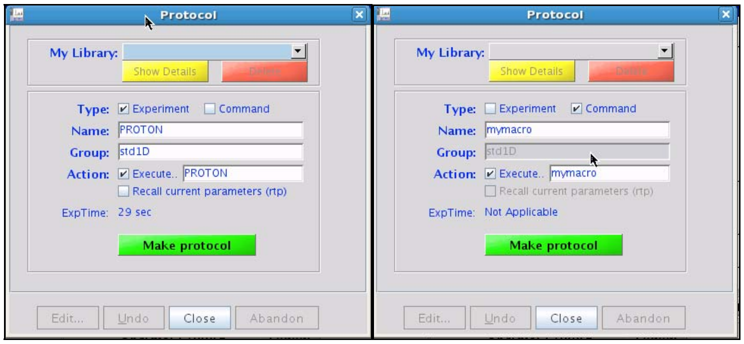


Table 1 Study Clones… Submenu

Item	**Function**
Clone Current Study	Use to assign a name for the button associated with a study of a list of experiments assembled in the Study Queue.
Clone Current Experiment	Allows user to assign a name for the button option associated with the currently loaded or displayed experiment. For example, use this option if an FID from a previous study is retrieved in the current workspace, and the user wants to create a clone of the single experiment that had been acquired on that sample.
Clone Location Queue	Allows creation of a button associated with the study completed for the selected tray sample location.
Command and Protocol Buttons	Use the Protocols menu option to devise a method to create a button to run an old-fashioned macro to setup an experiment. In the Type field, select either Experiment or Command. If Command is selected, a button option is created to call a macro that for example, analyzes the lineshape. Based on the result, the user can decide to call a group of non-spin shim routines. A Study Clone could then be composed of a PROTON experiment customized appropriately for running 1H lineshape, followed by the command protocol to analyze lineshape in the Study Clone. This Study clone can be submitted at any time and automates the task of refining shims.  Type: ☑ Experiment ☐ Command Name: PROTON Group: std1D Action: ☑ Execute.. PROTON ☐ Recall current parameters (rtp) ExpTime: 29 sec Type: ☐ Experiment ☑ Command Name: mymacro Group: std1D Action: ☑ Execute.. mymacro ☐ Recall current parameters (rtp) ExpTime: Not Applicable
Edit Parlib…	Enables users to view the details of any valid VnmrJ 3 protocol. Edit/Make parlib can also create new protocols based on the contents of the parameter set in the current workspace. Use the Study Clones tool to create a modified version of an already existing protocol, such as PROTON or CARBON experiments with specific parameters for a quick or long experiment; do not use Edit/Make Parlib. In order to properly utilize the tool, the user must have a basic understanding of the concept of modules and locked parameters (Plock). Following is an example view of Edit/Make parlib for a band-selected 2D experiment.

	
	
	(ExpType: bsHSQCAD)
	Name: bsHSQCAD
	apptype: hetero2D
	modules: esat wet gradient Dch_adiabatic dpfgse impress sel2D
	Lock parameters... dm ni nt seltype shape180
	By default, start with... HSQCAD gHSQCAD HSQC gHSQC wet1D PRESAT PROT
	(Queued acquisition only!)
	Customization:
	`if (satmode ='y') then`
	`  satmode='yy'`
	`endif`
	(macro format - overrides all defaults)
	Both the apptype and the list of modules are different from the PROTON protocol. The user can query the values and modules for the apptype parameter by using the apptype? and modules? commands before creating a new protocol from a pulse sequence and parameter set that originated from outside VnmrJ 3, such as the user library. Assign the value for modules to the Lock parameters list and add any parameter needed to be kept at setup time.
	
	In the above PROTON experiment, basic protocols include the following key concepts:
	Name—Displays the name of the set parameter and the protocol's button.
	apptype—Displays a generic helper macro name for setting up a group of protocols. In VnmrJ 3 apptype is optional. Common apptypes are std1D, homo2D, hetero2D, and lstd1Dmodules. The names for the little min-parameter sets that are combined to create the existing parameter set.
	module—Displays a module and all of the values for the parameters in that module, type on the command line module ('popup','modulename'), for example module ('popup','presat').
	Default Starting Experiment—Define a default experiment as a starting point to morph the default experiment into the desired experiment. Type REQexp? from the command line to query the value for the default starting experiment.
	Customization—Shows the contents of the macro name_setup or in the case of this example PROTON_setup. Not editable in this tool but it can be edited in a text editor.
	User Customization—Creates a macro from the text typed in this field. userprotocolname or for this example userPROTON

Show Library	Opens a graphical tool to backup then remove old buttons and to review the executable actions of buttons.

Study Queue Actions…

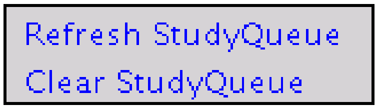
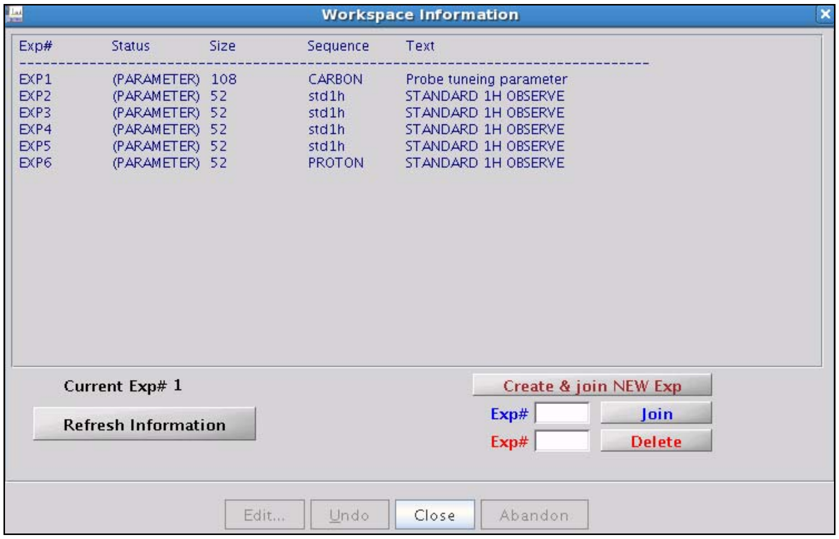


Table 2 Study Queue Actions…

Item	Function
Refresh StudyQueue	The menu options in the Study Clones submenu are Refresh Study Queue: Updates the study information in the Study Queue pane.
Clear StudyQueue	Clear Study Queue: Clears the Study Queue pane.

Workspace Information…



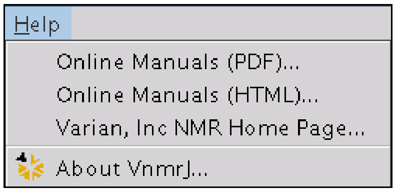
```
Exp#        Status        Size        Sequence     Text
------------------------------------------------------------------------------------
EXP1        (PARAMETER)   108         CARBON       Probe tuneing parameter
EXP2        (PARAMETER)   52          std1h        STANDARD 1H OBSERVE
EXP3        (PARAMETER)   52          std1h        STANDARD 1H OBSERVE
EXP4        (PARAMETER)   52          std1h        STANDARD 1H OBSERVE
EXP5        (PARAMETER)   52          std1h        STANDARD 1H OBSERVE
EXP6        (PARAMETER)   52          PROTON       STANDARD 1H OBSERVE
```

Current Exp# 1

Table 3 Workspace Information…

Workspace Information…	The Workspace Information menu displays the status of all workspace ongoing processes.

2.1.9 Help Menu



Item	Function
Online Manuals (PDF)…	Opens manuals in PDF format.
Online Manuals (HTML)…	Opens manuals in HTML format.
Varian, Inc NMR Home Page…	Opens the Varian NMR home page.

2.2 System Toolbar

The system toolbar provides access to common functions.

2.3 User Toolbar

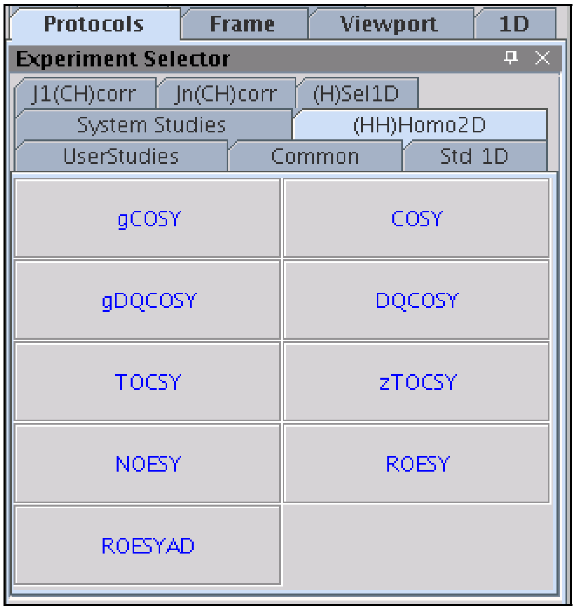
Save the current screen layout: graphics, parameter panel, locator sizes.

2.4 Protocols

Use the Protocols menu option to devise a method to create a button to run an old-fashioned macro to setup an experiment.

2.4.1 Experiment Selector

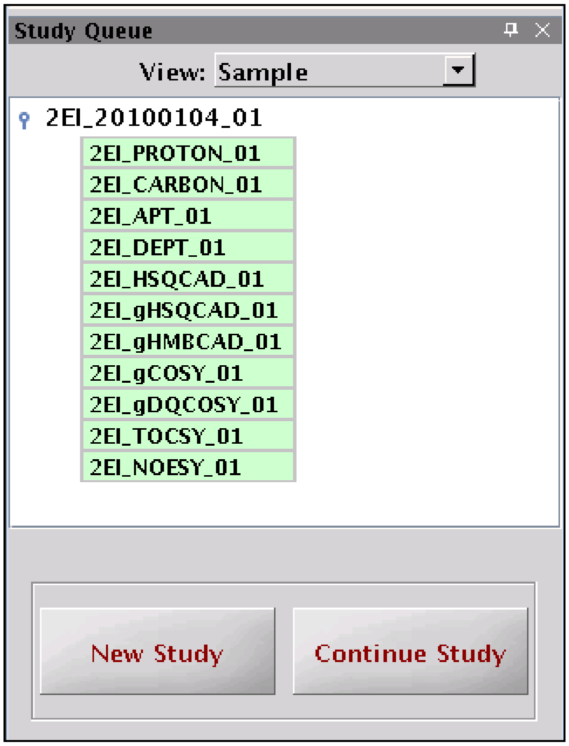
When an experiment is selected in Experiment Selector, all submissions to automation can be selected. Configure families of experiments and content by using the Experiment Selector. Simple or complex experiments, based on an account or an operator within an account, are accessible as needed.



2.4.2 Study Queue

VnmrJ 3 allows the ability to construct a linked list of experiments as a Study Queue, that can be performed on any given sample. For more information, see Chapter 4 Studies.

Use New Study or Continue Study to process sample submissions. New Sample can also be used to process sample submission.

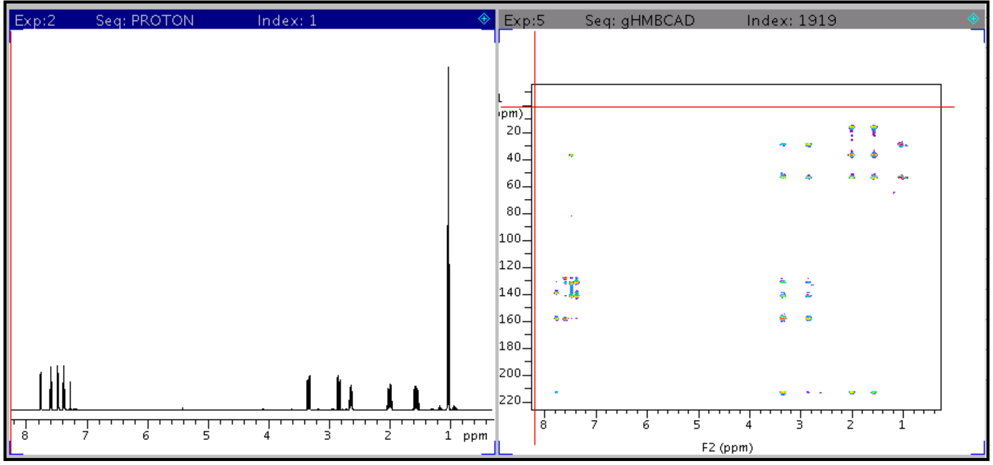


View: Sample

2EI_20100104_01
- 2EI_PROTON_01
- 2EI_CARBON_01
- 2EI_APT_01
- 2EI_DEPT_01
- 2EI_HSQCAD_01
- 2EI_gHSQCAD_01
- 2EI_gHMBCAD_01
- 2EI_gCOSY_01
- 2EI_gDQCOSY_01
- 2EI_TOCSY_01
- 2EI_NOESY_01

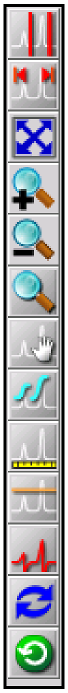
2.5 Graphics Canvas

The Graphics canvas is used to display and interact with graphic and text information. For more information, see 5.4 Interact with the spectrum using the graphical toolbar.



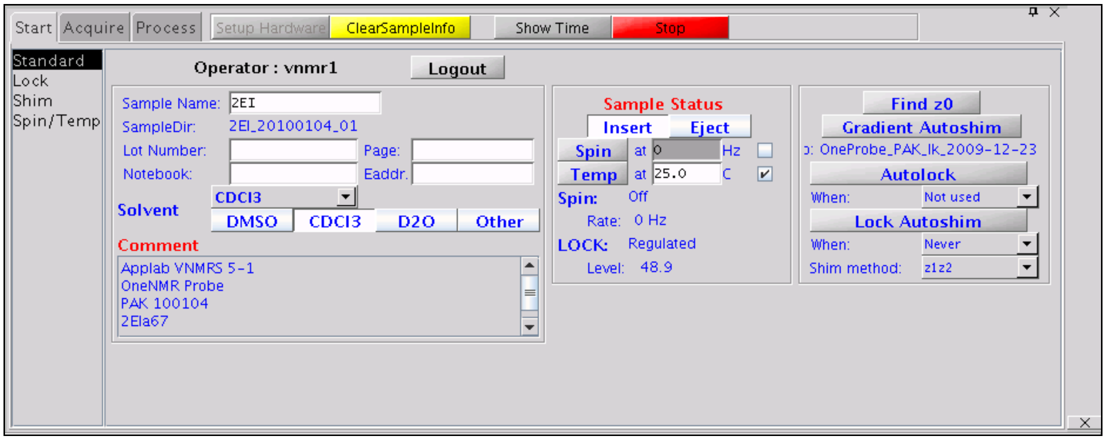
2.6 Graphics Toolbar

The Graphics toolbar is used to control the interactive display in the graphics canvas. For more information, see 5.4 Interact with the spectrum using the graphical toolbar.



2.7 Parameter Panel

The Parameter panel is below the graphics window and above the Hardware toolbar. The Parameter panel shows the pulse sequence, context-specific information, menus, and text entry. The panels under the Acquire and Process tabs change on a pulse-sequence to pulse-sequence basis. Parameter panel information is made for the experiment being set up or processed.



Operator : vnmr1

Sample Name: 2EI
SampleDir: 2EI_20100104_01
Lot Number:
Page:
Notebook:
Eaddr.
Solvent: CDCl3

Comment:
Applab VNMRS 5-1
OneNMR Probe
PAK 100104
2EIa67

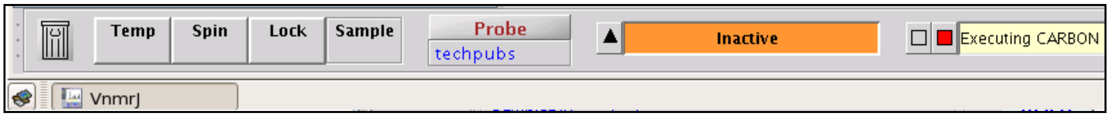
Spin at 0 Hz ☐
Temp at 25.0 C ☑
Spin: Off
Rate: 0 Hz
LOCK: Regulated
Level: 48.9

ɔ: OneProbe_PAK_lk_2009-12-23
When: Not used
When: Never
Shim method: z1z2

Each pane can be resized, reduced to a tab, or closed. Setup, acquire, or process NMR data using the point and click feature of the interface. The interface can also be accessed using the command line, above the large graphics window. There are three tabs labeled Start, Acquire, and Process below the graphics window. The top page on each tab displays commonly used functions. Lower pages displayed detailed functions. Buttons below the Graphics window and to the right of the tabs represent tab-specific actions and differ with each tab.

2.8 Hardware Toolbar

The Hardware toolbar, located at the bottom of the VnmrJ window, shows a trash can icon and a display area dedicated to real-time hardware information.



Sample temperature, spin rate, lock level, and current sample changer location are displayed to the left of the Hardware toolbar. If the user is logged in as an administrator, the probe files are displayed next to the toolbar. Real time events such as system is idle, locking, shimming, or acquiring data are displayed in the field located to right of the probe file. If the system is active, each event's remaining time is displayed. To the right of the Hardware toolbar is a system message box.

Chapter 3 Prepare and Load Samples

Sections in this chapter include:

- 3.1 Preparing the Sample
- 3.2 Ejecting and Inserting the Sample

How samples are prepared and positioned in the turbines greatly influences how reliable and fast the data accumulation is from multiple samples and how auto shimming is performed. The discontinuities in the bulk magnetic susceptibilities at the various interfaces (air-to-glass, glass-to-solvent, and solvent-to-air) can generate a lot of line-broadening variability from sample-to-sample, regardless of whether automation or manual operation is used. The time spent shimming, or the need to shim, is largely dependent on the care used to control the effects of these discontinuities.

3.1 Preparing the Sample

3.1.1 Solvent Selection

Although samples are typically run as solutions, they can also be run as neat liquids. The solvents used to make up solutions should be inert (does not react with the sample) and ideally available in deuterated form. Resolution is better with a deuterium lock, especially for lengthy accumulations. The instrument can be run unlocked without locking onto the deuterium of a deuterated solvent. Commonly used solvents are deuterated versions of chloroform, DMSO, water, acetone, and methylene chloride.

3.1.2 Solute Concentration

A typical NMR sample contains the solute in 1:100 mM concentration. Because the signal-to-noise of an NMR spectrum increases as the square root of the time of the experiment, halving the sample concentration increases the required time four-fold. The minimum sample concentration is often controlled by the minimum sample height, see Sample Height, which is more critical to optimize than the sample concentration. Sample concentrations of up to 100% can be used in NMR, but unusual spectral effects may occur. Typically, “dilute solutions" are used.

3.1.3 Solution Preparation

Filtration

Samples for solution-state NMR should be homogeneous liquids. Samples that contain heterogeneous material in the tube, solids, bubbles, or immiscible liquids exhibit broad NMR lineshapes, and this broadening cannot be shimmed out. Such samples should be filtered before use, if high-resolution spectra are desired.

Salt

Samples of increased ionic strength detune the probe and cause reduced signal-to-noise. The probe may need to be retuned for these samples, to improve both the signal-to-noise and the 90-degree pulse width, but not back to the original values.

3.1.4 Sample Height

To minimize bulk magnetic susceptibility discontinuities at each end of a sample (the "end effects"), the liquid column length of the sample must be at least three times the length of the observe coil. This suggests a column length of close to 50 mm for most modern probes. Solvent volumes of 0.6 ml in a 5-mm tube (and 3.1 ml in a 10-mm tube) are adequate for removing the end effects. (The 50-mm length is ideal for 5-mm tubes in 5-mm probes. The 10-mm probes are particularly sensitive to shorter column heights. The 3-mm probes are less sensitive about maintaining this sample height as compared to 5-mm tubes and probes.)

Signal-to-noise cannot be increased by reducing the sample volume to attain higher concentration; shorter samples produce spectra with broader lines. Typically, a 0.4 ml sample generates a spectrum with a lower signal-to-noise than the same solution diluted to 0.6 ml. Shorter samples take longer to shim and require more re-shimming between each sample. (All samples have some variability in sample volume. The amount of re-shimming needed to compensate for this variability is more pronounced in 0.4 mL samples than in 0.6 to 0.8 mL samples. This problem gets worse if the samples get much shorter than 0.5 mL.

For best results and minimum shimming time, samples should be prepared to be of similar heights. As long as the bottom of the tube is positioned properly, there is little sensitivity to sample length above 0.7 ml. The user should make every sample up to the same height and obtain shim values using samples of that height.

For Wilmad 528 or 535 tubes with no restricting plugs, samples containing the volumes listed in Table 4 should be placed at the depths shown in that table. Depth is the distance from the bottom of the standard spinner turbine to the bottom of the sample tube.

Table 4 Sample Tube Depths

Volume	Length	Depth (Range)
700 µL	50 mm	68 mm (65-69 mm)
600 µL	42 mm	65 mm (63-67 mm)
500 µL	36 mm	62 mm (60-64 mm)
400 µL	28 mm	59 mm (59-62 mm)

3.1.5 Sample Position

Use the sample depth indicator provided with the spectrometer to set the sample position to a repeatable position. Note that each spinner turbines accept only one size tube. There are spinner turbines designed for 5-mm tubes, as well as for 10-mm, 4-mm, 3-mm, and 1.7-mm tubes.

Figure 2 Sample Depth Gauge

To Position a Sample Using the Depth Gauge

1. Carefully insert the NMR sample tube into the spinner turbine by inserting the tube into the wider top side of the turbine.

NOTE: Do not place your hand under the hole in the bottom of the spinner turbine, as the sample tube will protrude from this opening when the sample is correctly positioned in the turbine.

---

2. Put the sample-turbine assembly into the top of the depth gauge, and push the assembly down gently until it rests on the bottom of the depth gauge.

   Because the position of the bottom plug within the depth gauge is adjustable by users, take note of the depth (in mm) that the plug is currently set to, and ensure that is the value recommended by the local NMR administrator.

3. If you need to use less than 0.7 ml of solvent for any reason, you can center the liquid volume in the cross-hatched area. This centers the sample in the receiver coil, indicated by the center line symbol.

3.1.6 Sample Tubes

Buy the best quality NMR sample tubes, and clean the outside of each tube with a solvent such as isopropyl alcohol. Cleaning should be followed by a careful wiping with a wiper tissue before placing the tube in the probe. This helps keep the inside of the magnet bore (the "upper barrel") clean, to make the sample spinning more reliable (when needed). The use of good quality sample tubes is especially important when the sample spinning is used. Low quality sample tubes often generate larger spinning sidebands, or scratch the inside of the probe.

3.2 Ejecting and Inserting the Sample

All modern spectrometers have an automatic insert and eject feature. The spectrometer is equipped with hardware and software to provide computer control of sample ejection, insertion, spinning, locking, and shimming. This section covers computer-controlled sample ejection and insertion.

Manual control of ejection and insertion is also provided on each of these systems to enable the user to withdraw samples if necessary. Although it is recommended that the user rely on computer control.

NOTE: Solids probes do not support sample insertion and ejection.

3.2.1 To Eject a Sample, Without a Sample Changer

Always eject first, even if no sample is in the magnet, to start the airflow for carrying the sample. After ejection air is turned on and under computer control, if the sample is present it will rise back to the top of the upper barrel. Remove the sample, and replace it with another sample.

Using the Command Line

Enter `e` (or `enter`) in the command line.

Using Automation
If you have a sample changer (7510-AS, 7600-AS, Carousel, or SMS), the sample is changed automatically under computer control (in the sample order listed in the StudyQ).

Manual Ejection

This is applicable for the VNMRS and 400MR systems only.
Flip-up the switch on the top of the upper barrel (located on the top of the magnet bore) to eject the sample.

3.2.2 To Insert a Sample, Without a Sample Changer

When inserting a sample, the sample tube gradually lowers down the upper barrel under computer control.

Using the Command Line:

1. Perform a sample ejection (even if no sample is in the magnet) to start the airflow for carrying the sample.
2. Insert the sample by placing it in the top of the upper barrel.
3. Enter **`i`**(or **`insert`**) in the command line.

Using Automation

If you have a sample changer (a 7600-AS, a Carousel, or an SMS), the sample is changed automatically under computer control (in the sample order listed in the StudyQ).

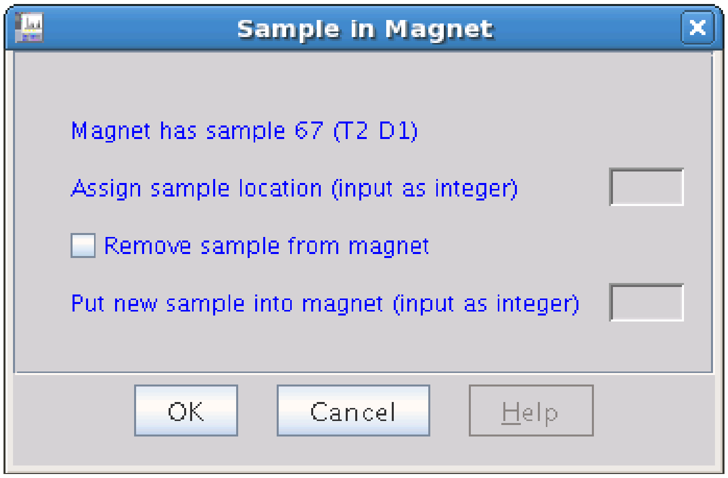
Manual Ejection

This is applicable for the VNMRS and 400MR systems only.

Flip-down the switch on the top of the upper barrel (located on the top of the magnet bore).

3.2.3 To Change a Sample, With a Sample Changer

The Sample in Magnet tool is located in the Tools menu. This tool allows samples to be removed or inserted by a robot sample changer. It can also be used to specify the tray or slot location for a sample in the magnet.



Sample in Magnet

Magnet has sample 67 (T2 D1)

Assign sample location (input as integer)

☐ Remove sample from magnet

Put new sample into magnet (input as integer)

Figure 3 Sample in Magnet Tool

To remove a sample from the magnet:

1. Select the **Remove sample from magnet** check box and click **OK**.

To insert a sample in the magnet:

1. Put the sample into the robot sample holder.
2. Type the location into the **Put new sample into magnet** box and click **OK**.
3. To designate the tray location of a sample in the magnet, type the desired location into the **Assign sample location** box and click **OK**.

Chapter 4 Studies

VnmrJ 3 allows the ability to construct a linked list of experiments as a Study Queue, that can be performed on any given sample. When data is collected using the Study Queue information regarding that sample, including raw data, processed data, plots, are linked together and automatically collected into a Sample Directory. This allows customized information from one experiment (*i.e.*, calibrated pulse widths, optimized sweep widths, solvent suppression conditions, *etc*.) to be automatically incorporated into subsequent acquisitions, and the automatic addition of high-resolution reference spectra to plots of 2D data sets. The location and structure of each Sample Directory is controlled by choices made in the User Preferences panel under the Templates tab, see Templates Tab.

Sections in this chapter include:

- 4.1 Introduction
- 4.2 Operations Using a Robot Sample Changer
- 4.3 Operations Without a Robot Sample Changer

4.1 Introduction

A Study Queue can be created, modified, extended, and saved using different tools described in this chapter. It is important to note that these tools can be applied with or without the use of a robot sample changer.

The sequence of steps for data acquisition using Study Queues is simple and straightforward. The process is initiated by clicking the New Study button on the lower portion of the Study Queue vertical panel. This places the software into “submit mode” where the tools are available to build, customize, and submit a Study Queue. (In contrast, the other operating mode is “Review mode” and is used for all other tasks, such as manual data collection, processing and plotting, and so on.)

While the Study Queue is used to collect data, it also serves other functions. The Study Queue presents a graphical representation of the status of all experiments submitted to acquisition in the current automation run. Experiment nodes that have completed are displayed as green, while the active experiment is blue and experiments that have been submitted but not yet begun are considered “pending” and are shown in yellow. Double-clicking on a completed node loads the associated data and processes that data in the current workspace.

Finally, when used on a system equipped with a robot sample changer, the Study Queue can display an overview of all the studies submitted to automation by choosing View → Spectrometer from the top of the Study Queue pane. Lists of nodes under the headings of Active Study, Completed Studies, Studies in Progress, Errored Studies, Priority Studies, Studies in Queue are displayed, as well as the estimated completion time for each active and pending experiment node.

The various options and choices available for using the Study Queue mechanism will be discussed in this chapter.

4.2 Operations Using a Robot Sample Changer

The Study Queue pane changes to reflect movement between the two modes of software operation: Review mode and Submit mode.

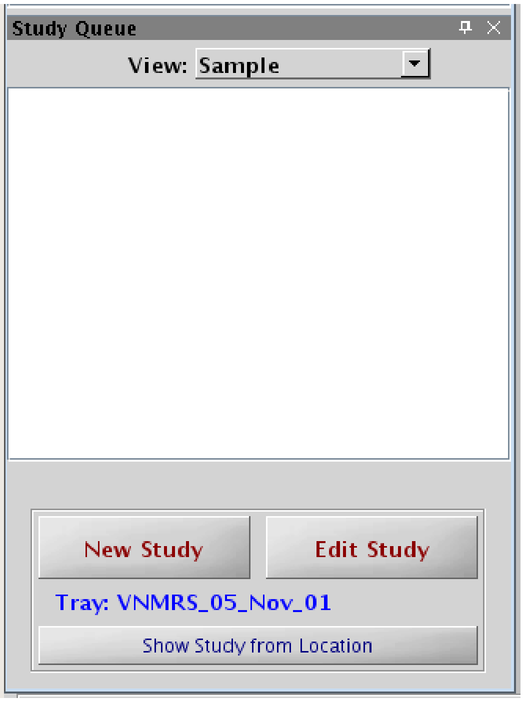


Figure 4 Study Queue—Review Mode

4.2.1 Study Queue Controls—Review Mode

In Review mode, the Study Queue pane is used to display information about the current automation run.

The View pull-down selects what information is displayed:

- Spectrometer – shows all studies in the current automation run
- Sample – shows the study linked to the data in the current workspace
- Active Sample – shows the study that is currently running

The New Study button initializes a new study and moves the software to “submit mode.” The Edit Study button allows one to modify the study currently displayed in the Study Queue. The Show Study from Location button loads a study from the tray display.

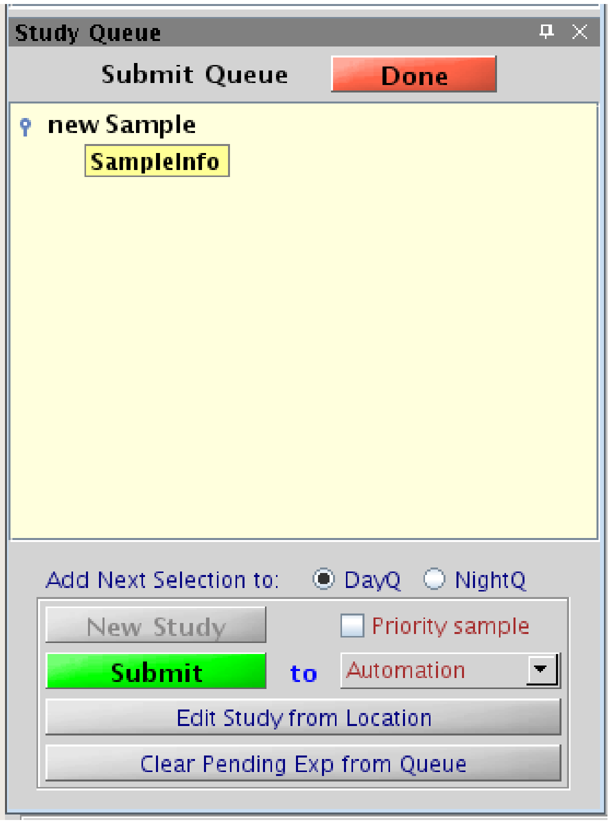


Figure 5 Study Queue—Submit Mode

4.2.2 Study Queue Controls—Submit Mode

In Submit mode, the Study Queue pane is used to build a study and submit it to acquisition.

The Done button abandons any changes made to the current study and returns the software to Review mode. The DayQ and NightQ buttons explicitly selects when an experiment will be run. The schedule for when the DayQ and NightQ begin is set by the account administrator through the Preferences panel, see Automation Tab.

The Priority sample check box allows a sample to be submitted ahead of all other samples in the current automation run. This feature is controlled by the system administrator on an operator-by-operator basis.

The New Study button creates a new study.

The Submit button submits the current study to acquisition. The associated pull-down menu determines how the acquisition is to be performed:

- Automation – submits the study to the Spectrometer Queue.
- Foreground exp – submits the study to acquisition in the current workspace.
- Background – submits the study to a background copy of VnmrJ.

The Edit Study from Location button loads a study into the Study Queue from the tray display in preparation for modification of that study.

The Clear Pending Exp from Queue button deletes all pending experiments from the current Study Queue.

4.2.3 Using the Study Queue – Full Automation

To collect a series of spectra in a Study Queue:

1. Place the sample in a spinner turbine and adjust its position using the sample depth gauge. Place the sample/spinner into the robot sample rack and note its position.
2. Click **New Study** in the Study Queue vertical panel. This initializes the Study and moves the software into “submit mode.”
3. Click on the buttons in the Experiment Selector to add the desired experiments to the Study Queue. The order of the experiments in the Study Queue can be changed using a click-and-drag action. Dragging an experiment node to the trash can deletes it from the queue.
4. Experimental parameters for an individual experiment can be customized by double-clicking on the experiment node in the study queue. (This is dependent upon the rights and privileges permitted for each user by the System Administrator.) The parameter set is loaded into the current workspace where any desired changes can be made using the horizontal panels under the “Acquisition” tab. Once the desired changes are complete, click **Save**.
5. Fill in the information associated with the sample, such as sample name, solvent, email address, comments about the sample, and so on. The sample name parameter is typically required as part of the automatic data saving routine, and the solvent information is used to determine where the lock must be set to in order to get correct referencing.
6. Select the checkboxes to control automatic printing, locking, shimming, and tuning, as appropriate.
7. Select the location in the tray display that corresponds to your sample. Once selected, the location will show a flashing highlight ring.
8. Click **Submit**. (Depending on the settings in the preferences panel, the system remains in “submit mode” with the current queue still loaded in the Study Queue window. The same queue can be submitted to a new sample location by editing the sample information, clicking the appropriate location in the tray, and clicking **Submit** again.)
9. Click **Done** to return the interface to Review mode.

4.2.4 Day Queue, Night Queue, and Priority Queue

1. The account administrator can choose to restrict the time available for any given sample. (See Automation Tab) Depending on preferences chosen by the account administrator, the Study Queue either moves any experiment that exceeds the time limit into the Night Queue or will not allow further additions to the study. The user has the ability to explicitly direct an experiment to occur at night by selecting the “NightQ” radio button in the Study Queue window before adding an experiment to the study.
2. If allowed by the account administrator, users can select the “Priority sample” checkbox before submitting a Study Queue to acquisition. The priority sample will be inserted into the magnet as soon as the current sample is completed, as compared to being added to the Spectrometer Queue in the order that the studies were created.

4.2.5 Editing experiments in a pending study

Until acquisition has begun on a queued experiment, that experiment can be modified or deleted from the study.

1. Load the study to be edited into the Study Queue pane by clicking the location in the tray display and then clicking **Edit Study**.
2. Double -click the experiment node to be edited, or simply drag the node to the trash can to delete it.
3. After changing the parameters, click **Save**.
4. Select the location of the sample from the tray display, and click **Submit**.
5. Click **Done** to return the interface to Review mode.

4.2.6 Extending an existing study

After a study has been created, it is possible to add experiments to the study. Study status (pending, active, completed) does not affect adding experiments to the study. The Study does not have to have been collected on an instrument. Studies can be continued on any VnmrJ 3 spectrometer. After a Study is loaded and ready for edit, the workflow is identical to the creation of a new Study.

1. Load the study to be edited into the Study Queue pane.\
    a. Select the location in the Tray display.
    b. Select a data set from the study.

       File→Open→select the file or FID to open→Go to Main menu→Tools→Study Queue Actions→Refresh Study Queue.

       For more information, see File Menu and See Tools Menu.
    c. By recalling a previous Automation file using the Tray Archives tool (see Automation Menu) and clicking the appropriate location in the tray display.
2. After the study is loaded into the Study Queue, click **Continue Study** or **Edit Study** (for active/pending Studies).
3. Add experiments to the queue from the Experiment Selector and customize, see Editing experiments in a pending study.
4. Select the location of the sample in the tray display and click **Submit**.
5. Click **Done** to return the interface to Review mode.

4.2.7 Deleting a Sample From the Queue

To delete a sample from the Study Queue:

1. Select **New Study.**
2. Select the location to be deleted from the tray display.
3. Select **Edit Study from Location.**
4. Select **Clear Pending Exp from Queue.**
5. Select **Submit.**

4.3 Operations Without a Robot Sample Changer

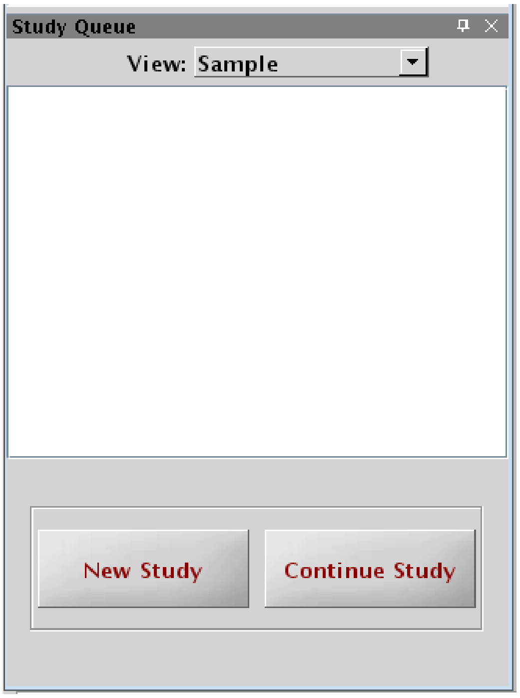


Figure 6 Study Queue—Review Mode

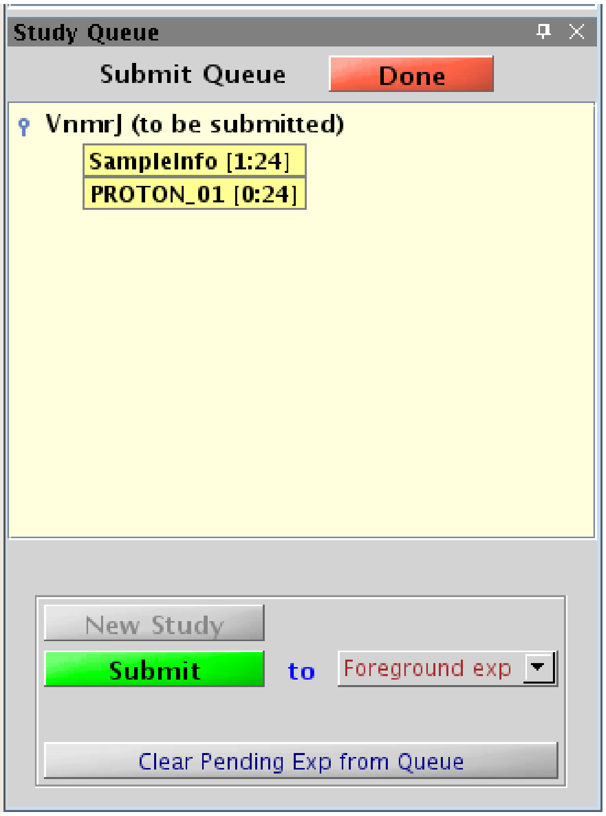
4.3.1 Study Queue Controls—Review Mode

When in Review mode the Study Queue pane is used to display information about the current study.

The View pull-down selects what information is displayed:

- Sample – shows the study linked to the data in the current workspace
- Active Sample – shows the study that is currently running

The New Study button initializes a new study and moves the software to Submit mode. The Continue Study button modifies a completed study currently displayed in the Study Queue.



4.3.2 Study Queue Controls—Submit Mode

In Submit mode, the Study Queue pane is used to build a study and submit it to acquisition.

Button	**Function**
Done button	Abandons any changes made to the current study and returns the software to Review Mode.
New Study button	Creates a new study.
Submit button	Submits the current study to acquisition. The associated pull-down menu determines how the acquisition is to be performed.
Foreground exp	Submits the study to acquisition in the current workspace.
Background	Submits the study to a background copy of VnmrJ.
Clear Pending Exp from Queue button	Deletes all pending experiments from the current Study Queue.

4.3.3 Using the Study Queue—Full Automation

To collect a series of spectra on a sample using the Study Queue:

1. Place the sample in a spinner turbine and adjust its position using the sample depth gauge. Insert the sample/spinner into the magnet. For more information, see 3.2.2 To Insert a Sample, Without a Sample Changer.
2. Click **New Study** in the Study Queue vertical panel. This initializes the Study and moves the software into “submit mode.”
3. Click on the buttons in the Experiment Selector to add the desired experiments to the Study Queue. The order of the experiments in the Study Queue can be changed using a click-and-drag action. Dragging an experiment node to the trash can deletes it from the queue.
4. Experimental parameters for an individual experiment can be customized by double-clicking on the experiment node in the study queue. (This is dependent upon the rights and privileges permitted for each user by the System Administrator.) The parameter set is loaded into the current workspace where any desired changes can be made using the horizontal panels under the “Acquisition” tab. Once the desired changes are complete, click **Save**.
5. Fill in the information associated with the sample, such as sample name, solvent, email address, comments about the sample, *etc*. The sample name parameter is typically required as part of the automatic data saving routine, and the solvent information is used to set the spectral window.
6. Select the checkboxes to control automatic printing, locking, shimming, and tuning, as appropriate.
7. Click **Submit**.
8. The software will submit the Study Queue to acquisition and return to Review mode.

4.3.4 Creating a Study Queue from data acquired manually

A common workflow in the research laboratory is to collect multiple proton spectra on a sample as the user optimizes conditions, such as shimming or solvent suppression, for that sample. Once the experimental conditions are deemed correct, a series of spectra can be acquired. The Study Queue supports this common workflow.

1. Acquire a PROTON spectrum (any experiment could be used but PROTON is most common) in foreground using manual acquisition. (See Chapter 1, *Spectroscopy User Guide*).
2. Use the File → Autosave function to save the fid using the data save template established in User Preferences (See Templates Tab). This action creates the directory structure required by the Study Queue.
3. In the Study Queue pane, select View → Sample to refresh the Study Queue display.
4. Click **Continue Study** to open the Study and move the software into “submit mode.”
5. Click on the buttons in the Experiment Selector to add the desired experiments to the Study Queue. The experiments are created based on the optimizations done in the PROTON experiment, retaining optimized parameters such as sweep width, solvent suppression, and so on.
6. Once the Study has been built and customized as desired, click **Submit**.

4.3.5 Editing experiments in an active study

Until acquisition has begun on a queued experiment, that experiment can be modified or deleted from the active study.

1. Load the active study into the Study Queue pane by choosing View → Sample.
2. Click **Edit Study**.
3. Double - click the experiment node to be edited, or simply drag the node to the trash can to delete it.
4. After changing the parameters as desired, click **Save**.
5. Click **Submit**.

4.3.6 Extending a completed study

Once a Study has been created, it is possible to add more experiments to that Study whether the study is active or completed. The Study does not have to have been collected on any particular instrument. Studies can be continued at any time on any spectrometer under VnmrJ 3. Once the The workflow is identical to the creation of a new study once the study to be edited is loaded.

1. Load the study to be edited into the Study Queue pane
    a. by choosing View → Sample in the Study Queue pane when the current workspace holds a data set from the study of interest.
    b. by recalling a data set from the study of interest using File → Open (see File Menu) and then selecting Tools → Study Queue Actions → Refresh Study Queue. (See Tools Menu)
2. Once the desired Study has been loaded into the Study Queue, click **Continue Study**.
3. Experiments can now be added to the queue from the Experiment Selector and customized as described above.
4. Click **Submit**.

Chapter 5 Reprocessing Data

This chapter describes how to retrieve the dataset, Fourier Transform (FT) the dataset, alter the dataset parameters, use the graphical toolbar, display parameters, and save.

Sections in this chapter include:

- 5.1 Retrieve the dataset from the disk
- 5.2 Fourier Transform the Dataset
- 5.3 Alter Processing Parameters
- 5.4 Interact with the spectrum using the graphical toolbar
- 5.5 Display parameters
- 5.6 Save Current Process / Display parameters

5.1 Retrieve the dataset from the disk

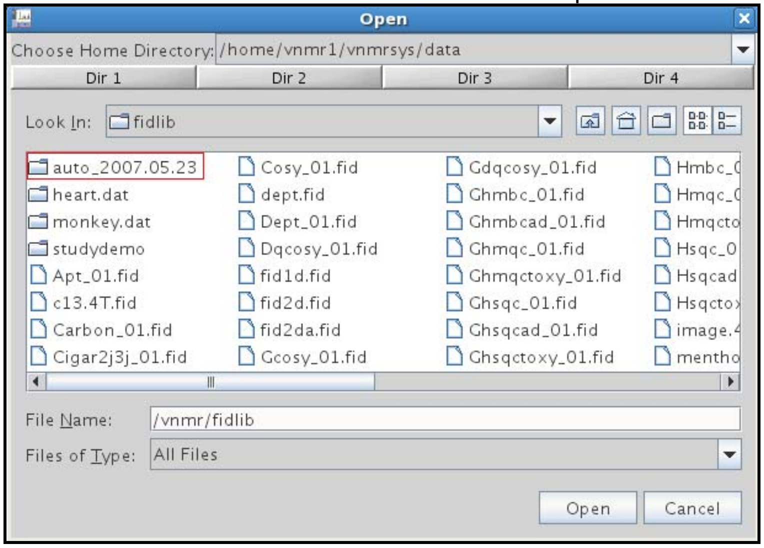
5.1.1 Using the Open window

Before any re-processing can be done on a dataset, the desired dataset must be in the current workspace. (To change to a different workspace/experiment/viewport, see the *Spectroscopy Guide*.) Browser and Locator can also be used to retrieve data, see 5.1.2 and

To retrieve the data: File→Open.

The resulting pop-up window lets you access the desired data directory.

1. Select the desired file.
2. Click the **Open** button.
   The selected dataset is loaded into the current workspace.



Choose Home Directory: /home/vnmr1/vnmrsys/data

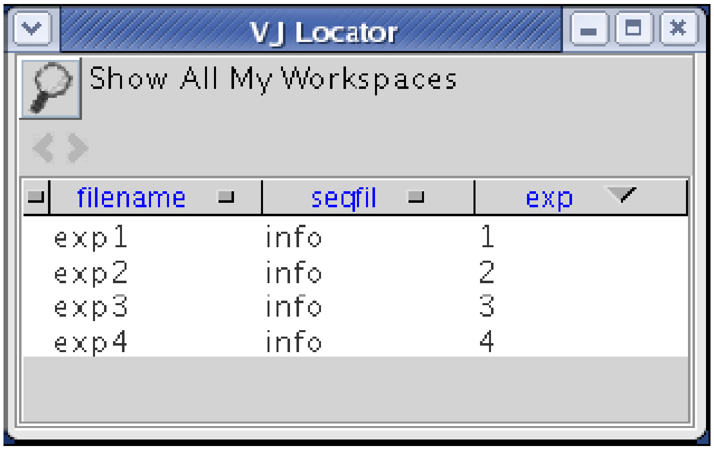
Look In: fidlib

File Name: /vnmr/fidlib

Files of Type: All Files

5.1.2 Using the Locator and Browser windows

The Locator provides access to data sets and experiments.



filename	seqfil	exp
exp1	info	1
exp2	info	2
exp3	info	3
exp4	info	4

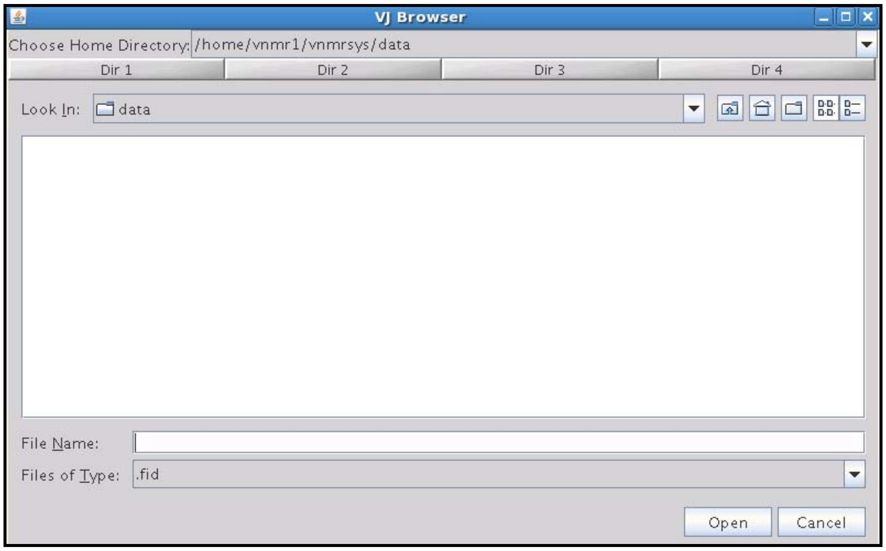
Figure 7 VJ Locator popup menu

1. Click **Tools** from the main menu.
2. Click **Locator…**

Click the magnifying glass icon to open a menu of searches. Selecting a search changes the search sentence (next to the magnifying glass icon). The results of the search are displayed in the list. Those items in the white part of the list satisfy the search sentence. Those in the gray part do not. Three attributes are displayed for each item that is found by the search. The attributes correspond to the three columns in the list. Click the attribute name at the top of the list opens a menu of attribute choices.

Click an item in the Locator list to select that item. The selected item can be dragged to the graphic area or the parameter panel area to cause the appropriate action.

For example, dragging a data set to the graphic canvas retrieves that data set into the current workspace (experiment). Dragging a workspace to the graphic canvas causes that workspace (experiment) to be joined with the graphic area. Double-clicking an item performs the same action as dragging the item to the graphics canvas.



Choose Home Directory: /home/vnmr1/vnmrsys/data

Look In: data

File Name:

Files of Type: .fid

Browser Buttons

Icon	Icon Description
⇧	Go up one level in the directory tree.
⌂	Go to user’s home directory.
▭	Make a new folder in the current directory.
⊞	Show list of files and directories at the current directory level.
☰	Show details of files and directories at the current directory level.

5.1.3 Using the Study Queue

If a dataset was run in VnmrJ 3 or later, all related data in the corresponding study can be brought into the Study Queue window by clicking on the Refresh Study Queue button in Tools→Study Queue Actions. This facilitates the review or re-processing of the other datasets in that study.

5.2 Fourier Transform the Dataset

5.2.1 Fourier Transform of One-Dimensional Data

The data is typically Fourier transformed into a spectrum before analysis. The data is stored as time-domain data; the FT converts it to frequency-domain data. FT can be done using any of the following buttons/ menu:

- The blue-green Transform button in the Action Bar
- The Transform All button on the upper left of the Process / Default page (it is also on the Process/Weighting page)

- The Process button in the middle bottom of the Process / Basic page
- The Process menu, which has options for either Process or Display 1D and Full Process

5.2.2 Fourier Transform of Two-Dimensional Data

The FT of a 2D dataset can also be done in several equivalent ways:

- The blue-green Transform button in the Action Bar
- The Full 2D Transform button on the upper left of the Process / Default page (it is also on the Process/Weighting page and the Process/More 2D page)
- The Process button in the middle bottom of the Process / Basic page
- The Process menu, which has options for Full Process 2D
- The full processing of a 2D spectrum requires a Fourier Transform along two perpendicular directions, called t1 and t2. These two processing steps can be done one at a time if desired. This allows you to customize the processing parameters in each direction (if desired). For more details, see the corresponding *Spectroscopy Guide*.

5.3 Alter Processing Parameters

More information can be extracted from an NMR spectrum if the processing parameters are optimized specifically for that spectrum (optimized in ways which help answer the questions being asked). Examples include changing the parameters that control processes like zero-filling, weighting functions (and apodizations), linear prediction, referencing, or integral reset points. These parameters are typically controlled using the Process tab.

5.3.1 Zero-Filling

Zero-filling is controlled by the Transform Size controls, which are located on multiple pages, such as Basic, Default, Weighting, and More 1D pages. If the number of points used is larger than the number of points acquired, zero filling is being performed. This helps to better define shapes of the peaks. It is common and advantageous to transform twice as many points as were acquired. The user can transform fewer acquired data points, which allow the FT calculation to be faster, but at the expense of a less well-defined lineshape.

5.3.2 Weighting and Apodization

The detailed parameters controlling the weighting functions and functions that are applicable to the FID before the FT, are defined in the Weighting page in the Process tab. There is also a simplified set of controls on the Default page. Typically, weighting functions are used to increase signal-to-noise, at the expense of broader lines. These functions are used to remove truncation wiggles from the spectrum as needed. The “apodization” process is when the weighting functions are used to remove truncation wiggles

5.3.3 Linear Prediction

All of the detailed parameters that control linear prediction are located in the More 1D page. There is a simplified set of controls on the Default page. The Auto buttons set up a default set of conditions for either forward or backward linear prediction. Many of the protocols in the Experiment Selector set up appropriate parameters for using linear prediction automatically. For more detailed information about controlling linear prediction, see the *Spectroscopy Guide*.

5.3.4 Referencing

Automatic processing uses a routine to provide default referencing for the spectrum. To alter the referencing, use the controls located in either the Default or the Display pages. Referencing By Solvent or By TMS automatically analyzes the spectrum. Referencing the spectrum to a certain cursor position requires the user to place the cursor at the desired location in the spectrum and type in the desired numerical value for that position in the Reference cursor to entry box.

5.3.5 Integration

Integral regions are automatically set up for Proton spectra during automated processing. Further manipulations of the integral can be made by using the controls in the Integration page on the Process tab, or in the graphical tool bar, see Interact with the spectrum using the graphical toolbar.

5.3.6 Phasing

Proper spectral phasing is automatically set up for all spectra during automated processing. Further manipulations of the phasing can be made using the graphical toolbar.

5.4 Interact with the spectrum using the graphical toolbar

The graphics control bar for the active viewport is to the right of the graphics canvas. Use the buttons in the bar to control the interactive display in the graphics canvas.

5.4.1 Common Graphics Display Toolbar Controls

The following tools are common to 1D, nD, and FID display toolbars.

Table 5 Graphic Display Tool Bar Controls

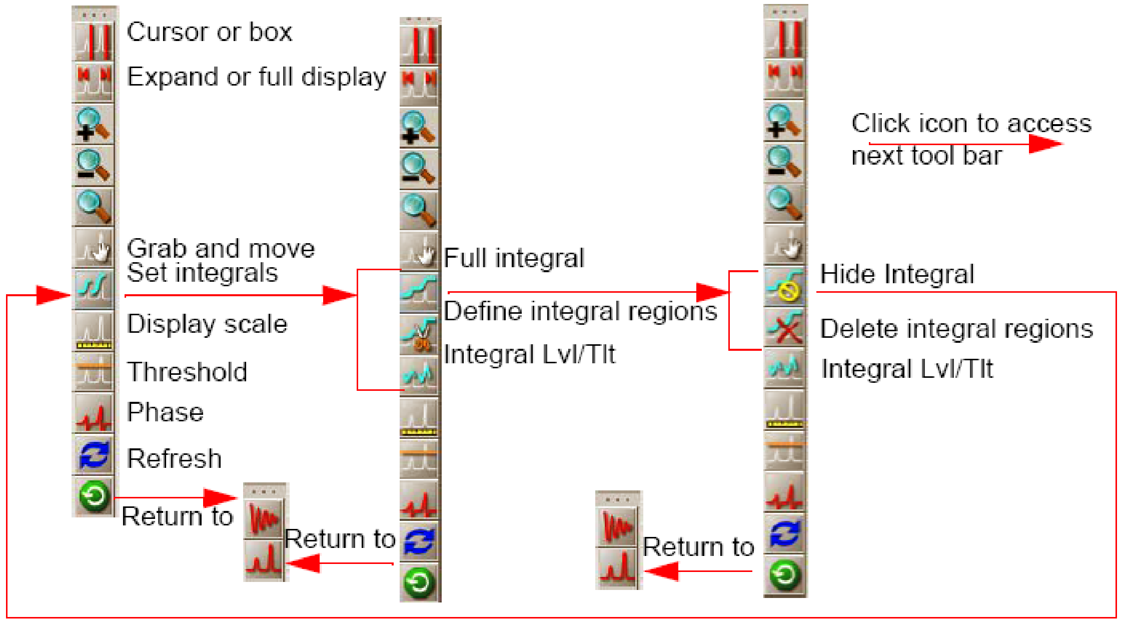
Icon	Description
⛶	Reset to full display.
🔍+	Zoom in.
🔍−	Zoom out.
🔍	Select zoom region.
🔄	Redraw display.
↺	Return to previous tool menu.

5.4.2 1D Display Spectrum Toolbar Controls

Icon	Description
	One cursor in use, click to toggle to two cursors.
	Two cursors in use, click to toggle to one cursor.
	Click to expand to full spectral display.
	Pan or move spectral region.
	Display integral.
	Display scale.
	Toggle threshold on or off.
	Phase spectrum.

5.4.3 Integration and Graphics Controls

This section describes methods and tools for displaying, and plotting integrals.



5.4.4 nD Display Toolbar Controls

Icon	Description
	Display color map and show common nD graphics tool.
	Display contour map and show common nD graphics tool.
	Display stacked spectra and show common nD graphics tool.
	Display image map and show common nD graphics tool.
	One cursor in use, click to toggle to two cursors.
	Two cursors in use, click to toggle to one cursor.
	Click to expand to full display.
	Pan and stretch.
	Show trace.
	Show projections.
	Shows horizontal maximum projection across the top of the 2D display.
	Shows horizontal sum projection across the top of the 2D display.
	Shows vertical maximum projection across the top of the 2D display.
	Shows vertical sum projection down the left side of the 2D display.
	Rotate axes.
	Increase vertical scale 20%.

Icon	Description
	Decrease vertical scale 20%.
	Phase spectrum tool bar selection.
	First spectrum selection.
	Second spectrum selection.
	Enter peak pick menu.

5.5 Display parameters

The parameters that control the appearance of the spectrum can be saved in temporary display parameters files, which allow the user to easily jump between different displays of one dataset. There are eight display-parameter files available for each experiment (workspace) in each user account. Each display-parameter file saves the parameters that describe the appearance of the spectrum like **`sp, wp, sp2, wp2, sc, wc, sp2, wc2, vs, is, vs2d, th,`** and the integral reset regions). This file does not store or alter phasing, referencing, or baseline-correction information.) The files are created with the typed command `s1, s2, s3,` up to `s8`, and recalled with the typed commands **`r1`**, **`r2, r3,`** up to **`r8`**. The typed commands **`fr1`** through **`fr8`** ("full recall 1") also recall the integral reset regions, which are not recalled with the simpler r1 style command.

These display-parameter files can be copied to other experiments by using the typed command **`md (m,n)`** (where m is the experiment with the files and n is the experiment to which you want to copy the files). This copy function can also be accomplished by using the Edit / Move Display parameters menu. This capability is quite useful for looking at exactly the same expansion of multiple NOESYs run with different mixing times, or looking at exactly the same expansion region in multiple NOESY, TOCSY, ROESY, and COSY datasets.

The display-parameter files remain in the local experiment until the experiment is either deleted or the files removed. The display-parameter files are not useful for experiments run with different spectral widths; for example, if an s1 was saved on a proton spectrum, if a carbon spectrum is later brought into this same experiment, the r1 will give a non-useful expansion.

5.6 Save Current Process / Display parameters

In the Basic and Default pages of the Process tab, there is a button labeled Save Current Process / Display Parameters. Click this button to save a parameter set in the dataset with all the current processing and display information, in a manner that is suitable for GLP requirements. This action stores parameters such as phasing, integral regions (resets points and `isadj`), and the display information that would be stored by the `s1` and `r1` commands (described above). (It does not store information about referencing, symmetrization, baseline correction, or drift correction.) The next time any Process button is clicked, the last-stored set of display parameters will be used to display the resulting spectrum.

For more detailed information about processing NMR data, see the corresponding *Spectroscopy Guide*, Chapter 6 (for 1D data) and Chapter 11 (for 2D data), where each of the steps discussed above is covered in more detail.

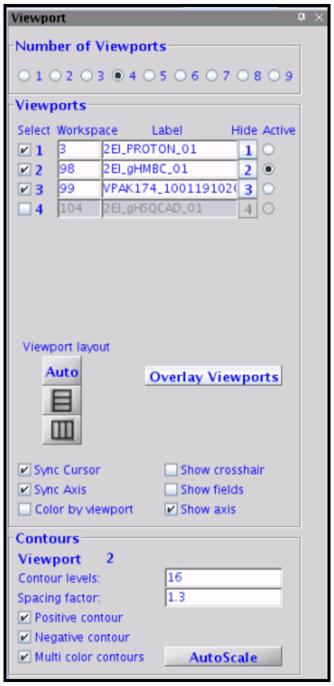
Chapter 6 Interpreting Data

This chapter includes:

- 6.1 Viewport Tab
- 6.2 Working with Viewports and Inset Frames
- 6.3 Requirements for Aligning and Stacking Spectra
- 6.4 Setting up Stacked Aligned Spectra
- 6.5 Interactive Zero- and First-Order Baseline Correction Mode
- 6.6 Displaying Integrals Step-by-Step
- 6.7 Baseline Correction
- 6.8 Baseline Correction Commands
- 6.9 Integral Reset Points Commands
- 6.10 Integral Regions Commands
- 6.11 Integral Display and Plotting Commands

6.1 Viewport Tab

Click the Viewport tab to display the viewport controls. If not visible, click View on the main menu and select Viewport. Refer to Viewport Tab, for description of the tools and buttons.



Number of Viewports

○ 1 ○ 2 ○ 3 ● 4 ○ 5 ○ 6 ○ 7 ○ 8 ○ 9

Viewports

Select		Workspace	Label	Hide	Active
☑	1	3	2EI_PROTON_01	1	○
☑	2	98	2EI_gHMBC_01	2	●
☑	3	99	VPAK174_100119102	3	○
☐	4	104	2EI_gHSQCAD_01	4	○

☑ Sync Cursor
☑ Sync Axis
☐ Color by viewport
☐ Show crosshair
☐ Show fields
☑ Show axis

Contours

Viewport 2
Contour levels: 16
Spacing factor: 1.3
☑ Positive contour
☑ Negative contour
☑ Multi color contours

Figure 8 Viewport Tab

6.1.1 Viewport Tab

Field	Function
Viewports	Select the number of viewports to display.
Viewport Layout	Select the layout for the viewport and cursor, axis , and othe viewport controls.
Contour	The contour panel displays if the active viewports contain an Nd spectrum. More extensive controls provided on the 2D vertical panel.

6.1.2 Inset Frame Buttons

Use Inset Frame buttons to delete one or all inset frames and to restore the default frame to full size.

Button	Function
Delete Inset	Delete the selected inset.
Delete all	Delete all inset frames.
Full size	Restore the default frame to its full size.

6.1.3 Display Check Boxes

Display Check boxes controls optional display features.

Check box	Function
Cross hair	Display cross hair and chemical shift(s) of the cursor position when mouse is moved over the spectrum. A useful function when the fields are not shown, not in cursor mode (default mode), or when chemical shift of a peak without moving the left cursor is required while in the cursor mode.
Fields	Display cr, delta, vp etc.fields at the bottom of the viewport.
Axis	Show scale of the axis.
Show frame border	Select the check box to display a box around the frame. Clear this check box to display the four corners of the selected frame as *hot spots* for resizing. No border or corner will be displayed if a frame is not selected. An empty frame is not visible until it is selected.

6.2 Working with Viewports and Inset Frames

All VnmrJ graphics are displayed in frames. The viewport has a default frame that occupies the entire viewport graphics area. An inset frame initially shares the same workspace and data as the original frame and is configured in the same way as the default frame.

6.2.1 Creating an Inset Frame

An inset frame has the full capability of the default frame. The only difference is that the default always exists, while an inset frame can be created and removed.

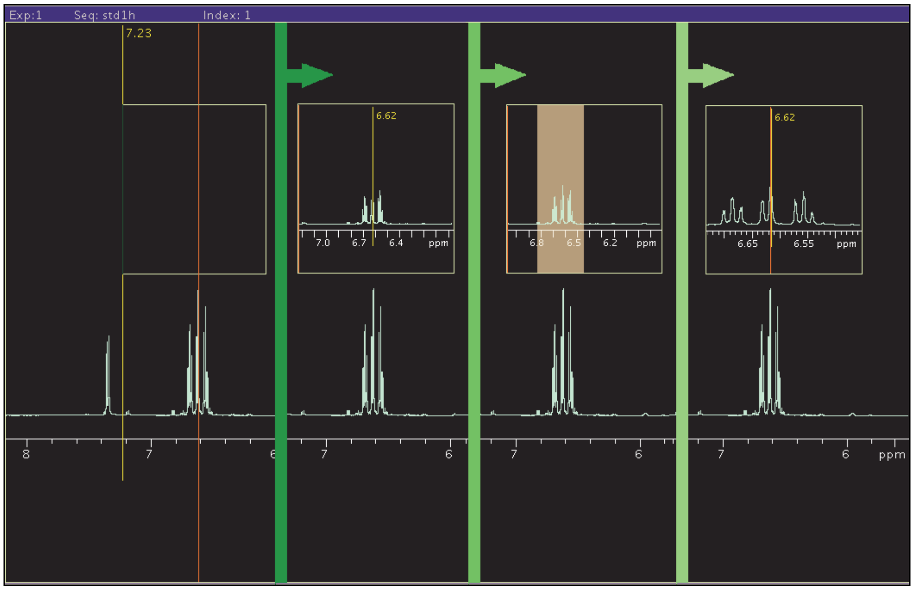


Figure 9 Creating an Inset Frame

Create an inset frame within the default viewport frame as follows:

1. Select the inset mode tool
2. Place the cursor at the low field (left) side of the region to be expanded as shown in Figure 9, frame 1a.
3. Hold the left mouse button down and drag the inset window to the high field (right) side of the region.
4. Drag the cursor down to set the height of the inset frame as shown in Figure 9, frame 1b.
5. Release the mouse button to create the inset frame, see Figure 9, frame 2.

6.2.2 Zooming in on a Region Within an Inset Frame

1. Select the default mode tool
2. Click inside the frame to make the frame active.
3. A frame has a yellow border when it is active and white border when it is inactive (these are the default colors of inactive and active frames).
4. Select the zoom mode tool
5. Place the cursor at the low field (left) side of the region to be expanded as shown in Figure 4 frame 3a.

6. Hold the left mouse button down and drag the inset window to the high field (right) side of the region, Figure 4 frame 3b.
7. The region selected is indicated by a transparent gray rectangle.
8. Release the mouse button and the selected region expands to fill the inset box, Figure 4 frame 4.

6.2.3 Resizing an Inset Frame

1. Select the default mode tool
2. Click inside the frame to make the frame active. An active frame has a yellow border.
3. Move the mouse cursor to a corner of the inset frame. The cursor changes from a single-headed arrow to a double-headed arrow.
4. Hold down the left mouse button and grab the corner of the frame.
5. Drag the corner to resize the frame.
6. Release the left mouse button when the frame is at the desired size.

6.2.4 Moving an Inset Frame

1. Select the default mode tool
2. Click inside the frame to make the frame active. An active frame has a yellow border.
3. Move the mouse cursor to an edge of the inset frame. The cursor changes from a single-headed arrow to a four-headed arrow.
4. Hold down the left mouse button and grab the edge of the frame.
5. Drag the frame to the new position.
6. Release the left mouse button when the frame is at the desired position.

6.3 Requirements for Aligning and Stacking Spectra

Spectra can be a mixture of 1D and 2D data sets, all 2D data sets, or all 1 D data sets, provided the following requirements are met:

- All selected viewports need to use a common scale.
- Data in the viewports may have different nuclei, different spectral widths, or different spectral regions. The common scale is determined based on data in all selected viewports and determines whether alignment or stacking is possible. Overlaid and stacked spectra are drawn based on the common scales.
- Alignment is enabled if more than one axis in more than one viewport has the same axis (H1, C13 etc).
- Stacking is enabled when data in all viewports have the same axis/axes.

6.4 Setting up Stacked Aligned Spectra

1. Select the **Viewport** tab from the vertical tabs panel.
2. Load each data set into a different viewport and process the data. Data must meet the Requirements for Aligning and Stacking Spectra.

3. Select viewports containing spectra to overlay by selecting the check box under **Select**.
4. Click **Overlay viewports** button to overlay all selected viewports.

   The Stack Spectrum button, Figure 10, is displayed below Overlay Viewports button, if all spectra have the same dimension (all 1D or all 2D) and all axis/axes (nuclei) match. Stacked spectra are aligned and each spectrum is shifted along x and y. The shift between spectra is specified by x and y offset in the entry fields below Stack Spectrum button. Spectral axes are also synchronized to enable zoom and pan of the spectrum without losing the alignment.

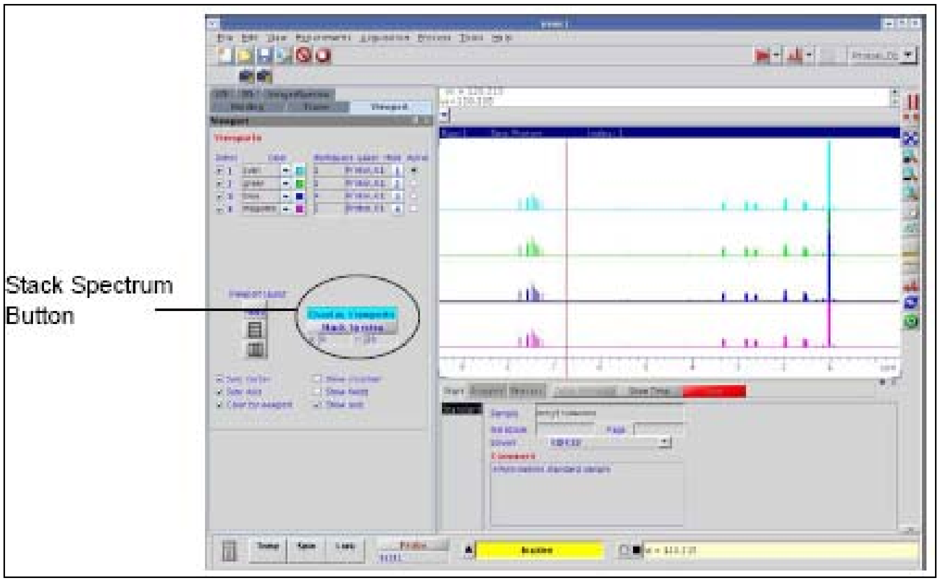


Figure 10 1D Stacked Spectra

The Align spectra button, Figure 11, is available when multiple spectra are overlaid - a mix of 1D and 2D spectra. All 2D spectra must have matching axes. All 1D data must match one of the 2D axes. 1D spectrum is aligned and displayed at the margins of the 2D spectrum. 1D spectrum is rotated if necessary to align with the 2D spectrum. Zoom and pan tools are synchronized when the spectra are aligned.

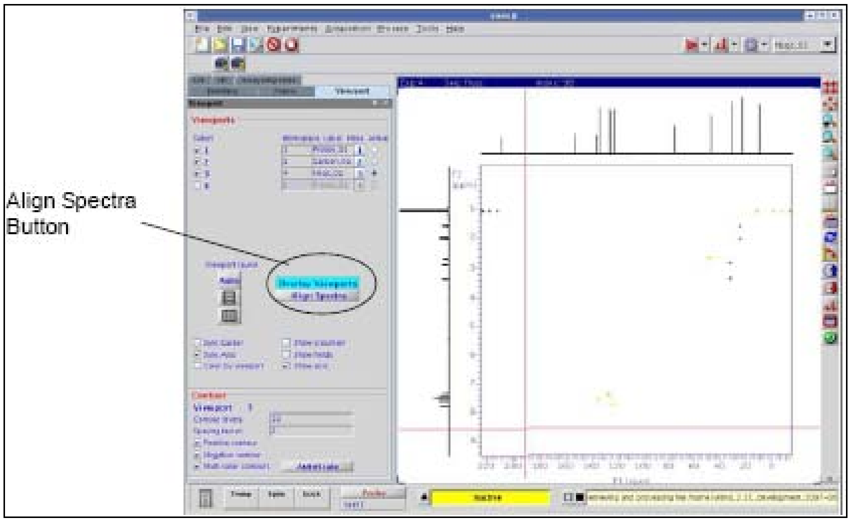


Figure 11 2D Spectra with Overlaid 1D's

The stacked spectrum button for 2D spectra, Figure 12, is displayed below Overlay Viewport if all 2D spectra axes and nuclei match. Spectral axes are synchronized to enable zoom and pan of the spectra without losing alignment.

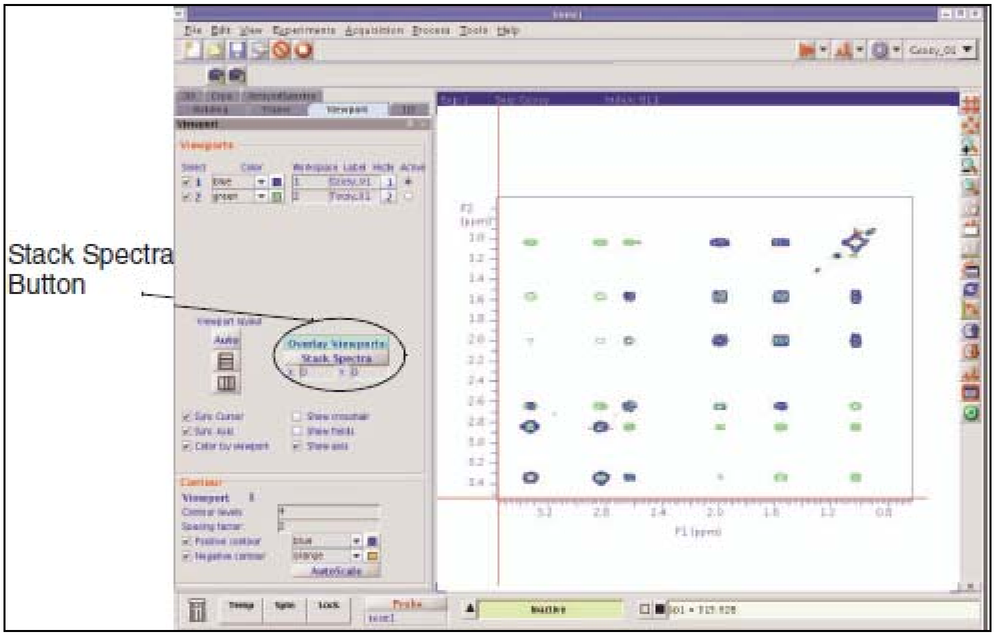
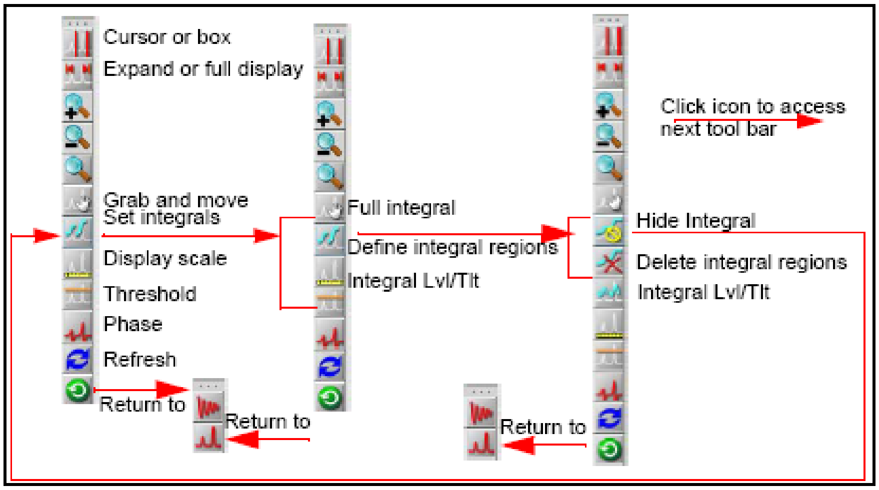


Figure 12 Stacked Overlaid 2D Spectra

6.5 Integration and Level and Tilt

Methods, and tools for displaying and plotting integrals are described below.



6.5.1 Interactive Zero- and First-Order Baseline Correction Mode

The Integral Lvl/Tlt button activates interactive zero and first order baseline correction mode. The zero order correction is represented by the **lvl** parameter; the first order correction is represented by the **tlt** parameter. If no integral is displayed when the Integral Lvl/Tlt button is clicked, the integral is automatically displayed.

1. Click the left mouse button on an integral region of interest, about halfway vertically up the screen.

   A horizontal cursor intersects at the mouse arrow. Two vertical cursors are placed on either side of the mouse arrow.

2. Click the left or the right mouse button above or below the horizontal cursor, but within the two vertical cursors, to adjust the zero-order baseline correction parameter lvl.

- Clicking above the horizontal cursor increases lvl.
- Clicking below the horizontal cursor decreases lvl.
- Clicking on the horizontal cursor restores the initial baseline correction value.

3. Click the left mouse button on another region of the spectrum, outside the vertical cursors.

   A new horizontal cursor displays at the mouse arrow, two new vertical cursors display on either side of the mouse arrow, and a single vertical cursor displays in the middle of the region where lvl was being updated. The mouse now controls the first-order baseline correction parameter tlt**.**

4. Click the left or the right mouse button above or below the horizontal cursor to increase or decrease **tlt**, and change **lvl** so that the total drift correction at the single vertical cursor in the middle of the previous region is held constant.

   This process eliminates or substantially reduces the necessity to iteratively adjust the two parameters lvl and tlt. As with the zero-order correction, placing the mouse arrow right on the horizontal cursor and clicking the mouse button will restore the initial baseline correction values.

   Each time the mouse is clicked outside the two vertical cursors, new vertical and horizontal cursors display.

   Using the left and right mouse buttons, you can adjust both adjust the baseline correction parameters differing only in their sensitivities. The left mouse button causes changes a factor of eight times larger than the right mouse button making the left button a “coarse” adjust and the right button a “fine” adjust. The overall sensitivity of these adjustments can also be controlled by the parameter lvltlt. The parameter can be set according to the following:

- The default value is 1.
- Make lvltlt larger than 1.0, to make larger changes.
- Set lvltlt to be between 0.0 and 1.0, to have finer control.

  The middle mouse button adjusts the integral scale (parameter is) or the integral offset (parameter io), exactly as whenever an integral is displayed.

5. Exit the interactive baseline correction mode by clicking another graphics control button.

6.5.2 Displaying Integrals Step-by-Step

The following methods provide an opportunity to compare procedures. Before starting each procedure, be sure to obtain a typical spectrum by entering:

1. Load a data file into the active viewport using the file browser or the Locator.
2. Transform the data, if necessary.
3. Click the **Process** tab.
4. Select the **Integration** page.
5. Click any one of the Integration Display Mode option buttons:

- Full: Shows integrals over the entire spectrum, including the noise.
- Partial: Shows even integrals regions and hides all the odd integral regions.
- Off: Turns off the integral display.

6. Click **Auto Find Integrals** to automatically set the integral resets, and display the data as set by the Integration Display Mode option button.
7. Click **Scale display to fit** button to automatically scale the display.

8. Set the integral area:
    c. Enter a value in the Integral Area field.
    d. Click one of the following option buttons under the Normalize Area to: page region.
    - Single Peak — select the region or peak under the cursor as the reference and set the single peak integral to the value in the Integral Area field when the Set Integral Value button is clicked.
    - Sum — sets the entire integral to the value in the Integral Area field.

      Do not click the Set Integral Value button; this button sets the single peak reference.
9. Display the integral results as follows:

- Single Peak — both the Show Integral Values and Show Normalized Value buttons are active.
- Integral Values

  Click **Show Integral Values** to display the values of the integral regions on the screen below the spectrum.

  Click **Display Lists of Integrals** to list the display regions and the value of the integral over each region.
- Normalized Integral Values

  Click **Show Normalized Values** to display the values of the integral regions normalized to the reference region on the screen below the spectrum.

  Click **Display Normalized Integrals** to list the display regions and the value of the integral over each region normalized to the reference region.

6.5.3 Manual Method

1. Load a data file into the active viewport using the file browser or the Locator.
2. Transform the data if necessary.
3. Click the **Process** tab.
4. Select the **Integration** page.
5. Click **Clear Integrals**.
6. Any currently defined integral reset points are cleared.
7. Set up the integral resets from left to right (down field to up field).
    a. Click **Interactive Resets** button.
    b. Place the cursor to the left of the first integral region.
    c. Click the left mouse button.
    d. Move the cursor to the right of the first integral region.
    e. Click the left mouse button.
8. Repeat Step 7b.
9. Click **Scale display to fit** button to automatically scale the display.

10. Set the integral area as follows
    a. Enter a value in the Integral Area field.
    b. Click one of the following radio buttons under the Normalize Area to: page region.

- Single Peak — select the region or peak under the cursor as the reference and set the single peak integral to the value in the Integral Area field when the Set Integral Value button is clicked.
- Sum — sets the entire integral to the value in the Integral Area field.

  Do not click the Set Integral Value button; this button sets the single peak reference.

11. Display the integral results as follows:

- Single Peak — both the Show Integral Values and Show Normalized Value buttons are active.
- Integral Values
- Click **Show Integral Values** to display the values of the integral regions on the screen below the spectrum.
- Click **Display Lists of Integrals** to list the display regions and the value of the integral over each region.
- Normalized Integral Values
- Click **Show Normalized Values** to display the values of the integral regions normalized to the reference region on the screen below the spectrum.
- Click **Display Normalized Integrals** to list the display regions and the value of the integral over each region normalized to the reference region.

6.6 Baseline Correction

Most operations performed on spectra assume a “good” baseline. Line lists, integrations, resolution measurements, 2D volume integrations, and so on measure intensities from “zero” and do not perform any baseline adjustments. Perform a baseline correction operation before performing further data reduction if the baseline in your spectrum is not “good.” Two types of baseline corrections are provided, linear and non-linear, and are available using the buttons on the Display page.

6.6.1 Baseline Correction Commands

The `dc` command turns on a linear baseline correction, using the beginning and end of the displayed spectrum to define a straight line to be used for baseline correction.

`dc` calculates a zero-order baseline correction parameter **`lvl`** and a first-order baseline correction parameter **`tlt`**. The `cdc` command turns off this correction. The results of the `dc` or `cdc` command is stored in the **`dcg`** parameter, which can be queried to determine whether drift correction is active by typing `dcg`? on the command line. If active, **`dcg=''`**; if inactive, **`dcg='cdc'`**.

The `bc` command performs a 1D or 2D baseline correction. The 1D baseline correction uses spline or second to twentieth order polynomial fitting of predefined baseline regions. `bc` defines every other integral, that is, those integrals that disappear in partial integral mode `(intmod='partial')` as baseline and attempts to correct these points to zero. A variety of parameters can be used to control the effect of the `bc` command.

For more information about the `bc` command, refer to the entry for `bc` in the *Command and Parameter Reference*.

6.7 Integral Reset Points Commands

The `z` command (or the equivalent button or icon) resets the integral to zero at the point marked by the displayed cursor. `z(reset1,reset2,...)` allows the input of the reset points as part of a command, instead of using the position of the cursor. Reset points do not have to be entered in order. The resets are stored as frequencies and will not change if the parameter **fn** is changed. The command `cz` (or the equivalent button) removes all such integral resets. `cz(reset1,reset2,...)` clears specific integral resets.

The **liamp** parameter stores the integral amplitudes at the integral resets points, and the **lifrq** parameter stores the frequencies of integral reset points, for a list of integrals. To display the values of liamp, enter `display ('liamp')` with a **Text Output** page selected. Frequencies are stored in Hz and are not adjusted by the reference parameters **`rfl`** and **`rfp.`**

6.8 Integral Regions Commands

The region command divides a spectrum into regions containing peaks. A variety of parameters can be used to control the effect of the region command; see the *Command and Parameter Reference* for details.

6.9 Integral Display and Plotting Commands

Displaying and plotting the integral trace is independent of the values of the integrals. The height of the trace is controlled by the parameter and can interactively be adjusted after the spectrum is displayed with the `ds` command. Also, the macro isadj(height) (or the equivalent button) adjusts the integral height so that largest integral fits the paper or is set to a specific height (in mm) if the “height” argument is provided, for example, isadj(100).

The command `dli` (or the equivalent button) displays a list of integral values at the integral reset points. The frequency units of the reset points are defined by the parameter axis. The reset points are stored as Hz and are not referenced to `rfl` and `rfp`. The amplitudes are stored as actual values; they are not scaled. The integral values are scaled by the parameters `ins` and `insref` and the Fourier number. Typically, `ins` is set to the number of nuclei in a given region. For example, if a region represented a single methyl group, the following procedure would scale the integral values of that region:

1. Set `ins=3`.
2. Set `insref` to the Fourier-number-scaled-values of that integral.
3. Enter `dli`. The integral value of that region is displayed as 3 and all other integral values are accordingly scaled.

Integral value scaling can be interactively set with the `ds` command. The setint macro can also be used to adjust integral value scaling. setint sets the value of an integral and is used in conjunction with the command `dli` to scale integral values. Normalized integral values can also be selected. In this case, `ins` represents the total number of nuclei. The individual integral values will be scaled so that their sum is equal to ins. The normalized mode may be selected by setting insref to “not used.” The integral is scaled by **`ins`** and **`insref`**.

Two commands are closely related to `dli`:

- `nli` is equivalent to `dli` except that no screen display is produced.
- `dlni` normalizes the values from `dli` using the integral normalization scale parameter ins and then displays the list.

The `dpir` command displays numerical integral values below the appropriate spectral regions, using the integral blanking mode in which only every other integral is plotted. The command `dpirn` shows the normalized integral values in an analogous fashion.

The `pir` command plots digital integral values below the spectrum, using the integral blanking mode in which only every other integral is plotted. The command `pirn` plots the normalized integral values in an analogous fashion.

6.9.1 Using Viewports Region Controls

The viewport controls are present if there are two or more view ports.

Showing and Hiding Viewports

The selected viewports are arranged on the graphics canvas based the layout selection. See Setting Viewport Layout.

1. Select the check box next to each viewport to show on the graphics canvas.
2. Clear the check box next to a view port to hide the viewport.
3. Point the cursor to the box next to the viewport label and hold down the left mouse button to temporarily hide a viewport.
4. Release the mouse button to show the viewport.

   The viewports do not change their layout on the graphics screen. This tool is used when overlay viewports is selected.

Making a Viewport Active

1. Click the radio button associated with a viewport to make the viewport active.

   The title bar of the active viewport is colored. The inactive viewports have gray title bars.
2. Use the horizontal and vertical panel tools to work on the data in active viewport or begin data acquisition using the active panel.

   Experiments started from the current active panel are run in the order of submission. Systems running an automated sample changer use only experiment 1 (which is in viewport 1) to submit samples to the automation queue. All other viewports are used for data processing and analysis.

Adding a Label to the Viewport

The default label for a viewport is the currently loaded experiment’s file name.

1. Click inside a viewport’s label box (viewport does not have to be active).
2. Select the contents of the box and overwrite the text with new text.
3. Click outside the text box.

   A new label is now associated with this viewport.

6.9.2 Setting Viewport Layout

Select a layout icon to arrange the view ports on the graphics canvas. Click the Overlay Viewports to stack viewport on view port. The overlay is useful for placing high resolution 1D spectra on the appropriate 2D axes.

Icon	Function
Auto	Auto layout arranges viewports in an optimized row by column matrix
▤	Horizontal layout of viewports
▥	Vertical layout of viewports

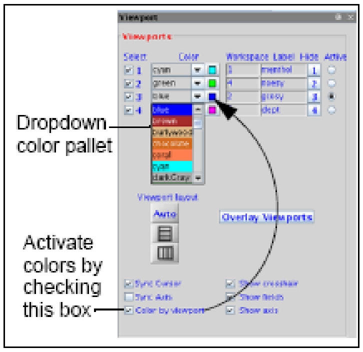
6.9.3 Synchronizing Cursors and Axes

Check Box	Function
Sync cursor	Select this checkbox to link and synchronize the cursors and cross hairs in multiple viewports.
Sync Axis	Select this checkbox to link and synchronize axes in multiple viewports. Axis is synchronized to the current active viewport.

6.9.4 Setting Crosshair, Fields, and Axis Display Options

Check Box	Function
Show crosshair	Select this check box to show crosshairs and display current position.
Show fields	Select this check box to show information fields at the bottom of the active viewport canvas:  vs sp(ppm) wp(ppm) first last step 111.7 35.78 65.00 1 4 1
Show axis	Select this check box to show the axis or remove the check to hide the axis.

6.9.5 Assigning Colors to Spectra by Viewport

Check Box	Function
Color by viewport	Select this check box to display the spectral data using colors assigned by the viewport, see Figure 13. **Default color assignment**: Spectra are displayed using a different color for each viewport if the box is checked. The spectra a displayed using the defaults assigned in the Display options window if this box is not checked. **Change a color assignment:** Click on the dropdown color menu for a viewport and select a color for the spectral display in the viewport.  *Figure 13 Setting Spectra Colors by Viewport*

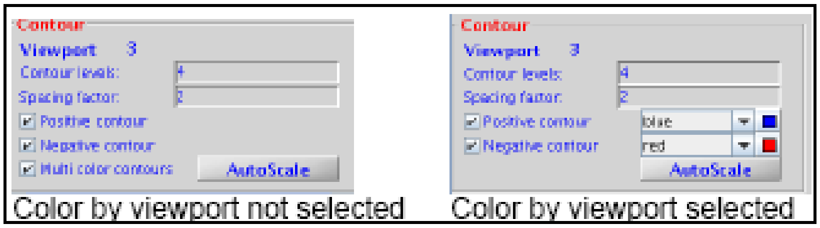
Display Check Boxes

The check boxes control of optional display features.

Check box	**Function**
Cross hair	Display cross hair and chemical shift(s) of the cursor position when mouse is moved over the spectrum. A useful function when the fields are not shown, not in cursor mode (default mode), or chemical shift of a peak without moving the left cursor is require while in the cursor mode.
Fields	Display **cr, delta, vp** etc... fields at the bottom of the viewport.
Axis	Show scale of the axis.
Show frame border	Select this check box to display a box around the frame. Un-check the box to display the four corners of the selected frame as *hot spots* for resizing. No border or corner will be displayed for if a frame is not selected. An empty frame is not visible until it is selected.

6.9.6 Contour

The contour sub-panel, Figure 14, appears exclusively for the active viewport with 2D data loaded and displayed in contour mode (dpcon).



Viewport 3
Contour levels: 4
Spacing factor: 2
☑ Positive contour
☑ Negative contour
☑ Multi color contours

Color by viewport not selected

Viewport 3
Contour levels: 4
Spacing factor: 2
☑ Positive contour blue 🟦
☑ Negative contour red 🟥

Color by viewport selected

Figure 14 Contour Controls

The Contour panel has the following controls:

Control	**Function**
Contour levels	Enter a number of contours between 4 and 32 in the text field.
Spacing factor	Enter a number in the text field to specify the spacing between contours. A number between 1.1 and 2 is recommended.
Positive contour	Select this check box to show positive contours using the default color red.
Negative contour	Select this check box to show negative contours using the default color blue.
Color dropdown	Select a color from the menu to use a color other then the default color. Each contour has a color dropdown menu.
Multi color contours	Select this option to use the colors defined in Display Option. If you select the Color by Viewport box,options are not displayed,
AutoScale	Automatically scale the spectrum by clicking.

6.9.7 Using Viewports as a Spectral Interpretation Tool

The Viewports tool displays and presents data and provides a powerful interface to interpret spectral data. A collection of NMR data sets acquired on a given sample are typically more informative when considered together and simultaneously. Information presented in a 1D data set is often complimentary to that provided by a homonuclear or heteronuclear 2D data set, and many 2D data sets are closely linked from an interpretational point of view. Viewports provide powerful options for interrogating spectral data.

6.9.8 Overlaying Homonuclear Data Sets

When interpreting homonuclear data, concurrently consider COSY or scalar coupling interactions (*i.e.*, through-bond) with NOESY or dipolar coupling interactions (*i.e.*, through-space).

For example, Figure 15 shows the graphics window with COSY and NOESY data sets in Viewports 1 and 2, respectively, collected on the ethylindanone standard sample. The Overlay and Align Spectra buttons have been used to provide a composite display. By selecting the Show Crosshair check box, a yellow cursor line, or crosshair, is displayed simultaneously in each window. The crosshairs are linked, allowing responses from one data set to be immediately interpreted with respect to the other. This display clearly differentiates those responses observed in the NOESY spectrum that are derived from long-range relaxation from the vicinal and geminal responses.

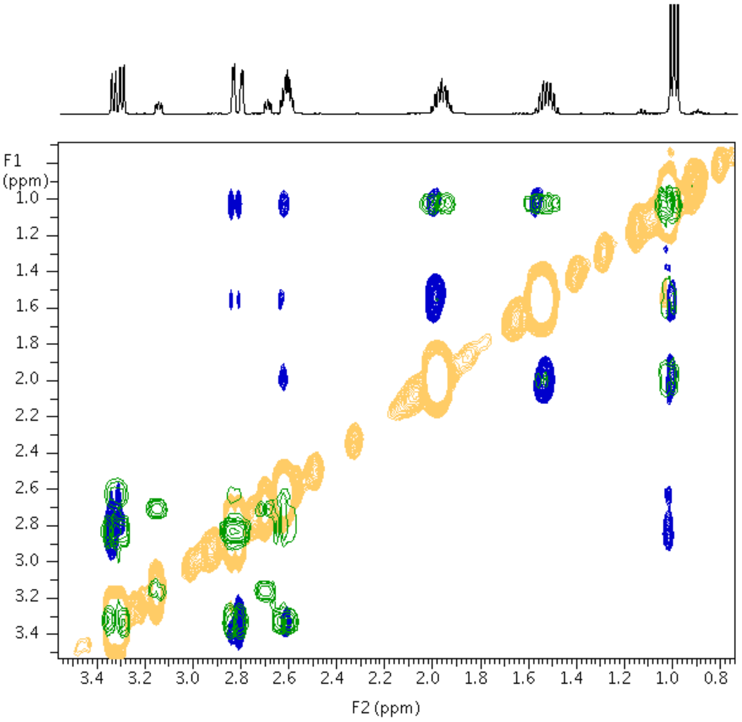


Figure 15 Overlaid COSY and NOESY spectra of ethylindanone.

COSY responses are shown in green, while the phase-sensitive NOESY data are displayed in blue and yellow.

6.9.9 Cross Referencing Heteronuclear Data Sets

Many heteronuclear data sets provide complimentary insights into molecular structure. As shown in Figure 16, a Viewports overlay of HSQC and HMBC data allows visualization of both 1-bond and long-range coupling in a single, easy- to -interpret arrangement. This type of display is very valuable when spectra become congested, and allows unambiguous assignment of long-range responses to 1-bond J_{CH} spin pairs.

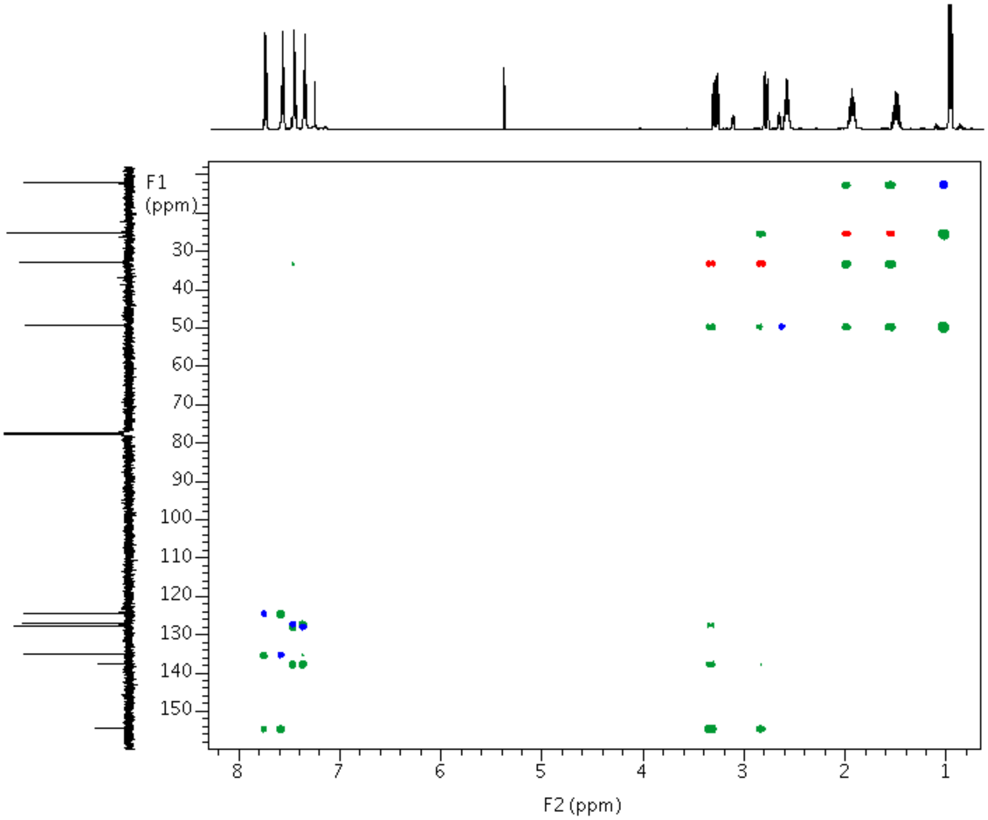


Figure 16 Overlaid HSQC and HMBC spectra of ethylindanone

HMBC responses are shown in green, while the phase-sensitive multiplicity-edited HSQC data are displayed in blue (odd number of protons) and red (even number of protons).

Chapter 7 Printing, Plotting, and Data Output

Printing and plotting of data are highly individualized activities. Each user has specific ideas about proper formats, necessary expansions, and so on.

Sections in this chapter:

- 7.1 Printing of the Graphics
- 7.2 Plotting
- 7.3 Color Printing and Plotting
- 7.4 Sending a Plot via email
- 7.5 Pasting text into a Text Editor or another Application
- 7.6 Advanced Printing Commands

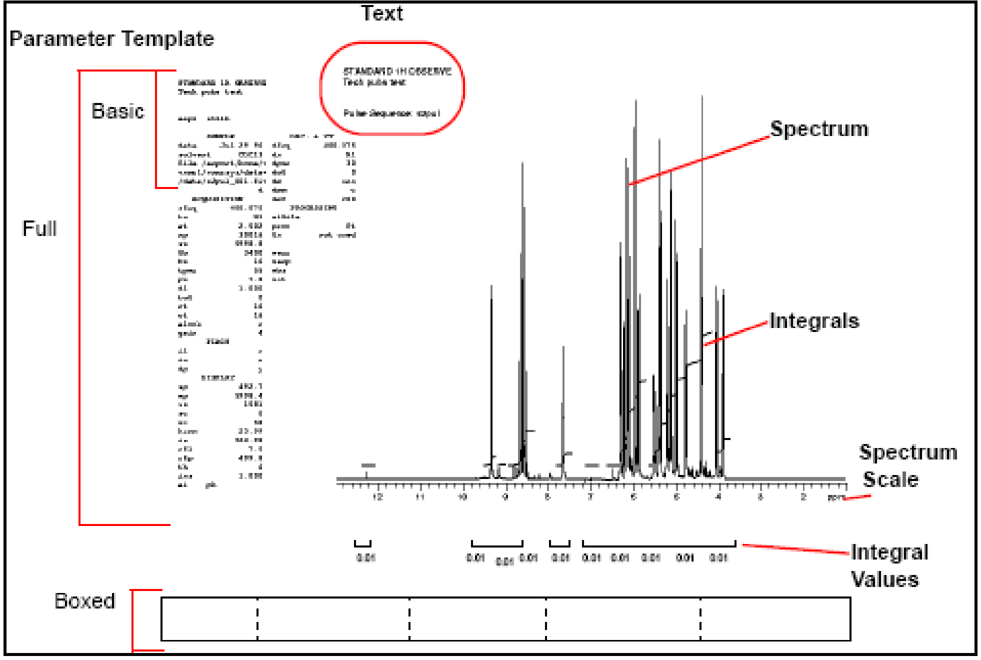


Figure 17 Printout Example

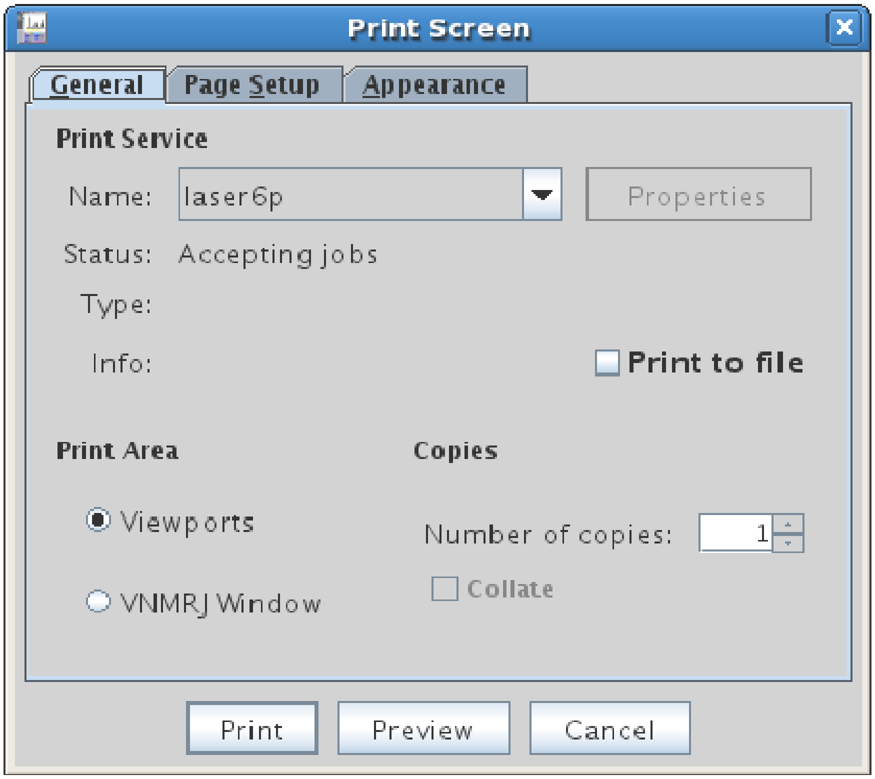
7.1 Printing of the Graphics

After processing, the 1D or 2D spectrum is displayed in the graphics canvas so that the scale, expansion, and threshold can be adjusted.

To print the spectrum:

1. Click **File** from the Menu.
2. Select **Print Screen…**

   The pop-up in Figure 18 appears.
3. Select the **Name** of the printer.
4. Select **ViewPorts** to print the Graphics area.
5. Choose the **number of copies** to print.
6. Click the **Print** button.



Print Service

Name: laser6p

Status: Accepting jobs

Type:

Info:

☐ Print to file

Print Area

● Viewports

○ VNMRJ Window

Copies

Number of copies: 1

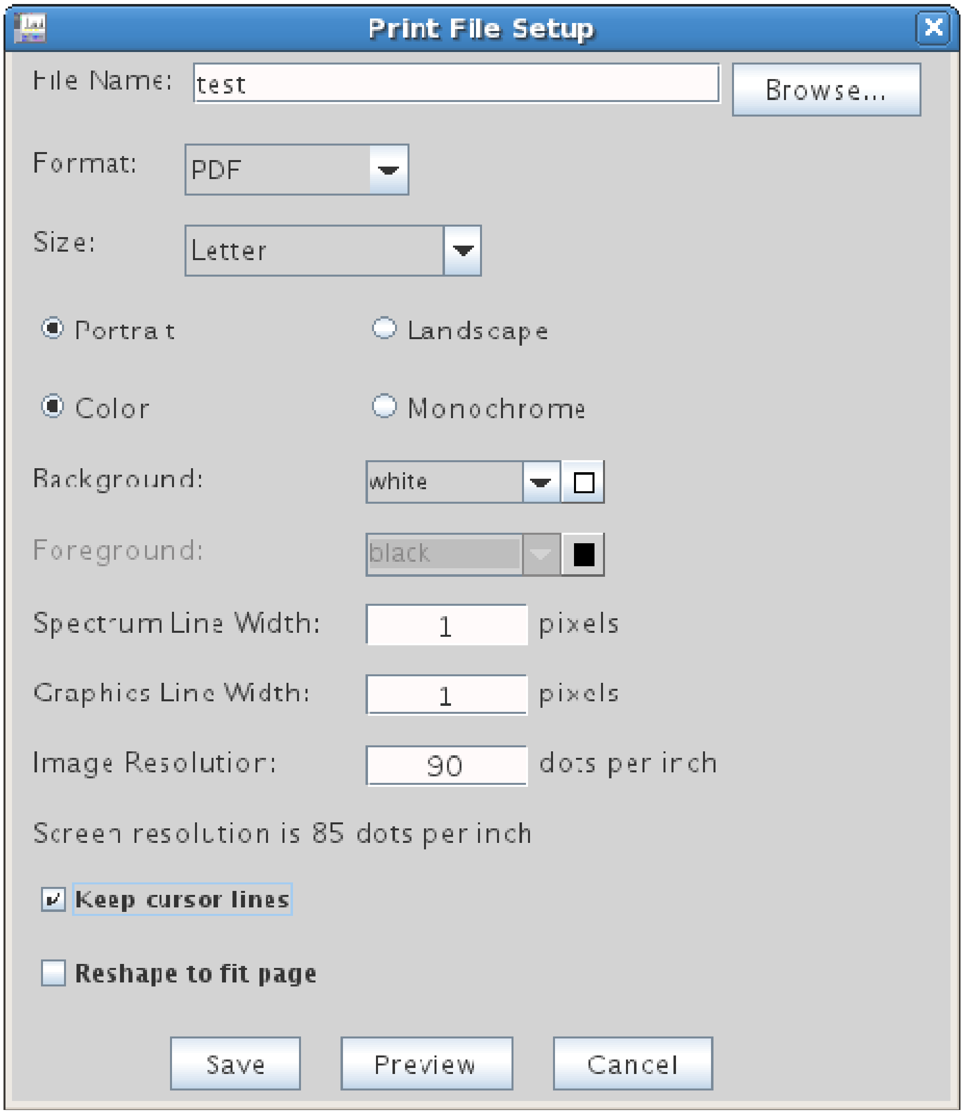
☐ Collate

Figure 18 General tab of the Print Graphics pop-up

NOTE: The VnmrJ interface can be printed by selecting the VNMRJ Window option.

To print a file:

1. Select the **Print to file** check box.
2. Figure 19 shows the pop-up that appears when the **Print to file** check box is selected and the **Print** button is clicked.



File Name: test

Format: PDF

Size: Letter

● Portrait ○ Landscape

● Color ○ Monochrome

Background: white ☐

Foreground: black ■

Spectrum Line Width: 1 pixels

Graphics Line Width: 1 pixels

Image Resolution: 90 dots per inch

Screen resolution is 85 dots per inch

☑ Keep cursor lines

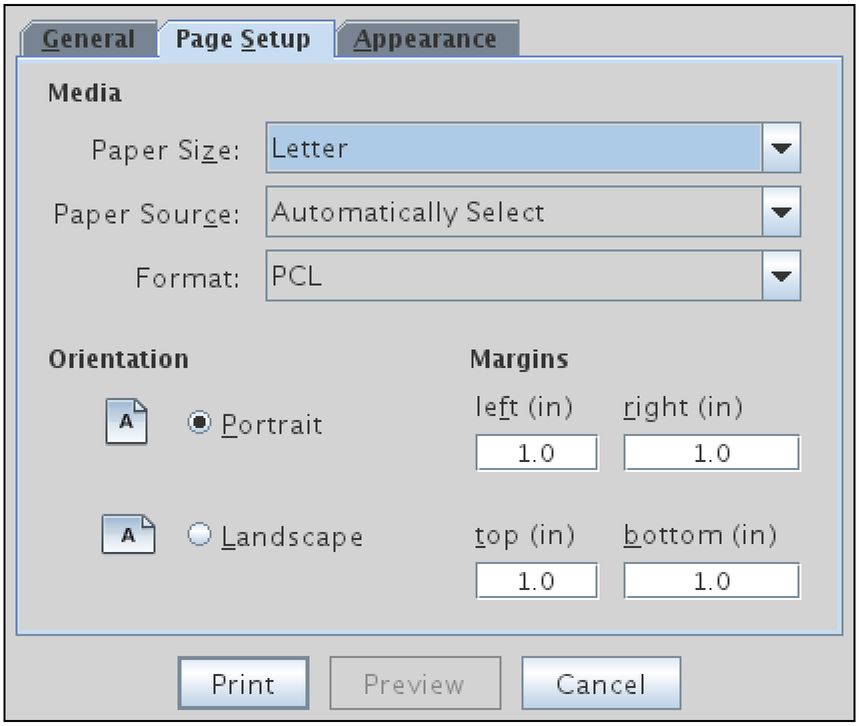
☐ Reshape to fit page

Figure 19 The Print File Setup

Item	**Description**
Sync cursor	Select this checkbox to link and synchronize the cursors and cross hairs in multiple viewports.
Sync Axis	Select this checkbox to link and synchronize axes in multiple viewports. Axis is synchronized to the current active viewport.
File Name	Name of the File to be saved.
Browse	Brose a folder to set the location of where the file will be saved.
Format	A dropdown menu choices of various outputs.
Size	Dropdown menu choices for paper size.

Item	**Description**
Orientation	Select Portrait or Landscape.
Appearance	Use to select Color of Monochrome output.
Spectrum Line Width	Enter a line width in pixels.
Graphics Line Width	Enter a line width in pixels.
Image Resolution	Enter the desired image resolution in dots per inch (dpi).
Screen Resolution	Displays the current screen resolution in dots per inch (dpi).
Keep Cursor Lines	Select to include cursor lines as they appear in the display.
Reshape graphics to fit paper	Select to resize graphics to fit paper size.
Save	Saves output to File name.
Preview	Starts Adobe Acrobat Reader and displays (without saving) output.
Cancel	Closes Print to File pop-up without saving.

The Page Setup and Appearance tabs allow for settings similar to the Print to File setup to be applied to the printed output. Additionally, entries are available for paper source, margins, and print quality (Draft, Normal, and High) while finer control of image resolution and preview is unavailable.



Media

Paper Size: Letter

Paper Source: Automatically Select

Format: PCL

Orientation

● Portrait

○ Landscape

Margins

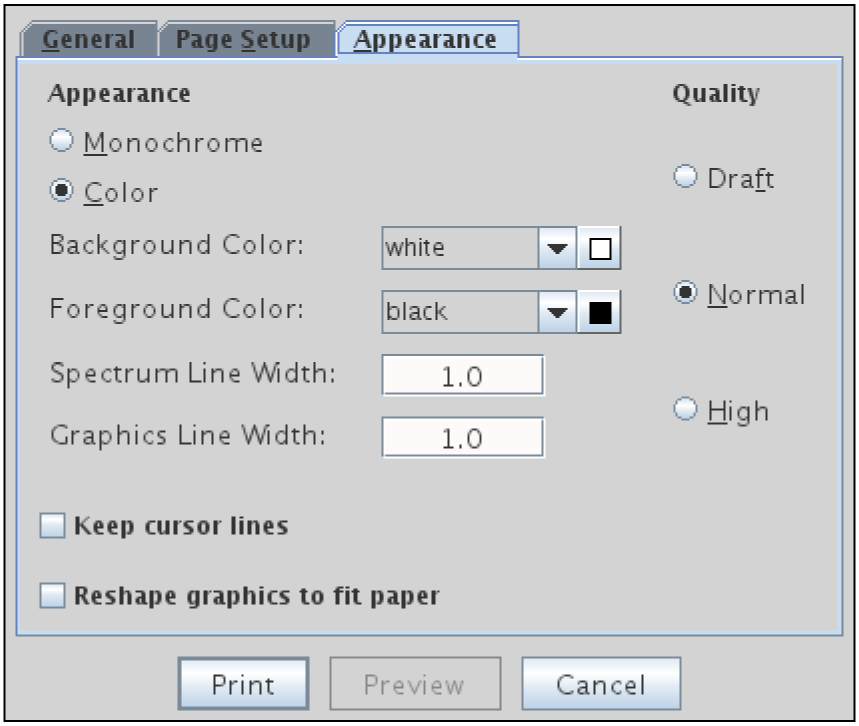
left (in) 1.0

right (in) 1.0

top (in) 1.0

bottom (in) 1.0

Figure 20 The Page Setup tab of the Print Screen pop-up



Appearance

○ Monochrome

● Color

Background Color: white

Foreground Color: black

Spectrum Line Width: 1.0

Graphics Line Width: 1.0

☐ **Keep cursor lines**

☐ **Reshape graphics to fit paper**

Quality

○ Draft

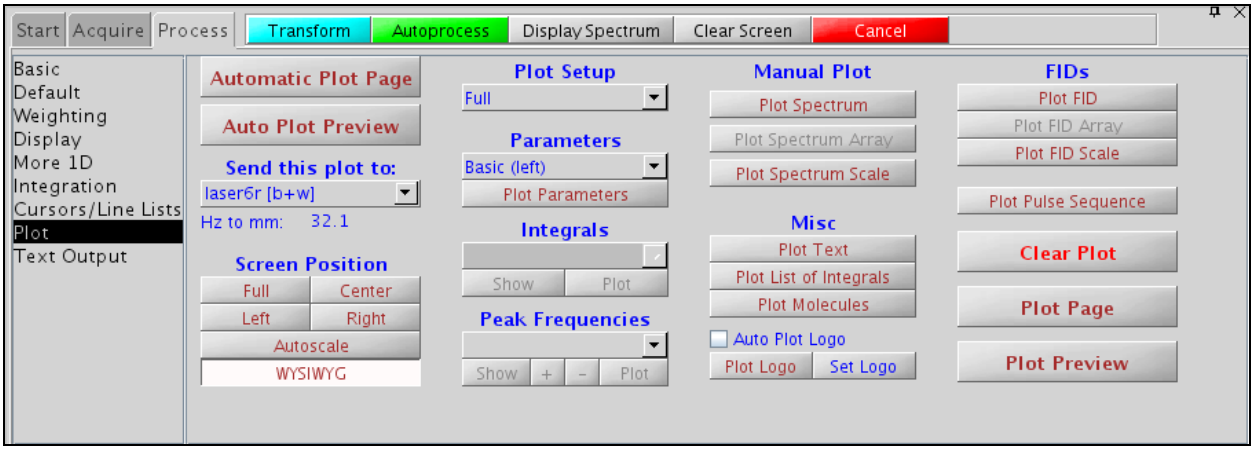
● Normal

○ High

Figure 21 The Appearance Setup tab of the Print Screen pop-up

7.2 Plotting

Plotting is based around the concept of a plot file. Set up and submit a plot using the selections on the Plot Parameter page under the Process tab after the spectrum or FID is displayed. Items selected on the Plot Parameter page (Figure 22) are added to a temporary plot file, and the Plot Page button submits the plot file to the plotter. The Clear Plot button removes the plot file. The Plot Preview button starts Adobe Reader and displays the output of the plot file. Some of the menu options in Adobe Reader are available for use. The PlotView pop-up also appears during a preview, which allows the saving of the view to a pdf format file, sending the output to the default plotter, a file and to one or more e-mail addresses.



Send this plot to: laser6r [b+w]

Hz to mm: 32.1

Plot Setup: Full

Parameters: Basic (left)

Figure 22 Plot Parameter page for a 1D data set

To Plot:	**Select:**	**Click:**
Pulse sequence		Plot Pulse Sequence
FID		Plot FID, Plot Page
FID and scale		Plot FID, Plot FID Scale, Plot Page
Spectrum		Plot Spectrum, Plot Page
Spectrum and scale		Plot Spectrum, Plot Spectrum Scale, Plot Page
Spectrum, scale, and text		Plot Text, Plot Spectrum, Plot Spectrum Scale, Plot Page
Spectrum, scale, and parameters	Parameter Template option	Plot Spectrum, Plot Spectrum Scale, Plot Page
Spectrum, scale, and peak frequencies	Peak Frequencies option	Plot Spectrum, Plot Spectrum Scale, Plot Page
Spectrum, scale, and integrals		Plot Spectrum, Plot Spectrum Scale, Integrals Plot, Plot Page

To Plot:	Select:	Click:
Spectrum, scale, and integrals, integral values	Integrals option	Plot Spectrum, Plot Spectrum Scale, Integrals Plot, Plot Page
Parameters only	Parameter Template option	Plot Page
Text only		Plot Text, Plot Page
Peak frequencies only	Peak Frequencies option	Plot Page
Integrals only	Integrals option	Integrals Plot, Plot Page
Scaled integral values only	Integrals option	Integrals Plot, Plot Page
Normalized integral values only	Integrals option	Integrals Plot, Plot Page
Molecules only		Plot Molecules, Plot Page
Logo only		Plot Logo, Plot Page
Using default settings to the printer		Automatic Plot Page
Using default settings to Adobe Reader for preview		Auto Plot Preview

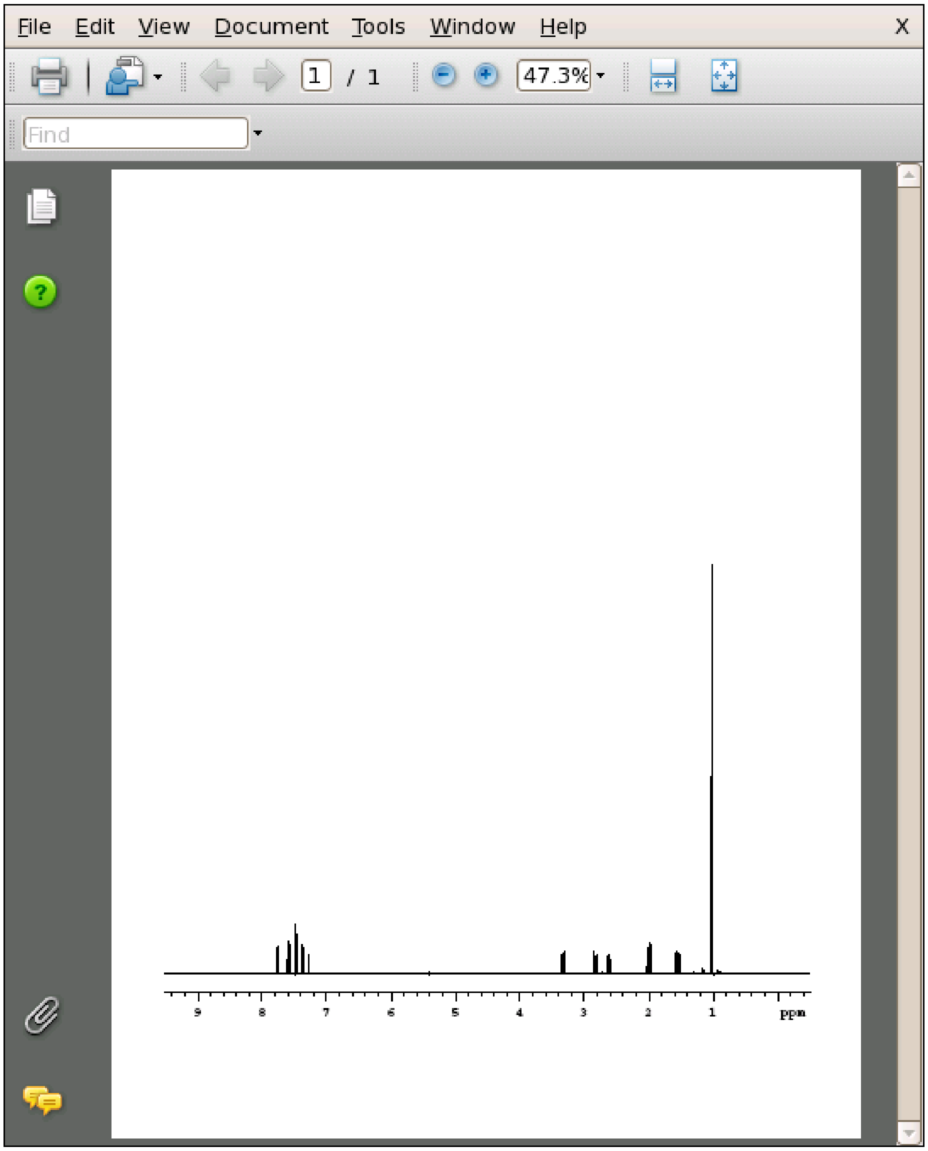
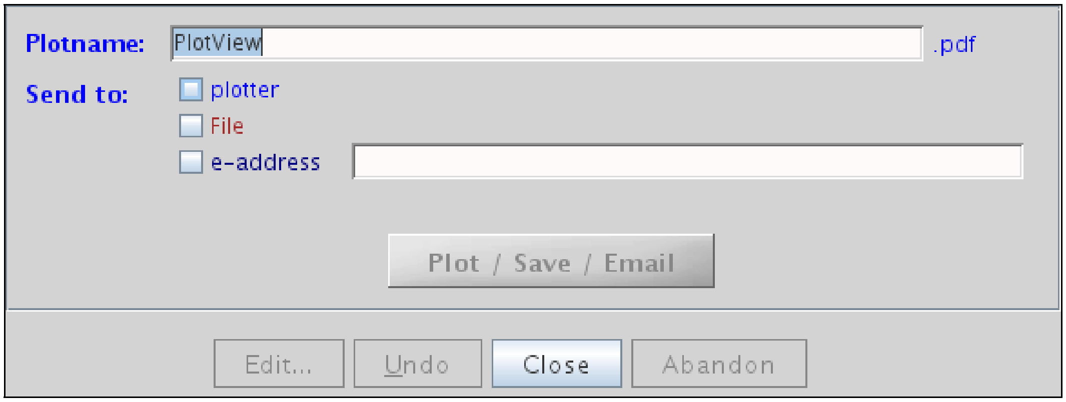


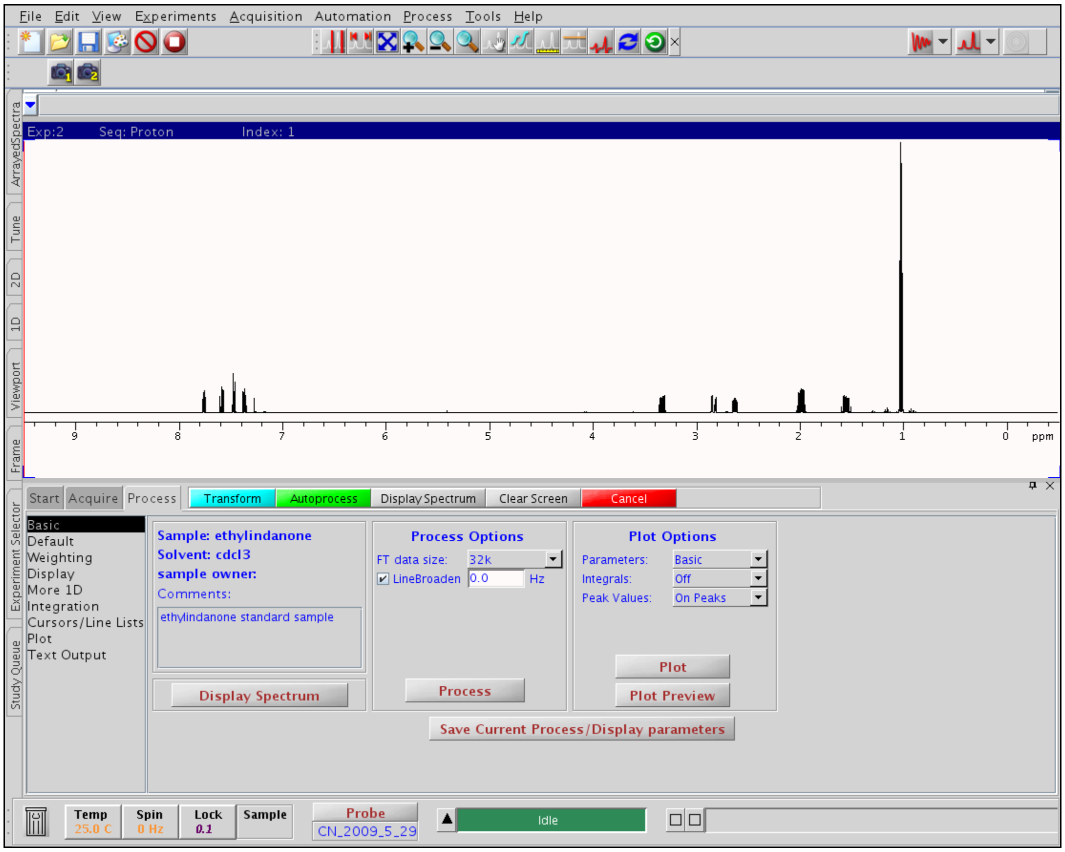
Figure 23 Preview of the plot file initiated from pressing the Plot Preview button


Plotname: PlotView .pdf

Send to:
☐ plotter
☐ File
☐ e-address

Figure 24 The PlotView pop-up

The Basic Parameter page (Figure 25) contains a Plot and a Plot Preview buttons; both have the same function as the Automatic Plot Page and Auto Plot Preview buttons found on the Plot Parameter page.



Exp:2 Seq: Proton Index: 1

Sample: ethylindanone
Solvent: cdcl3
sample owner:
Comments:
ethylindanone standard sample

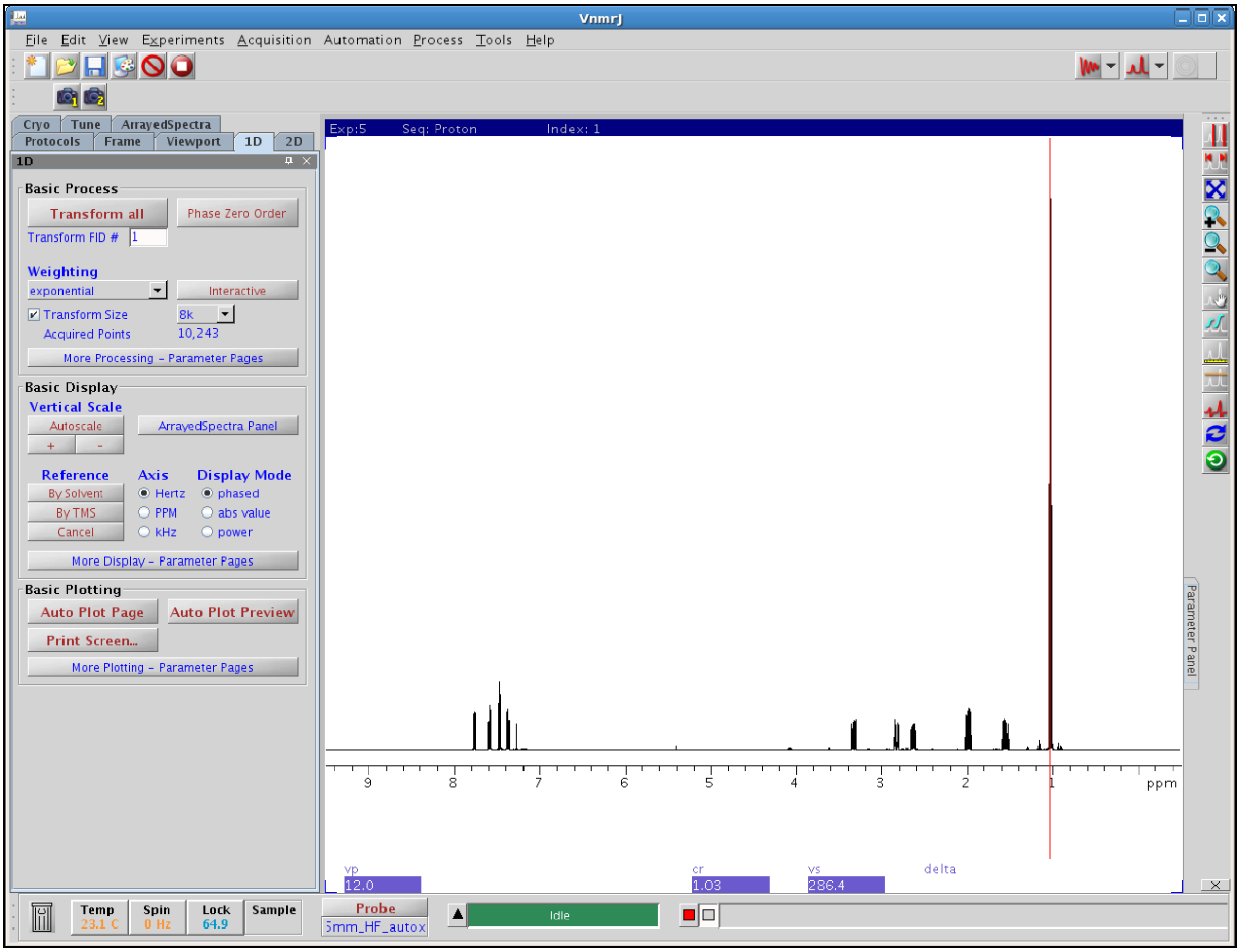
FT data size: 32k
☑ LineBroaden 0.0 Hz

Parameters: Basic
Integrals: Off
Peak Values: On Peaks

Temp 25.0 C
Spin 0 Hz
Lock 0.1
Probe CN_2009_5_29

Figure 25 The Basic Parameter Page for a 1D data set

The 1D Vertical Parameter page contains three buttons to direct the output plot, to a Printer (Auto Plot Page), to Adobe Reader for previewing (Auto Plot Preview), and to call up the Print Screen window, which is identical in function as selecting File from the menu and then selecting Print Screen…



Exp:5 Seq: Proton Index: 1

Transform FID # 1

Weighting: exponential

☑ Transform Size 8k

Acquired Points 10,243

Axis: ● Hertz ○ PPM ○ kHz

Display Mode: ● phased ○ abs value ○ power

vp 12.0

cr 1.03

vs 286.4

Temp 23.1 C

Spin 0 Hz

Lock 64.9

Sample

Probe 5mm_HF_autox

Figure 26 The 1D Vertical Parameter page for a 1D data set

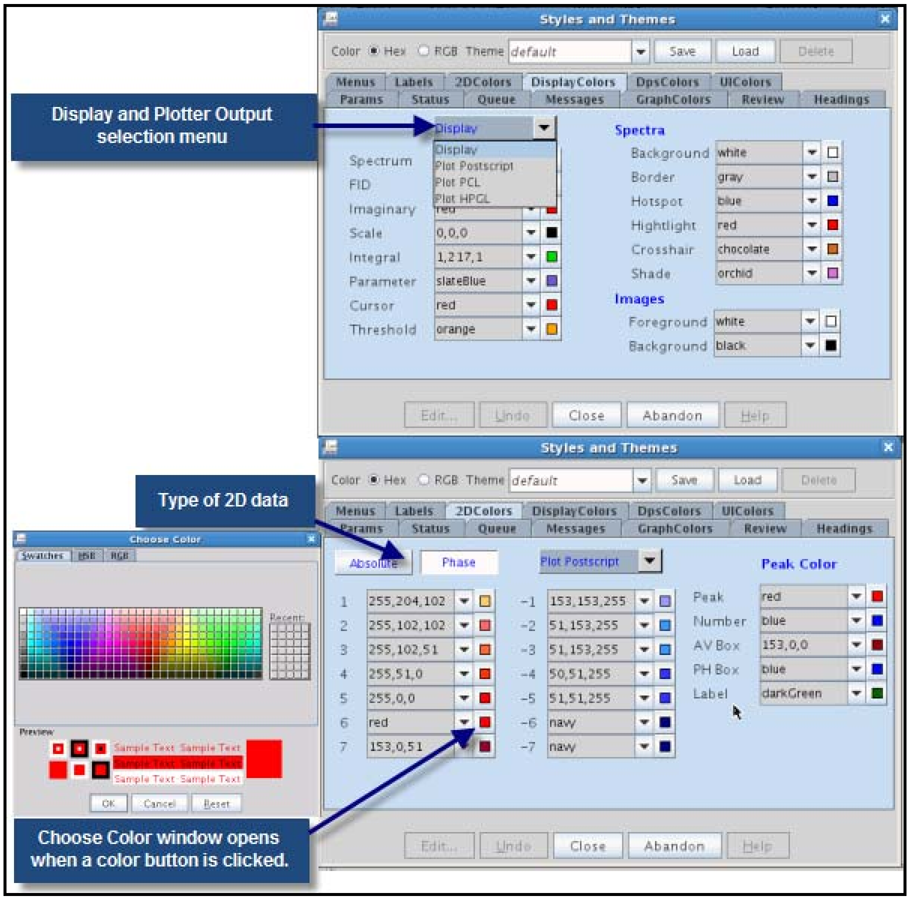
7.3 Color Printing and Plotting

Printer and Plotter color output is defined using the Styles and Themes window, which provides access to the display colors and the VnmrJ interface colors.

7.3.1 Setting Colors

1. View the current settings or define new color settings as follows:
2. Click **Edit**.
3. Select **Display options…**

   The Style and Themes window opens, Figure 27.



Spectrum
FID
Imaginary red
Scale 0,0,0
Integral 1,217,1
Parameter slateBlue
Cursor red
Threshold orange

Spectra
Background white
Border gray
Hotspot blue
Hightlight red
Crosshair chocolate
Shade orchid

Images
Foreground white
Background black

Plot Postscript

1	255,204,102	-1	153,153,255
2	255,102,102	-2	51,153,255
3	255,102,51	-3	51,153,255
4	255,51,0	-4	50,51,255
5	255,0,0	-5	51,51,255
6	red	-6	navy
7	153,0,51	-7	navy

Peak Color
Peak red
Number blue
AV Box 153,0,0
PH Box blue
Label darkGreen

Figure 27 Styles and Themes for Display and 2D Colors

4. Select **Display** tab to set the colors for the spectra, axis, parameters, etc.
5. Click **Display and Output** selection drop down menu.
6. Select the output device: Display, Plot Postscript, Plot PCL, or Plot HPGL.
7. Select a color or keep the current color for each display or function shown.
8. Enter a **name** in the field next to the Save button to save the selection to a user defined file or continue with the next step to overwrite the current file.
9. Click **Save** to save the color selections to the specified file.
10. Click **2D colors**.
11. Click **Phase** to set the colors for this 2D display mode.
12. Select the output device: Display, Plot Postscript, Plot PCL, or Plot HPGL.
13. Select a **color** or keep the current color for each contour level.
14. Click **Absolute** to set the color for this 2D display mode.
15. Select a **color** or keep the current color for each contour level.
16. Enter a **name** in the field next to the Save button to save the selection to a user defined file or continue with the next step to overwrite the current file.
17. Click **Save** to save the color selections to the specified file.

7.3.2 Loading a Color File

To retrieve a color file:

1. Select a theme file from the T**heme** drop-down menu.
2. Click **Load**.

7.3.3 Changing or Renaming a Color File

To change the colors in a file:

1. Select a theme file from the T**heme** drop-down menu.
2. Click **Load**.
3. Follow procedure in Setting Colors 7.3.1.
4. Click **Save** to save the file.

To change the name of a color file:

1. Select a theme file from the Theme drop-down menu.
2. Click **Load**.
3. Enter a **new name** in the field next to the Save button.
4. Click **Save** to save the file.

Optional: To delete the file with the old name, 7.3.4.Removing a Color File.

7.3.4 Removing a Color File

To remove a color file from the list:

1. Select a **theme file** from the Theme drop-down menu.
2. Click **Load**.
3. Click **Delete**. The deleted file is removed from the bottom list box.
4. Click **OK**, when prompted, to delete the file, or **Cancel** to keep the file.

7.3.5 Closing the Color Selection Window

1. Click **Close** to exit the window.

7.4 Sending a Plot via email

After selecting the options to and clicking the Plot Preview button in the Plot Parameter page.

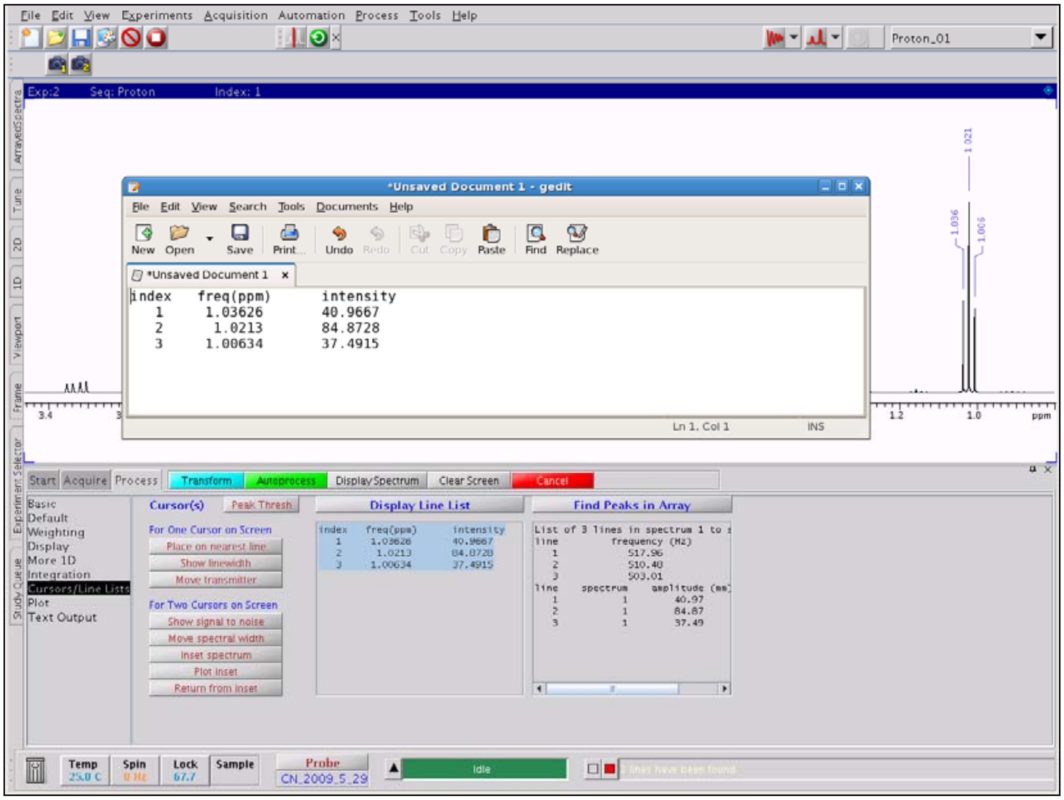
1. Enter a **Plot name** in the PlotView pop-up.
2. Check **File**.
3. Check **e-address**.
4. Enter valid email addresses in the entry field of e-address.
5. Press the **Plot / Save / Email** button.
6. Press the **Close** button when done.

7.5 Pasting text into a Text Editor or another Application

Text output which appears in the Integration, Cursors / Line Lists / Text Output Parameter pages can be pasted into a text editor or other application as shown in Figure 28, to be saved or used elsewhere.

1. Highlight the text to be pasted by clicking the left mouse button and dragging the mouse to the end of the desired text.

   Release the mouse button at the end of the desired text. The selected text is highlighted indicating what has been selected.
2. Start the text editor or application and place the mouse cursor on the active document.
3. Click the middle mouse button to paste the highlighted text into the text editor.



```
index    freq(ppm)        intensity
   1      1.03626         40.9667
   2       1.0213         84.8728
   3      1.00634         37.4915
```

```
List of 3 lines in spectrum 1 to
line          frequency (Hz)
  1              517.96
  2              510.40
  3              503.01
line     spectrum     amplitude (mm
  1          1           40.97
  2          1           84.87
  3          1           37.49
```

Figure 28 Contents of the Display Line List text box copied into the Linux gedit text editor

7.6 Advanced Printing Commands

Printing from within VnmrJ is initiated with the `printon` command. All output which normally appears in the text output window is saved and, when the `printoff` command is issued, sent to a printer. This output includes the following:

- Parameter listings from `dg, dgl, da, etc.`
- Line listings from `dll`.
- Integral listings from `dli`.
- System configuration parameters generated by `config('display')`.

- Text files using the `text` command .
- Results of calculations from h2cal, adept, tl, t2, etc.
- Any other information that some program or macro may write to the text window.

This output is saved in a temporary file in /vnmr/tmp. The VnmrJ parameter **`printer`** determines the printer to which the output is directed. When the `printoff` command is issued, VnmrJ executes a UNIX script called `vnmrprint` that sends the temporary file to the printer using standard UNIX printing utilities. This script is supplied with the name of the temporary file to be printed, the name of the printer (corresponding to a **`printcap`** entry), and the type of printer (corresponding to an entry in /vnmr/devicetable. If desired, the script vnmrprint affords a place for user customizing.

NOTE: Information in /vnmr/devicetable is used to distinguish PostScript printers.

The macro ptext prints out the text file given as an argument. For example, the command `ptext`('/vnmr/psglib/DEPT. c') prints the text file **DEPT.c**.

Print jobs for the currently active printer in VnmrJ are held in a print queue. The showprintq macro displays the current print jobs in the print queue. The killprint macro will stop a print job and remove it from the print queue. Unless the user executing this macro is root (superuser), only that user's print job is deleted from the print queue.

Table 6. Printer-Associated Commands and Parameters

Commands	**Parameter**
`killprint` `printoff«'clear'lfile»`	Stop print job and remove from print queue
`printon`	Stop sending text to printer & start print operation
`ptext (file)`	Direct text output to printer Print out a text file
`showplotter`	Display currently defined plotters and printers
`showprintq`	Display print jobs in print queue
`vnmrprint*`	Print text files (UNIX)
`* vnmrprint`	
`printer` {string}	Printer device

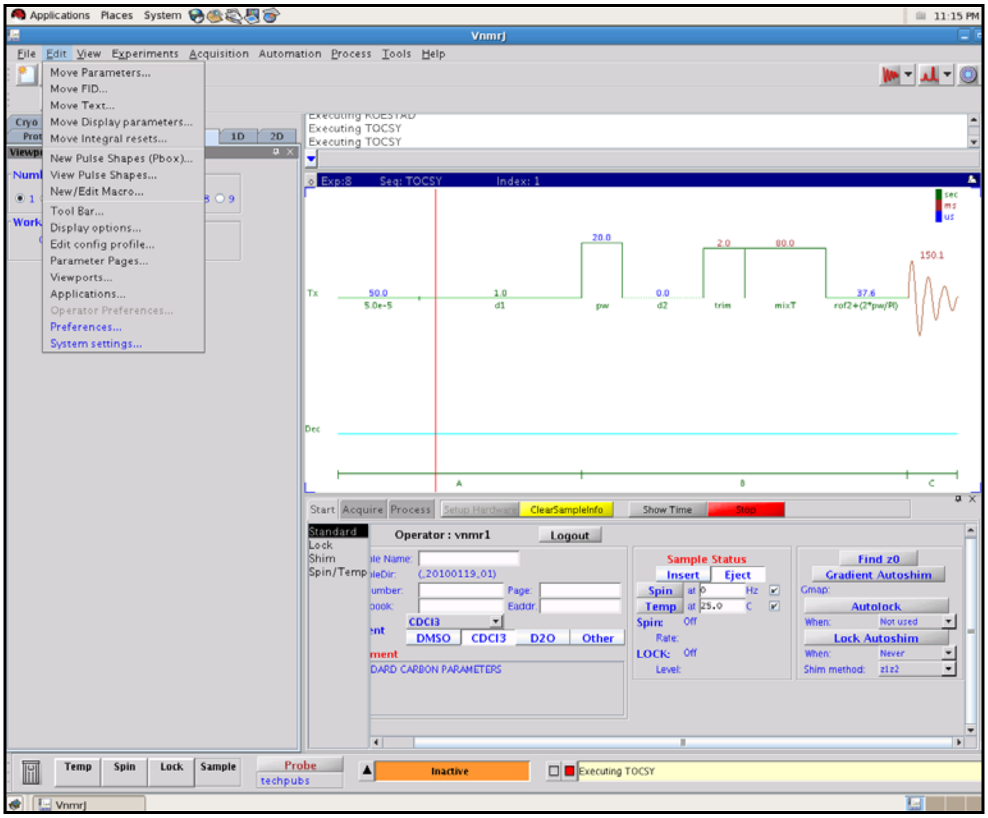
Chapter 8 VnmrJ 3 Preferences

The default behavior of VnmrJ at the user account level has several customizable options. These options are different from those that can be accessed by the administrator of VnmrJ. The optimal operation of these customization options requires a properly setup probe file for the probe in use.

Select the Preferences from the Edit menu on the main VnmrJ window to access the VnmrJ preferences setup window. Note that some of the options appear only if the Enable Email Options check box has been selected. Ensure that an email server has been properly configured at the host computer.

Sections in this chapter include:

- 8.1 Templates Tab
- 8.2 Automation Tab
- 8.3 Queue Tab
- 8.4 eOptions Tab
- 8.5 Data Mirror Tab
- 8.6 SampleTags Tab
- 8.7 UserPrefs Tab

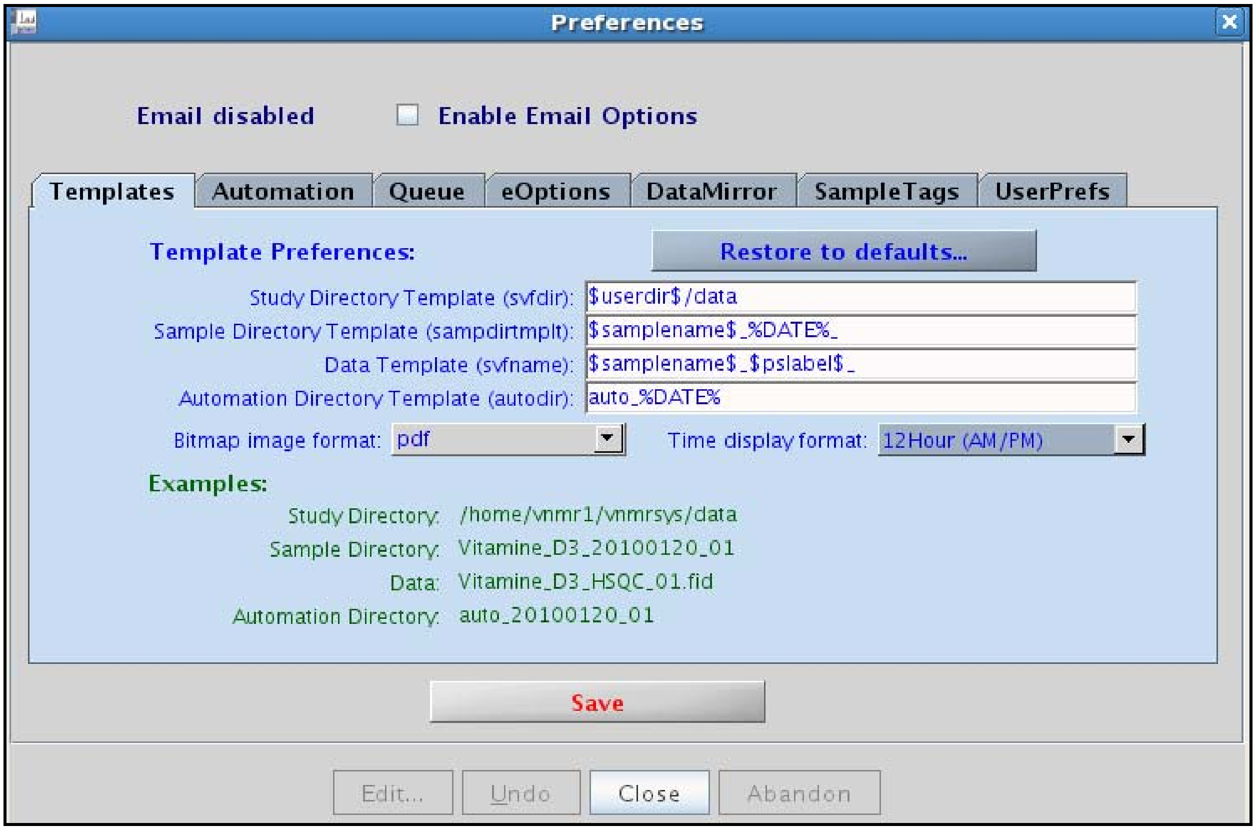


```
Executing ROESYAD
Executing TOCSY
Executing TOCSY
```

le Name:
leDir: (_20100119_01)
umber: Page:
ook: Eaddr:
CDCl3
Spin: at 0 Hz ☑
Temp: at 25.0 C ☑
Spin: Off
Rate:
LOCK: Off
Level:
Gmap:
When: Not used
When: Never
Shim method: z1z2
DARD CARBON PARAMETERS
Executing TOCSY

8.1 Templates Tab

Any VnmrJ parameter in the Templates tab can be used to select where the data is saved and how data is to be named.



Email disabled ☐ Enable Email Options

Template Preferences:

Study Directory Template (svfdir): $userdir$/data
Sample Directory Template (sampdirtmplt): $samplename$_%DATE%_
Data Template (svfname): $samplename$_$pslabel$_
Automation Directory Template (autodir): auto_%DATE%
Bitmap image format: pdf
Time display format: 12Hour (AM/PM)

Examples:

Study Directory: /home/vnmr1/vnmrsys/data
Sample Directory: Vitamine_D3_20100120_01
Data: Vitamine_D3_HSQC_01.fid
Automation Directory: auto_20100120_01

Figure 29 Template Tab— Preferences

The following describes the five entries for data saving:

1. Study Directory Template: Defines where the data for all the samples is stored. It defines the parameter **`svfdir`**. Refer to Format of the Entry Level, for the exact format of this entry.
2. Sample Directory Template: Defines the directory inside the study directory where all the data for any sample are stored. It defines the parameter **`sampdirtmplt`**. This is always a subdirectory for the study directory, svfdir. Refer to Format of the Entry Level, for the exact format of this entry.
3. Data Template: Defines the actual name that is given to the individual fid files. It defines the parameter **`svfname`**. This file is saved inside sampdirtmplt. Refer to Format of the Entry Level, for the exact format of this entry.
4. Automation Directory Template: Defines location of the automation directory, where the system stores all the information to do the automation run. It defines the parameter **`autodir`**.
5. Bitmap Image Format: Allows the definition of the format to be used when saving or e-mailing bitmaps of the spectra recorded. The following are available options:
    a. tif. This creates a TIFF format document, typical for accurate representation of bitmap images. A TIFF document can be read with most common image and word processing programs.

    b. pdf. This creates a document according to the Adobe PDF™ document format. A PDF document can be read using Adobe Acrobat Reader. The PDF documents created from VnmrJ require Adobe Reader version 5 or higher.
    c. pcx. This creates a document using the PCX protocol, adequate for representations of bitmap images. PCX documents can be read with most common image and word-processing documents.
    d. jpg. This creates a JPEG format document accurate for the representation of real-life photos. JPEG documents can be read with most common image and word processing programs.

8.1.1 Format of the Entry Level

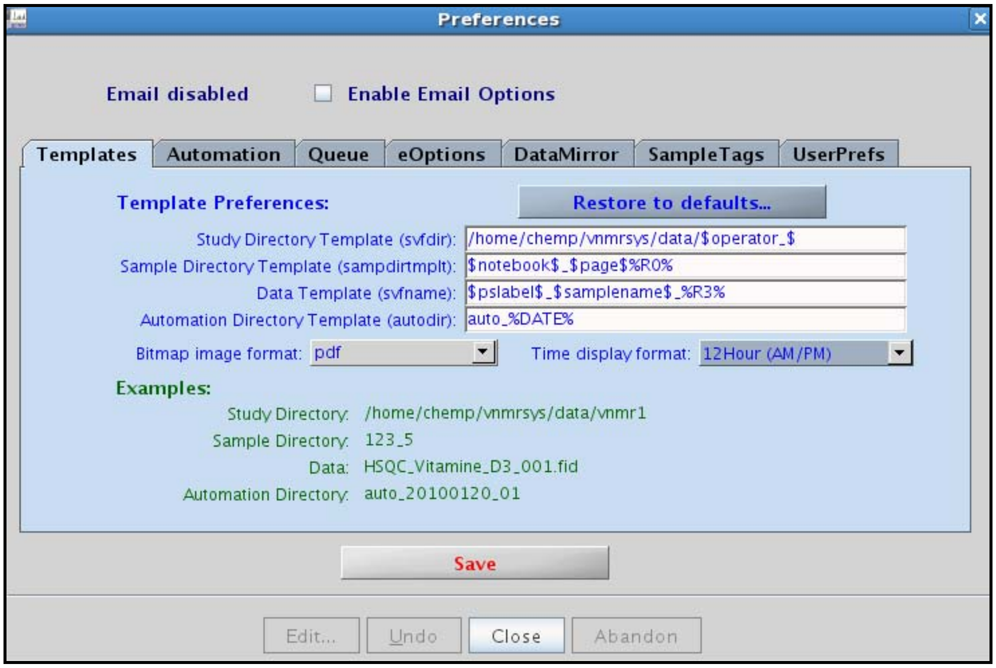
The format used to enter the directories in the first three fields is the same as was used previously for “autodir.” A detailed description of it appears under the description of “autodir” in the *Command and Parameter Reference Manual* for VnmrJ. A brief description is shown here.

There are three types of text that can be entered in these fields. All the options can be mixed together generating very powerful saving options.

1. Fixed text, like “/home/vnmr1/data.” This is interpreted as is without any special translations. If the text defines an absolute directory path then this path will be used (example /vnmr/data). If there is no absolute path then the directory will be created as a subdirectory of the previously defined directory path. So if sample directory is defined as “mysamples/today” then the subdirectory will be created for the study directory.
2. Text enclosed in “$” signs. This will substitute the enclosed text with the value of the VnmrJ parameter with the same name. For example $**`samplename`**$ will be substituted with “mysample” if **`samplename`**=’mysample’. Any VnmrJ parameter can be used for this. The most useful ones will be the ones defining sample parameters (**`studyowner`**, **`samplename`**, **`solvent`**, etc.) and experiment parameters (**`pslabel`** which shows what experiment is run for example). So “$**`samplename`**$/$**`pslabel`**$” will translate into “mysample/PROTON” in the case of a proton spectrum.
3. Text enclosed in “%” signs. This will substitute the enclosed text with the entry on the actual enterQ file being used. This can be very cryptic but some useful options are outlined here. For more detailed description the user is referred to the *Command and Parameter Reference Manual*.
    a. %DATE%: This will be substituted with the date that the spectrum is acquired.
    b. %RX% where X is a number: This will be substituted with a numerical extension in the form “_00”, “_01”, “_02” etc. The number X defines the number of digits that will be used for the extension, so %R2% will generate “_01” while %R3% will generate “_001” etc. This ensures that a unique name is generated for every directory created. A %R2% will be appended automatically to the study, sample and automation directory templates if one is not explicitly defined. One can suppress this by using %R0% however this is not recommended as it may accidentally overwrite data.

Examples shows what each of the strings entered translates to. Some useful examples are displayed below.

The example shown in Figure 30 will direct all study data to home/chemp/vnmrsys/data/<operator name>. This is accomplished by the use of the `$operator_$` argument. Note that the more proper parameter to use in VnmrJ 3 would be `$studyowner$`. The data for each sample will further be saved in a directory named <notebook>_<page>, where notebook and page are the notebook and page numbers that were entered in the Start tab of the parameter panel. The revision number is suppressed here. The actual FIDs will be saved as <experiment>_<samplename> with a three digit revision after it.



Template Preferences:

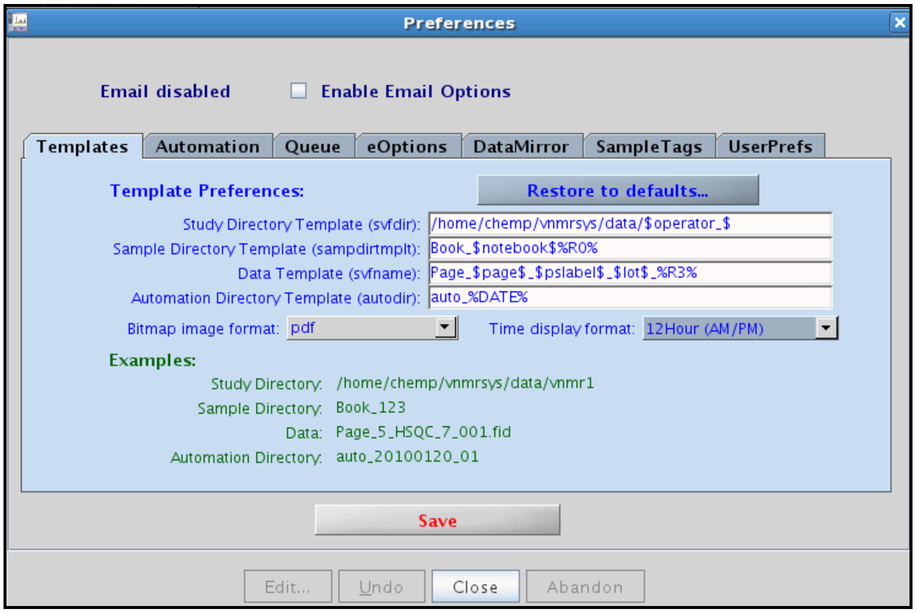
Study Directory Template (svfdir): /home/chemp/vnmrsys/data/$operator_$
Sample Directory Template (sampdirtmplt): $notebook$_$page$%R0%
Data Template (svfname): $pslabel$_$samplename$_%R3%
Automation Directory Template (autodir): auto_%DATE%
Bitmap image format: pdf
Time display format: 12Hour (AM/PM)

Examples:

Study Directory: /home/chemp/vnmrsys/data/vnmr1
Sample Directory: 123_5
Data: HSQC_Vitamine_D3_001.fid
Automation Directory: auto_20100120_01

Figure 30 Setting the Preferences Window

The example shown in Figure 31 illustrates the inclusion of a fixed text in the sample directory name (Book_<notebook>) and dates.

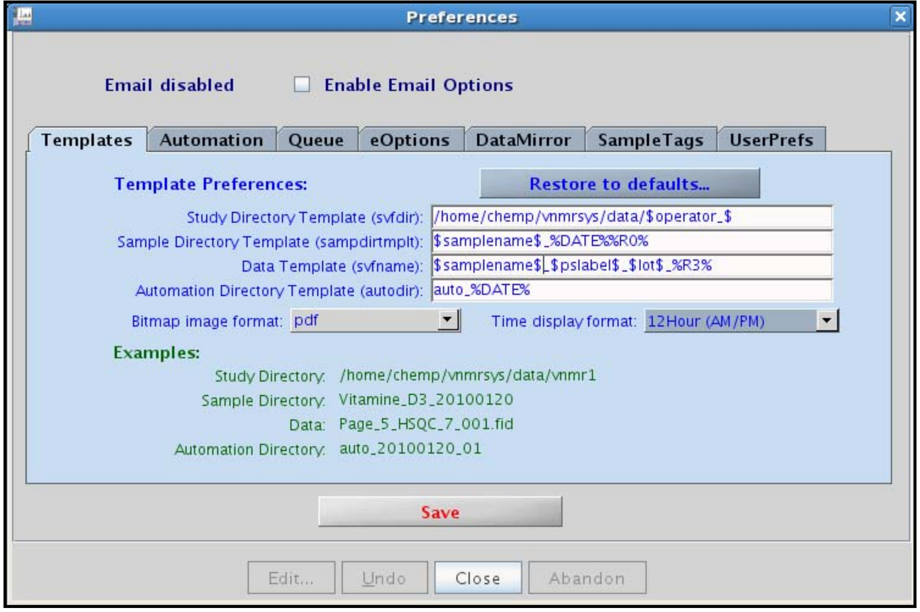


Template Preferences:
Study Directory Template (svfdir): /home/chemp/vnmrsys/data/$operator_$
Sample Directory Template (sampdirtmplt): Book_$notebook$%R0%
Data Template (svfname): Page_$page$_$pslabel$_lot_%R3%
Automation Directory Template (autodir): auto_%DATE%
Bitmap image format: pdf
Time display format: 12Hour (AM/PM)

Examples:
Study Directory: /home/chemp/vnmrsys/data/vnmr1
Sample Directory: Book_123
Data: Page_5_HSQC_7_001.fid
Automation Directory: auto_20100120_01

Figure 31 Example—Inclusion of text in the sample directory

The example in Figure 32 shows the date is part of the directory name.

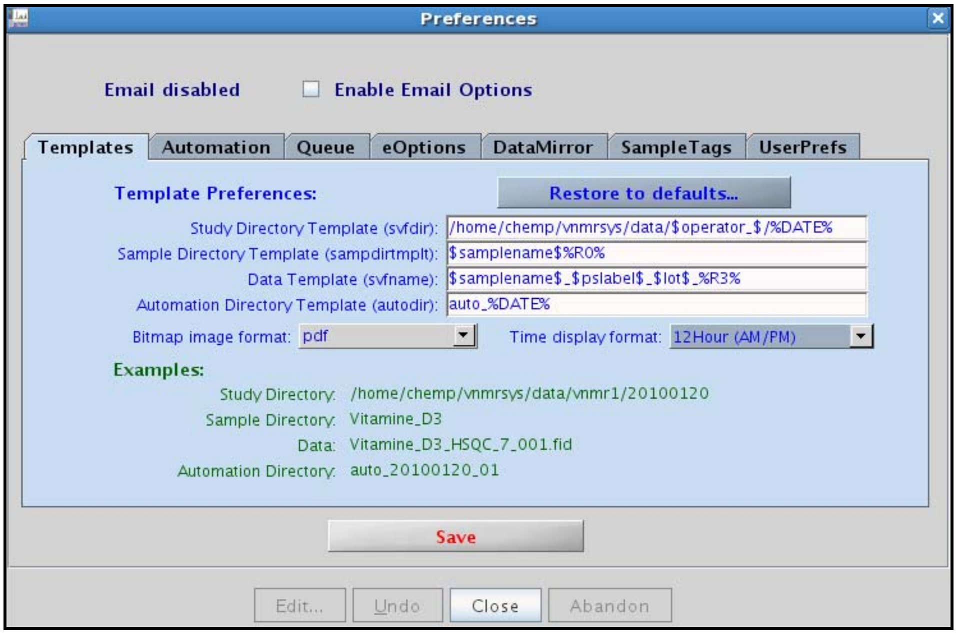


Template Preferences:
Study Directory Template (svfdir): /home/chemp/vnmrsys/data/$operator_$
Sample Directory Template (sampdirtmplt): $samplename$_%DATE%%R0%
Data Template (svfname): $samplename$_$pslabel$_lot_%R3%
Automation Directory Template (autodir): auto_%DATE%
Bitmap image format: pdf
Time display format: 12Hour (AM/PM)

Examples:
Study Directory: /home/chemp/vnmrsys/data/vnmr1
Sample Directory: Vitamine_D3_20100120
Data: Page_5_HSQC_7_001.fid
Automation Directory: auto_20100120_01

Figure 32 Example—Inclusion of the date in the directory name

Data from one operator can be sorted to different subdirectories according to the date the data was recorded.



Email disabled ☐ Enable Email Options

Template Preferences:

Study Directory Template (svfdir): /home/chemp/vnmrsys/data/$operator_$/%DATE%
Sample Directory Template (sampdirtmplt): $samplename$%R0%
Data Template (svfname): $samplename$_$pslabel$_lot_%R3%
Automation Directory Template (autodir): auto_%DATE%
Bitmap image format: pdf
Time display format: 12Hour (AM/PM)

Examples:

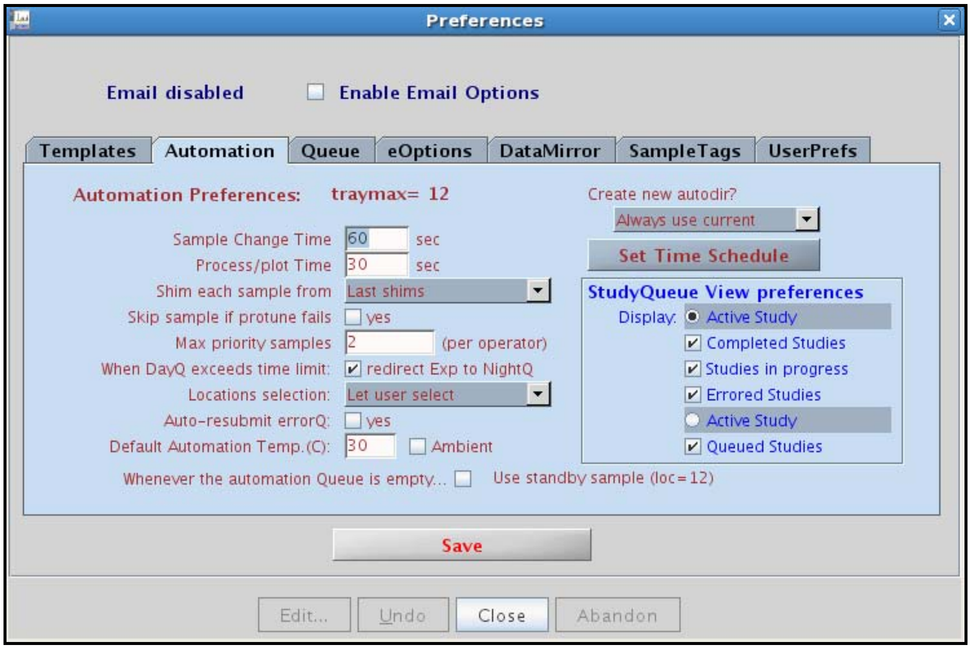
Study Directory: /home/chemp/vnmrsys/data/vnmr1/20100120
Sample Directory: Vitamine_D3
Data: Vitamine_D3_HSQC_7_001.fid
Automation Directory: auto_20100120_01

Figure 33 Example—Inclusion of the date in the study directory template

---

NOTE: Any changes done on this window should be saved before exiting the window; otherwise, changes are lost. The changes usually require the creation of a new automation run to become active.

8.2 Automation Tab



Email disabled ☐ Enable Email Options

Automation Preferences: traymax= 12

Create new autodir? Always use current

Sample Change Time 60 sec
Process/plot Time 30 sec
Shim each sample from Last shims
Skip sample if protune fails ☐ yes
Max priority samples 2 (per operator)
When DayQ exceeds time limit: ☑ redirect Exp to NightQ
Locations selection: Let user select
Auto-resubmit errorQ: ☐ yes
Default Automation Temp.(C): 30 ☐ Ambient
Whenever the automation Queue is empty... ☐ Use standby sample (loc=12)

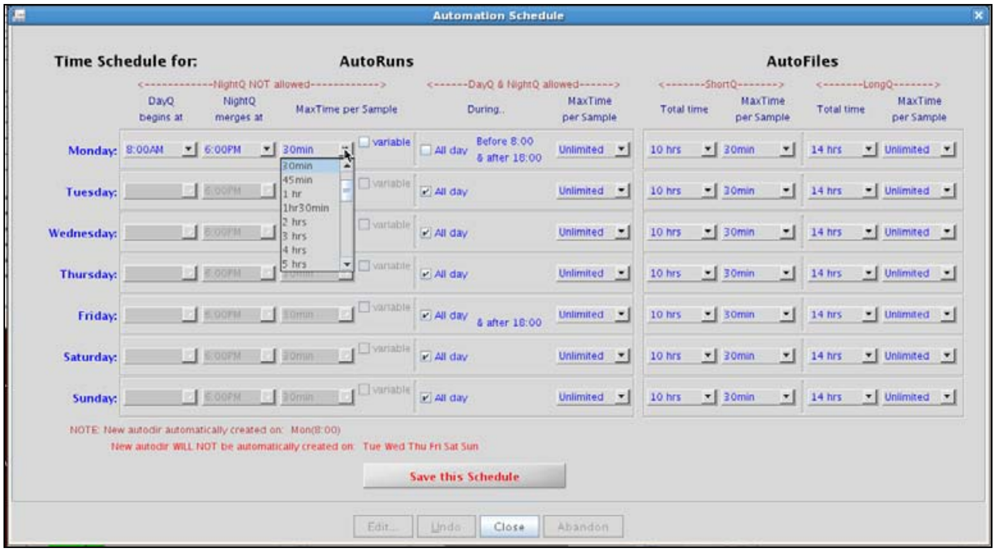
StudyQueue View preferences

Display: ● Active Study
☑ Completed Studies
☑ Studies in progress
☑ Errored Studies
○ Active Study
☑ Queued Studies

The Automation tab defines the defaults that will be used for automation experiments. Defaults are described in the following table.

Field	**Description**
Traymax	This parameter indicates what type of autosampler is actually used. If this is not set up properly then it needs to be changed in the System Preferences window. Only vnmr1 can make this change.
Sample change time	This is the approximate time required for the sample change using the autosampler, the finding of the z0 value for the automatic locking, the automatic tuning (ProTune) and the automatic shimming. The default value is 120 seconds which should be sufficient for most cases. It is generally advised to round the time rounded up, so if the system needs 100 seconds to enter instead 120. This ensures that the time calculations will not fall off because of too short estimates.
Process/plot time	This is the time required to process, plot and save the spectrum. The default value of 10 seconds should be sufficient. Use a longer value only if you are using a slower computer.
Shim each sample from	This defines the shims started by the system when the shim tries to shim a new sample. There are two options: Last shims: Last shims use the shims from the previous sample run Default shims: Default shims load the shim file that is in the probe file. In actual operation with properly prepared sample the outcome should be equivalent. If however that range of samples and tubes used varies widely then the use of Default shims give more consistent results.
Skip sample if Protune fails	This check-box appears only if ProTune is installed. If checked then the system skips the sample in the unlikely event that the automatic tuning fails. When the check box is not selected, the data acquisition proceeds.
Maximum priority samples	This defines the maximum priority samples an operator can submit in each automation run. The right to have priority samples is granted from the VnmrJ administrative interface.
When DayQ exceeds time limit	Select redirect Exp to NightQ if the DayQ has exceeded it’s time.
Locations selection	This drop-down menu defines whether users will be allowed to choose the sample location manually (Let User Select option) or not (Next Available option). The Next Available option will ignore the user input and submit the experiment queue for the next available sample position in the autosampler.
Auto-resubmit errorQ	Select yes to automatically have the software resubmit samples back into the queue if it has errorred.
Edit/resubmit option	This option either allows or not the right to edit and resubmit a study queue that has already been submitted.
Default Automation	Defines the temperature default for all samples run in

Field	Description
Temp. (C)	automation. If at the time New Automation run is started, temperature control is enabled in the account, a new temperature can be specified at customization time. The software checks if the chosen temperature is safe given the choice of solvent for that sample. In addition, if variable temperature is allowed in sample changer automation, it is important to set the value for the **`tin`** (temperature interlock) parameter contained in ~/vnmrsys/modules/cpQdefaults be set to “w” so that the sample changer will not insert any sample until the probe has reached the requested temperature.
Whenever the automation queue is empty	If you select this checkbox, a standby sample is inserted into the magnet whenever the automation run finishes and there are no more samples in the queue. This removes the last sample run from the magnet thus allowing its owner to retrieve it from the autosampler tray. The standby sample needs to be placed in the position indicated in the field, position 96 in the case of the figure above. That particular sample location is not available for use.
Set Time Schedule	Opens a tool for defining how time is managed in automation
StudyQueue View preferences	Displays options for what is displayed in the Study Queue when the Spectrometer view is selected.



Time Schedule for: AutoRuns AutoFiles

	<------------NightQ NOT allowed------------>				<------DayQ & NightQ allowed------>		<-------ShortQ------->		<-------LongQ------->	
	DayQ begins at	NightQ merges at	MaxTime per Sample		During..	MaxTime per Sample	Total time	MaxTime per Sample	Total time	MaxTime per Sample
Monday:	8:00AM	6:00PM	30min	☐ variable	☐ All day Before 8:00 & after 18:00	Unlimited	10 hrs	30min	14 hrs	Unlimited
Tuesday:		6:00PM		☐ variable	☑ All day	Unlimited	10 hrs	30min	14 hrs	Unlimited
Wednesday:		6:00PM		☐ variable	☑ All day	Unlimited	10 hrs	30min	14 hrs	Unlimited
Thursday:		6:00PM	30min	☐ variable	☑ All day	Unlimited	10 hrs	30min	14 hrs	Unlimited
Friday:		6:00PM	30min	☐ variable	☑ All day & after 18:00	Unlimited	10 hrs	30min	14 hrs	Unlimited
Saturday:		6:00PM	30min	☐ variable	☑ All day	Unlimited	10 hrs	30min	14 hrs	Unlimited
Sunday:		6:00PM	30min	☐ variable	☑ All day	Unlimited	10 hrs	30min	14 hrs	Unlimited

NOTE: New autodir automatically created on: Mon(8:00)
New autodir WILL NOT be automatically created on: Tue Wed Thu Fri Sat Sun

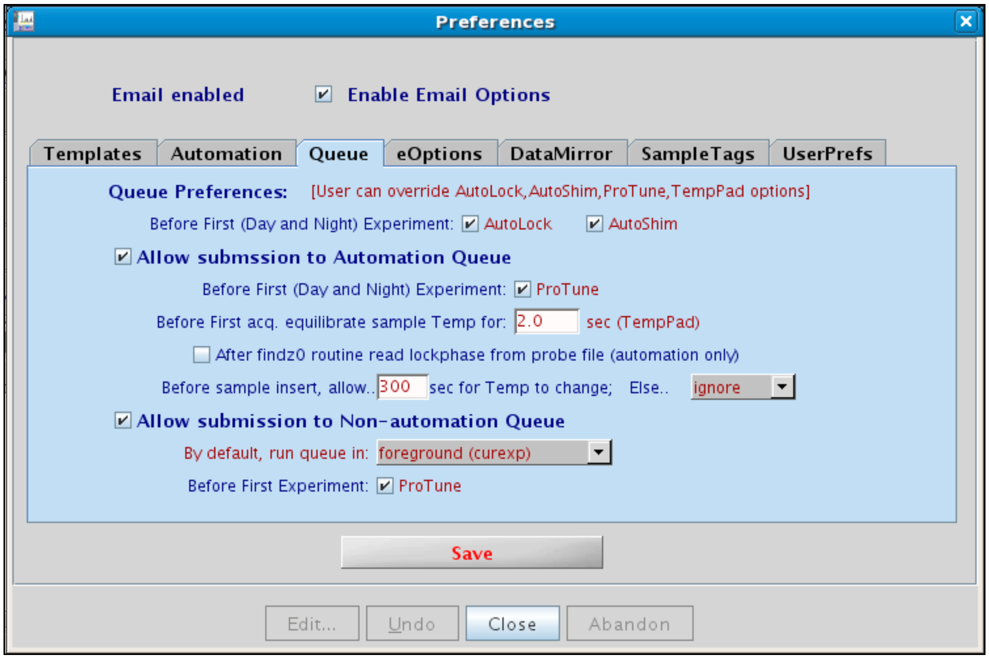
The Automation Schedule tool allows the account administrator to define how time is allocated during the day or night for AutoRuns (typically on demand sample automation) and AutoFiles (sample queues built for submission at a time chosen by the account administrator). Selecting the All Day check simply turns- off time management and is a better choice for a research environment as opposed to an open system being accessed by a large number of chemists. Using the Automation Schedule tool, the user can define different queue times for different times of the day as well as after hours’ queues. At sample submission time with the NightQ enabled, a chemist can submit certain experiments to the day and others to the night.

There are definitions for each day of the week. The first two columns define when does the day and the night start They are only active if the All day option is not selected. The third column, Max Time per Sample, defines how much experiment time will be allocated per sample during the day. The column with the same name further to the right defines the maximum time per sample during day and night runs.

The definitions for the AutoFiles are similar, with the fields defining how much should the total time for short and long queues be and how long should the maximum time per sample allowed for short and long queues be

Please note that any changes done on these two windows need to be saved (red button Save) before exiting the window, otherwise they will be lost. The changes will usually require the creation of a new automation run in order to become active.

8.3 Queue Tab



Email enabled ☑ Enable Email Options

Queue Preferences: [User can override AutoLock,AutoShim,ProTune,TempPad options]

Before First (Day and Night) Experiment: ☑ AutoLock ☑ AutoShim

☑ Allow submssion to Automation Queue

Before First (Day and Night) Experiment: ☑ ProTune

Before First acq. equilibrate sample Temp for: 2.0 sec (TempPad)

☐ After findz0 routine read lockphase from probe file (automation only)

Before sample insert, allow.. 300 sec for Temp to change; Else.. ignore

☑ Allow submission to Non-automation Queue

By default, run queue in: foreground (curexp)

Before First Experiment: ☑ ProTune

Figure 34 Queue tab - Preferences window

The Queue tab offers options for the actual automation queue. All settings at this page are default settings. Each sample submission will start with these default settings. They can be overwritten during the sample submission.

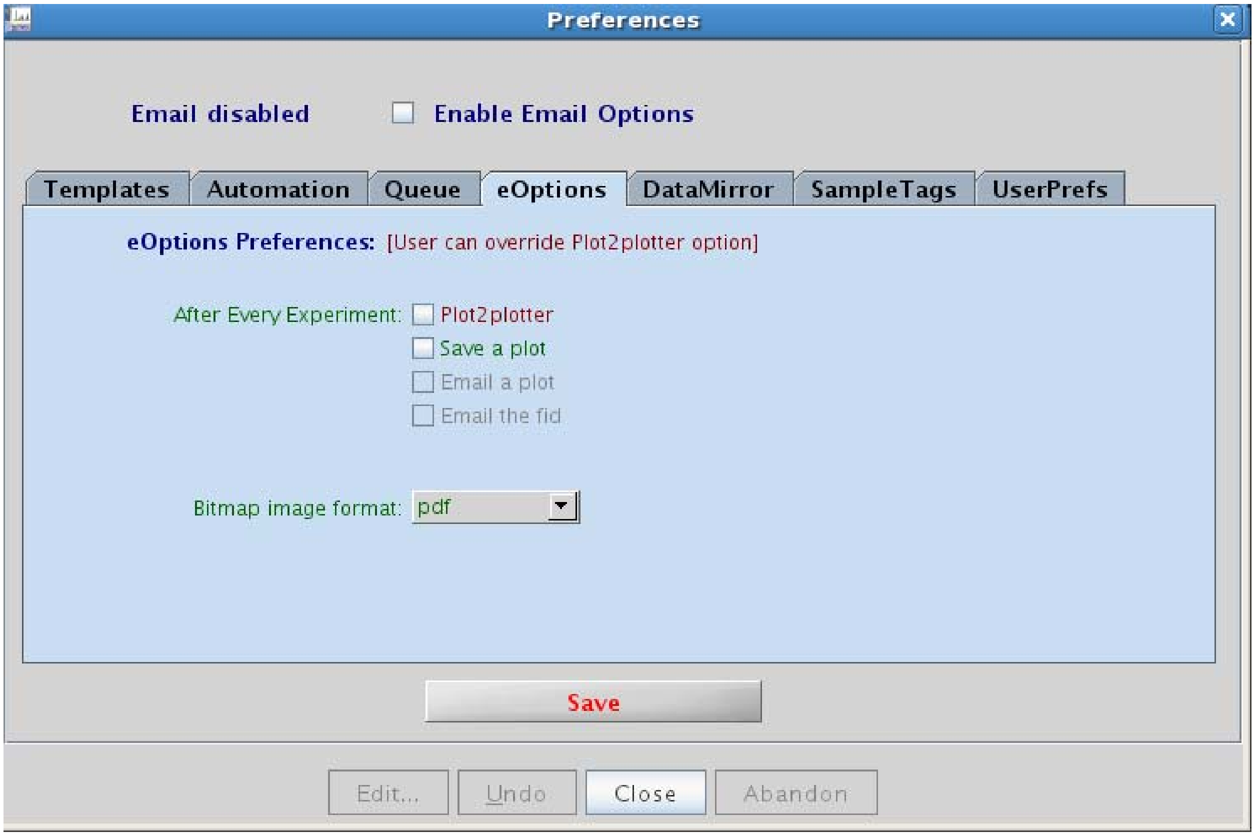
The first field defines whether the system will be automatically locked and shimmed before the first experiment of a sample queue. The normal behavior is for each sample to be automatically locked and shimmed and this is set here. If, for some reasons, one does not want the samples in the queue to be automatically shimmed and locked then these boxes should be unchecked.

Allow submission to Automation Queue	This setting activates the automation queue. If this is not desired then this should be unchecked. If the system is equipped with a ProTune module then the option for automatic tuning before the first experiment appears here If automatic tuning before the first experiment is not desired then this box should be unchecked.
Before first acq. Equilibrate sample temp for	This sets the amount of time that the system will wait for the temperature to equilibrate after it inserts the sample in the magnet but before recording any spectra. The default value is 2 seconds and it should be sufficient for experiments conducted at or near ambient temperatures. Increasing this value has got the advantage of having the temperature better equilibrated in the sample but the disadvantage of increasing the time required for each sample. Before sample After findz0 routine read lockphase from probe file (automation only). This is a setting for optimizing the lock system before spectra are recorded. If selected then the lock phase is set to the value in the probe file overwriting any value that has been set manually. The normal value for this box is to be unchecked. It should only be checked if there is a suspicion that the lock phase is not set properly. In this case the probe file should be updated with the correct value for the lock phase.
Insert allow XXX seconds for Temp to change, Else..	This value selects the amount of time the system waits after retrieving a completed sample but before inserting the next sample for the temperature to equilibrate. This is a useful feature when operators are allowed to run samples in automation at different temperature. It is placed there in order to avoid the case where a sample was run at a temperature higher than the boiling point or lower than the freezing point of the next sample solvent. The time defined here is the maximum time that the system will wait for the temperature to equilibrate. If the temperature equilibrates faster, the run continues. The actions in the pull-down menu define what will happen if such a condition is met and there is no waiting time defined. The possible actions are: “Ignore”, which causes the system to ignore the condition and proceed with the sample submission, “Abort”, which causes the system to abort the automation run and “Alert” which gives a warning to the operator.

Allow submission to Non-automation Queue	By default run queue in: This option defines how the automation will be run. The two options are “In the background (autodir)” and “In the foreground (curexp)”. The background option runs the AutoFiles automation in the same way as the standard automation run by holding all the information in the autodir directory. The foreground option will run all the automation in the current VnmrJ experiment. If the system is equipped with a ProTune module, the option for automatic tuning before the first experiment appears here If automatic tuning before the first experiment is not desired then this box should be unchecked.

NOTE: Any changes done on this window need to be saved (red button Save) before exiting the window, otherwise they will be lost. The changes will usually require the creation of a new automation run in order to become active.

8.4 eOptions Tab



Email disabled ☐ Enable Email Options

eOptions Preferences: [User can override Plot2plotter option]

After Every Experiment: ☐ Plot2plotter
☐ Save a plot
☐ Email a plot
☐ Email the fid

Bitmap image format: pdf

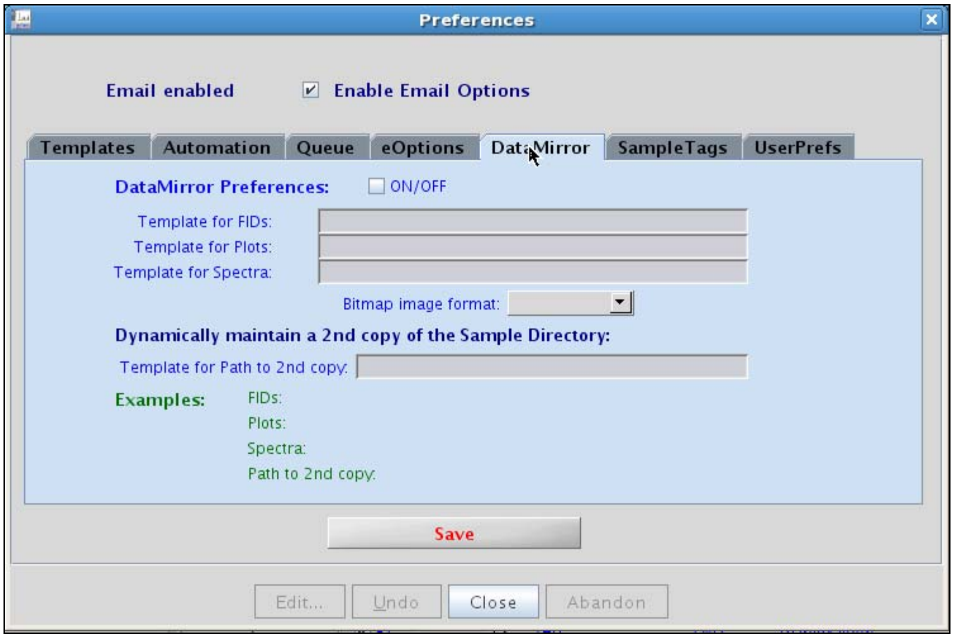
The eOptions tab defines actions that will be taken to produce an electronic output of the recorded spectra. The default options set on this tab will be used with every new study queue. Each operator has the option to override these in the Start parameter panel during the submission of the queue.

The available options define what happens after every experiment. The available options are:

plot2ploter	This produces a hardcopy output to a defined plotter.
Save a plot	This saves the plot in the format defined below
Email a plot	This option is enabled only if the “Enable Email Options is checked at the top of the window. If it is then the system will send a plot of the recorded spectrum as an e-mail attachment to the e-mail address of the operator who submitted the sample.
Email the FID	This option is also enabled if the “Enable Email Options” is checked at the top of the window. If it is then the system will send the entire fid with the recorded spectrum as an e-mail attachment to the e-mail address of the operator who submitted the sample. This option should be used with care as NMR spectra, especially multidimensional ones, can grow to very large sizes which may not be accommodated by the available capacity and bandwidth of the network.
Bitmap image format	This pull-down menu defines the format of the plot that will be saved or e-mailed. Options are: • tif. This creates a TIFF format document, typical for accurate representation of bitmap images. A TIFF document can be read with most common image and word processing programs. • pdf. This creates a document according to the Adobe PDF™ document format. A PDF document can be read using Adobe Acrobat Reader. The PDF documents created from VnmrJ require Adobe Reader version 5 or higher. • pcx. This creates a document using the PCX protocol, adequate for representations of bitmap images. PCX documents can be read with most common image and word-processing documents. • jpg. This creates a JPEG format document accurate for the representation of real-life photos. JPEG documents can be read with most common image and word processing programs

NOTE: Any changes done on this window need to be saved (red button Save) before exiting the window, otherwise they will be lost. The changes will usually require the creation of a new automation run in order to become active.

8.5 Data Mirror Tab



Email enabled ☑ Enable Email Options

DataMirror Preferences: ☐ ON/OFF

VnmrJ 3 provides the option to automatically store another copy of the data recorded in automation on a local or remote disk. Use the Data Mirror tab to setup this feature is defined.

If the option is turned on (ON/OFF check box at the top of the window) the feature is activated and each dataset is saved twice: first at the locations defined in the Templates tab, second at the locations defined on this tab. The syntax for the templates is identical to the ones in the Templates Tab.

There are three types of files that are mirrored: FIDs, Plots and Spectra. The entire sample directory can be mirrored and defined in the last field on this tab.

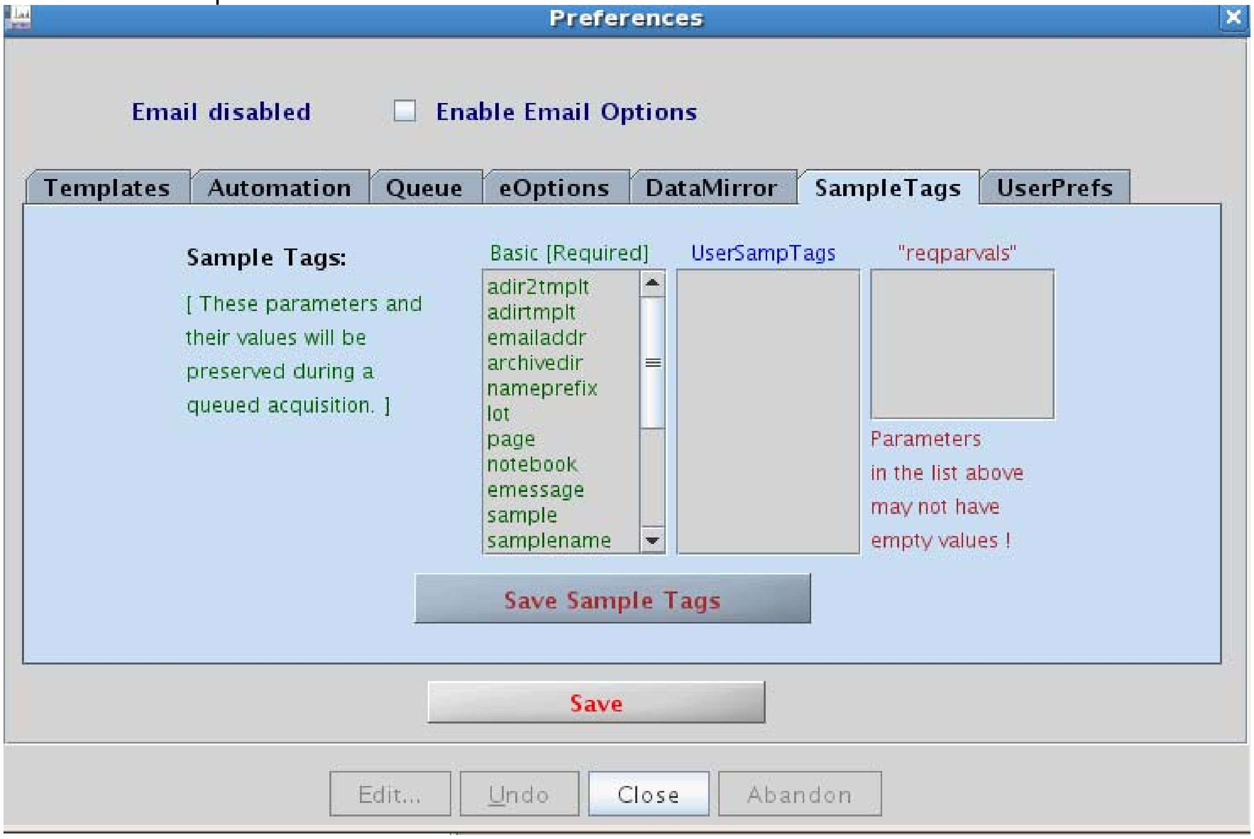
Data mirroring can be useful for backup. When used with externally mounted or network drives, users can access data at other systems. This can have several benefits: reducing the load on the spectrometer computer host, reducing the need for extensive network transfers of data.

A complementary tool to this is the UNIX function of rsync. The way to setup rsync is described in most UNIX manuals.

NOTE: Any changes done on this window need to be saved (red button Save) before exiting the window, otherwise they will be lost. The changes will usually require the creation of a new automation run in order to become active.

8.6 SampleTags Tab

The SampleTags tab allows the system administrator to define which parameters required for each sample will be recorded.



[These parameters and their values will be preserved during a queued acquisition.]

adir2tmplt
adirtmplt
emailaddr
archivedir
nameprefix
lot
page
notebook
emessage
sample
samplename

Parameters in the list above may not have empty values !

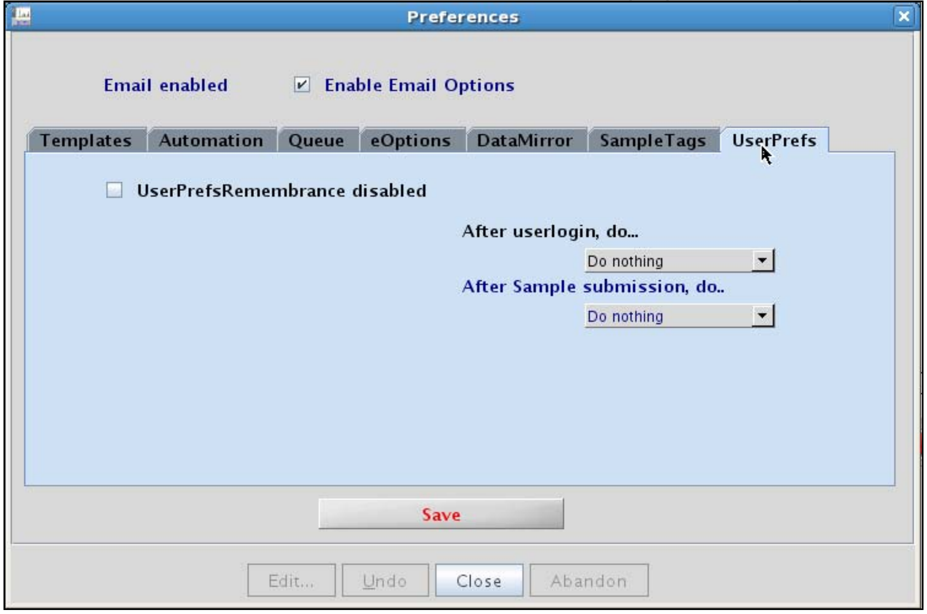
The window displays three lists of parameters:

Basic (required)	These are parameters that are required for each sample, like the samplename, the data directory where data are saved etc.
UserSamp Tags	Any extra parameters that the system administrator may want the users to be entering. Normally this would require a respective entry into the userficpar macro. Such parameters may be, for example, the department which the operator belongs etc.
“reqparvals”	This column contains the parameters that will be absolutely required to be entered otherwise the automation run will not be able to proceed. Note that any parameter appearing as data saving parameter in the Templates tab will automatically be required and one does not need to enter it again here.

NOTE: Any changes done on this window need to be saved (red button Save) before exiting the window, otherwise they will be lost. The changes will usually require the creation of a new automation run in order to become active.

8.7 UserPrefs Tab

The UserPrefs tab allows the account administrator to setup options for operators.



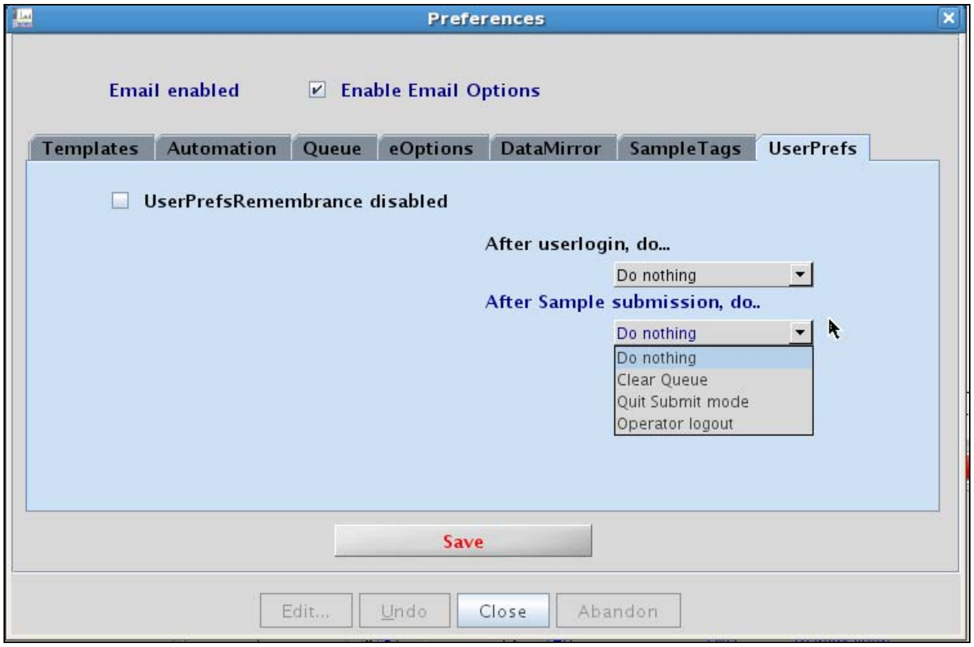
Email enabled ☑ Enable Email Options

☐ UserPrefsRemembrance disabled

After userlogin, do... Do nothing

After Sample submission, do.. Do nothing

The first task the account administrator must perform using the UserPrefs tab is to decide the action to be taken after sample submission.



Email enabled ☑ Enable Email Options

☐ UserPrefsRemembrance disabled

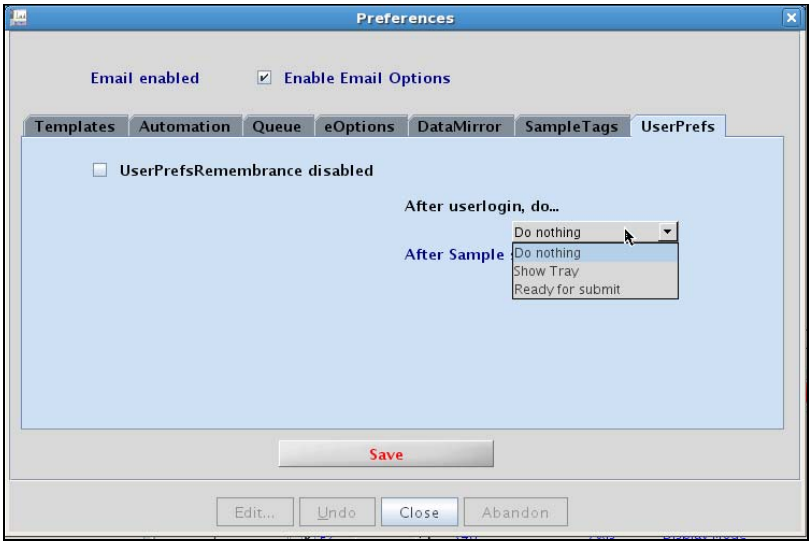
After userlogin, do... Do nothing

After Sample submission, do.. Do nothing

The choices:

Do nothing	Enables the user to leave the list of experiments in the StudyQueue in place for submission in full or in part to another location.
Clear Queue	Clears the experiment list while leaving the interface in submission mode for easy creation of a new queue for other sample locations.
Quit Submit mode	Automatically clicks the **Done** button and exit sample submission mode and leaves the operator in Data Review mode.
Operator logout	Quits submit mode and logs that operator out automatically. This is an excellent choice for a high-volume NMR service lab with lots of operators who tend to generally submit one sample at a time.

The account administrator next defines the interface personality at the time of operator login.

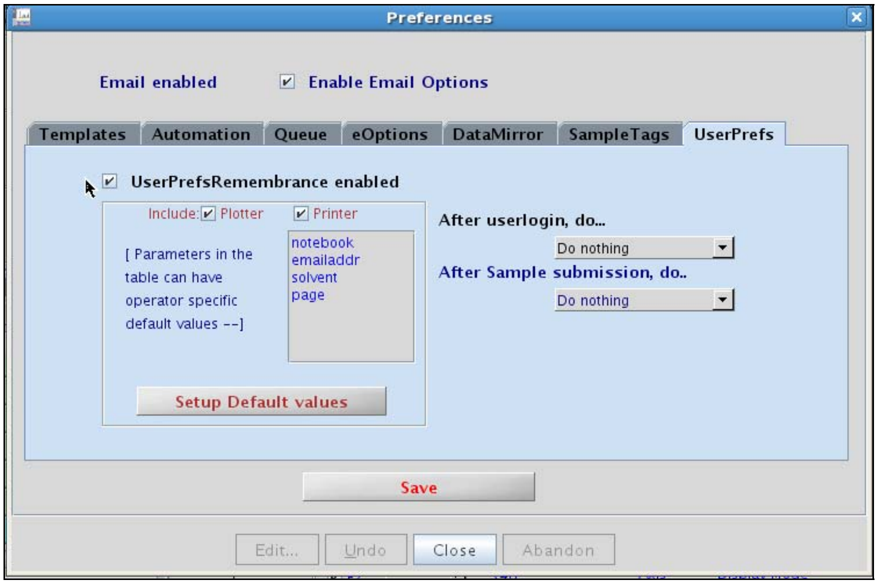


The choices are as follows:

Choice	Description
Do nothing	Lets the operator choose the way
Show Tray	Displays the automation tray
Ready for submit	Automatically clicks “**New Sample**” button to enable the submission mode of the interface

In the UserPrefs tab, the option UserPrefsRemembrance enables the user to set values for any desired parameters that can be pre-set upon entry of sample submission mode. The parameters can be unique for each operator, allowing users to easily manage pre-filling of items such as preferred solvent, notebook, email address, and so on.

Note that once enabled and the parameter list for remembrance is defined by the account administrator, individual operators can manage their own preferences for those parameter via the menu Edit=>Operator Preferences.



Email enabled ☑ Enable Email Options

☑ UserPrefsRemembrance enabled

Include: ☑ Plotter ☑ Printer

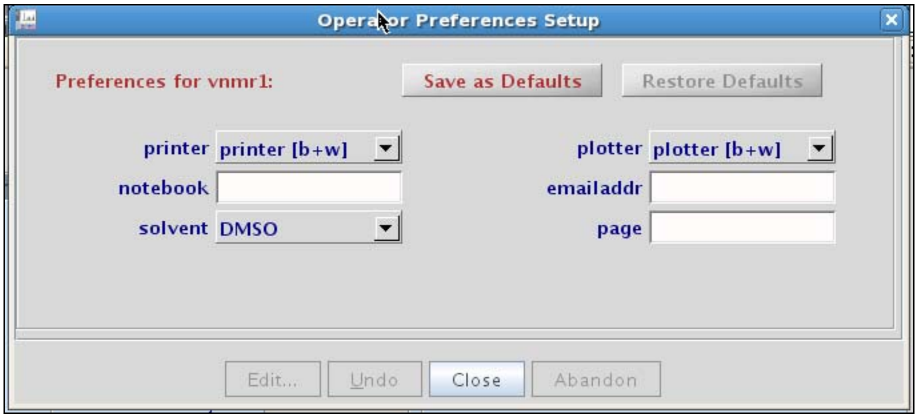
[Parameters in the table can have operator specific default values --]

notebook
emailaddr
solvent
page

After userlogin, do... Do nothing

After Sample submission, do.. Do nothing

In this example, the operator choices for printer, plotter, notebook, email address, solvent, and page have been defined by the account administrator by entering those checks or parameter names into the entry box. After the first time, the account administrator must select the Setup Default values button to assign defaults for the user to start modifying values.



Preferences for vnmr1:

printer: printer [b+w]
plotter: plotter [b+w]
notebook:
emailaddr:
solvent: DMSO
page:

The menu for plotter and printer read valid devices known to the NMR spectrometer and each operator can choose the location where the data hard copy is plotted. The userRemembrance tools help prevent keying errors of items such as a laboratory notebook and preferred solvents. In this example the email address is controlled by the operator and not an administrative task for the account administrator. An operator can choose to direct PDF plot emails to any desired email address.

NOTE: Any changes done on this window need to be saved (red button Save) before exiting the window, otherwise they will be lost. The changes will usually require the creation of a new automation run in order to become active.

Chapter 9 Quantification Tools

VnmrJ 3 provides two new tools to facilitate NMR quantitative measurements: Quanttools located in an application directory, and the qEstimate tool.

The package of programs in Quanttools provides a mechanism to assay any sample for a specific chemical substance. This method uses a user-generated template that must first be interactively created from an NMR spectrum, acquired on a standard sample. This method supports automated analysis and report generation once user-generated template is in place. The use of internal standards is required to perform these analyses.

Use the qEstimate tool to provide an estimate of absolute concentration for any sample. This capability is based on the linearity of the digital receiver and does not require the use of reference signals or the addition of reference standard compounds to the NMR sample. A simple calibration must be performed once for each probe before this tool can be used, see Calibrating probe parameters for quantitation.

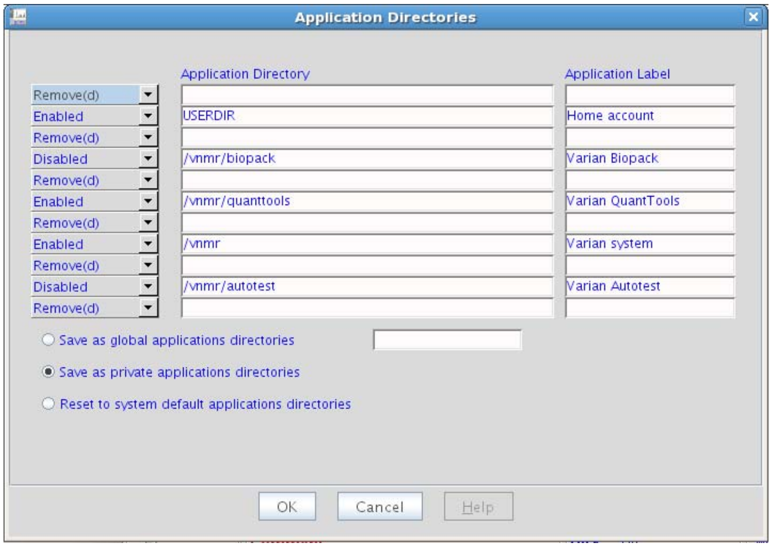
Sections in this chapter include:

- 9.1 Quanttools Application Directory
- 9.2 The qEstimate tool
- 9.3 Automated Creation of Quantification Reports

9.1 Quanttools Application Directory

To access the tools:

1. Enable the Varian QuantTools application directory.
2. Select **Applications…** from the Edit menu,
3. Select **Enabled** from the pull-down menu next to /vnmr/quanttools.
4. Exit and restart VnmrJ to load the new menus and panel items, after the application directory is enabled.



	Application Directory	Application Label
Remove(d)		
Enabled	USERDIR	Home account
Remove(d)		
Disabled	/vnmr/biopack	Varian Biopack
Remove(d)		
Enabled	/vnmr/quanttools	Varian QuantTools
Remove(d)		
Enabled	/vnmr	Varian system
Remove(d)		
Disabled	/vnmr/autotest	Varian Autotest
Remove(d)		

○ Save as global applications directories

● Save as private applications directories

○ Reset to system default applications directories

Figure 35 QuantTools application directory as enable.

9.2 The qEstimate tool

9.2.1 Calibrating probe parameters for quantitation

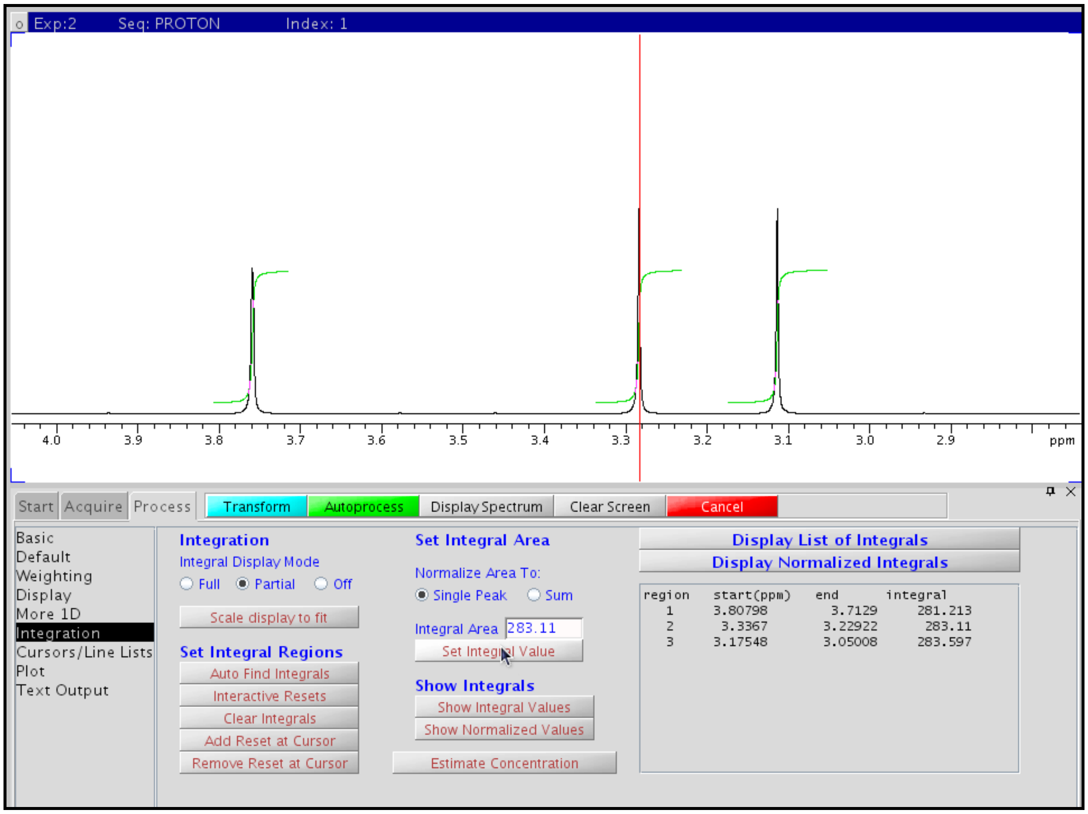
Before the qEstimate tool can be used, a simple calibration step must be performed. This calibration is specific to each probe and receiver combination.

1. Carefully prepare a sample of known concentration. Any sample in the 40 to 100 mM range should be suitable, assuming that a.) it contains a resonance, or group of resonances, that are well separated from all other signals, b.) that resonance is reasonably sharp, and c.) the number of protons represented by that resonance is known. The ^{31}P signal to noise standard sample provided with the system can be used with reasonable results.

2. Insert the calibration sample and ensure that the probe is properly tuned and shimmed. Set the number of transients to 1 (*i.e.*, nt=1) and turn off the steady state transients (*i.e.*, ss=0). Set the receiver gain manually to a value that provides good signal-to-noise without giving a receiver overflow error (letting the system set the gain value automatically could introduce relaxation errors). Acquire a spectrum.

3. After processing the spectrum, navigate to the Integration panel under the Process tab. Establish an integral region around the resonance to be used for calibration using the Set Integral Regions tools. Make sure to include some baseline on each side of the peak in the integral region. Place the cursor within the selected integral region.

4. Multiply the concentration of the calibration standard by the number of protons represented under the resonance chosen in step 3, then enter that result into the Integral Area field. For example, if the standard sample was created at a concentration of 94.36 mM and the resonance used for calibration was a methyl group (*i.e.*, 3 protons), the number entered into the Integral Area field would be 283.11 (*i.e.*, 94.36 × 3 = 283.11; see Figure 36). Select the Set Integral Region button.



Integral Display Mode ○ Full ● Partial ○ Off

Normalize Area To: ● Single Peak ○ Sum

Integral Area 283.11

```
region   start(ppm)    end       integral
  1      3.80798      3.7129     281.213
  2       3.3367     3.22922      283.11
  3      3.17548     3.05008     283.597
```

Figure 36

Setting the integral value for a calibration standard of caffeine dissolved at a concentration of 94.36 mM, see Figure 36. The integral region for the methyl signal at approximately 3.28 ppm has been selected and a value of 283.11 has been entered into the Integral Area field.

If the ^{31}P S/N sample is used to calibrate the system, integrate the all the aromatic resonances as a group and enter the value of 727.5 into the Integral Area box. The sample is approximately 0.485 molar and the aromatic resonances represent 15 protons from 3 phenyl rings. The small amount of residual CHCl3 also in the region does not significantly effect the calibration.

5. Type `qEstimate(‘setproBefile’)` on the command line and hit Return. This will create the parameters required for the qEstimate function and store appropriate values for those parameters in the probe file. The parameters that are created by qEstimate(‘setproBefile’) are H1Qgain, H1Qins, H1Qinsref, H1Qconc, and H1Qmult. H1Qmult refers to the tip angle scaling factor where a value of 1 equals a 90 degree tip angle.

Once these values are on the probefile, sample concentration is accessible by a mouse click. The results are still dependent on the physics and relaxation properties of the sample. Typically, reasonable results are obtained from normal survey ^{1}H spectra. More precise results can be obtained by using a single scan fid acquired with a 90 degree tip angle and fixed receiver gain.

9.2.2 Using qEstimate

Whenever qEstimate is evoked the gain and tip angle for the current sample are noted and compared with the values used for during calibration. The parameter ins (concentration for the standard) is loaded into the current experiment and the parameter insref is adjusted from the value shown on the probe file for the scaling factors defined by receiver gain and tip angle.

To determine the concentration of an NMR sample:

1. Acquire a proton spectrum.
2. Integrate a resonance (or group of resonances) representing a known number of protons.
3. Select the **Estimate Concentration** button on the Integration panel.
4. Divide the integral area displayed for a give peak by the number of protons represented under that peak.

   This yields the millimolar concentration of the sample is the result from step 4.

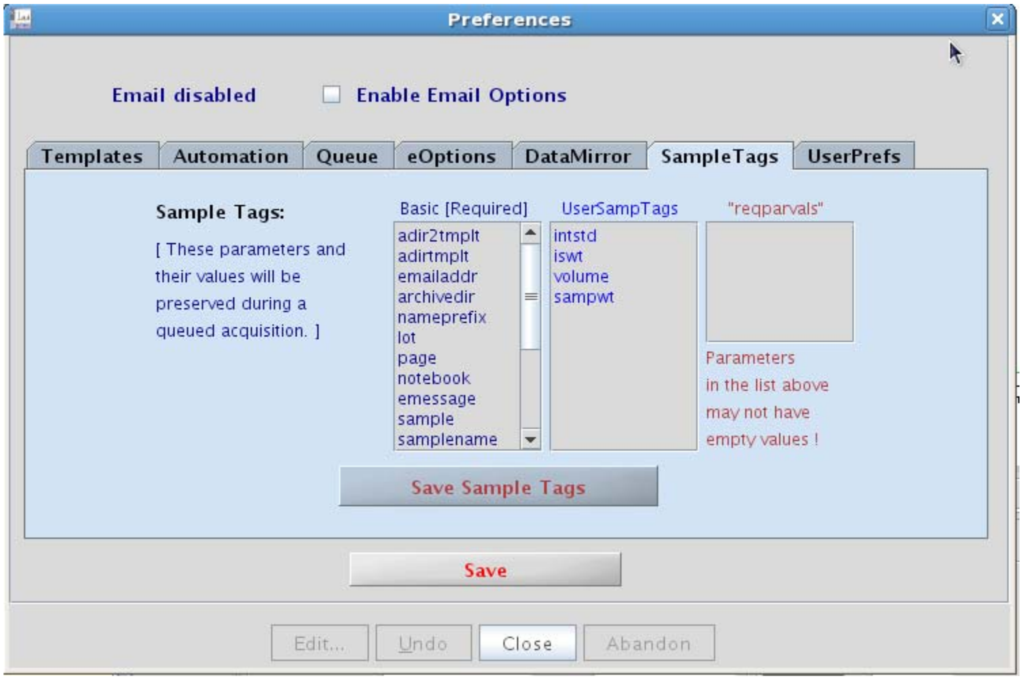
For example, the methyl resonance of an isopropyl group represents 6 protons. If integration of the isopropyl methyl signals from an unknown sample yielded a value of 339.6 after clicking **Estimate Concentration**, then the concentration of the sample is 56.6 mM (339.6 ÷ 6 = 56.6).

9.3 Automated Creation of Quantification Reports

A precise answer with respect to the exact purity of a substance is often required. In this situation the best solution is to add a precise amount of a reference compound and compare the integral results for the compound to that of the internal reference standard. The purpose of the reporting macro is to allow a completely automated output of results with the answer in the output rather than generating raw data for manual analysis after the fact`. In order to generate a reporting macro prepare a standard sample and include a reference standard and acquire a spectrum suitable to precise quantification. Either a single scan 90º pulse experiment with fixed receiver gain or a multi-scan data set with the relaxation delay long enough to ensure complete relaxation. Four internal standards are supported, maleic acid, dimethylsulfone, dimethylfumarate, or methenamine.

Quantification requires a number of new parameters that are available via a userfixpar macro inside of the quanttools maclib. To use an existing userfixpar macro to use the automatic reporting tools in the quanttools applications directory, edit your local userfixpar macro to include what is found in the applications directory.

Once the new parameters are created, go to Edit > Preferences > SampleTags and add userSamptags field, volume, sampwt, iswt, and intstd. This step allows software to know to keep track of the values for all of these new parameters just as it does for samplename, notebook, page.



Email disabled ☐ Enable Email Options

Sample Tags:

[These parameters and their values will be preserved during a queued acquisition.]

Basic [Required]: adir2tmplt, adirtmplt, emailaddr, archivedir, nameprefix, lot, page, notebook, emessage, sample, samplename

UserSampTags: intstd, iswt, volume, sampwt

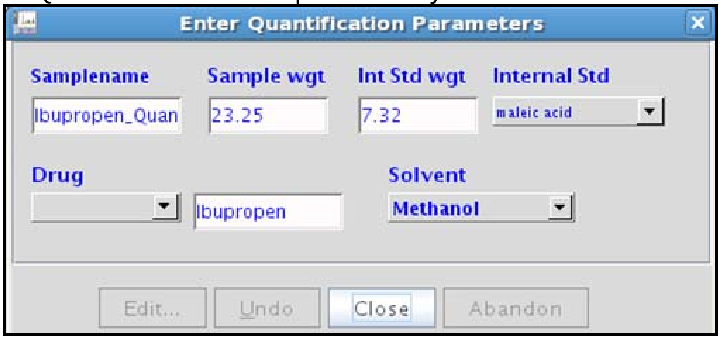
"reqparvals"

Parameters in the list above may not have empty values !

Figure 37

In the Sample Tags page:

1. Enter the parameters.
2. Click Save Sample Tags.
3. Click Save.
4. Restart VnmrJ.
5. After the restart. you can enter the quantification-specific parameters in the Standard page in the Start folder.
6. Click SetQuantPars button to open an entry box.



Samplename: Ibupropen_Quan

Sample wgt: 23.25

Int Std wgt: 7.32

Internal Std: maleic acid

Drug: Ibupropen

Solvent: Methanol

Figure 38 Enter Quantitation Parameters window

After a data set has been acquired with the reference standard of a compound of interest, navigate to the automatic report creation menu found in the Tools Menu. An interactive entry form and help menu opens to assist the user through the rest of the process.

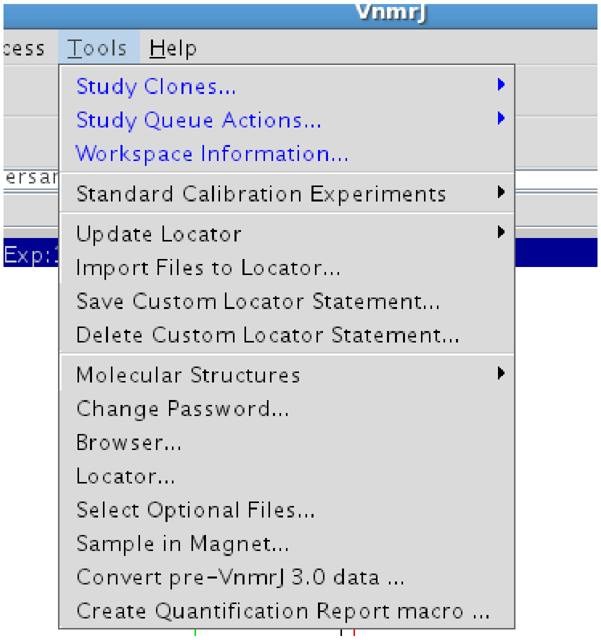
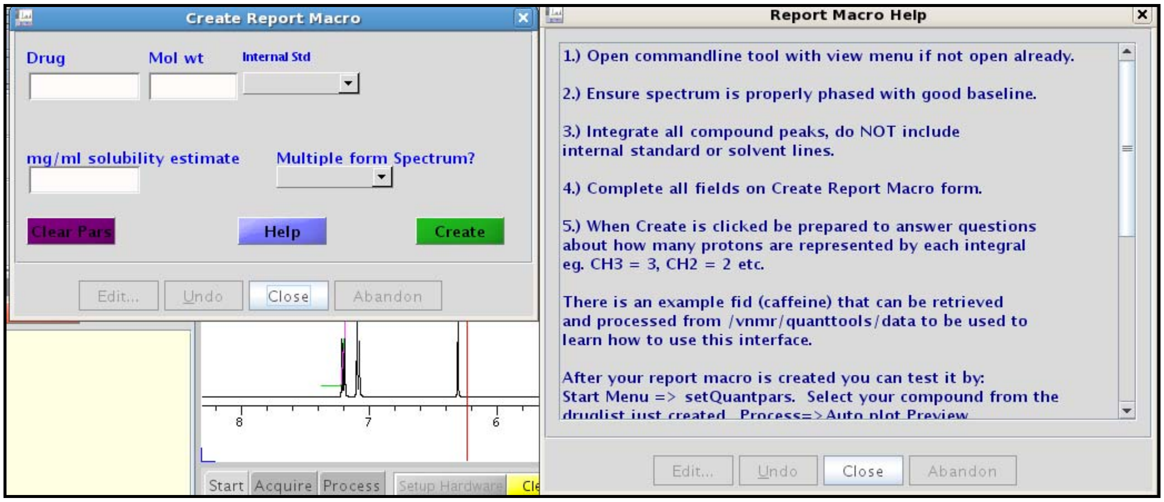


Figure 39 Automatic Report Creation menu

The form that is opened by running the Create Quantification Report macro is shown below



1.) Open commandline tool with view menu if not open already.

2.) Ensure spectrum is properly phased with good baseline.

3.) Integrate all compound peaks, do NOT include internal standard or solvent lines.

4.) Complete all fields on Create Report Macro form.

5.) When Create is clicked be prepared to answer questions about how many protons are represented by each integral eg. CH3 = 3, CH2 = 2 etc.

There is an example fid (caffeine) that can be retrieved and processed from /vnmr/quanttools/data to be used to learn how to use this interface.

After your report macro is created you can test it by:
Start Menu => setQuantpars. Select your compound from the druglist just created. Process=>Auto plot Preview

Figure 40 Create Quantification Report Form

The following example is of creating a reporting macro for ibuprofen. First integrate the spectrum, being careful to NOT include the internal standard or the solvent peaks. The internal standard of the example was maleic acid near 6.3 ppm and the solvent was deuterated methanol with peaks near 5.0 ppm and 3.3 ppm.

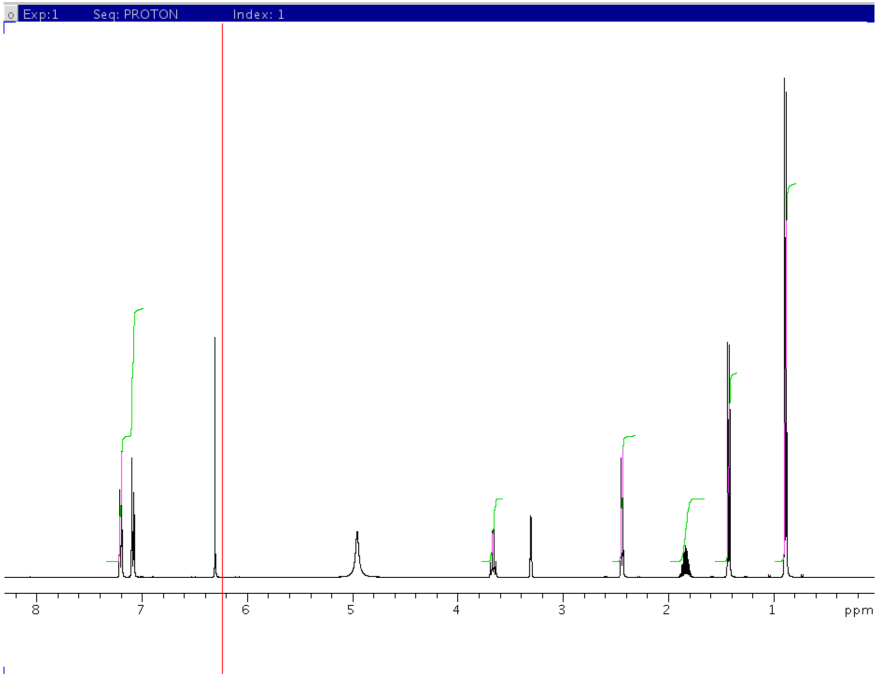
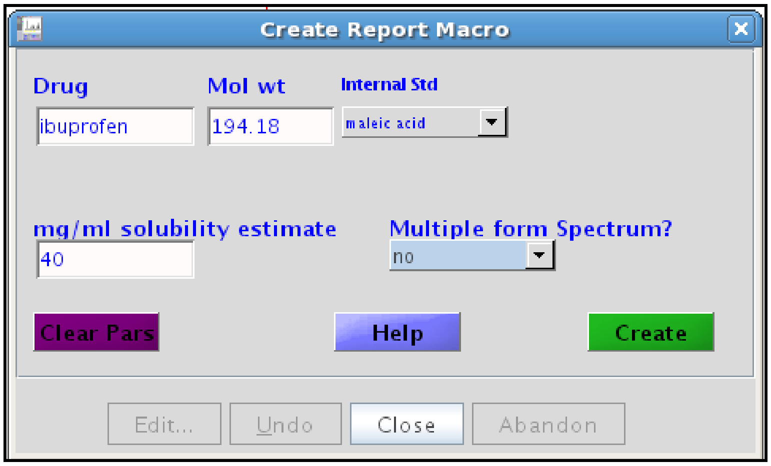


Figure 41 Integrating the Spectrum

Evoke the Create Quantification Report macro in the Tools menu and fill out the form.



Drug: ibuprofen

Mol wt: 194.18

Internal Std: maleic acid

mg/ml solubility estimate: 40

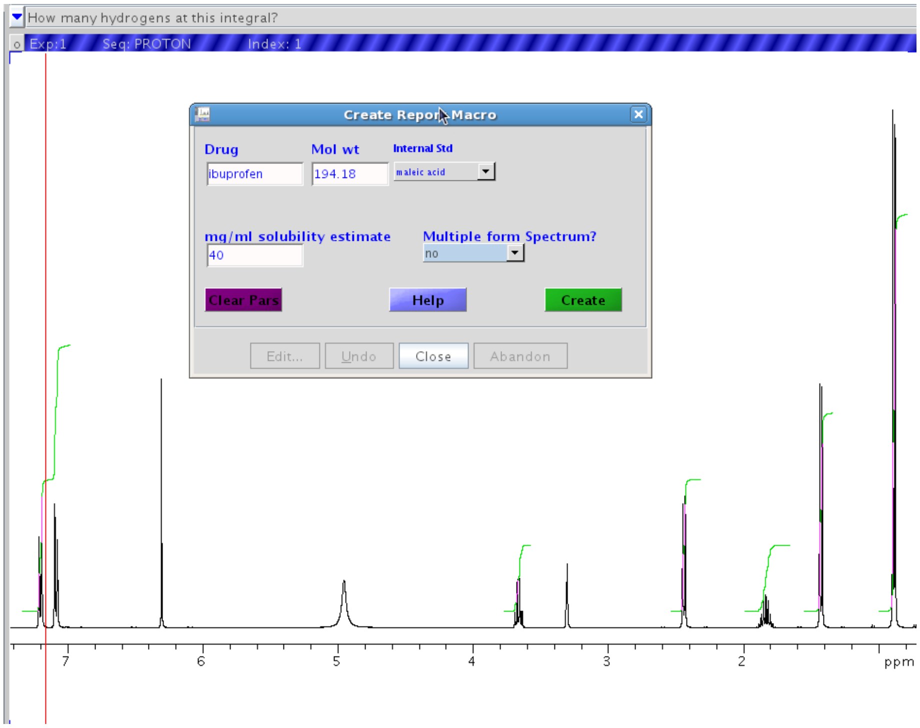
Multiple form Spectrum?: no

Figure 42 Create Report Macro for Ibuprofen

A guess for the solubility estimate field is sufficient.

Select **No** in the Multiple form Spectrum field if there is no exchange on the NMR timescale mixing populations.

Click **Create** and a cursor moves through the spectrum and you are prompted to enter the number of protons represented by each integral region.



How many hydrogens at this integral?

Exp:1 Seq: PROTON Index: 1

Create Report Macro

Drug: ibuprofen

Mol wt: 194.18

Internal Std: maleic acid

mg/ml solubility estimate: 40

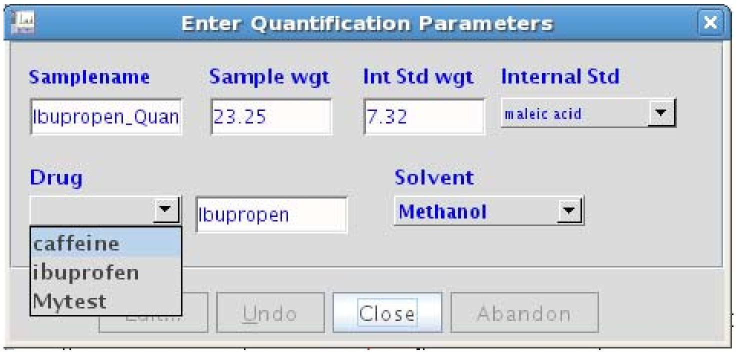
Multiple form Spectrum?: no

Figure 43 Filling in the number of protons window

After the questions are answered a macro will be created in the local user's maclib with a name based on the answers to the questions. In this example the macro will be called "quant_ibuprofen_cd3od_maleic" because the drug name was given as ibuprofen, the solvent was cd3od, and the internal standard was maleic acid.

If the purity of the internal standard is 100%, edit the macro and update the entry for $purity on line 3 to the correct value.

Creating the report automatically adds the name of the drug to the source for a menu in the SetQuantPars tool.



Enter Quantification Parameters

Samplename: Ibupropen_Quan

Sample wgt: 23.25

Int Std wgt: 7.32

Internal Std: maleic acid

Drug: Ibupropen

Solvent: Methanol

Figure 44 Enter Quantification Parameters window

When a purity quantification analysis calling the specific reporting macro is finished the macro will generate a custom output for that sample. There are two ways to hook into automation. Either one Experiment Selector button with output directed by the parameters entered at the time of submission or a single Experiment Selector button for each drug. Both strategies require creating a experiment button.

To create a quantification-specific experiment button begin by first acquiring a fid for the desired sample. If the parameter **`execplot`** does not exist (type `execplot?` on the command line) then create the parameter by entering:

```
create(‘execplot’,’string’)
```

Next open the Edit Parlib tool from the Tools menu.

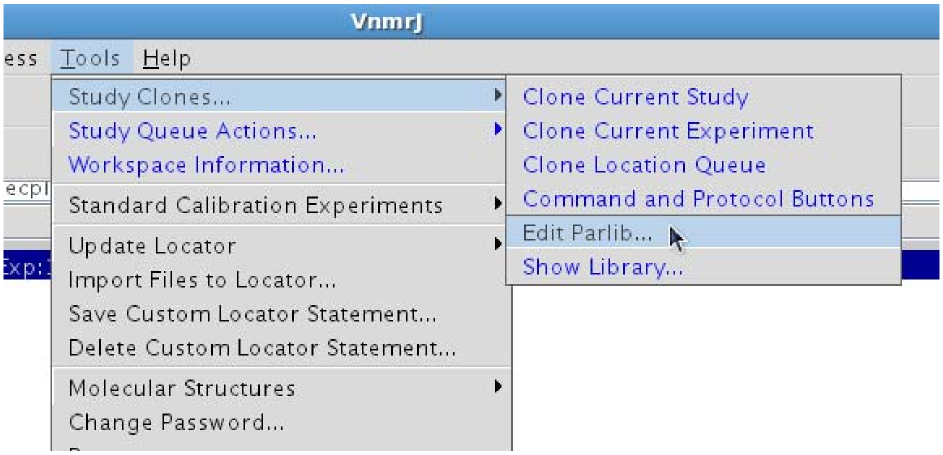
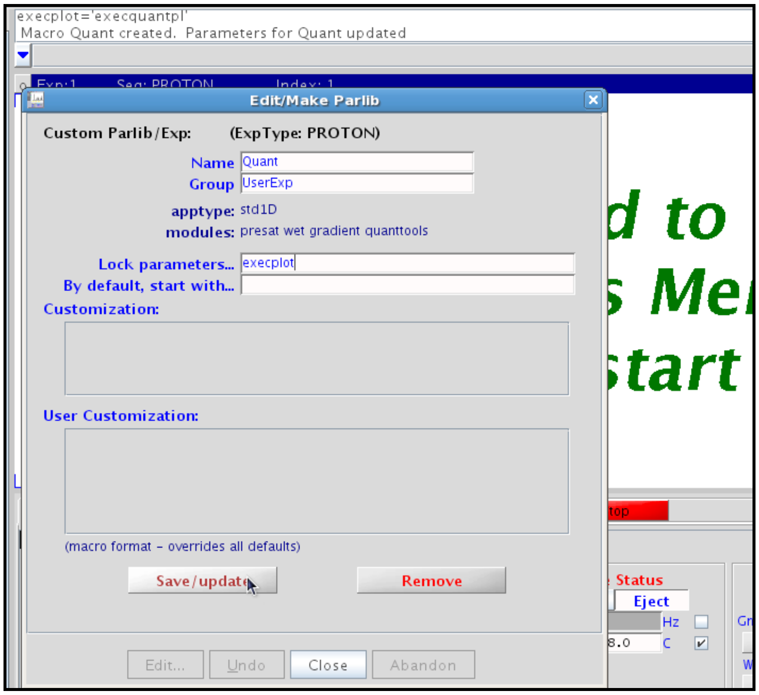


Figure 45 Edit Parlib –Tools menu


```
execplot='execquantpl'
 Macro Quant created.  Parameters for Quant updated
```

Custom Parlib/Exp: (ExpType: PROTON)

Name: Quant

Group: UserExp

apptype: std1D

modules: presat wet gradient quanttools

Lock parameters...: execplot

By default, start with...:

Customization:

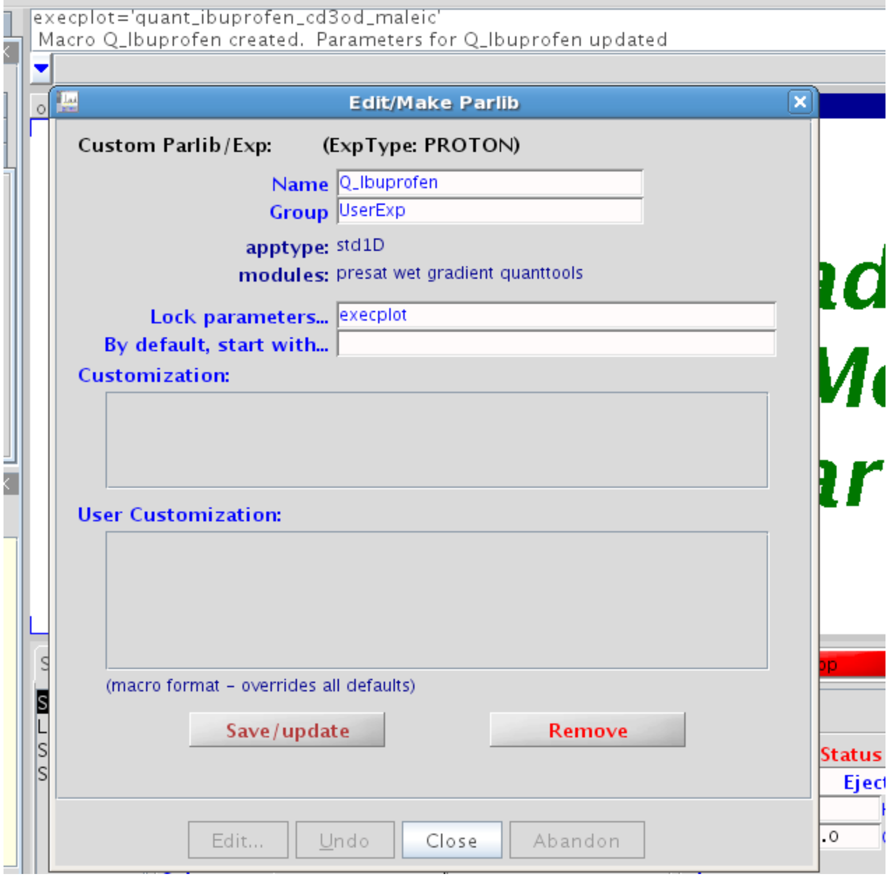
User Customization:

(macro format – overrides all defaults)

Figure 46 Setting the parameters in the Edit/Make Parlib page

For a single Quant Experiment button where the reporting macro is called by the values in parameters, **`drug`**, **`solvent`**, and **`intstd`** assign `execplot='execquantpl'` from the command line. Then in the Edit Parlib tool add **`execplot`** to the Lock parameters list, enter a name for the button, and click Save/update.

In the example, to create a button called Q_ibuprofen, enter the name into the name field and assign `execplot='quant_ibuprofen_cd3od__maleic'` before opening the Edit Parlib tool.

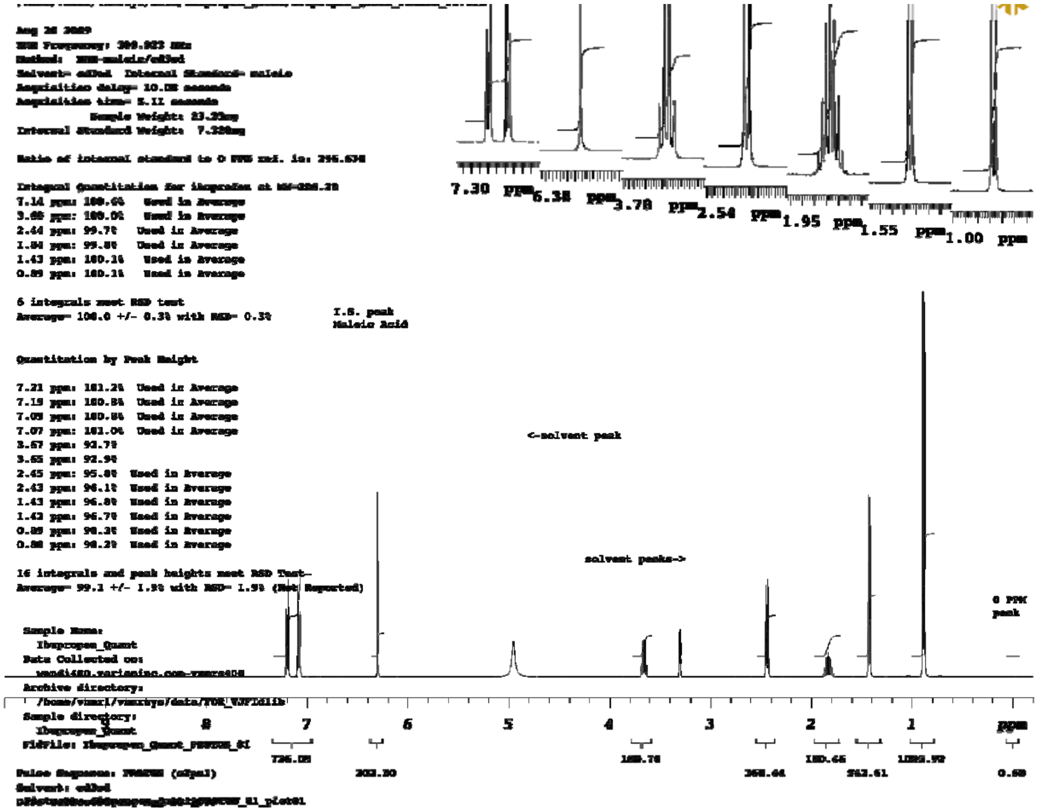


```
execplot='quant_ibuprofen_cd3od_maleic'
Macro Q_Ibuprofen created.  Parameters for Q_Ibuprofen updated
```

Custom Parlib/Exp: (ExpType: PROTON)

Name: Q_Ibuprofen
Group: UserExp
apptype: std1D
modules: presat wet gradient quanttools
Lock parameters...: execplot
By default, start with...:

The automation report output for ibuprofen is shown below.



```
Aug 28 2009
NMR Frequency: 399.923 MHz
Method:  NMR-maleic/cd3od
Solvent= cd3od  Internal Standard= maleic
Acquisition delay= 10.00 seconds
Acquisition time= 5.11 seconds
           Sample Weight: 23.20mg
Internal Standard Weight:  7.320mg

Ratio of internal standard to 0 TMS ref. is: 295.670

Integral Quantitation for ibuprofen at MW=206.28
7.14 ppm: 100.0%   Used in Average
3.66 ppm: 100.0%   Used in Average
2.44 ppm: 99.7%   Used in Average
1.84 ppm: 99.8%   Used in Average
1.43 ppm: 100.1%   Used in Average
0.89 ppm: 100.1%   Used in Average

6 integrals meet RSD test
Average= 100.0 +/- 0.3% with RSD= 0.3%

Quantitation by Peak Height

7.21 ppm: 101.2%  Used in Average
7.19 ppm: 100.8%  Used in Average
7.09 ppm: 100.8%  Used in Average
7.07 ppm: 101.0%  Used in Average
3.67 ppm: 92.7%
3.65 ppm: 92.9%
2.45 ppm: 95.8%  Used in Average
2.43 ppm: 96.1%  Used in Average
1.43 ppm: 96.8%  Used in Average
1.42 ppm: 96.7%  Used in Average
0.89 ppm: 98.2%  Used in Average
0.88 ppm: 98.2%  Used in Average

16 integrals and peak heights meet RSD Test-
Average= 99.1 +/- 1.9% with RSD= 1.9% (Not Reported)

 Sample Name:
   Ibuprofen_Quant
 Data Collected on:
   [illegible]
 Archive directory:
   /home/vnmr1/vnmrsys/data/TOK_VJFIdlib
 Sample directory:
   Ibuprofen_Quant
 FidFile: Ibuprofen_Quant_PRESAT_01

Pulse Sequence: PRESAT (s2pul)
Solvent: cd3od
[illegible]_01_plot01
```