Quantum NMR Spectrometer

SpinStudioJ

User Manual

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Contents

Chapter 1 Overview

1.1 Introduction

SpinStudioJ is a professional software package for NMR data acquisition and processing in the products of Zhongke-niujin. The User Manual thoroughly describes all aspects of SpinStudioJ, which include system requirements, installation, introduction to menus and panels, NMR experiments, data processing and analysis, as well as the routine 1D and 2D experiments, data plotting and pulse programming. This manual enables users to quickly get acquainted with the main functions and operations of the software and be quite capable of running experiments and processing data. All users of SpinStudioJ are recommended to carefully read this manual.

1.1.1 Definitions and Conventions

In order to make the text in the manual more clear and easier to be understood by users, following conventions are used in this manual:

1. Commands to be entered are all in ***bold italic***, e.g. ***wft.***
2. Menus, buttons are all in **bold**, e.g. **File**, **Acquire**.

Table 1.1 Term definition and abbreviation

Term abbreviation	Description
FID	Free Induction Decay
APT	Attached Proton Test
BIRD	BIlinear Rotation Decoupling
DEPT	Distortionless Enhancement by Polarization Transfer
DOSY	Diffusion Ordered Spectroscopy
GARP	Globally optimized Alternating-phase Rectangular Pulses
T_1	Spin-Lattice Relaxation Time

T_2	Spin-Spin Relaxation Time

1.2 Start the System

Double click the icon of SpinStudioJ in your desktop to start the software, the user login dialog will pop up, as shown in Figure 1.1. Then enter the user name and password, the first login can be logged in with the user name “admin”, the password is “zhongkeMR”. The user name and password are case-sensitive.

Figure 1.1 User login dialog

After click **OK** in Figure 1.1, the main interface will be open(Figure 1.2). The main interface includes menu bar, toolbar, navigation panel, data window, log panel, command line, status bar.

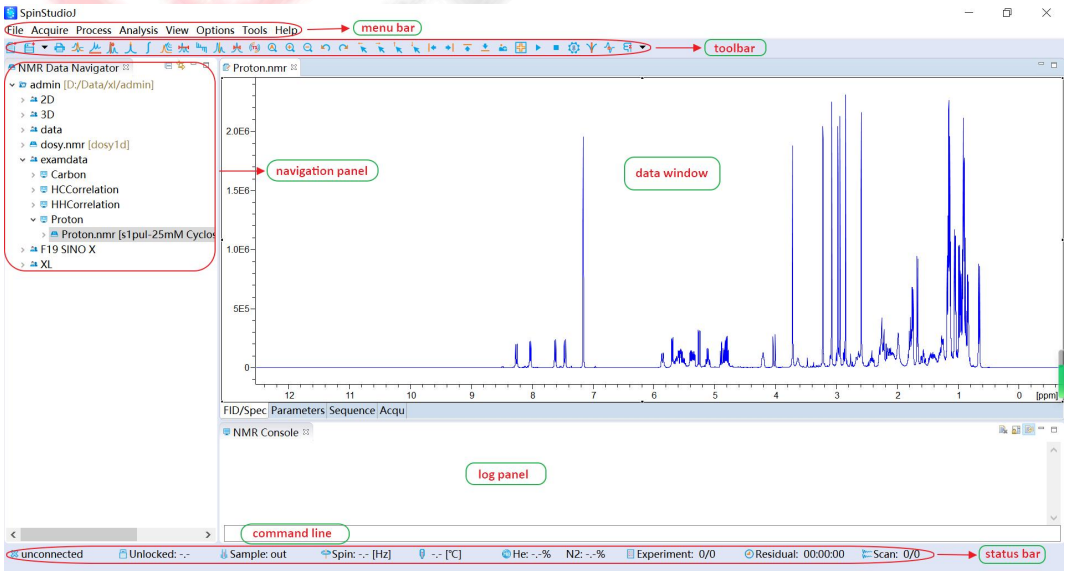


Figure 1.2 Software main interface

1.3 Menu bar

Menu bar includes several menus as shown in Figure 1.3, each of which will be introduced in detail. User can click the selected menu to reveal a drop-down menu, which may also include several options that can be selected for execution.

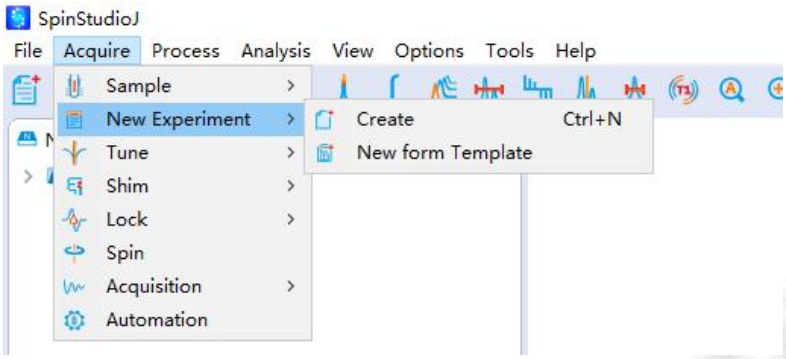


Figure 1.3 Menu bar

1.3.1 File

File menu is shown in Figure 1.4.

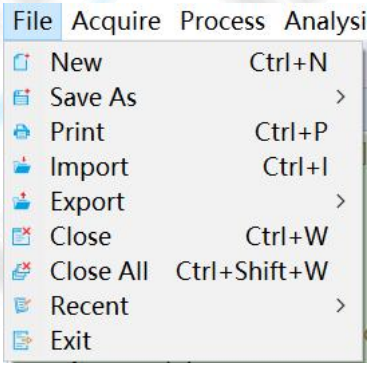


Figure 1.4 File menu

New:

Open New Experiment dialog box, which will be explained in detail in 2.5 New Experiment.

Save As:

Save data in current workspace as **Template** or **SpinStudioJ Data** (Figure 1.5).

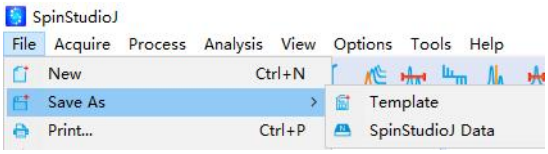


Figure 1.5 Save As option

Click **Template** to open a dialog box (Figure 1.6). In **Save Directory** item you can select **General** to save as general template or **Auto** to save as automation template. User can give a **Name** to your template. The item **Class** and **Dim** are decided by the current data, while **Type** can be selected.

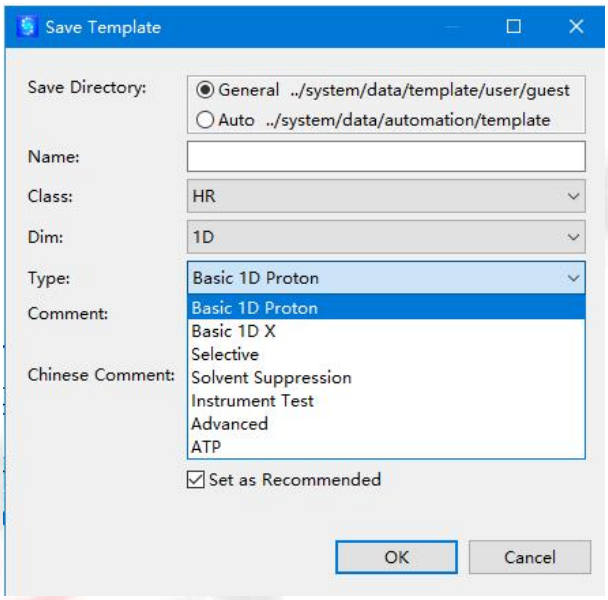


Figure 1.6 Save template dialog

User can also set up parameters for automation experiments by clicking Add Automation Parameter button to open a dialog (Figure 1.7), where both **General** and **Advanced** parameters can be selected according to your requirement.

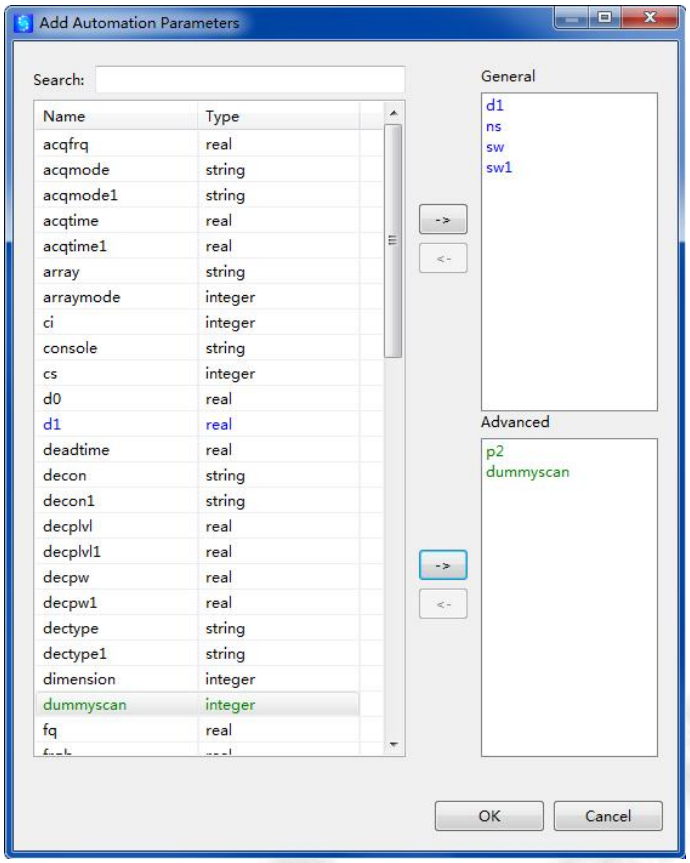


Figure 1.7 Add parameters dialog

Click **SpinStudioJ Data** to open a dialog box for data saving (Figure 1.8).

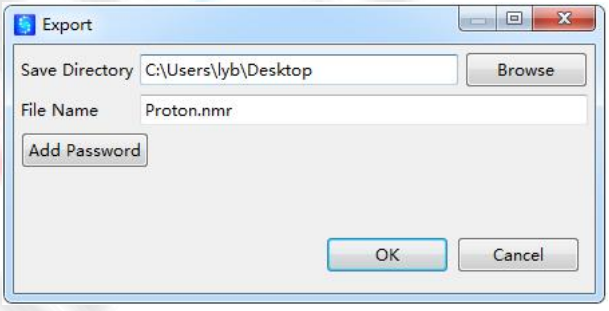


Figure 1.8 Save experiment data dialog

Print:

Enter the print/plot interface. User can edit spectra here. Please refer to **Chapter 9**

Spectrum Printing for detailed description.

Import:

Data import. User can import data of different format, including SpinStudioJ, Bruker and Agilent for processing. You don't need to select the specific format of data. Click **Browse** to find the directory where the data are saved, and the format will be recognized automatically. User can select all or some of the data under the defined directory. User have to choose the destination directory through **Import to folder** box. Check the box under Options will keep all your directory structure retained in destination directory. You can set password for your imported data by clicking **Add password** button (Figure 1.9).

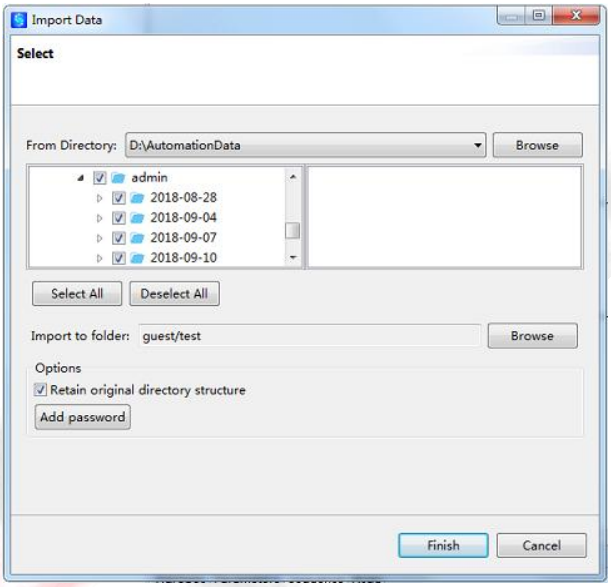


Figure 1.9 Data import dialog

Export:

Data export. User can export the data in current workspace to designated directory in **SpinStudioJ**, **Data format 1**or **Data format 2**(Figure 1.10). You can give a name to your experimental data as you like. If in **SpinStudioJ** format, you can set password for your exported data by clicking **Add password** button.

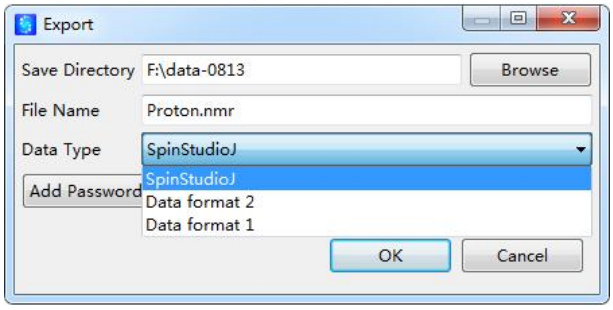


Figure 1.10 Data export dialog

If you want to export one of the array data, enter the command ***export1DArray***, and the dialog box will pop up as shown in Figure 1.11. Fill in the corresponding number of the data (the number range is displayed in the parent brace), that is, export the data to the current array data. Under the directory, and automatically named "current data name_export data number".

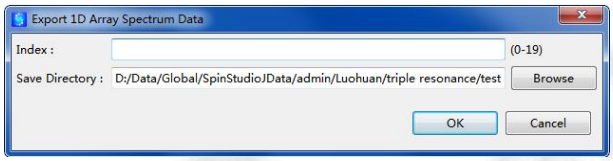


Figure 1.11 Export one of the array data

Close:

Close current workspace. This can also be done by clicking ⊠ button on the workspace tab. The third way is to put the mouse over the tab and righ-click to choose **Close**.

Close All:

Close all workspaces. You can also put the mouse over the tittle bar of the data window and righ-click to choose **Close All**.

Exit:

Exit the software. You can also click  button on the top right of the main window to exit the software.

1.3.2 Acquire

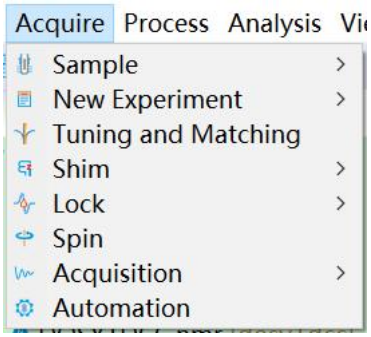


Figure 1.12 Acquire menu

Sample: Click **Eject** or **Inject** to control sample in and out.

New Experiment: Create New Experiment or New Template.

Tuning and Matching: Open tuning interface, referring 2.7 Tuning and Matching Shimming for details.

Shim: Open shimming interface, referring 2.8 Shimming for details.

Lock: Open lock interface, referring 2.6 Locking for detail.

Spin: Open Spin Rate Edit dialog box.

Acquisition: Start or stop acquisition, equivalent to the commands ***go*** or ***aa***.

Automation: Open automation interface, user name and password needed. Refer to 2.11 Automation for details.

1.3.3 Process

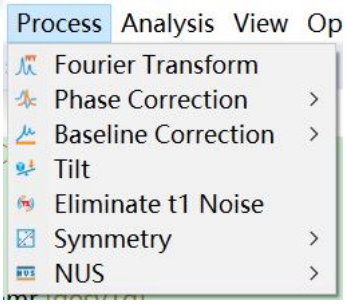


Figure 1.13 Data processing menu

Fourier Transform:

To open **Fourier Transform** dialog box. Details are referred to 4.3 Window and Fourier Transform and 5.2 2D Window and Fourier Transform.

Phase Correction:

To open Automatic or Manual phase correction dialog boxes, referring to 4.5 Phase Correction for details.

Baseline Correction:

There are three options included, **Automatic**, **Semi-automatic** and **Manual**.

Automatic: enables automatic baseline correction;

Semi-automatic: similar to **Manual** but with different parameters, referring to 4.6.3 Semi-automatic baseline correction for details;

Manual: enables manual base line correction by opening a dialog window to set appropriate parameters, referring to 4.6.4 Manual baseline correction for details.

Tilt:

Open a dialog box for tilt processing, referring to chapter 5.6 J Spectrum Tilt.

Elimination T1 Noise:

Eliminate T1 noise of 2D spectra, referring to 5.7 t1 Noise Elimination.

Symmetry:

Symmetrize the spectra to improve the quality. Two options for COSY-like and J-Resolved are included. Please refer to 5.8 Symmetry.

NUS:

Data processing for non-uniform sampling, referring to *NUS Manual*.

1.3.4 Analysis

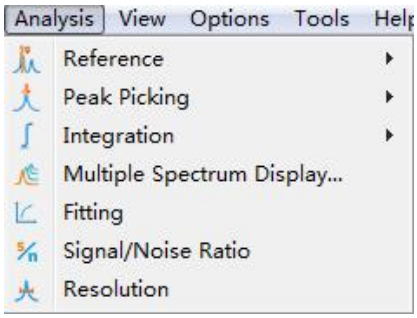


Figure 1.14 Data analysis menu

Reference:

Include **Automatic** and **Manual** methods. Clicking **Automatic** enables directly automatic referencing, while clicking **Manual** will open a dialog box to set the reference shift, referring 6.1 Reference.

Peak Picking:

Include **Automatic** and **Manual** methods. Clicking **Automatic** will directly display the results of peak picking, while clicking **Manual** will enter manual peak picking mode, referring to 6.2 Peak Picking.

Integration:

Enter manual integration mode, referring to 6.3 Integration.

Multiple Spectrum Display:

Enter Multiple Spectrum Display mode to manage the display and processing of multiple spectra in different workspace, referring to 6.4 Multiple Spectrum Display.

Fitting:

Enter data fitting mode for T1 relaxation, T2 relaxation and DOSY data, referring to 6.6 Fitting.

Signal/Noise Calculation:

Calculate signal to noise ratio, referring to 6.7 Signal/Noise Ratio.

Resolution:

Calculate the digital resolution of the spectrum and the line shape data for the highest peak within the displayed region of spectrum, equivalent to command ***res.***

1.3.5 View

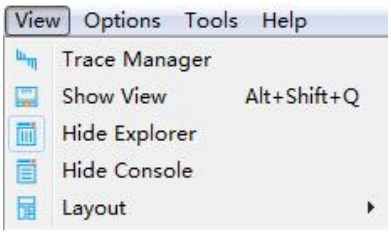


Figure 1.15 View menu

Trace Manager:

Open **Trace Manager** window tor manage the display of arrayed data, referring to 6.5 Array Data Display and Processing

Show view:

Open a window for display setting of the software appearance (Figure 1.16). User can choose to open the windows you want to display.

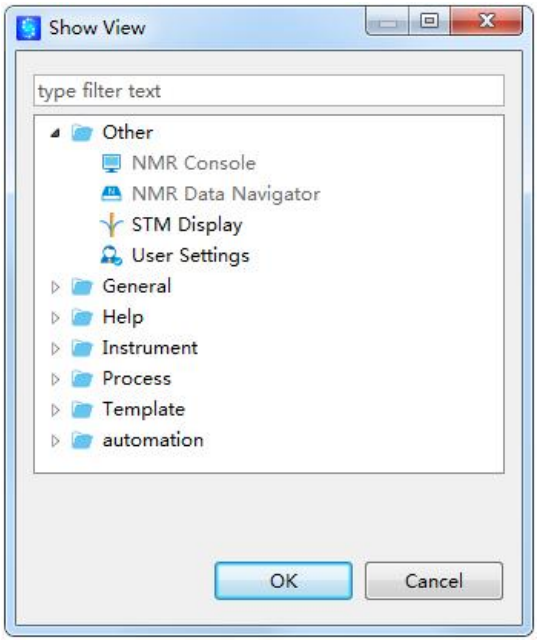


Figure 1.16 Show view dialog

Hide Explorer/Show Explorer:

Hide or show the Data Navigator.

Hide Console/Show Console:

Hide or show NMR Console window below the workspace window, including log panel and command line.

Layout:

Control the layout of multiple spectra display (Figure 1.17), which includes Horizontal (Figure 1.18), Vertical (Figure 1.19) and Stacked display (Figure 1.20). For Stacked layout, an offset must be set in pixel unit (Figure 1.21).

Figure 1.17 Layout menu

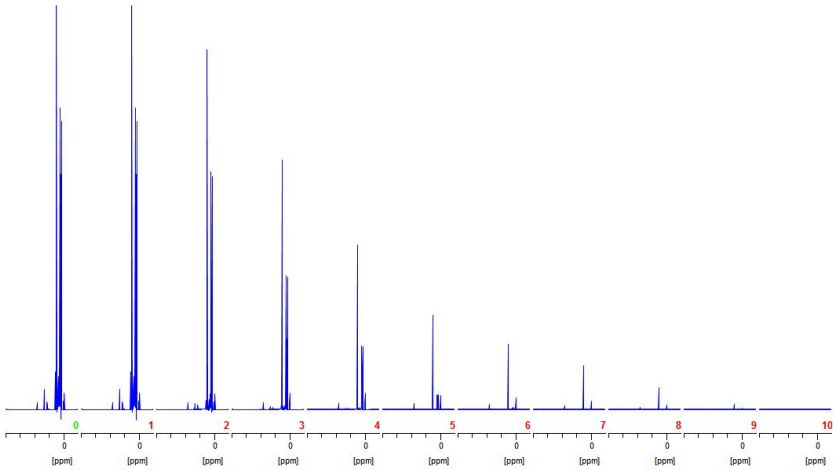


Figure 1.18 Horizontal layout view

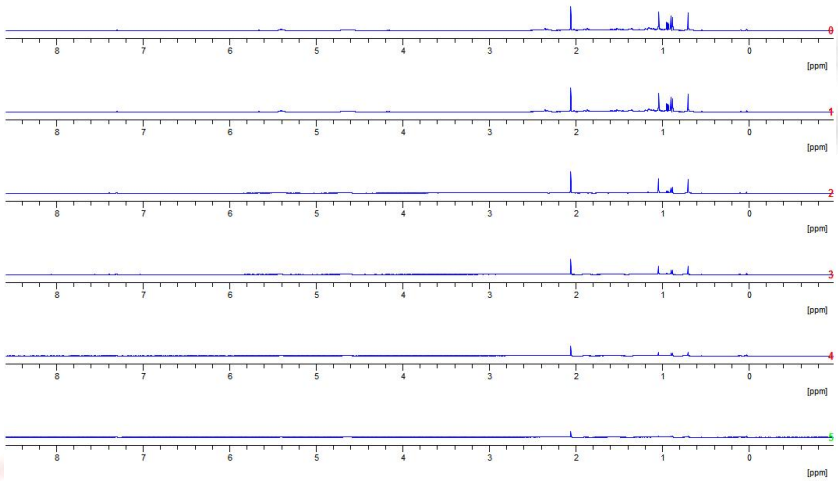


Figure 1.19 Vertical layout view

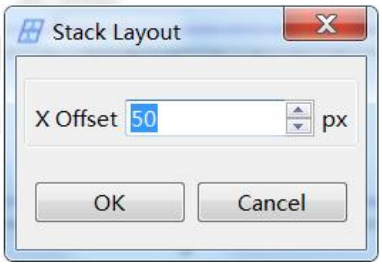


Figure 1.20 Stack Layout Offset dialog

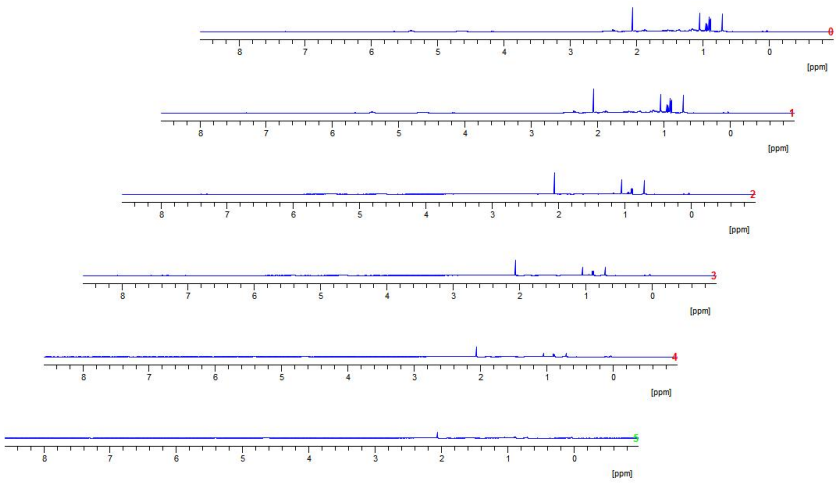


Figure 1.21 Stack layout view

1.3.6 Options

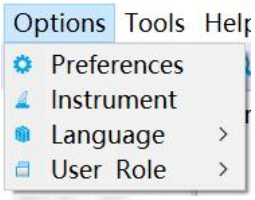


Figure 1.22 Option menu

Preferences:

To Configure the appearance of software interface, Automation experiments, Data Process, Shimming methods for the instrument, NMR Editor, Navigator, Print and Pulse Sequence Diagram (Figure 1.23).

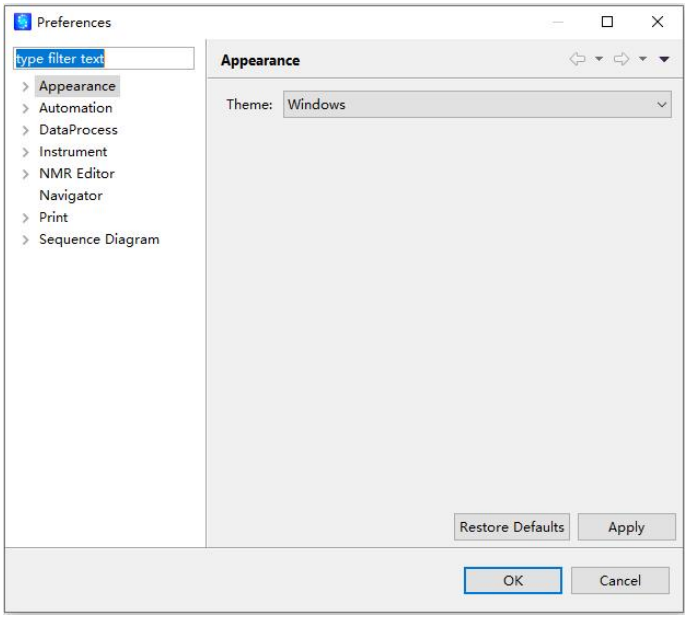


Figure 1.23 Software preference configuration interface

Appearance: to set the theme of the whole interface(Figure 1.24). User can select: Classic, Dark, Windows, Windows XP Blue, Windows XP Olive or Windows Classic.

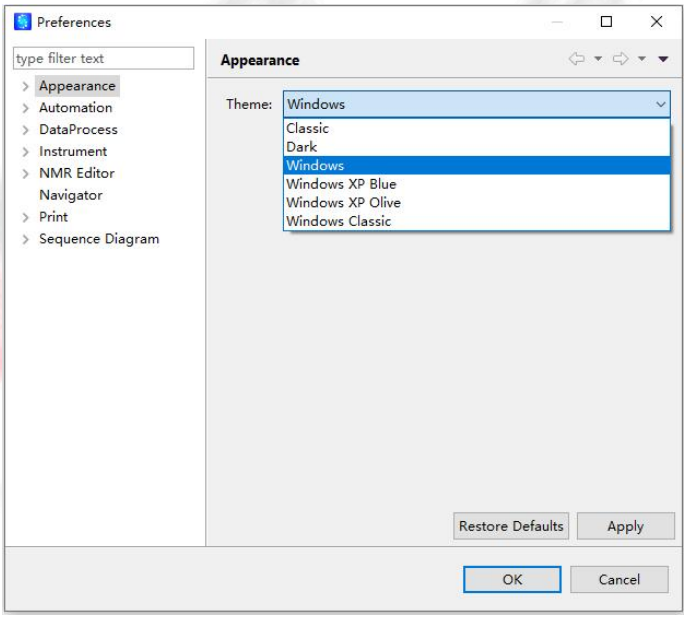


Figure 1.24 Software appearance configuration

Fonts: to configure the fonts used in Console, Navigator, Print interface, Status Line, and Automation Navigator(Figure 1.25). Click **Edit** to show the dialog box (Figure 1.26) for font selection, then the selected font name will be shown in the Preview window.

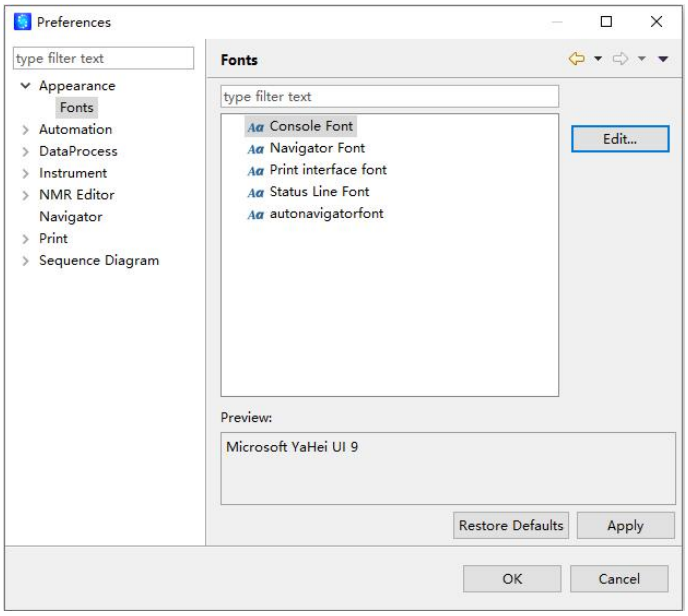


Figure 1.25 Font configuration dialog

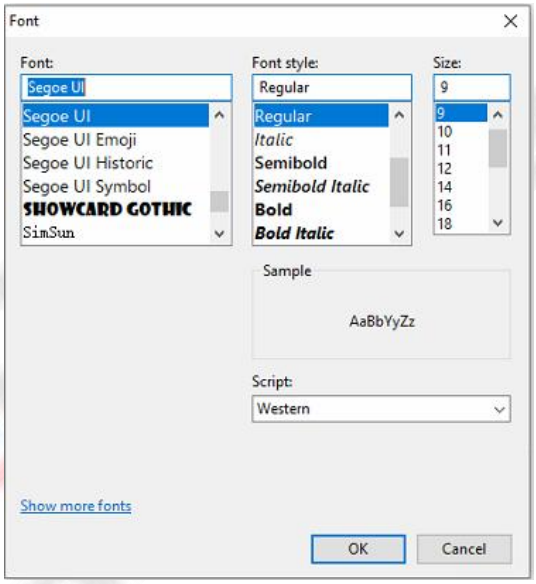


Figure 1.26 Font editing dialog

Automation: to set the conditions of log files for automation experiments (Figure 1.27). The page can be configured with: a. History limit, above which the earlier history is automatically cleared in the software interface. b. The history file interval is limited by the number of days, and a new history file is automatically created. c. History file size limit beyond which a new history file is automatically created. d. The history rolls back the initial number, and when the history file is re-created, the latest pieces are kept in the software

interface.

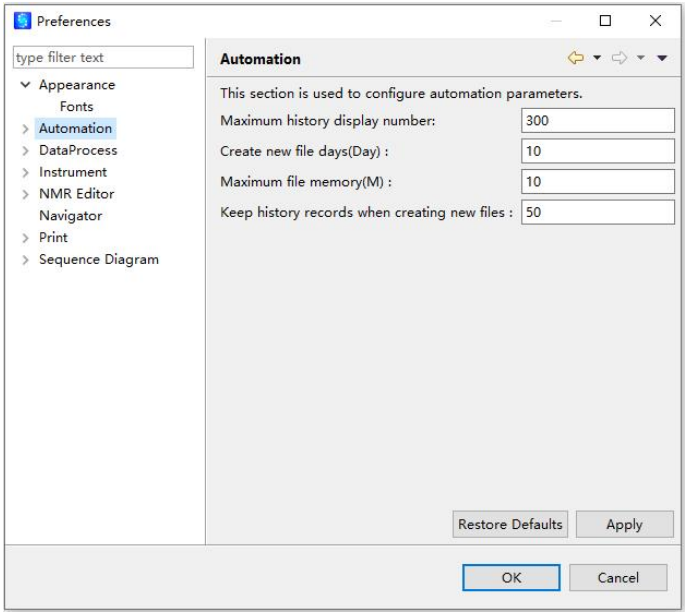


Figure 1.27 Automation history configuration

Automation > **Tuning After Gradient Shimming**: to configure the combination of shim coils for search shimming after auto shimming (Figure 1.28). User can add or delete shim coil in this window, or change the sequences of coil combinations.

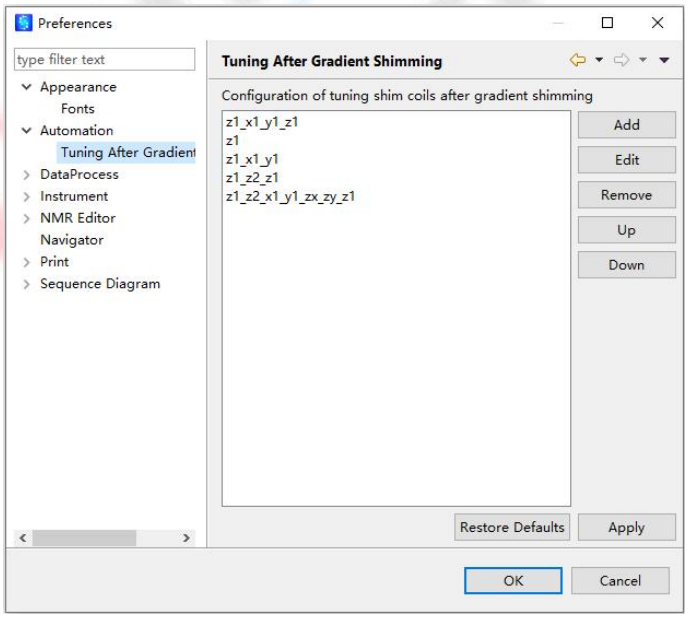


Figure 1.28 configure the combination of shim coils for search shimming after auto shimming

DataProcess: to set the MestReNova installation path for the data processing software(Figure 1.29). After the configuration is completed, open the data in the workspace,

and enter the command ***mnova*** in the command line to open the data of the current active workspace in the MestReNova installed in the workstation for post-processing of the data.

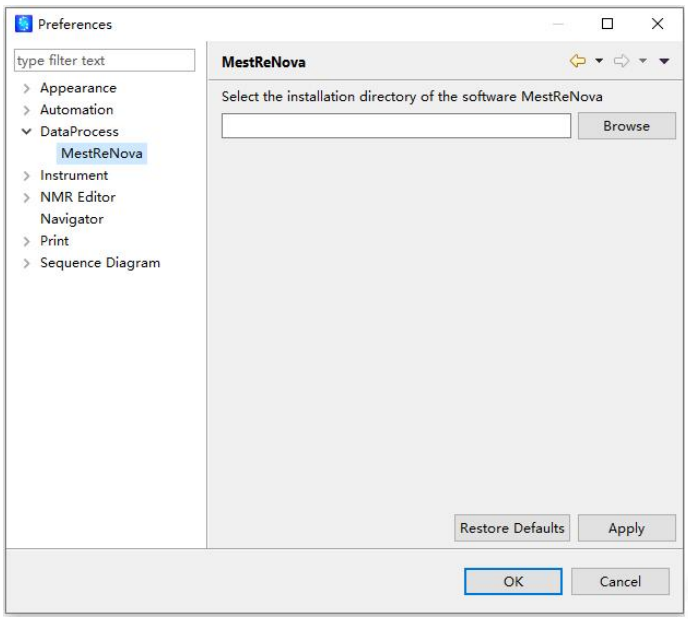


Figure 1.29 MestReNova installation path setting

Instrument > **Shimming** > **3D Smart Shimming**: to configure the shim coil combinations for search shim before and after 3D Smart Shimming (Figure 1.30).

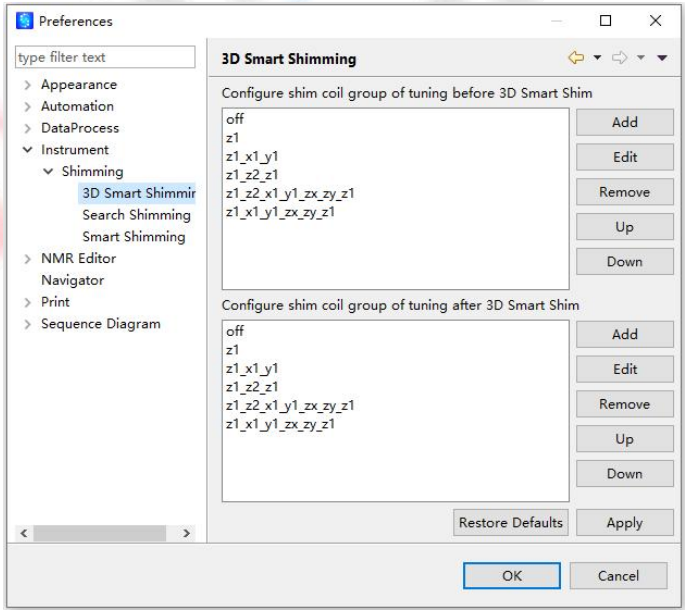


Figure 1.30 Search shimming configuration before/after 3D Smart Shimming

Instrument > **Shimming** > **Search Shimming**: to set the configurations for search shim (Figure 1.31). User can add or delete shim coils and their combinations or change the

sequences of them.

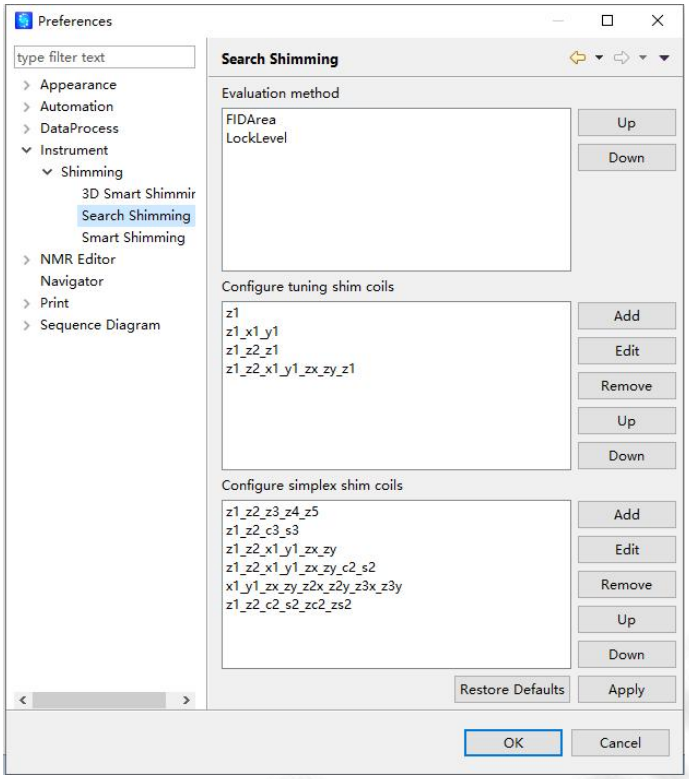


Figure 1.31 Search shimming configuration

Instrument > **Shimming** > **Smart Shimming**: to set the configuration of tuning shim before and after Smart Shimming (Figure 1.32).

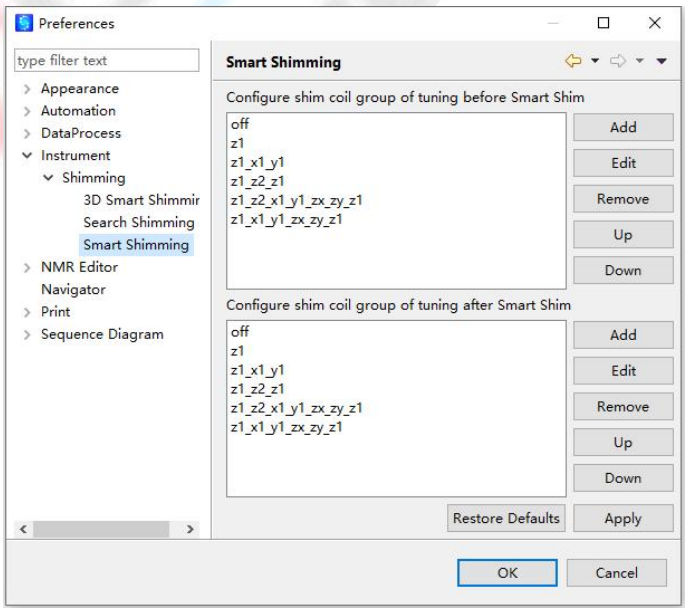


Figure 1.32 Smart shimming configuration

NMR Editor: to set the maximum number of opened workspace, the maximum number for history display, maximum spectral number for 1D array display and maximum spectral number for multiple spectrum display (Figure 1.33).

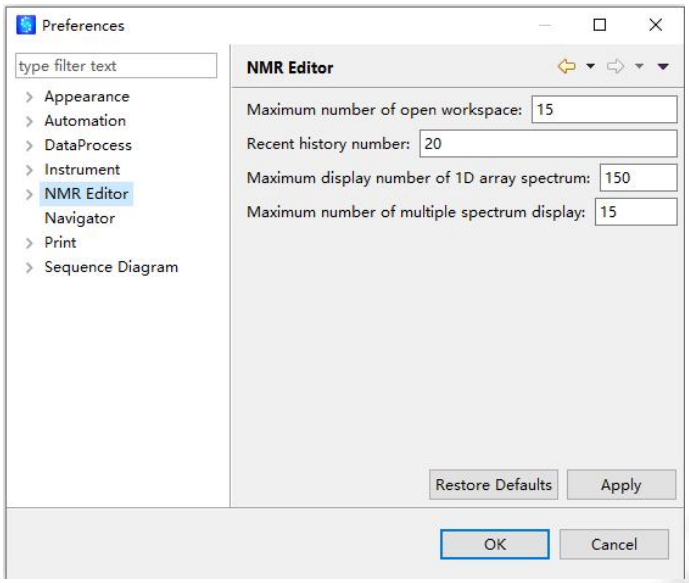


Figure 1.33 NMR Editor configuration

NMR Editor > **FID/Spectrum**: to set the parameters for FID/spectrum display (Figure 1.34). If no specific display parameter was correlated to specified experiment, the default parameters in this window will be used.

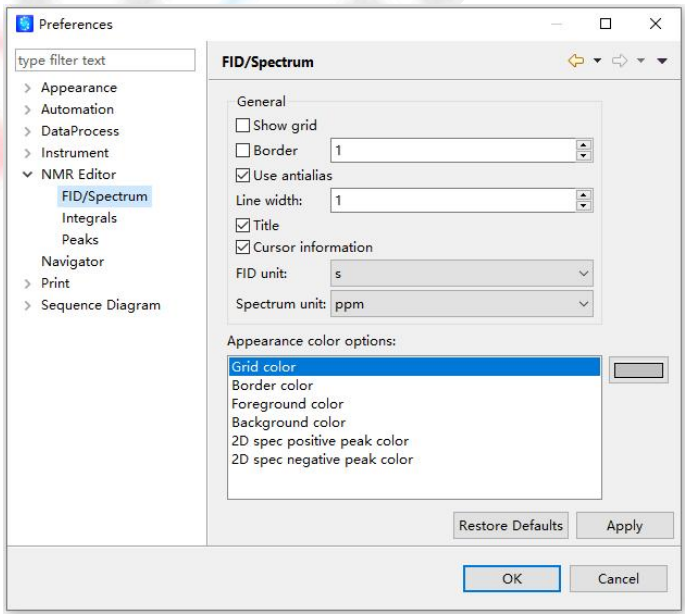


Figure 1.34 FID/spectrum display configuration

NMR Editor > **Integrals**: to set the integral accuracy, that is, set the decimal place of the integral value (Figure 1.35).

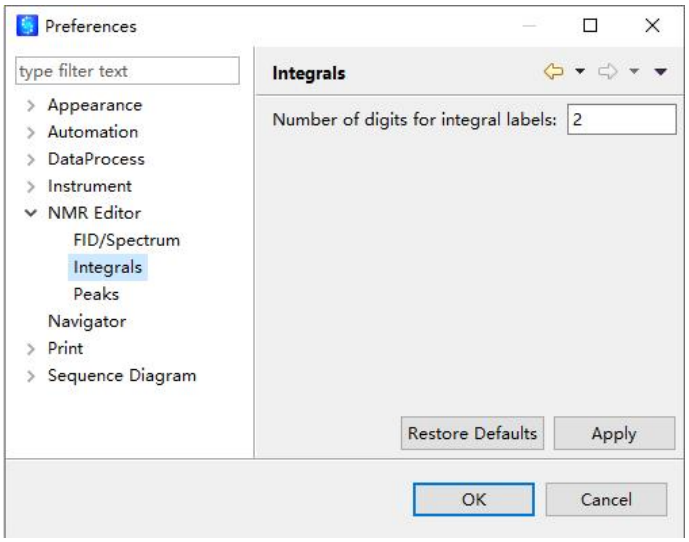


Figure 1.35 Accuracy configuration for integral

NMR Editor > **Peaks**: to set the peak accuracy, that is, set the decimal place of the peak value (Figure 1.36).

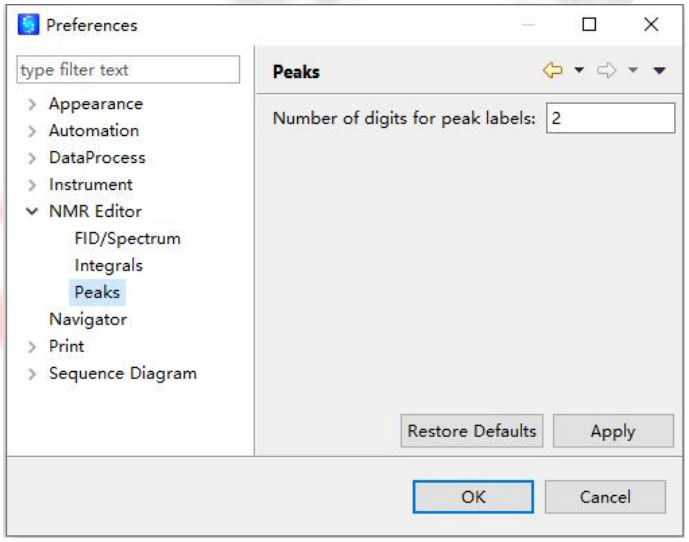


Figure 1.36 Accuracy configuration for peaks

Navigator: configure to select whether to show pulse sequence name in experiment node and file path on the right side of the username(Figure 1.37).

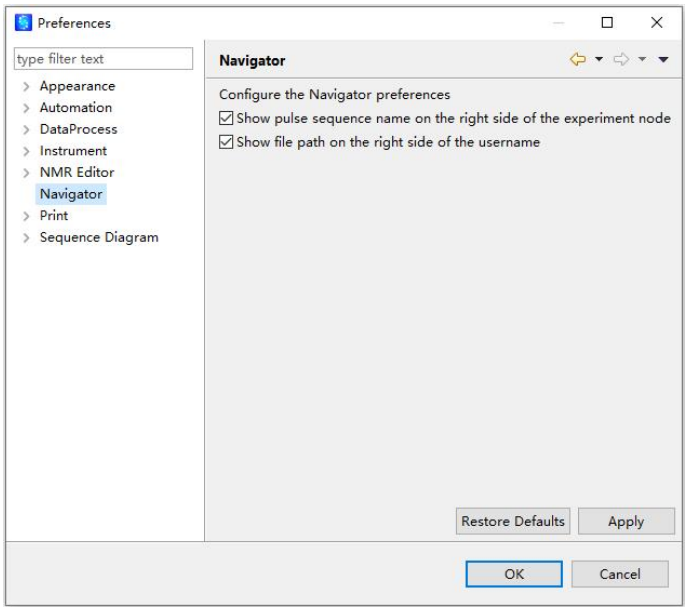


Figure 1.37 Navigator configuration

Print > Export: Configure the default resolution (dpi) for exporting images from the print interface(Figure 1.38). The optional values are 120, 360, 600, and the default setting is "120".

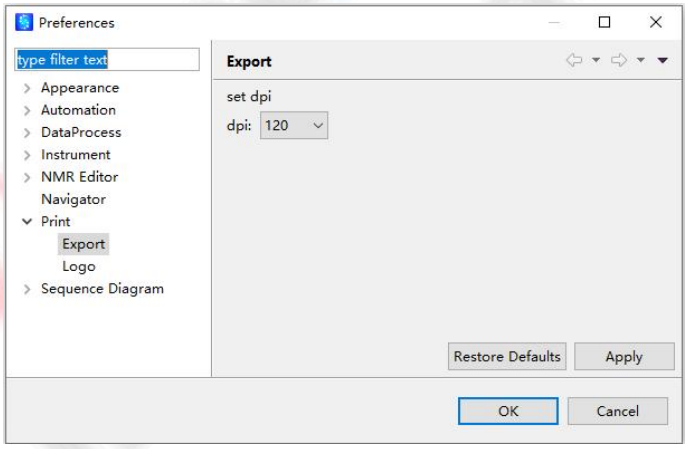


Figure 1.38 Default resolution settings for exporting images

Print > Logo: The default logo configuration for the print interface(Figure 1.39). Use the default blank to display our logo, or you can also click the **Browse** button to select other image as logo. The image format can only be PNG or JPG.

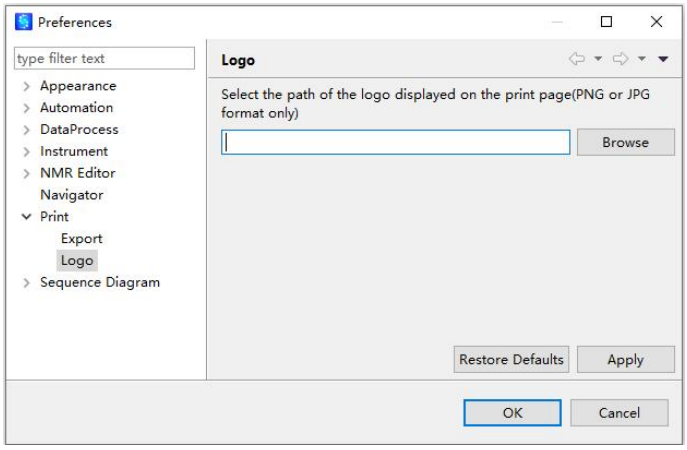


Figure 1.39 Default logo configuration for the print interface

Sequence Diagram > **Appearance**: to configure the colors to be used for pulse sequence display (Figure 1.40).

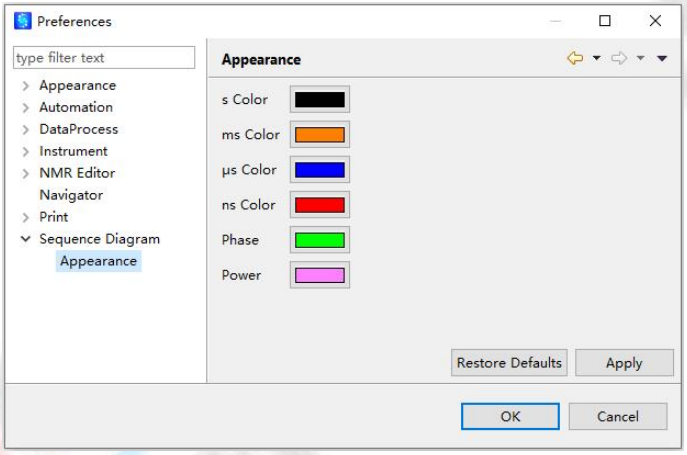


Figure 1.40 Pulse sequence appearance configuration

Instrument:

The inherent parameters for the spectrometer preset by installation engineer, user changing not recommended.

Language:

Switch languages between Chinese and English.

User＆Role:

For user management and role management, user management and role management

dialog boxes can be opened respectively. A workstation can have multiple operating system users. When switching to different operating systems and logging in to SpinStudioJ, there will be a default administrator account, that is, “admin”.

You can only use admin to log in to the software for the first time. After logging in, you can open the user management dialog box for user management at any time. Before opening the dialog box, the password input box will pop up. Enter the correct administrator password (the default password is “zhongkeMR”) to open the user management dialog box, as shown in Figure 1.41.

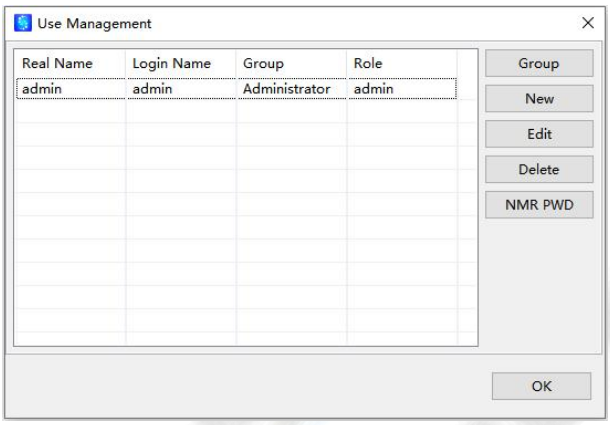


Figure 1.41 User management dialog box when administrator account is logged in

Click the button **Group** to manage operating system users, as shown in Figure 1.42.

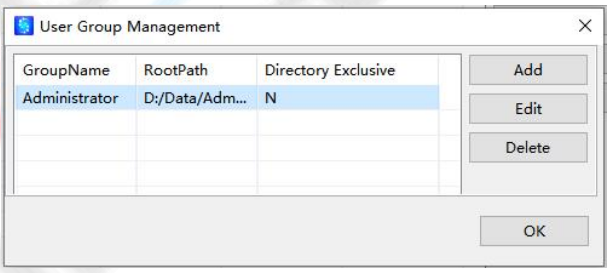


Figure 1.42 Operating system user management dialog box

Click the **Add** button to add an operating system user(Figure 1.42). After filling in the “Group Name”, the “Root Path” will be automatically generated. The “Directory Exclusive” below, the current operating system user can only set whether their own file directory is exclusive after logging in, so in Figure 1.43, it’s not editable. After adding, click the **OK** button to automatically create an operating system account in the background.

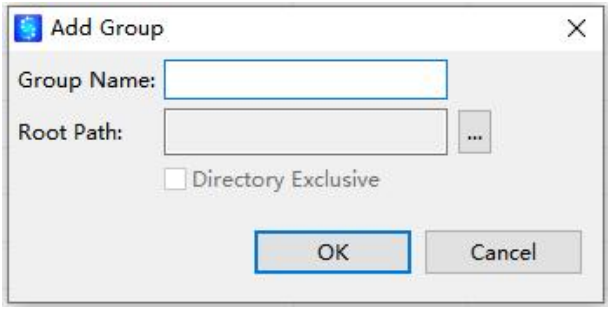


Figure 1.43 Add operating system user

Select an operating system user and click **Edit** to make relevant settings for the operating system user. As shown in Figure 1.44, select the current operating system account. At this time, “Directory Exclusive” can be checked, and settings such as Group Name and Root Path can be edited and changed. If other operating system accounts are selected, a prompt box will pop up (Figure 1.45).

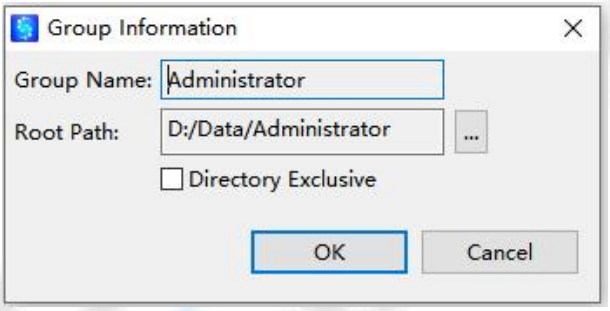


Figure 1.44 Edit current operating system account information

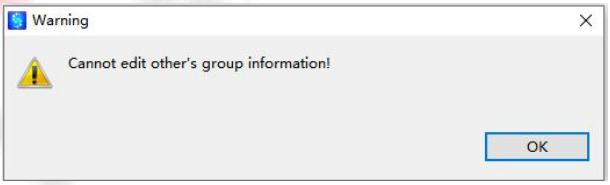


Figure 1.45 Pop-up warning when editing other operating system accounts

Click the button **New** in the user management dialog box (Figure 1.41) to create a new user, as shown in Figure 1.46. Enter the real name (Real Name), login name (Login Name), password (Password), select the user role (Role), and select the user's operating system account (User Group) from the drop-down list. Check whether the experimental data is encrypted. If it is encrypted, enter the password in the Data Password below. You can also set

the relevant permissions of electronic signature (ESignature Meanings), including review, approval, responsibility, authorship, etc.

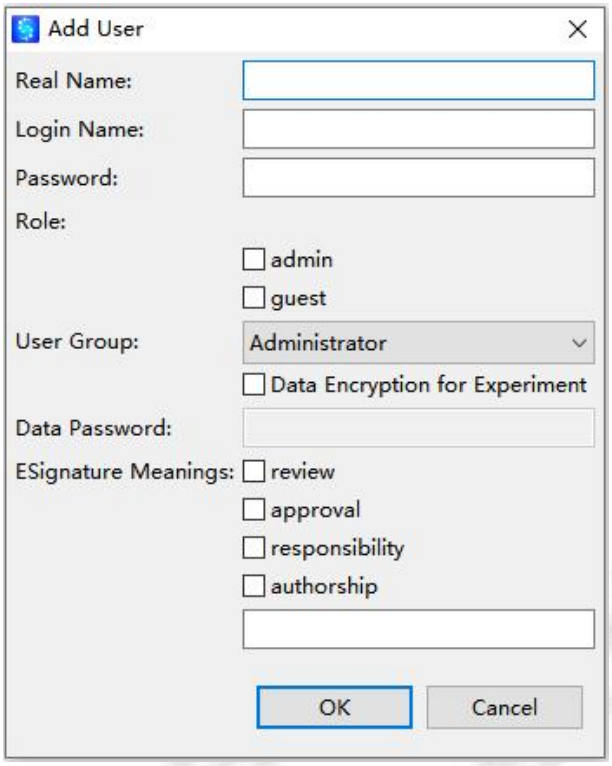


Figure 1.46 Add a new user

Click **Edit** in the user management dialog box (Figure 1.41) to edit the user. The edit dialog box is shown in Figure 1.47, which is basically the same as the new user dialog box (Figure 1.46). The only difference is that the user group cannot be selected at this time.

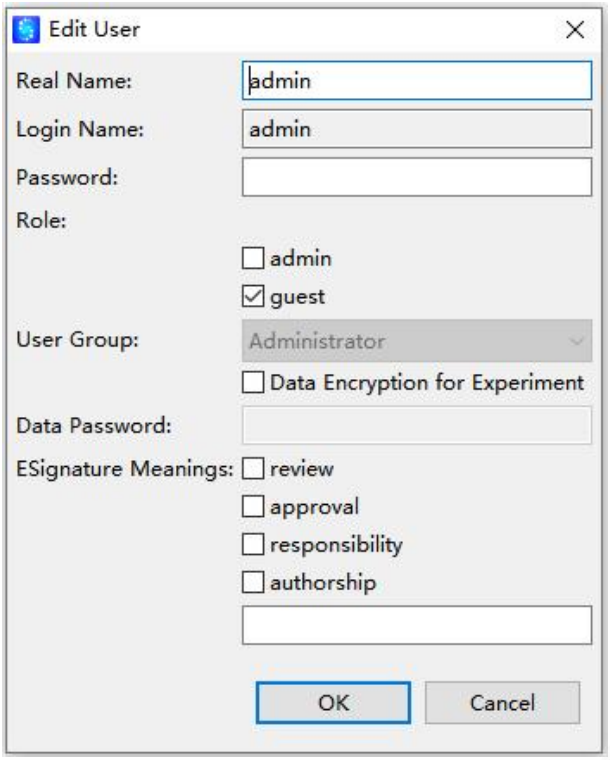


Figure 1.47 Edit User

If you log in to SpinStudioJ as an ordinary user, the user management dialog box is shown in Figure 1.48, and there is no permission to edit the user.

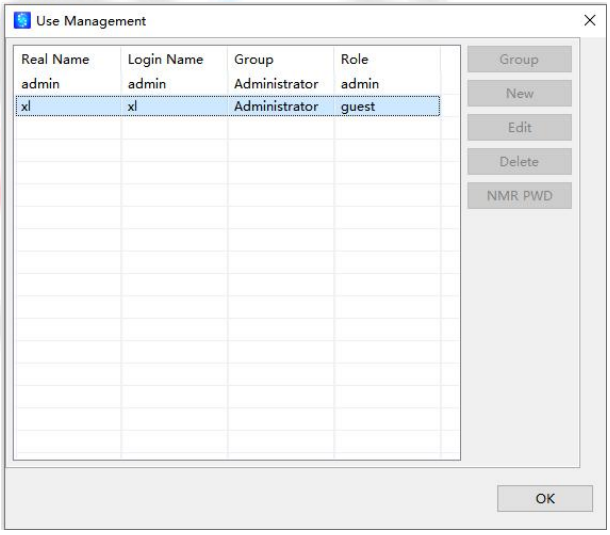


Figure 1.48 User management dialog box when ordinary user is logged in

1.3.7 Tools

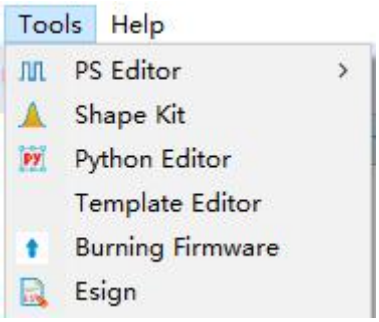


Figure 1.49 Tool menu

PS Editor:

Pulse sequence editor. User can create new pulse sequence by editing an existing pulse sequence in library, or open an pulse sequence in other place to edit. Detailed function description refers to 3.4 PS Editor.

Shape Kit:

Shape pulse tool. For detailed function description, refer to the manual *Shape Kit*.

Python Editor:

Python editor. For detailed function description, refer to the *Python Editor Manual*.

Template Editor:

Template editor. After clicking, a template selection box will pop up (Figure 1.50). Select any template and click **Select** to open the template editor (Figure 1.51). At this time, you can directly modify parameter values and attributes, and the changed cell contents will turn red for differentiation.

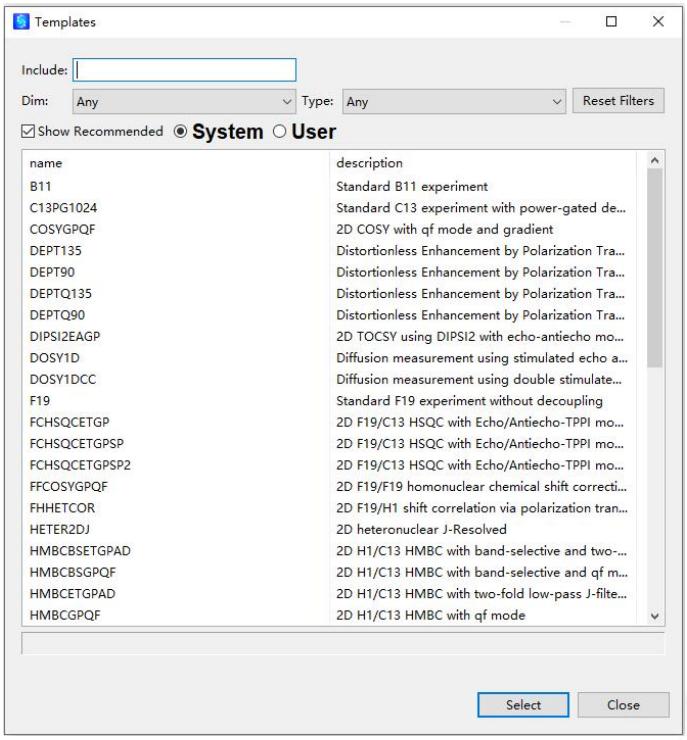


Figure 1.50 Template selection box

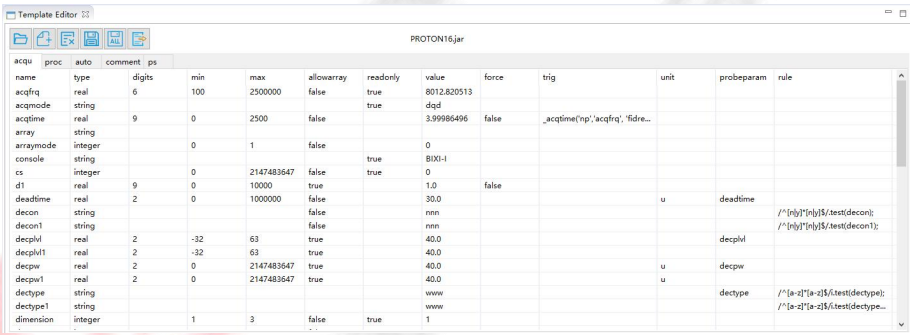


Figure 1.51 Template editor interface

: Open Template. After entering the template editor, if the current template has not been modified, click this button, the template selection box (Figure 1.50) will pop up directly, and you can switch to other experimental templates. If the current experimental template is changed, a box will pop up to remind you (Figure 1.52). Select **Yes** to pop up the template selection box, and select **No** to return to the current template.

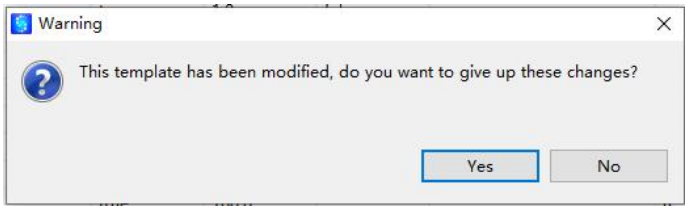


Figure 1.52 Prompt box for switching templates

: Add Parameter. After filling in the name and other information of the new parameter （Figure 1.53）, click the **OK** button to add it successfully. The text in the added parameter line is all red.

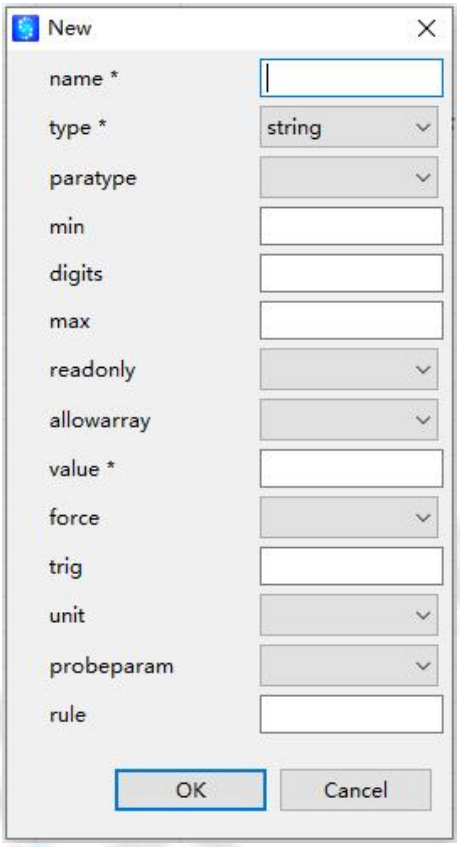


Figure 1.53 Dialog box for adding parameters

: Delete. Select the parameter row or attribute column, and click the delete button. The selected row or column will be marked with a black shading to indicate that the deletion was successful.

: Save. After the template is changed, click the **Save** button, and the parameter modification record table will pop up (Figure 1.54). After confirming, click the **OK** button, and the box will pop up again to remind whether the current template is covered (Figure 1.55). Selecting **Yes** will directly cover the system template. Selecting **No** will be saved under the current system user.

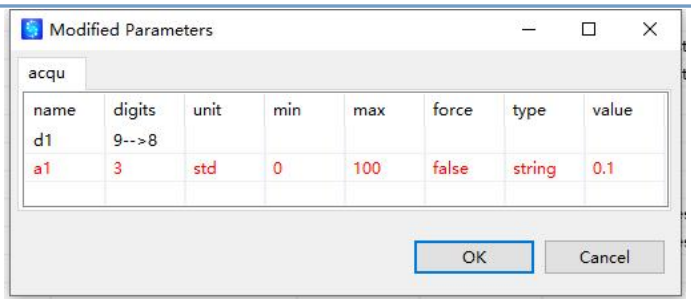


Figure 1.54 Parameter modification record

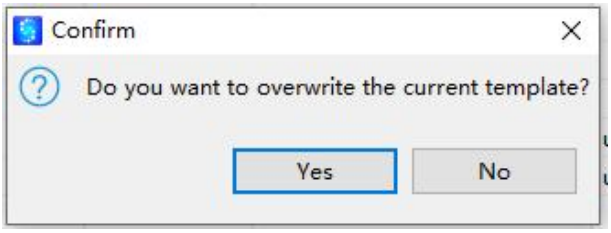


Figure 1.55 Confirmation box for overwriting template

: Save All, save changes to all templates. After the current template modification is completed, click , the template selection box will pop up (Figure 1.56). You can check part of the templates on the left alone, or you can check **Select All** above to directly select all templates, and then click **OK**. The cover template confirmation box in Figure 1.55 will pop up at this time. Selecting **Yes** will directly cover the corresponding template of the system, and selecting **No** will save it under the current system user.

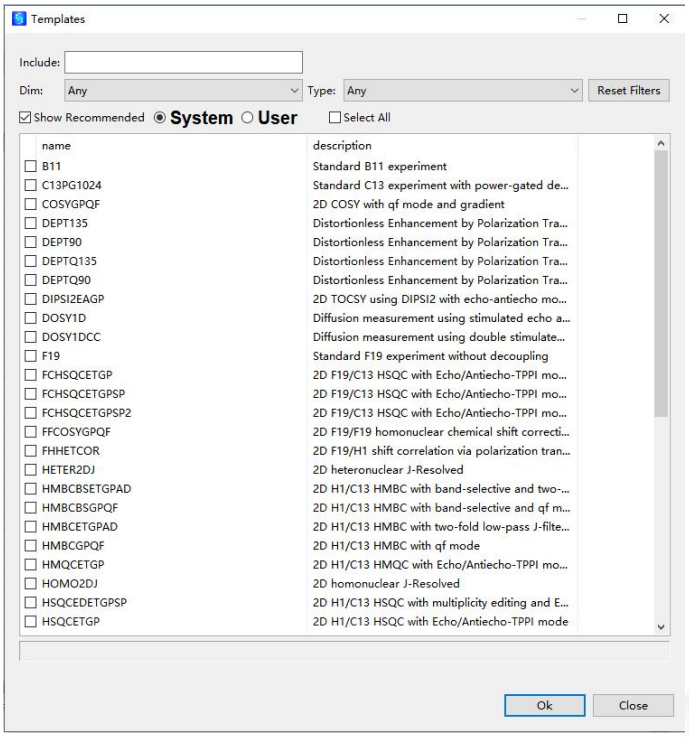


Figure 1.56 Template selection box

: Exit. Exit the template editor.

Burning Firmware:

Open the burning program dialog box (Figure 1.57), select the firmware module in the TargetFW drop-down box, then click **Browse** to select the program file path, and click the **Burning** button to burn the program. After the firmware module is selected, click the **Get Version** button to obtain the current firmware version number, and click the **Get All Version** button to display the version information of all modules in the log bar of the software main interface.

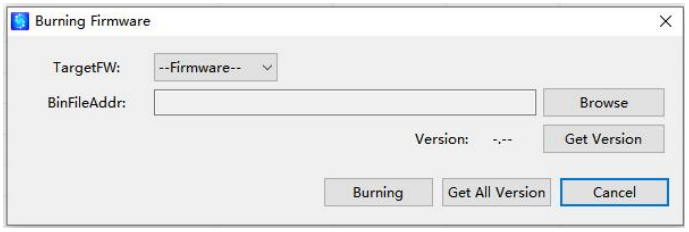


Figure 1.57 Burning program dialog

Esign:

Electronic Signature. After the current user is given electronic signature permission, click it to open the electronic signature dialog box (Figure 1.58), and then you can evaluate the current workspace data.

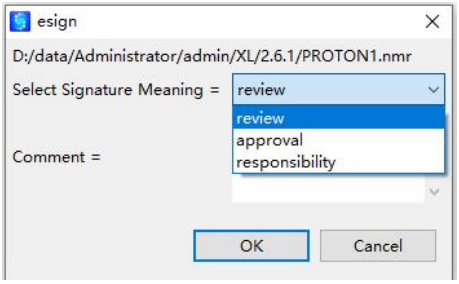


Figure 1.58 Electronic signature dialog

The details of the electronic signature can be viewed by right-clicking on the data navigation bar and selecting “Show Electronic Signature”, as shown in Figure 1.59.

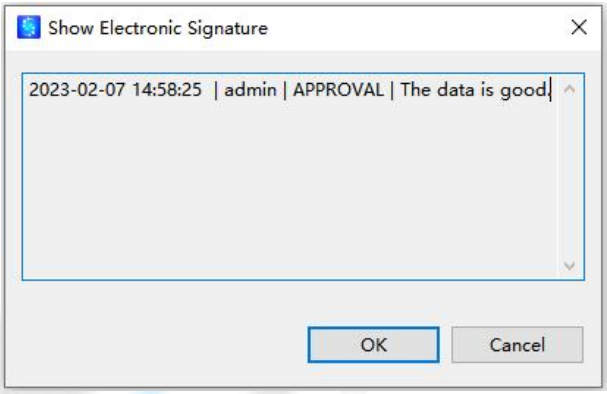


Figure 1.59 Electronic signature

1.3.8 Help

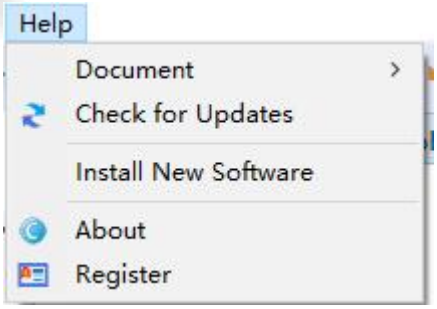


Figure 1.60 Help menu

Document: Help file. Click to open the *SpinStudioJ User Manual* or *SpinStudioJ*

Command and Parameter Manual.

Check for Updates: to check if there is any updates.

Install New Software: to install software plug-ins. Please refer to *SpinStudioJ Installation Manual* for details.

About: to display the software information of current version.

Register: Registration authorization for SpinStudioJ. Please refer to *SpinStudioJ Installation Manual* for details.

1.4 Toolbar

Toolbar includes buttons for acquisition and display.

Acquisition related buttons

Acquisition related buttons are shown in Figure 1.61, most of which have corresponding items in menu bar. For example, user can click button for peak picking or click **Analysis** > **Peak Picking**.

Figure 1.61 Acquisition related buttons

- New Experiment
- Save Template
- Print
- Manual Phase Correction
- Manual Base Line Correction
- Manual Calibration
- Manual Peak Picking
- Manual Integration
- Multiple Spectral Display
- Peak Distance
- Trace Manager
- Set Spectral Center

- Set Spectral Width
- Eliminate t1 Noise
- Start
- Stop
- Automation
- Tuning
- Lock
- Shimming

Display related Buttons

Display related buttons are shown in Figure 1.62, which include scaling and moving of FID and spectrum displayed in workspace, as well as insert display, row and column display.

Figure 1.62 Display related buttons

1.5 NMR Data Navigator

NMR Data Navigator provide an experimental data management tool for users. User can open, create, copy, paste, display and delete data in it. You can choose navigator template according to your occupation. The default template is for university, while templates for testing center and pharmaceutical company are also included (Figure 1.63). New template can be created by administrator using command ***syscf***, referring to *SpinStudioJ Installation Manual*.

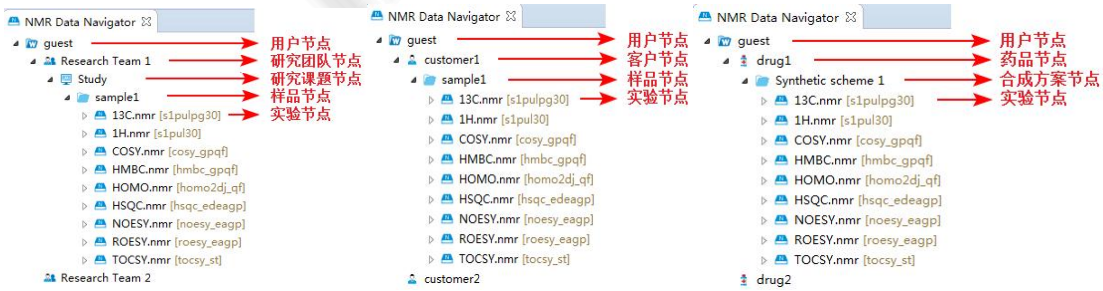


Figure 1.63 NMR Data Navigator

There are two useful buttons in the right of the title bar of navigator area. You can click the button to close all the pop-down structure in navigator, and click button to spread it and navigate to the position corresponding to the active workspace. Click the button to minimize the window or button to maximize it, and click button to recover it.

In the next section we will introduce the right-click menus based on university template. The templates for testing center and pharmaceutical company are similar.

1.5.1 User Node

The username displayed in user node is the same as the login username. Username includes admin and user created name. Only administrator can use admin to log in for setting up the system. Normal users can use self-defined username to log in.

The right-click menu is shown in Figure 1.64. User can create research team (New Research Team), or directly create experiment (New Experiment). New Research Team can include directories, while New Experiment is a .nmr file.

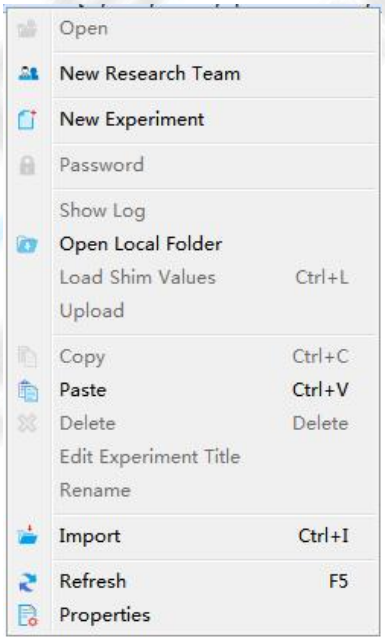


Figure 1.64 The right-click menu of user node

① New Research Team: Create a directory for new team (Figure 1.65). The default directory is under the current user node. Fill in the name of your team, then click **Finish** to close the window.

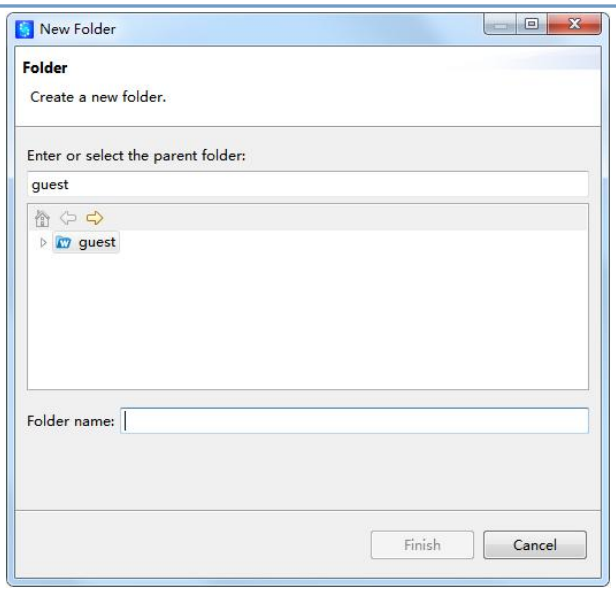


Figure 1.65 Create a new research team dialog on the user node

② New Experiment: Create new experiment in current user (Figure 1.66). You can use the current directory or choose a new one, then fill in a name for the experiment, and choose the parameter type. If there is now data opened in workspace, the default parameter type is template and the default template is PROTON16; on the other hand, the default type is Current workspace parameter, i.e., using the parameters of the active workspace. Finally, click **Finish** to complete the creation.

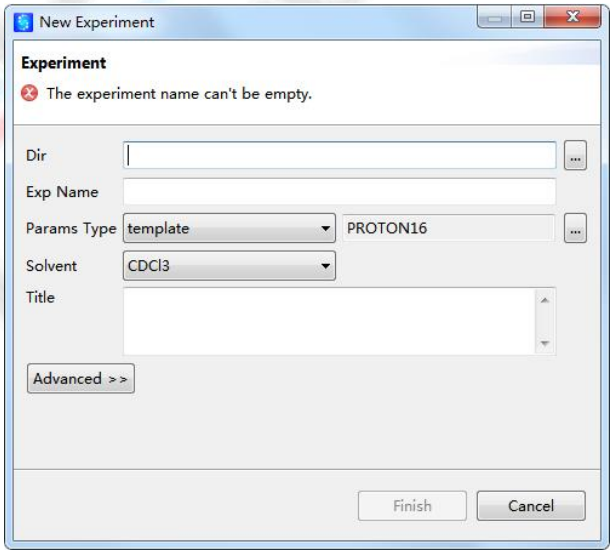


Figure 1.66 Create a new experiment dialog on the user node

③ Open Local Folder: open the directory of current user in File Manager.

④ Upload: upload data to the predefined FTP server. The FTP server can be configured using command ***syscf***.

⑤ Import: import data. A dialog box will be opened and you can select the directory to find the data you want to import. The default directory is the user's directory (Figure 1.67). Refer to 1.3.1 File for other functions.

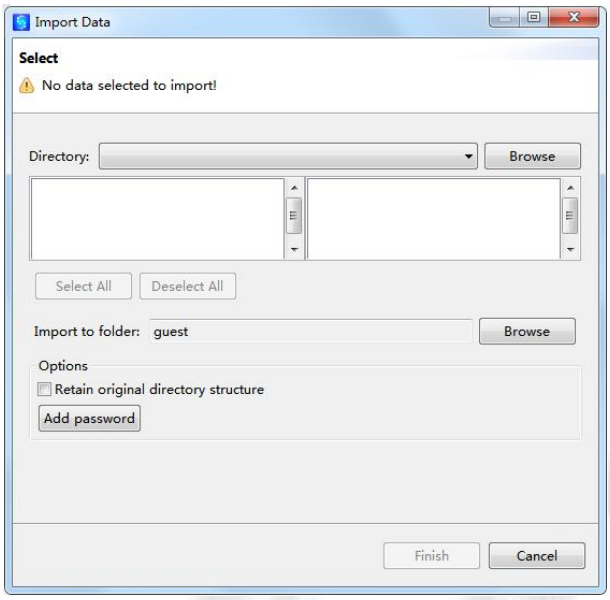


Figure 1.67 Data import dialog in user node

⑥ Refresh: Refresh the data navigation interface

⑦ Properties: The property panel will pop up on the right side of the workspace, which mainly contains the user name, local path and last modification time, as shown in Figure 1.68.

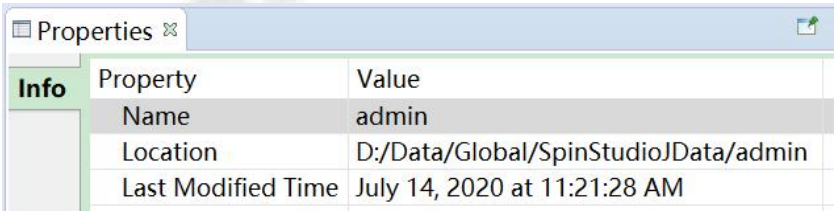


Figure 1.68 Right-click property panel for user node

1.5.2 Research Team node

When a Research Team node is created, you can also use right-click menu to execute many functions (Figure 1.69).

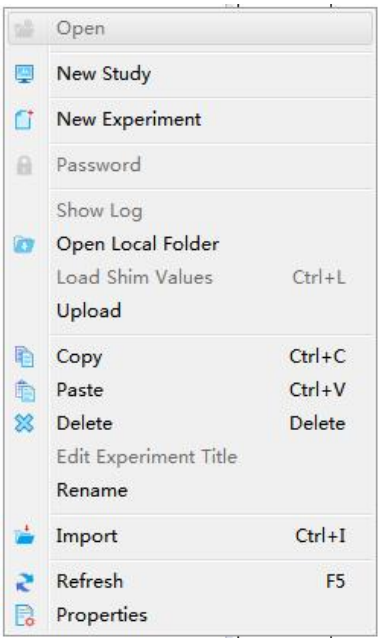


Figure 1.69 Right-click menu in research team node

① New Study: Create a new research project (Figure 1.70), which is under Research Team path and can further contain directories.

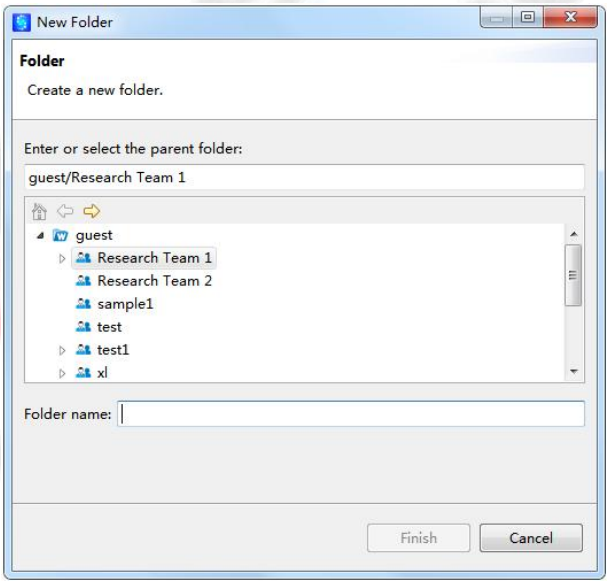


Figure 1.70 New research project dialog in the research team node

② New Experiment: create new experiment under current research team directory. A dialog box will be opened (Figure 1.71), and the settings are similar to those described in the New Experiment under User node.

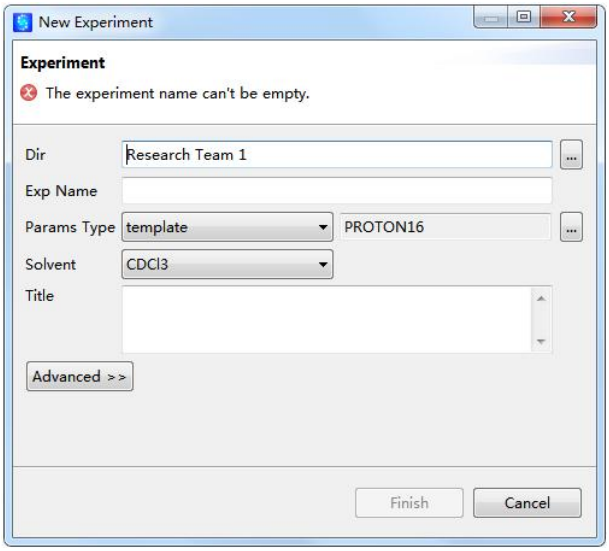


Figure 1.71 New experiment dialog under the research team node

③ Open Local Folder: open the directory of current Research Team in File Manager.

④ Upload: upload data to the predefined FTP server. The FTP server can be configured using command ***syscf***.

⑤ Delete: Delete the selected Research Team node.

⑥ Rename: Rename the Research Team.

⑦ Import: import data. A dialog box will be opened and you can select the directory to find the data you want to import. The default directory is the user's directory.

⑧ Refresh: Refresh all data under the research team.

⑨ Properties: The property panel will pop up on the right side of the workspace, which mainly contains the name, local path, and last modification time of the research team, as shown in Figure 1.72.

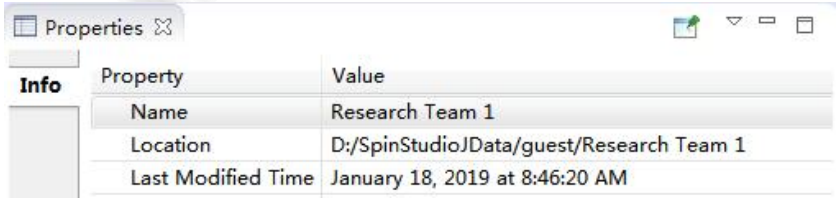


Figure 1.72 Right-click property panel for research team node

1.5.3 Research Project node

User can create new research project (Study) under current Team node, and the

right-click menu is shown in Figure 1.73.

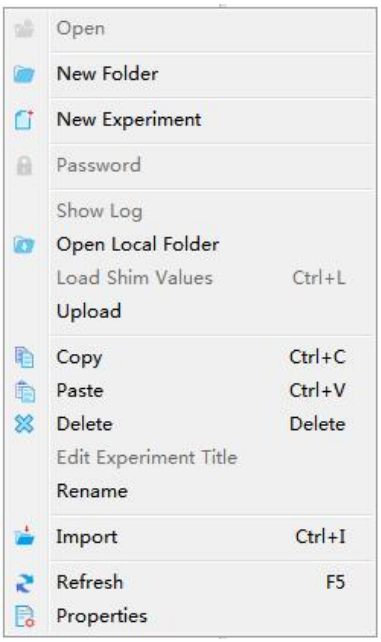


Figure 1.73 Right-click menu in research project

All operations are the same as previous description, but under current Study directory.

1.5.4 Folder node

User can create new Folder under Study node, which may be the directory for some sample. The right-click menu and correlated operations are similar to the previously described.

1.5.5 Experiment node

This may be the last node under the whole directory structure, created by New Experiment in right-click menu, with the name of any kind of structure correlated with current experiment. It is a .nmr file composed of several components. Double-clicking the node will open a new window in workspace to show the data.

The right-click menu is shown in (Figure 1.74), which is a little bit different from previous menus.

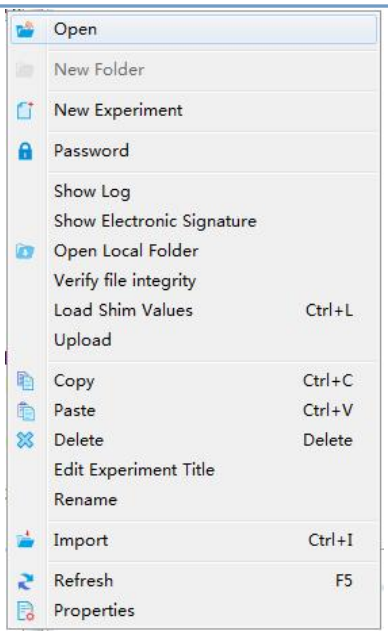


Figure 1.74 Right-click menu in experiment node

① Open: open experimental data in workspace.

② New Experiment: create new experiment parallel to the current experiment. The default Params Type is current node parameters.

③ Password: add password to the experiment (Figure 1.75). After entering the password and confirming it in the pop-up dialog box, click the button OK to save it.

Figure 1.75 File encryption dialog

④ Show Log: Display data acquiring, processing and other records, as shown in Figure 1.76.

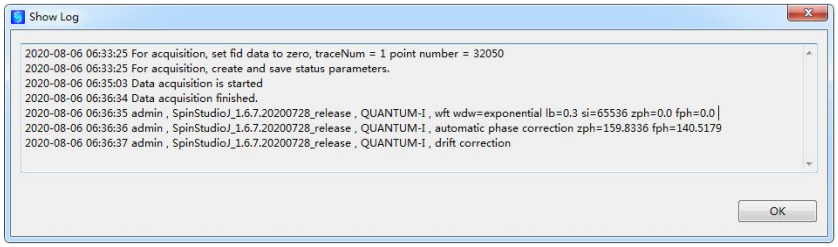


Figure 1.76 Dialog of showing log

⑤ Show Electronic signature: Display the electronic signature information of the data, as shown in Figure 1.77.

Figure 1.77 Electronic signature dialog

⑥ Open Local Folder: open the directory of current experiment data in File Manager.

⑦ Verify file integrity: Verify the integrity of the data. This item can be used to check whether the data has been illegally modified, as shown in Figure 1.78.

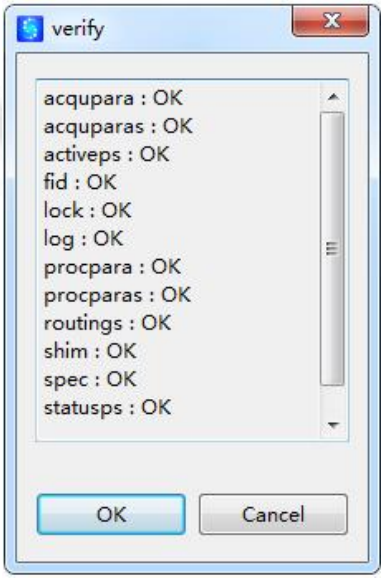


Figure 1.78 Data integrity verification

⑧ Load Shim Values: retrieve the shim values from a shim file. If your shimming is not good, you can select a saved data with good shimming to retrieve it.

⑨ Upload: upload data to the predefined FTP server.

⑩ Copy: copy data.

⑪ Paste: paste copied data. Using Copy and Paste can copy experimental data to any nodes.

⑫ Delete: delete the node.

⑬ Edit Experiment Title: edit the experimental title, which may contain any information you want to be recorded, which will be shown in the bracket following the experiment node name, and will be shown above the spectrum when you print or export it (Figure 1.79).

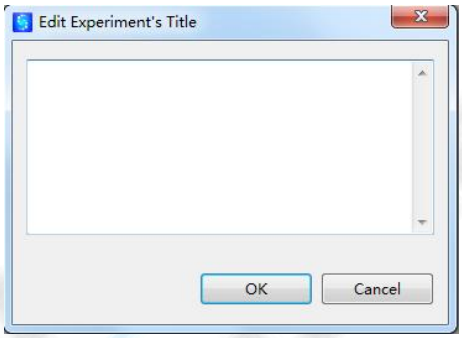


Figure 1.79 Edit title dialog

⑭ Rename: rename the experiment.

⑮ Import: import data. The default destination directory is one layer above current experiment node.

⑯ Refresh: Refresh current experiment.

⑰ Properties: The property panel will pop up on the right side of the workspace, which mainly contains the name, local path, last modification time and Title of the experiment, as shown in Figure 1.80.

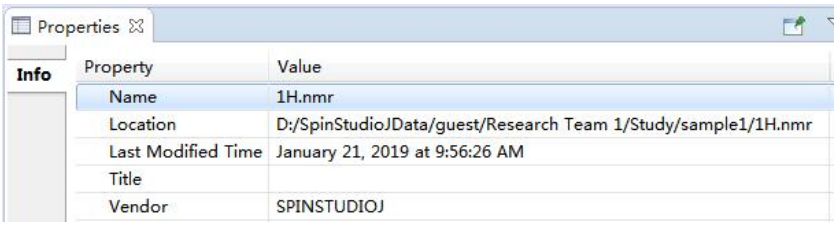


Figure 1.80 Right-click property panel for experiment node

1.6 Workspace

The workspace looks like Figure 1.81.

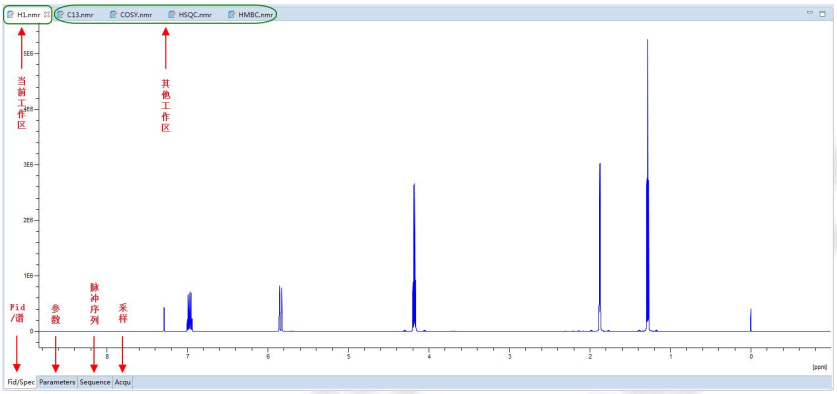


Figure 1.81 Workspace interface

The top bars display the names of opened experimental nodes, with the active window in white and others in blue. The workspace where data acquisition is in progress is labeled with a black star.

At the bottom of workspace are tabs for **FID/Spectrum**, **Parameters**, **Sequence** and **Acquisition**. Clicking each tab to show corresponding information in the current workspace window. The active tab is in white while others in blue.

FID/Spec:

To display the FID or spectrum of current experiment. The FID will be displayed at the end of acquisition, or can be shown during acquisition by typing command ***showfid***. Right-clicking within window area will allow user to select FID or Spec and Real or Imaginary part (Figure 1.82).

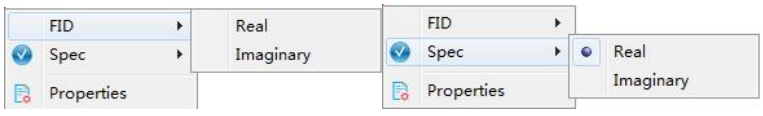


Figure 1.82 Right-click display menu of FID/Spec interface

Parameters:

To display acquisition and processing parameters. In these windows, user can set and modify parameters, which can also be done in command line, e.g., typing in ***p1=10*** will set the pulse width to 10 μs, and ***si=32768*** will set the data points to 32768.

Sequence:

Display Diagram and Source file of pulse sequence. User can check and edit pulse sequence here.

Acqu:

Display acquired data in real time during acquisition. Both FID and spectrum can be displayed by right-clicking as in the FID/Spec tab.

1.7 Log Panel

Log panel is used to show all the log information of backstage operations and all the commands and parameters that user typed in the command line (Figure 1.83).

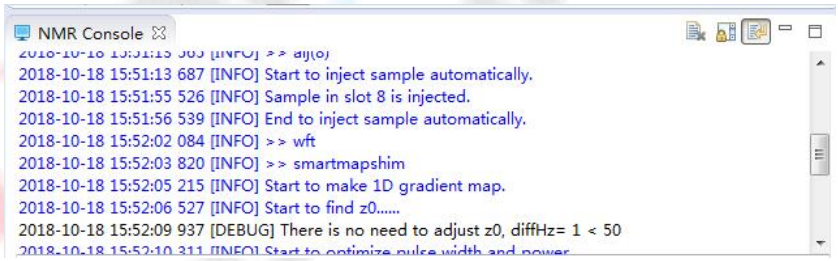


Figure 1.83 Log panel

User can click to clear log information; click to lock the scrolling side bar to stop the scrolling of log information; click to force the log information displayed in a new line, which is the default mode; click to minimize and to maximize the log panel, and click at the right side of workspace to restore it.

1.8 Command Line

The Command Line is just below the Log Panel. User can type any commands to execute it, or just type in parameter name to check its value, or assign a value to parameter by typing parameter name followed by an equal sign and a value, e.g., ***si=32768*** means set si to 32768.

1.9 Status bar

Status bars are located at the bottom of software interface (Figure 1.84). They are used to display the major status information of the spectrometer in real time, which includes connection, lock, sample, spin, temperature, helium level, queue and acquisition information.

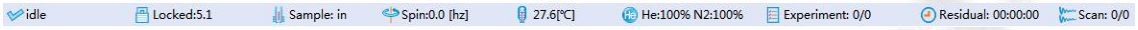


Figure 1.84 Status bars

1.9.1 Connection Status

Display the current status of connection between computer and console. means connected and means connection failed.

1.9.2 Lock Status

The icon for lock status is , demonstrating whether the field is locked and the lock level.

Double-clicking the icon or button on toolbar will open the LockShim box (Figure 1.85). Adjust z0 and lock power and gain to let the lock level to a certain value, then select the **LockOn** option, the status will be shown as **Locked**; deselecting the **LockOn** option will prompt an **Unlocked** status.

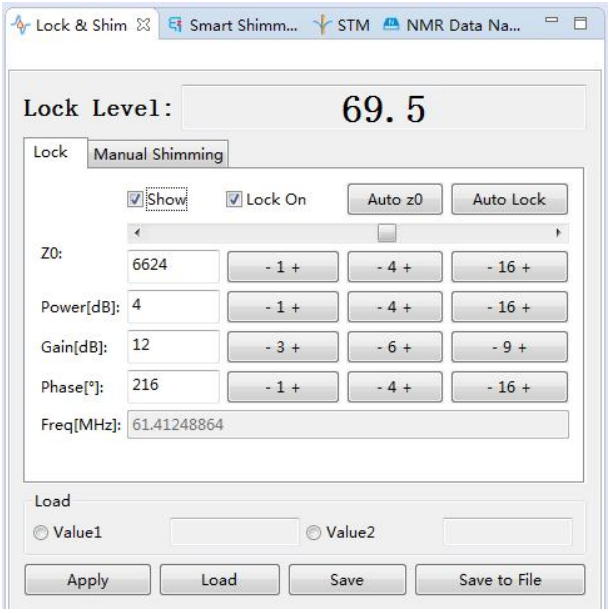


Figure 1.85 Lock & Shim box

1.9.3 Sample Status

Display whether there is a sample in the magnet. Right-clicking the icon, you will be prompted to select **Inject** or **Eject** sample. If there is a sample changer, right-clicking the icon to select Sample Status will open a window, as is shown in Figure 1.86 for 24 slots sample changer. User can right-click position number to select **Inject** or **Eject** sample.

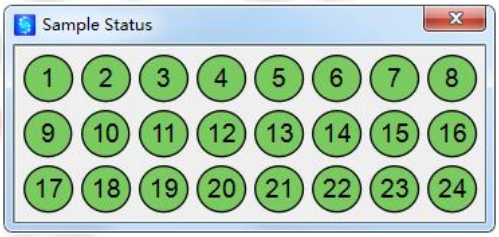


Figure 1.86 Sample status diagram

1.9.4 Sample Spinning

The current sample spinning rate is displayed beside the spinning status icon in the Status Bar at the bottom of software interface.

Right-click on the spinning status and then select **Start Edit** to open the Spin Edit dialog window. The appearance of Spin Edit window is different for Quantum-I (Figure 1.87) and Quantum-I ^{Plus} (Figure 1.88).

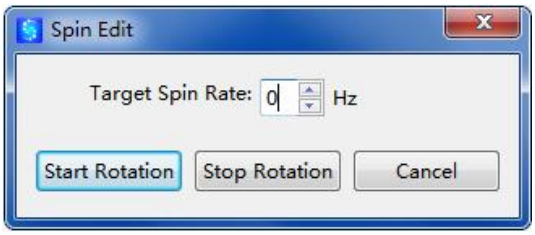


Figure 1.87 Spin Edit window for Quantum-I console

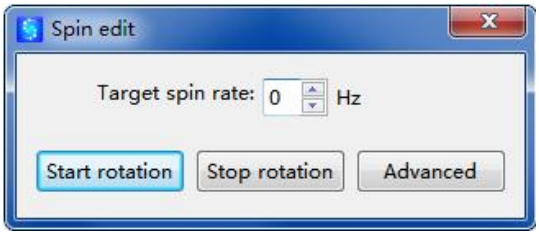


Figure 1.88 Spin Edit window for Quantum-I ^{Plus} console

For Quantum-I console (Figure 1.87), you can directly type in the value of spinning rate, or set the value by clicking the up and down arrows. Then, click Start Rotation to rotate the sample, or click Stop Rotation to stop sample spinning. Click Cancel to unset the setting and quit the window.

For Quantum-I ^{Plus} console (Figure 1.88), you can also directly type in the value of spinning rate, or set the value by clicking the up and down arrows. Then, click Start Rotation to rotate the sample, or click Stop Rotation to stop sample spinning. Click Advanced to enter advanced pneumatic setting window, which includes **Spin information**, **Spin information** and **Warning message** (Figure 1.89).

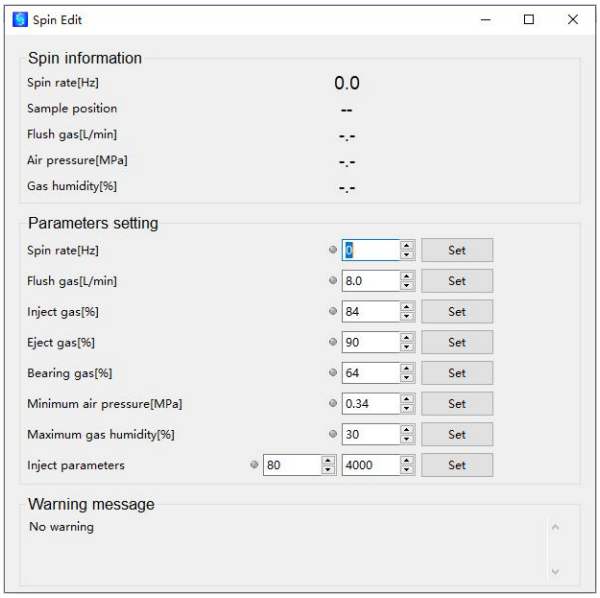


Figure 1.89 Advanced setting for pneumatic unit

1. **Spin information**

Spin rate[Hz]: sample spinning rate in Hz.

Sample position: status of sample within the upper barrel. Top means that there is a sample at the top of the upper barrel; Bottom means that there is a sample at the bottom of the upper barrel; No Sample means that no sample is detected at the upper barrel.

Flush gas[L/min]: the current flow of flush gas in L/min.

Air pressure[MPa]: the air pressure in pneumatic system in MPa.

Gas humidity[%]: the humidity of compressed air in pneumatic system.

2. **Parameters setting**

Users can set up pneumatic parameters here.

Spin rate[Hz]: sample spinning rate in Hz. After entering the value on the left, click the **Set** button on the right to set the speed value.

Flush gas[L/min]: the current flow of flush gas in L/min. After entering the value on the left, click the **Set** button on the right to set the Flush gas flow rate.

Inject gas[%]: air flow for sample insertion, range 1-100.

Eject gas[%]: air flow for sample ejection, range 1-100.

Bearing gas[%]: air flow for bearing sample, range 1-100.

Minimum air pressure[MPa]: minimum pressure of compressed air into pneumatic unit, in MPa.

Maximum gas humidity[%]: maximum humidity of compressed air into pneumatic unit.

Inject parameters: Set the parameters of the injection flow. Enter the number of steps required for the injection flow to gradually reduce from the set value to 0 on the left, enter the amount of steps for each decrease of the injection flow on the right, and then click the **Set** button on the far right.

3. Warning message

Display warning messages related to pneumatic unit.

1.9.5 Temperature Status

The current temperature (℃) is displayed beside the temperature status icon in the Status Bar at the bottom of software interface.

Right-click on the temperature status area and then select **Start Edit** to open the Temperature Edit dialog window. The appearance of Temperature Edit window is different for Quantum-I (Figure 1.90) and Quantum-I Plus (Figure 1.91).

Figure 1.90 Temperature Edit window for Quantum-I console

Figure 1.91 Temperature Edit window for Quantum-I Plus console

For Quantum-I console (Figure 1.90), the user can directly type in the value of spinning rate, or set the value by clicking the up and down arrows. Then, click **Start** to rotate the sample, or click **Stop** to stop sample spinning. Click **Cancel** to quit the current setting.

For Quantum-I Plus console (Figure 1.91), the user can directly type in the value of spinning rate, or set the value by clicking the up and down arrows. Then, click **Start** to rotate the sample, or click **Stop** to stop sample spinning. Click Advanced to open a dialog window for temperature related parameter setting (Figure 1.92).

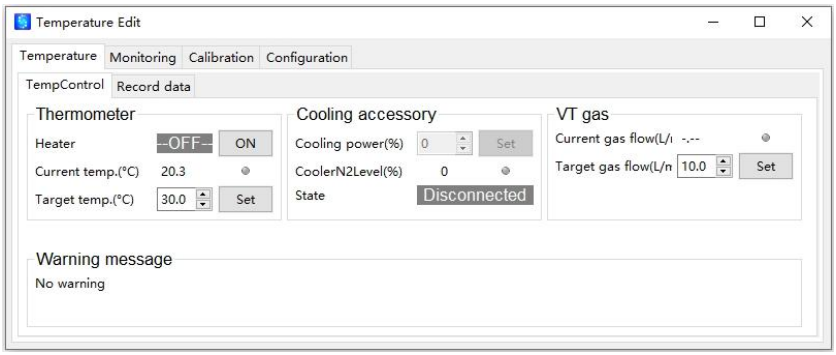


Figure 1.92 Window for VT control

1. Temperature

Set up VT parameters for current experiment, such as target temperature, VT gas flow, whether to use VT accessory and record the VT process.

1) TempControl

a. Thermometer

Heater: VT switch. The status sign is displayed on the left side, where green --ON-- indicates VT is on (Figure 1.93) and gray --OFF-- indicates VT is off (Figure 1.92). On the right side is the VT control button toggling between ON and OFF. The user can click the button to turn on/off the VT controller.

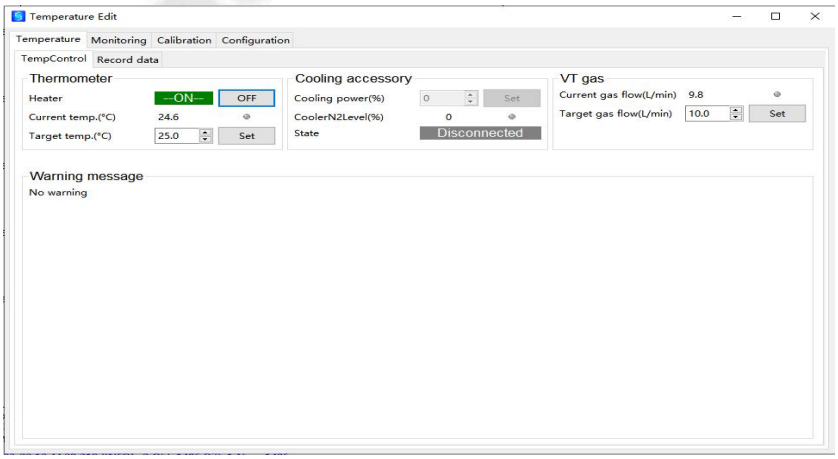


Figure 1.93 Temperature control opening interface

Current temp: Current temperature in ℃.

Target temp: Targeting temperature for VT experiment in ℃. Enter the temperature value in the temperature box and click the **Set** button on the right to set the target temperature.

b. **Cooling Accessory**

Cooling power: Cooling system power control. After entering the cooling power value on the left side, click the Set button on the right side to set the target power.

CoolerN2Level: Level of liquid nitrogen in Dewar.

State: Status displaying whether the cooling system is connected.

c. **VT gas**

Current gas flow: Current VT gas flow in L/min.

Target gas flow: Target VT gas flow in L/min. Enter the value on the left and click the **Set** button on the right to set the target gas flow rate.

d. Warning message: Warning messages related to VT control.

2) Record data

Set parameters related to VT data recording (Figure 1.94).

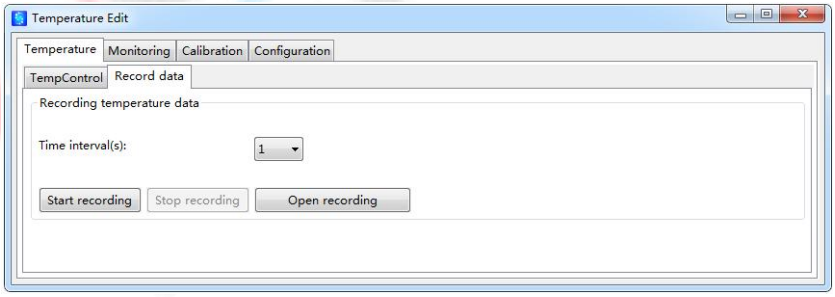


Figure 1.94 Settings for VT data recording

Time interval: Tine interval between each recording, in seconds. The user can select the value through pull-down menu.

Start recording: Start to record the temperature.

Stop recording: Stop recording the temperature.

Open recording: Open the window to check the recorded temperature data.

2. Monitoring

The **Monitoring** tab is used for real time monitoring of temperature (Figure 1.95). Target temp, Sample temp and Sensor temp can be displayed when corresponding boxes are checked. The displayed temperature range can be changed by scrolling the mouse wheel over the vertical axis.

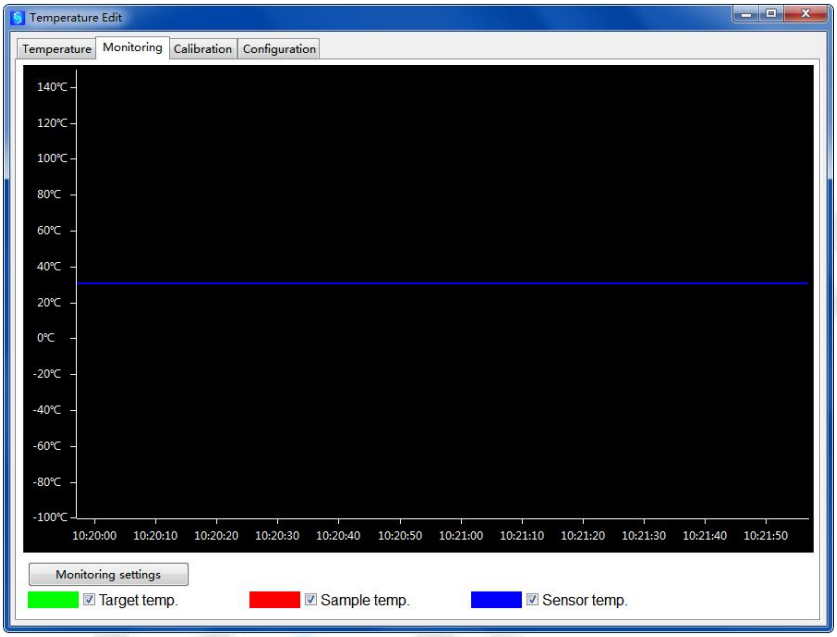


Figure 1.95 Window for temperature monitoring

Click **Monitoring settings** to open the monitoring setting window, where the user can change the displayed time duration and time interval for refreshing the data (Figure 1.96).

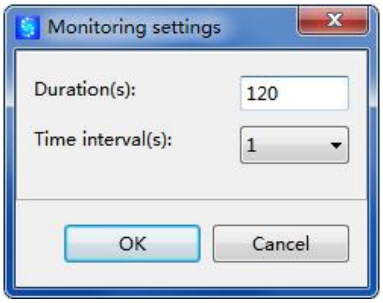


Figure 1.96 Monitoring settings window

3. Calibration

The parameter settings related to temperature calibration are displayed in this window (Figure 1.97).

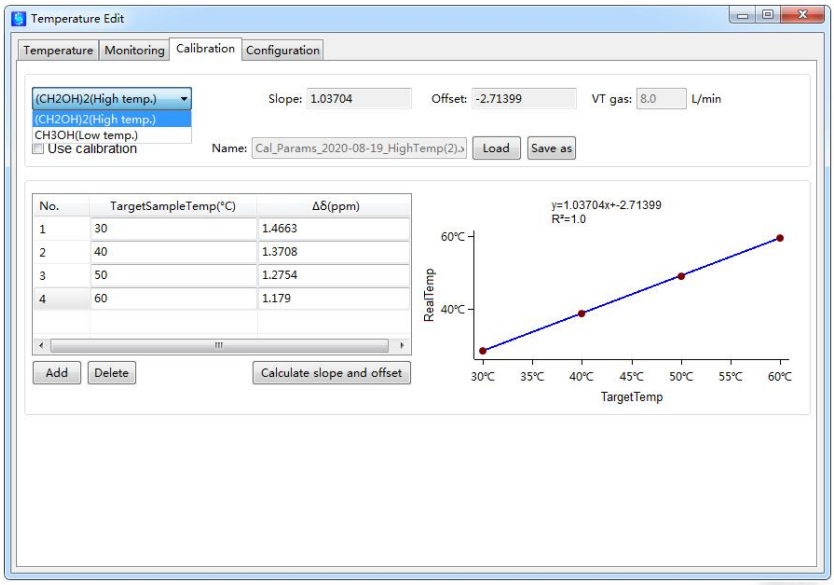


Figure 1.97 Settings for temperature calibration

(CH2OH)2(High temp.): Click to open the pull-down menu and select the sample for high/low temperature calibration.

Slope and Offset: Display the slope and offset derived from the calibration curve.

VT gas: VT gas flow (L/min) used for temperature calibration.

Use calibration: Option for whether to use calibrated data when turning on VT control.

Name: name of the calibration file. The user can click **Load** to select a saved file for use, or click **Save** to save the current calibrated parameters to a file.

The used temperature points and corresponding chemical shift differences are displayed in the table. The user can click **Add** or **Delete** to add more points or delete the current points for calibration. Click **Calculate slope and offset** to carry out temperature calibration using displayed data, and the resulting calibration curve will be displayed at the right side.

4. Configuration

The basic settings for a designed NMR probe are placed here (Figure 1.98).

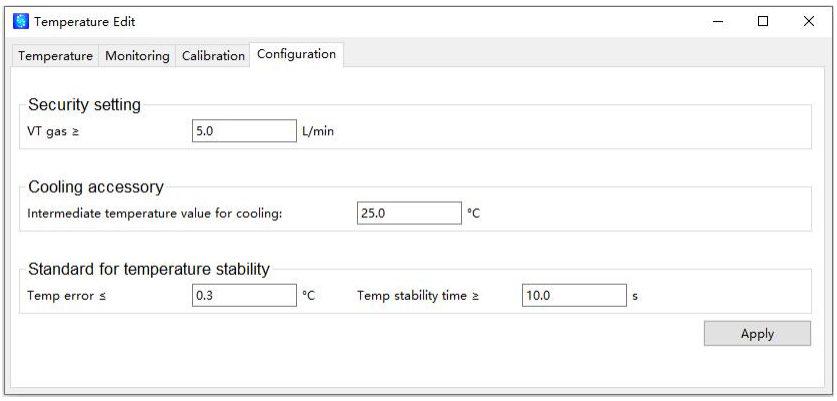


Figure 1.98 Basic settings for VT control

VT gas: Minimum allowed VT gas flow.

Intermediate temperature value for cooling: Temperature threshold for turning on cooling accessory. When the targeting temperature is lower than this value, cooling accessory is turned on.

Temp error and Temp stability time: Thresholds for temperature stabilization judgement. When the temperature fluctuation is smaller than the Temp error value within Temp stability time, it is regarded as stabilized.

Apply: Click to apply the above settings.

1.9.6 Cryogen Level Status

Display the current levels of liquid helium and nitrogen. Double-clicking or right-clicking the icon then selecting **Display Parameters**, will open a tab in Navigator area for level reading and setting (Figure 1.99). Only the administrator can set up new values, and the normal user can only browse it.

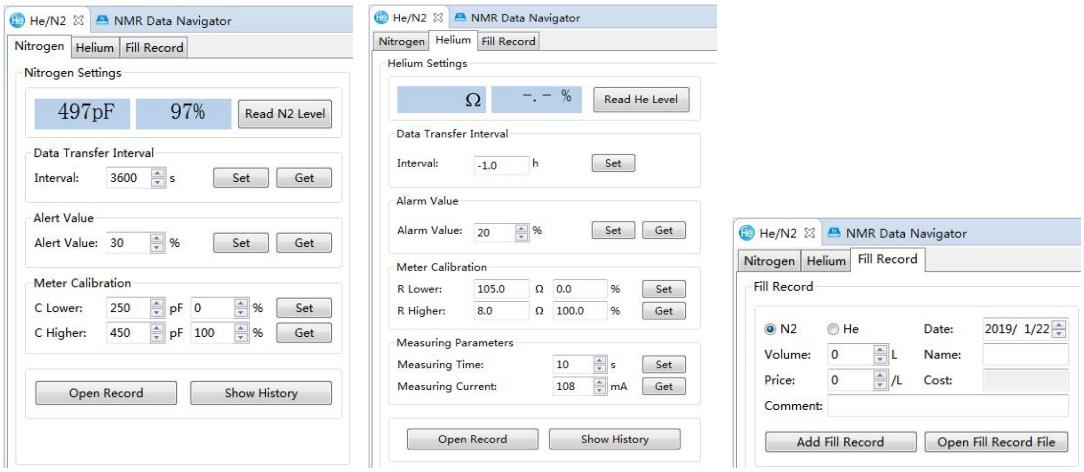


Figure 1.99 Level reading and parameter setting interface

The liquid nitrogen level is shown as percentage value as well as corresponding capacitance value. You can also click Read N2 Level to read the value in real time.

Liquid nitrogen level is continuously monitored. Administrator can set **Data Transfer Interval** in seconds for uploading the level data to computer. The commonly used value is 3600s, i.e., uploading data every hour, and a new data point will be added in the record curve.

Testing the liquid helium level will cause relatively large consumption, so it is generally not measured in real time. Instead, the software performs the test when the data is read, and uploads the current test results after the test is completed. The administrator can set the time interval for data reading and uploading (ie, the liquid level test time interval). Set **Data Transfer Interval** to a negative number or 0 to indicate no automatic reading, usually set it to 168h, which means reading once a week. The administrator can click the button Set to save the settings after modification.

Alert Value: minimum value for alerting, with a default value of 30%. Changes on this value is not recommended.

Meter Calibration: used to calibration of the capacitance values for 0% and 100% level. Normally done by installation engineer and changes is not recommended.

Clicking **Open Record** will open an Excel file with records, which includes record time, capacitance value and percentage level. Clicking **Show History** will open a diagram shown recorded curves (Figure 1.100).

The liquid helium level is shown as percentage value as well as corresponding resistance

value. You can also click Read He Level to read these values in real time.

Alert Value: minimum value for alerting, with a default value of 20%. Changes on this value is not recommended.

Meter Calibration: used to calibration of the resistance values for 0% and 100% level. Normally done by installation engineer and changes is not recommended.

The Measuring Parameters are used to set the duration and electric current values each time the helium level is measured. Normally done by installation engineer and changes is not recommended.

Refilling Record can facilitate the administrator to record the time, refilled volumes and price, and add them to record files.

Right-clicking the icon in status bar then selecting **Display Level History** will open a figure in the workspace, which includes the curves of recorded helium and nitrogen levels, as well as preset alerting level curves (Figure 1.100). User can scroll the mouse wheel to room, or click and hold the left button, then move around to check the suitable range of level and date.

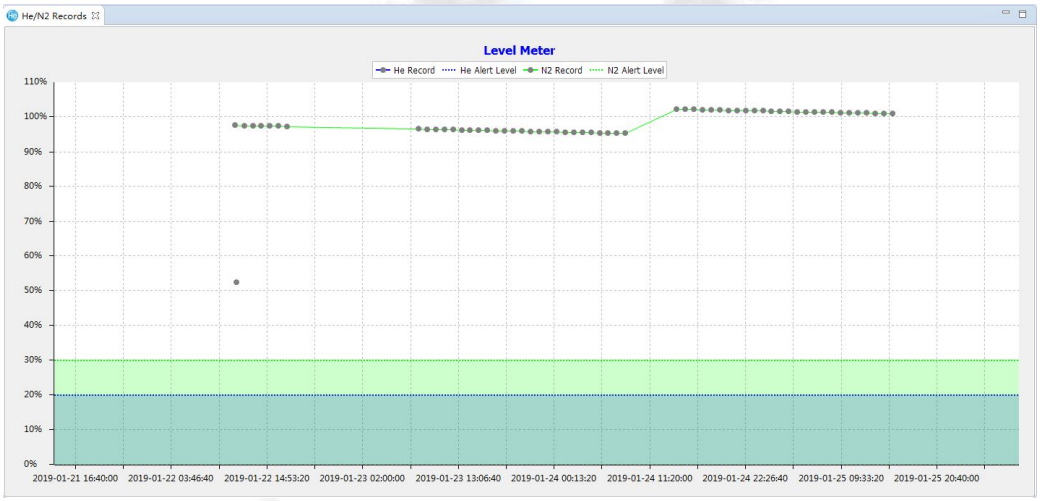


Figure 1.100 Liquid level history curve

Right-clicking over the above window, you will be prompted for options (Figure 1.101).

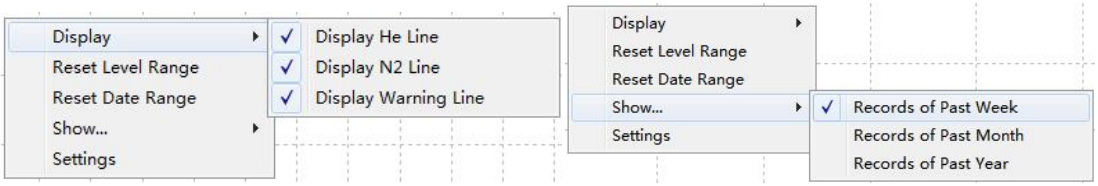


Figure 1.101 Right-clicking option for liquid level recording curve page

Display: select whether to show helium, nitrogen and warning curve.

Reset Level Range: the default level range for display is 0%~110%, which can be reset to 0%~100%.

Reset Date Range: reset the displayed time period defined by **Show** option.

Show: options for displaying records of last week, month or year.

Settings: open the cryogen status window.

1.9.7 Queue Information

Display which experiment is currently executed and the total amounts of experiments. For example, 1/4 means totally there are 4 experiments to be done and currently the first one is running. Double-clicking the Experiment will open the Experiment Queue table showing detailed information (Figure 1.102). User can use right-clicking options to change the position of any experiment or delete it from list.

Experiment Queue | NMR Data Navigator

Experiment Queue

No.	Path	Status	Type	Sequence	Start Time	End Time
1	D:/SpinStudioJ...	Acquiring	acquiring	s1pul30	14:22:59	14:24:28
2	D:/SpinStudioJ...	Queued	acquiring	s1pulpg30	14:24:28	14:58:37
3	D:/SpinStudioJ...	Queued	acquiring	cosy_gpqf	14:58:37	15:12:53
4	D:/SpinStudioJ...	Queued	acquiring	hmbc_gpqf	15:12:53	15:51:10

Figure 1.102 Experimental queue list

Type include acquiring, again, alock, autoz0, stm, gmap1d, gshim1d, gmap3d, gshim3d, searchshim, autosample and tune.

1.9.8 Acquisition Related Information

Residual: residual time for current experiment.

Scan: completed scans and total scans.

Chapter 2 Getting Started

2.1 Sample Preparation and

1. Sample Preparation and Spectrometer Startup

Add appropriate amount of sample into dry and clean NMR tube of the same diameter as that of the probe.

For 5mm NMR tube, a volume of 0.5~0.65 mL will result in a sample height of 4~5cm height. It is recommended that all samples are kept with the same height.

Wipe the tube with lens paper and insert it into spinner, then put them into the gauge at one end of the sample rack, adjust the position of tube in spinner until it touch the bottom of the rack (Figure 2.1). Note, sample surface should be higher than the brown plate.

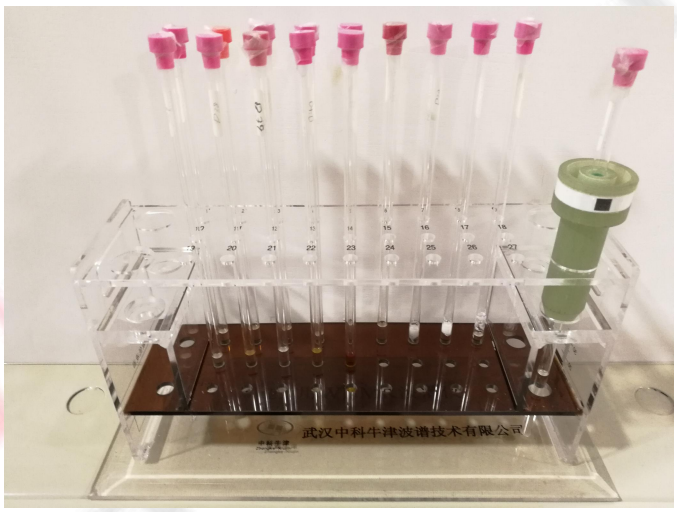


Figure 2.1 Sample rack, spinner and NMR tube

2. Spectrometer Status Checking

Spectrometer turned on?

All wiring correct?

Spectrometer work well?

Pressure of compressed air appropriate?

Right probe?

2.2 Startup SpinStudioJ

1. Startup

Double-click the SpinStudioJ icon on the desktop, or go to the SpinStudioJ folder in the software installation directory and double-click SpinStudioJ.exe to startup the software.

2. Checking the connection

The connection status between computer and console is displayed at the left side of the status bar. means connected and means unconnected.

If unconnected, user can type ***hi*** command to start connection. If the connection is still failed, you need to check:

A. Is the IP address and port number of console network correctly set up?

B. Is the network cable connected and router working?

C. Is there any malfunctioning in the console?

2.3 VT Control

Users are recommended to regulate the sample temperature before starting a experiment, so that the experiment is done under a stable sample temperature, referring to 1.9.5 **Temperature Status** for detailed operation.

2.4 Inserting and Ejecting Sample

There are three methods for inserting and ejecting samples:

(1) Right-clicking the Sample Status icon Sample:in, then select **Inject** or **Eject**

(2) In the menu bar, select **Acquire** > **Sample** > **Inject** or **Eject**

(3) On the command line, enter ***inject*** or ***eject***

When equipped with a sample changer, you can type ***aij***(n) to insert sample or ***aej*** to eject sample, or right-click the Sample Status icon Sample:in to select **Sample Status**, then right-click on the slot number to select **Inject** or **Eject**, referring to 1.9.3 .

After inserting the sample, a dialog box will be popped up (Figure 2.2), where you can

enter the sample information which will be displayed when printing (optional) and select a solvent, which will be used as the solvent in **New Experiment**.

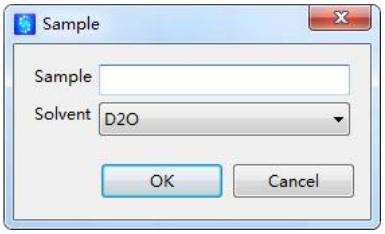


Figure 2.2 Injection dialog

2.5 New Experiment

User can open a **New Experiment** dialog box by clicking icon in tool bars, clicking **File** > **New** or **Acquire** > **New Experiment** > **Create**, or type ***newexp*** on command line. This can also be done by right-clicking to select **New Experiment** under **User**, **Research Team**, **Research Project**, **Folder** or **Experiment** in **NMR Data Navigator**. Directory (Dir) and experiment name (Exp Name) will be different if you create new experiment under different node of NMR Data Navigator, referring to **1.5** NMR Data Navigator. The dialog box is shown in Figure 2.3, the default Params Type will depend on if there were opened data in workspace.

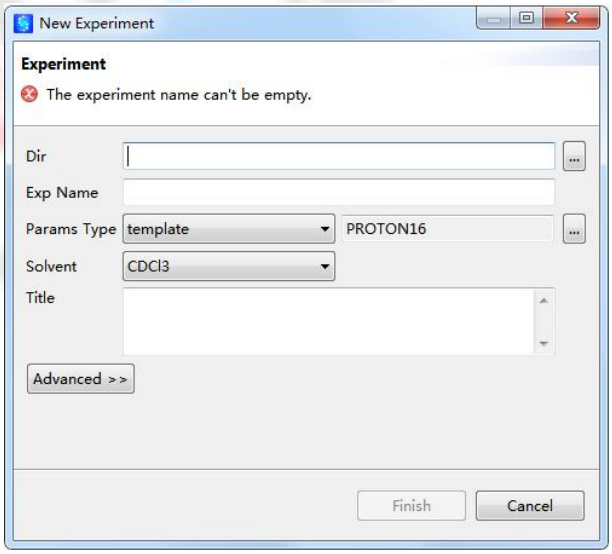


Figure 2.3 New experiment dialog

You can also select **Acquire** > **New Experiment** > **New form Template** to open a

Template dialog box (Figure 2.4). Clicking the specified template will also pop up a dialog box shown in Figure 2.3, but with specified template information.

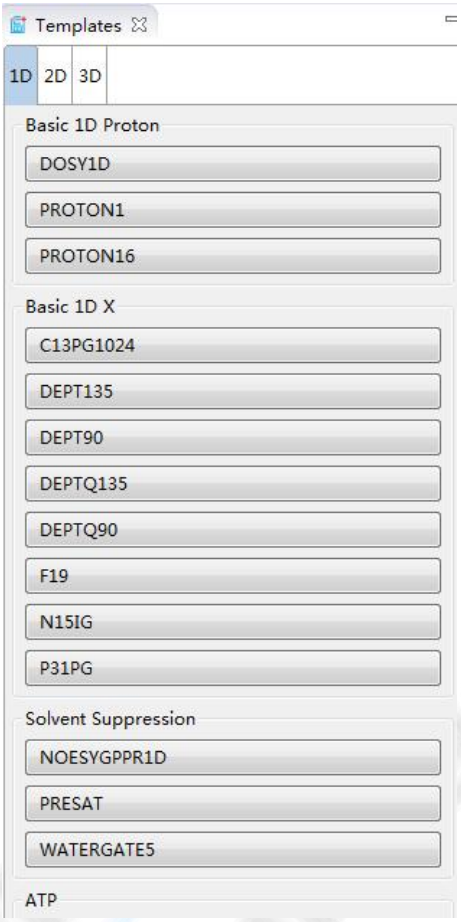


Figure 2.4 New experiment selection interface from template

Dir: directory where the data is saved. You can click ... to select. If there are data in the specified directory.

Exp Name: user can give a name to your experiment. Chinese, English letters, numbers and symbols are supported.

Params Type: type of parameter, which can be **current workspace parameters** or **template**. If there is any data open in workspace, the option is current workspace parameters, i.e., using the parameters of the active workspace. If no data is opened, the option is template, and the default template is PROTON16. You can also click ... to open a dialog box (Figure 2.5). There there are two options for **Dim**, 1D and 2D, and many options for Type, which includes Basic 1D Proton, Basic 1D X, Selective 1D, Solvent Suppression, Instrument Test, ATP, Basic 2D H-H, Basic 2D H-X, Basic 2D X-H and J Spectra. User can specify a type to

display or enter a key work in Include to find the template. Show Recommended is enabled by default. Clicking **Reset Filters** to display all templates.

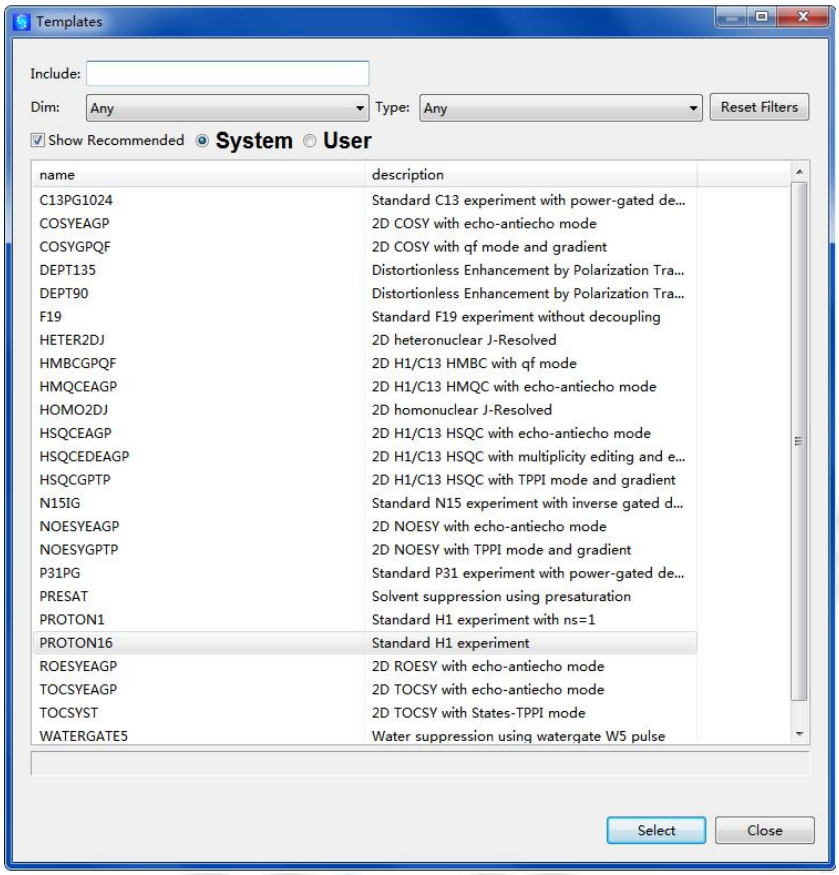


Figure 2.5 Template selection dialog

Solvent: deuterated solvent used for the sample. You click the down-arrow to select one from the list, which should be the same as that in **Sample** dialog box (Figure 2.2).

Title: optional information about the sample, e.g. sample name, solvent, concentration, date.

Advanced: advanced option where you can choose to get parameters from probe file or copy them from specific experiments for each channel, or add a password for the new experiment (Figure 2.6). If you want to copy parameters from some experimental data, it must have been opened in workspace, then you can choose it from the drop-down list of the Channel. The number of Channels displayed depends on the type of experiment you chose.

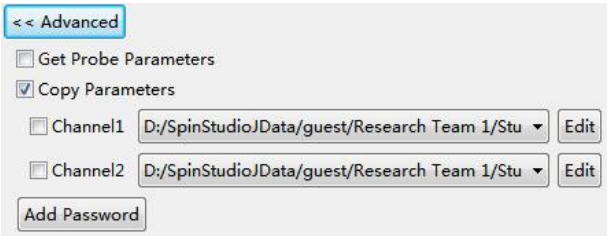


Figure 2.6 Advanced expansion item

Only two parameters sw and frqo can be copied. You can click Edit button to open a dialog box (Figure 2.7), where you can edit to copy one or two parameters.

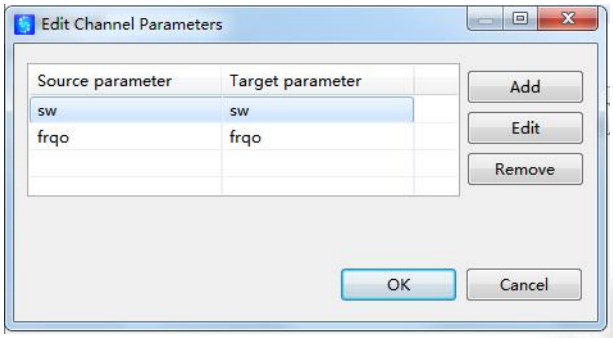


Figure 2.7 Edit parameter dialog

After all needed information are selected, click **Finish** to dismiss the window (Figure 2.3), and the new experiment will be displayed in the corresponding position of Data Navigator.

2.6 Locking

User can open Lock tab in **Lock & Shim** panel overlapped with the **Data Navigator** by clicking button in tool bar, clicking **Acquire** > **Lock** > **Manual Lock** in the menu bar, or double-clicking icon in Status panel (Figure 2.8).

Select **Show** check box to open a window in the workspace to show lock sweep. The red curve represents the lock level and the blue curve represents error level. The lock level value is displayed in **Lock & Shim** panel.

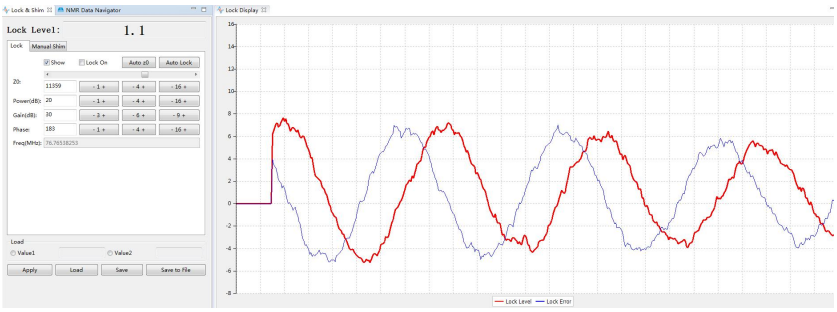


Figure 2.8 Lock & Shim panel

Three parameters, Lock Power, Lock Gain and Lock Phase, are used to control the lock signal. These parameters can be adjusted by left- and right-clicking mouse buttons or rolling the mouse wheel up and down over the buttons corresponding to different increments. These parameters may need an adjustment before and after locking.

There are boxes and buttons under the lock parameters area used to save or load lock or shim parameters during manual locking and shimming.

Value1 and **Value2**: temporarily saved lock and shim parameters, used in combination with **Apply** and **Save** buttons.

Apply: apply temporarily saved **Value1** or **Value2** to the current lock. You have to select one of these two values first.

Save: temporarily save current lock or shim parameters to **Value1** or **Value2**. You have to select one of these two values first.

Load: load a *.ini file and apply the parameters for lock and shim. A dialog box will be opened to enable you select a file.

Save to File: save the current lock and shim parameters including z0 in a .ini file in shimvalue directory under currently used solvent.

2.6.1 Manual Locking

Adjust the value of z0 to maximize the red lock curve and make the blue curve as close as possible to zero (Figure 2.9). You can adjust z0 by: moving the slider, left- and right-clicking mouse buttons or rolling the mouse wheel up and down over the buttons corresponding to different increments.

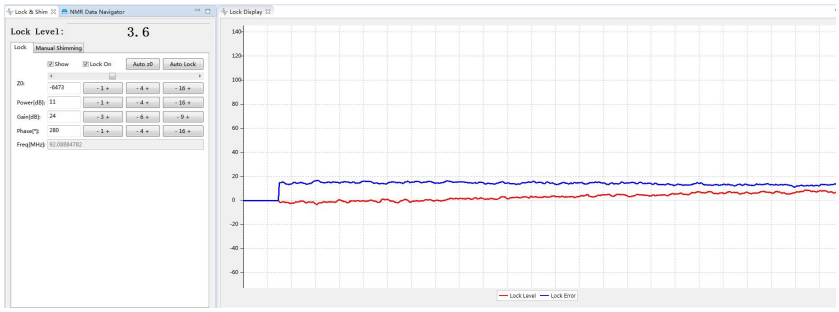


Figure 2.9 The lock horizontal line and the lock error line are separated

If the lock signal is too small that the two lines can not be separated, adjust Lock Power and Lock Gain. If the red line is negative and the blue line not near zero, adjust Lock Phase. When correct parameters are set, the red line is maximized and stable (Figure 2.10). You may have to alternatively adjust z0 and lock phase to maximize the lock level. If Lock Level is too large and saturated (>100), slightly lower Lock Power and Lock Gain. When Lock Level is above 50, select **Lock On** check box to finish locking.

Figure 2.10 Adjusted lock line

2.6.2 Auto Locking

Click **Auto Lock** in the Lock tab or type ***alock*** in command line, the software will automatically find z0, optimize Lock power, Lock Gain and Lock Phase to complete locking. You can also click **Auto z0** to find correct z0 only, then manually optimize Lock power, Lock Gain and Lock Phase.

In the process of locking, if you want to stop, you need to enter the command ***stopalock*** or the command ***aaa***.

2.7 Tuning and Matching

Click **Acquire** > **Tune**, or click button in tool bar to open the tuning interface. A parameter window is opened in left side and a window opened in workspace showing a wobble curve with a dip (Figure 2.11).

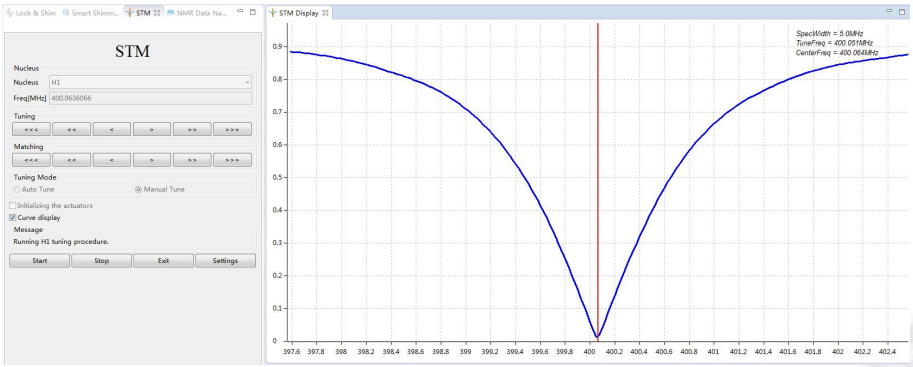


Figure 2.11 Automatic tuning interface

There are two options for **Nucleus** and **Freq** under **Nucleus**.

Nucleus: Tuning nucleus. Users can choose according to the needs of the experiment.

Freq: The frequency corresponding to the tuning nucleus. It will be updated automatically when you switch **Nucleus**. The red vertical line in the right window is the location of the frequency, located in the center of the window.

There are **Tuning** and **Matching** tool icon buttons under **Tuning and Matching**. When manually tuning, you can click on these icons to adjust the **Tune** and **Match** of the probe.

There are two options in **Auto Tune** and **Manual Tune** in **Tune Mode**, you can choose auto tuning or manual tuning as needed.

Initialize the actuators: Set the probe's corresponding nucleus to the initial position before tuning. This option must be checked when switching the tuning nucleus, otherwise the tuning will fail.

Curve display: The display switch of the tuning curve. Check it to open the tuning interface and display the curve, otherwise, the interface does not open.

Message: Displays the tuning related status.

Start and **Stop**: Start and stop tuning.

Exit: Exit the tuning mode.

Settings: Open the Settings dialog box to set the tuning scan frequency range **TuneSpecWidth** and the tuning scan point **TunePoint** (Figure 2.12).

TuneSpecWidth: sweep width in MHz for tuning, 2 is recommended. If no signal is found, larger value can be used.

TunePoint: sweep points for tuning. If TunePoint is larger than 1, frequency sweep is used, with a recommended value of 200; if TunePoint equals to 1, point tuning method is used, and the tuning value will be shown on the LED screen of preamplifier.

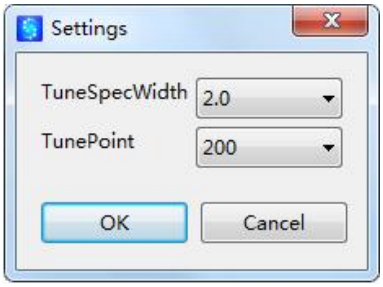


Figure 2.12 Settings dialog

Two display modes, **ABS** and **Complex**, for tuning curve can be selected by right-clicking in the window. **ABS** (absolute value) mode is commonly used (Figure 2.13), while **Complex** mode can also be used (Figure 2.14). During the tuning process, you can click and drag the mouse to zoom the display range, right-click and select **ShowFull** to resume to full display.

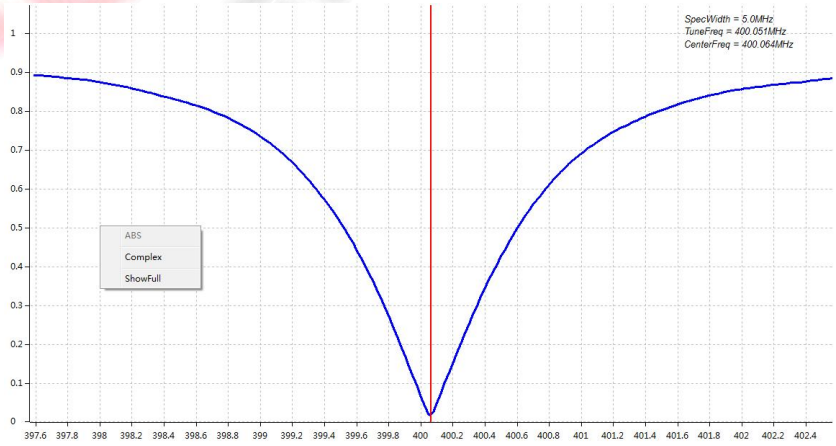


Figure 2.13 Absolute value mode tuning window

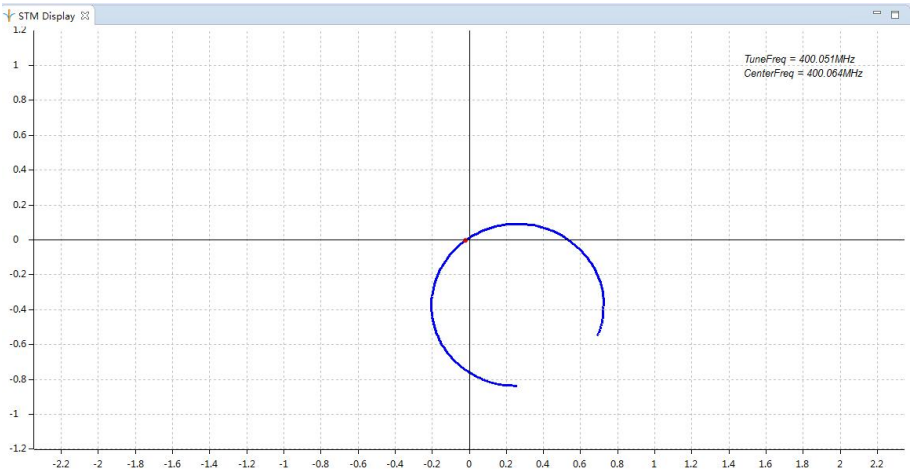


Figure 2.14 Complex mode tuning window

1. Automatic Tuning and Matching

Select the nucleus to be tuned in the Nucleus column and Freq will automatically switch to the corresponding frequency. Select "**Auto Tune**" in **Tune Mode** and it is recommended to check the "**Initialize the actuators**" option. Click the **Start** button and the software will automatically perform the tuning operation. After the tuning is completed, it stops automatically.

If there are multiple nuclei that require tuning, repeat the above. It is recommended to tune the order of the nuclear resonance frequencies from low to high.

You can also enter the auto-tuning command ***stm***. The software will read the nuclei that need to be called according to the selected workspace, and automatically perform the tuning operation in the order of the nuclear resonance frequency from low to high. If no data opened, ***stm*** command will open the tuning and matching windows , select ^{1}H nucleus by default but without further operation.

2. Manual Tuning and Matching

After opening the tuning window, select the nucleus to be tuned in the **Nucleus** column. Select "**Manual Tune**" in **Tune Mode** and unselect the "Initializing the implements" option. Click the **Start** button to start tuning. The icon under the **Tuning and Matching** column is activated and the tuning curve display area shows the tuning curve. Adjust the **Tune** and **Match** of the probe by clicking the arrow buttons corresponding to the **Tuning** and **Matching** icons. After the adjustment is completed, click the **Stop** button to finish tuning.

If more than one nucleus should be tuned, start from that with the lowest resonant frequency.

Note: When switching nucleus for tuning, it is recommended to use **Auto Tune**, otherwise it's easy to fail. After the **Auto Tune** is finished, you may need to use **Manual Tune** for further fine tuning.

2.8 Shimming

Shimming is the process of small compensation (positive or negative) to the main magnetic field to improve its homogeneity for better spectral resolution. Every time when changing probe or sample, it is necessary to shim the magnet.d

SpinStudioJ provides four methods for shimming: Manual Shimming, Smart Shimming, Search Shimming and 3D Smart Shimming. The last three methods belong to auto shim. User can select proper method according to your situation.

2.8.1 Manual Shim

User can manually adjust the current values for room temperature shim coils. Currently, the room temperature shim set for 400MHz spectrometer consists of 23 coils including z0, which can be further divided into five groups based on their effects on magnetic field (Table 2.1).

Table 2.1 Room temperature shim coils

Axial	Z1 Z2 Z3 Z4 Z5 Z6
Low Order	X1 Y1 ZX ZY C2 S2
3rd Order	Z2X Z2Y ZC2 ZS2 C3 S3
4th Order	Z3X Z3Y Z2C2 Z2S2

For manual shim, the criteria for field homogeneity is lock level. Higher lock level normally indicates higher field homogeneity.

Note Lock level is not only criteria for field homogeneity. The area of FID and line shape can also indicators of field homogeneity.

The process for Manual Shim is as follows:

1. Click **Acquire > Shim > Manual Shimming**, or click the down arrow of button on tool bar then select **Manual Shimming** to open **Lock & Shim** panel, with Manual Shim tab being active (Figure 2.15).

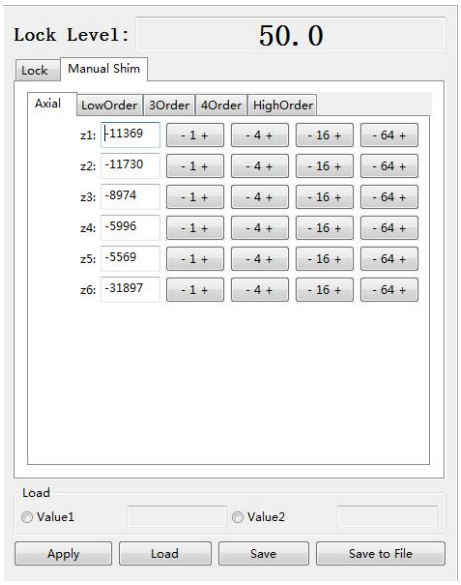


Figure 2.15 Manual shimming interface

2. Click **Load** button to retrieve the shim values from a .ini shim file.
3. Adjust z1 value by using one of the following methods to increase the lock level, until it maximized or even begins to fall.
   (1) Right- or left-click the buttons showing different increments (-1+, -4+, -16+ and -64+).
   (2) Turn the mouse wheel up or down while the cursor is over these buttons.
   (3) Enter a number in the box corresponding to each coil. The range of the number for shim coil is between -32768 ~ +32767.
   (4) In the command line, assign a value to the shim coil in the form of , e.g. z1=2000.
4. Repeat step 3 for z2.
5. The z1 may need to be adjusted again, then z2 again until the lock level no longer increases.
6. Click **LowOrder** tab to display the low order radial shim coils. Adjust x1 to

maximize lock level, then adjust y1 in the same way.

7. Click **Axial**, slightly adjust z1 and z2 again to maximize the lock level.

You can click **Save** to temporarily save the shim values during manual shimming, then click **Apply** to retrieve it. After finishing the manual shimming, click **Save to File** to save all the shim values to a file that ends in the .ini extension, which can be retrieved later by clicking **Load.**

The administrator can type ***ssh*** command to save all the shim values to the currently used shim file, which can be retrieved by typing ***lsh*** in the command line.

Note If common user try to use manual shim, it is recommended to adjust z1-z2 (Axial) and x1-y1 (Low Order) only.

Note The shim coils in 3Order and 4Order tabs can only be adjusted by engineer and administrator.

2.8.2 Search Shimming

Search Shimming can automatically search and optimize the compensation value for each shim coil, with consideration of coupling effects between coils. It can result in good field homogeneity if used in combination with **Smart Shimming**.

There are two kinds of methods, **Tuning** and **Simplex**, for **Search Shimming** based on currently used solvent. ①**Tuning** is a simple shim method for low order shim coils, shimming on one coil each time, without considering the coupling between coils. Less coils and thus less time are used for shimming. ②**Simplex** is a complex shim method considering the coupling between coils, used for spectrometer calibration or after probe changing. More coils and thus much time are used for shimming.

Two kinds of criteria, **FIDArea** and **LockLevel**, are used for judging shim results. You can get best shim result by comparing the area of resulting FID (**FIDArea**) or by comparing the lock level (**LockLevel**).

This will result in four combination of shimming modes (Figure 2.16), which can be flexibly used according to the situation of current field homogeneity. Tow of then will be

introduced here, and the others are similar.

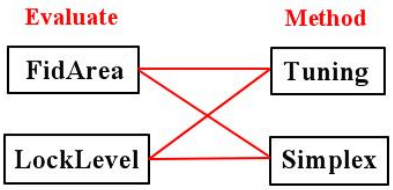


Figure 2.16 Search shimming mode

1. Shimming using Simplex with FIDArea as Criterion

(1) Click **Acquire** > **Shim** > **Search Shimming**, or click the down arrow of button on tool bar then select **Search Shimming** to open **SearchShim** window (Figure 2.17).

Figure 2.17 Search shimming interface

(2) In the drop-down menu next to **Solvent**, select correct one.

(3) Click the drop-down menu next to **Evaluate** and select **FIDArea**.

(4) Select a combination of shim coils in the drop-down menu next to **Simplex**, click **Add** to put into the searching list in **Groups** to immediately start search shim. You can click **Open** button to display the real time result in the searchshim window in workspace (Figure 2.18).

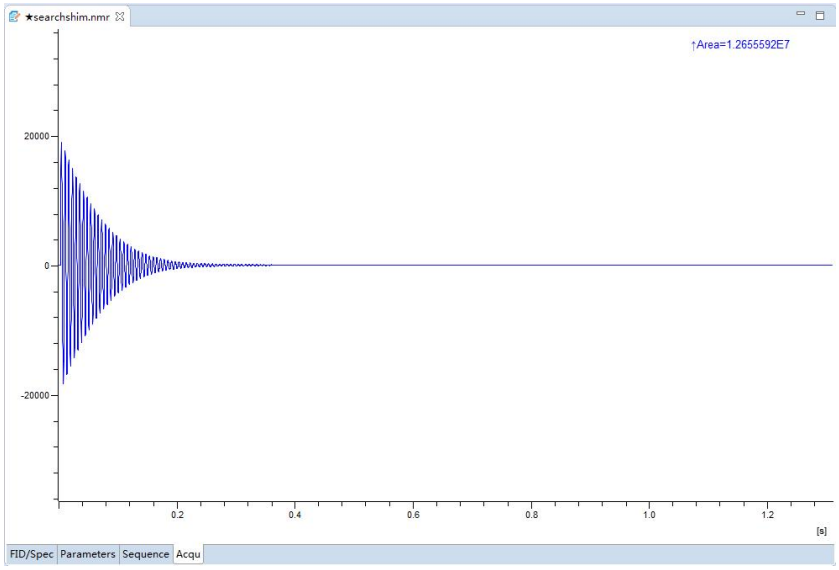


Figure 2.18 Search shimming window

(5) You can continue to add shim coils combinations to the search list during the shimming process, and change the order by clicking **Remove**, **Up** and **Down** buttons. The shimming process can be interrupted any time by clicking **Stop** button (Figure 2.19).

Figure 2.19 Simplex shimming with reference to FIDArea

2. Shimming using Tuning with Lock Level as Criterion

(1) Open the **SearchShim** window as described above (Figure 2.19).

(2) In the drop-down menu next to **Solvent**, select correct one.

(3) Click the drop-down menu next to **Evaluate** and select **LockLevel**.

(4) In the Lock tab (see 2.6 Locking), make sure that the check boxes for **Show** and **Lock On** are selected. Look at the lock level and check if it is stable (fluctuation small than 0.1, e.g. between 24.4\24.5\24.6). If not, slightly lower the lock power.

(5) Select a combination of shim coils in the drop-down menu next to **Tuning**, click **Add** to put into the searching list in Groups to immediately start search shim. You can open the Lock window to check the real time result by lock level.

(6) You can continue to add shim coils combinations to the search list during the shimming process, and change the order by clicking **Remove**, **Up** and **Down** buttons. The shimming process can be interrupted any time by clicking **Stop** button (Figure 2.20).

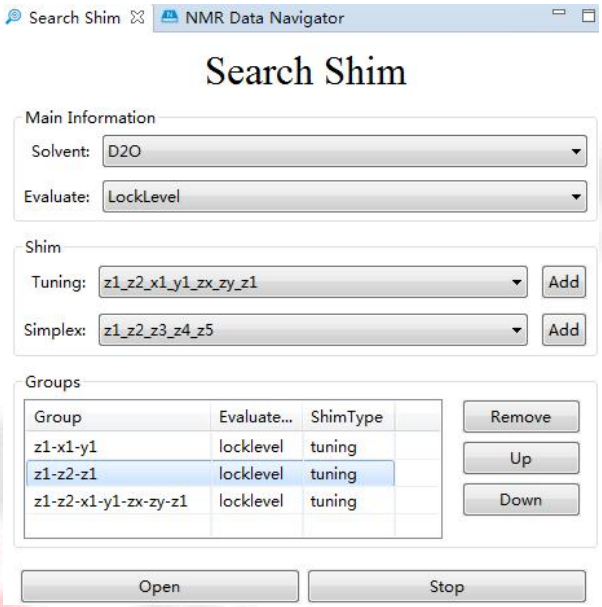


Figure 2.20 Tuning shimming with reference to Locklevel

The default iteration times are 15 for each coil combination. You can add or edit the coil combinations by clicking **Options** > **Preferences** > **Instrument** > **Shimming** > **Search Shimming** (refer to 1.3.6 Options).

After finishing the search shim, please save the shim values to a file, and readjust the Lock again.

Note Tuning and Simplex search shim methods are very effective for lower order shim coils. Users are net recommended to use these methods for high order shim coils if you are not familiar with shimming.

2.8.3 Smart Shimming

In NMR spectrometers equipped with pulse field gradient system, user can utilize gradients for automatic shimming. Smart Shim is thus designed for the automatic shimming on axial (z axis) shim coils.

Smart Shim is based on Shim Map, which ref the effects of each shim coil on the magnetic field as a function of sample position. User can retrieve a saved map for Smart Shim or create a new map for shimming, as revealed in Figure 2.21.

Figure 2.21 Smart Shim interface

1. Click **Acquire** > **Shim** > **Smart Shim**, or click the down arrow of button on tool

bar then select **Smart Shimming** to open **Smart Shim** window (Figure 2.21).

2. In the drop-down menu next to **Solvent**, select the solvent used for the sample. If a mixture of 90% H_2O+10% D_2O is used, select H_2O+D_2O.

3. Smart Shim based on existing Field Map

(1) Select **Shim Map** to use existing Field Map for shimming. Displayed in the box is a map named default, i.e., the system's default Field Map for current solvent. It will be displayed under the Smart Shim window, as shown in the left of Figure 2.22. Click **Browse** to open a window for the selection of user created Field Maps, as shown in the right of Figure 2.22. The maps coming along with the system is in the directory of default under current solvent, for example, if the solvent is D_2O, the path is *D:\SpinStudioJ-1.4.9alpha-20190624\system\data\solvents\D2O\gradshim1d\default*) . The user created maps are saved in the directory of gradshim1d under current solvent, with a name in the format of yyyymmdd(n), where n is the sequential number, starting from 0, for these maps created in the same day. For example, 201900622(2) is the 3rd map created at June 22, 2019.

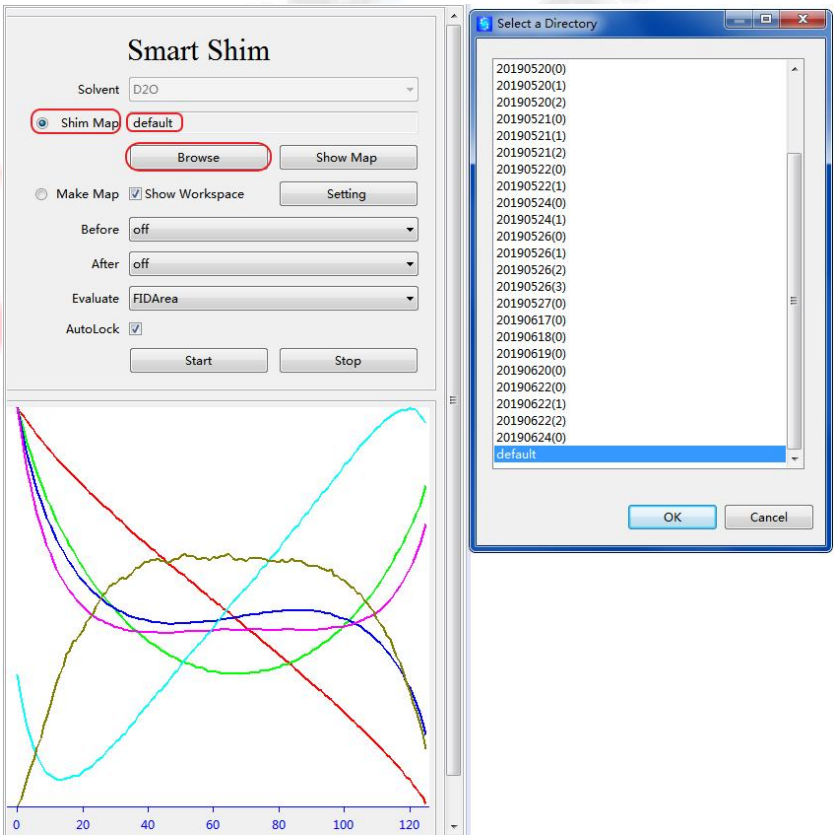


Figure 2.22 Select the user-created shim map

(2) Click **Setting** button to open the dialog box for Shimming Parameters. User can select Get parameters from field map file, or select Custom parameters to give new values for delta and iteration (Figure 2.23).

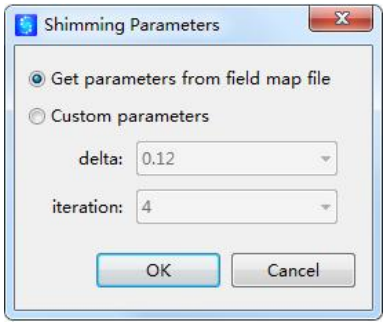


Figure 2.23 Shimming Parameters dialog

(3) User can click **Show Map** button to open a window showing the map in the workspace area (Figure 2.24). Clicking **Start** button here or in the Smart Shim panel will initiate the gradient shimming using current field map. This process can be seized by clicking **Stop** button. If the currently used map was made by user, you can click **As Default** to save it as the system default one for current solvent. Click **Close** to close the map window. At this point, a dialog box will pop up (Figure 2.25), and you can choose whether the name of the default field diagram is Default or Default 2.

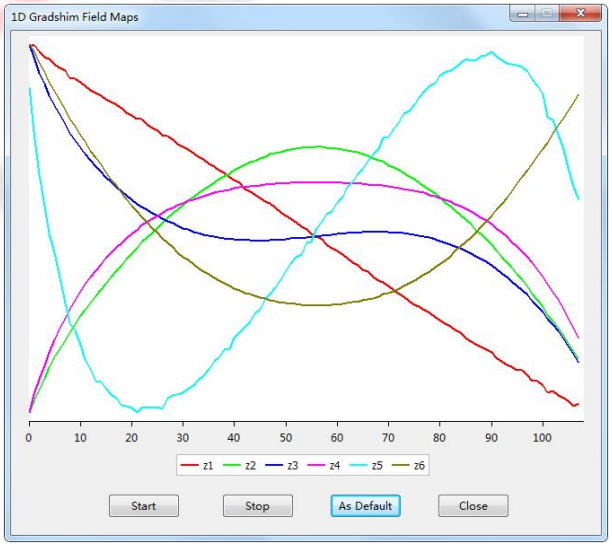


Figure 2.24 Show Map dialog

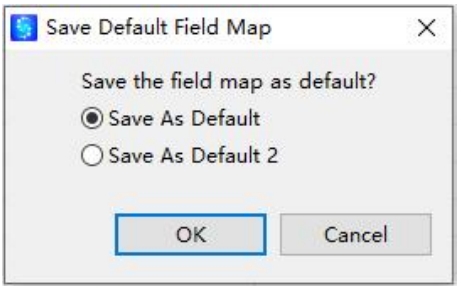


Figure 2.25 Save default field map selection box

(4) Clicking **Show Workspace**, will open the gradshim1d window in workspace (Figure 2.26), showing the whole process of gradient shimming.

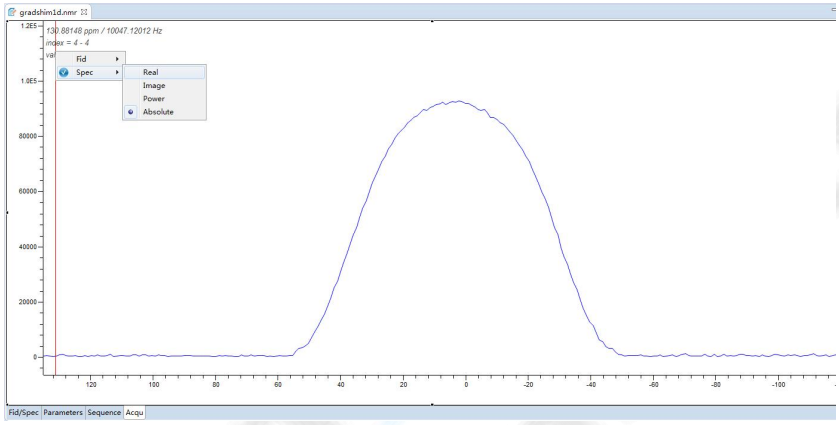


Figure 2.26 Process of gradient shimming

(5) Typing ***smartshim*** in command line can also start Smart Shim.

Note If the result using saved field maps is not satisfactory, it might be that the map is not suitable, you can make new map for shimming.

4. Smart Shim based on new Field Map

(1) If **Make Map** is selected, a new field map will be made first. The default way is to make field map using automatically optimized parameters. User can click **Setting** button to open a dialog box, then select “Set parameters manually” to set up proper values for each parameter, as shown in Figure 2.27.

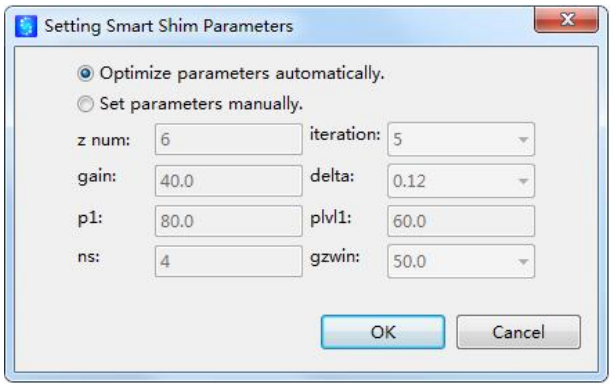


Figure 2.27 Parameter setting dialog

z num: number of axial shim coils used for making field map, 6 as default. User can change it according to the situation.

iteration: number of times for for gradient shim iteration. If data still dose not converge after this number of iteration, it will be stopped automatically.

gain: receiver gain for data acquisition during making map. The optimum value is the maximum one without overflow.

delta: waiting time before gradient pulse.

p1: pulse width used in the pulse sequence for field map.

plvl1: corresponding power for p1.

ns: number of scan for acquisition, 4 by default, or 8 for solvents with few deuterium atoms. This value can be changed according to different solvents.

gzwin: the percentage of gradient image profile over the spectral width used in gradient shimming. This should be adjusted when making new field map.

(2) Click **Start** button to make new field map. After the new map is made, the program will automatically start gradient shimming using the map. Clicking **Show Workspace** next to **Make Map** will open the gradshim1d window in workspace (Figure 2.26), showing the whole process of gradient shimming.

(3) At the end of making field map, a window will be open shown the new map (Figure 2.28). Right-clicking over it then select Show Field Map, or double-clicking the map, will open a window showing the map in the workspace area as in the case of using existing map (Figure 2.24).

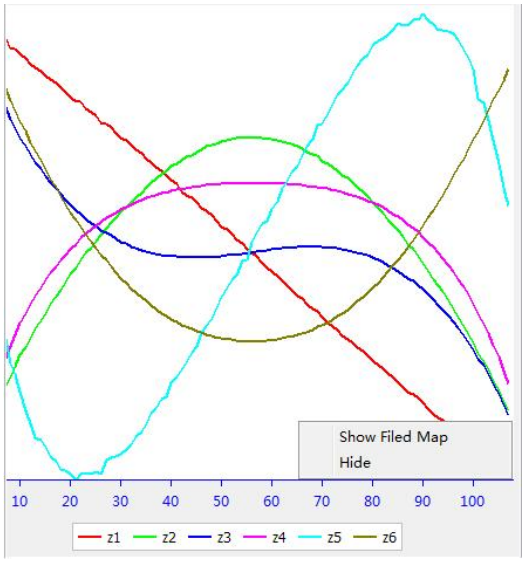


Figure 2.28 Shim map view box

(4) Typing s***martmapshim*** in command line can also start Smart Shim with new map.

5. Search Shim before and after Smart Shim

Whether using existing map or by making new map for Smart shim, combined use of search shim **Before** and **After** Smart Shim will result in much better field homogeneity. These options can be set in Smart Shim panel (see Figure 2.21)

(1) Click down-arrow next to **Before** and **After** to open the drop-down menu, then select one of the coil combinations for search shim (Figure 2.29). You can also click **Options > Preferences > Instrument > Shim > Smart Shim**, to customize the combination, iteration and steps for yourself, referring to 1.3.6 Options. There are two options, **FIDArea** and **LockLevel**, for Evaluate, as the same in Search Shim.

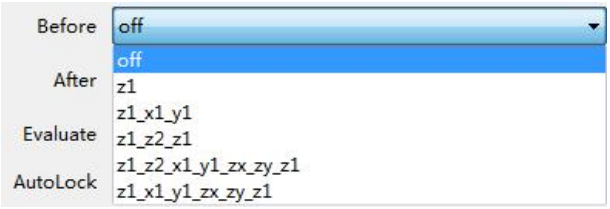


Figure 2.29 Tuning coil combinations of **Before**

(2) These setting can also be done by using commands:

gshim: smart shim using default map, without auto locking after shimming.

smartshimaa: smart shim using default map, plus search shim on z1-x1-y1-zx-zy, with auto locking after shimming.

smartshimaxyz: smart shim using default map, plus search shim on z1-x1-y1, with auto locking after shimming.

smartshimaz: smart shim using default map, plus search shim on z1, with auto locking after shimming.

smartshimba: search shim on z1-x1-y1-zx-zy, then smart shim using default map, with auto locking after shimming.

smartshimbxyz: search shim on z1-x1-y1, then smart shim using default map, with auto locking after shimming.

smartshimbz: search shim on z1, then smart shim using default map, with auto locking after shimming.

gmapshim*:* smart shim using new map, without auto locking after shimming.

smartmapshimaa: smart shim using new map, plus search shim on z1-x1-y1-zx-zy, with auto locking after shimming.

smartmapshimaxyz: smart shim using new map, plus search shim on z1-x1-y1, with auto locking after shimming.

smartmapshimaz: smart shim using new map, plus search shim on z1, with auto locking after shimming.

smartmapshimba: search shim on z1-x1-y1-zx-zy, then smart shim using new map, with auto locking after shimming.

smartmapshimbxyz: search shim on z1-x1-y1, then smart shim using new map, with auto locking after shimming.

smartmapshimbz: search shim on z1, then smart shim using new map, with auto locking after shimming.

6. Selecting the check box of **AutoLock** will enable the field being auto locked on after gradient shim, else the field will be unlocked. The default is on.

2.8.4 3D Smart Shimming

The principal of 3D Smart Shim is similar to Smart Shim, but with with XYZ three directional gradient encoding, which results in longer time for shimming.

The interface for 3D Smart Shim is shown in Figure 2.30. User can retrieve a saved map or create a new map for shimming.

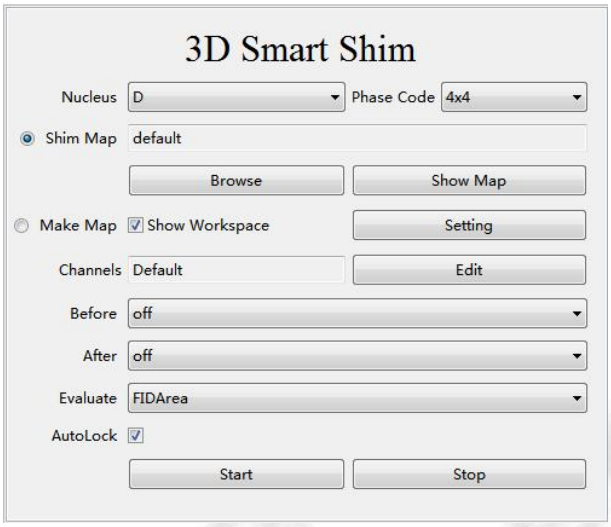


Figure 2.30 3D Smart Shim interface

As shown in above, the interface of 3D Smart Shim is similar to Smart Shim, with several additional options.

Nucleus: the nucleus used for 3D shimming, set as deuterium by default. It can be changed as H1 if your solvent is 90% H_2O+10% D_2O.

Phase Code: phase encoding matrix for 3D shimming, 4×4 by default. 8×8 or 16×16 can be used.

Channels: the shim coils used for 3D shimming, all being used by default. User can click **Edit** button to edit your own combinations of shim coils.

The detailed 3D Smart Shim procedures are explained as follows:

1. Click **Acquire** > **Shim** > **3D Smart Shimming**, or click the down arrow of button on tool bar then select **3D Smart Shimming** to open **3D Smart Shim** window (Figure 2.30).
2. Select **Nucleus,** the nucleus used for 3D shimming. The default nucleus is D (deuterium),

however, if the solvent has high content of H_2O, such as 90% H_2O+10% D_2O, it should be changed to H1.

3. Select **Phase Code** for phase encoding matrix, 4×4 by default. The 8×8 or 16×16 matrix can also be used.

4. 3D Smart Shim based on existing Field Map

(1) Select **Field Map** to use existing Field Map for shimming. Displayed in the box is a map named default, i.e., the system's default Field Map for current solvent as shown in the left of Figure 2.31. The default map will be displayed under the 3D Smart Shim window.

(2) Click **Browse** to open a window for the selection of user created Field Maps, as shown in the right of Figure 2.31. The maps coming along with the system is in the directory of default under current solvent, for example, when the solvent is D_2O, the path is *D:\SpinStudioJ-1.4.9alpha-20190624\system\data\solvents\D2O\gradshim3d\default*. The user created maps are saved in the directory of gradshim3d under current solvent, with a name in the format of yyyymmdd(n), where n is the sequential number, starting from 0, for these maps created in the same day. For example, 20190620(0) is the first map created at June 20, 2019.

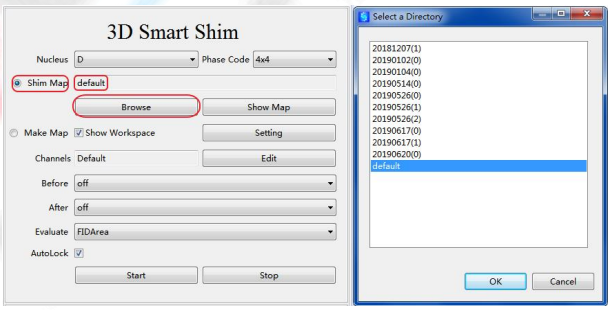


Figure 2.31 Select the user-created 3D shim map

(3) Click **Setting** button to open the dialog box for shimming parameters. User can select **get parameters from field map file**, or select **custom parameters** to give new values for delta and iteration, and select the shim coils to be used (Figure 2.32).

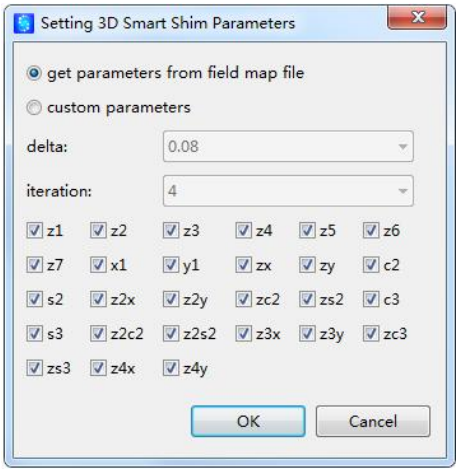


Figure 2.32 Setting dialog when Shim Map is selected

(4) Select **Channels**, the shim coils coils used for 3D Smart Shim. The default is composed of all coils for current spectrometer. User can click **Edit** to open a edit window, then click **Show** button to check the default coils (Figure 2.33), or click **Add** button to define your own coil combinations and give a name in the **Channel Name** box (Figure 2.34).

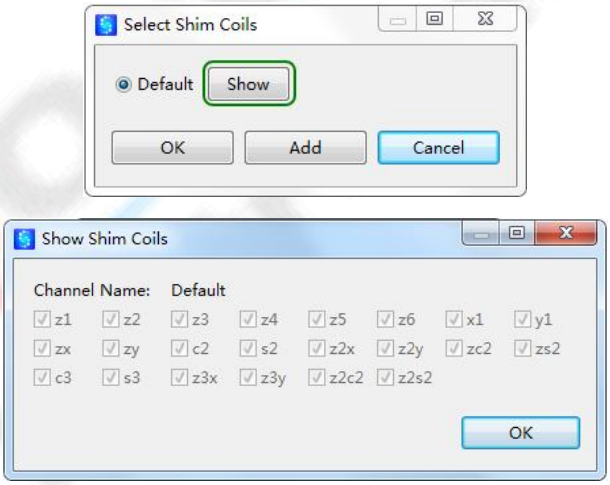


Figure 2.33 Show button function in the Edit dialog

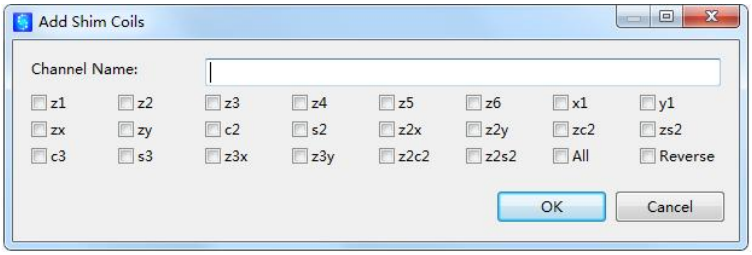


Figure 2.34 Add button function in the Edit dialog

(5) User can click **Show Map** button to open a window showing the map in the workspace area (Figure 2.35). You can check each coil by clicking **Next** or **Back**. Clicking **Start** button here or in the 3D Smart Shim panel will initiate the 3D gradient shimming using current field map. This process can be seized by clicking **Stop** button. If the currently used map was made by user, you can click **As Default** to save it as the system default one for current solvent. Click **Close** to close the map window.

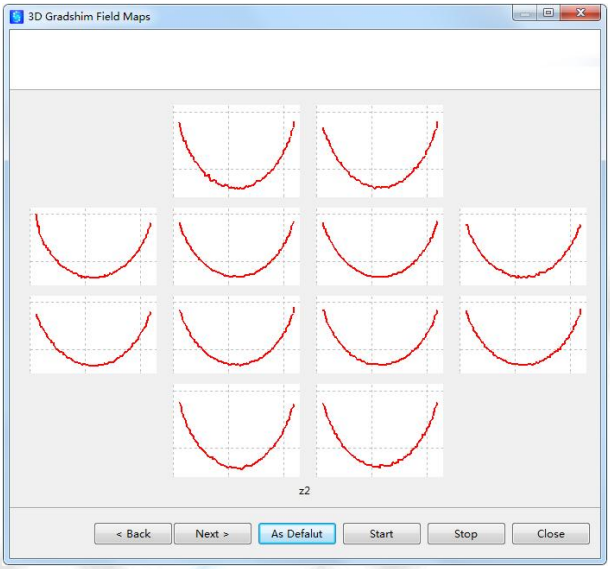


Figure 2.35 Show map dialog

(6) Clicking **Show Workspace**, will open the gradshim1d window in workspace (Figure 2.36), showing the whole process of gradient shimming.

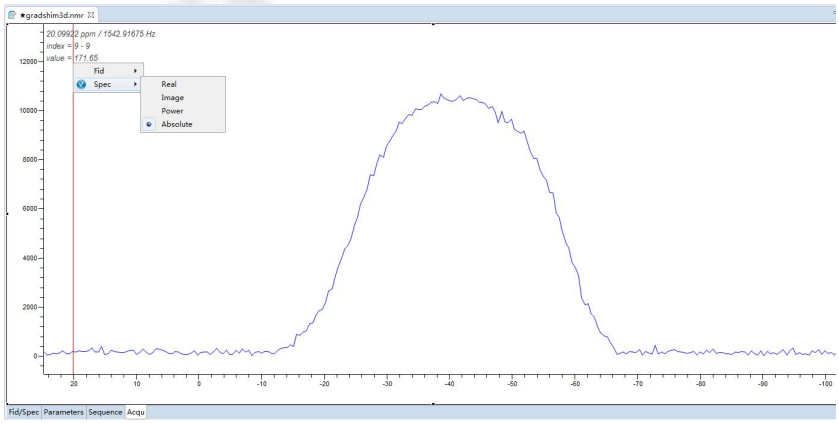


Figure 2.36 Process of gradient shimming

(7) Typing ***smartshim3d*** in command line can also start 3D Smart Shim.

Note If the result using saved field maps is not satisfactory, it might be that the map is not suitable, you can make new map for shimming.

5. 3D Smart Shim based on new Field Map

(1) If **Make Map** is selected, a new field map will be made first. The default way is to make field map using automatically optimized parameters. User can click **Setting** button to open a dialog box, then select Set parameters manually to set up proper values for each parameter, as shown in Figure 2.37.

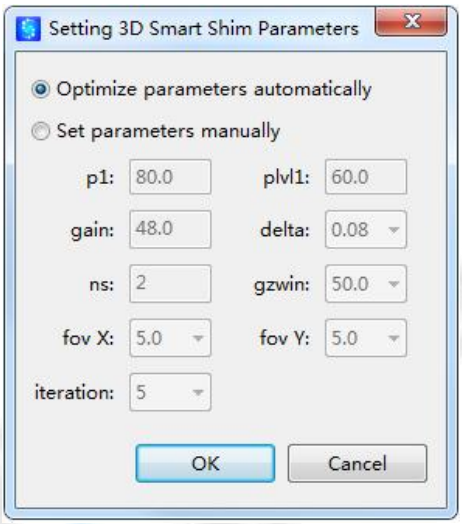


Figure 2.37 Setting dialog when Make Map is selected

p1: pulse width used in the pulse sequence for field map.

plvl1: corresponding power for p1.

gain: receiver gain for data acquisition during making map. The optimum value is the maximum one without overflow.

delta: waiting time before gradient pulse.

ns: number of scan for acquisition, 2 by default, 4 is also ok.

gzwin: the percentage of gradient image profile over the spectral width used in gradient shimming.

fov X: field of view in X direction.

fov Y: field of view in X direction.

iteration: number of times for for gradient shim iteration.

(2) Select **Channels**, the shim coils coils used for 3D Smart Shim. The default is composed of all coils for current spectrometer. User can edit for your own coil combinations. Please refer to above section 3D Smart Shim based on existing Field Map for details.

(3) Click **Start** button to make new field map. After the new map is made, the program will automatically start gradient shimming using the map. Clicking **Show Workspace** next to **Make Map** will open the gradshim3d window in workspace (Figure 2.36), showing the whole process of gradient shim.

(4) Typing s***martmapshim3d*** in command line can also start 3D Smart Shim.

6. Search Shim before and after Smart Shim

Whether using existing map or by making new map for Smart shim, combined use of search shim **Before** and **After** Smart Shim will result in much better field homogeneity. These options can be set in 3D Smart Shim panel (Figure 2.30)

Click down-arrow next to **Before** and **After** to open the drop-down menu, then select one of the coil combinations for search shim (Figure 2.38). You can also click **Options** > **Preferences** > **Instrument** > **Shim** > **Smart Shim**, to customize the combination, iteration and steps for yourself, referring to 1.3.6 Options. There are two options, **FIDArea** and **LockLevel**, for Evaluate, as the same in Search Shim.

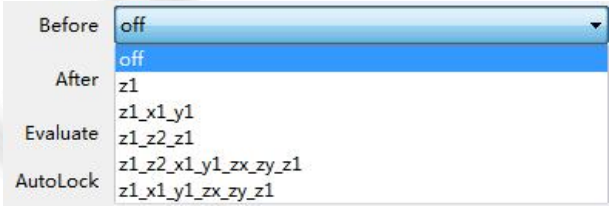


Figure 2.38 Tuning coil combinations of **Before**

7. Selecting the check box of **AutoLock** will enable the field being auto locked on after gradient shim, else the field will be unlocked. The default is on.

2.9 Experiment Setup

2.9.1 Parameter Setup

Acquisition related parameters can be setup in **AcquParams** tab of an NMR experiment in workspace, such as observe nucleus (nuc) and corresponding basic frequency (frqb) and offset (frqo), power (plvl1), spectral width (sw/swhz), acquisition time (acqtime) , receiver gain (gain); decoupling nucleus (nuc1) and corresponding basic frequency (frqb1) and offset (frqo), power (plvl2), as shown in Figure 2.39. These parameters are listed under one of the following groups: Base, Experiment, Constant, RF Channels, Duration and Pulse. The icon A means that the current display is active for parameter setup, you can enter or change values for parameters with their value boxes in white. Clicking A will switch it to S, representing a status only for parameters display, in which no parameter can be changed.

The parameters can be searched by entering any letters in **Filter** box.

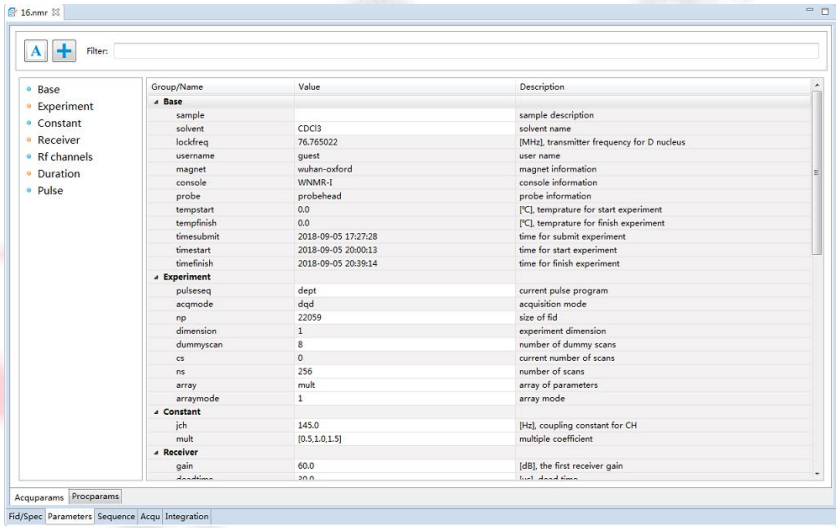


Figure 2.39 Experimental parameter setting page

There are three ways for parameter setup: (1) directly entering a value for parameter, such as ns, gain, sw, frqo; (2) clicking to open a drop-down list for selecting a value for the parameter, such as solvent, arraymode, nuc; (3) clicking to activate the ... button, clicking which will open a dialog box displaying options for selection, such as pulseseq (Figure 2.40). User can select pulse sequence from **System** library or **User** library, with Filter to

help you select quickly. Click **OK** to close the window.

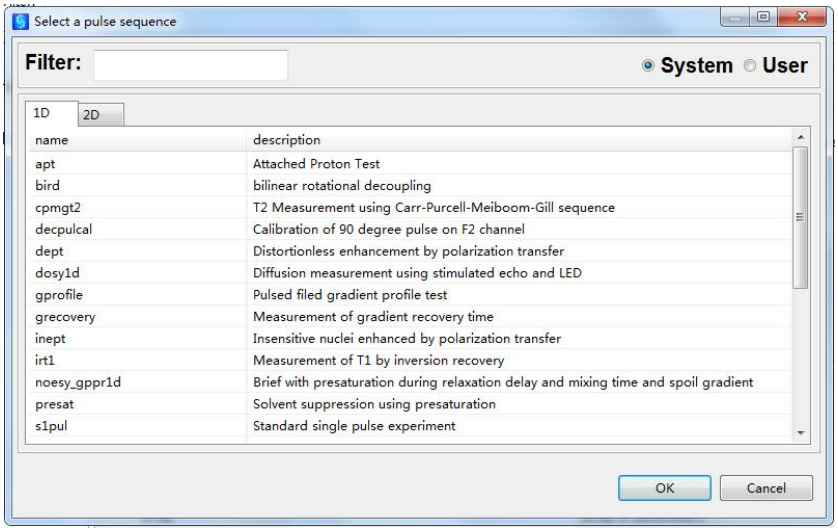


Figure 2.40 Pulse sequence selection dialog

The parameters can also be setup in command line. For example, you can type ***ns=8*** to set the number of scans to 8; type ***pulseseq='apt'*** to set the pulse sequence as apt.

Different pulse sequence includes different parameters. When right-clicking over the parameter line, a menu will appear with options where you can select one for manipulation (Figure 2.41).

Figure 2.41 Right-clicking function of parameter line

User can select Add Parameter, or click the  button in parameter panel to add parameter for display. A dialog box will appear as shown in Figure 2.42. If you want to add some parameter for your currently used pulse sequence, simply select the check box of the parameter, or click **New** button to open a dialog box for creating new parameter (Figure 2.43). You must give a value to the field with asterisk.

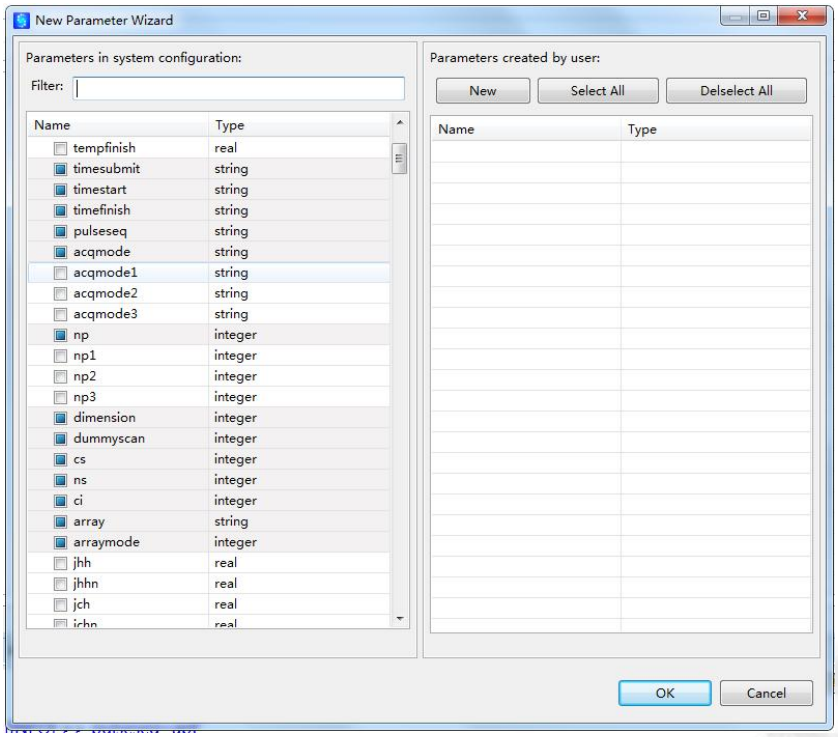


Figure 2.42 Add parameter dialog

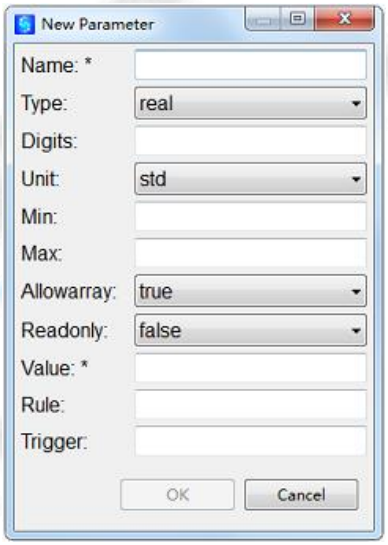


Figure 2.43 Create parameter dialog

Compared with 1D NMR experiment, 2D NMR experiment includes additional parameters for indirect dimension, e.g. sw1, np1 for observation, si1, sb1, sbs1 for processing.

When np1 > 1, a 1D experiment becomes 2D experiment.

2.9.2 Displaying Pulse Sequence

Pulse sequence is displayed in **Sequence** window of the workspace. The pulse sequence diagram and the source file are separately displayed in **Diagram** and **Source** tabs. Some parameters are also displayed in the diagram, as shown in Figure 2.44. The source file can be directly edited (Figure 2.45), and the revised version will be saved in current experiment, without changing the system's original pulse sequence.

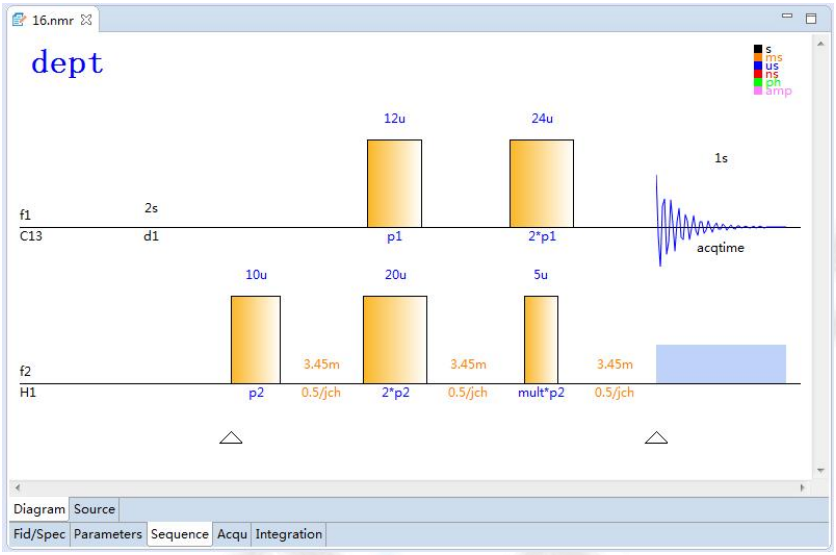


Figure 2.44 Pulse sequence graphic display

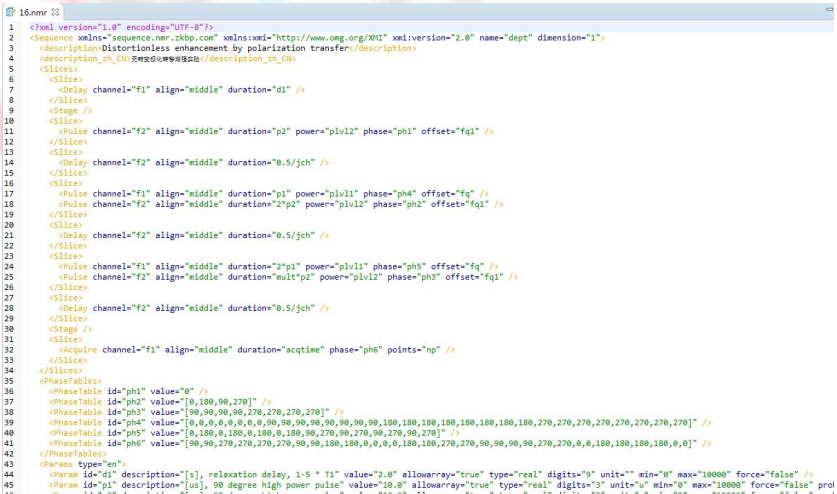


Figure 2.45 Pulse sequence text display

2.10 Data Acquisition and Storage

2.10.1 Starting and Stopping Acquisition

Firstly, type ***again*** in command line to get optimized gain value, then type ***go***, or click  button in tool bar to start data acquisition (FID will be displayed when acquisition is finished) , or type ***ga*** to execute data acquisition and processing (Fourier transformation, automatic phase correction and spectrum display). If the acquisition need to be stopped, type command ***aa*** to stop acquisition in current workspace or ***aaa*** to stop acquisitions in all workspaces.

2.10.2 Repetitive Acquisition

Repetitive acquisition is designed for interactive parameter optimization. User can look at the results after changing parameter in real time.

Open or activate the experiment with parameters to be optimized in workspace. Type ***gs*** in command line to open the gs Editor window (Figure 2.46), and to start the repetitive acquisition at the same time. User can optimize your parameter by changing value directly in the box, by left-and right-clicking the  button, or by rolling mouse wheel forward and backward over the button to change the value based on the increment shown left. The increment can be changed by clicking the down-arrow and select other values. Click the 

button to apply the new value and start a new acquisition. The resulting spectrum will show the effect of the revised parameter. To exit this mode, you need to click  button to stop first, then click  button to exit.

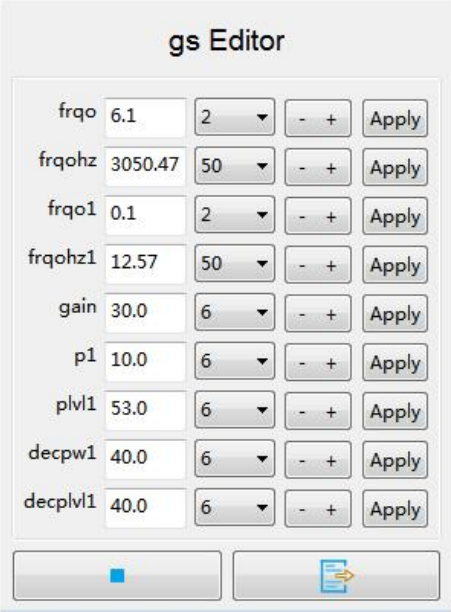


Figure 2.46 gs editor interface

If you exit the Gs Editor without stopping it first, you can type ***aa*** in command line to stop it, or type ***gsedit*** to open the window again, then stop it.

2.10.3 Data Archiving

SpinStudioJ provide two methods for data archiving:

(1) After acquisition, the data will be automatically saved in the current directory.

(2) Save data to other designated directory. Click **File** > **Save As** in menu bar, or click the down-arrow of the button in tool bar and select **SpinStudioJ Data**. A dialog box will appears as shown in Figure 2.47,where you can enter the whole path in **Save Directory** box or click **Browse** button to select the destination directory for data archiving. You can enter a name for your data in **Exp Name** box, and even add a password to protect your data by clicking **Add Password** button, then entering the password and confirming it (Figure 2.48).

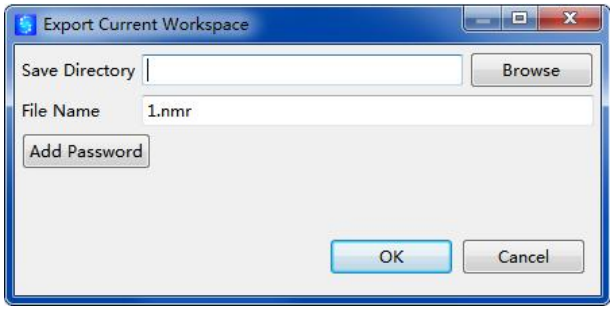


Figure 2.47 Dialog for saving experiment data

Figure 2.48 Add password dialog

2.10.4 Data Acquisition for 2D Experiments

The start of data acquisition for 2D experiments is the same as for 1D experiment. After all preparation is done, type again in command line to get optimized gain value, then type go, or click button in tool bar to start data acquisition.

2.11 Automation

Automation can simplify experiments and save much time for large number of samples in routine work. User only need to assign the sample information, solvent and pulse sequence for each sample, then the program will automatically complete other things including sample inserting, auto locking, auto tuning, auto shimming, auto gain, data acquisition and processing. Most routine 1D and 2D experiments can be done in automation.

2.11.1 Automation Login

Click **Acquire** > **Automation** on menu bar, or click button on tool bar. The

Automation login window appears, you can enter user name and password here to log in (Figure 2.49). The administrator can use admin as username and password for first time login. Other users and passwords are created by admin.

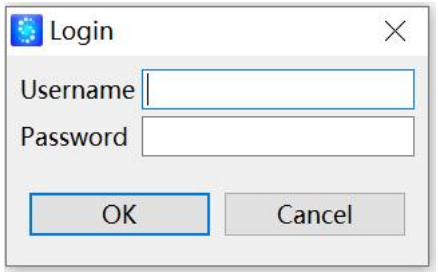


Figure 2.49 Automation login dialog

After opening the automation experiment interface, the automation experiment data navigation panel will appear on the left side of the data navigation panel to display the automation experiment data of the current logged-in user. If the administrator admin logs in, the automation experiment data of all users will be displayed, as shown in Figure 2.50. You can right-click in any level directory to refresh the data or open the current folder (**Open Local Folder**).

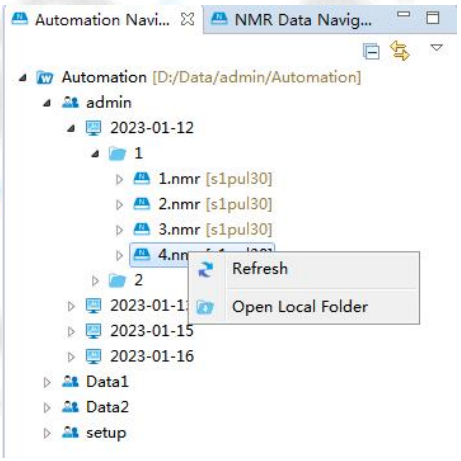


Figure 2.50 Automation experiment data navigation panel

After login, the Automation interface will be shown in the workspace, which includes tool bar, experiment queue list and history list, as shown in Figure 2.51.

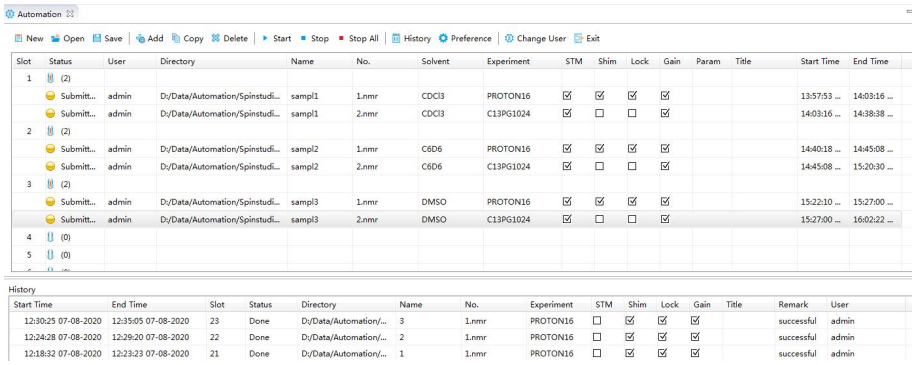


Figure 2.51 Automation interface

1. Toolbar for automation

Figure 2.52 Toolbar for automation

New: Empty all experiments under all sample slots and open a new blank experiment list.

Open: Empty all experiments under all sample slots and open the selected list of experiments that have been saved before. Note: You must have saved the experimental queue before you can select the files that need to be opened.

Save: Save the current experiment queue information to a file.

Add: Add an experiment, a sample slot must be selected first.

Copy: Copy experiments. You can copy a single experiment or all experiments under a Slot.

Clear Done: Clear all experiments with Done status in the experimental cohort.

Delete: Delete a single experiment or all experiments under a Slot.

Start: Start the experiment for all current users in the experiment queue. Except for administrators who submit experiments with the Editable status of all users in the queue.

Stop: Stop the experiment for all current users in the experiment queue.

Stop All: Stop experiments for all users in the experimental queue. You need to enter an administrator password to perform this operation.

Pause: Pause the current experimental queue, after pausing the button name becomes **Continue**. The pause operation completes the experiment currently in progress and the

experiment in Queued state, and the other experiments remain in the Submitted state. At this time, you can add and submit automated experiments normally, and keep the Submitted state after the experiment is submitted. Other manual operations can also be performed, such as tuning, locking field, shimming, etc. When the manual operation is completed, that is, the software is in the Idle state, you can click the **Continue** button to continue to complete the automated experimental queue. For the remaining experimental queue, tuning, shimming, locking field, etc. will be judged again according to the configuration to determine whether to check. Both pause and continue operations require an administrator password to use.

History: List of automation experiment histories.

Preference: Automation experiment preferences, configure automation related information. This button is displayed when the administrator admin logs in, and only the administrator user can configure it. Referring to 2.11.2 Preference for details.

Slot Status: Display interface for the experimental state of all slots.

Accounting Record: Open the accounting record interface. When the administrator admin logs in, the button will be displayed, and after clicking, the password text box will pop up. Enter the administrator password to open the accounting record interface (Figure 2.53) to view and manage.

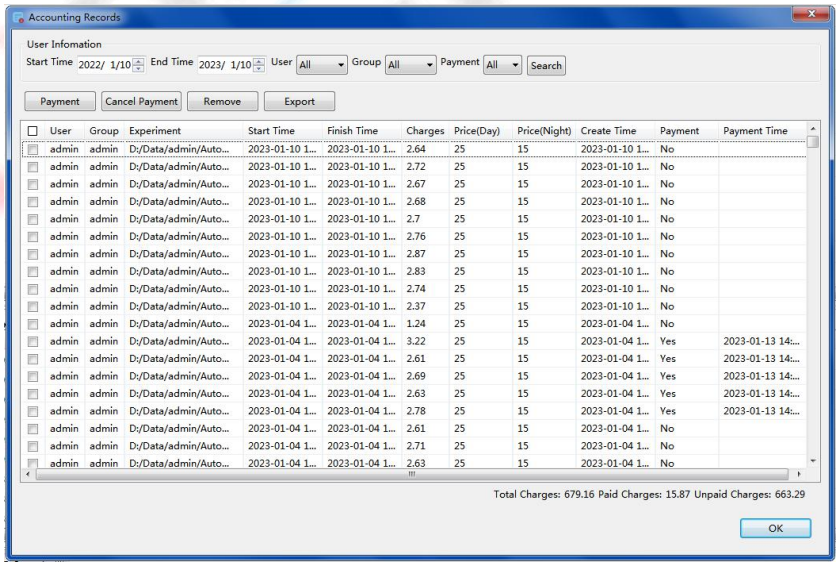
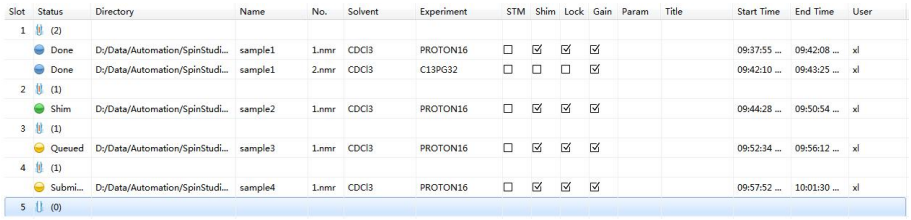


Figure 2.53 Accounting record

Change User: Switch the logged-in user.

Exit: Exit the automation interface.

2. List of automation



Slot	Status	Directory	Name	No.	Solvent	Experiment	STM	Shim	Lock	Gain	Param	Title	Start Time	End Time	User
1	(2)														
	Done	D:/Data/Automation/SpinStudi...	sample1	1.nmr	CDCl3	PROTON16	☐	☑	☑	☑			09:37:55 ...	09:42:08 ...	xl
	Done	D:/Data/Automation/SpinStudi...	sample1	2.nmr	CDCl3	C13PG32	☐	☐	☐	☑			09:42:10 ...	09:43:25 ...	xl
2	(1)														
	Shim	D:/Data/Automation/SpinStudi...	sample2	1.nmr	CDCl3	PROTON16	☐	☑	☑	☑			09:44:28 ...	09:50:54 ...	xl
3	(1)														
	Queued	D:/Data/Automation/SpinStudi...	sample3	1.nmr	CDCl3	PROTON16	☐	☑	☑	☑			09:52:34 ...	09:56:12 ...	xl
4	(1)														
	Submi...	D:/Data/Automation/SpinStudi...	sample4	1.nmr	CDCl3	PROTON16	☐	☑	☑	☑			09:57:52 ...	10:01:30 ...	xl
5	(0)														

Figure 2.54 Automated experimental queue display interface

Slot: Displays the numbers of all slots.

Status: Displays the status of the current experiment (Editable, Cancelled, Submitted, Queued, Tuning, Lock, Shim, Gain, Acquire, Done, Error, Temp, Spin).

Use: The user corresponding to the experiment.

Directory: Displays the local path to the current experiment data .nmr file. The path is automatically generated according to the configuration in **Preference** when creating a new experiment, and the path can also be switched by pulling down.

Name: The file name corresponding to the current experiment.

No.: The number corresponding to the current experiment.

Solvent: Solvent name corresponding to the current experiment.

Experiment: Experiment template corresponding to the current experiment.

TempSpin: Whether to perform temperature control or rotation operation on the current experiment. While setting the temperature control and rotation parameters in the Edit dialog box, the check box here must be checked to perform the operation.

STM: Automatic tuning and matching, check means do automatic tuning operation, not check means not to do.

Shim: One-dimensional gradient shimming, check means to do one-dimensional shimming operation, not check means not to do.

Lock: Auto lock. Check means to do automatic locking operation, not check means not to do.

Gain: Automatic gain, check means to do automatic gain operation, not check means not

to do.

Param: Editing experimental parameters. The experiment can be edited by clicking **Edit** in Editable state, referring to 4 **Checking and Modification of Parameters** for details.

Title: Detailed remarks or description information of the current experiment, the user can choose whether to fill in according to needs.

Start Time: The start time of the current experiment.

End Time: The end time of the current experiment.

3. History of automation

Displays the automation experiment history, completed experiments, failed experiments, and error messages. Double-clicking on a selected history experiment will open the experiment data in the workspace.

4. Right-click menu of automation

(1) Right-click menu of slot

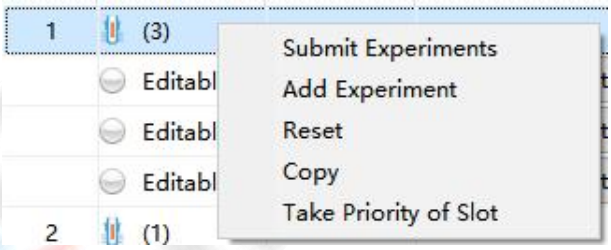


Figure 2.55 Right-click menu of slot

Submit Experiments: Submit all experiments under the current slot.

Add Experiment: Add experiment.

Reset: Reset the slot. This option can be used to clear all experimental information in the current slot when all experimental states in the current slot are Done or Error.

Copy: Copy all experiments under the slot into the specified slot. This operation has nothing to do with the experimental state and the user, that is, the experiment in any state of any user can be copied.

Take Priority of Slot: Prioritize all experiments under the current slot.

(2) Right-click menu of experiment

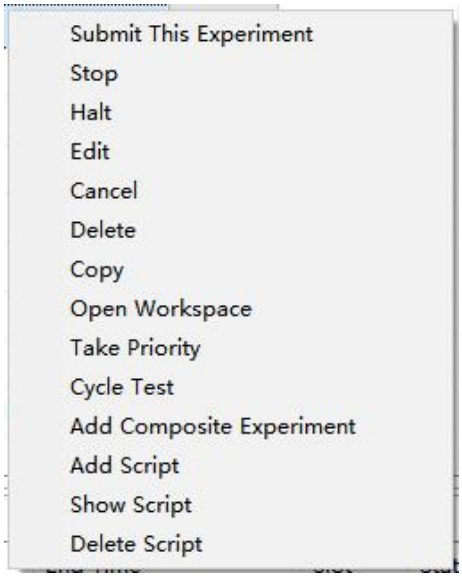


Figure 2.56 Right-click menu of experiment

Submit This Experiment: Submit the currently selected experiment. This operation can only be performed on experiments under the currently logged-in user, except for administrator.

Stop: Stop the currently selected experiment. This operation can only be performed on experiments under the currently logged-in user, except for administrator.

Halt: Suspend sampling and perform data processing, data conversion and data uploading according to software configuration.

Edit: Edit the currently selected experiment and switch it to the Editable state. This can be performed for experiments in Done, Error, and Cancelled states. This operation can only be performed on experiments under the currently logged-in user, except for administrator.

Cancel: Switch the Editable state to the Cancelled state. After switching, the experiment cannot edit the experimental parameters.

Delete: Delete the selected experiment.

Copy: Copy the selected experiment into the specified slot. Experiments for all users and in any state can be copied.

Open Workspace: Opens the workspace for the current experiment. Experiments that have not yet been created in the workspace cannot be opened. When the experiment status

changes to Queued after the experiment is submitted, the workspace for the current experiment is created. This operation can only be performed on experiments under the currently logged-in user, except for administrator.

Take Priority: Prioritizing the currently selected experiment.

Cycle Test: After checking, all experiments will be done from the beginning. This function is only used when engineers install and debug.

Add composite Experiment: This function is mainly used to optimize spectral width and frequency offset.

2.11.2 Preference

After login, the administrator can click the Preference button on the tool bar to open the Preference setting window (Figure 2.57). Two parts, the general settings for automation experiments (**Automation Configuration**) and user related settings (**User Settings**), are included in Preference, which will be introduced in the following section.

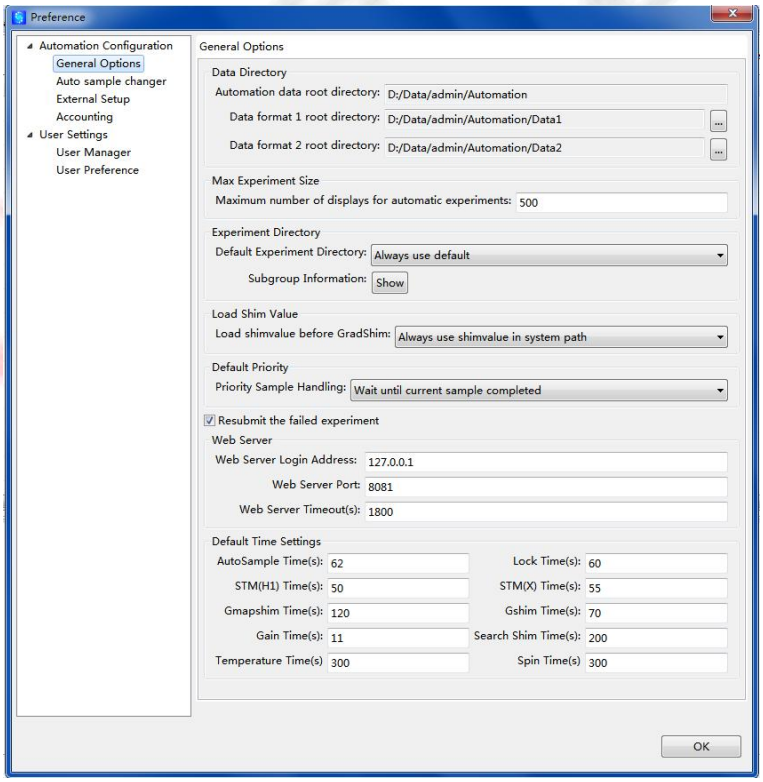


Figure 2.57 Automation configuration interface

1. Automation Configuration

(1) General Options

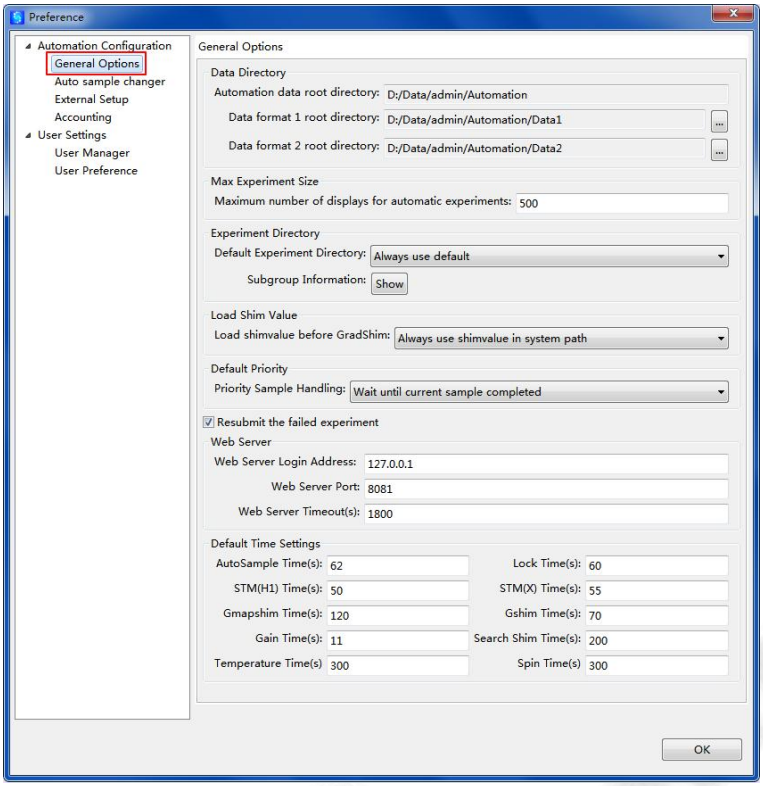


Figure 2.58 General Options configuration interface

Data Directory

Automation data root directory: the root directory where the data of all automation experiments are saved. When user create new automation experiments, this directory is used as default value.

Data format 1 root directory: the root directory for saving data converted to Varian format.

Data format 2 root directory: the root directory for saving data converted to Bruker format.

Max Experiment Size

Maximum number of displays for automatic experiments: The default value is 500, and the value range is [100, 1000].

Experiment Directory

Default Experiment Directory: There are two options, “Always use default” and “Consistent with the last experiment under the selected slot”. When configured as the default option “Always use default”, all new experiments use the default path of the user configured in User Preference; When configured to “Consistent with the last experiment under the selected slot”, if there are no other experiments under the slot when the new experiment is created, the default path is used. If there is an experiment, it will be consistent with the path of the previous experiment.

Subgroup information: Click the **Show** button, the subgroup dialog box pops up (Figure 2.65). Then click the **Add** button to pop up the **Add Subgroup** dialog box (Figure 2.66). After adding subgroups, check the corresponding data path in the **User Preference**. When a new experiment is created in the automated experiment interface, the user has multiple subgroup paths to choose from.

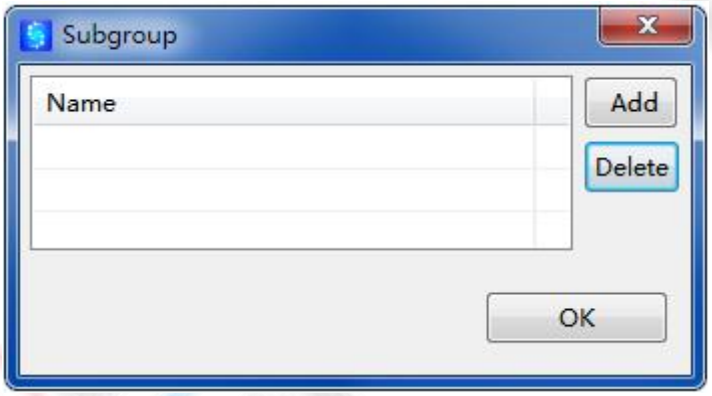


Figure 2.59 Subgroup dialog

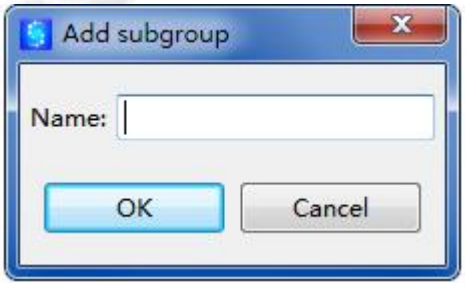


Figure 2.60 Add Subgroup dialog

Load Shim Value

Load shimvalue before GradShim: To load the shim values before gradient shimming,

there are two options: “Always use shimvalue in system path” and “Use shimvalue in current solvent path”. The default configuration is “Always use shimvalue in system path”. When “Use default shimming value before gradient shimming” in **Use Preference** is checked, the shim values under the system path or under the current sample solvent will be load according to the current configuration.

Default Priority

Priority Sample Handing: There are two options, “Wait until current sample completed” and “Do Priority Samples immediately”. When configured as the default option “Wait until current sample completed”, all experiments in the slot where the current Queued state experiment is located will be completed before the priority experiment; when configured as “Do Priority Samples immediately”, only the Queued state experiment will be completed, and then the priority experiment will be carried out immediately.

Resubmit the failed experiment

If this option is checked, when the experiment fails, the History column will mark the reason for the experiment failure and write "will be resubmitted", the log line will also prompt the experiment failure and "will be submitted again!", and then the experiment will be resubmitted again. If there is no Queued state experiment behind the failed experiment, resubmit it and do it again immediately; if there is a Queued state experiment behind the failed experiment, the resubmitted experiment will be given priority, and after the Queued state experiment is finished, go back to do the resubmitted experiment immediately.

Web Server

Web Server IP Address: IP address of the Web server for visiting automation interface from web. This is set by engineer, not allowed to be changed by user.

Web Server Port: port number for the Web server. This is set by engineer, not allowed to be changed by user.

Web Server timeout: The timeout period of the web server is generally 1800s by default. After the interface is not operated, the interface information is no longer updated, and the

dialog box prompts the login timeout. When you click **OK**, it will exit to the login interface.

Default Time Settings

AutoSample Time: The time of injecting sample automatically through auto sampler changer in s.

Lock Time: The time of automatic lock in s.

STM(H1) Time: Time of auto tune for H1 in s.

STM(X) Time: Time of auto tune for X in s.

Gmapshim Time: Time of making 1D field map and shimming in s.

Gshim Time: Time of Shimming with default 1D field map in s.

Gain Time: Time of auto gain in s.

Search Shim Time: Time of search shim in s.

Temperature Time: The time it takes to reach the set temperature and stabilize in s.

Spin Time: The time it takes to reach the set speed and stabilize in s.

Each estimated time needs to be further optimized by the engineer based on the actual software and hardware conditions. The time of automatic lock and shimming is based on the user's common solvent.

(2) **Auto Sample Changer**

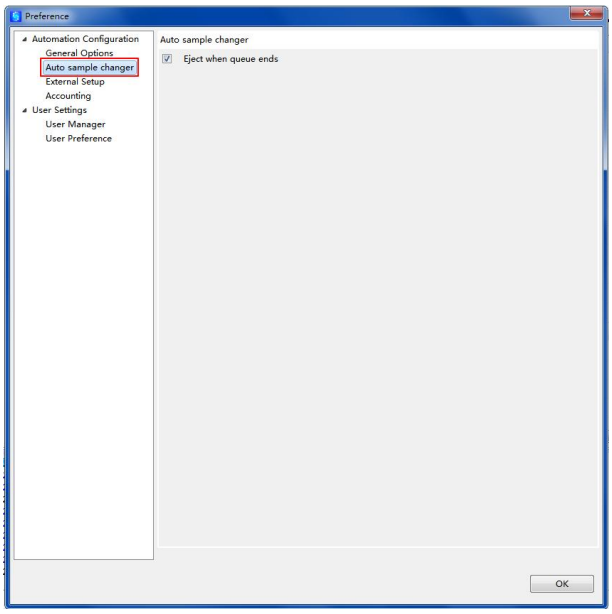


Figure 2.61 Configuration for auto sample changer

Eject when queue ends: decide whether to eject the sample after all experiments in queue are finished.

(3) External Setup

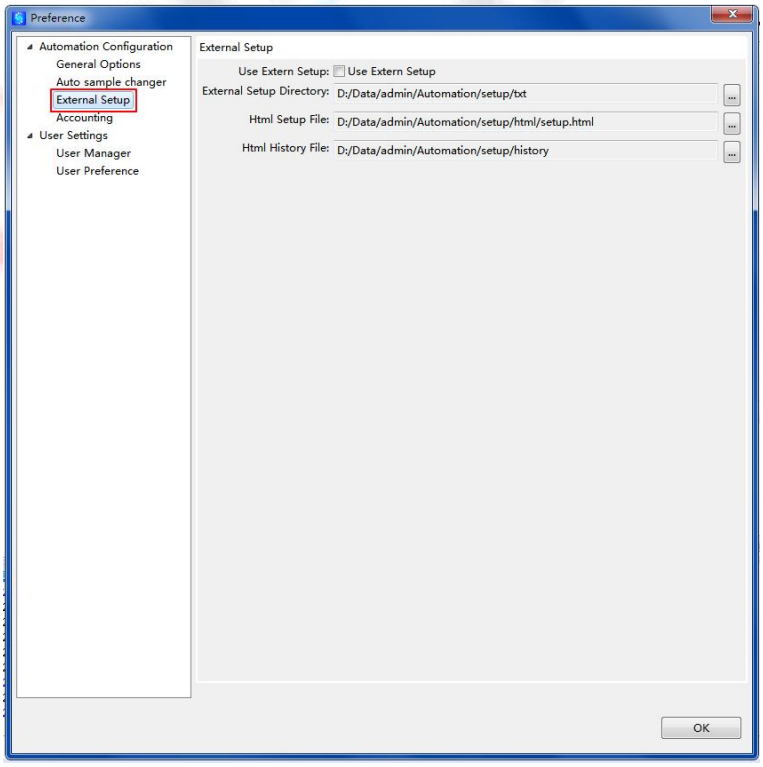


Figure 2.62 Configuration for external Sutup

Use Extern Setup: Whether to use external Setup function.

External Setup Directory: The path of the setup file when submitting an experiment using the Setup function.

Html Setup File: The path of the log file of the experiment submitted using the Setup function.

Html History File: The path of the history file of the experiment submitted using the Setup function.

(4) Accounting

Click **Accounting** and a password box will pop up, as shown in Figure 2.63. After entering the administrator's password, the accounting configuration interface will open (Figure 2.64).

Figure 2.63 Password box

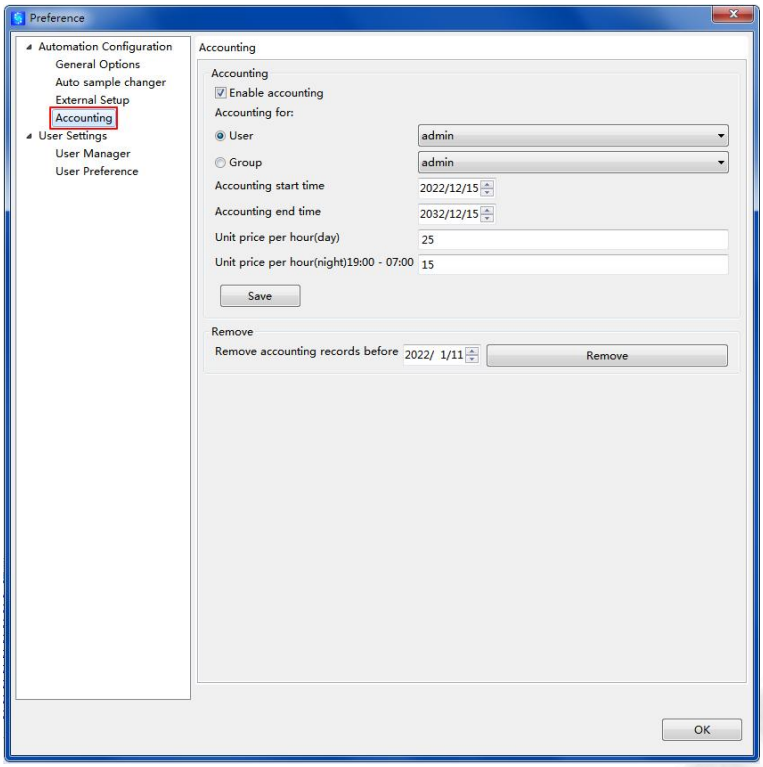


Figure 2.64 Accounting configuration

Accounting

Enable accounting: Enable the accounting function. The default is not checked, that is, not enabled.

Accounting for: You can select **User** or **Group**. Select “Group” to unify settings for all users under the operating system; select “User” to personalize the user. The settings include the starting and ending time and charging price below. After the settings are completed, click the **Save** button below to take effect.

Accounting start time: The time when accounting starts. The default time is the time when the interface is first opened.

Accounting end time: The time when accounting ends. The default time is ten years after the first time the interface is opened.

Unit price per hour(day): Unit price per hour for daytime, the default value is 25.

Unit price per hour(night)19:00-07:00: Unit price per hour for night, the default value is 15.

Save: Save settings button. After the above settings are completed, you need to click the

button to take effect.

Remove

Remove accounting records before: Remove the accounting records of all users under all operating systems before a certain date. After setting the date, click the **Remove** button. The default date is one year before the date of opening the interface.

2. User Settings

(1) User Manager

The administrator can use **User Manager** to add, edit or delete Automation users (Figure 2.65).

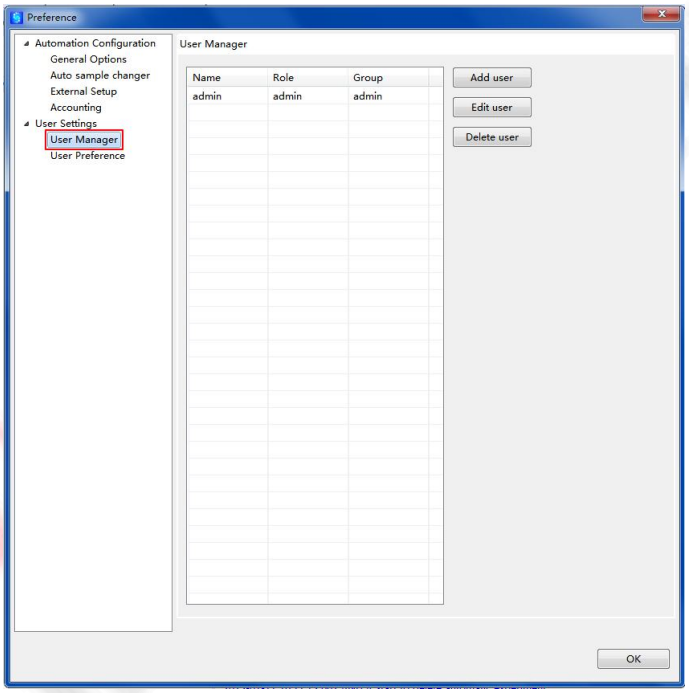


Figure 2.65 Interface of User Manager

Click **Add User** to open a dialog box (Figure 2.66). The administrator can set the user name and password for a new user and assign its **Role**. The **Role** is "guest" for all users created by administrator. **Group** is divided into "General" and "Advanced". If it is "General", the user can only modify the General parameters and does not have the authority to modify Advanced parameters.

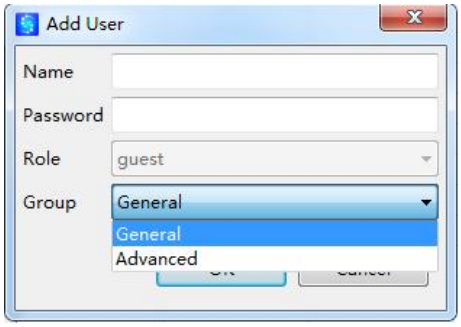


Figure 2.66 Add a new user

The administrator can select a user and click **Edit User** to modify its password (Figure 2.67), or click **Delete User** to delete it.

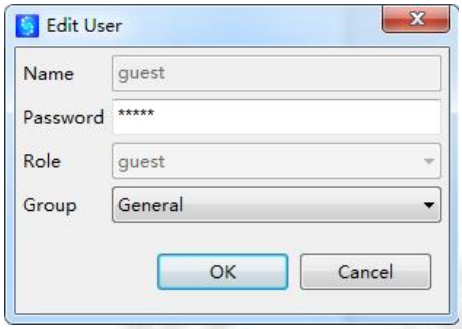


Figure 2.67 User editing interface

(2) User Preference

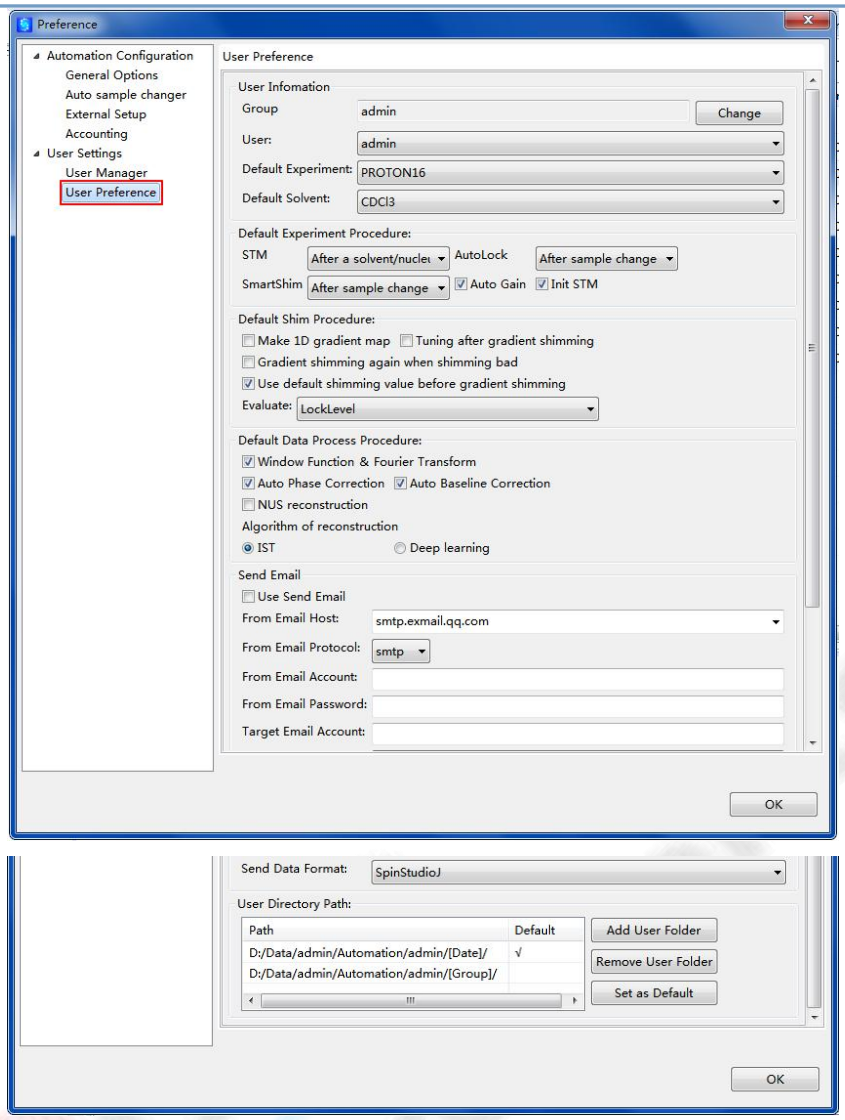


Figure 2.68 User Preference configuration

Information related to the automation experiments for each user is setup in **User Preference**. User will use these information as default conditions to created new automation experiments.

The first four items are basic. **Group** is the operating system user of the workstation. **User** can only select from the drop-down list of current operating system users (**Group**) in **User Manager**. There are many options for **Default Experiment** template and **Default Solvent**.

The second part is **Default Experiment Procedure**, which include **STM**, **Auto Lock**, **Smart Shim** and **Auto Gain**. **STM** has three options: “Always”, “Never”, “After a solvent/nucleus change”. The default setting is “After a solvent/nucleus change”, that is,

when the experiment is submitted, each experiment in the queue will be compared with the previous experiment that performed STM. If the nuclei are different, check STM. If they are the same, further judge whether the solvent is the same. Do not check if the solvent is the same, and check if the solvent is different. When STM is set to “Always” or “Never”, check or uncheck STM for each experiment. **Init STM** is the operation of initializing the motor during STM. It is recommended to check it.

AutoLock and **SmartShim** have four options: “Always”, “Never”, “After solvent change”, and “After sample change”. The default setting is “After sample change”, that is, “AutoLock/SmartShim” will be checked every time the sample is changed. If it is set to “After solvent change”, when the experiment is submitted, each experiment in the queue will be compared with the previous experiment that performed AutoLock/SmartShim. If the solvent is different, check AutoLock/SmartShim, if the solvent is the same, uncheck it .

The third part is the **Default Shim Procedure**, that is, choose whether to do one-dimensional gradient map to shimming (Make 1D gradient map), whether to do search shimming(Tuning) after gradient shimming (Tuning after gradient shimming) and the evaluation method of search shimming (Evaluate), whether to do gradient shimming again when shimming effect becomes poor (Gradient shimming again when shimming bad), the standard of shimming deterioration is that the final residual error of gradient shimming is greater than the threshold set by the engineer, and the system field value will be load at this time , and then shim again; and whether to load the default field value before each gradient shimming (Use default shimming value before gradient shimming). At this time, the system shim value or the shim value under the current solvent can be load, which is determined by the settings in **General Options**.

The fourth part is the Default Data Process Settings. These selections decide whether to perform ***wft***, ***aph*** and ***dc*** after data acquisition. Whether to perform data reconstruction processing on NUS data and which algorithm to use for data reconstruction, IST or Deep Learning.

The fifth part is the mail setting (**Send Email**). The first is to check whether to use this function (Use Send Email), and then select “From Email Host”. The software gives the commonly used smtp email host. If you use other hosts, you can directly edit in the “From

Email Host" column, and then select "From Email Protocol". The default is "smtp" (Simple Mail Transfer Protocol), or you can also select "pop3" (Post Office Protocol Version 3). For example, using 163 mailboxes, if "From Email Host" selects smtp.163.com, then "From Email Protocol" selects "smtp"; if "From Email Host" selects pop.163.com, then "From Email Protocol" selects "pop3". The two must match correctly, otherwise, the email cannot be sent normally. Finally, select the data format you want to send (Send Data Format), the default format is SpinStudioJ, or you can drop down and select Data format 1 or Data format 2.

The last part is **User Directory Path**. The administrator can set several directories for a user for data storage. The default root directory for a user is a subdirectory of user name under the root directory already set in **General Options**. Click **Add User Folder** to open a dialog box (Figure 2.69). If Subgroup Folder is checked, there will be an additional data path with sub-group users as subdirectory, that is, the path *D:/Data/admin/Automation/admin/[Group]/* in Figure 2.68. You can assign a name to the **User Folder** for the user, which is under user root directory. If **Date Folder** or **Slot Folder** is selected, a subdirectory with the format of [Date Time] or [Slot Index] under **User Folder** will be created. If both of them are selected, then the **Custom Directory** will be followed by /[Date Time]/ [Slot Index] subdirectory. Select a directory in the box under **User Directory Path**, then click **Remove User Folder** to remove it, or click **Set as Default** to set it as default directory of the user.

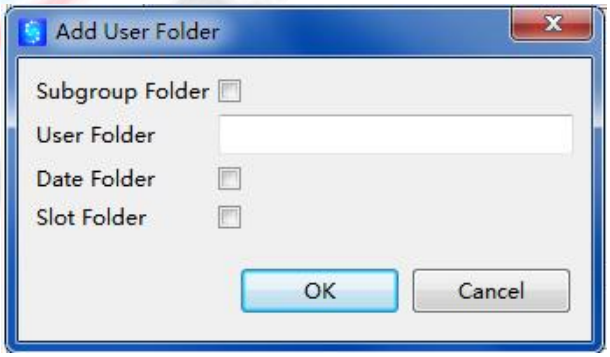


Figure 2.69 Set user directory

2.11.3 Operations of Automation experiments

1. New

When you want to start a new automation experiment process, click New button in tool bar. A window will appear warning that all experiments will be cleared (Figure 2.70). Click **OK** to proceed. There are two methods for creating automation experiments, one by utilizing saved automation experiments list, another by adding new experiments.

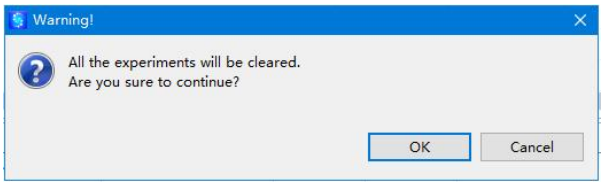


Figure 2.70 New automation experiment prompt box

2. Open

If user want to run the same automation experiments done before, or part of them will be repeated, you can click Open button in tool bar. A window the same as in Figure 2.70 will be popped up to warn you. Click **OK**, and the file manager window will be open. You can find the automation list file saved before, click **Open** to apply it to the current experiments. The list can be further edited.

3. Add

Select a Slot line in the Automation list table window, the click Add button in tool bar or right-click and select **Add Experiment**. When the selected Slot is empty or the experiment under the Slot belongs to the current user, an experiment will be added to the Slot. If there are experiments of other users under the selected Slot, you need to wait for the experiment status to change to Done or Error, and then use the right-click function **Reset** or the Delete button in the toolbar to clear the Slot before adding experiments. The experiment is the default one as defined in "**User Preference**", and can be modified further.

4. Checking and Modification of Parameters

After adding new experiments, user can check them and modify the parameters through

the drop-down list or tick box, as shown in Figure 2.71. You can also give a name to the experiment in the box under **File**.

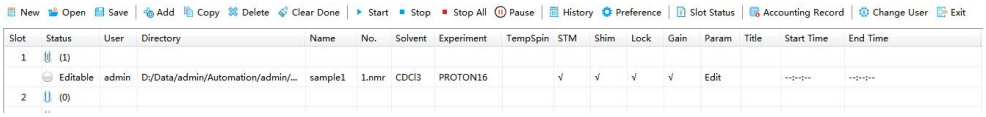


Figure 2.71 Automation experiment parameter modification and editing

Click the **Edit** button under **Param** to open a dialog box, where you can edit parameters for Acquisition (Acqu), Shim, Data Process, Temperature control and rotation parameters (TempSpin) as shown in the left of Figure 2.72.

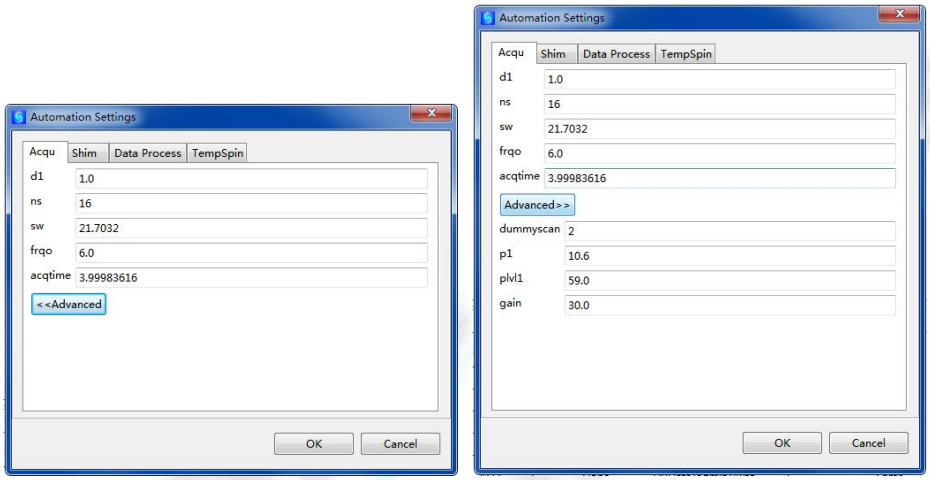


Figure 2.72 Edit parameters for Acquisition

By default the **Acqu** tab is active, showing parameters according to the experimental template. Click the **< < Advanced** button, if the current user's Group is Advanced, all parameters related to the experiment that are not displayed will be opened, and all parameters can be modified, as shown in the right figure of Figure 2.72; if the currently logged in user Group is General, a prompt box will pop up in Figure 2.73 below, and there is no permission to open the Advanced parameter table.

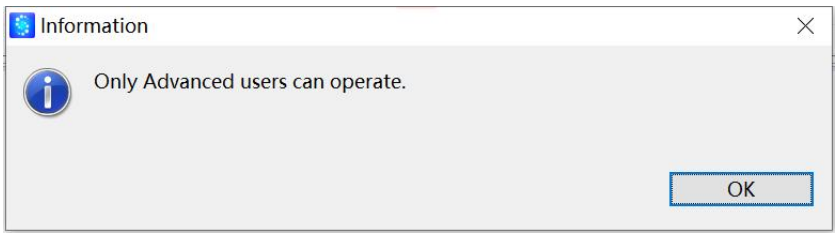


Figure 2.73 Prompt box when viewing Advanced parameters

The options in Shim and Data Process tabs are almost the same as in User Preference, see Figure 2.74.

When checking “Tuning after gradient shimming” in the Shim tab, you can toggle the selection of the channel combination of the shimming coil below and the shimming evaluation method (Evaluate).

If “Optimize z1 for Metabonomics” is checked, the Z1 current value will be added to 21 after the shimming is finished. The default value is 21, which can be configured according to the actual situation using the ***syscf*** command.

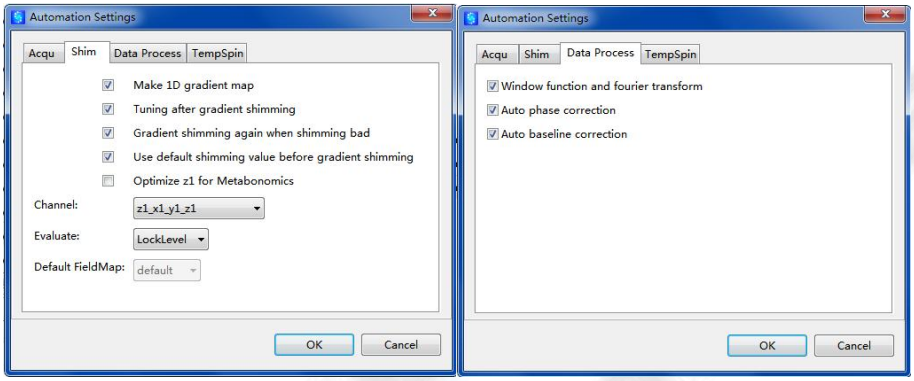


Figure 2.74 Options in Shim and Data Process

When “Make 1D gradient map” is not checked, that is, when using the default field map to smooth the field, you can switch the default field map at the **Default FieldMap** below, as shown in Figure 2.75. Select “default” or “default2”, the default is “default”. The premise of selection is that two default field maps have been stored for the current solvent before. For example, for samples of different heights, adjust the parameters to make two default field maps. In actual use, select the appropriate field map according to the current sample height to achieve the best effect.

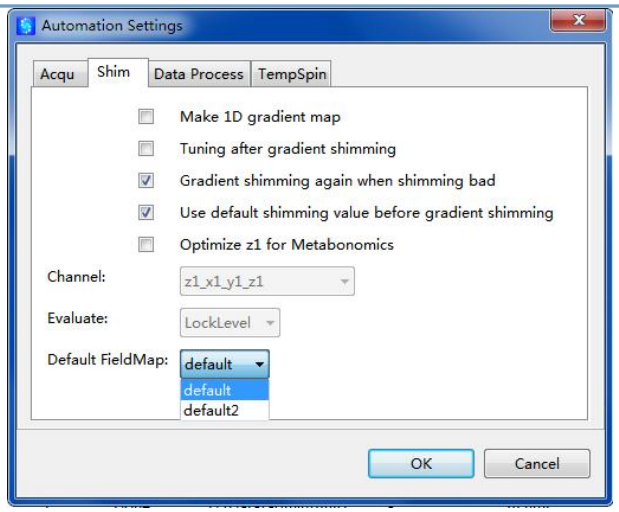


Figure 2.75 Select default field map

If the current experiment template is a non-uniform (NUS) sampling experiment, the data reconstruction related configuration (Figure 2.76) appears in the data processing settings, and the default configuration is the same as the previous settings in **User Preference**.

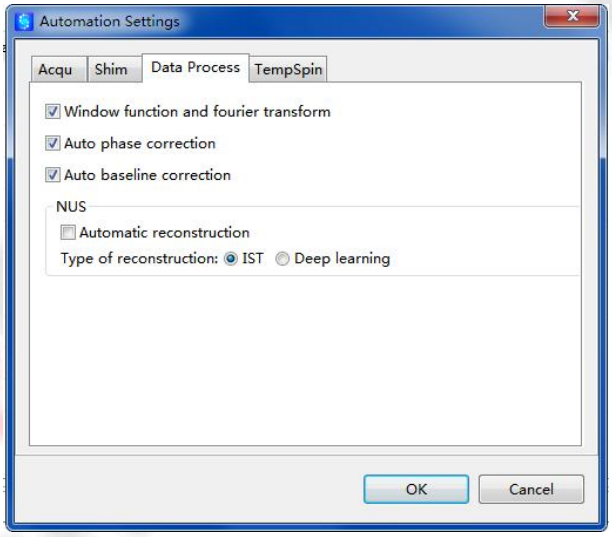


Figure 2.76 Processing parameter configuration for non-uniform sampling

If according to the experimental requirements, the temperature or rotation control of the sample needs to be performed after the injection is completed, as shown in Figure 2.77. You can check “Temperature control” or “Spin control”, and then set the target temperature (unit: ℃) or target speed (unit: Hz) in the **Target value** column below. “Maximum waiting time” is the longest waiting time, the default value is 300s, if the waiting time does not reach the set value, it is considered temperature control or rotation failure, the software will

feedback error information. “Stable time” default value is 40s, that is, after reaching the target temperature or target speed, there will be 40s stable time. Check “Stop temp after experiment” and “Stop spin after experiment” to stop temperature control and rotation after the experiment is complete.

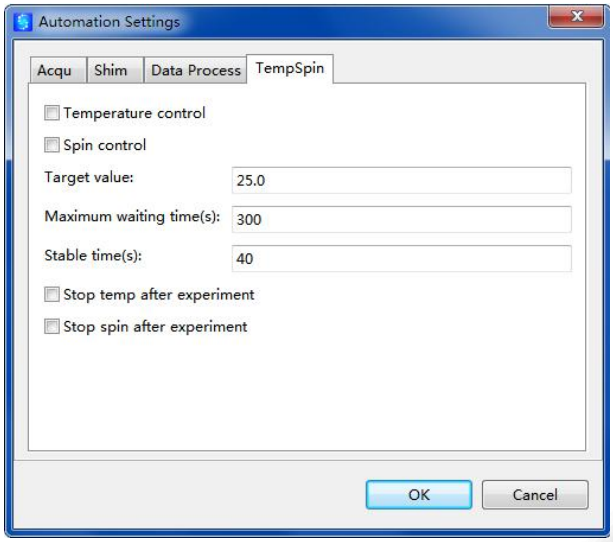


Figure 2.77 Temperature control and rotation configuration

5. Copy

There are two methods to copy experiments to destination slot:

(1) Copy single experiment to designated slot

Select the experiment line to be copied, then click Copy button in tool bar, or right-click and select **Copy**. A dialog box is open to let the user select destination slot. After selecting the wanted slot, click OK button to close the box, as shown in Figure 2.78.

(2) Copy all experiments in a slot to destination slot

Select the slot to be copied, then click Copy button in tool bar, or right-click and select **Copy**. A dialog box is open to let the user select destination slot. After selecting the wanted slot, click OK button to close the box. It can also be selected by the button Select All and the **Select Invert** and **Choose** buttons, as shown in Figure 2.78.

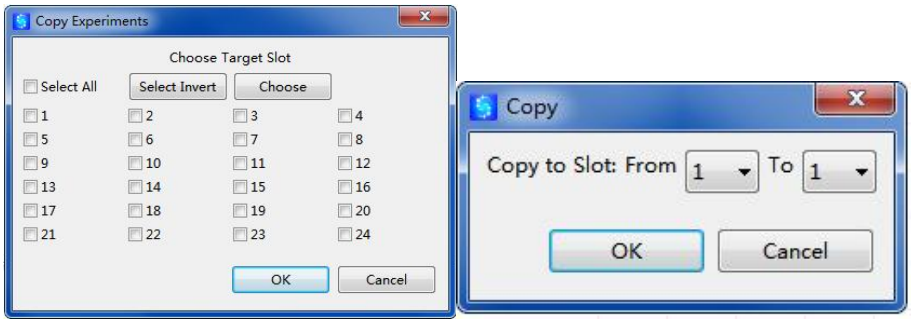


Figure 2.78 Copy dialog

6. Delete

(1) Select the experiment you want to delete, click the toolbar button Delete , or right click and select **Delete**. You can delete experiments with current user experiment statuses of Queued, Submitted, Done, Error, Cancelled and Editable. If the experiment is in progress, it will first become Editable and then be deleted. This operation can only be performed on experiments under the currently logged-in user, except for admin.

(2) Select the Slot line, click the toolbar button Delete , or right-click to select **Reset**, you can delete all the experiments under the Slot whose status is Done or Error. If there are experiments with other status under the Slot, the operation will not be executed. This operation has nothing to do with the logged-in user, only related to the experimental state.

7. Start

After finishing setting and checking of experimental parameters, you can click Start button in the tool bar to start automation experiments. If logging in as admin, this button is to submit all experiments in Editable and Cancelled state. If it is a normal user, this button is to submit all the editable and Cancelled experiments of the current user.

You can also select a single experiment, right-click and select **Submit This Experiment** to submit the currently selected experiment. This operation can only be performed on experiments under the currently logged-in user, except for admin.

After submitting the experiment, the experiment will generally be carried out in the order in which the experiment was submitted. However, if there is an unfinished experiment under a certain slot, then add an experiment under that slot and submit it. The newly

submitted experiment will be carried out immediately after the last experiment under that slot. This can effectively avoid multiple out-and-out of the same sample and repeated STM, SmartShim, Auto Lock, etc., to save time.

8. Stop

(1) Stop any experiment in the queue, and continue to execute other experiments.

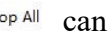
Select any experiment, then right-click and select **Stop**. After stopping the experiment, the state of the experiment will switch to Editable. This operation can only be performed on experiments of the currently logged-in user, except for admin.

(2) Stop all experiments of the current user.

Clicking the button 

can only stop all experiments of the current user, including the submitted and ongoing experiments. This operation authority is no exception for admin.

(3) Stop all users' experiments.

Click the button 

can stop all experiments for all users, including the submitted and ongoing experiments.

(4) Pause any experiment for the current user(Halt)

Select any experiment, and then right-click Halt. Pause an experiment. If the experiment state is Acquire, the state will be switched to Cancelled, and data processing, data conversion and data upload will be carried out according to the software configuration; if it is a state other than Acquire, such as Submitted, shim, etc., the state will be switched to Editable. This operation can only be performed on the experiment of the current user, except admin.

9. Take Priority

(1) Prioritize a single experiment(Take Priority)

This operation gives priority to the experiment in Submitted state. Select the experiment, then right-click to select **Take Priority**. This experiment will be prioritized to the top of all Submitted experiments. The administrator account admin can give priority to all users' experiments, and ordinary users can only give priority to their own experiments.

(2) Prioritize a slot

Select a slot in the experimental queue, right-click and select **Take Priority of slot**. This

slot will be prioritized to the top of all Submitted experiments. The administrator account admin can give priority to all users' slots, and ordinary users can only give priority to their own slots.

(3) Prioritize the composite experiments

If the selected experiment is any one of the composite experiments, a pop-up box will ask if you want to have priority together, select **Yes** to give priority to all, and select **No** to have no priority.

In addition to the above priority rules, all priority operations follow one principle: first priority, first do. At the same time, we should also follow the software configuration. When configured as the default option “Wait until the current sample is completed”, all experiments under the slot where the current Queued state experiment is completed, and then do the priority experiment or slot. When configured to “Do Priority Samples immediately”, it will do priority experiments or slots immediately after the Queued state experiment is completed.

10. Cycle Test

If you want to carry out repeated acquisition for some experiment, select an experiment line, right-click to select **Cycle Test**. This experiment will be repeatedly acquired after the queue is finished. This function is displayed only when logging in with admin, and is generally used when debugging the auto sampler changer.

11. Add Composite Experiment

The combined experiment is used to optimize the spectrum width and frequency offset of the target experiment. The conventional usage is to acquire a hydrogen spectrum before acquiring the target experiment, and use the script program to set the optimized spectrum width and frequency offset to the target experiment.

Take the experiment COSYGPQF as an example. After filling in the experiment **Name**, left click to select the experiment, and then right click to select **Add Composite Experiment**. At this time, you can set the parameters in the pop-up dialog box, as shown in Figure 2.79. The script execution time can only use the default option Acquire, and the experiment can use the default PROTON1. According to the target experiment COSYGPQF, choose to optimize the F1 and F2 dimensions at the same time, check Use this script, and click **OK**. Then a dialog box for setting script sequence will pop up, as shown in Figure 2.80. Currently the software only supports this script for optimizing parameters, so just click **OK** to confirm. At this time, an experiment with the same Name will be generated in front of the target experiment, as shown in Figure 2.81. After submitting the experiment by clicking the start button in the toolbar, the relevant experiment will be completed in order.

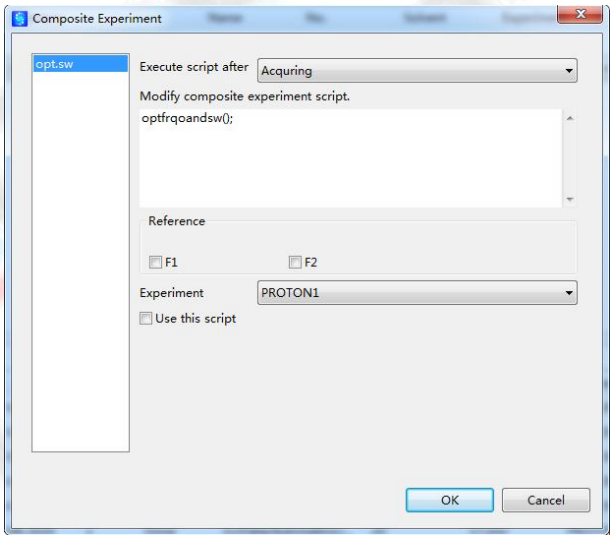


Figure 2.79 Dialog for composite experiment

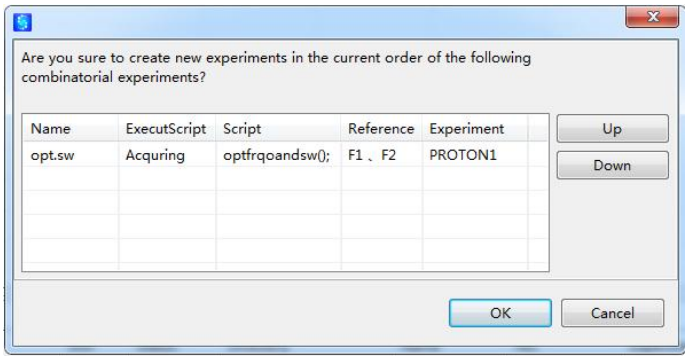


Figure 2.80 Dialog to set script sequence

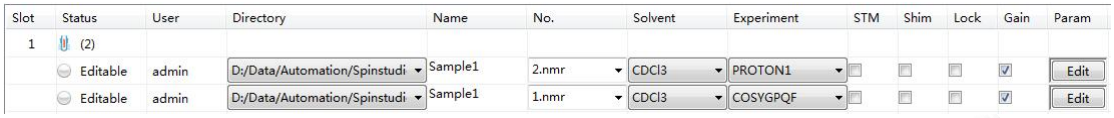


Figure 2.81 Composite experiment

12. Add/Show/Delete Script

The software only supports adding temperature control script programs currently. After selecting the target experiment, right-click and select **Add Script**, and the Add Script dialog box will pop up, as shown in Figure 2.82. You can drop down to select the script execution time. There are five options: Tune, Shim, Lock, Gain and Acquire, the default is after Acquire, modify the temperature value in the Script box and click **OK**. After submitting the experiment, the temperature control will start after the target experiment is acquired.

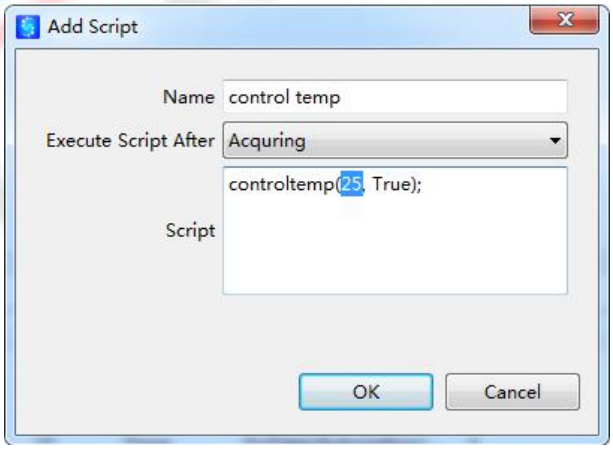


Figure 2.82 Add script dialog

If you want to check whether a script is added to an experiment, or to view the content

of the script, you can select the target experiment, then right-click and select **Show Script**, and the script display box will pop up, as shown in Figure 2.83. If no temperature control script has been added to the target experiment, a prompt box will pop up as shown in Figure 2.84.

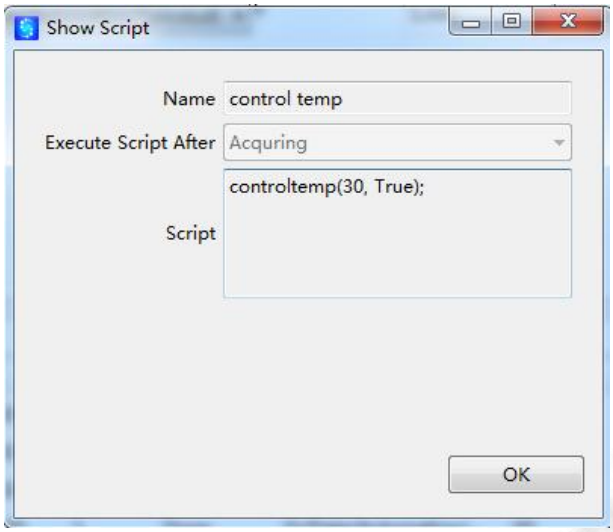


Figure 2.83 Show script dialog

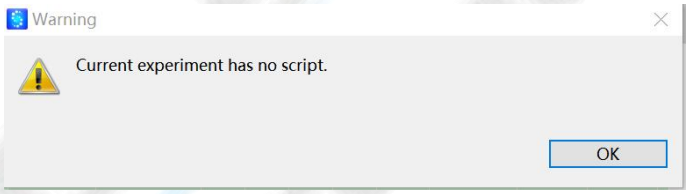


Figure 2.84 Dialog when the experiment has no script

To delete the temperature control script, you can select the target experiment, right-click and select **Delete Script**, select **OK** in the pop-up dialog box, as shown in Figure 2.85.

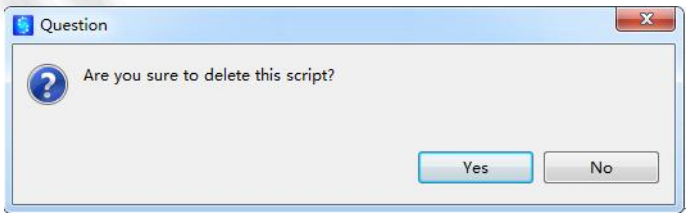


Figure 2.85 Delete script dialog

13. Open Workspace

Select an experiment line, right-click to select **Open Workspace**. A window will be opened in Workspace.

14. Save

Clicking Save button in tools bar will save the information of the current queue list to a file, which can be retrieved later by clicking Open button.

15. History Log of Automation

The history log record the all the information about completed automation experiments and the error messages. Double clicking the log item will open a window corresponding to the experiment in the workspace.

2.11.4 Web Client

The automation user can log into the Automation interface through Web client, using the same user name and passwords as for the SpinStudioJ Automation Experiment. User can add new experiments, edit, delete, copy, submit and stop experiments. These operations will be synchronized to the SpinStudioJ software.

The URL of Automation Experiment Web Client is: http://localhost:8081/. It is recommended to use the 360 browser and use the speed mode (compatibility mode is not available), localhost represents the workstation's IP address, which is the Web Server IP Address configured in the automated lab preferences.

The login page is shown in Figure 2.86.

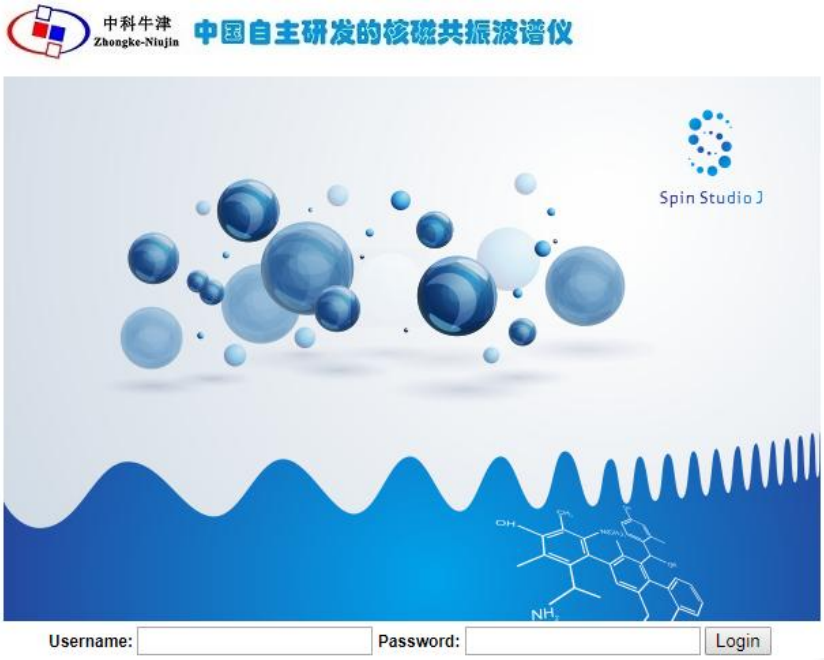


Figure 2.86 Login interface

The username and password are the username and password set for the SpinStudioJ automation lab.

When the web interface exceeds the Web Server timeout set by the automation experiment preferences, the interface information is no longer updated, and the dialog box prompts the login timeout. After clicking the confirmation button, it will return to the timeout login interface (Figure 2.87).

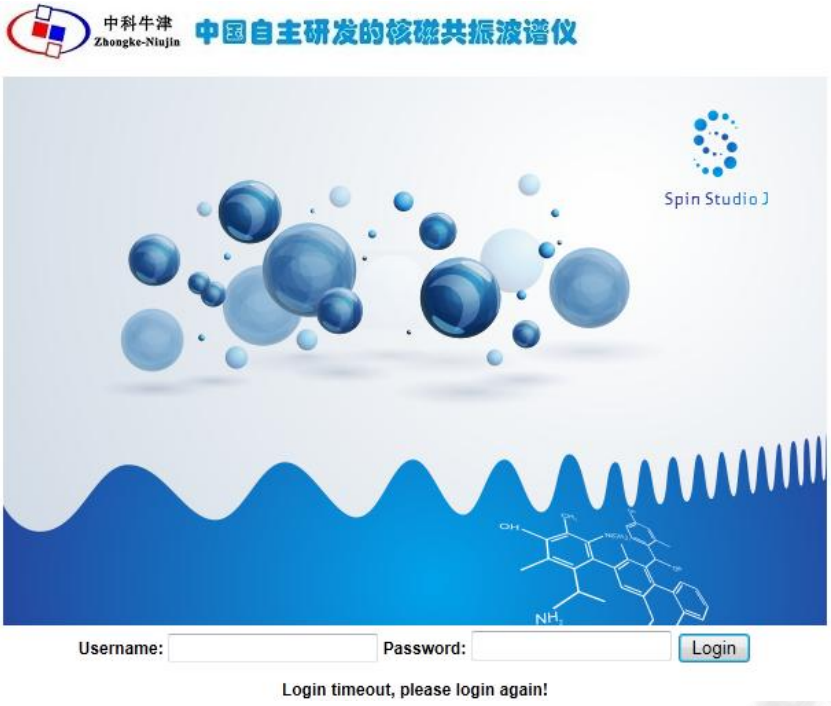


Figure 2.87 Timeout login interface

The interface of Web client is shown in Figure 2.88.

Figure 2.88 The interface of Web client

New Experiment: click button in the tools bar or button at the end of slot line under **Operation** column. A window will be opened as Figure 2.89.

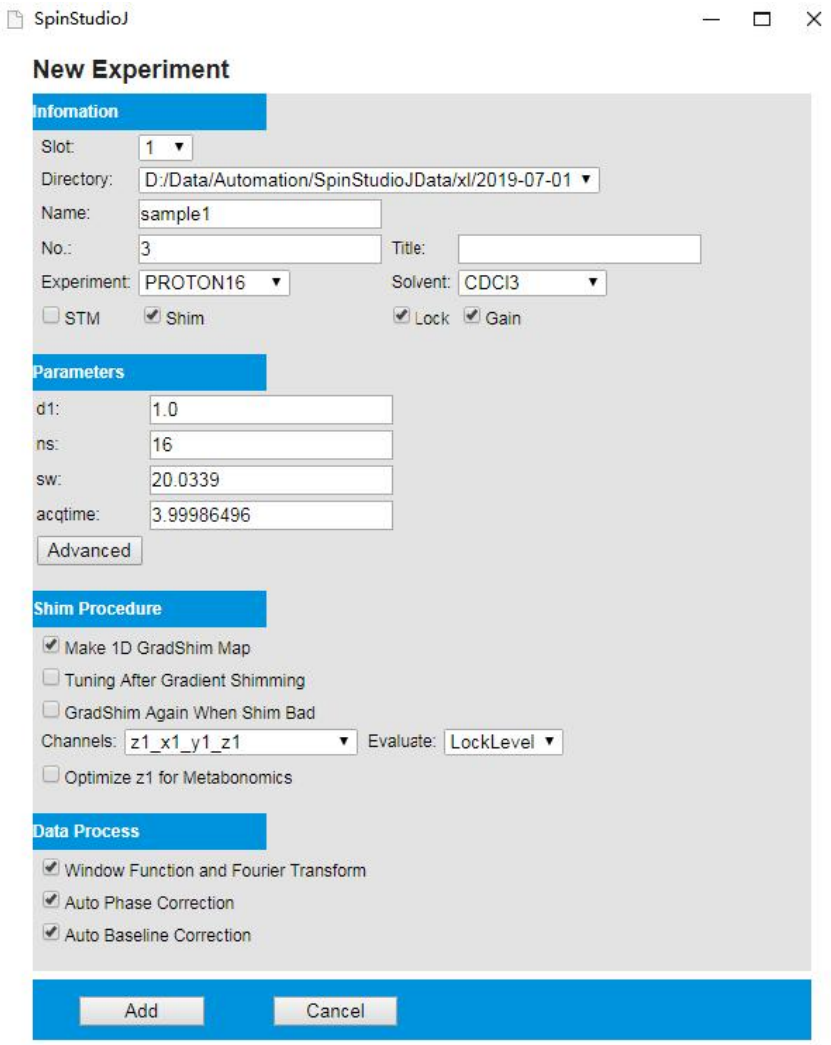


Figure 2.89 New experiment interface

The parameters in this window are the same as in the SpinStudioJ. After editing the parameters, click **Add** button to add this experiment to the list.

Start: click button in the tools bar to start all the experiments in the list. Clicking button at the end of experiment line under **Operation** column will start the current experiment. All users can only submit their own experiments, with the exception of administrators.

Stop: click button in the tools bar to stop all the experiments in the list. Clicking button at the end of experiment line under **Operation** column will sop the current experiment.

Delete: clicking button at the end of experiment line under **Operation** column will

delete the current experiment.

Edit: for those experiment in editable **Status**, you can clicking button at the end of experiment line under **Operation** column to edit it. This can only be done for the experiment of the currently logged in user, and the administrator is no exception.

Copy: clicking button at the end of slot line under **Operation** column will copy the experiment information of current slot to the next slot.

Reset: You can clear all experiments under the current Slot by clicking the corresponding button under Operation in the Slot line. After the reset, other users can use the Slot to add experiments again.

History log: the history of automation experiments will be displayed in the lower part of the Web client, as shown in Figure 2.90.

History

Start Time	End Time	Slot	Status	Directory	Name	No.	Solvent	Experiment	STM	Shim	Lock	Gain	Title	Remark	User
2019-07-05 14:45:39	2019-07-05 14:45:45	2	Done	D:/Data/Automation/SpinStudioJData/xl/2019-07-05	b	4.nmr	CDCl3	PROTON1	☐	☐	☐	☐		successful	xl
2019-07-05 14:45:32	2019-07-05 14:45:38	2	Done	D:/Data/Automation/SpinStudioJData/xl/2019-07-05	b	3.nmr	CDCl3	PROTON1	☐	☐	☐	☐		successful	xl
2019-07-05 14:45:25	2019-07-05 14:45:31	2	Done	D:/Data/Automation/SpinStudioJData/xl/2019-07-05	b	2.nmr	CDCl3	PROTON1	☐	☐	☐	☐		successful	xl
2019-07-05 14:45:19	2019-07-05 14:45:24	2	Done	D:/Data/Automation/SpinStudioJData/xl/2019-07-05	b	1.nmr	CDCl3	PROTON1	☐	☐	☐	☐		successful	xl
2019-07-05 14:44:11	2019-07-05 14:44:17	1	Done	D:/Data/Automation/SpinStudioJData/xl/2019-07-05	a	2.nmr	CDCl3	PROTON1	☐	☐	☐	☐		successful	xl

Figure 2.90 History of automation experiments

Chapter 3 Pulse Programming

The pulse sequence in SpinStudioJ is written in Extensible Markup Language (XML). You can use text editor such as Notepad++ to create pulse sequence in XML, then save it with ps as extension. The XML text of a pulse sequence is shown in Figure 3.1, which corresponds to the pulse sequence diagram in Figure 3.2.

Figure 3.1 pulse sequence text

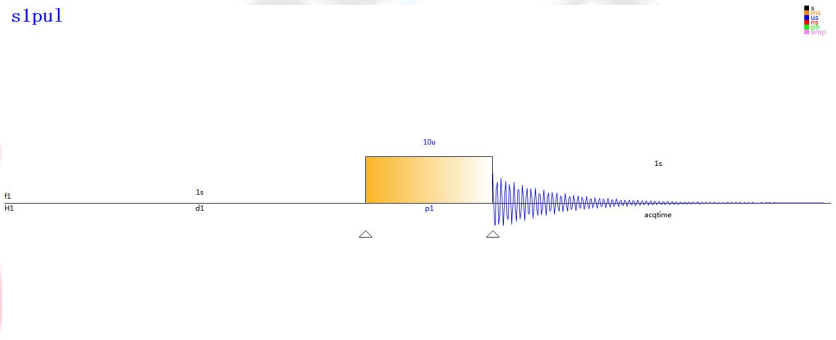


Figure 3.2 Pulse sequence diagram

3.1 Grammar of Pulse Sequence Editing

3.1.1 Modules of Pulse Sequence

A pulse sequence consists of the following parts (see Figure 3.1):

1. Basic information. The content of **name** is the name of the pulse sequence, while the **dimension** represent the dimension of experiment, 1 for 1D and 2 for 2D.

2. Description of the pulse sequence. This includes English and Chinese descriptions.
3. Pulse sequence elements. The pulse sequence is based on the time slice structure. All elements are embedded in time slices. <Slice> is the start symbol of a time slice, and </Slice> is the end symbol. Each time slice may consists of one or several elements. The pulse sequence shown in Figure 3.1 consists of three time slices, each of which contains Delay, Pulse and Acquire element respectively.

There are totally 11 elements defined in pulse sequence, some of which are related with RF channels, while others are not. There are 6 values for channel property in pulse sequence, i.e., observe channel **f1**, decoupling channel **f2**, **f3** and **f4**, gradient channel **gz** and shim channel **shim**.

Eight channel-related elements are: **Pulse**, **Delay**, **Acquire**, **Gradient**, **ShpaeGradient**, **ShapePulse**, **CompositePulse** and **Shim**; three elements not related to channel are: **Loop**, **If** and **Stage**.

In a time slice, there are three ways of alignment for elements: **left**, **middle** and **right**.

4. Phase table. All phase cycle used in the pulse sequence should be listed here.
5. Parameters definition. All parameters used in the pulse sequence must be defined here one by one. The first part is in English and the second part is in Chinese. Each parameter consists of several properties:

   id: name of the parameter.

   description: text describing the meaning of the parameter.

   value: parameter value.

   allowarray: determine whether array is allowed. The value can be "true" or "false".

   type: type of parameter: real, integer, long, string.

   digits: define the precision of the parameter, expressed in number of total digits.

   unit: the unit of parameter: "M", "K", "m", "μ", " ". The last one means standard international units.

   min: the minimum value.

   max: the maximum value.

   force: whether to use the value given here as the parameter value.

   probeparam: retrieve pulse width or pulse power when reading prove file. Only used

for pulse and power parameters.

3.1.2 Grammar for channel-related elements

1. Delay

The example of delay element is shown as

```
<Slice>
  <Delay channel="f1" align="middle" duration="d1" />
</Slice>
```

delay: element symbol

channel: "f1", "f2", "f3", "f4", "g"z or "shim"

align: "left", "middle", "right"

duration: time of delay. Reference values are d0~d20 in second.

2. Pulse

The example of pulse element is shown as

```
<Slice>
  <Pulse channel="f1" align="middle" duration="p1" power="plvl1" phase="ph1" offset="fq" />
</Slice>
```

Pulse: element symbol

channel: "f1", "f2", "f3", "f4"

align: "left", "middle", "right"

duration: length of pulse. Reference values are p0~p20 in microsecond, ms.

power: RF power of the pulse. Reference values are plvl0~plvl20 in dB.

phase: the phase of the pulse. Reference values are ph1~ph10.

offset: the offset of RF transmitter. Reference values are fq~fq10 in Hz.

3. Acquire

The example of Acquire element is shown as

```
<Slice>
  <Acquire channel="f1" align="middle" duration="acqtime" phase="ph1" points="np" />
</Slice>
```

Acquire: element symbol

channel: "f1"

align: "left", "middle", "right"

duration: length of acquisition time. Reference values are acqtime~acqtime3 in second.

phase: the phase of the pulse. Reference values are ph1~ph10.

points: acquisition points. Reference values are np~np3.

4. Gradient

The example of Gradient element is shown as

```
<Slice>
  <Gradient channel="gz" align="middle" duration="gtime" amplitude="gzlevel" />
</Slice>
```

Gradient: element symbol

channel: "gz"

align: "left", "middle", "right"

duration: length of gradient pulse. Reference values are gtime~gtime9 in microsecond.

amplitude: amplitude of gradient pulse. Reference values are gzlevel~gzlevel9, in percentage value of the maximum gradient (G).

5. ShapeGradient

The example of ShapeGradient element is shown as

```
<Slice>
  <ShapeGradient channel="gz" align="middle" duration="gtime" amplitude="(1-2*parseInt(ci%2))*2*gzlevel" shape="gshape" />
</Slice>
```

ShapeGradient: element symbol

channel: "gz"

align: "left", "middle", "right"

duration: length of gradient pulse. Reference values are gtime~gtime9 in microsecond.

amplitude: amplitude of gradient pulse. Reference values are gzlevel~gzlevel9, in percentage value of the maximum gradient (G).

shape: shape of the gradient. Reference values are gshape~gshape9.

6. ShapePulse

The example of ShapePulse element is shown as

```
<Slice>
  <ShapePulse channel="f1" align="middle" duration="sp1" power="splvl1" phase="ph1" offset="fq1" shape="pulseshape1" />
</Slice>
```

ShapePulse: element symbol

channel: "f1", "f2", "f3", "f4"

align: "left", "middle", "right"

duration: length of pulse. Reference values are sp~sp20 in microsecond, ms.

power: RF power of the pulse. Reference values are plvl0~plvl20 in dB.

phase: the phase of the pulse. Reference values are ph1~ph10.

offset: the offset of RF transmitter. Reference values are fq~fq10 in Hz.

shape: shape of the pulse. Reference values are pulseshape~pulseshape9.

7. CompositPulse

The example of CompositPulse element is shown as

```
<Slice>
  <CompositePulse channel="f1" align="middle" duration="p1" power="plvl1" phase="ph1" offset="fq1" shape="cpdprg" />
</Slice>
```

CompositPulse: element symbol

channel: "f1", "f2", "f3", "f4"

align: "left", "middle", "right"

duration: length of pulse. Reference values are sp~sp20 in microsecond, ms.

power: RF power of the pulse. Reference values are plvl0~plvl20 in dB.

phase: the phase of the pulse. Reference values are ph1~ph10.

offset: the offset of RF transmitter. Reference values are fq~fq10 in Hz.

shape: shape of the pulse. Reference values are pulseshape~pulseshape9. Refer to the files in *~/system/data/shapelib* directory.

8. Shim

The example of CompositPulse element is shown as

```
<Slice>
  <Shim channel="shim" align="middle" duration="st" shimchannel="recchann[i]" shimvalue="recvalue[i]" />
</Slice>
```

Shim: element symbol

channel: "shim"

align: "left", "middle", "right"

duration: lasting time for shimming

In the pulse sequence text, the phase used by the pulse sequence element must give detailed information in the phase list, and the parameters used must also give detailed information in the pulse sequence parameter list.

If the parameter used in an element is numeric type, it can be directly given a numeric value. For example, a delay of 0.2s can be assigned directly, but double quotes must be used.

```
<Slice>
  <Delay channel="f1" align="middle" duration="0.2" />
</Slice>
```

The value of a property in an element can not only equal to a parameter name or numeric value, but also to an expression. For example, a gradient element can be expressed as (1-2*parseInt(ci/2))*0.8*gzlevel, Where parseInt(ci%2) indicates that the remainder of ci divided by 2 is rounded, and gzlevel represents the gradient field strength value of the Z direction set by the current experiment. Note that the parameters used in expression must have been defined in the parameter definition of the pulse sequence.

```
<Slice>
  <ShapeGradient channel="gz" align="middle" duration="gtime" amplitude="(1-2*parseInt(ci%2))*2*gzlevel" shape="gshape" />
</Slice>
```

Only hard gradients and shape gradients can exist in the gradient channel, and other elements cannot exist in the gradient channel; hard gradients and shape gradients cannot exist in the observation channel and the decoupling channel.

3.1.3 Grammar for elements not related to channels

Three elements are not related to channels: condition element **If**, loop element **Loop** and decoupling element **Stage**. These elements are not within time slice, but before or after time

slices. They separate time slices.

1. If

The condition element consists of start and end parts. The start part contains condition information, whereas the end part is just an end symbol.

(1) Start of the condition element

Example:

```
<If condition="0<mult">
```

If: start symbol

condition: the content of condition

(2) End of the condition element is just an end symbol

```
</If>
```

2. Loop

The loop element consists of start and end parts. The start part contains loop counting information, whereas the end element is just an end symbol.

(1) Start of Loop

Example:

```
<Loop count="nx+ds" index="i">
```

Loop: start symbol

count: number of loops, can be a parameter or an expression.

index: index of loop, starting from 0.

(2) End of the loop element is just an end symbol

```
</Loop>
```

3. **Stage**

When decoupling channel exists in pulse sequence, the decoupling information can be included in the pulse sequence. This information is expressed by using **Stage** to separate

pulse sequence into several time periods. The decoupling status for each period is controlled by the setting of the parameter decon, which consists of a string of letters whose number equals to the total number of periods. The value for each letter can only be "n" or "y", corresponding to decoupling off or on. If you want to separate the pulse sequence in some place, simply insert a line with content of **<Stage />**. This is represented as a triangle in pulse sequence diagram, at the bottom of the position for separation, as shown in Figure 3.3. If ***decon*** = ***"nny"***, the decoupling will be turned on during acquisition, and a light-blue square will be displayed at the decoupling channel just under acquisition time in the diagram (Figure 3.3).

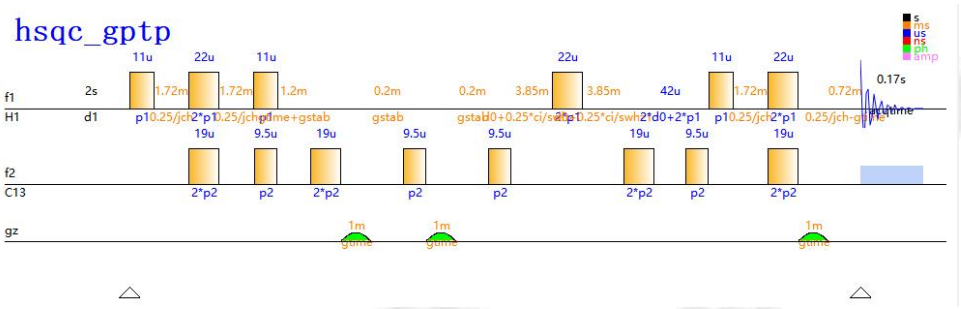


Figure 3.3 Pulse sequence with decoupling separator

3.2 Display and Editing of Pulse Sequence

3.2.1 Pulse sequence display

For any experiment displayed in workspace, click the **Sequence** tab, then click **Diagram** or **Source** tab to display the diagram or the source code of the pulse sequence. Figure 3.4 is the diagram of a pulse sequence.

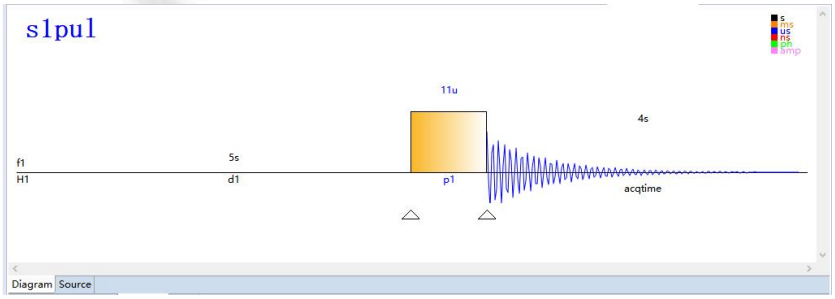


Figure 3.4 Pulse sequence graphic display

3.2.2 Change Pulse Sequence

Open the workspace, switch the tab of the workspace to the **Parameters - Acquparams** page, click the button to the right of pulseseq, as shown in the red circle in Figure 3.5, a pulse sequence selection dialog will pop up, as shown in Figure 3.6. The pulse sequence displayed in the System group in this dialog box is in *~/system/data/pslib* in the directory where the software is located. The pulse sequence displayed in the User group is in *~/system/data/pslib/user* in the directory where the software is located. Select a pulse sequence and click the **OK** button. At this point, switch the tab of the workspace to the **Sequence** page. The **Sequence** page will display the selected pulse sequence.

Figure 3.5 Display file list

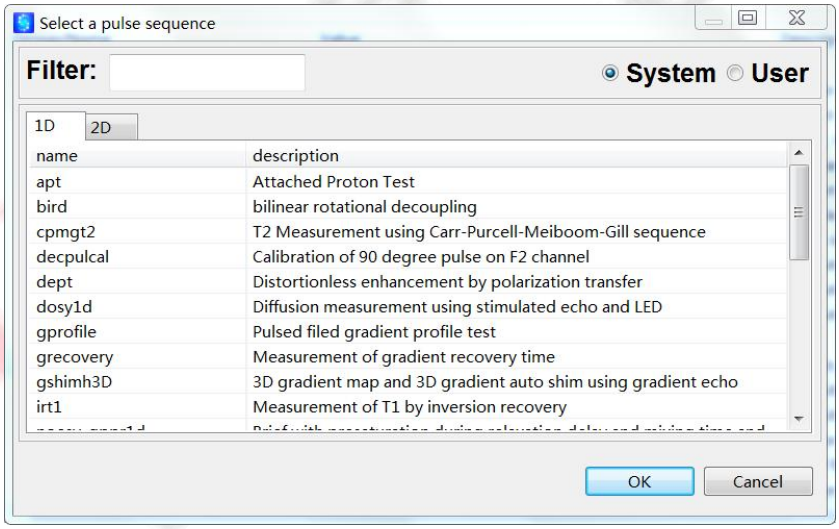


Figure 3.6 Pulse sequence selection dialog

3.2.3 Editing Pulse Sequence

1. Create New Pulse Sequence

There are two ways to create a new pulse sequence:

(1) Create the text file of the new pulse sequence using an editor such as Notepad++,

then save it with an extension of .ps to the directory *~/system/data/ pslib*.

(2) Using PS Editor to create the new pulse sequence. Refer to 3.4.1 Create New Pulse Sequence for details.

2. Edit the pulse sequence text

Three ways can be used for pulse sequence editing.

(1) Modify the text directly. The pulse sequence text saved in the ~/system/data/pslib directory is opened in a text editing software such as Notepad++, and can be saved after modification. This method will modify the pulse sequence in the configuration file.

(2) Using PS Editor to edit the pulse sequence. Refer to 3.4.2 and 3.4.3 for details. This method will modify the pulse sequence in the configuration file.

(3) Edit the pulse sequence in the workspace. To start the software and open a workspace, switch the tab of the workspace to Sequence, which has two subpages: Diagram and Source. The Diagram page is shown in Figure 3.7. This method only modifies the pulse sequence in the workspace.

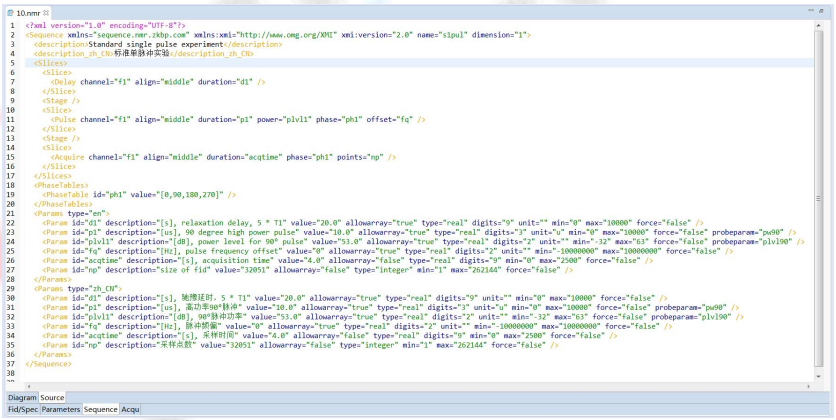


Figure 3.7 Pulse Sequence Source Page

3.3 Synchronization of pulse sequence and experimental parameters

1. When opening a workspace, changing pulse sequence or after editing a pulse sequence, the program will try to retrieve the property of each parameter. For a parameter

with the property of **force="true"**, the value in the parameter list of pulse sequence will be given to this parameter; if **force="false"**, the value of the parameter will not be changed.

2. The values displayed in the pulse diagram are the true values of parameters. When the values are changed in command line or in **Parameters** tab, the diagram will be refreshed in real time. There are two ways to modify the experimental parameters: (1) edit in the parameter panel Parameters; (2) modify the parameters by command, for example, enter ***d1=3*** in the command line and press Enter to take effect.

3.4 PS Editor

3.4.1 Create New Pulse Sequence

Click the menu bar **Tools** > **PS Editor** > **New PS** to open the New Pulse Sequence dialog box, as shown in Figure 3.8.

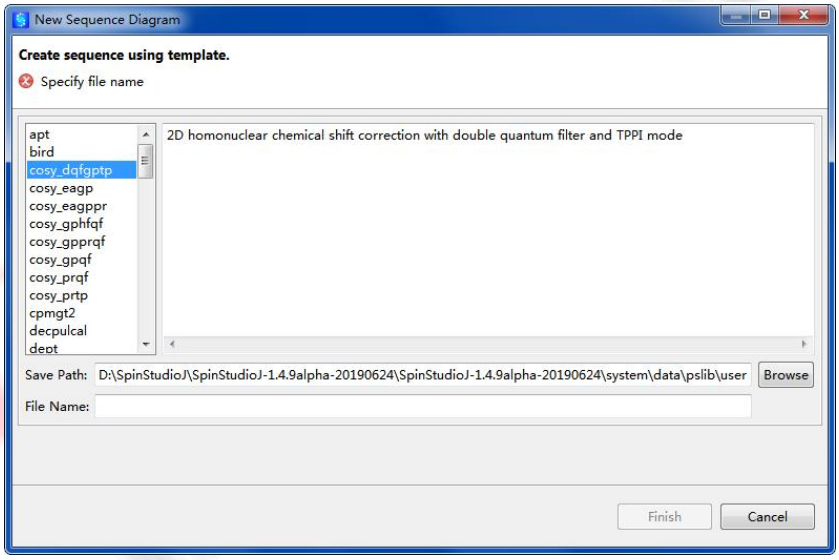


Figure 3.8 New Pulse Sequence dialog box

In the left frame, select a sequence in the sequence library as the template, and the right side is the description of the currently selected sequence. The **Save Path** below is the newly edited sequence storage path. You can also click the button **Browse** to select another path. **File Name** is the newly edited sequence name. Click **Finish** when you are finished and the pulse sequence editor will open. The selected template pulse sequence is loaded in the editor.

The user can edit the sequence, that is, change it to save. For the specific editing method, see 3.4.3 Edit Pulse Sequence.

3.4.2 Opening an existing pulse sequence

Click on the menu bar **Tools** > **PS Editor** > **Open PS** to open the pulse sequence selection dialog box as shown in Figure 3.9. Select the pulse sequence file to be modified and open it. The pulse sequence editor loads the selected pulse sequence. The user can edit the sequence. For details, see 3.4.3 Edit Pulse Sequence.

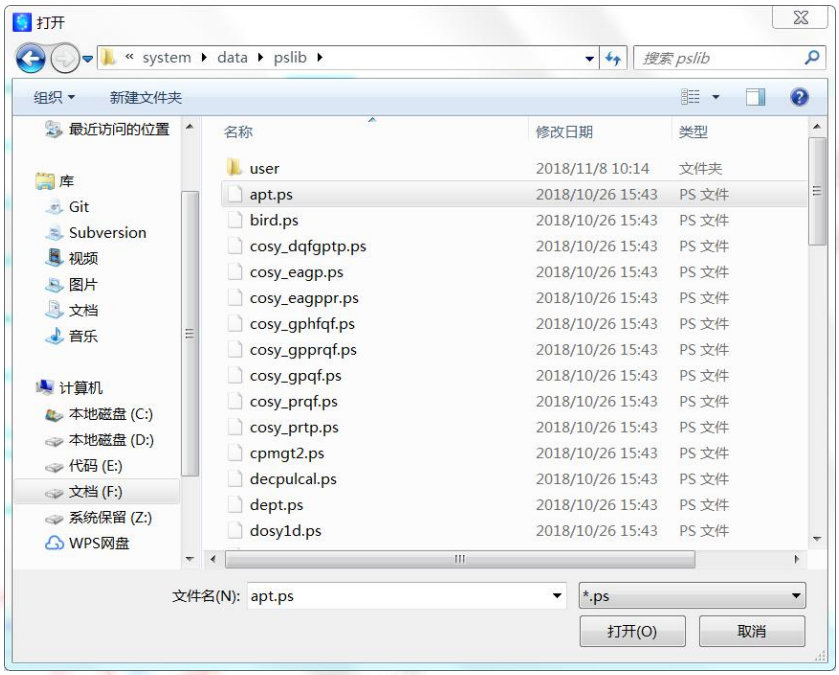


Figure 3.9 Pulse sequence selection dialog box

3.4.3 Edit Pulse Sequence

There are two subpages in the Pulse Sequence Editor: **Diagram** and **Source**. The Diagram page is shown in Figure 3.10.

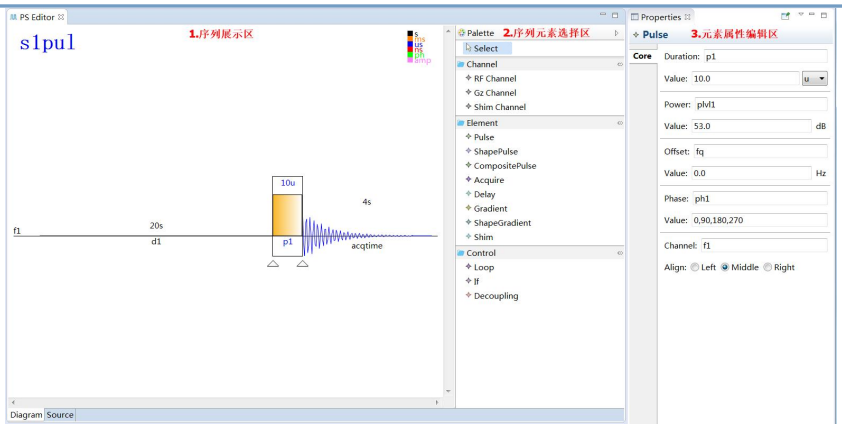


Figure 3.10 Diagram page

In the Diagram page, users can add elements to the sequence by dragging, and can easily edit the element attributes, as follows.

Add new element: in the right area where all elements are listed, click the element you want to add, then move the mouse cursor to the position in sequence diagram where you want to put it, click again. The new element will be added at this position.

Move an element: in sequence diagram, click on the element you want to move, then drag it to the new position and release the mouse button. The selected element will be moved to the new position.

Edit an element: Double-click on any element, or right-click on the element to display the menu shown in Figure 3.11. Select Properties to open the element property editing area (see Figure 3.10). After editing the element properties in the property editing area, it will be valid in real time and saved in real time.

Right click in the sequence display area will also pop up the right-click menu as shown in Figure 3.11.

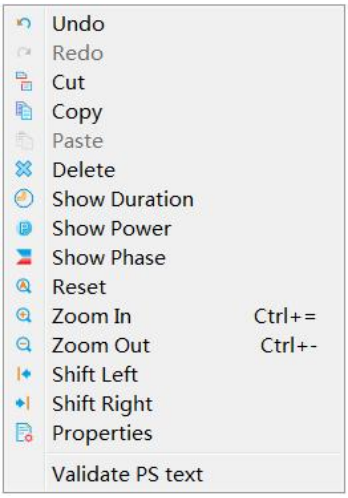


Figure 3.11 Pulse sequence editing related right-click menu

Undo, Redo, Cut, Copy, Paste: editing operations on elements

Show Duration, Show Power, Show Phase: display time, power or phase

Reset, Zoom In, Zoom Out, Shift Left, Shift Right: operation on the diagram

Properties: open element property tab

Validate PS text: check the grammar of the sequence text

The Source tab is shown in Figure 3.12. User can directly edit the source text of the pulse sequence according to the grammar described in 3.1 Grammar of Pulse Sequence Editing. Type **Ctrl** + **S**, or right-click the mouse button then select **Save** to save the changes. You can also right-click and select **Content Format** to format the text, or select **Validate PS text** to check the grammar of the text.

Figure 3.12 Pulse Sequence Editor Source Page

Chapter 4 1D Data Processing

4.1 Workspace Introduction

Workspace has 4 label pages in total, they are respectively “FID/Spectrum”, “Parameters”, “Sequence” and “Acqu”, as shown in Figure 4.1.

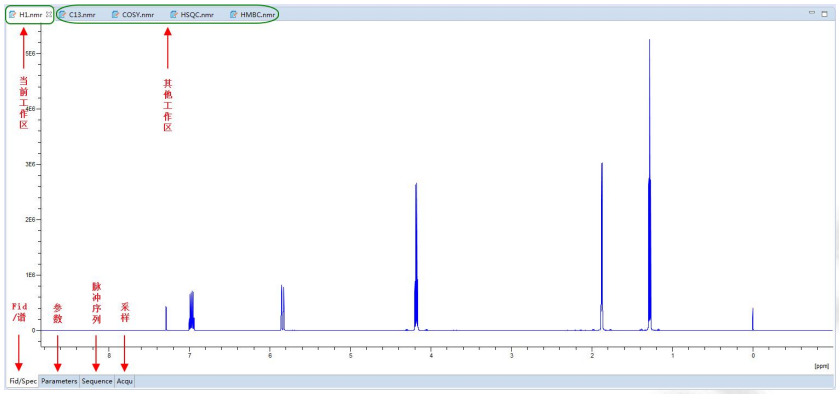


Figure 4.1 Workspace interface

The “FID/Spectrum” page displays the acquired FID data and the Fourier transformed spectral data, which are switched by the right-click menu. There are 2 sub-pages in the “Parameter” page: the acquiring parameter page displays the parameters (including the pulse sequence parameters) that are called when acquiring, and the processing parameter page displays the parameters that are called when processing the data. The “Sequence” page displays the text and graphics of the pulse sequence, which can be switched by clicking the Graphics and Source tabs at the bottom left of the page. The “Acqu” page records real-time acquiring information.

Right-click on FID interface to pop up FID and display the menu as shown in Figure 4.2. You can select to display real and imaginary part of the FID, or display real and imaginary part of the spectrum. You can also open the FID property page.

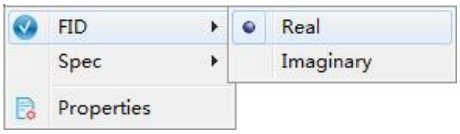


Figure 4.2 FID page right-click menu function

The FID property page is divided into two sub-pages: Appearance and Components. The appearance page is shown in Figure 4.3, and its properties are listed as follows:

X Unit: Horizontal axis unit, optional values are s, ms, μs, pt

Foreground Color: Foreground color, ie FID curve color

Background Color

Show Grid

Grid Color

Show Border

Border Color

Border Line Width

Line Width: FID curve line width

Anti-aliasing

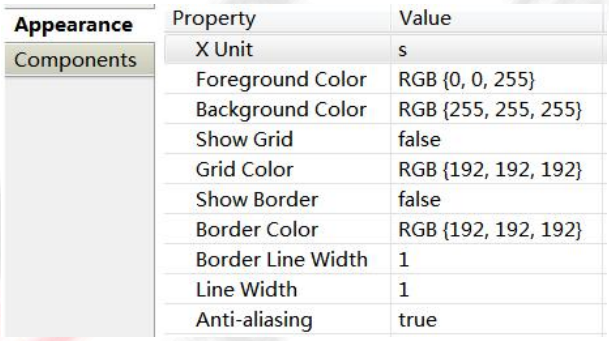


Figure 4.3 FID property page Appearance

The component page is shown in Figure 4.4, and its properties are described as follows:

Title: Whether to display the title. The title can be edited through the right-click menu of the navigation bar and displayed in the upper left corner of the FID in the workspace.

Show CursorInfo: Whether to display mouse information. The mouse information shows the FID information of the current mouse point, which is displayed in the upper left corner of the FID in the workspace.

Left Axis: Whether to display the left axis.

Right Axis: Whether to display the right axis.

Top Axis: Whether to display the top axis.

Bottom Axis: Whether to display the bottom axis.

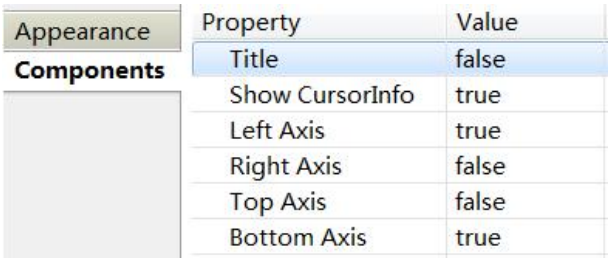


Figure 4.4 Components of the FID property page

The Appearance page option of the Spectrum property page is the same as the FID Appearance page, except that the horizontal axis unit selectable values are ppm, Hz, KHz, MHz, pt.

The component page of the spectrum is different from the FID, and there are three more items, as shown in Figure 4.5. The properties of these three components are described below.

Integral Lines: Whether to display the integral lines.

Integral Label: Whether to display the integral label.

Peaks: Whether to display the peak information.

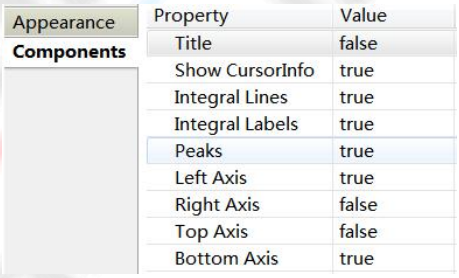


Figure 4.5 Components of the spectrum property page

4.2 Spectrum Operation

Here are some general spectral operations. The operation of the spectrum can be done by selecting the corresponding icon on the toolbar. Both the FID and the spectrum after the Fourier transform can perform the following spectrum operations.

4.2.1 Spectrum zoom

Zoom in or zoom out the spectrum vertically. Roll the mouse wheel up and down on the spectrum and the spectrum will be zoomed in or zoom out in the vertical direction. Or use the toolbar icon and , you can also zoom in the vertical direction of the spectrum.

Zoom in or zoom out the spectrum horizontally. Drag the mouse over the spectrum to achieve horizontal scaling. The specific operation is as follows: select the left starting point on the spectrum, click the left mouse button to drag to the right to release the left mouse button, and the spectrum within the dragging range will be enlarged horizontally. Drag the mouse from right to left and the spectrum will return to full spectrum in the horizontal direction. Or using the icon will restore the full spectrum display both horizontally and vertically.

Perform Fourier transform related operations after the spectrum is zoomed vertically or horizontally, it will maintain the same display range and amplitude as before the transformation.

4.2.2 Move the spectrum

Move the spectrum in the horizontal direction. The display region can not display the entire spectrum after the spectrum is zoomed in, at this moment, you need to move the spectrum horizontally to see the details of each part of the spectrum. Place the mouse in the area of the horizontal coordinate. At this point, the mouse will become a graphic with a small double arrow above the arrow. Hold down the left mouse button and drag it left or right to move the spectrum horizontally. Or click on the toolbar icon and to move the spectrum left and right, click to move a small step; click the icon and move to the leftmost or rightmost end of the spectrum at once.

Place the mouse in the ordinate area, and the mouse will change to the top of the arrow with a small vertical double-headed arrow. Hold down the left mouse button and drag it up and down to move the spectrum vertically. Click the toolbar icon and you can also move the spectrum up and down in the vertical direction, click to move a small step; click the icon to center the spectrum baseline, and click to move the spectrum baseline to the

bottom.

4.2.3 Illustration

Open the one-dimensional spectrum data, click the toolbar icon to enter the illustration mode, and the left mouse button selects the area that needs to be enlarged. Release the left mouse button, and the partial magnification spectrum is displayed in the upper left corner of the spectrum, as shown in Figure 4.6. In this mode, if you want to delete the illustration, right click on the illustration and select Delete Selected Illustrations. You can also right-click to set the color, line width, etc. of the illustration. For parameter definitions and settings in Properties, please refer to the introduction to the 4.1 Workspace Introduction.

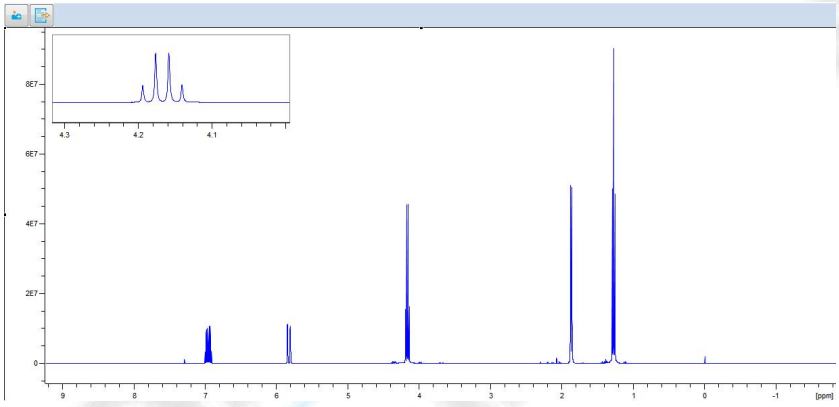


Figure 4.6 Show illustration

In illustration mode, place the cursor in the spectrum area of the illustration, and you can drag the entire illustration. Place the cursor in the upper left and upper right corners of the illustration, and a circle scattered icon will appear. At this time, drag the mouse to zoom in to the upper left corner or the upper right corner. Place the cursor near the midpoint of the left and right edges of the illustration, and a two-way arrow will appear. At this time, you can drag one side of the illustration to zoom in or out.

In illustration mode or after exiting illustration mode, you can place the cursor under the coordinate axis of the illustration. At this time, a two-way arrow will appear. Dragging the mouse left and right can change the illustration area, and scrolling the mouse pulley can zoom it horizontally; If the cursor is placed on the spectrum area of the illustration, you can scroll the mouse pulley to zoom it vertically.

After exiting the illustration mode, placing the cursor in the spectrum area will become a hand shape. At this time, dragging left and right can locally zoom in and out of the spectrum.

4.3 Window and Fourier Transform

Window and Fourier transform can be done by the command or dialog window.

4.3.1 Window and Fourier transform via command

Before using the window function, you need to set the window parameter: “window function”, “window factor”, and Fourier transform related parameters.

Users can set it in “Procparams” interface, as shown in Figure 4.7. Click the button and the window function selection window will pop up. There are mainly the following window functions: Gaussian, Exponential, Sine, Squared Sine, Cos, Squared Cos. You can check the required window function and set the value of the corresponding factor below. Note that the window function can be selected multiple times, indicating that several window functions can be used in superposition. It can also be set in the command line. For example, type ***wdw=‘exponential’*** in the command line to set the window function to exponential, type ***lb=3*** to set line broadening factor to 3 Hz.

When the value of wdw is set to sine, the values of sb and sbs are automatically changed to 0.5 and 0, respectively; when the value of wdw is set to cos, the values of sb and sbs are automatically changed to 1 and 0, respectively.

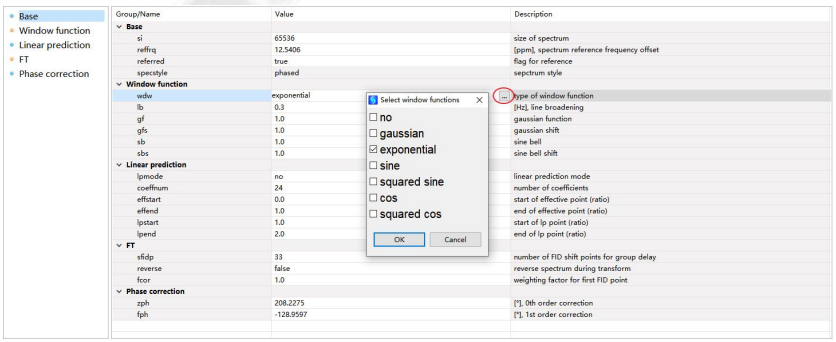


Figure 4.7 Window parameter

Fourier transform correlation parameters include si in the **Base** column, and the other

three parameters in the **FT** column. The parameters can be modified on the parameter panel or set on the command line.

Si represents the number of spectral data points, that is, the number of points in the Fourier transform. Unlike np, si must be a power of 2, such as 4096, 8192, 16384, 32768, and so on. If the value of np is between two powers of 2 at the time of sampling, then si is preferably taken as a power higher, and the value of the partial data point larger than np is filled with 0, which is commonly referred to as zero-filling. sfidp represents the number of group delay shift points. If reverse selects true, the inverse Fourier transform is performed; if false is selected, the positive Fourier transform is performed. fcor is the weighted multiple of the first point in the FID.

The following are window and Fourier transform related commands:

wft: Window function+Fourier transform

wftabs: Window function+Fourier transform+Display absolute spectrum

wftpwr: Window function+Fourier transform+Display power spectrum

4.3.2 Window and Fourier transform via dialog

Click on the menu bar **Process** > **Fourier Transform** to open the window and Fourier transform dialog on the right side of the spectrum, as shown in Figure 4.8.

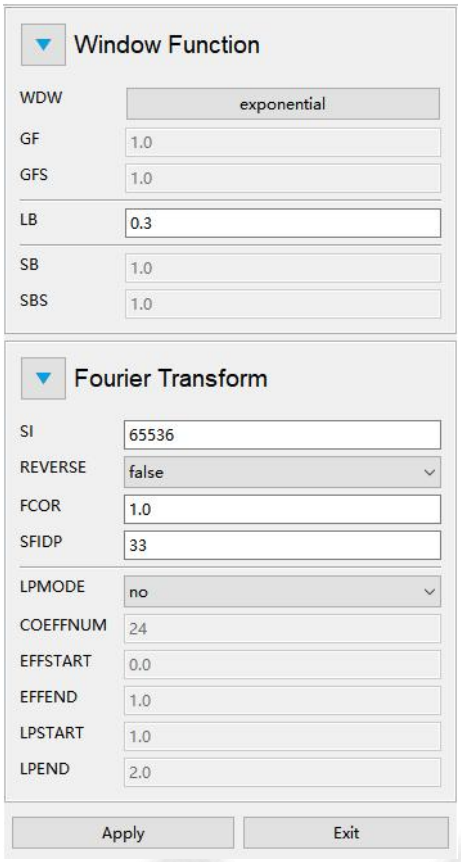


Figure 4.8 Window and Fourier transform dialog

After opening the window and Fourier transform dialog box, you can check the required windowing functions, mainly in the following categories: Gaussian, Exponential, Sine, Squared Sine, Cos, Squared Cos. Multiple window functions can be selected at the same time, but Sine and Squared Sine cannot be selected at the same time. Cos and Squared Cos cannot be selected at the same time. After selecting the window function, set the corresponding parameters. The basic parameters of the Fourier transform and the linear prediction related parameters can be edited in the **Fourier Transform** column. After setting the parameters, click the **Apply** button.This operation is equivalent to setting the parameter and then typing the command ***wft***. During the Fourier transform, the parameter lpmode is identified to determine whether to perform linear prediction. After the Fourier transform, the workspace automatically switches to the spectrum display page, as shown in Figure 4.9.

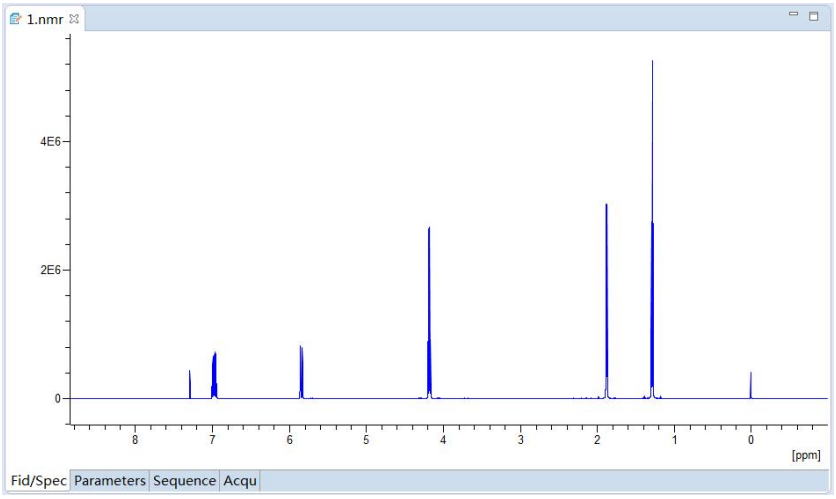


Figure 4.9 Spectrum display page

4.4 Linear Prediction

Linear prediction, as a sub-process in the Fourier transform process, can be performed by a Fourier transform correlation command or Fourier transform dialog.

4.4.1 Linear prediction via command

The linear prediction related parameters need to be set before using the command for linear prediction. The user can set it under the **Linear prediction** column on the **Procparams** page of the parameter page, as shown in Figure 4.10. It can also be set on the command line, such as typing ***lpmode="forward"*** at the command line and typing ***coffnum=24*** to set the number of prediction coefficients to 24.

Linear prediction		
lpmode	no	linear prediction mode
coffnum	24	number of cofficients
effstart	0.0	start of effective point (ratio)
effend	1.0	end of effective point (ratio)
lpstart	1.0	start of lp point (ratio)
lpend	2.0	end of lp point (ratio)

Figure 4.10 Linear prediction parameters

“lpmode” indicates the prediction mode. If "no" is selected, no linear prediction is performed. If "backward" is selected, backward prediction is performed (this function is temporarily not supported). If "forward" is selected, forward prediction is performed.

"coeffnum" indicates the number of prediction coefficients, and the number of points participating in the prediction must be greater than 5 times of "coeffnum", otherwise the prediction will fail.

"effstart" and "effend" represent the starting point index and ending point index of the participating predictions, and the value is a percentage of the total number of points relative to the FID. For example, forward and backward predict the default values of "effstart" and "effend" are usually 0 and 1.0 respectively. In fact, both can take any value between 0-1, but you must ensure that "effstart <effend" is met.

"lpstart" and "lpend" represent the starting point index and the ending point index of the predicted data, and the value is a percentage of the total number of points relative to the FID. For example, forward and backward predict the default values of "lpstart" and "lpend" are usually 1.0 and 2.0 respectively. But when "effstart" and "effend" do not use the default value, the values of "lpstart" and "lpend" must satisfy (lpend - lpstart) ≦ (effend - effstart).

After setting the parameters, type the Fourier transform related commands, such as ***ft***, ***wft***, etc. During the Fourier transform, the parameter "lpmode" is identified to determine whether to perform linear prediction.

4.4.2 Linear prediction via dialog

Click the menu bar **Process** > **Spectrum** > **Fourier Transform** to open the Fourier Transform dialog box, as shown in Figure 4.11.

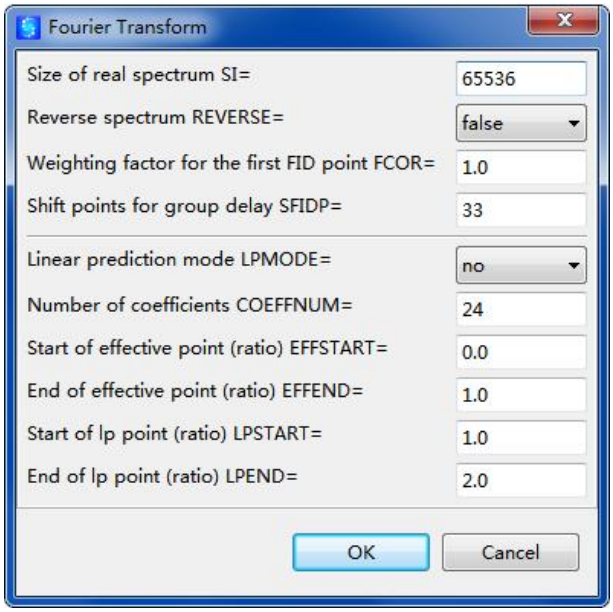


Figure 4.11 Fourier transform dialog box

Set the corresponding parameters and click **OK**. The operation performed at this time is equivalent to setting the parameter and then typing the command ***ft***. During the Fourier transform, the parameter lpmode is identified to determine whether to perform linear prediction.

4.5 Phase Correction

Phase correction is divided into automatic phase correction and manual phase correction.

4.5.1 Automatic phase correction

Automatic phase correction can be done by typing the command ***aph*** on the command line or by clicking on the menu bar **Process** > **Phase Correction** > **Automatic**. The spectra before and after doing automatic phase correction are shown in Figure 4.12 and Figure 4.13 respectively.

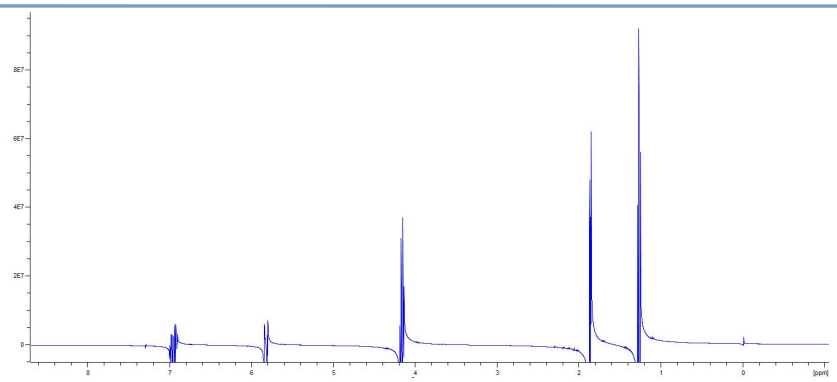


Figure 4.12 Before ***aph***

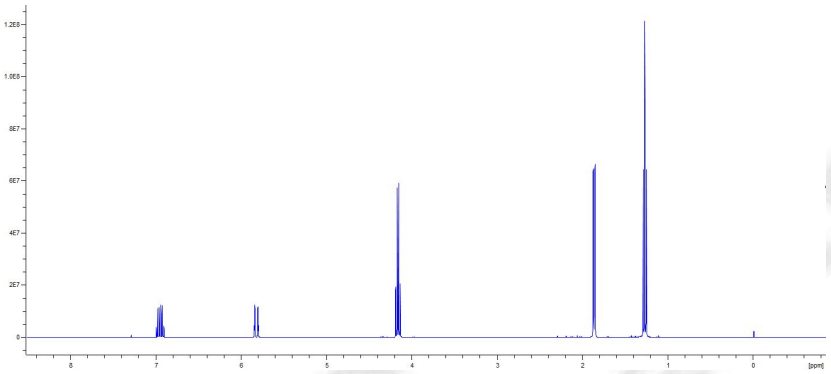


Figure 4.13 After ***aph***

4.5.2 Manual phase correction

Click **Phase Correction** > **Manual** under the **Process** menu, or click the toolbar icon to enter the manual phase correction mode. The phase correction toolbar will appear at the top of the spectrum, as shown in Figure 4.14.

Figure 4.14 Manual phase correction toolbar

A vertical green line will appear on the spectrum interface (the default position is the highest peak center of the spectrum), which is the baseline pivot, as shown in Figure 4.15. You can set the mouse location to the new pivot by selecting the right-click menu **Set Pivot Point**.

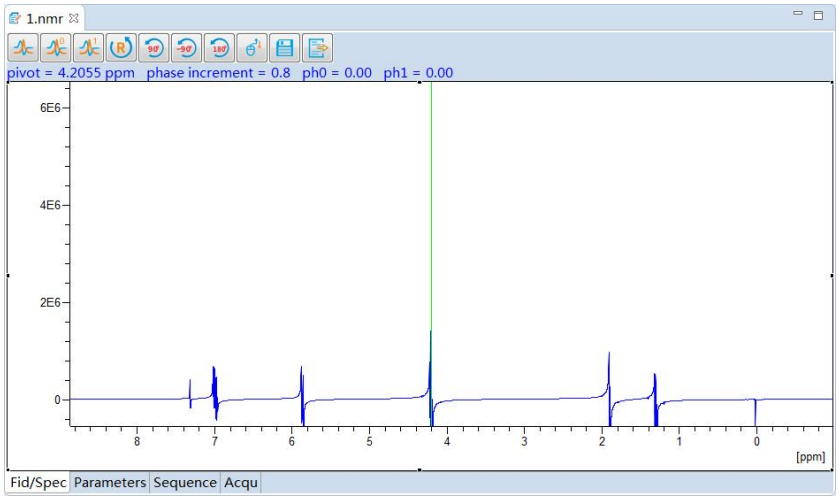


Figure 4.15 Manual phase correction

Now move the mouse to the phase correction toolbar button, then slide the mouse wheel to adjust the 0 order phase, as shown in Figure 4.16 . Clicking the button or will increase or decrease the zero-order phase by 90 degrees. Clicking the button will increase the zero-order phase by 180 degrees.

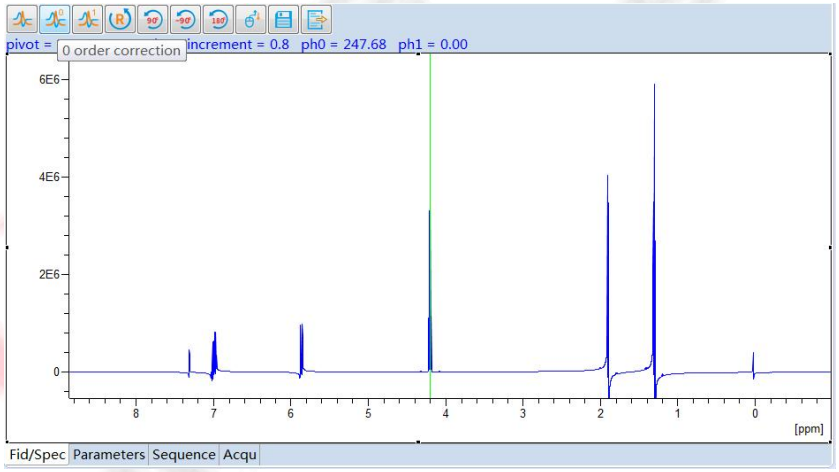


Figure 4.16 Adjusting the 0 order phase

Then move the mouse to the phase correction toolbar button, slide the mouse wheel to adjust the first-order phase, and the phase-aligned spectrum is shown in Figure 4.17 . If you are not satisfied with the calibration result, click the button to return to the initial state and re-calibrate. Click the button to save the current phase correction spectrum and exit. Click the button to exit without saving the phase correction result.

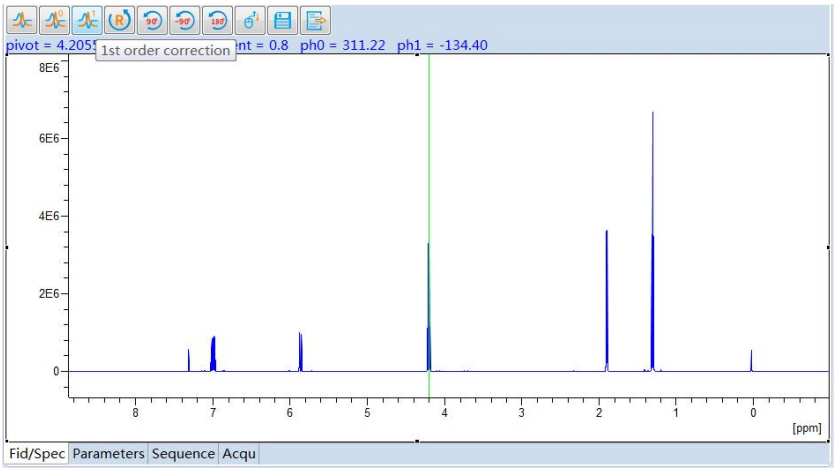


Figure 4.17 Spectrum after adjusting the phase

Below the phase correction toolbar, there is a status information bar as shown in Figure 4.18. The value of pivot indicates the position of the red reference line; the “phase increment” indicates the sensitivity of the phase adjustment, and the larger the value, the greater the change of the phase of the mouse sliding in the 0 order and 1st order phase adjustment. It can be adjusted by the button . Put the mouse on the button and slide the mouse wheel up and down to increase or decrease the phase increment. Ph0 represents the value of the current 0 order phase, and ph1 represents the value of the current 1st order phase.

Figure 4.18 Phase correction information bar

4.6 Baseline Correction

4.6.1 Drift correction

Drift correction can be done by typing the command ***dc*** on the command line. This function is equivalent to automatic calculation of parameters for zero-order polynomial automatic baseline correction of the spectrum.

4.6.2 Automatic baseline correction

Automatic baseline correction can be done by typing the command ***abs*** or ***abs2*** on the

command line. They corresponds to BCRI and ARPLS under the menu bar **Process** > **Baseline Correction** > **Automatic** respectively. The spectra before and after doing automatic baseline correction ***abs*** are shown in Figure 4.19 and Figure 4.20 respectively.

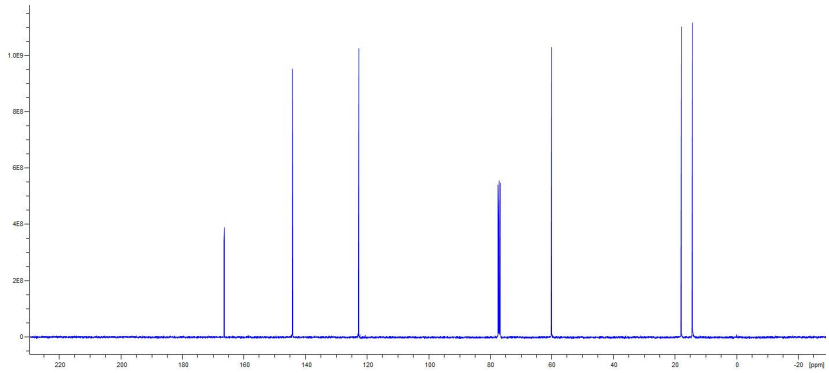


Figure 4.19 Before ***abs***

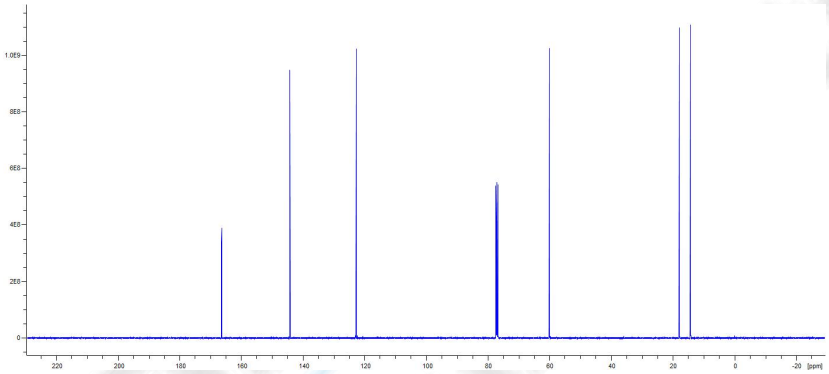


Figure 4.20 After ***abs***

When selecting ARPLS, parameter setting panel will pop up on the right side of the spectrum (Figure 4.21). There are two parameters, the wavelet transform factor (Cwt factor) and the smoothing coefficient (Lamda). The Cwt factor value range is [0.00001, 0.1], the recommended value is 0.1, the Lamda value range is [10^5, 10^10], and the default recommended value is 10^8.

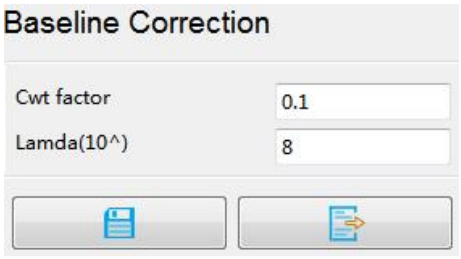


Figure 4.21 ARPLS parameter setting panel for one-dimensional spectrum

4.6.3 Semi-automatic baseline correction

Click the menu bar **Process** and select **Baseline Correction** > **Semi-automatic** to enter the semi-automatic baseline correction mode. The semi-automatic baseline correction dialog shown in Figure 4.22 is displayed on the right side of the spectrum.

The semi-automatic baseline correction uses an iterative algorithm. There are two parameters affecting the iterative algorithm: Noise Factor and Cwt Factor, which can be modified as needed.

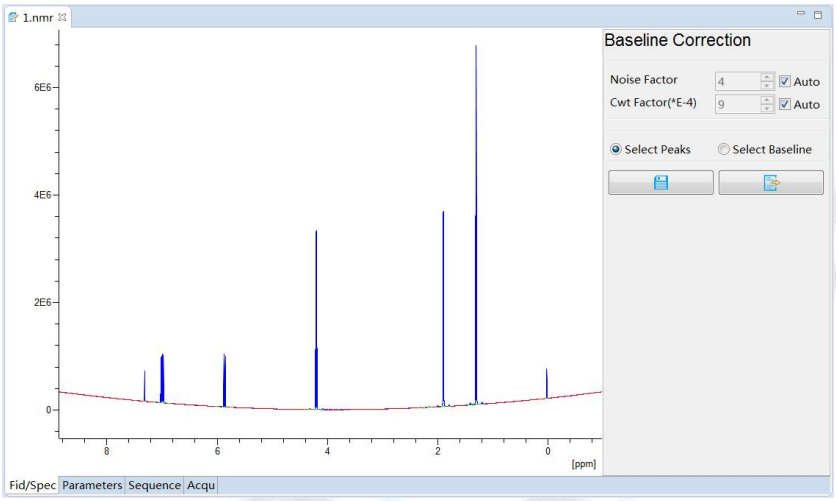


Figure 4.22 Semi-automatic baseline correction dialog

When initially entering the semi-automatic baseline correction mode, the program automatically simulates the baseline using an iterative algorithm based on the spectrum and displays the automatically calculated “Noise Factor” and “Cwt Factor” on the dialog. The area selected as the peak in the correction algorithm, the baseline color is green; the area selected as the baseline, the baseline color is red.

If you are not satisfied with the simulated baseline effect, you can uncheck the “Auto” option after the “Noise Factor” and “Cwt Factor” and manually adjust these two factors to make the simulated baseline and the spectral baseline as close as possible.

If there is still a partial baseline simulation effect is not ideal, you can select the peak or select the baseline manually. The dialog box has Select Peak and Select Baseline for selection. After selecting an option, the user can drag the mouse to select a region as a baseline or peak, and the program will simulate the baseline again according to the user's settings.

If the user needs to zoom the spectrum to view the local baseline simulation, click the right mouse button on the spectrum and select the right-click menu “Enter zoom in/out” to enter the zoom mode. Then drag the mouse from left to right on the spectrum to zoom in on the spectrum and drag the mouse from right to left to display the full spectrum. Click the right mouse button on the spectrum again and select “Exit zoom in/out” to exit the zoom mode.

Click the button to perform baseline correction on the spectrum based on the current simulated baseline and save and exit. Click the button to exit directly.

4.6.4 Manual baseline correction

Click the menu bar **Process** to select the **Baseline Correction** > **Manual** option, or click the button in the toolbar to enter the manual baseline correction mode. The manual baseline correction dialog shown in Figure 4.23 is displayed on the right side of the spectrum.

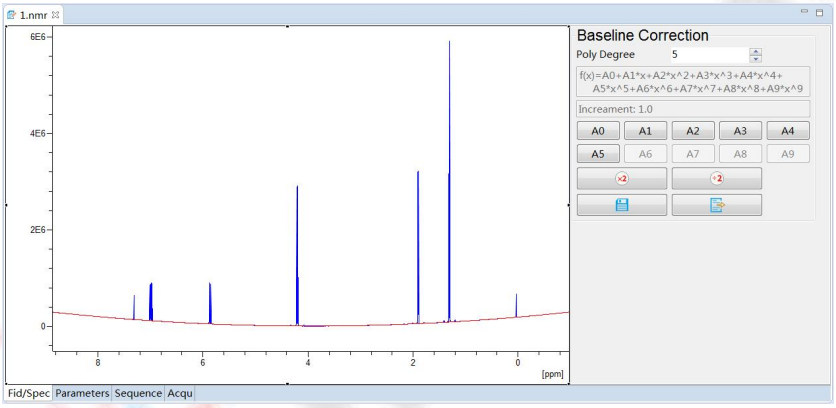


Figure 4.23 Manual baseline correction dialog

When entering the manual baseline correction mode initially, the default polynomial degree (Poly Degree) is 5, and the program automatically simulates the baseline using a polynomial algorithm. You can also manually modify the polynomial order, and the program will automatically simulate a new baseline based on the current order and display it. The adjustable range of the polynomial order is 0~9.

The lower text box of **Poly Degree** shows the polynomial formula, and the polynomial coefficients can be adjusted by buttons A0~A9. Place the mouse on any of the coefficient buttons, and the corresponding coefficient value of the current button will be automatically displayed on the interface, as shown in Figure 4.24. Slide the mouse wheel and the

corresponding factor will increase or decrease. If the order is 5, only A0~A5 can be edited, and A0~A5 are adjusted in turn to make the red line and the spectrum baseline overlap as much as possible.

"Increment" means sensitivity, the larger the value, the greater the change in the coefficient of the mouse's sliding circle. You can increase or decrease the sensitivity by clicking the button ×2 and ÷2.

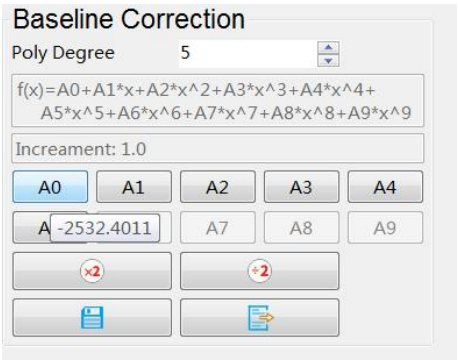


Figure 4.24 Polynomial coefficient

If you need to zoom the spectrum to see the local baseline simulation, drag the mouse from left to right on the spectrum to zoom in, and drag the mouse from right to left to display the full spectrum.

Click the button to perform baseline correction on the spectrum based on the current simulated baseline and save and exit. Click the button to exit directly.

Chapter 5 2D Data Processing

5.1 2D Spectrum Operation

Here are some general 2D spectral operations.

5.1.1 Spectrum zoom

Drag the mouse over the spectrum to zoom in on the spectrum. The specific operation is as follows: select the starting point on the spectrum, click the left mouse button and drag to the end point to release the left mouse button, and the spectrum within the dragged rectangle will be enlarged. Using the toolbar will restore the full spectrum display.

Perform Fourier transform related operations after the spectrum is zoomed , it will maintain the same display range as before the transformation.

5.1.2 Move the spectrum

The display area cannot display all the spectrum after the spectrum is enlarged. Then you need to move the spectrum to see the details of each part of the spectrum.

Move the spectrum in the horizontal direction. The display region can not display the entire spectrum after the spectrum is zoomed in, at this moment, you need to move the spectrum horizontally to see the details of each part of the spectrum. Click and drag the upper or lower axis to drag the spectrum horizontally. Use the toolbar to move the spectrum in the horizontal direction. shows the left shift, shows the right shift, shows moving to the top left end of spectrum, shows moving to the top right end of spectrum.

Move the spectrum in the vertical direction. Use the toolbar to move the spectrum in the vertical direction. shows the up shift, shows the down shift, shows the spectrum base line is in the middle, shows the the spectrum baseline is is set at the bottom.

5.1.3 Spectrum display

Click the right mouse button and select the “Edit Contour Levels” option in the spectrum display area. The dialog will open as shown in Figure 5.1.

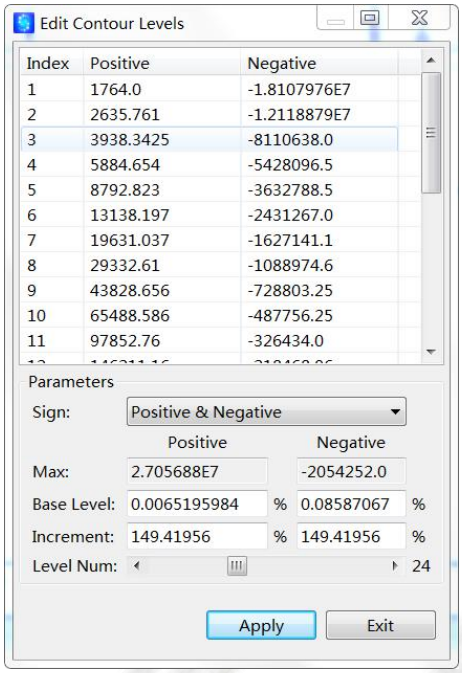


Figure 5.1 Contour editing dialog

The user can modify the display sign. If the current spectrum is non-phase sensitive, then Sign can only select positive signal. If the current spectrum is phase sensitive, Sign can choose to display positive and negative signals simultaneously, positive signals or Negative signal. The maximum value (Max Value) is automatically calculated based on the spectrum and cannot be edited. The user can modify the contour level (Base level), the contour growth factor (Increment), and the Number of contour (Level Num). Wherein, the Base level is a percentage relative to the Max Value; the Increment represents the growth ratio of the adjacent two contour lines; the Level Num represents the total number of contour lines calculated, and currently several of them can be selected for display.

Users can place the cursor on the spectrum and slide the mouse wheel to control the number of displayed contour lines. You can also control the displayed contour line number by clicking one of the contour lines with the left mouse button in the Contour dialog box. As shown in Figure 5.2, the white background contour is a calculated but not displayed contour,

and the blue background is the currently displayed contour.

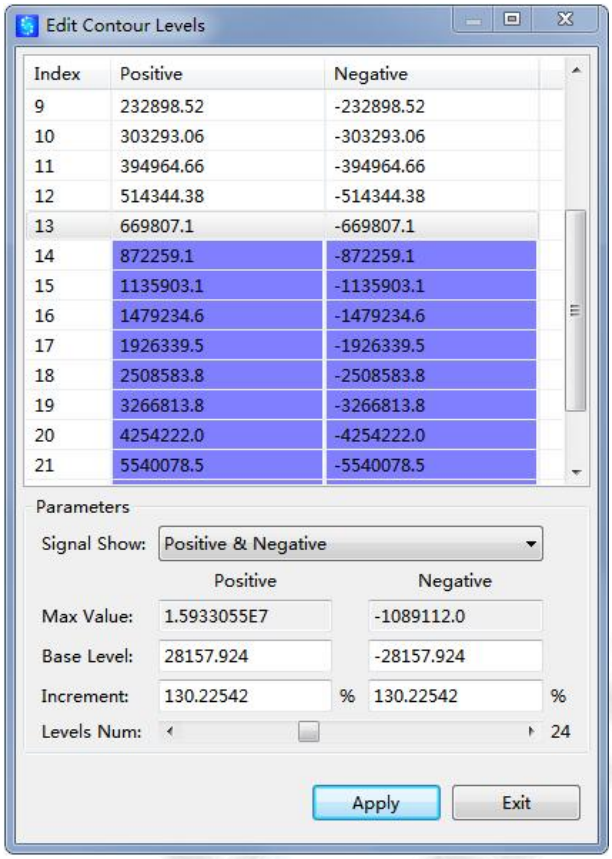


Figure 5.2 Contour line diagram

Click the “Apply” button to apply the set value to the current spectrum, and click “Exit” to exit directly.

5.1.4 Row and column display

First, open the 2D spectrum that needs to display the row and column data, click the icon  in the toolbar to enter the row and column display mode, and place the cursor at any point in the spectrum, it will display the hydrogen spectrum data corresponding to the current row and column, as shown in Figure 5.3. You can also click on the icon  or  separately display the hydrogen spectrum corresponding to the row or column at any position, and Figure 5.4 shows the hydrogen spectrum corresponding to a row of the 2D spectrum..

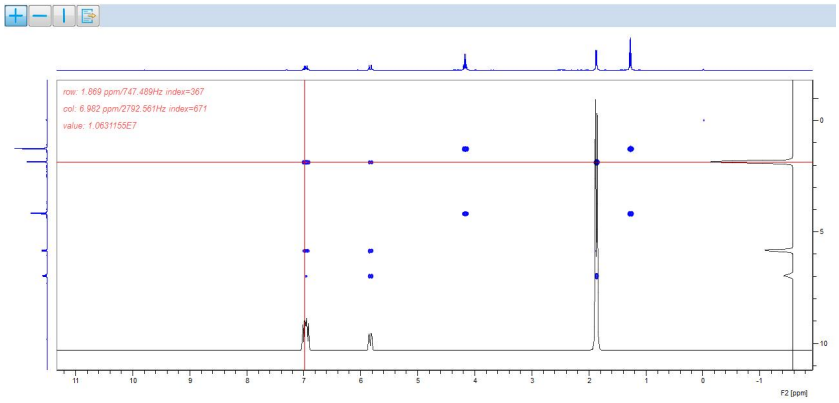


Figure 5.3 Row and column display for 2D spectrum

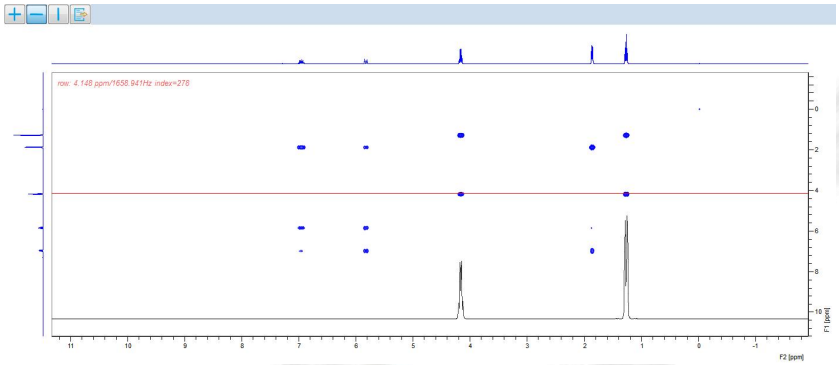


Figure 5.4 Row display for 2D spectrum

In this mode, the right mouse button can export the hydrogen spectrum of a row or column (Figure 5.5). Click to select “Export F2 Dimension Data”, a dialog box will pop up as shown in Figure 5.6, fill in the corresponding number of the data (the number range is displayed in the right parenthesis), and the default number is the row and column number corresponding to the red cross line intersection in Figure 5.5. By default, the data is exported to the same directory of the current array data. You can also click the **Browse** button to select the path and automatically name it as “current data name _row/col_ filled in the row and column number”. The F2 dimension data is exported by default “Row”.

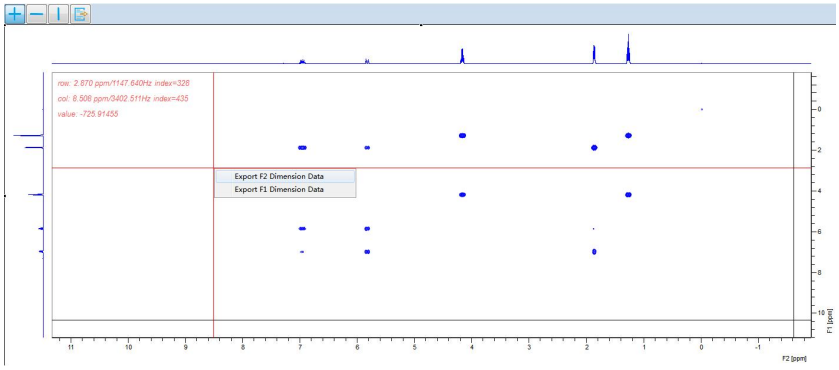


Figure 5.5 Row or column data export for 2D spectrum

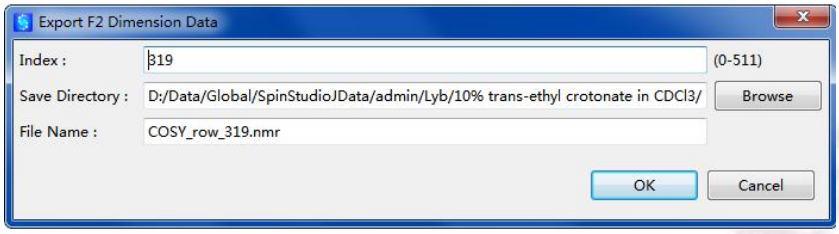


Figure 5.6 Row or column data export dialog for 2D spectrum

5.1.5 Projection Display

On the side of the two-dimensional spectral display area, the corresponding one-dimensional spectrum can be projected and displayed. Right-click on **Properties** in the 2D spectrum display area and open the property page, as shown in Figure 5.7. You can control the display/non-display of projections within **Components**.

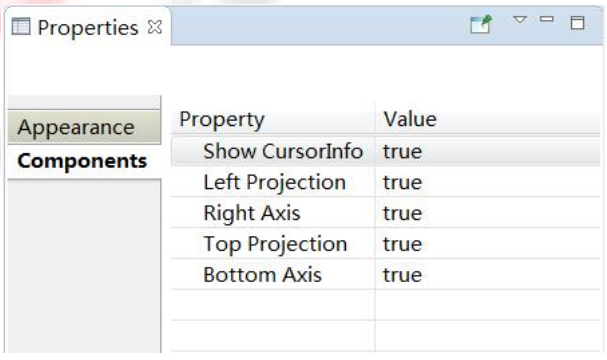


Figure 5.7 Projection display property page

After the projection is displayed, right-clicking on the projection area will display the menu shown in Figure 5.8. The display of the internal projection or the external projection

can be switched by the right-click menu.

If it is currently displayed as an internal projection, the default is to display “Positive”, and you can select “Positive”, “Negative”, or “Both” by right-click menu. If the current display is an external projection, the default is to display the positive and negative peaks (Both) and cannot be modified.

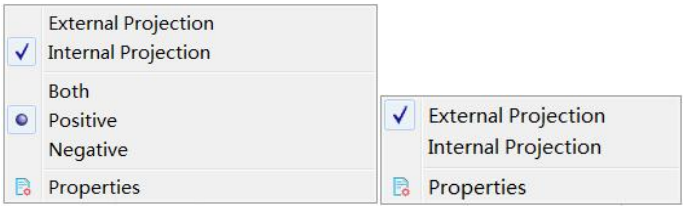


Figure 5.8 Right-click menu in projection state

Click on “External Projection”, if no external projection has been imported, the dialog shown in Figure 5.9 will pop up. You can import an external projection by selecting the external projection data directory.

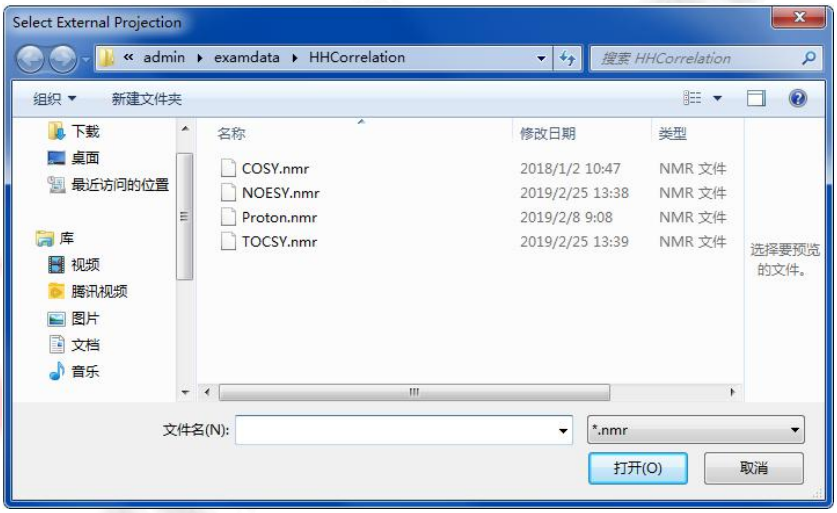


Figure 5.9 Select external projection data directory

If an external projection has been imported, the data of the last imported external projection will be saved in the .nmr file of the current path. When the user clicks on External Projection, the dialog box shown in Figure 5.10 will pop up first, allowing the user to select whether to display the “Projection in current file” or “Projection in other file”. If the user selects “Projection in current file”, it will directly display the external projection of the last import. If “Projection in other file” is selected, the dialog box shown in Figure 5.9 above will

pop up to reselect the one-dimensional projection data.

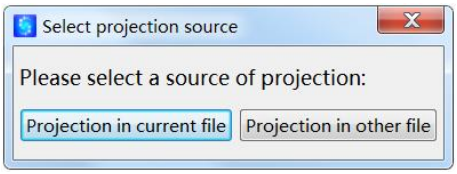


Figure 5.10 Import external projection dialog

5.2 2D Window and Fourier Transform

2D window and Fourier transform is similar to 1D, and can be done through command or dialog. The parameter type of the 2D windowing is the same as that of the 1D, and only the corresponding parameters in the F1 dimension are added. For example, the window function on the F2 dimension is “wdw”, and the window function on the F1 dimension is “wdw1”; the Fourier transform points on the F2 dimension are si, and the Fourier transform points on the F1 dimension are si1.

5.2.1 Window and Fourier transform via command

Before using the window, you need to set the window parameter: window function, and window factor. Users can set it in Window function of “Procparams” interface, as shown in Figure 5.11. It can also be set in the command line. For example, type ***wdw1=‘exponential’*** in the command line to set the window function of F1 dimension to exponential, type ***lb1=3*** to set line broadening factor of F1 dimension to 3 Hz.

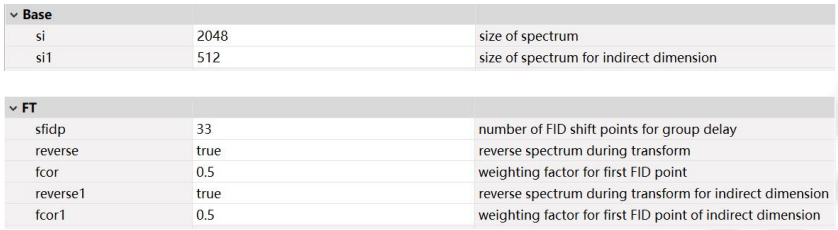
Window function		
wdw	sine	type of window function
lb	1.0	[Hz], line broadening
gf	1.0	gaussian function
gfs	1.0	gaussian shift
sb	0.5	sine bell
sbs	0.0	sine bell shift
wdw1	sine	type of window function for indirect dimension
lb1	3.0	[Hz], line broadening for indirect dimension
gf1	1.0	gaussian function for indirect dimension
gfs1	1.0	gaussian shift for indirect dimension
sb1	0.5	sine bell for indirect dimension
sbs1	0.0	sine bell shift for indirect dimension

Figure 5.11 Window parameter

When the corresponding parameters sb and sbs of the sine window are 0.5 and 0,

respectively, when the parameters sb and sbs corresponding to the cos window are 1, 0, respectively, a better effect can be obtained. Therefore, when the value of wdw1 is set to sine, the values of sb1 and sbs1 are automatically changed to 0.5 and 0, respectively; when the value of wdw1 is set to cos, the values of sb1 and sbs1 are automatically changed to 1 and 0, respectively.

The parameters invoked by the command to perform the Fourier transform are the Fourier related parameters in the "Procparams" parameter panel, including the si in the "Base" column, and the other five parameters in the "FT" column, as shown in Figure 5.12 The parameters can be modified on the parameter panel or set on the command line.



Base		
si	2048	size of spectrum
si1	512	size of spectrum for indirect dimension

FT		
sfidp	33	number of FID shift points for group delay
reverse	true	reverse spectrum during transform
fcor	0.5	weighting factor for first FID point
reverse1	true	reverse spectrum during transform for indirect dimension
fcor1	0.5	weighting factor for first FID point of indirect dimension

Figure 5.12 2D Fourier transform parameter

"si" represents the number of spectral data points, "sfidp" represents the number of group delay shift points. If reverse selects true, the inverse Fourier transform is performed; if false is selected, the positive Fourier transform is performed. "fcor" is the weighted multiple of the first point in the FID.

The following is the window and Fourier transform related command, which is exactly the same as the one-dimensional Fourier transform command.

wft: Window function+Fourier transform

wftabs: Window function+Fourier transform+Display absolute spectrum

wftpwr: Window function+Fourier transform+Display power spectrum

5.2.2 Window and Fourier transform via dialog

Click on **Spectrum** > **Fourier Transform** under the **Process** menu to display the window and Fourier transform dialog on the right side of the spectrum, as shown in Figure 5.13.

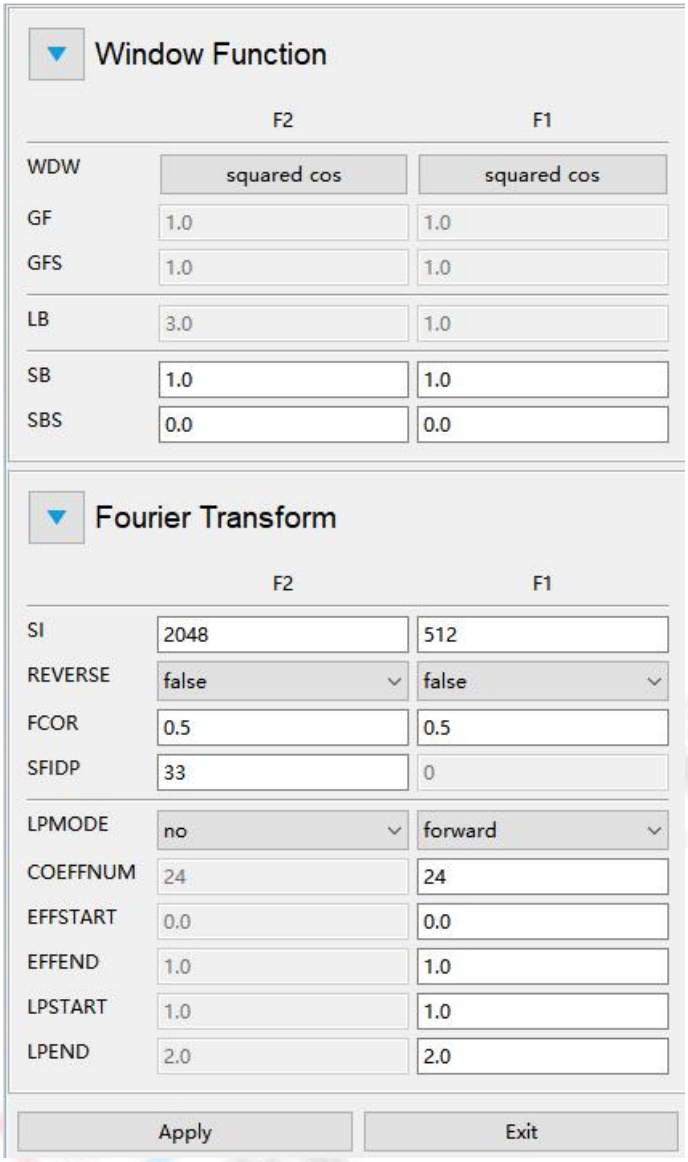


Figure 5.13 Window and Fourier transform dialog

After opening the window and Fourier transform dialog box, you can check the required windowing functions, mainly in the following categories: Gaussian, Exponential, Sine, Squared Sine, Cos, Squared Cos. Multiple window functions can be selected at the same time, but Sine and Squared Sine cannot be selected at the same time. Cos and Squared Cos cannot be selected at the same time. After selecting the window function, set the corresponding parameters.

The basic parameters of the Fourier transform and the linear prediction related parameters can be edited in the **Fourier Transform** column. After setting the parameters, click the **Apply** button. The operation performed at this time is equivalent to setting the

parameter and then typing the command ***wft***. During the Fourier transform, the parameter lpmode and lpmode1 are identified to determine whether to perform linear prediction. After the Fourier transform, the workspace automatically switches to the spectrum display page.

5.3 2D Linear Prediction

Linear prediction is a sub-process in the Fourier transform process. 2D linear prediction is the same as 1D, and can be performed by Fourier transform related commands or Fourier transform dialog. The parameter type of 2D windowing is the same as that of 1D, and only the corresponding parameters in the F1 dimension are added. For example, the prediction mode on the F2 dimension is “lpmode”, and the window function on the F1 dimension is “lpmode1”.

5.3.1 Linear prediction via command

The linear prediction related parameters need to be set before using the command for linear prediction. The user can set it under the Linear prediction column on the “Procparams” page of the parameter page, as shown in Figure 5.14. It can also be set on the command line, such as typing ***lpmode="forward"*** at the command line and typing ***coffnum=24*** to set the number of prediction coefficients to 24.

Linear prediction		
lpmode	no	linear prediction mode
coeffnum	24	number of coefficients
effstart	0.0	start of effective point (ratio)
effend	1.0	end of effective point (ratio)
lpstart	1.0	start of lp point (ratio)
lpend	2.0	end of lp point (ratio)
lpmode1	forward	linear prediction mode for indirect dimension
coeffnum1	24	number of cofficients for indirect dimension
effstart1	0.0	start of effective point (ratio) for indirect dimension
effend1	1.0	end of effective point (ratio) for indirect dimension
lpstart1	1.0	start of lp point (ratio) for indirect dimension
lpend1	2.0	end of lp point (ratio) for indirect dimension

Figure 5.14 Linear prediction parameters

“lpmode” indicates the prediction mode. If "no" is selected, no linear prediction is performed. If "backward" is selected, backward prediction is performed (this function is temporarily not supported). If "forward" is selected, forward prediction is performed.

“coffnum” indicates the number of prediction coefficients, and the number of points participating in the prediction must be greater than 5 times of “coffnum”, otherwise the

prediction will fail.

"effstart" and "effend" represent the starting point index and ending point index of the participating predictions, and the value is a percentage of the total number of points relative to the FID. For example, forward and backward predict the default values of "effstart" and "effend" are usually 0 and 1.0 respectively. In fact, both can take any value between 0-1, but you must ensure that "effstart <effend" is met.

"lpstart" and "lpend" represent the starting point index and the ending point index of the predicted data, and the value is a percentage of the total number of points relative to the FID. For example, forward and backward predict the default values of "lpstart" and "lpend" are usually 1.0 and 2.0 respectively. But when "effstart" and "effend" do not use the default value, the values of "lpstart" and "lpend" must satisfy (lpend - lpstart) ≦ (effend - effstart).

After setting the parameters, type the Fourier transform related commands, such as ***ft***, ***wft***, etc. During the Fourier transform, the parameter "lpmode" is identified to determine whether to perform linear prediction.

5.3.2 Linear prediction via dialog

Click on **Spectrum** > **Fourier Transform** under the **Process** menu to open the Fourier Transform dialog box, as shown in Figure 5.15.

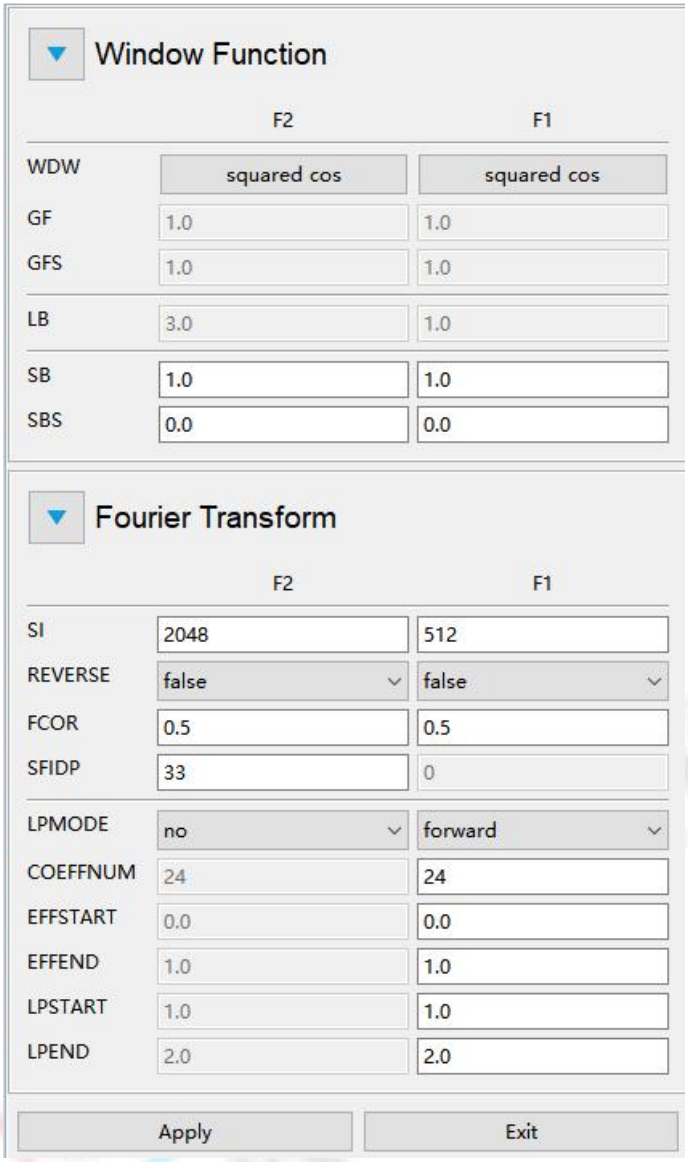


Figure 5.15 2D Fourier transform dialog

Set the corresponding parameters and click **Apply**. The operation performed at this time is equivalent to setting the parameter and then typing the command ***ft***. During the Fourier transform, the parameter “lpmode” and “lpmode1” are identified to determine whether to perform linear prediction for F2 dimension and F1 dimension.

5.4 2D Phase Correction

Phase correction is divided into automatic phase correction and manual phase correction.

5.4.1 Automatic phase correction

Automatic phase correction can be done by typing the command ***aph*** on the command line or by clicking on the menu bar **Process** > **Phase Correction** > **Automatic**. The spectra before and after doing automatic phase correction are shown in Figure 5.16 and Figure 5.17 respectively.

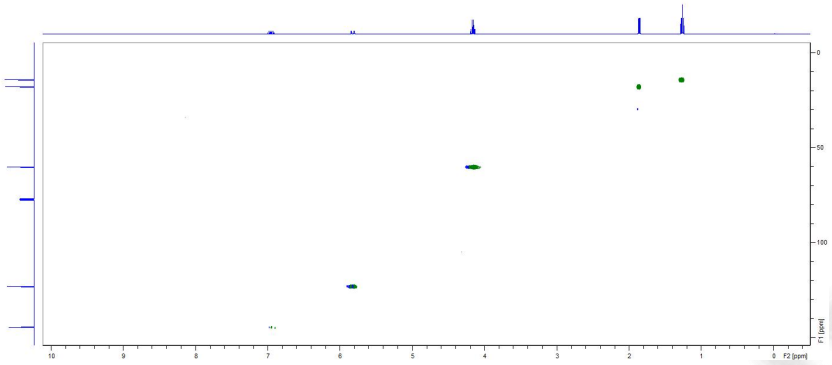


Figure 5.16 Before ***aph***

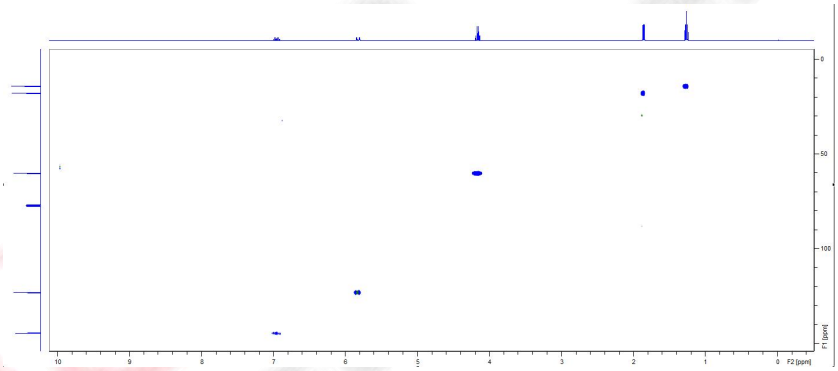


Figure 5.17 After ***aph***

5.4.2 Manual phase correction

Click the toolbar icon  or click **Phase Correction** > **Manual** under the **Process** menu. The spectrum will enter the manual phase correction mode. The phase correction toolbar will appear at the top of the spectrum. The toolbar is shown in Figure 5.18.

Figure 5.18 Manual phase correction toolbar for 2D spectrum

Move the mouse to the 2D spectrum in the workspace and right click to display the context menu, as shown in Figure 5.19. Select “Add Peak”, the peak of the current position of the mouse is marked with a small red circle and a crosshair to indicate that the peak is selected. “Remove” indicates canceling the currently selected peak, and “Remove All Peaks” means canceling all currently selected peaks. You can select 2~4 points as needed.

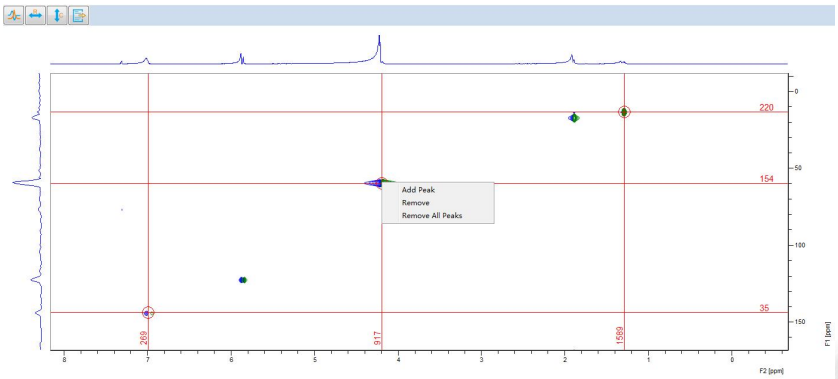


Figure 5.19 Schematic diagram of peak selection for 2D spectrum phase correction

After selecting the peak, click to enter the row phase mode, and click to enter the column phase mode. The row or column corresponding to the selected point is extracted and arranged vertically for phase modulation, as shown in Figure 5.20. The green line on the spectrum is the baseline pivot, and the red line position is the highest point of the absolute value of the spectrum.

The phase adjustment method is the same as 1D, and the workspace reflects the spectrum phase change in real time.

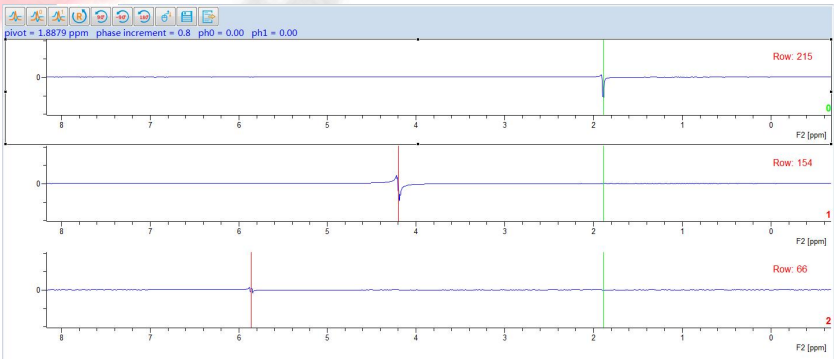


Figure 5.20 2D spectrum interactive phase adjustment

The spectrum effect of the two-dimensional spectral phase correction is shown in Figure 5.21.

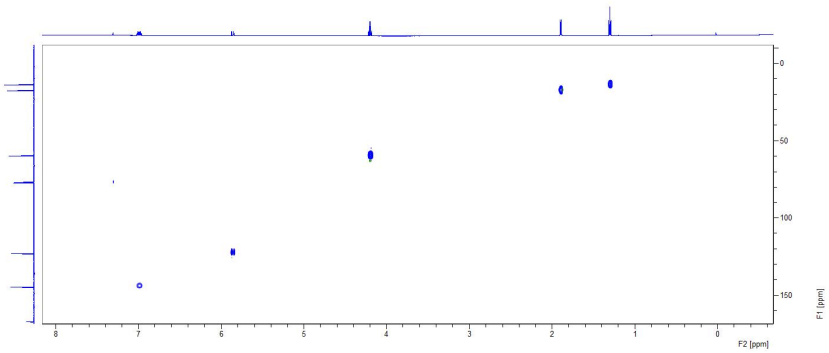


Figure 5.21 2D spectrum phase correction effect diagram

5.5 2D Baseline Correction

The 2D spectrum only supports automatic baseline correction now. Automatic baseline correction can be done by typing the command ***abs*** or ***abs2*** on the command line. They corresponds to BCRI and ARPLS under the menu bar **Process** > **Baseline Correction** > **Automatic** respectively. The spectra before and after doing automatic baseline correction ***abs*** are shown in Figure 5.22 and Figure 5.23 respectively.

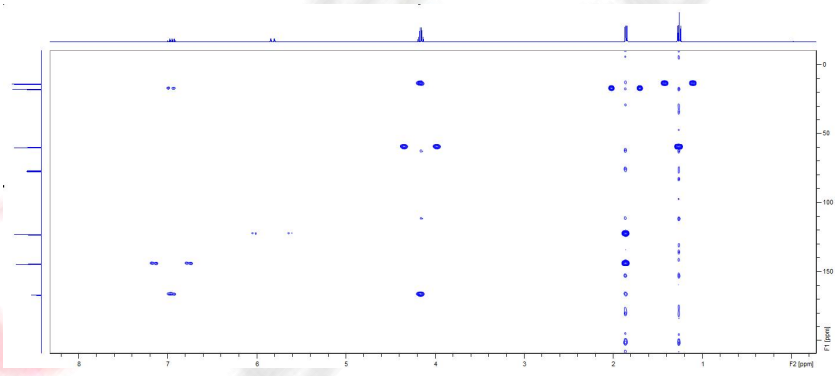


Figure 5.22 Before ***abs***

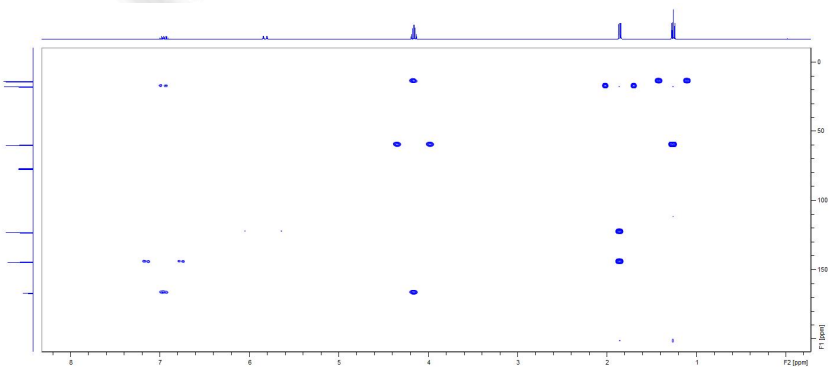


Figure 5.23 After ***abs***

When selecting ARPLS, parameter setting panel will pop up on the right side of the spectrum (Figure 5.24). There are two parameters, the wavelet transform factor (Cwt factor) and the smoothing coefficient (Lamda). The Cwt factor value range is [0.00001, 0.1], the recommended value is 0.008, the Lamda value range is [10^5, 10^10], and the default recommended value is 10^6.

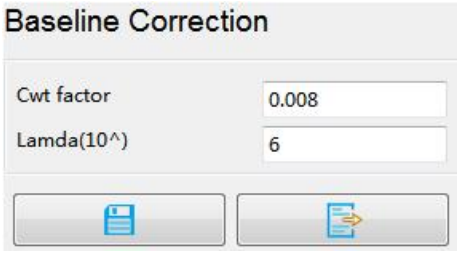


Figure 5.24 ARPLS parameter setting panel for two-dimensional spectrum

5.6 J Spectrum Tilt

Click the **Tilt** option in the menu **Process**, open J spectrum tilt dialog as shown in Figure 5.25.

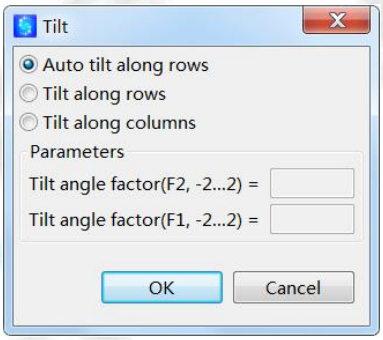


Figure 5.25 J Spectrum Tilt dialog

Auto tilt along rows:

Automatically tilt along the row. This can also be done by entering the command ***tilt*** on the command line.

Tilt along rows:

Tilt in row direction. When you select this option, you need to manually enter the Tilt angle factor (F2, -2...2) on the dialog. The range of this parameter is between -2 and 2. Tilt angle θ=arctan(factor), when θ is a positive value, the point above the axis in the horizontal

level of the spectrum is tilted to the right, and the point below the horizontal center axis is tilted to the left. When θ is a negative value, the tilt direction is opposite.

Tilt along columns:

Tilt in row direction. When you select this option, you need to manually enter the Tilt angle factor (F1, -2...2) on the dialog. The range of this parameter is between -2 and 2. Tilt angle θ=arctan(factor), when θ is a positive value, the point on the left side of the axis of the spectrum is tilted upward, and the point on the right side of the center axis is tilted downward. When θ is a negative value, the tilt direction is opposite.

5.7 t1 Noise Elimination

Click the **Eliminate t1 Noise** option under **Process**, or click the toolbar icon to open the "Eliminate t1 Noise" parameter settings panel, as shown in Figure 5.26.

The parameter "Sliding window" indicates the size of the sliding window and takes a positive integer. The maximum value is one-half of the data processing parameter si1, and the default value is 4. The smaller the parameter value, the more accurate the calculation when performing the t1 noise elimination operation, and the parameter value can be adjusted according to the processing effect. After setting the parameters, click the button to apply to the current workspace spectrum. The effect after noise elimination is shown in Figure 5.27, and then click the button to exit.

You can also enter the command ***et1*** directly in the command line and use the default parameters to eliminate t1 noise.

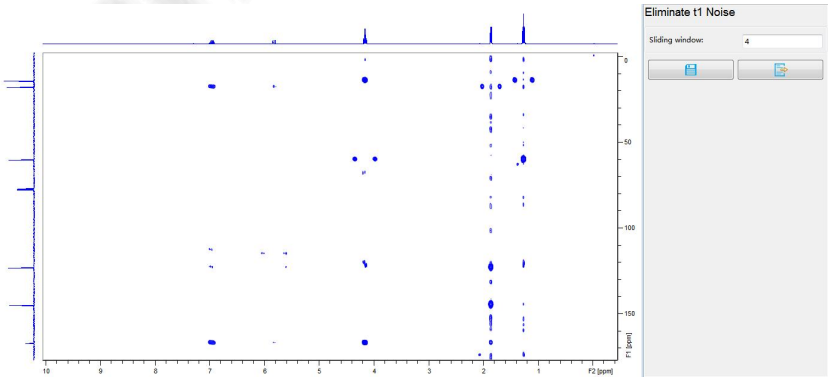


Figure 5.26 Enter to t1 noise elimination mode

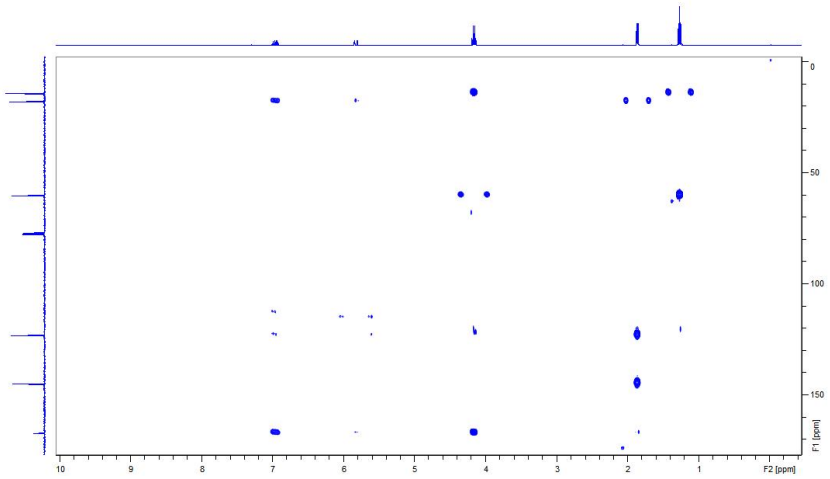


Figure 5.27 t1 noise elimination effect

5.8 Symmetry

5.8.1 COSY-like

COSY spectrum symmetry can be performed by clicking the **Process** menu, selecting the **Symmetry** > **COSY-like** option, or entering the command ***sym*** directly.

Note: When the COSY spectrum is symmetrized, the F2 dimension Fourier transform points are the same as the F1 dimension Fourier transform points, that is, si is equal to si1.

5.8.2 J-Resolved

J-spectrum symmetry can be performed by clicking on the menu bar **Process**, selecting the **Symmetry** > **J-Resolved** option, or entering the command ***symj*** directly. Before the symmetry of the homonuclear J-spectrum, it should be tilted first, see 5.6 J Spectrum Tilt.

Chapter 6 Data Analysis

6.1 Reference

There are two modes of calibration: automatic calibration and manual calibration.

6.1.1 Automatic calibration

Automatic calibration is based on the solvent selected when the field is locked. Click the menu bar **Analysis** to select the **Reference** > **Automatic** option, or enter the ***aref*** command in the command bar to perform the auto-calibration operation (the calibration operation is performed by default when the sampling command is executed).

Note: If the wrong solvent is selected during the injection or new experiment, the automatic calibration will produce the wrong result. In this case, you can type ***rearef*** on the command line, pop up the solvent selection box (Figure 6.1), select **Lock Solvent** and **Real Solvent**, click the **OK** button, then the solvent information in the base information column of the **Acquparams** will be automatically modified, and perform the calibration operation again based on the current solvent.

Figure 6.1 Solvent selection box

6.1.2 Manual calibration

Click the **Reference** > **Manual** option under the menu bar **Analysis**, or click the toolbar icon to enter the manual calibration mode. A green line will appear in the spectrum, as shown in Figure 6.2.

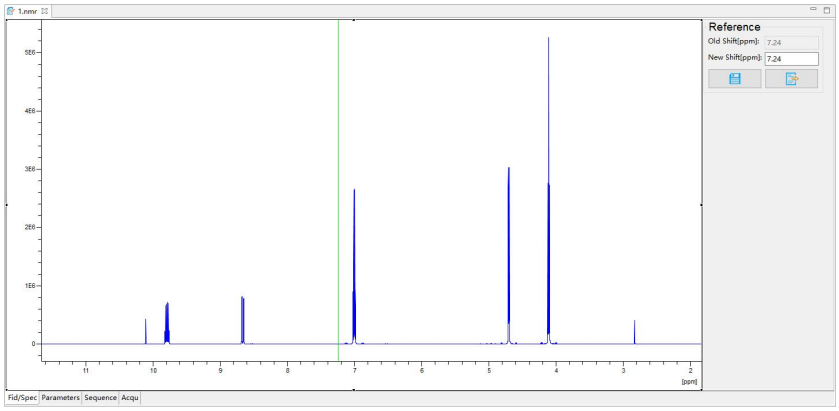


Figure 6.2 Manual calibration interface

Old Shift: The horizontal position of the green vertical line in the spectrum.

New Shift: Reset the horizontal position of the green vertical line.

: Save and exit the calibration mode.

: Exit calibration mode without saving.

If you want to set the coordinates of the rightmost peak of the spectrum to 0 ppm, just click the rightmost peak and set the value of "New Shift" to 0 on the right side of the pop-up page. Click the Save button to complete the calibration. The result is shown in Figure 6.3, and the rightmost peak coordinate has been set to 0 ppm.

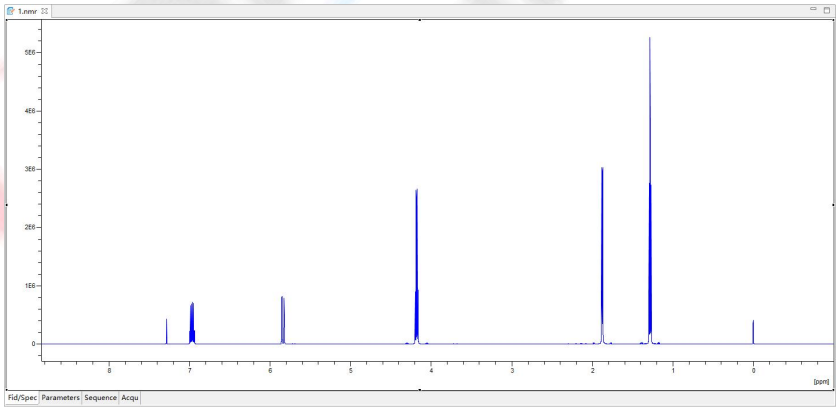


Figure 6.3 Calibration effect spectrum

After entering the calibration mode, the spectrum cannot be scaled horizontally. Click the right mouse button in the blank space to pop up the "Enter Zoom" option. Click this option to enter the spectrum zoom mode. Click the right mouse button in zoom mode to pop up the "Exit Zoom" option, click this option to exit the spectrum zoom mode.

6.2 Peak Picking

There are two types of peak picking modes: automatic and manual. After the peak picking result is saved, the “Peak” item will be generated on the right side of **Acqu** below the workspace. You can select a row or a column and right-click to copy or delete, or export the entire table data into .txt or .xls format file for post-processing.

6.2.1 Automatic peak picking

Click the menu bar **Analysis** to select the **Peak Picking** > **Automatic** option, or type ***pps*** in the command line to perform an automatic peak picking operation. The peak picking results are shown in Figure 6.4. When ***pps*** is input, the threshold is automatically calculated, that is, the spectrum is divided into 32 segments, the difference between the maximum and minimum intensities of each segment is calculated, and then the smallest difference in the 32 segments is multiplied by 4. You can also manually set the threshold according to the ordinate value of the spectrum. For example, you can type ***pps(1000000)*** or ***pps 1000000***, which is equivalent.

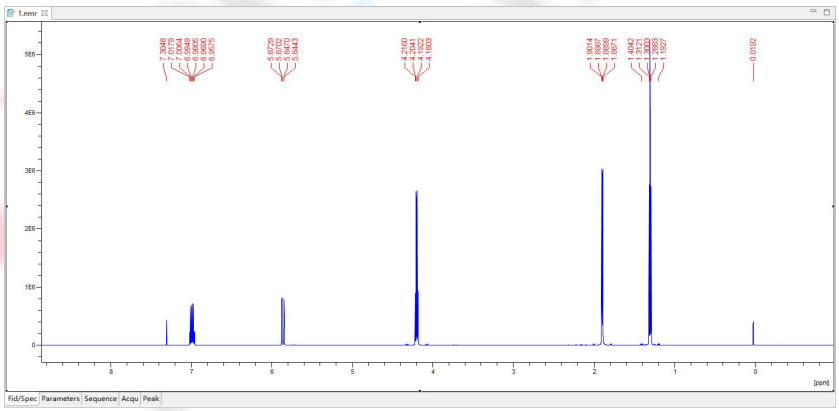


Figure 6.4 Peak picking results

6.2.2 Manual peak picking

Click on the menu **Analysis** to select the **Manual** option under **Peak Picking**, or click the toolbar button to enter the manual peak picking mode. The toolbar shown in Figure 6.5 will appear at the top left of the spectrum.

Figure 6.5 Peak picking toolbar

: Peak picking by threshold. See 1 **Peak picking by threshold** below for details.

: Peak picking manually. See 2 **Peak picking manually** below for details.

: Pick max peak in selected range. Hold down the left mouse button to select the range, and when you release it, automatically pick the peak with the highest intensity in the range. In this mode, the right mouse button function is exactly the same as the “peak picking manually”. Refer to 2 **Peak picking manually**.

: Pick peaks in selected ranges. See 3 **Pick peaks in selected ranges** below for details.

: Delete the selected peak. First select the peak to be deleted, and then click this icon to clear the currently selected peak information label.

: Remove all peaks. Clicking on this icon will clear all peak information labels displayed on the spectrum. In the array spectrum, click the icon to clear the peak information label for the currently selected spectrum.

: Save and exit peak picking mode. The peak information obtained by the “peaks” is saved as a node to the system directory, the node name is “peaks”, and then the peak picking mode is exited. The system log records the full path to the saved file.

: Exit peak picking mode without saving. The system does not save the peak information and exits the peak-picking mode, but the peak-picking label is temporarily retained on the workspace interface.

A detailed description of threshold peak picking and manual peak picking is as follows.

1. Peak picking by threshold

When entering the manual peak picking mode, the default is “Peak picking by threshold”. At this time, a green line is displayed in the spectrum, as shown in Figure 6.6.

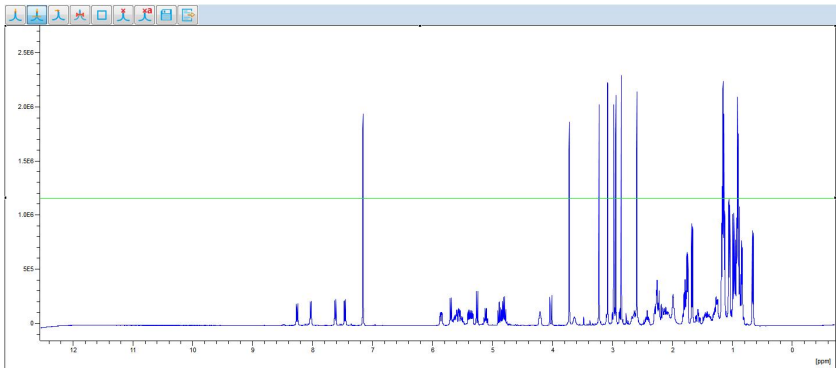


Figure 6.6 Interface for “Peak picking by threshold”

Click the right mouse button in the spectrum area to pop up the menu shown in Figure 6.7.

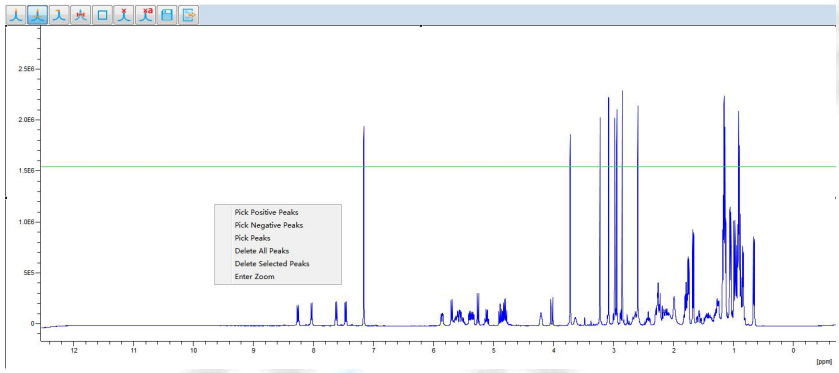


Figure 6.7 Right-click menu in “Peak picking by threshold” mode

Pick Positive Peaks: Pick all positive peaks larger than this threshold, and the results are shown in Figure 6.8;

Pick Negative Peaks: Pick all negative peaks whose absolute value is larger than this threshold;

Pick Peaks: Pick all positive peaks and negative peaks whose absolute value larger than this threshold;

Delete All Peaks: Clear all peak information in the spectrum (Figure 6.9);

Delete Selected Peaks: First select the peak to be deleted, and then click this option to clear the currently selected peak information label (Figure 6.9);

Enter Zoom: Enter zoom mode. After entering the peak picking mode, the spectrum cannot be scaled by dragging the mouse horizontally. You need to click this option to zoom. Peak picking is not supported in zoom mode. After zooming to the desired area, click “Exit

Zoom" to exit the spectrum zoom mode (Figure 6.9).

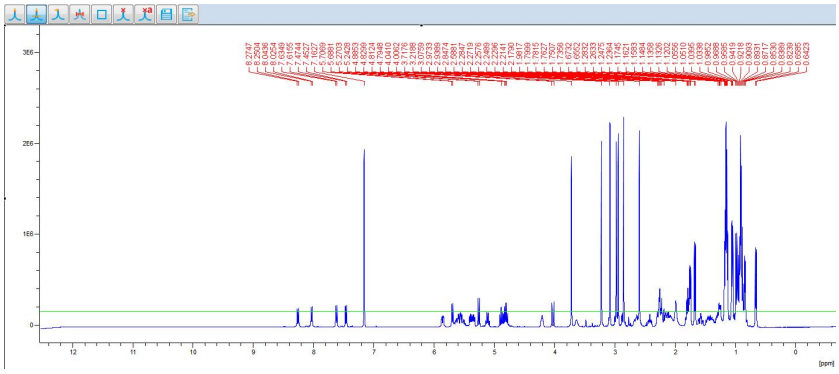


Figure 6.8 Spectrum with picking positive peaks

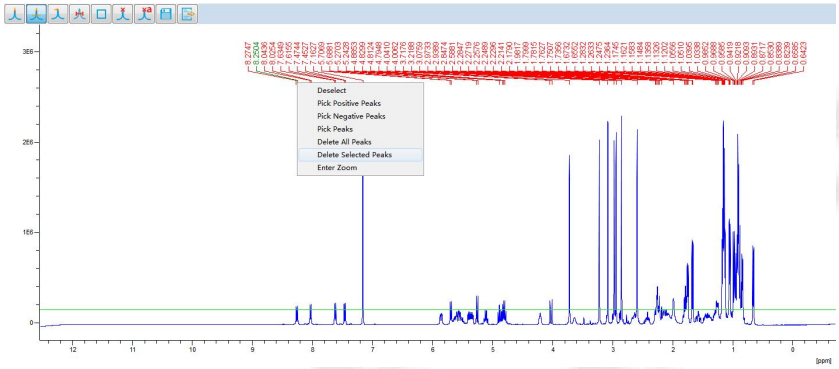


Figure 6.9 Right-click menu when there is peak information

2. Peak picking manually

Click the icon  in the peak picking toolbar to enter the "Pick peaks in selected ranges" mode. In this mode, the right click menu is shown in Figure 6.10. "Delete All Peaks" means to delete all peaks. "Delete Selected Peaks" means to delete the currently selected peaks. Click "Enter Zoom" to enter zoom mode to zoom the spectrum, click "Exit Zoom" to exit zoom mode.

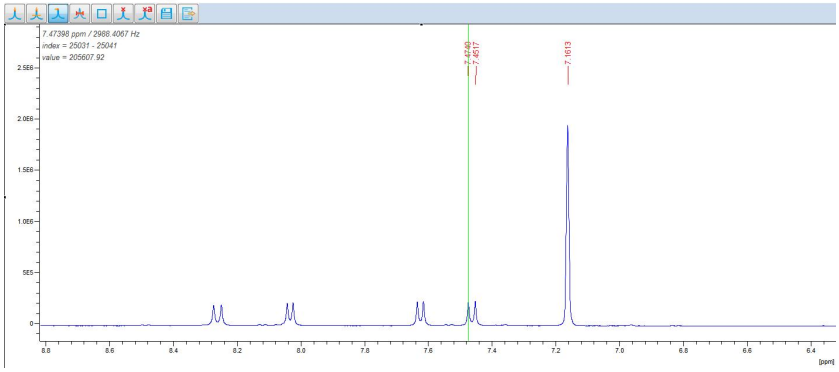


Figure 6.10 Peak picking manually

3. **Pick peaks in selected ranges**

Click the manual peak picking icon to enter the "Pick peaks in selected ranges" mode. Hold down the left mouse button and drag to form a square area (Figure 6.11). Release the left mouse button and the information label of all the peaks at the top of the peak will be displayed, as shown in Figure 6.12. In this mode, the right mouse button function is exactly the same as "peak picking manually" and you can refer to 2 **Peak picking manually**.

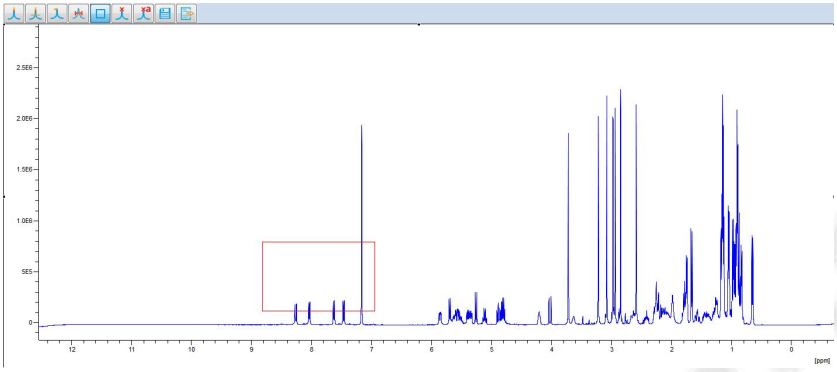


Figure 6.11 Select peak ranges in "Pick peaks in selected ranges" mode

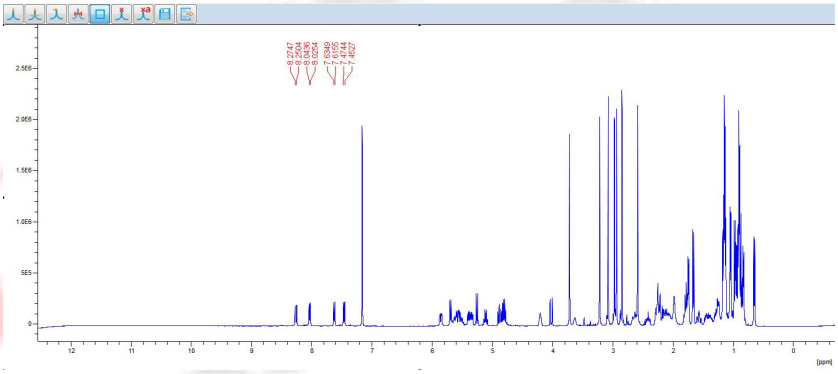


Figure 6.12 Spectrum after "Pick peaks in selected ranges"

6.2.3 Peak-picking via dialog

Type the command ***pp*** to open the peak-picking dialog, as shown in Figure 6.13.

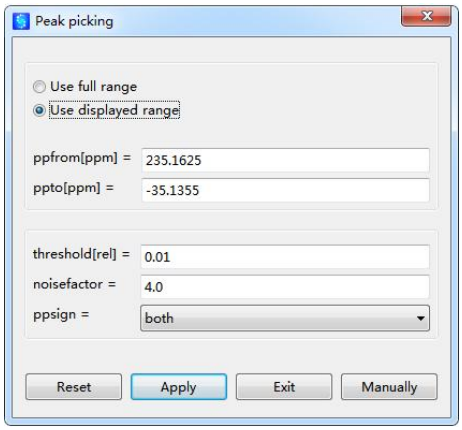


Figure 6.13 peak-picking dialog

Select “Use full range”, “ppfrom” and “ppto” will be set to the full range of the current spectrum. Select “Use displayed range ”, “ppfrom” and “ppto” will be set to the displayed range of the current spectrum. The user can also enter the value of “ppfrom” and “ppto” manually.

The product of “threshold” and the strongest peak is stored as the first threshold. The product of “noisefactor” and the noise level is stored as the second threshold. The larger of the two threshold values, will be used as the final peak threshold.

The value of “ppsign” has three choices: both, positive and negative. Respectively represent: pick both of the positive and negative peaks, pick the positive peaks only, pick the negative peaks only.

Click on **Reset** to reset the values in this dialog to the workspace active parameters. Click on **Apply** to pick peaks with the values in this dialog. Click on **Exit** to close this dialog. Click on **Manually** to enter the manual peak-picking mode.

6.3 Integration

Click on the menu **Analysis** to select **Integration** > **Manual**, or click on the icon ∫ in the toolbar to enter the manual integration mode. The integration toolbar will appear at the top left of the spectrum, as shown in Figure 6.14.

Figure 6.14 Integration toolbar

: Delete the selected integrals. First select the integrals you want to delete, then click this icon to clear the currently selected integration information.

: Delete all integrals. Click this icon to clear all the integrals displayed on the spectrum.

: Save and exit the integration mode. After saving the integration result, an "Integration" item will be generated on the right side of **Acqu** below the workspace. You can select a row or a column and right-click to copy or delete, or export the entire table data into .txt or .xls format file for post-processing.

: Exit the integration mode without saving the integration information.

A detailed description of other functions is as follows.

1. Add new integral manually

After entering the manual integration mode, place the mouse in the spectrum display area, at which point the mouse will become the integral line graph and a vertical red line will appear. Drag the red line to the appropriate position on the left side of the peak that needs to be integrated, press the left button and drag to the appropriate position on the right side of the peak, release the mouse button, and mark the corresponding integral line and integral value on the spectrum. Repeat the same operation to integrate the peaks that need to be integrated (Figure 6.15).

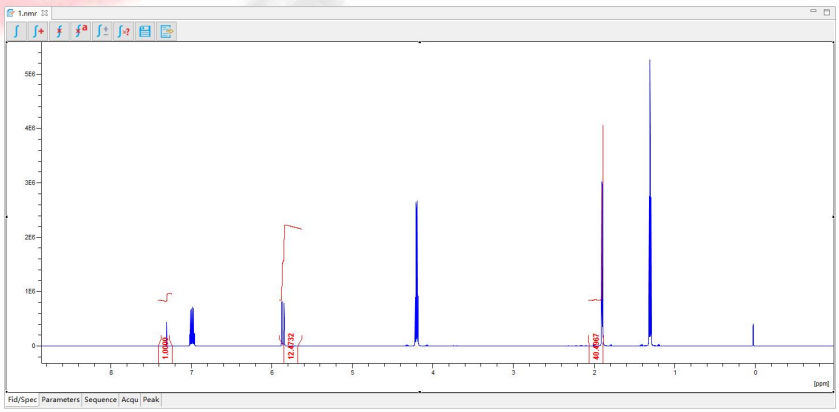


Figure 6.15 Schematic diagram of manual integration

2. Add new integral via dialog

Clicking the Integral toolbar button will pop up the Add Integral dialog shown in Figure 6.16. The value of the "Unit" drop-down box indicates the numerical unit in the text boxes "From" and "To". The default is ppm, and you can drop down to select others. In the "From" text box, enter the abscissa of the starting point, and enter the abscissa of the end of the point in the "To" text box. Click "OK" and the result will be displayed in the spectrum. Click "Cancel" to cancel the operation of adding new integral.

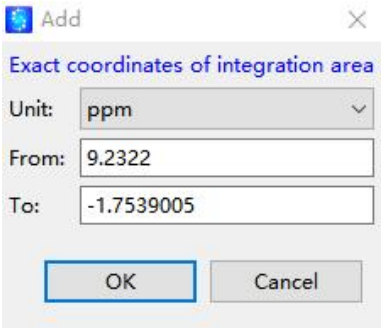


Figure 6.16 Add integral dialog

3. Expand by 1.15

Click the integration toolbar button to zoom in on the integral line level. Place the mouse on the icon and click the left or right button once to zoom in or out all the integration lines in the spectrum by 1.15 times, or use the scroll wheel to zoom.

4. Expand by X

Click the Integral toolbar button and the Zoom Integral dialog box will pop up, as shown in Figure 6.17. Enter the scaling factor in the "Scaling Factor" text box. After clicking "OK", the system will scale the integration line according to the input scale factor. Click "Cancel" to cancel the zoom.

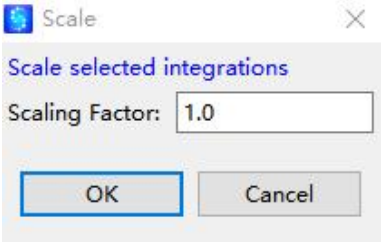


Figure 6.17 Integral scaling dialog

5. Delete integrals

Place the mouse on the information label position of the integral segment to be deleted. At this time, the number will turn green. Click the left mouse button and the color will turn dark green, indicating that the peak information to be deleted has been selected. Click the icon and the system will delete the currently selected integral segment. Right click and select “Delete selected integrals” from the right-click menu to get the same result. If you want to delete all the integrals, click on the icon or select “Delete all integrals” in the right-click menu (Figure 6.18).

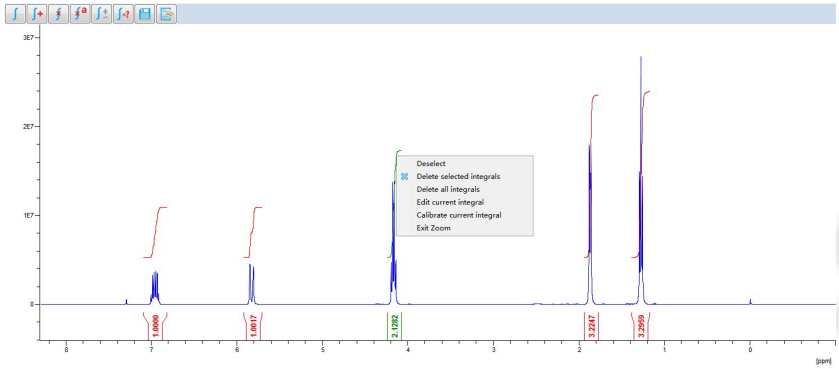


Figure 6.18 “Delete integrals” in right-click menu

6. Edit current integral

Select the integral segment as described above, and then select “Edit current integral” from the right-click menu, and a menu similar to Figure 6.16 will pop up. You can re-select the values of “From” and “To” to precisely adjust the integral segment.

7. Calibrate current integral

Select the integral segment to be used as the integral reference value as described above, and select “Calibrate Current Integral” from the right-click menu to bring up the dialog box (Figure 6.19). You can set the integral segment to the value of the nucleus it represents as a reference.

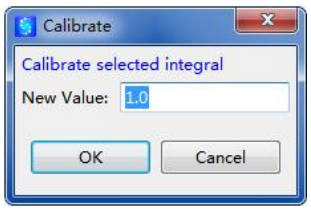


Figure 6.19 Dialog box for integral calibration

6.4 Multiple Spectrum Display

6.4.1 Multiple spectrum display

Open the spectrum data, click the **Multiple Spectrum Display** option under the menu **Analysis**, or click the system toolbar button to enter the multiple spectrum display mode. The spectrum of the current workspace is the reference spectrum, as shown in Figure 6.20.

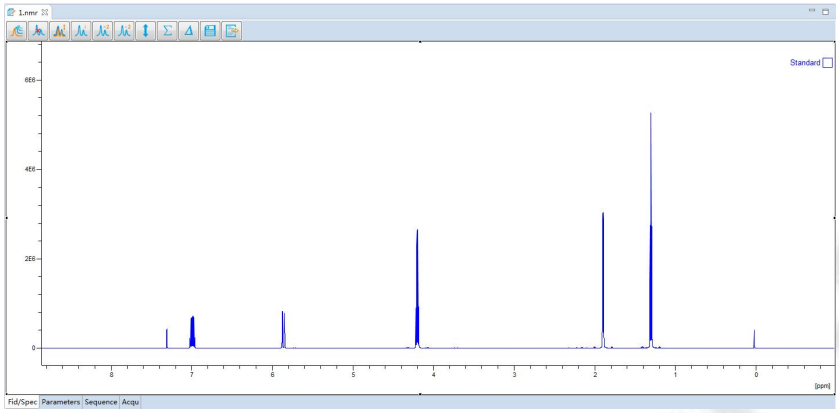


Figure 6.20 Multiple spectrum display interface

The multiple spectrum display toolbar at the top left is shown in Figure 6.21.

Figure 6.21 Multiple spectrum display toolbar

In “multiple spectrum display” mode, in the data navigation panel on the left, double-click the data you want to use, and the spectrum of the data will be displayed in the current workspace, together with the previous spectrum display. In the same way, you can continue to add spectrum data. It should be noted that the data in the multiple spectrum display area must be the same nucleus, but the acquisition parameters such as the spectrum width may be different, and the actual displayed area is based on the reference spectrum. Three spectra are displayed simultaneously in different colors as shown in Figure 6.22.

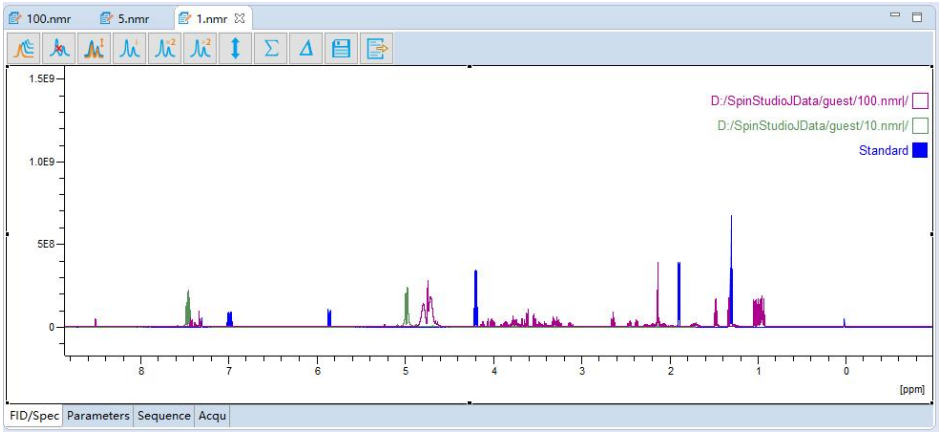


Figure 6.22 Schematic of multiple spectrum display

Note that the spectra are displayed in overlapping at this time. The upper right corner area displays the data path information of each spectrum in different colors. If the box on the right side is filled, it indicates that it is the current active spectrum, and some operations can be performed on it. Click the icon to switch between displaying/not displaying information.

If you choose the wrong one or no longer need one of the spectra, you can click on the corresponding path information area box, select the spectrum, and then click the icon to delete it. Note that the system cannot remove the base spectrum.

Click on the icon to change the spectrum from overlapping display to top and bottom (Figure 6.23). Click this icon repeatedly to switch between the two display modes.

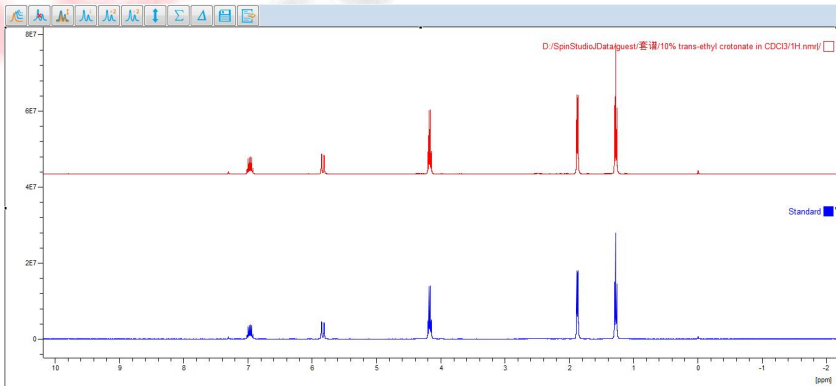


Figure 6.23 Spectra display separately

Click on the icon and respectively to zoom in or out twice the selected spectrum. If you place the mouse directly in the spectra display area and scroll the wheel, all selected

spectra will be zoomed in or out vertically.

Place the mouse over the icon and scroll the wheel to move the selected spectrum vertically.

6.4.2 Spectral addition and subtraction

In the “multiple spectrum display” mode, addition or subtraction of the spectrum can also be performed. In the process of addition and subtraction, always use the reference spectrum as one of them, then select another spectrum, click Σ, the system will add the reference spectrum to the selected spectrum, and display the added spectrum at the top (Figure 6.24).

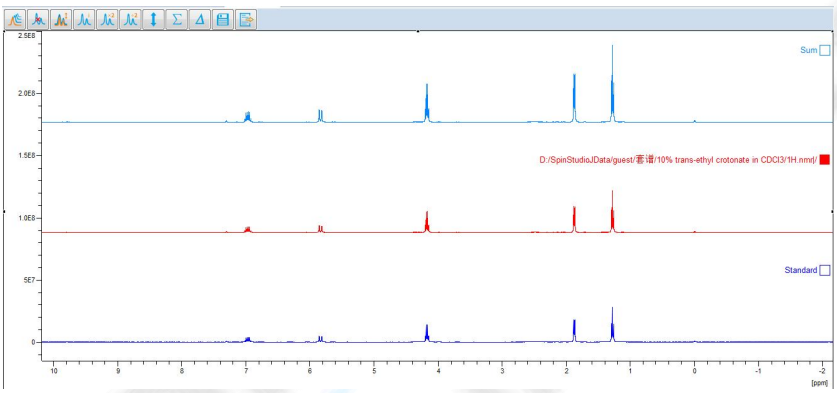


Figure 6.24 Adding spectra

If a spectral subtraction is to be performed, the reference spectrum is also used as the starting spectrum. Select another spectrum and click Δ, the system will subtract the selected spectrum from the reference spectrum and display the subtracted spectrum at the top (Figure 6.25).

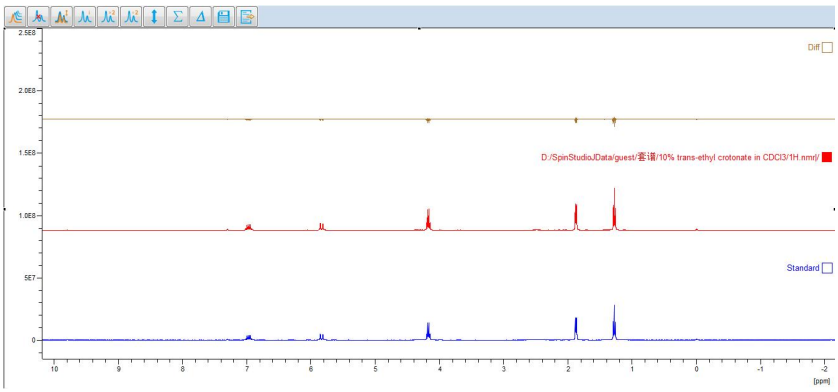


Figure 6.25 Spectrum subtraction

After completing the spectral addition and subtraction operation, clicking will pop up the dialog box shown in Figure 6.26 to save the result of the addition and subtraction of the spectrum. The user can change the data storage address or experiment name on the dialog box as needed.

Clicking does not save and exits the “multiple spectrum display” mode directly.

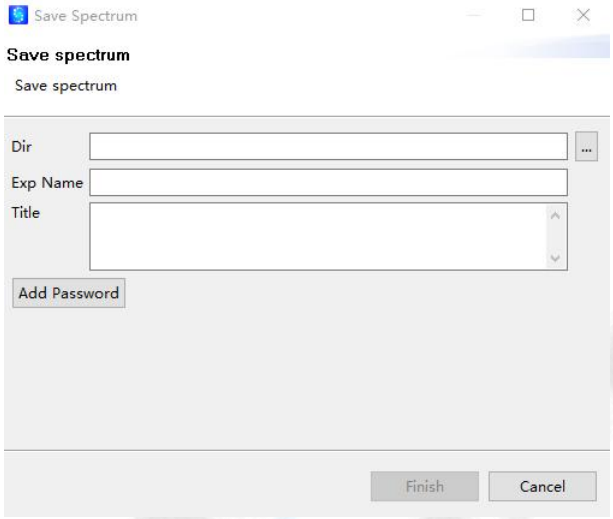


Figure 6.26 Save spectrum addition and subtraction results

6.5 Array Data Display and Processing

Array data is a set of values that are set during the experiment. In the experiment, one of these array values is taken to perform an experiment, and obtain a set of experiment data.

Before the experiment, some experiment parameters are set to a set of values (such as setting the pulse width p1 to a series of values to find the 90-degree pulse width). In the experiment, one of these parameter array values is taken to perform experiments to obtain a set of experimental data, the array data is formed.

6.5.1 Array data display

The display of array data in the workspace is in the form of multiple spectra arrangements. Multiple data can be arranged horizontally or vertically by clicking the

Horizontal or **Vertical** option under the menu bar **View** > **Layout** (Figure 6.27).

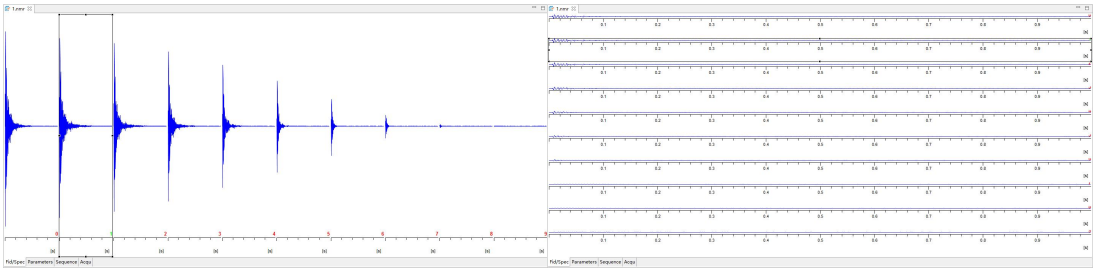


Figure 6.27 Horizontal (left) and vertical (right) display of array FID

Clicking on the **Stacked** option under the menu bar **View** > **Layout** will cause the data to be stacked in a certain degree of inclination. A window pops up, allowing the user to set the horizontal misalignment value for the stack display (Figure 6.30). The data displayed after setting will be displayed in a stack according to the horizontal misalignment between each data (Figure 6.31).

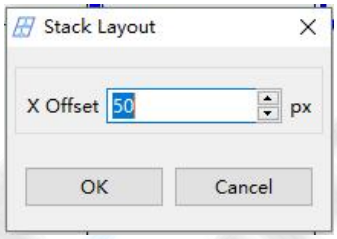


Figure 6.28 Horizontal misalignment value when stacked

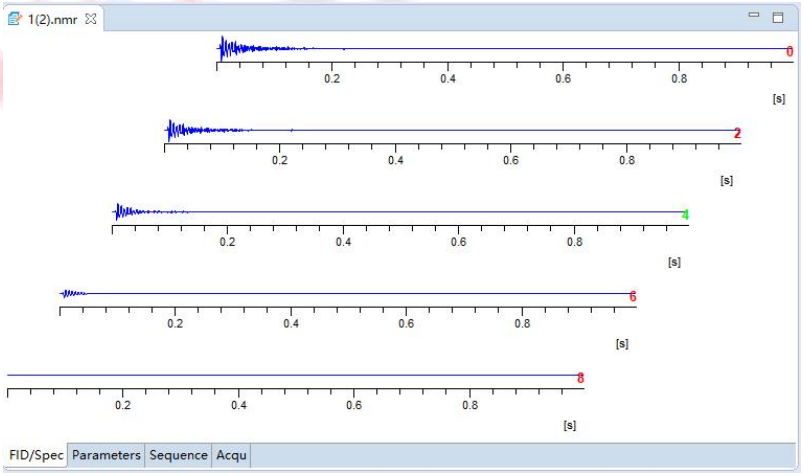


Figure 6.29 Stack display of array data

Clicking on the menu has the option to get more array data images. You can click on the

menu bar **View** to select **Trace Manager**, or click the toolbar icon . The array data selection panel shown in Figure 6.30 will pop up on the right side of the spectrum. Figure 6.31 shows the functions of the various buttons on the toolbar above the array data selection panel.

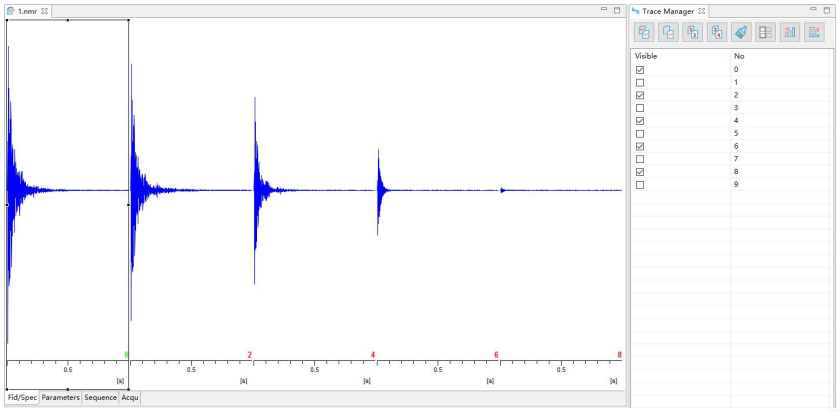


Figure 6.30 Array data selection panel

Figure 6.31 Array manager toolbar

Any data in the array data can be displayed in the workspace by using the function buttons of the array data selection panel. Users can selectively display certain data by checking the way. Clicking on the icon will display the selection in turn, and clicking the icon will display all the data. You can also click on the icon or to display odd or even digit data. Clicking on the icon will bring up a window that lets you select the starting and ending range of the data you want to display (Figure 6.32). Clicking on the icon or will cause the data to be displayed horizontally or horizontally (as shown in Figure 6.27 above).

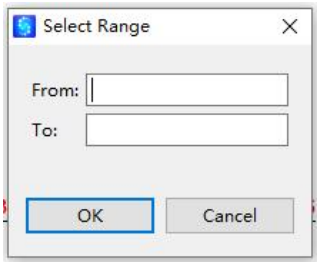


Figure 6.32 Select array spectrum display range

In the array spectrum display mode, use the left mouse button to click on any spectrum. At this time, the number of the spectrum becomes green, and the user can change the display range, height, peak picking, and integral operation of the spectrum. Click the format brush icon to apply the results of the previous actions to all spectra simultaneously.

Fourier transform of the array FID will result in an array spectrum, and the array spectrum can also perform the operations described in this section.

6.5.2 Array data processing

To perform array data processing, you need to activate a current data (the spectrum with the green number in the lower right corner is active). The data is processed first, and other data requires the same operation to use the format brush button . Figure 6.33 shows the results of the Fourier transform and phase correction of the array FID in Figure 6.27.

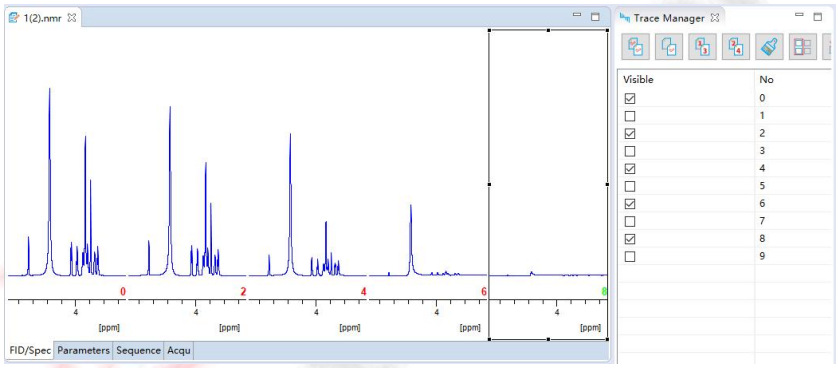


Figure 6.33 Array data activation display

If you need to do peak picking or integration, the format brush can't meet the requirements of applying to other data. You need to enter the peak/integration mode. After completing the peak/integration operation, click the icon on the peak/integration toolbar to apply the result to all the spectra (Figure 6.34).

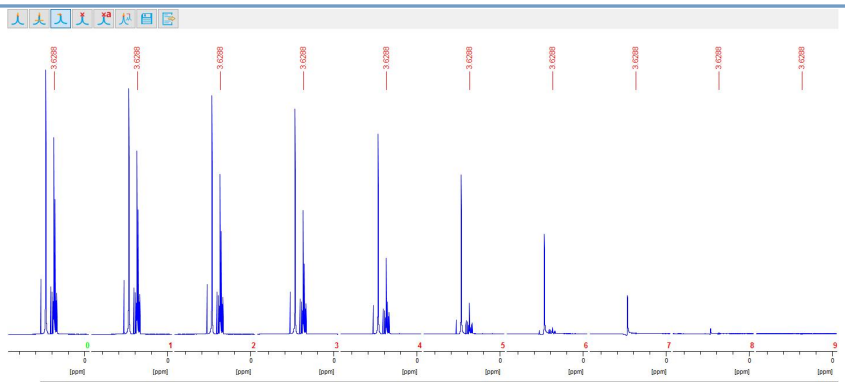


Figure 6.34 Array data peaking display

6.6 Fitting

Open the array spectrum data, and then click the **Fitting** option under the **Analysis** menu to enter the fitting interface, as shown in Figure 6.35.

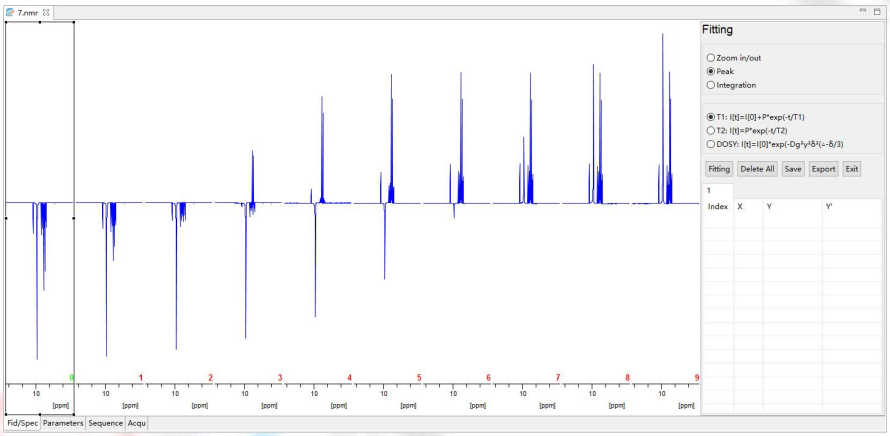


Figure 6.35 Fitting interface

Zoom in/out: Spectral scaling. Select this option to scale the spectrum horizontally. You can select the signal you want to fit to scale (scaling a signal or all signals in a certain area).

Peak: Select this option to perform peak-picking operations.

Integration: Select this option to perform integration operations.

T1: Select this option to perform T1 relaxation fitting.

T2: Select this option to perform T2 relaxation fitting.

DOSY: Select this option to perform DOSY fitting.

6.6.1 T1 relaxation fitting

Open the T1 array spectra, first adjust the phase of the last spectrum to pure absorption through the Trace Manager tool, and then apply the same phase to the other sub-spectra. Click the **Fitting** option under the **Analysis** menu, the system will enter the data fitting page, and various options will be displayed on the right side of the workspace, as shown in Figure 6.35. Through the data fitting interface, the user can complete the fitting of T1, T2 or DOSY data.

Use the Trace Manager tool to select the last spectrum and check Zoom in/out to enlarge the spectrum to the appropriate range and height. Check Peak (or Integration, the same operation) to enter peak picking mode. Hold down the left mouse button and drag to select a region, the system will automatically mark the chemical shift value of the strongest signal peak in the region (only one chemical peak value of the signal peak can be marked at a time), and then select the desired peak of the spectrum, as shown in Figure 6.36. If you want to delete the selected peak, click the button Delete All in the dialog box.

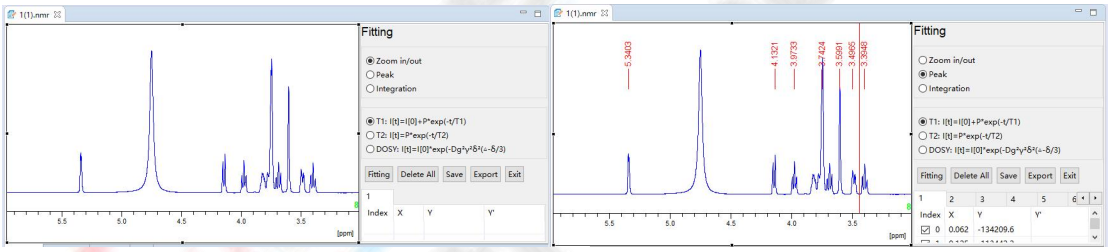


Figure 6.36 Amplification and peak picking in the fitting interface

Peaking results will work for all spectra. At this point you can operate the Trace Manager to display all the spectra (Figure 6.37), or still only show one spectrum, and the result is the same.

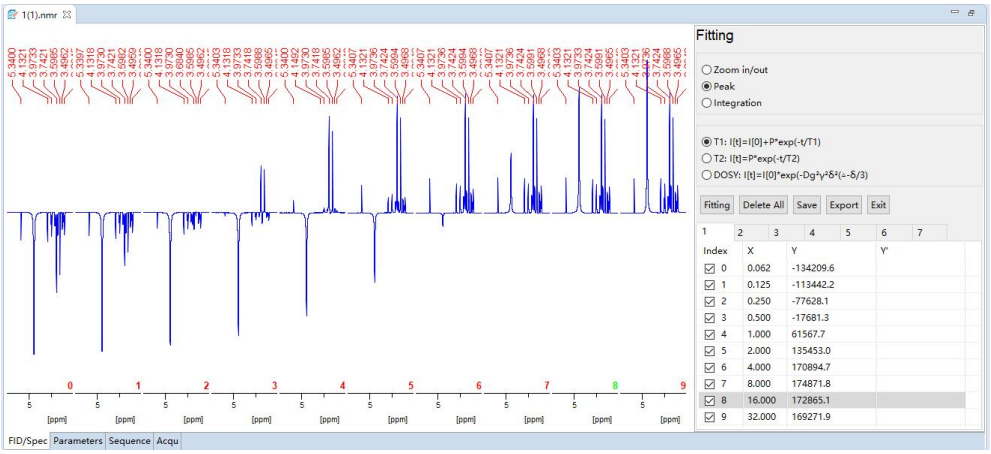


Figure 6.37 Show all spectra after peak picking

Select T1: I[t]=I[0]+P*exp(-t/T1), click the button Fitting, the system will automatically calculate and draw the T1 fit curve, as shown in Figure 6.38. Click the button Save, the system will save the peak information obtained by the peak picking as a node to the current data directory, the node name is "peaks". Click the button Export, the system can save the result of T1 relaxation fitting to text format and export (Figure 6.39), which can be used for other software for processing and analysis. Finally, click the button Exit to exit the fitting mode.

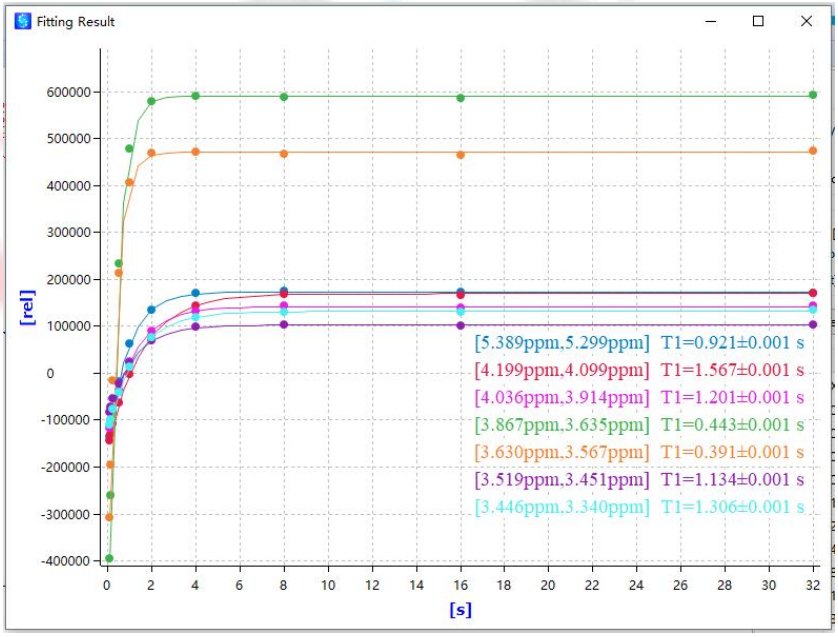
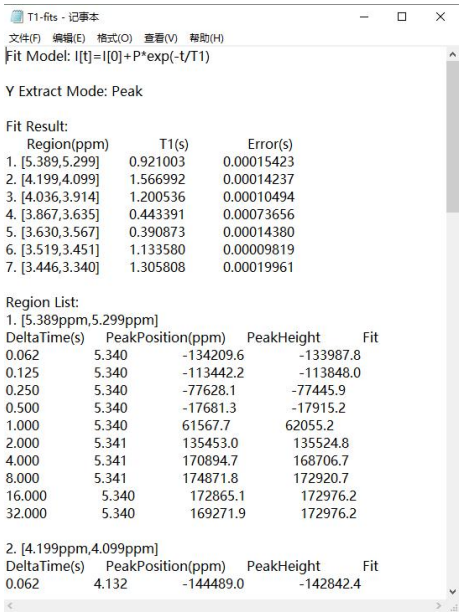


Figure 6.38 T1 fitting curve



T1-fits - 记事本

文件(F) 编辑(E) 格式(O) 查看(V) 帮助(H)

Fit Model: I[t]=I[0]+P*exp(-t/T1)

Y Extract Mode: Peak

Fit Result:

Region(ppm)	T1(s)	Error(s)
1. [5.389,5.299]	0.921003	0.00015423
2. [4.199,4.099]	1.566992	0.00014237
3. [4.036,3.914]	1.200536	0.00010494
4. [3.867,3.635]	0.443391	0.00073656
5. [3.630,3.567]	0.390873	0.00014380
6. [3.519,3.451]	1.133580	0.00009819
7. [3.446,3.340]	1.305808	0.00019961

Region List:

1. [5.389ppm,5.299ppm]

DeltaTime(s)	PeakPosition(ppm)	PeakHeight	Fit
0.062	5.340	-134209.6	-133987.8
0.125	5.340	-113442.2	-113848.0
0.250	5.340	-77628.1	-77445.9
0.500	5.340	-17681.3	-17915.2
1.000	5.340	61567.7	62055.2
2.000	5.341	135453.0	135524.8
4.000	5.341	170894.7	168706.7
8.000	5.341	174871.8	172920.7
16.000	5.340	172865.1	172976.2
32.000	5.340	169271.9	172976.2

2. [4.199ppm,4.099ppm]

DeltaTime(s)	PeakPosition(ppm)	PeakHeight	Fit
0.062	4.132	-144489.0	-142842.4

Figure 6.39 T1 fitting result output file

6.6.2 T2 relaxation fitting

The T2 relaxation fitting process is the same as the T1 relaxation fitting process, but the first sub-spectrum is selected to perform peak picking/integration on the signal to be fitted, then select T2: I[t]=P*exp(-t/T2) and click Fitting to fit. The fitting process and results are shown in Figure 6.40 and Figure 6.41, respectively.

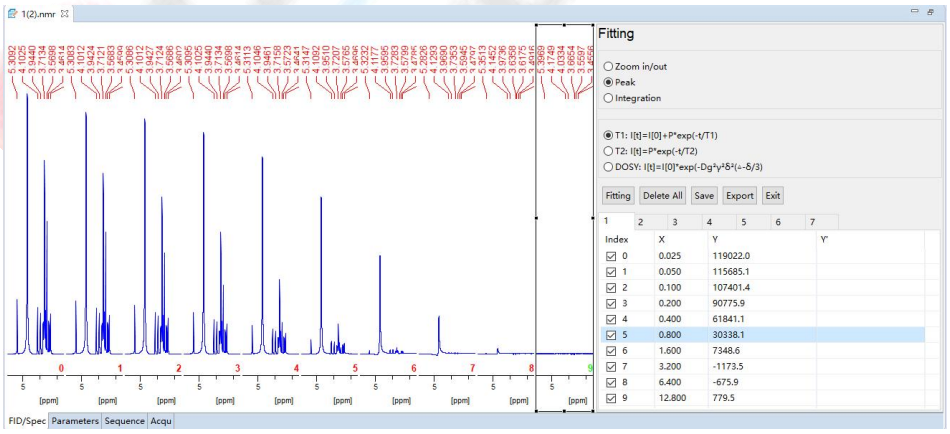


Figure 6.40 T2 relaxation fitting interface

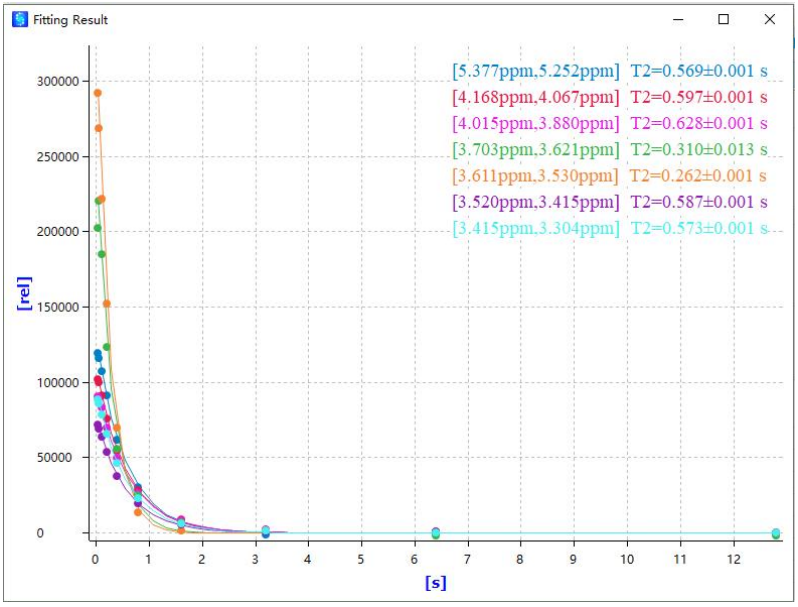


Figure 6.41 T2 fitting curve

6.6.3 DOSY fitting

The DOSY fitting process is the same as the T2 relaxation fitting, the fitting process is shown in Figure 6.42. Select the first sub-spectrum to pick peaks for the signal to be fitted, then select DOSY: I[t]=I[0]*exp(-Dg²γ²δ²(Δ-δ/3)) and click the button Fitting to fit. At this point, the box will pop up to select the fitting result display mode(Figure 6.43). Select **Standard** and the fit curve results are shown in Figure 6.44, select **2D Spectrum** and the results are shown in Figure 6.45.

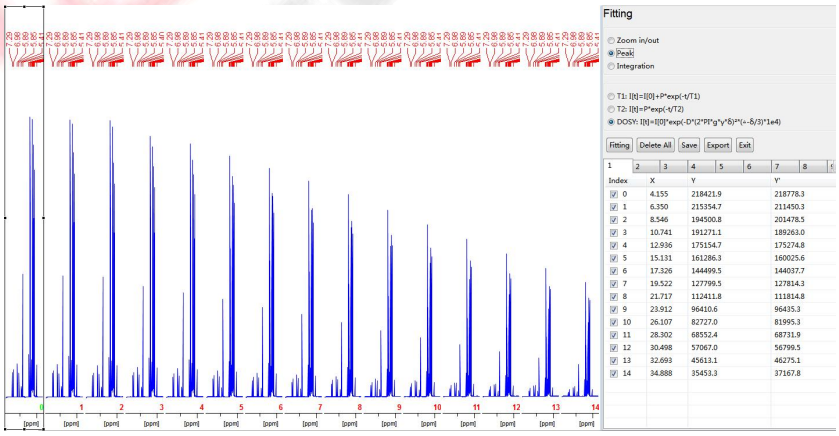


Figure 6.42 Peak picking for signals that need to fit

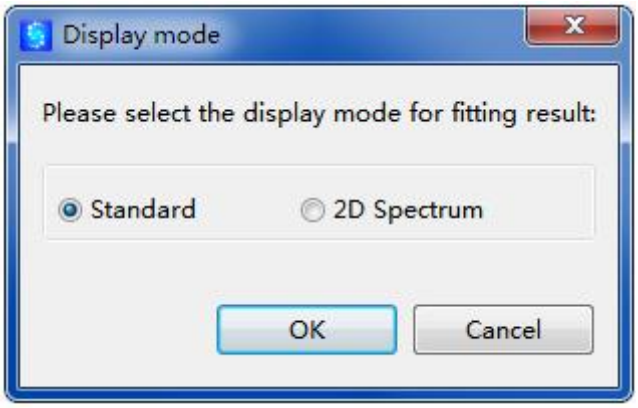


Figure 6.43 Selection box for fit result display mode

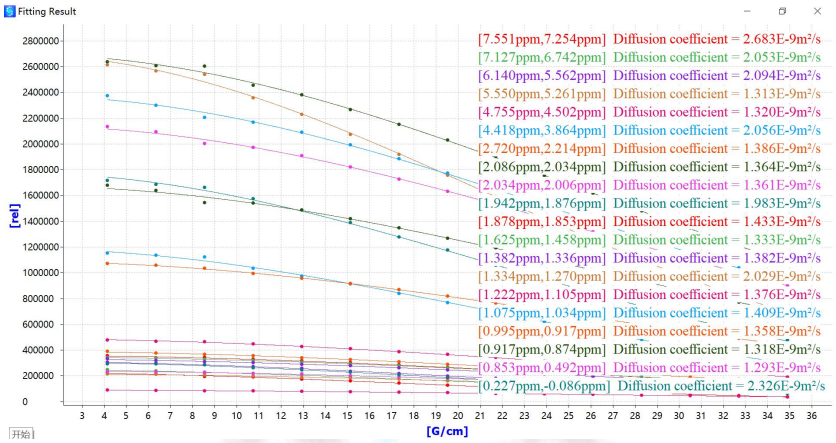


Figure 6.44 DOSY fitting curve

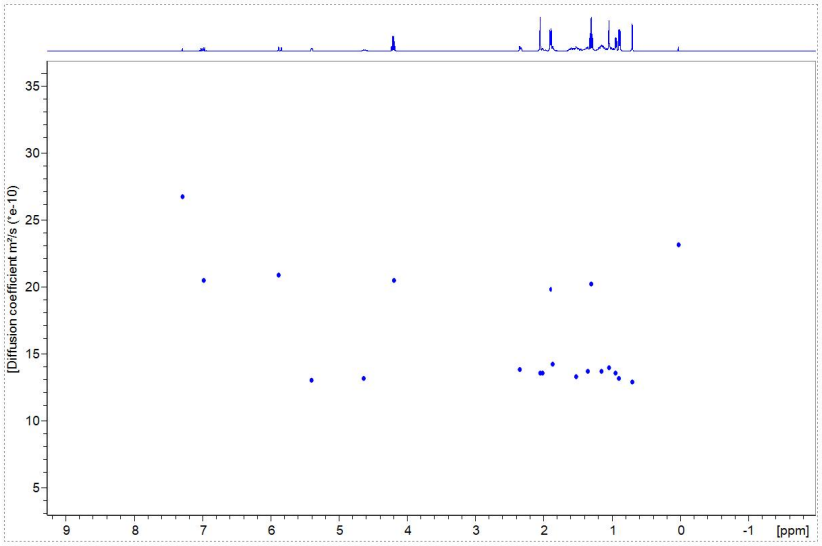


Figure 6.45 2D spectral display for DOSY fitting

6.7 Signal/Noise Ratio

This function is to calculate the signal to noise ratio of the currently displayed spectrum. Click the **Signal/Noise Ratio** option under the **Analysis** menu, or enter the command ***dsn*** in the command bar to enter the SNR calculation mode (Figure 6.46) . You can drag the mouse to select the calculated signal width and noise width respectively (Figure 6.47) , then right click and select “Start calculate SNR” (Figure 6.48) , the calculation result will pop up (Figure 6.49).

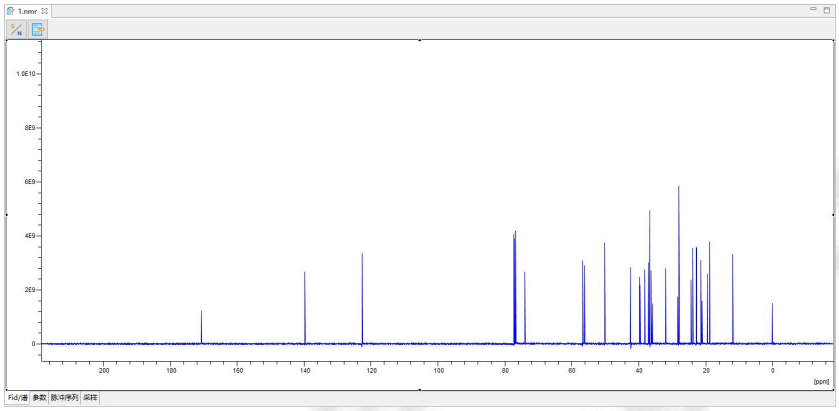


Figure 6.46 Signal/noise ratio calculation interface

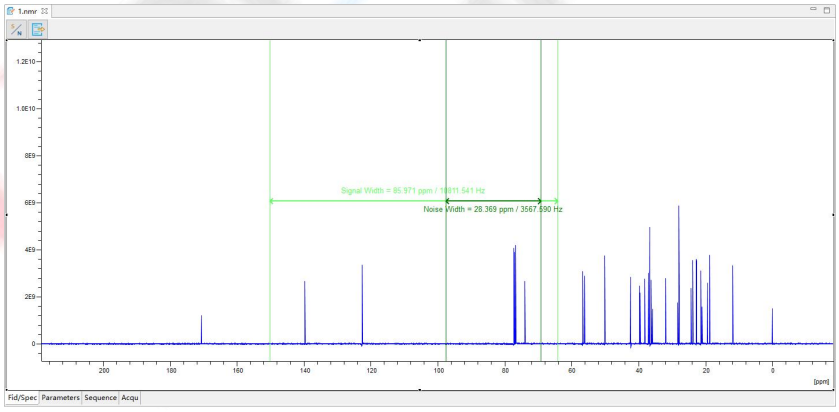


Figure 6.47 Select signal width and noise width

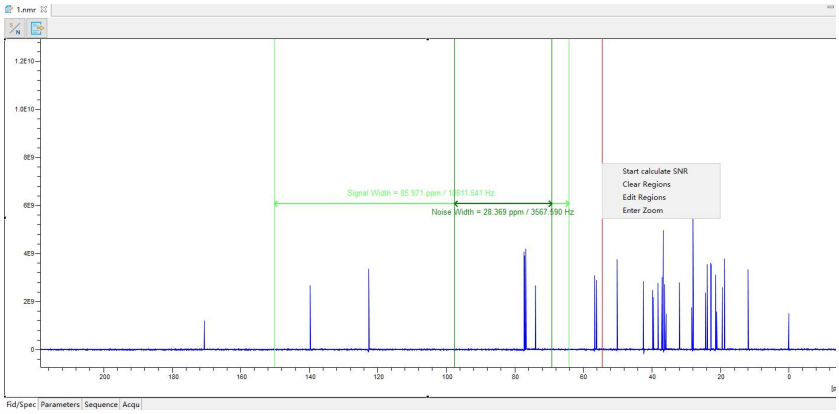


Figure 6.48 Right-click menu in SNR calculation mode

Figure 6.49 Signal to noise ratio calculation result

"Clear Regions" can also be selected through the right-click menu to clear the currently selected signal and noise range. When "Edit Regions" is selected, the system will pop up the dialog box shown in Figure 6.50, which can input the start and end points of the signal and noise and the noise width respectively. When "Enter Zoom" is selected, it will enter the zoom mode, and the horizontal zoom of the spectrum can be performed. Right click on "Exit Zoom" to exit zoom mode.

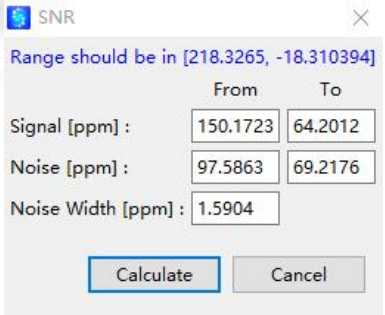


Figure 6.50 Edit dialog for signal to noise ratio calculation range

The signal/noise ratio of the currently displayed spectrum can be calculated using the command ***dsnmax*** or ***dsnmax(integer)***. Entering the command ***dsnmax*** will pop up the

calculation result directly, as shown in Figure 6.51. The command ***dsnmax*** is equivalent to ***dsnmax(200)***, ie the signal-to-noise ratio of the noise interval is 200 Hz, while ***dsnmax (1400)*** calculates the signal-to-noise ratio of the noise interval in the range of 1400 Hz.

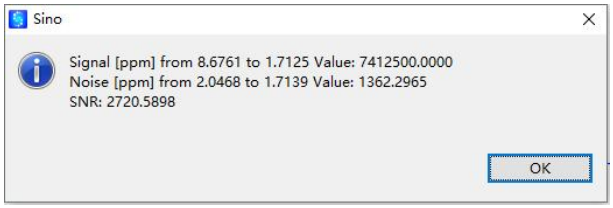


Figure 6.51 Signal to noise ratio results obtained by ***dsnmax***

6.8 Line Width and Digital Resolution

Open the experiment data in the workspace, click the **Resolution** option under the **Analysis** menu, or enter the command ***res*** in the command line to calculate the peak width of 50%, 0.55%, 0.11% of the peak intensity of the current display range, and the spectral digital resolution. The result will be displayed in the log line, as shown in Figure 6.52.

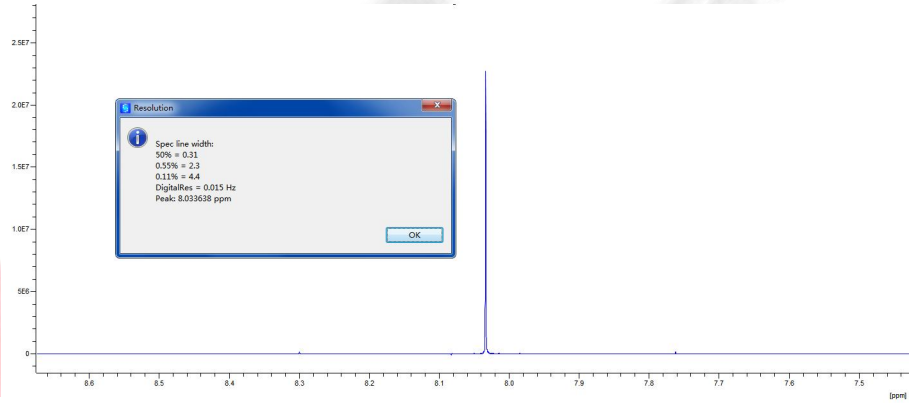


Figure 6.52 Line width and digital resolution

Chapter 7 1D NMR Experiments

7.1 1D ^{1}H Experiment

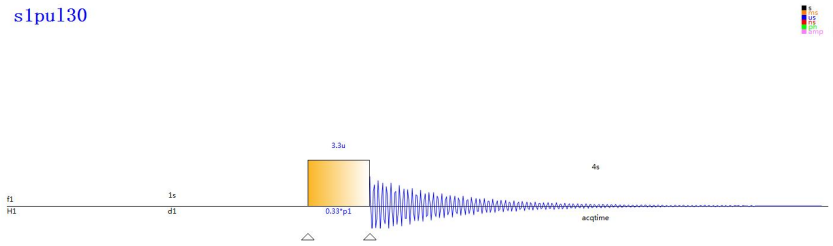


Figure 7.1 Pulse sequence of 1D ^{1}H experiment

1. Phase Cycle
   - ph1: 0 90 180 270
   - ph2: 0 90 180 270
2. Experiment Parameters
   - sw: Spectral width is around 12 ppm
   - gain: Receiver gain, set appropriate value to avoid overflow
   - frqb: Basic frequency of observed channel (^{1}H)
   - frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
   - d1: Relaxation delay 1 s (d1+acqtime=1~5 times T1)
   - p1: 90 degree pulse width of observed channel (^{1}H)
   - plvl1: Power level of pulse in observed channel (^{1}H)
   - acqtime: Acquisition time is around 3.5 s (np in the 32k point range)
   - ns: Number of the scans is 8, recommended in multiples of 4
3. Processing

Set si greater than np, generally 2 to the n which is similar to 2np, such as np=16384, you can set si=32768, or set si=65536 for higher digital resolution, and then perform Fourier transform. You can set si=32000 or si=64000, the program will automatically change it to

32768 or 65536. Phase correction is performed on the transformed spectrum to make the spectrum a purely absorptive signal. Eliminate baseline distortion by baseline correction. After scaling the peak, the peaks of interest are integrated and finally the spectrum is output.

7.2 1D ^{13}C Experiment

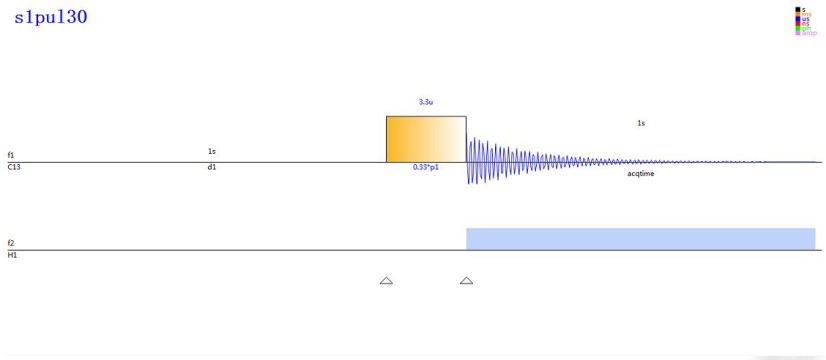


Figure 7.2 Pulse sequence of 1D ^{13}C experiment

1. Phase Cycle

- ph1: 0 90 180 270
- ph2: 0 90 180 270

2. Experiment Parameters

- sw: Spectral width is around 250 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{13}C)
- frqohz: Frequency offset of observed channel (^{13}C), the center of the spectrum
- p1: 90 degree pulse width of observed channel (^{13}C)
- plvl1: Power level of pulse in observed channel (^{13}C)
- d1: Relaxation delay 1 s
- frqb1: Basic frequency of decoupling channel (^{1}H)
- frqohz1: Frequency offset of decoupling channel (^{1}H), the center of the spectrum (^{1}H)
- decon1: ^{1}H decoupling section setting, usually "yyy", ie full decoupling, also "nny", ie sampling period decoupling
- decpw1: 90 degree pulse width of decoupling channel (^{1}H) is about 100μs

● dectype1: Decoupling mode of decoupling channel (^{1}H) is "www" (using waltz16 decoupling sequences)

● decplvl1: Power level of decoupling channel (^{1}H) is about 37 dB

● acqtime: Acquisition time is around 0.6 s

● ns: Number of the scans, depending on the sample concentration

● lb: Lorentzian broadening factor for exponential window multiplication, 2Hz is recommended

3. Processing

Set si greater than np, generally 2 to the n which is similar to 2np, such as np=16384, you can set si=32768, or set si=65536 for higher digital resolution, and then perform Fourier transform. You can set si=32000 or si=64000, the program will automatically change it to 32768 or 65536. Phase correction is performed on the transformed spectrum to make the spectrum a purely absorptive signal. Eliminate baseline distortion by baseline correction. The spectrum is output after peak picking.

7.3 ^{1}H T1 Relaxation

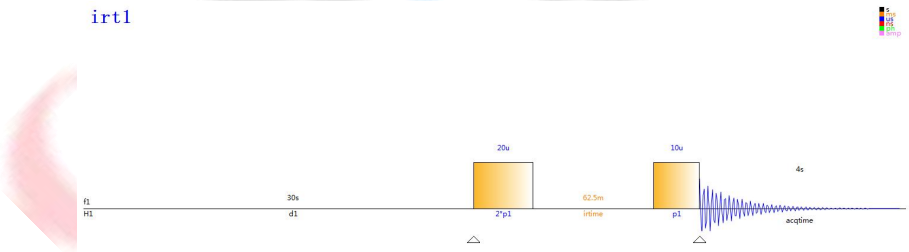


Figure 7.3 Pulse sequence of ^{1}H T1 Relaxation measurements (inversion-recovery)

1. Phase Cycle

● ph1: 0

● ph2: 0 180 90 270

● ph3: 0 180 90 270

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- d1: Relaxation delay is 20 s (＞5 times T1)
- irtime: Inversion-recovery time, set values as follows in array mode, [0.0625, 0.125, 0.25, 0.5, 1.0, 2.0, 4.0, 8.0, 16.0, 32.0]
- acqtime: Acquisition time is around 4 s
- ns: Number of the scans is in multiples of 4

3. Processing

Pretreatment can refer to hydrogen spectrum, post-treatment see 6.6.1 T1 relaxation fitting.

7.4 ^{1}H T2 Relaxation

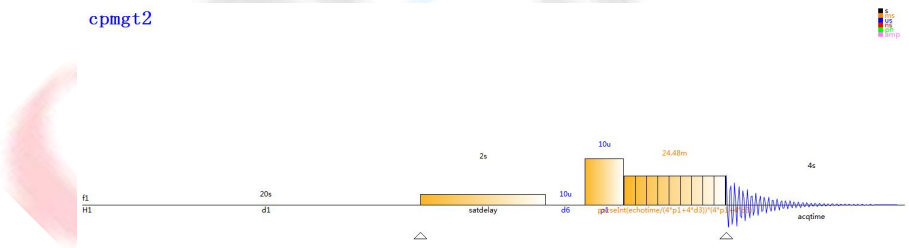


Figure 7.4 Pulse sequence of ^{1}H T2 Relaxation measurements (CPMG) with solvent presaturation

1. Phase Cycle

- ph1: 0 90 180 270
- ph2: 0
- ph3: 90 180

- ph4: 0 90 180 270

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- d1: Relaxation delay is 20 s (＞5 times T_1)
- d6: delay for power switching is 10μs
- echotime: Echo time, set values as follows in array mode, [0.025, 0.1, 0.4, 0.8, 1.6, 3.2, 6.4, 12.8, 25.6, 52.0]
- d3: Fixed echo time is about 0.00035 s, d3 should be << 1/J, but > (100 * p1)
- satdelay: Delay for presaturation is about 4 s (if presaturation is used)
- satplvl: Power level for presaturation is about 3 dB (if presaturation is used)
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center
- acqtime: Acquisition time is around 4 s
- ns: Number of the scans is in multiples of 4

3. Processing

Pretreatment can refer to hydrogen spectrum, post-treatment see 6.6.2 T2 relaxation fitting.

7.5 APT (Attached Proton Test)

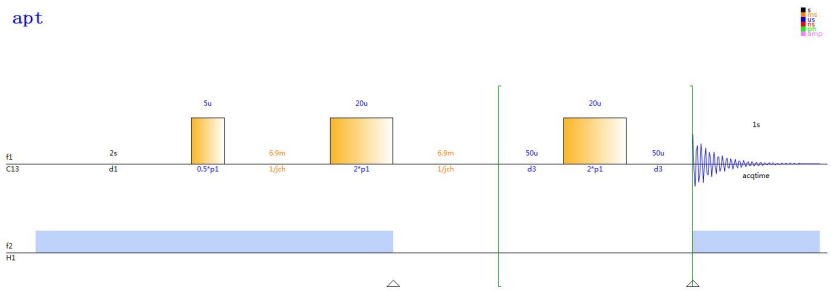


Figure 7.5 Pulse sequence of APT

1. Phase Cycle

- ph1: 0 0 180 180 90 90 270 270
- ph2: 90 90 270 270
- ph3: 90 270 90 270

2. Experiment Parameters

- sw: Spectral width is around 250 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{13}C)
- frqohz: Frequency offset of observed channel (^{13}C), the center of the spectrum
- frqb1: Basic frequency of decoupling channel (^{1}H)
- frqohz1: Frequency offset of decoupling channel (^{1}H), the center of the spectrum (^{1}H)
- d1: Relaxation delay is about 2 s
- jch: 145 Hz (CH one-bond scalar coupling constant)
- d3: Delay of the second echo is about 50 μs
- p1: 90 degree pulse width of observed channel (^{13}C)
- plvl1: Power level of pulse in observed channel (^{13}C)
- decon1: Stage of decoupling channel (^{1}H) is "nny", resulting ^{1}H decoupling in the acquisition
- decpw1: 90 degree pulse width of decoupling channel (^{1}H) is 100 μs
- dectype1: Decoupling mode of decoupling channel (^{1}H) is "www" (using waltz16

decoupling sequences)

- decplvl1: Power level of decoupling channel (^{1}H) is about 37 dB
- acqtime: Acquisition time is around 1 s
- ns: Number of the scans is in multiples of 8, depending on the sample concentration
- lb: Lorentzian broadening factor for exponential window multiplication, 2Hz is recommended

3. Processing

Set si greater than np, and then perform Fourier transform. The specific method is the same as the 7.2 1D ^{13}C Experiment.

7.6 DEPT (Distortionless Enhancement by Polarization Transfer)

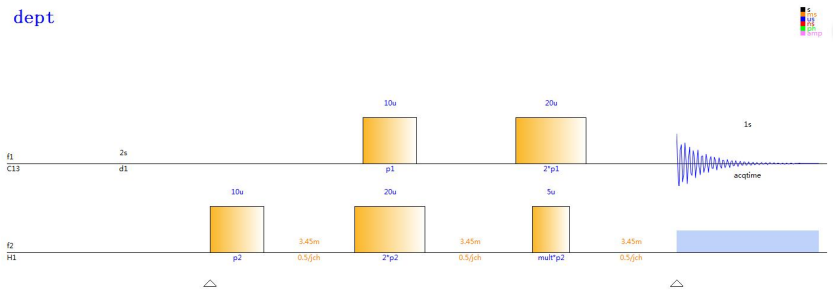


Figure 7.6 Pulse sequence of DEPT

1. Phase Cycle

- ph1: 0
- ph2: 0 180 90 270
- ph3: $(90)_4$ $(270)_4$
- ph4: $(0)_8$ $(90)_8$ $(180)_8$ $(270)_8$
- ph5: $(0\ 180)_4$ $(90\ 270)_4$
- ph6: $(90)_2$ $(270)_2$ $(180)_2$ $(0)_4$ $(180)_2$ $(270)_2$ $(90)_4$ $(270)_2$ $(0)_2$ $(180)_4$ $(0)_2$

2. Experiment Parameters

- sw: Spectral width is around 250 ppm
- gain: Receiver gain, set appropriate value to avoid overflow

● frqb: Basic frequency of observed channel (^{13}C)

● frqohz: Frequency offset of observed channel (^{13}C), the center of the spectrum

● p1: 90 degree pulse width of observed channel (^{13}C)

● plvl1: Power level of pulse in observed channel (^{13}C)

● p2: 90 degree high power pulse width of decoupling channel (^{1}H)

● plvl2: Power level of pulse in decoupling channel (^{1}H)

● d1: Relaxation delay is about 2 s

● jch: 145 Hz (CH one-bond scalar coupling constant)

● mult: Multiple of p2, set values as [0.5,1.0,1.5] in array mode, where 0.5, 1.0, 1.5 represent DEPT45, DEPT90 and DEPT135

● frqb1: Basic frequency of decoupling channel (^{1}H)

● frqohz1: Frequency offset of decoupling channel (^{1}H), the center of the spectrum (^{1}H)

● decon1: Stage of decoupling channel (^{1}H) is "nny", resulting ^{1}H decoupling in the acquisition

● decpw1: 90 degree pulse width of decoupling channel (^{1}H) is 63μs

● dectype1: Decoupling mode of decoupling channel (^{1}H) is "www" (using waltz16 decoupling sequences)

● decplvl1: Power level of decoupling channel (^{1}H) is about 37 dB

● acqtime: Acquisition time is around 1 s

● ns: Number of the scans is in multiples of 32, depending on the sample concentration

● dummyscan: Number of scans with no acquisition, 8

● lb: Lorentzian broadening factor for exponential window multiplication, 2Hz is recommended

3. Processing

(1) Perform Fourier transform to the array data (FID) then do phase correction to the first spectrum .

(2) Type "***deptproc***".

(3) Obtain 4 spectra representing CH_3, CH_2, CH and all proton attached carbon (CH_3 and CH_2 and CH).

7.7 DEPTQ (Distortionless Enhancement by Polarization Transfer with Quaternary carbon)

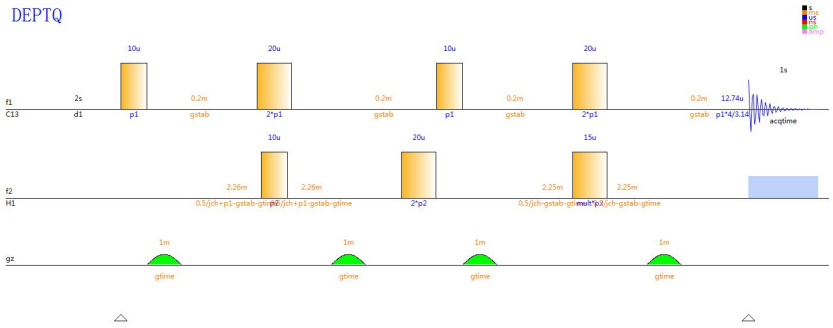


Figure 7.7 Pulse sequence of DEPTQ

1. Phase Cycle
   - ph1: 0
   - ph2: 270
   - ph3: 0 180 90 270
   - ph4: 90
   - ph5: 0 180
   - ph6: 90 90 270 270
2. Experiment Parameters
   - sw: Spectral width is around 250 ppm
   - gain: Receiver gain, set appropriate value to avoid overflow
   - frqb: Basic frequency of observed channel (^{13}C)
   - frqohz: Frequency offset of observed channel (^{13}C), the center of the spectrum
   - p1: 90 degree pulse width of observed channel (^{13}C)
   - plvl1: Power level of pulse in observed channel (^{13}C)
   - p2: 90 degree high power pulse width of decoupling channel (^{1}H)
   - plvl2: Power level of pulse in decoupling channel (^{1}H)
   - d1: Relaxation delay is about 2 s
   - gzlevel: Amplitude of z-gradient, set values as about 50%
   - gtime: Gradient time is 1.0 ~2.0ms

● gshape: Gradient pulse shape is hsine.grd

● gstab: Delay for gradient recovery is 0.1~0.2 ms

● jch: 145 Hz (CH one-bond scalar coupling constant)

● mult: Multiple of p2, set values as [0.5,1.0,1.5] in array mode, where 0.5, 1.0, 1.5 represent DEPT45, DEPT90 and DEPT135

● frqb1: Basic frequency of decoupling channel (^{1}H)

● frqohz1: Frequency offset of decoupling channel (^{1}H), the center of the spectrum (^{1}H)

● decon1: Stage of decoupling channel (^{1}H) is "nny", resulting ^{1}H decoupling in the acquisition

● decpw1: 90 degree pulse width of decoupling channel (^{1}H) is 63μs

● dectype1: Decoupling mode of decoupling channel (^{1}H) is "www" (using waltz16 decoupling sequences)

● decplvl1: Power level of decoupling channel (^{1}H) is about 37 dB

● acqtime: Acquisition time is around 1 s

● ns: Number of the scans is in multiples of 32, depending on the sample concentration

● dummyscan: Number of scans with no acquisition, 8

● lb: Lorentzian broadening factor for exponential window multiplication, 2Hz is recommended

3. Processing

Set si greater than np, and then perform Fourier transform. The specific method is the same as the 7.2 1D ^{13}C Experiment.

7.8 BIRD (BIlinear Rotation Decoupling)

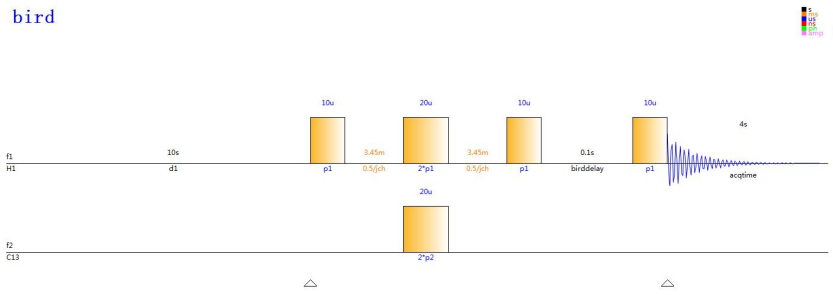


Figure 7.8 Pulse sequence of BIRD

1. Phase Cycle

- ph1: 0
- ph2: 180

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is 20 s (＞5 times T_1)
- birddelay: Bird delay, set values as follows in array mode, [0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1, 1.1, 1.2, 1.3, 1.4]
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- jch: 145 Hz (CH one-bond scalar coupling constant)
- acqtime: Acquisition time is around 4 s
- ns: Number of the scans is 1

3. Processing

(1) Perform Fourier transform to the array data (FID).

(2) Type "***ftabs***".

(3) Find the spectrum with the weakest signal (peak intensity) and the corresponding birddelay is the optimum value.

7.9 1D DOSY (Diffusion Ordered SpectroscopY)

Figure 7.9 Pulse sequence of 1D DOSY

1. Phase Cycle

- ph1: 0
- ph2: 0 0 180 180
- ph3: 0 0 0 0 180 180 180 180 90 90 90 90 270 270 270 270
- ph4: 0 180 0 180 180 0 180 0 90 270 90 270 270 90 270 90
- ph5: 0 0 0 0 180 180 180 180 90 90 90 90 270 270 270 270
- ph6: 0 180 180 0 180 0 0 180 270 90 90 270 90 270 270 90

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is about 5 s
- ecdelay: Eddy current delay is about 5 ms
- diffdelay: Diffusion time (big delta) is 20~200 ms
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- gzlevel: Variable z-gradient, set values as follows in array mode, [10.6, 16.2, 21.8, 27.4, 33, 38.6, 44.2, 49.8, 55.4, 61, 66.6, 72.2, 77.8, 83.4, 89]
- gzlevel1: Amplitude of spoiled z-gradient is 100%

- gtime: Gradient time (0.5 * little delta) is 1.0 ms
- gshape: Gradient pulse shape is hsine.grd
- gstab: Delay for gradient recovery is 0.2 ms
- acqtime: Acquisition time is around 1 s
- ns: Number of the scans is in multiples of 16, depending on the sample concentration

3. Processing

See 6.6.3 DOSY fittingfor the processing after the Fourier transform.

7.10 1D Solvent (Water) suppression Experiments

7.10.1 PRESAT (Presaturation)

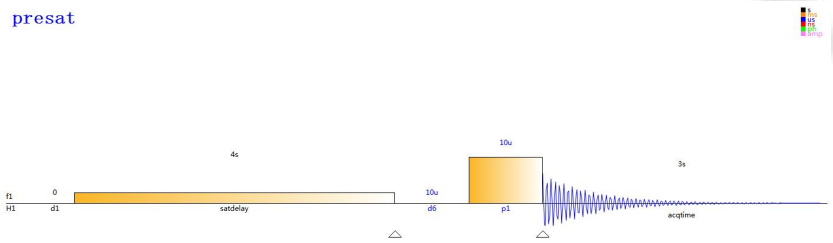


Figure 7.10 Pulse sequence of PRESAT

1. Phase Cycle

- ph1: 0
- ph2: 0 180 180 0 90 270 270 90
- ph3: 0 180 180 0 90 270 270 90

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is 0~2s

- d6: delay for power switching is 10μs
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- satdelay: Delay for presaturation is about 4 s
- satplvl: Power level for presaturation is about 3 dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center (this value can be set to 0 Hz and the center of the spectrum is aligned to the saturated peak)
- acqtime: Acquisition time is around 2.5 s
- ns: Number of the scans is in multiples of 4, depending on the sample concentration

3. Processing

Perform Fourier transform to obtain frequency domain data from time domain data (FID) created by acquisition with *si* larger than *td*, followed by processing steps like phase and baseline correction, resulting pure absorption signal in the spectrum. After reference calibration, peak picking and other analysis steps, the spectrum can be plotted.

7.10.2 WATERGATE

Figure 7.11 Pulse sequence of WATERGATE (using W3 sequence)

1. Phase Cycle

- ph1: 0 90 90 180 180 270 270
- ph2: 180 180 270 270 0 0 90 90

- ph3: 0 0 90 90 180 180 270 270

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is about 2 s
- wgdelay: Delay for binomial water suppression, is equal to 1/(2*d) where d is the distance of next null (in Hz)
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- gzlevel: Amplitude of z-gradient is about 15%
- gtime: Gradient time is 1.0 ~2.0ms
- gshape: Gradient pulse shape is hsine.grd
- gstab: Delay for gradient recovery is 0.1~0.2 ms
- acqtime: Acquisition time is around 3 s
- ns: Number of the scans is in multiples of 8, depending on the sample concentration

3. Processing

Perform Fourier transform to obtain frequency domain data from time domain data (FID) created by acquisition with *si* larger than *td*, followed by processing steps like phase and baseline correction, resulting pure absorption signal in the spectrum. After reference calibration, peak picking and other analysis steps, the spectrum can be plotted.

7.10.3 Solvent (Water) Suppression by 1D-NOESY

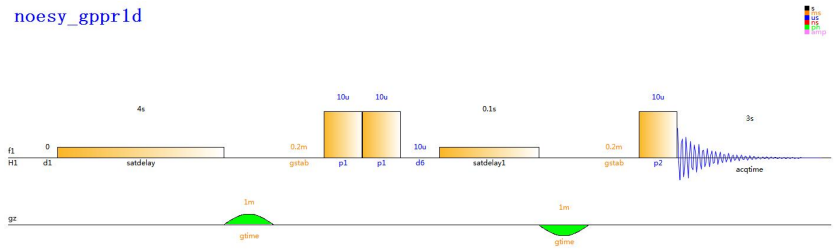


Figure 7.12 Pulse sequence of solvent water suppression by 1D NOESY (noesy_gppr1d)

1. Phase Cycle
   - ph1: 0 180
   - ph2: 0 0 0 0 0 0 0 0 180 180 180 180 180 180 180 180
   - ph3: 0 0 180 180 90 90 270 270
   - ph4: 0
   - ph5: 0 180 180 0 90 270 270 90 180 0 0 180 270 90 90 270
2. Experiment Parameters
   - sw: Spectral width is around 12 ppm
   - gain: Receiver gain, set appropriate value to avoid overflow
   - frqb: Basic frequency of observed channel (^{1}H)
   - frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
   - d1: Relaxation delay is about 0~2s
   - d6: delay for power switching is 10μs
   - p1: 90 degree pulse width of observed channel (^{1}H)
   - p2: 90 degree pulse width of observed channel (^{1}H)
   - plvl1: Power level of pulse in observed channel (^{1}H)
   - gzlevel: Amplitude of z-gradient, set values as about 20%
   - gtime: Gradient time is 1.0 ~2.0ms
   - gshape: Gradient pulse shape is hsine.grd

- gstab: Delay for gradient recovery is 0.1~0.2 ms
- satdelay: Delay for presaturation is about 4 s
- satdelay1: Second delay for presaturation is 0.1 s
- satplvl: Power level for presaturation is about 3 dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center (this value can be set to 0 Hz and the center of the spectrum is aligned to the saturated peak)
- acqtime: Acquisition time is around 2 s
- ns: Number of the scans is in multiples of 8, depending on the sample concentration

3. Processing

Perform Fourier transform to obtain frequency domain data from time domain data (FID) created by acquisition with *si* larger than *td*, followed by processing steps like phase and baseline correction, resulting pure absorption signal in the spectrum. After reference calibration, peak picking and other analysis steps, the spectrum can be plotted.

7.11 1D Selective Experiments

7.11.1 Selective 1D COSY Experiment

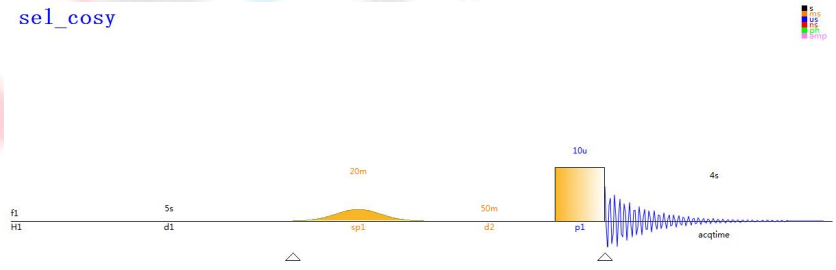


Figure 7.13 Pulse sequence of selective 1D COSY experiment (sel_cosy)

1. Phase Cycle

- ph1: 90 270 270 90 180 0 0 180
- ph2: 0 180 0 180 90 270 90 270

- ph3: 0 180 180 0 90 270 270 90

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is about 2 s
- sp1: 90 degree shape pulse width of observed channel (^{1}H)
- splvl1: Power level of shape pulse in observed channel (^{1}H)
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- acqtime: Acquisition time is around 2 s
- ns: Number of the scans is in multiples of 8, depending on the sample concentration

3. Processing

Perform Fourier transform to obtain frequency domain data from time domain data (FID) created by acquisition with *si* larger than *td*, followed by processing steps like phase and baseline correction, resulting pure absorption signal in the spectrum. After reference calibration, peak picking and other analysis steps, the spectrum can be plotted.

7.11.2 Phase-Cycled Selective 1D TOCSY Experiment

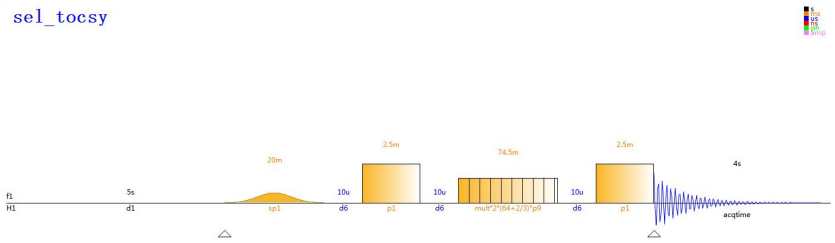


Figure 7.14 Pulse sequence of phase-cycled selective 1D TOCSY experiment (sel_tocsy)

1. Phase Cycle

- ph1: 90 270 270 90 0 180 180 0
- ph2: 0 180 0 180 90 270 90 270
- ph3: 0 180 0 180 90 270 90 270

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is about 2 s
- d6: delay for power switching is 10μs
- sp1: 90 degree shape pulse width of observed channel (^{1}H)
- splvl1: Power level of shape pulse in observed channel (^{1}H)
- p1: Trim pulse of observed channel (^{1}H) is 2.5 ms
- p9: Pulse for TOCSY-spinlock is about 40 μs
- slpltocsy: Power level of TOCSY-spinlock pulse in observed channel (^{1}H)
- mult: Cycle number of composite pulse is 18 (must be even number, make total TOCSY-spinlock time about 60~80 ms)
- acqtime: Acquisition time is around 2 s
- ns: Number of the scans is in multiples of 8, depending on the sample concentration

3. Processing

Perform Fourier transform to obtain frequency domain data from time domain data (FID) created by acquisition with *si* larger than *td*, followed by processing steps like phase and baseline correction, resulting pure absorption signal in the spectrum. After reference calibration, peak picking and other analysis steps, the spectrum can be plotted.

7.11.3 Gradient-Based Selective 1D TOCSY Experiment

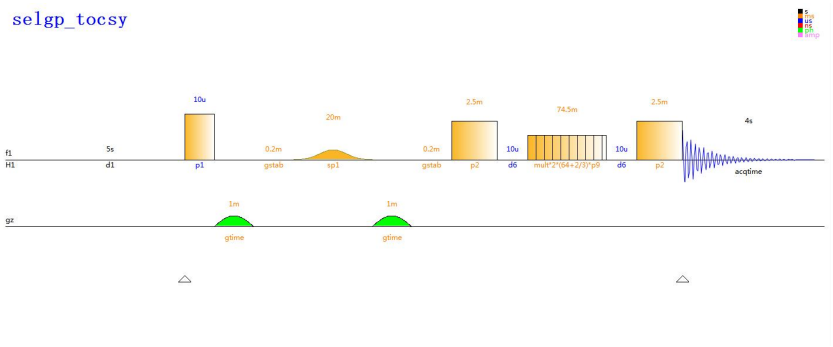


Figure 7.15 Pulse sequence of gradient-based selective 1D TOCSY experiment (selgp_tocsy)

1. Phase Cycle

- ph1: 0 180
- ph2: 0 0 90 90 180 180 270 270
- ph3: 90
- ph4: 0 180 180 0

2. Experiment Parameters

- sw: Spectral width is around 12 ppm
- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is about 2 s
- d6: delay for power switching is 10μs
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- sp1: 180 degree shape pulse width of observed channel (^{1}H)
- splvl1: Power level of shape pulse in observed channel (^{1}H)
- p2: Trim pulse of observed channel (^{1}H) is 2.5 ms
- p9: Pulse for TOCSY-spinlock is about 40 μs
- slpltocsy: Power level of TOCSY-spinlock pulse in observed channel (^{1}H)
- mult: Cycle number of composite pulse is 18 (must be even number, make total

TOCSY-spinlock time about 60~80 ms)

- gzlevel: Amplitude of z-gradient, set values as about 20%
- gtime: Gradient time is 1.0 ~2.0ms
- gshape: Gradient pulse shape is hsine.grd
- gstab: Delay for gradient recovery is 0.1~0.2 ms
- acqtime: Acquisition time is around 2 s
- ns: Number of the scans is in multiples of 8, depending on the sample concentration

3. Processing

Perform Fourier transform to obtain frequency domain data from time domain data (FID) created by acquisition with *si* larger than *td*, followed by processing steps like phase and baseline correction, resulting pure absorption signal in the spectrum. After reference calibration, peak picking and other analysis steps, the spectrum can be plotted.

7.11.4 Gradient-Based Selective 1D NOESY Experiment

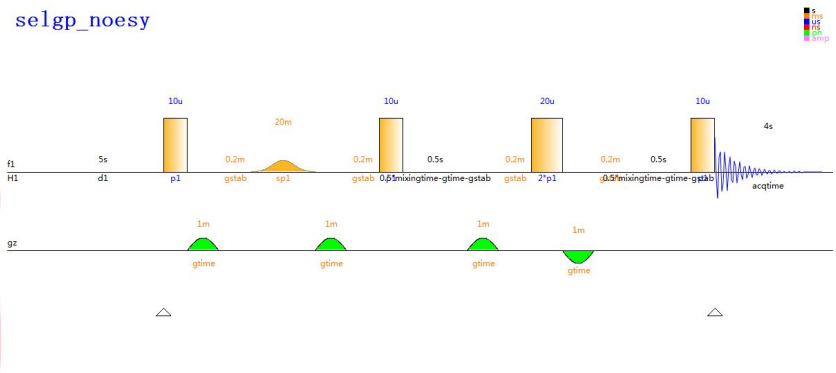


Figure 7.16 Pulse sequence of gradient-based selective 1D NOESY experiment (selgp_noesy)

1. Phase Cycle

- ph1: 0 180
- ph2: 0 0 90 90 180 180 270 270
- ph3: 0
- ph4: 0 180 180 0

2. Experiment Parameters

- sw: Spectral width is around 12 ppm

- gain: Receiver gain, set appropriate value to avoid overflow
- frqb: Basic frequency of observed channel (^{1}H)
- frqohz: Frequency offset of observed channel (^{1}H), the center of the spectrum
- d1: Relaxation delay is about 2 s
- p1: 90 degree pulse width of observed channel (^{1}H)
- plvl1: Power level of pulse in observed channel (^{1}H)
- sp1: 180 degree shape pulse width of observed channel (^{1}H)
- splvl1: Power level of shape pulse in observed channel (^{1}H)
- gzlevel: Amplitude of z-gradient is about 20%
- gtime: Gradient time is 1.0 ~2.0ms
- gshape: Gradient pulse shape is hsine.grd
- gstab: Delay for gradient recovery is 0.1~0.2 ms
- acqtime: Acquisition time is around 2 s
- ns: Number of the scans is in multiples of 8, depending on the sample concentration

3. Processing

Perform Fourier transform to obtain frequency domain data from time domain data (FID) created by acquisition with *si* larger than *td*, followed by processing steps like phase and baseline correction, resulting pure absorption signal in the spectrum. After reference calibration, peak picking and other analysis steps, the spectrum can be plotted.

Chapter 8 2D NMR Experiments

The two-dimensional sequence names provided by this software are as follows: experimental type + underline + acquisition mode and others. The experimental type includes cosy, tocsy and so on. The acquisition mode includes tppi, qf and so on. Others include such as gradient, bird, etc. Acquisition mode and others are sorted alphabetically.

The following is the meaning of the characters contained in the two-dimensional sequence names provided for this software:

bi: With BIRD fragment

dqf: Double quantum filtering

pr: Presaturation

gp: Gradient

qf: Qf mode of acquisition

sh: States mode of acquisition

st: States-TPPI Mode of acquisition

tp: TPPI mode of acquisition

ea: Echo-antiecho mode of acquisition

For example, cosy_prqf represents the absolute cosy spectrum with presaturation function. Users can choose different pulse sequences according to their needs.

8.1 ^{1}H-^{1}H COSY

8.1.1 cosy_prqf

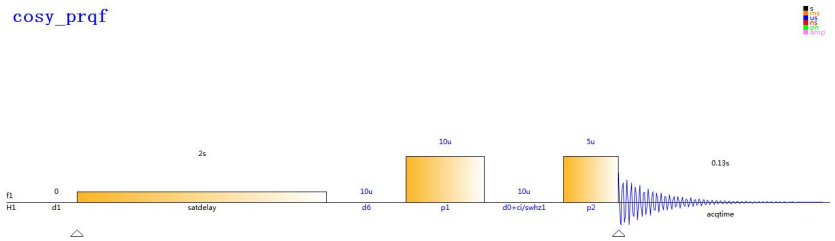


Figure 8.1 Absolute value COSY pulse sequence with presaturation

1. Phase table

- ph1: 0 0 90 90 180 180 270 270
- ph2: 0 90 270 0 180 270 90 180
- ph3: 0 180 90 270 180 0 270 90

2. Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- p2: 90° (or 30° ~ 60°) pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0s

- d6: delay for power switching is 10μs
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3. Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4. Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.1.2 cosy_prtp

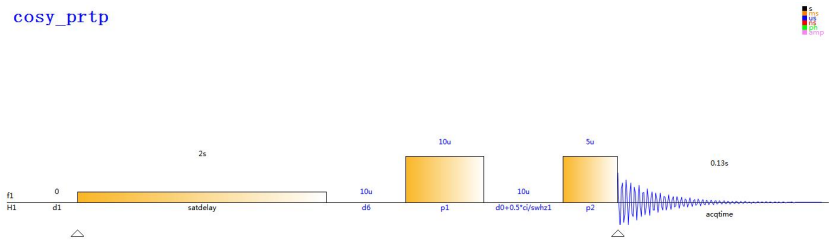


Figure 8.2 Phase sensitive COSY with presaturation

1.Phase table

- ph1: 0 180 180 0 90 270 270 90
- ph2: 0 180 0 180 90 270 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- p2: 90° (or 30° ~ 60°) pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4

- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3. Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4. Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in phase sensitive mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.1.3 cosy_gpqf

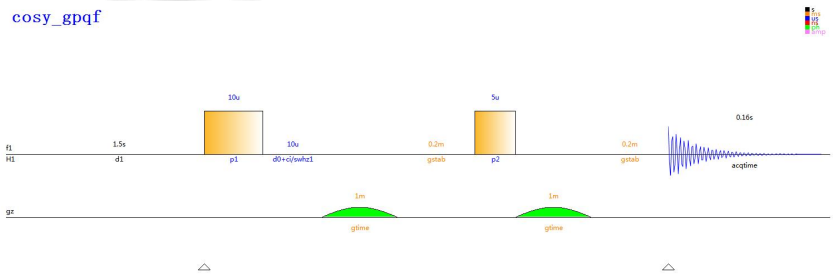


Figure 8.3 Gradient absolute value COSY pulse sequence

1.Phase table

- ph1: 0 180 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- p2: 90° (or 30° ~ 60°) pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 4 (or multiple of 4) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4

3. Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0

- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4. Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.1.4 cosy_gpprqf

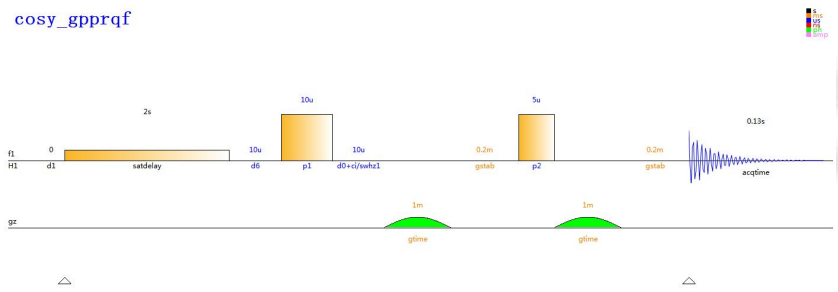


Figure 8.4 Absolute value COSY pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0 180 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum

- p1: 90° pulse width of ^{1}H transmitter in observed channel
- p2: 90° (or 30°~ 60°) pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3. Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4. Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.1.5 cosy_eagp/cosy_etgp

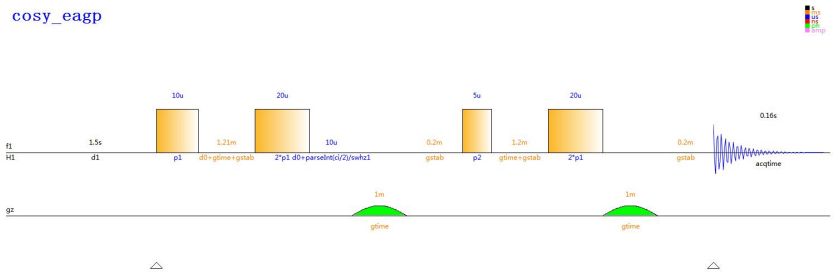


Figure 8.5 Phase sensitive COSY pulse sequence with gradient

1.Phase table

- ph1: 0 180 180 0 90 270 270 90
- ph2: 0 180 0 180 90 270 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90°pulse width of ^{1}H transmitter in observed channel
- p2: 90° (or 30° ~ 60°) pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms

- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.1.6 cosy_eagppr/cosy_etgppr

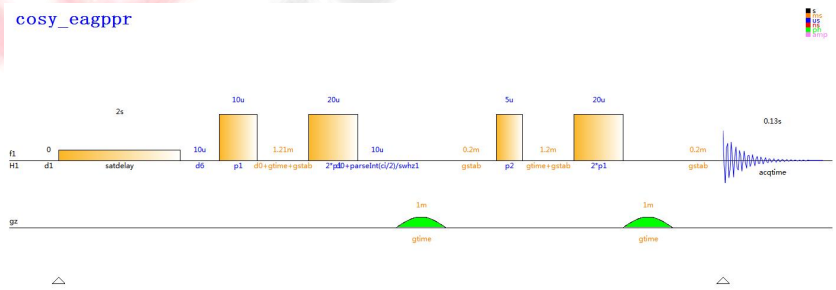


Figure 8.6 Phase sensitive COSY pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0 180 180 0 90 270 270 90

- ph2: 0 180 0 180 90 270 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- p2: 90° (or 30° ~ 60°) pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos

- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.1.7 cosy_dqfgptp

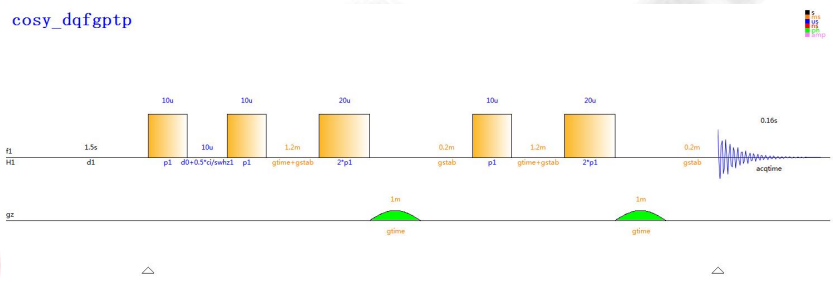


Figure 8.7 Phase sensitive COSY pulse sequence with gradient and double quantum filtering

1.Phase table

- ph1: 0 180 (Phase increases by TPPI)
- ph2: 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024

- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.2 ^{1}H-^{19}F COSY

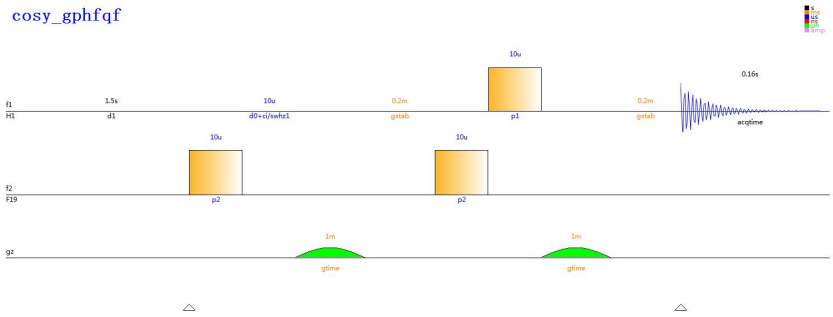


Figure 8.8 ^{1}H-^{19}F COSY pulse sequence with gradient

1.Phase table

- ph1: 0 180
- ph2: 0 0 180 180
- ph3: 0
- ph4: 0 180 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension (Obtained from the basic single pulse ^{19}F NMR spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{19}F transmitter
- frqohz1: Frequency offset of ^{19}F transmitter, the center of ^{19}F NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel

- p2: 90° pulse width of ^{19}F transmitter
- plvl2: The power of ^{19}F transmitter
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.3 TOCSY (TOtal Correlation SpectroscopY)

8.3.1 mlev_st

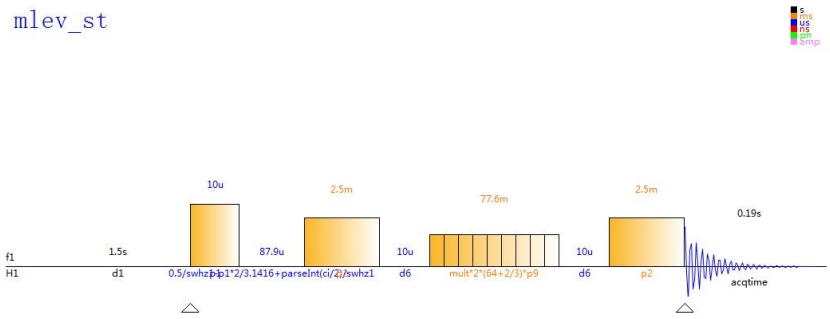


Figure 8.9 mlev_st pulse sequence

1.Phase table

- ph1: 0 180 180 0 90 270 270 90 (Phase increases by TPPI)
- ph2: 0 180 0 180 90 270 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p2: Trim pulse width of ^{1}H transmitter in Observed channel, 2.5 ms
- p9: 90° pulse width of Spin lock, 30 ~ 40 μs
- slpltocsy: The power of ^{1}H transmitter in observed channel corresponding with p9
- mult: The number of cycles of the spin lock combination pulse: 18 (must be even, so

that the total width of the spin lock combination pulse is about 60 ~80 ms)

- d1: Relaxation delay, 0.5-2 s
- d6: delay for power switching is 10μs
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.3.2 dipsi2_st

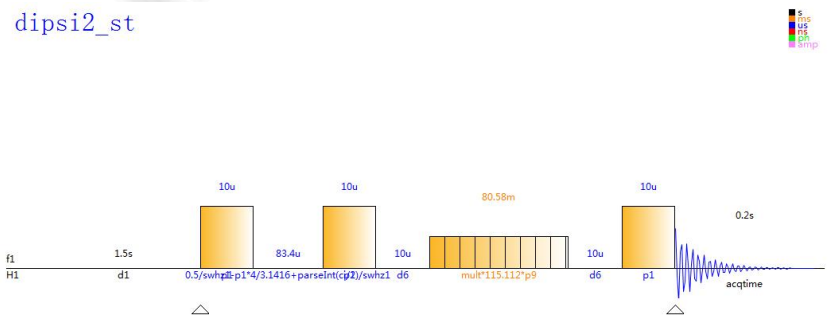


Figure 8.10 dipsi2_st pulse sequence

1.Phase table

- ph1: 0 180
- ph2: 0
- ph3: 0 0 0 0 180 180 180 180
- ph4: 0 0 180 180
- ph5: 0 180 180 0 180 0 0 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p2: Trim pulse width of ^{1}H transmitter in Observed channel, 2.5 ms
- p9: 90° pulse width of Spin lock, 30 ~ 40 μs
- slpltocsy: The power of ^{1}H transmitter in observed channel corresponding with p9
- mult: The number of cycles of the spin lock combination pulse: 18 (must be even, so that the total width of the spin lock combination pulse is about 60 ~80 ms)
- d1: Relaxation delay, 0.5-2 s
- d6: delay for power switching is 10μs
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512

- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.3.3 dipsi2_eagp/dipsi2_etgp

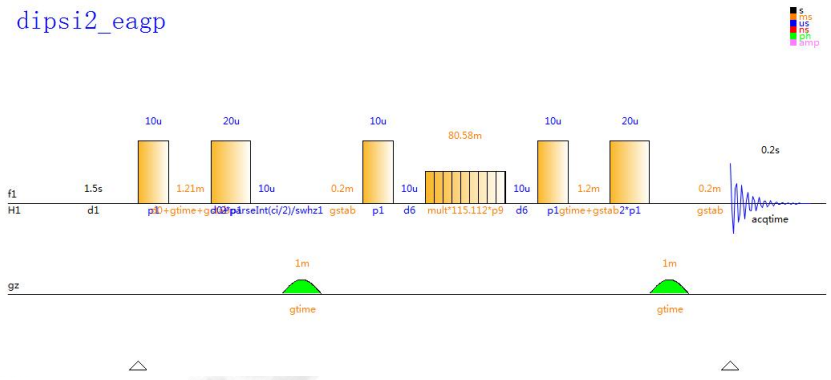


Figure 8.11 dipsi2_eagp pulse sequence

1.Phase table

- ph1: 0 180
- ph2: 0
- ph3: 0 0 0 0 180 180 180 180
- ph4: 0 0 180 180
- acq: 0 180 180 0 180 0 0 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p9: 90° pulse width of Spin lock, 30 ~ 40 μs
- slpltocsy: The power of ^{1}H transmitter in observed channel corresponding with p9
- mult: The number of cycles of the spin lock combination pulse: 18 (must be even, so that the total width of the spin lock combination pulse is about 60-80 ms)
- d1: Relaxation delay, 0.5-2 s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1

- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.3.4 dipsi2_eagppr/dipsi2_etgppr

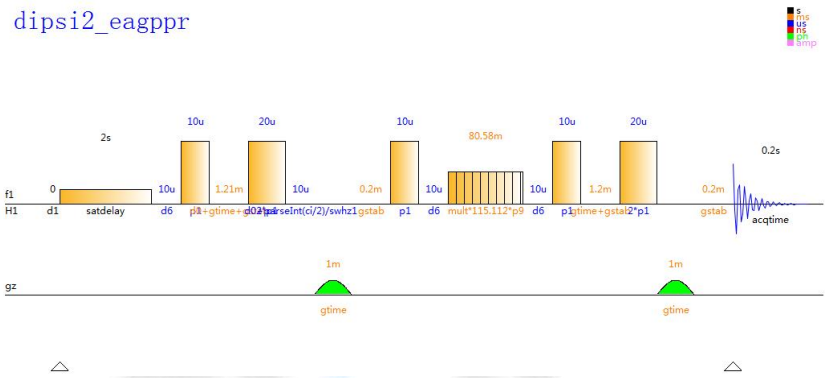


Figure 8.12 dipsi2_eagppr pulse sequence

1.Phase table

- ph1: 0 180
- ph2: 0
- ph3: 0 0 0 0 180 180 180 180
- ph4: 0 0 180 180
- ph5: 0 180 180 0 180 0 0 180

2.Processing parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024

- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p9: 90° pulse width of Spin lock, 30 ~ 40 μs
- slpltocsy: The power of ^{1}H transmitter in observed channel corresponding with p9
- mult: The number of cycles of the spin lock combination pulse: 18 (must be even, so that the total width of the spin lock combination pulse is about 60~80 ms)
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1

- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.3.5 dipsi2_gpstzqf

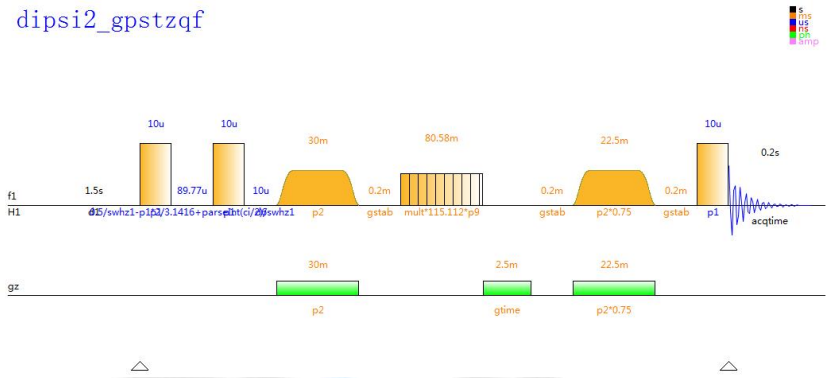


Figure 8.13 dipsi2_gpstzqf pulse sequence

1.Phase table

- ph1: 0 180
- ph2: 0
- ph3: 0 0 0 0 0 0 0 0 90 90 90 90 90 90 90 90
- ph4: 0
- ph5: 0 0 0 0 180 180 180 180
- ph6: 0 0 180 180
- ph7: 0 180 180 0 180 0 0 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)

- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p2: shape pulse width
- plvl2: Power level of shape pulse
- p9: 90° pulse width of Spin lock, 30 ~ 40 μs
- slpltocsy: The power of ^{1}H transmitter in observed channel corresponding with p9
- mult: The number of cycles of the spin lock combination pulse: 18 (must be even, so that the total width of the spin lock combination pulse is about 60 ~80 ms)
- d1: Relaxation delay, 0.5-2 s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 50（namely 50%）
- gzlevel2: Amplitude level of Z gradient, around 8（namely8%）
- gtime: Gradient time, 1~2ms
- gshape: hard.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1

- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.4 NOESY (Nuclear Overhauser Effect SpectroscopY)

8.4.1 noesy_eagp/noesy_etgp

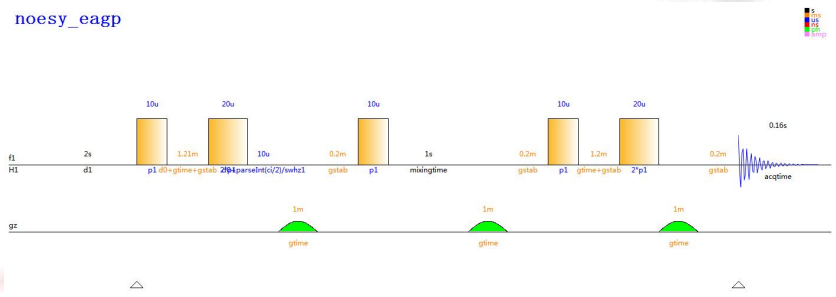


Figure 8.14 NOESY pulse sequence with gradient

1.Phase table

- ph1: 0 180
- ph2: 0
- ph3: 0 0 0 0 0 0 0 0 180 180 180 180 180 180 180 180
- ph4: 0 0 180 180 270 270 90 90
- ph5: 0 180 180 0 90 270 270 90 180 0 0 180 270 90 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic

single pulse ^{1}H NMR spectrum, optimized to the narrowest)

- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0.5-2 s
- mixingtime: Mixing time, about 1~2 s for small molecules and less than 1 s for macromolecules, which amounts to the T1 relaxation time of samples.
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 16 (or multiple of 16) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.4.2 noesy_gptp

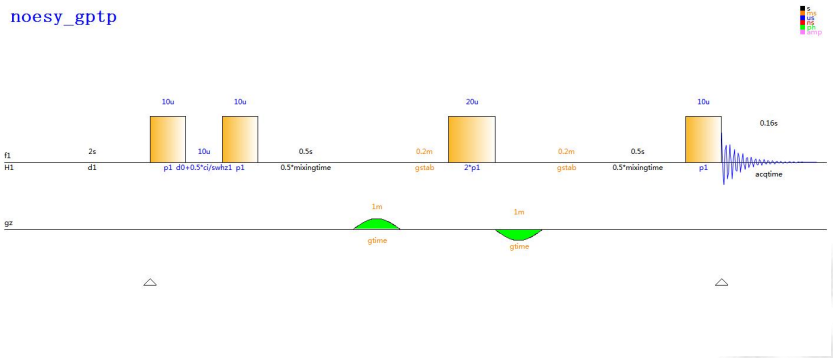


Figure 8.15 NOESY pulse sequence with gradient

1.Phase table

- ph1: 0 180 (Phase increases by TPPI)
- ph2: 0 0 0 0 0 0 0 0 180 180 180 180 180 180 180 180
- ph3: 0 0 180 180 90 90 270 270
- ph4: 0
- ph5: 0 180 180 0 90 270 270 90 180 0 0 180 270 90 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H

NMR spectrum

- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0.5-2 s
- mixingtime: Mixing time, about 1~2 s for small molecules and less than 1 s for macromolecules, which amounts to the T1 relaxation time of samples.
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 16 (or multiple of 16) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.4.3 noesy_gptpzqf

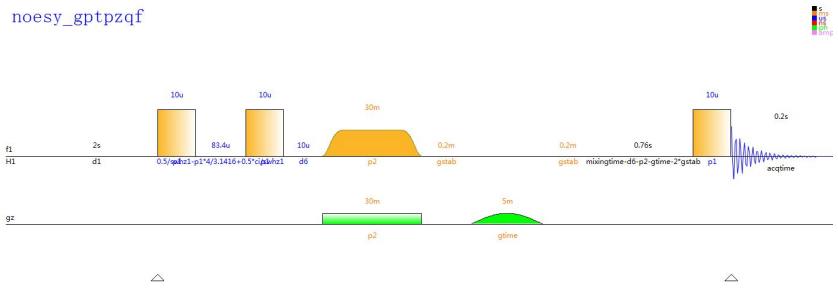


Figure 8.16 NOESY pulse sequence with Zero-Quantum Filter

1.Phase table

- ph1: 0 180 (Phase increases by TPPI)
- ph2: 0 0 0 0 0 0 0 0 180 180 180 180 180 180 180 180
- ph3: 0 0 180 180 90 90 270 270
- ph4: 0
- ph5: 0 180 180 0 90 270 270 90 180 0 0 180 270 90 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 256
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p2: shape pulse width
- plvl2: Power level of shape pulse
- d1: Relaxation delay, 0.5-2 s

- mixingtime: Mixing time, about 1~2 s for small molecules and less than 1 s for macromolecules, which amounts to the T1 relaxation time of samples.
- gzlevel: Amplitude level of Z gradient, around 30（namely 30%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gzlevel2: Amplitude level of Z gradient, around 8（namely 8%）
- gshape2: hard.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 16 (or multiple of 16) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.4.4 noesy_gpprtp

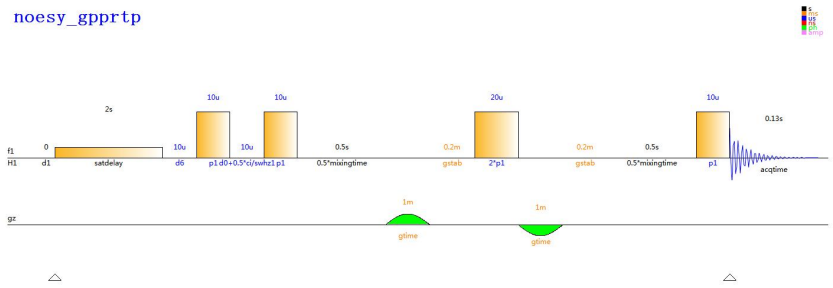


Figure 8.17 NOESY pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0 180 (Phase increases by TPPI)
- ph2: 0 0 0 0 0 0 0 0 180 180 180 180 180 180 180 180
- ph3: 0 0 180 180 90 90 270 270
- ph4: 0
- ph5: 0 180 180 0 90 270 270 90 180 0 0 180 270 90 90 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs

- mixingtime: Mixing time, about 1~2 s for small molecules and less than 1 s for macromolecules, which amounts to the T1 relaxation time of samples.
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 16 (or multiple of 16) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.5 ROESY (Rotating Frame Nuclear Overhauser Effect SpectroscopY)

8.5.1 roesy_prtp

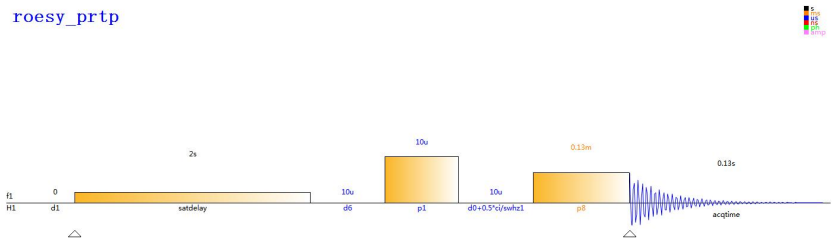


Figure 8.18 ROESY pulse sequence with presaturation

1.Phase table

- ph1: 0 180180 0 90 270 270 90 (Phase increases by TPPI)
- ph2: 0 180 0 180 90 270 90 270
- ph3: 0
- ph4: 0 180180 0 90 270 270 90

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p8: 90° pulse width of spin lock, around 130μs

- slplroesy: The power of ^{1}H transmitter in observed channel corresponding with p8
- mult: The number of cycles of the spin lock combination pulse: 600 (must be even, so that the total width of the spin lock combination pulse is about 300ms)
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.5.2 roesy_eagp/roesy_etgp

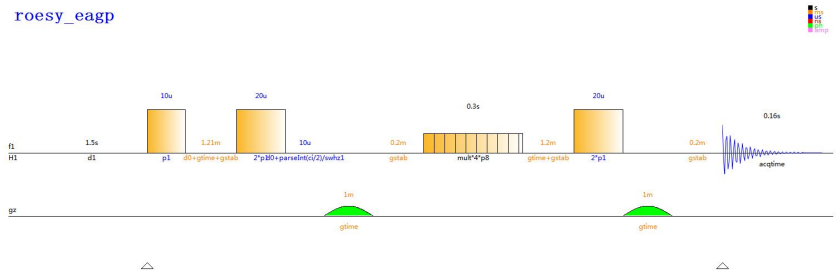


Figure 8.19 ROESY pulse sequence with gradient

1.Phase table

- ph1: 0 180
- ph2: 0
- ph3: 0 0 180 180
- ph4: 180 180 0 0
- ph5: 90 90 270 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p8: 90° pulse width of spin lock, around 130μs
- slplroesy: The power of ^{1}H transmitter in observed channel corresponding with p8
- mult: The number of cycles of the spin lock combination pulse: 600 (must be even,

so that the total width of the spin lock combination pulse is about 300ms)

- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1:0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.5.3 roesy_gptpw3

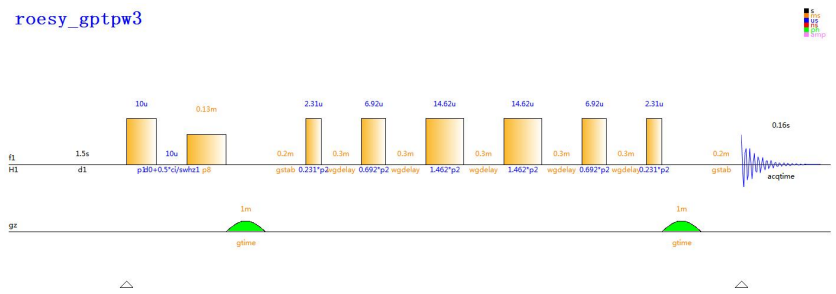


Figure 8.20 ROESY pulse sequence with gradient and water suppression

1.Phase table

- ph1: 0 180 180 0 90 270 270 90
- ph2: 0 180 0 180 90 270 90 270
- ph3: 0 180 0 180 90 270 90 270
- ph4: 180 0 180 0 270 90 270 90
- ph5: 0 180 180 0 90 270 270 90

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is equal to sw
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- p1: 90° pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- p2: 90° pulse width of ^{1}H transmitter in observed channel
- plvl2: The power of ^{1}H transmitter in observed channel

- p8: 90° pulse width of spin lock, around 130μs
- slplroesy: The power of ^{1}H transmitter in observed channel corresponding with p8
- mult: The number of cycles of the spin lock combination pulse: 600 (must be even, so that the total width of the spin lock combination pulse is about 300ms)
- d1: Relaxation delay, 0.5-2 s
- wgdelay: Binomial water suppression delay, around 0.3 ms
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs:0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.6 Homo2dj (Homonuclear 2D J-resolved Spectroscopy)

8.6.1 homo2dj_qf

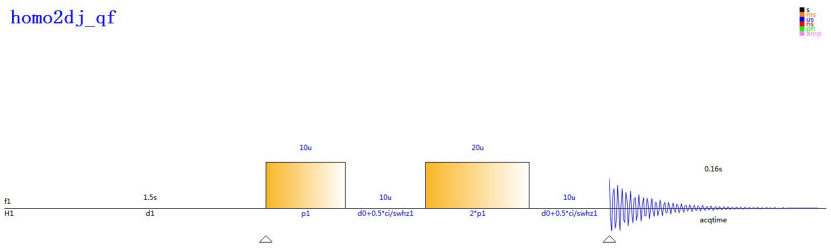


Figure 8.21 Homonuclear 2D J-resolved spectroscopy

1.Phase table

- ph1: 0 180 90 270
- ph2: 0 0 90 90 180 180 270 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- swhz1: Spectrum width in indirect dimension,100Hz (The maximum width of multiple peaks of ^{1}H-^{1}H coupling)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz1: 0 Hz
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel

- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0.5-2 s
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.6.2 homo2dj_prqf

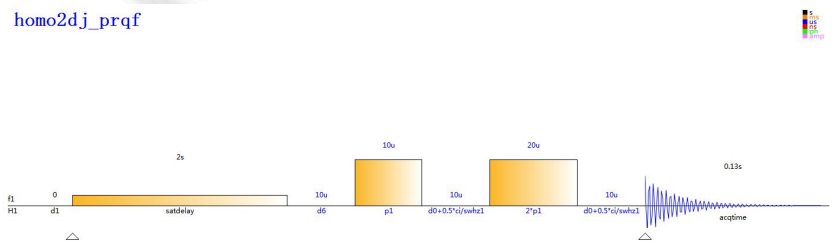


Figure 8.22 Homonuclear 2D J-resolved spectroscopy with presaturation

1.Phase table

- ph1: 0 180 90 270
- ph2: 0 0 90 90 180 180 270 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 12 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- swhz1: Spectrum width in indirect dimension, 100Hz (The maximum width of multiple peaks of ^{1}H-^{1}H coupling)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz1: 0 Hz
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel
- d1: Relaxation delay, 0 s
- d6: delay for power switching is 10μs
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512

- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.7 Heter2dj (Heteronuclear 2D J-resolved Spectroscopy)

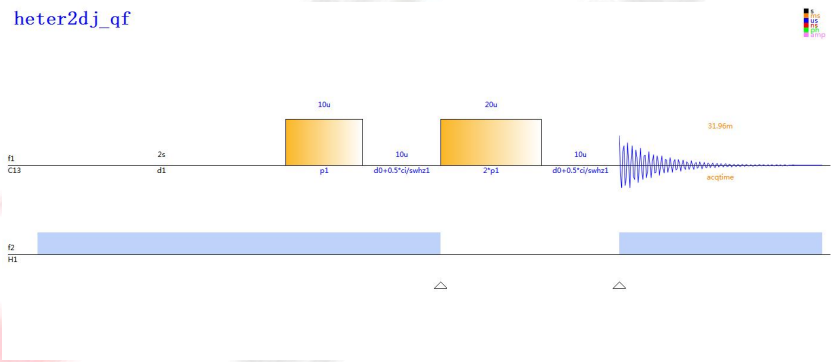


Figure 8.23 Heteronuclear 2D J-resolved Spectroscopy

1.Phase table

- ph1: 0 0 0 0 90 90 90 90 180 180 180 180 270 270 270 270
- ph2: 0 180 90 270 90 270 180 0 90 270 180 0 180 0 270 90
- ph3: 0 0 180 180 90 90 270 270

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 200 ppm (Obtained from the basic single pulse ^{13}C NMR spectrum, optimized to the narrowest)

- swhz1: Spectrum width in indirect dimension,300Hz (The half of maximum width of multiple peaks of ^{13}C-^{1}H coupling)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 64
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{13}C transmitter in observed channel
- frqohz: Frequency offset of ^{13}C transmitter in observed channel, the center of ^{13}C NMR spectrum
- frqb1: Basic Frequency of ^{1}H transmitter in decoupling channel
- frqohz1: 0 Hz
- p1: 90 degree pulse width of ^{13}C transmitter in observed channel
- plvl1: The power of ^{13}C transmitter in observed channel
- p2: 90 degree pulse width of ^{1}H transmitter in decoupling channel
- plvl2: The power of ^{1}H transmitter in decoupling channel corresponding with p2
- decon1: Decoupling switch 'yny'
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, www (waltz16 combined pulse decoupling)
- decplvl1: The decoupling power of ^{1}H transmitter in decoupling channel, around 37dB
- d1: Relaxation delay, 0.5-2 s
- ns: 16 (or multiple of 16) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 4

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5

- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.8 HMQC (Heteronuclear Multiple Quantum Coherence)

8.8.1 hmqc_bitp

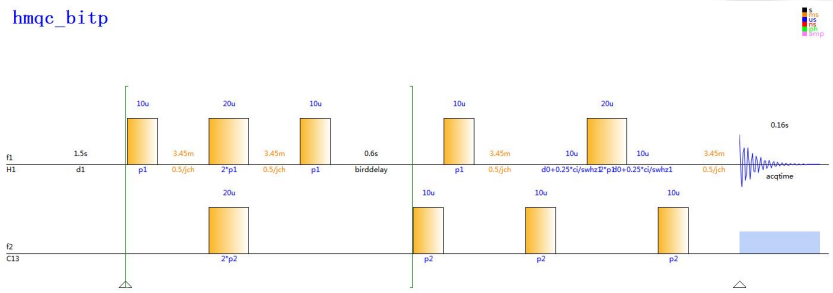


Figure 8.24 HMQC pulse sequence

1.Phase table

- ph1: 0
- ph2: 180
- ph3: 0 180 180 0
- ph4: 0 0 0 0 180 180 180 180
- ph5: 0 180（Phase increases by TPPI）
- ph6: 0 0 180 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic

single pulse ^{1}H NMR spectrum, optimized to the narrowest)

- sw1: Spectrum width in indirect dimension is around 165ppm (Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- birddelay: BIRD delay, which is about 0.6s to be optimized to minimize FID. Observe the received FID in the set mode and adjust the birddelay to minimize the intensity.
- mult: BIRD switch, 1:turn on, 0:turn off
- Jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048

- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb:1
- sb1:1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.8.2 hmqc_eagp/hmqc_etgp

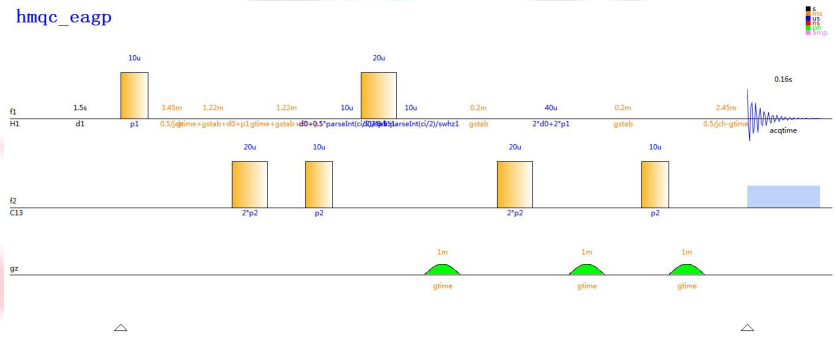


Figure 8.25 HMQC pulse sequence with gradient

1.Phase table

- ph1: 0
- ph2: 0 0 180 180
- ph3: 0 180
- ph4: 0 0 0 0 180 180 180 180
- ph5: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm (Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration

- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.8.3 hmqc_eagppr/hmqc_etgppr

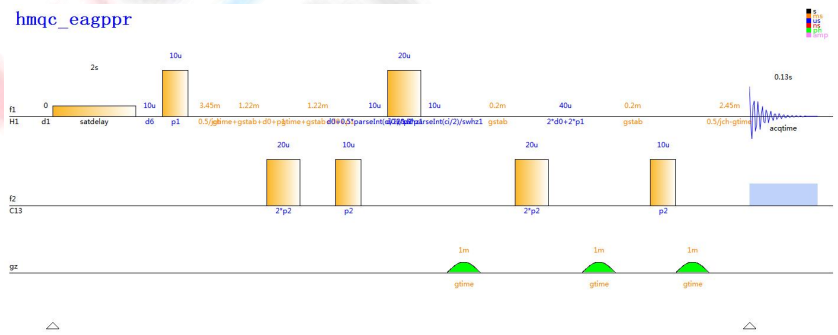


Figure 8.26 HMQC pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0
- ph2: 0 0 180 180

- ph3: 0 180
- ph4: 0 0 0 0 180 180 180 180
- ph5: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm(Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs

- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.9 HSQC (Heteronuclear Single Quantum Coherence)

8.9.1 hsqc_bitp

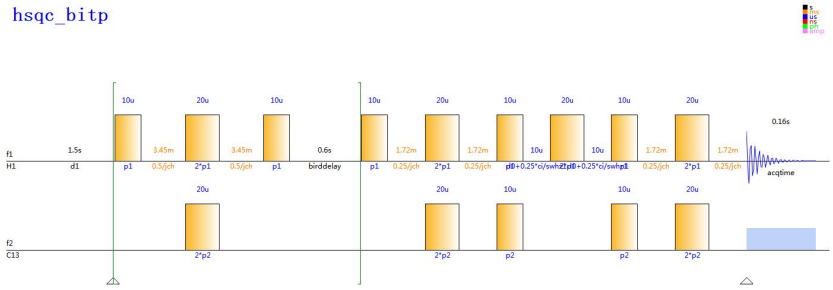


Figure 8.27 HSQC pulse sequence

1.Phase table

- ph1: 0
- ph2: 90
- ph3: 180
- ph4: 0 180 180 0
- ph5: 0 0 0 0 180 180 180 180
- ph6: 0 180
- ph7: 0 0 180 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm(Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H

NMR spectrum

- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- birddelay: BIRD delay, which is about 0.6s to be optimized to minimize FID. Observe the received FID in the set mode and adjust the birddelay to minimize the intensity.
- mult: BIRD switch, 1:turn on, 0:turn off
- Jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch 'nny'
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1:Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5

- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.9.2 hsqc_gptp

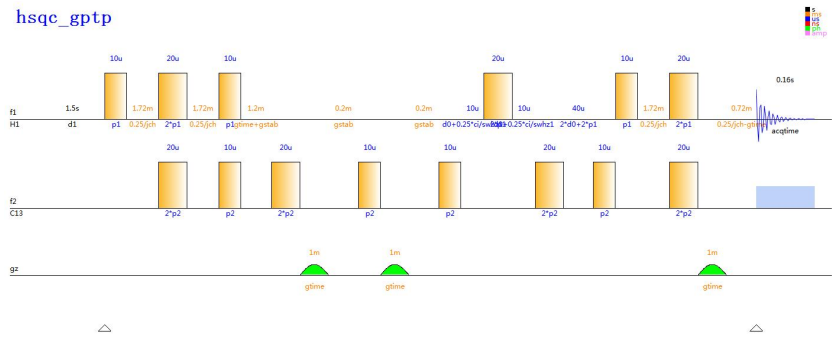


Figure 8.28 HSQC pulse sequence with gradient

1.Phase table

- ph1: 0
- ph2: 90
- ph3: 0 0 180 180
- ph4: 90 90 90 90 270 270 270 270
- ph5: 270
- ph6: 0 180
- ph7: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm(Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024

- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos

- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.9.3 hsqc_gpprtp

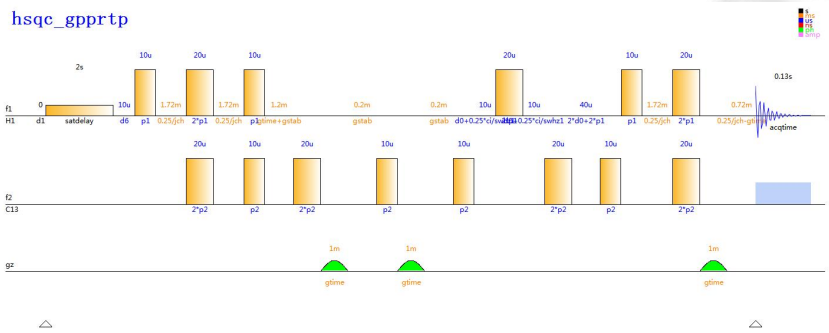


Figure 8.29 HSQC pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0
- ph2: 90
- ph3: 0 0 180 180
- ph4: 90 90 90 90 270 270 270 270
- ph5: 270
- ph6: 0 180
- ph7: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm(Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration

- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.9.4 hsqc_eagp/hsqc_etgp

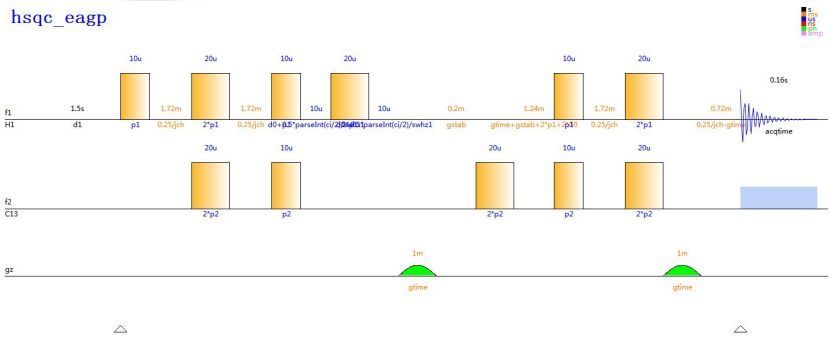


Figure 8.30 HSQC pulse sequence with gradient

1.Phase table

- ph1: 0
- ph2: 90
- ph3: 0 180
- ph4: 0 0 0 0 180 180 180 180
- ph5: 0 0 180 180
- ph6: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm (Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms

- jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.9.5 hsqc_eagppr/hsqc_etgppr

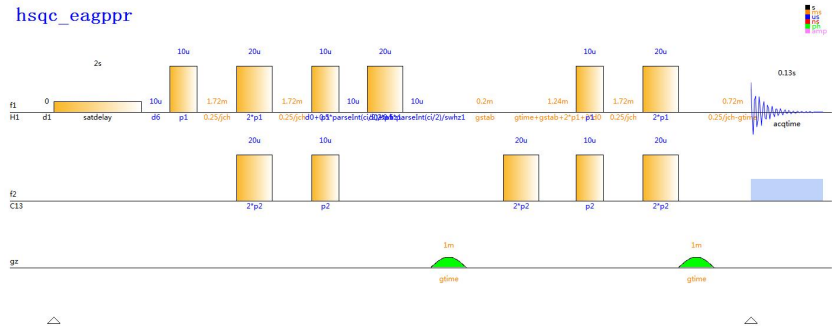


Figure 8.31 HSQC pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0
- ph2: 90
- ph3: 0 180
- ph4: 0 0 0 0 180 180 180 180
- ph5: 0 0 180 180
- ph6: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm (Obtained from the basic DEPT135 spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C

NMR spectrum

- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1

- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.9.6 hsqc_edeagp/hsqc_edetgp

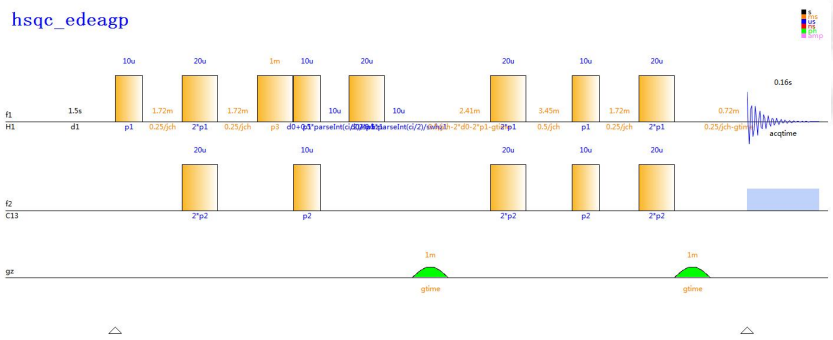


Figure 8.32 Editable gradient HSQC pulse sequence

1.Phase table

- ph1: 0
- ph2: 90
- ph3: 0 180
- ph4: 0 0 0 0 180 180 180 180
- ph5: 0 0 180 180
- ph6: 180 0 180 0 0 180 0 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 165ppm (Obtained from the basic DEPT135 spectrum, optimized to the narrowest)

- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- p3: Trim pulse width of ^{1}H transmitter in observed channel, 1ms
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- Jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 63μs
- dectype1: Decoupling modulation mode in decoupling channel, wwg (garp combined pulse decoupling)
- decplvl1: Decoupling power, around 37dB
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512

- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.10 HMBC (Heteronuclear Multiple Bond Correlation)

8.10.1 hmbc_eagp/hmbc_etgp

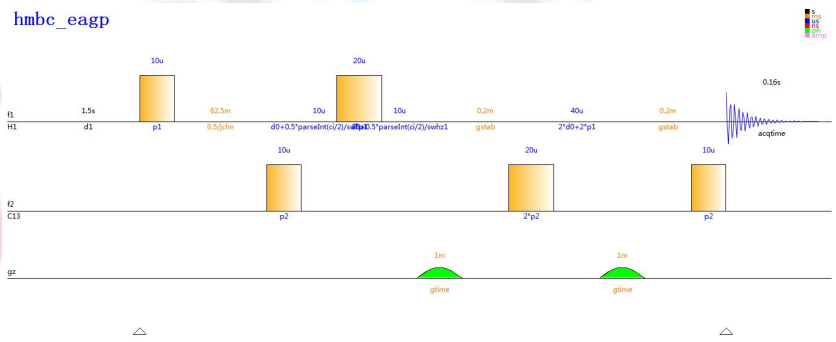


Figure 8.33 HMBC pulse sequence with gradient

1.Phase table

- ph1: 0
- ph2: 0 0 180 180
- ph3: 0 180
- ph4: 0 0 0 0 180 180 180 180

- ph5: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 220ppm (Obtained from the basic single pulse ^{13}C NMR spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jchn: 6~10 Hz（Long range coupling constant of CH）
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512

- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.10.2 hmbc_eagppr/hmbc_etgppr

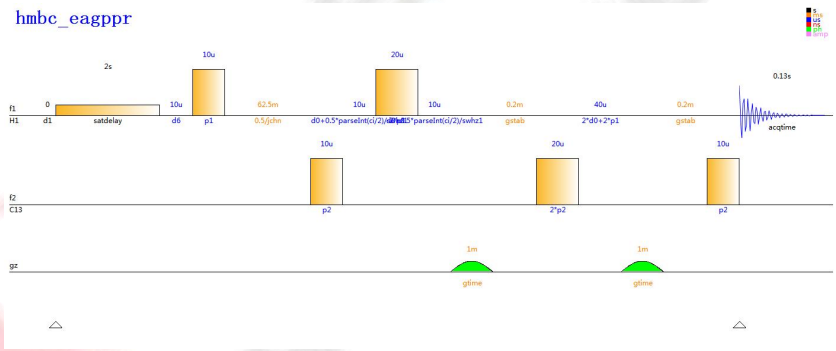


Figure 8.34 HMBC pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0
- ph2: 0 0 180 180
- ph3: 0 180
- ph4: 0 0 0 0 180 180 180 180
- ph5: 0 180 0 180 180 0 180 0

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 220ppm (Obtained from the basic single pulse ^{13}C NMR spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jchn: 6~10 Hz（Long range coupling constant of CH）
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared cos or cos
- wdw1: Squared cos or cos
- sb: 1
- sb1: 1
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.Processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the 2D spectrum in phase sensitive mode. The phase must be corrected in both two dimensions. The t1 noise can be further eliminated if the t1 noise is strong.

8.10.3 hmbc_gpqf

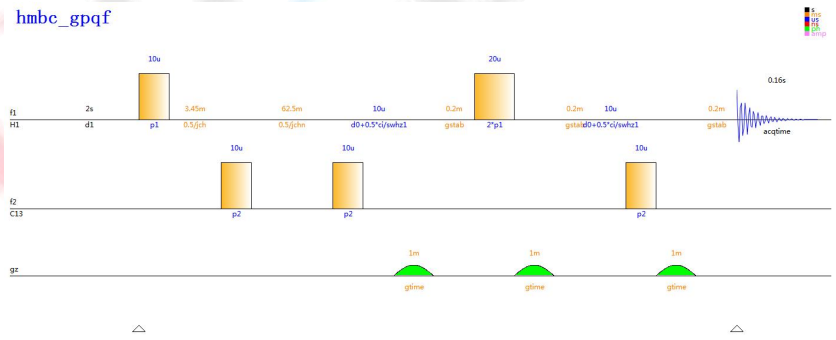


Figure 8.35 HMBC pulse sequence with gradient

1.Phase table

- ph1: 0
- ph2: 0 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 220ppm (Obtained from the basic single pulse ^{13}C NMR spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jch: 145 Hz　(Short range coupling constant of CH)
- jchn: 6~10 Hz (Long range coupling constant of CH)
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine

- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.10.4 hmbc_gpprqf

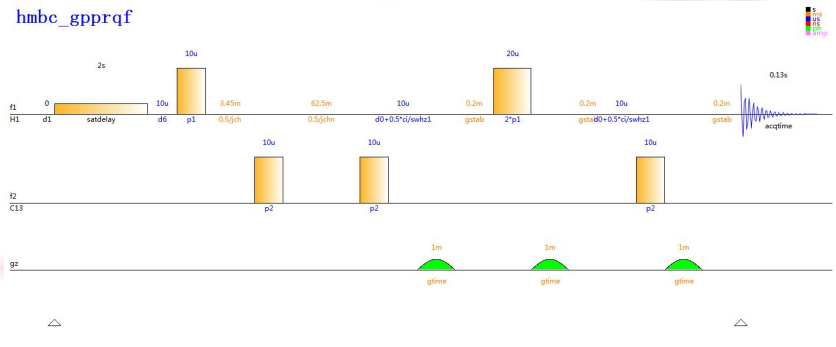


Figure 8.36 HMBC pulse sequence with gradient and presaturation

1.Phase table

- ph1: 0
- ph2: 0 180

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- sw1: Spectrum width in indirect dimension is around 220ppm (Obtained from the basic single pulse ^{13}C NMR spectrum, optimized to the narrowest)

- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{1}H transmitter in observed channel
- frqohz: Frequency offset of ^{1}H transmitter in observed channel, the center of ^{1}H NMR spectrum
- frqb1: Basic Frequency of ^{13}C transmitter in decoupling channel
- frqohz1: Frequency offset of ^{13}C transmitter in decoupling channel, the center of ^{13}C NMR spectrum
- p1: 90 degree pulse width of ^{1}H transmitter in observed channel
- plvl1: The power of ^{1}H transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{13}C transmitter in decoupling channel
- plvl2: The power of ^{13}C transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0s
- d6: delay for power switching is 10μs
- gzlevel: Amplitude level of Z gradient, around 20（namely 20%）
- gtime: Gradient time, 1~2ms
- gshape: hsine.grd
- gstab: Gradient stabilization time, 0.1~0.2ms
- jch: 145 Hz　(Short range coupling constant of CH)
- jchn: 6~10 Hz (Long range coupling constant of CH)
- ns: 8 (or multiple of 8) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 8
- satdelay: Presaturation time, ~2s
- satplvl: Presaturation pulse power, ~3dB
- satfreq: Presaturation pulse frequency offset, frequency difference between presaturation peak and spectral center

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512

- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5
- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

8.11 HCCOR (Heteronuclear Chemical Shift CORrelation)

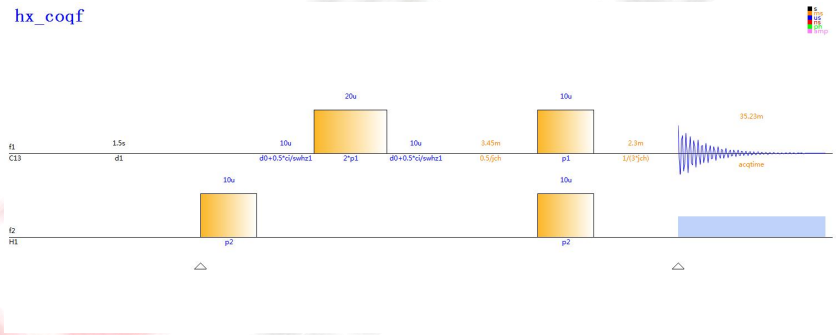


Figure 8.37 HCCOR pulse sequence

1.Phase table

- ph1: 0
- ph2: 0 180 90 270
- ph3: 0 0 0 0 180 180 180 180
- ph4: $(0)_8\,(90)_8\,(180)_8\,(270)_8$
- ph5: $(0\ 180\ 90\ 270)_2\ (90\ 270\ 180\ 0)_2\ (180\ 0\ 270\ 90)_2\ (270\ 90\ 0\ 180)_2$

2.Acquisition parameters

- sw: Spectrum width in direct dimension is around 220ppm (Obtained from the basic

single pulse ^{13}C NMR spectrum, optimized to the narrowest)

- sw1: Spectrum width in indirect dimension is around 10 ppm (Obtained from the basic single pulse ^{1}H NMR spectrum, optimized to the narrowest)
- np: Acquisition points in direct dimension, 1024
- np1: Incremental points in indirect dimension, 128
- gain: FID signal is not overflowed
- frqb: Basic Frequency of ^{13}C transmitter in observed channel
- frqohz: Frequency offset of ^{13}C transmitter in observed channel, the center of ^{13}C NMR spectrum
- frqb1: Basic Frequency of ^{1}H transmitter in decoupling channel
- frqohz1: Frequency offset of ^{1}H transmitter in decoupling channel, the center of ^{1}H NMR spectrum
- p1: 90 degree pulse width of ^{13}C transmitter in observed channel
- plvl1: The power of ^{13}C transmitter in observed channel corresponding with p1
- p2: 90 degree pulse width of ^{1}H transmitter in decoupling channel
- plvl2: The power of ^{1}H transmitter in decoupling channel corresponding with p2
- d1: Relaxation delay, 0.5-2 s
- jch: 145 Hz (Short range coupling constant of CH)
- decon1: Decoupling switch ‘nny’
- decpw1: Pulse width of decoupling, 90μs
- dectype1: Decoupling modulation mode, www (waltz16 combined pulse decoupling)
- decplvl1: Decoupling power, around 34dB
- ns: 32 (or multiple of 32) scans, depending on sample concentration
- dummyscan: Number of scans with no acquisition, 16

3.Processing parameters

- si: Fourier transform points in direct dimension, 2048
- si1: Fourier transform points in indirect dimension, 512
- wdw: Squared sine or sine
- wdw1: Squared sine or sine
- sb: 0.5

- sb1: 0.5
- sbs: 0
- sbs1: 0
- fcor: 0.5
- fcor1: 0.5

4.processing

Type ***wft*** in command line to perform two-dimensional Fourier transform and display the two-dimensional spectrum in absolute mode. The t1 noise can be further eliminated if the t1 noise is strong.

Chapter 9 Spectrum Printing

9.1 Start to print

Click on the menu bar **File** to select **Print**, or directly click the toolbar print button to open the print page shown in Figure 9.1.

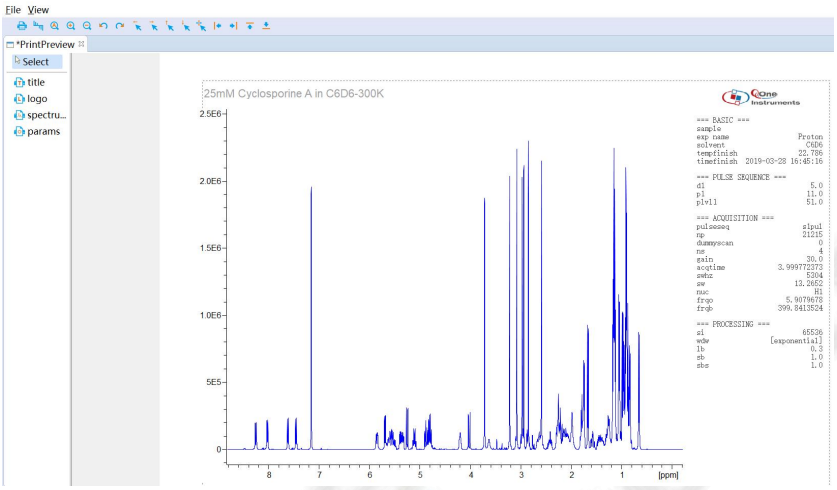


Figure 9.1 Print page

The top left corner is the descriptive information, which can be filled in or deleted as needed.

The toolbar of the printing interface includes an array manager, a spectrum display and adjustment tool, which can zoom the spectrum and move it up and down, left and right. The usage method is consistent with the toolbar of the software main interface.

Place the mouse in the spectral area. The right mouse button menu is shown in Figure 9.2. You can check whether to display peak search results (Show peaks), integral lines (Show integral lines), integral labels (Show integral labels), electronic signature (Show electronic signature), etc. You can also open the “Properties” interface to set the color and line width of the spectrum. Selecting “Enter Zoom” will enter the zoom mode to zoom in or out of the spectrum. Selecting “Delete” will delete the spectrum.

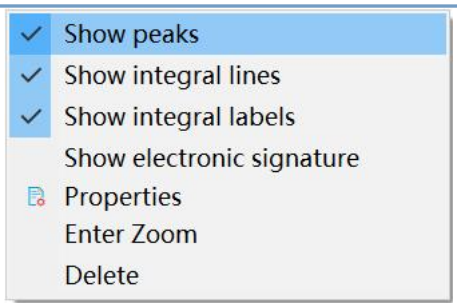


Figure 9.2 Right mouse menu in spectrum area

Place the mouse in the parameter area. The right mouse button menu is shown in Figure 9.3. Click “Edit” to open the parameter editing dialog box (Figure 9.4). You can select the acquiring parameters and processing parameters displayed on the printing interface. The check mark indicates that the parameters are displayed in the parameter area. Selecting “Delete” will delete the parameters in this area. Selecting “Load PS Parameters” will retrieve the pulse sequence related parameters and display them in the parameter area.

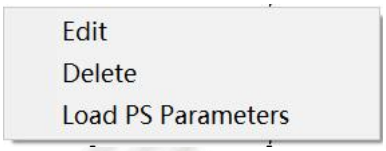


Figure 9.3 Right mouse menu in parameter area

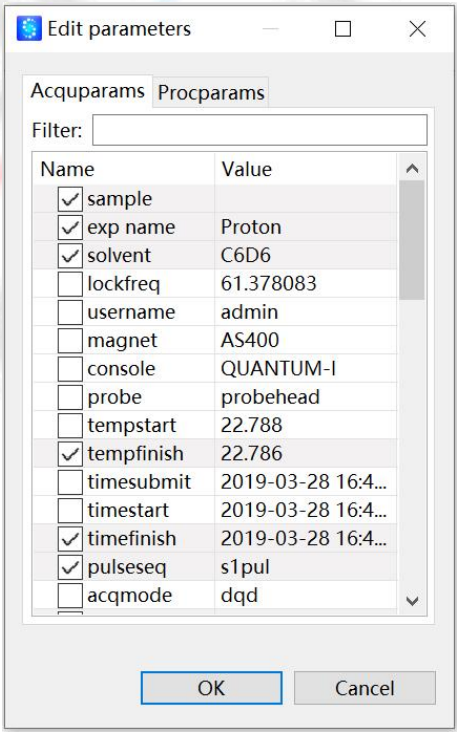


Figure 9.4 Parameter edit dialog

On the print page, click on the menu bar **View** and select “Actual Size/Adaptive Size” to adjust the page display size. Actual Size is shown in Figure 9.5 and Adaptive Size is shown in Figure 9.6.

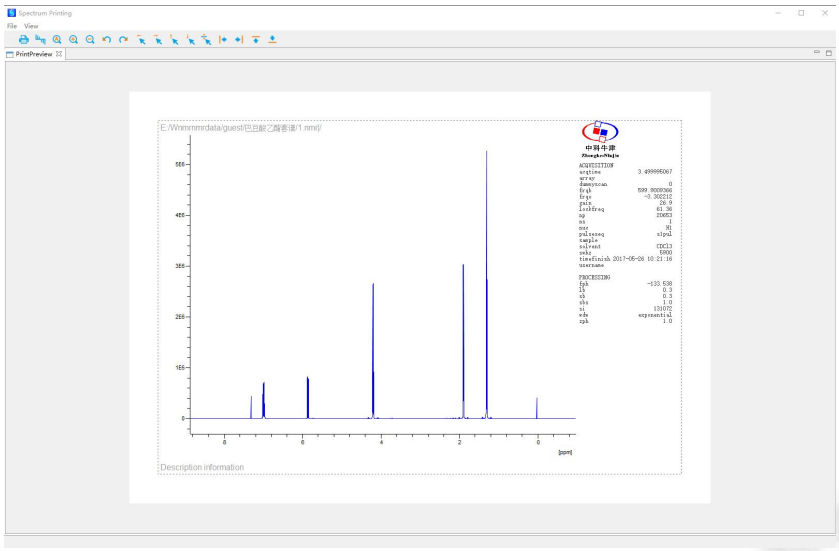


Figure 9.5 Actual size

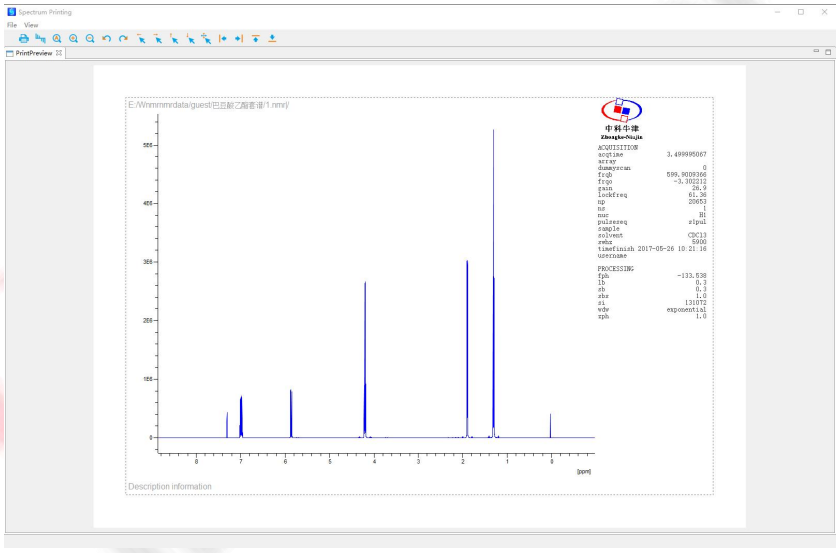


Figure 9.6 Adapt to window size

Clicking on the menu bar **View** to select **Page Setup** will bring up the page setup dialog shown in Figure 9.7.

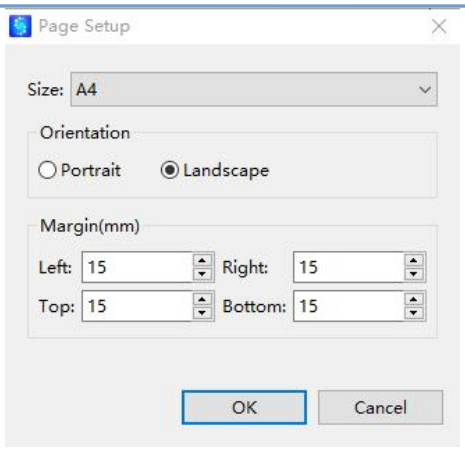


Figure 9.7 Page setup dialog

Size: Paper size, optional A3, A4 or A5.

Portrait: Vertical, as shown in Figure 9.8.

Landscape: Horizontal, as shown in Figure 9.9.

Margin (mm): Page margin, you can set up the left, right, top and bottom margins.

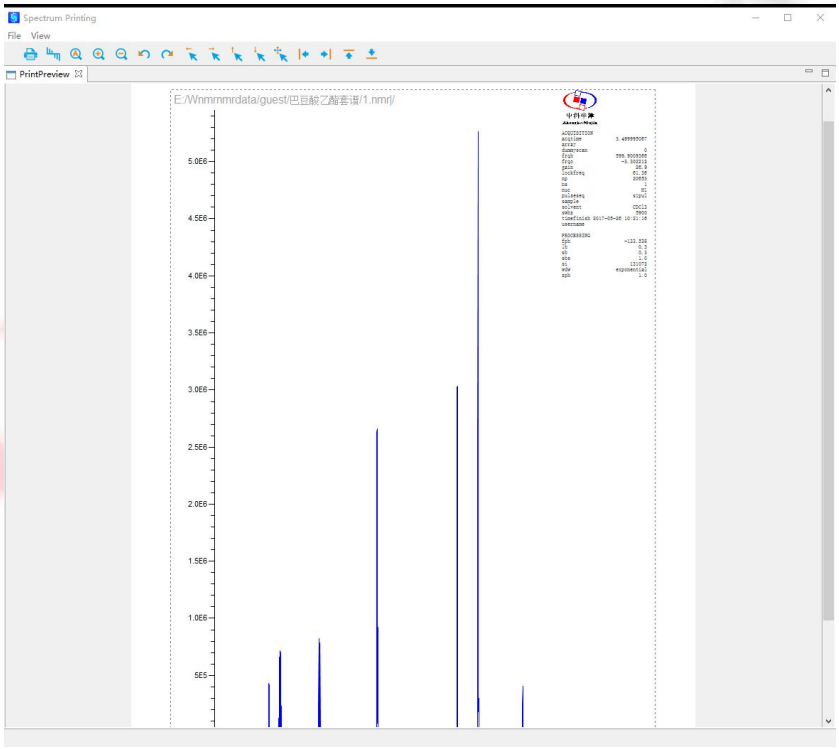


Figure 9.8 Vertical display

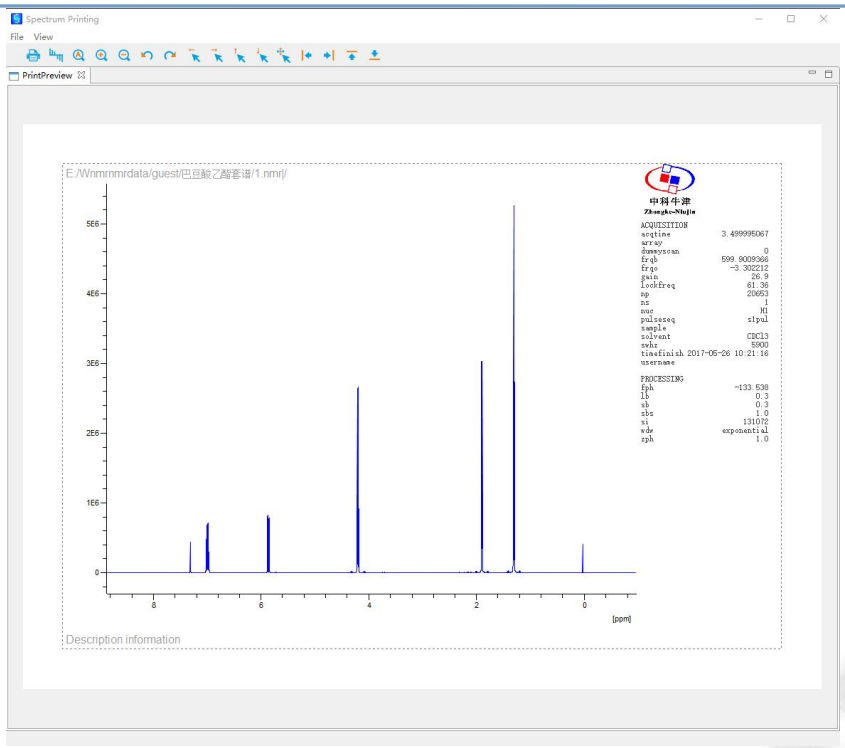


Figure 9.9 Horizontal display

Click **View** to select “Preferences” and the settings dialog box shown in Figure 9.10 will pop up. You can set colors, fonts, etc. as needed.

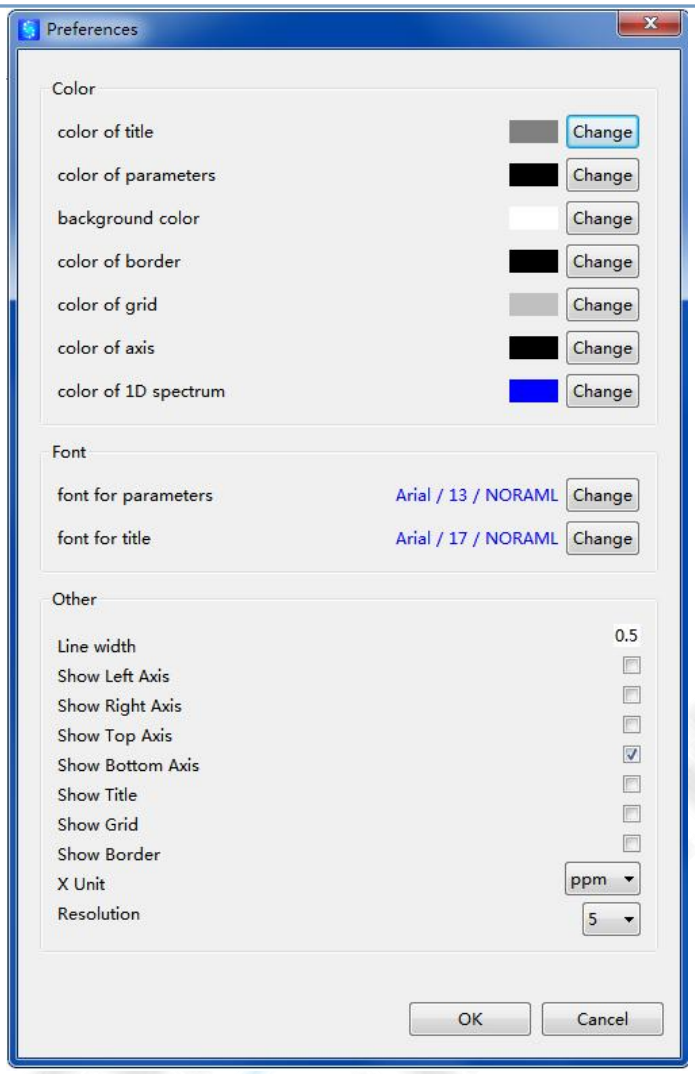


Figure 9.10 Settings dialog

9.2 Document Output

In the print interface, clicking on the menu bar **File**, as shown in Figure 9.11. There are the following Export options, which can be exported to image, pdf document, vector image (SVG) and other formats.

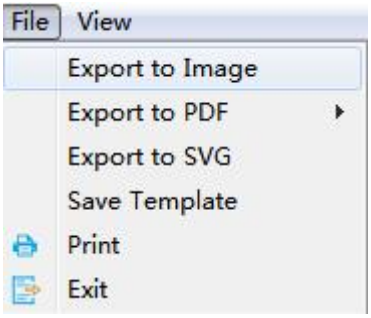


Figure 9.11 File options

Select “Export to Image” to export the picture format file, and the picture resolution selection dialog box will pop up first. As shown in Figure 9.12, the resolution parameter dpi can be set to 120, 360 or 600. The larger the value, the clearer the picture.

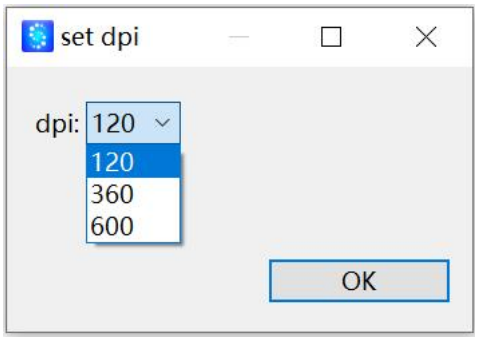


Figure 9.12 Picture resolution selection dialog

After selecting the resolution dpi, click the **OK** button. The picture save dialog box (Figure 9.13) pops up, and the output picture types include bmp, jpeg, and png. Users can choose the storage path, file name, and save type according to their needs, and click the Save button to complete the picture output operation.

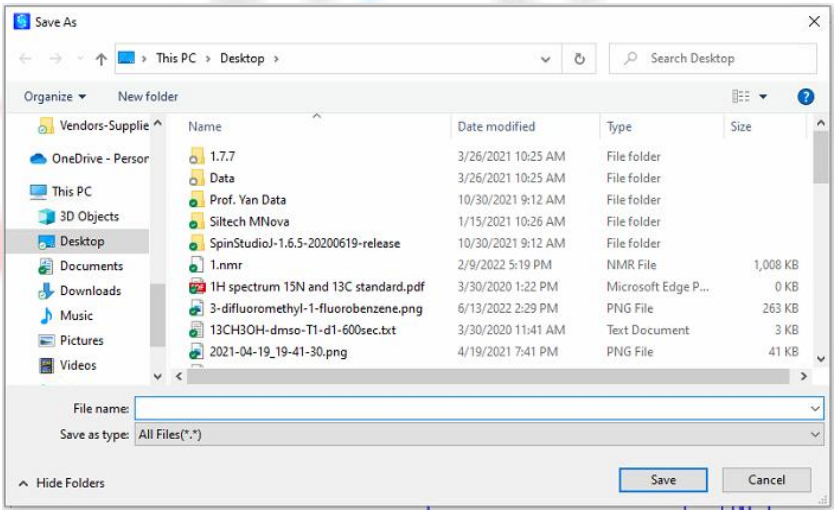


Figure 9.13 Save picture dialog

Select “Export to PDF” to export pdf documents, as shown in Figure 9.14. You can choose “Export to PDF (Default)” or “Export to PDF (High-resolution)”, and there is a difference in resolution between the two. After selection, the dialog for exporting pdf documents will pop up (Figure 9.15). The default file name is

"1_Year_month_day_hour_minute_ second". Users can also choose their own storage path, modify the file name, and click the **Save** button to complete the pdf document output operation.

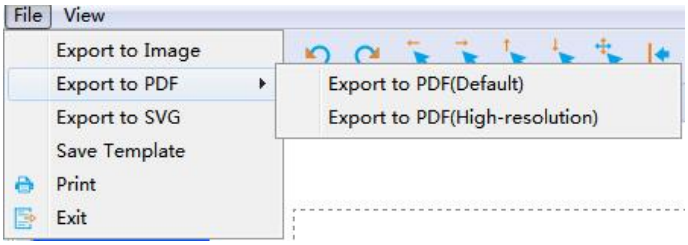


Figure 9.14 Selecting dialog for exporting pdf documents

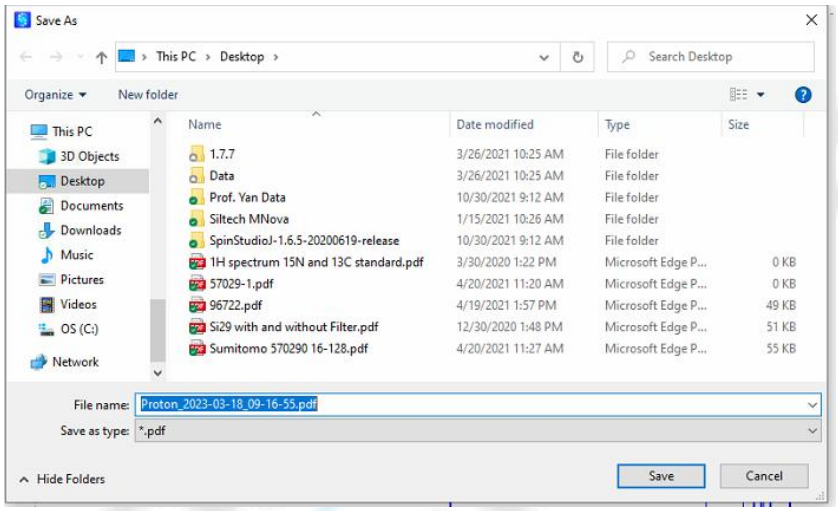


Figure 9.15 Dialog for saving pdf document

Select "Export to SVG" to export the vector diagram, and the dialog box for saving vector diagram (Figure 9.16) will pop up. The default file name is "1_Year_month_day_hour_minute_ second". Users can also choose the storage path and modify the file name independently. Click the **Save** button to complete the vector diagram output operation.

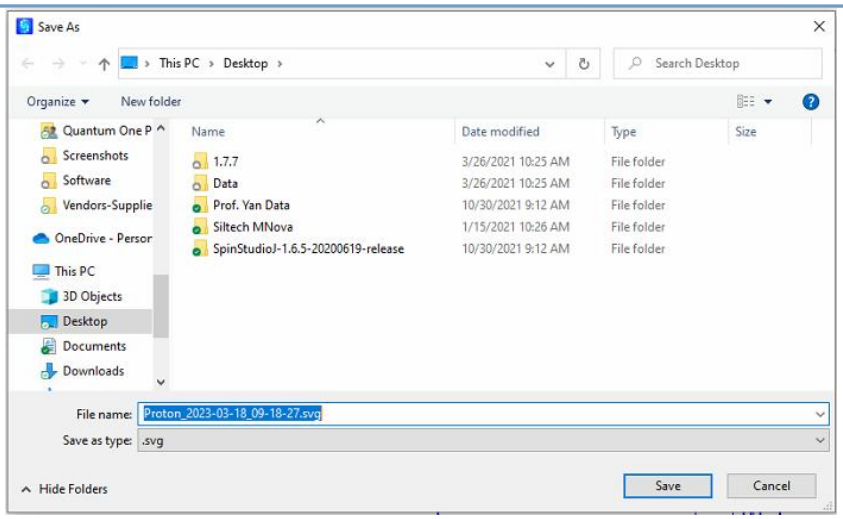


Figure 9.16 Dialog for saving the vector diagram

9.3 Save printing template

Click File on the menu bar of the print interface, as shown in Figure 9.11. Select Save Template, and a save print template dialog box will pop up (Figure 9.17). You can save the description information, the location, size, font, font size, and parameter names and parameter grouping information in the parameter area of the current print interface. The print template will be saved in the system print template directory, and users can directly switch to use it when selecting the print template in the processing parameter interface.

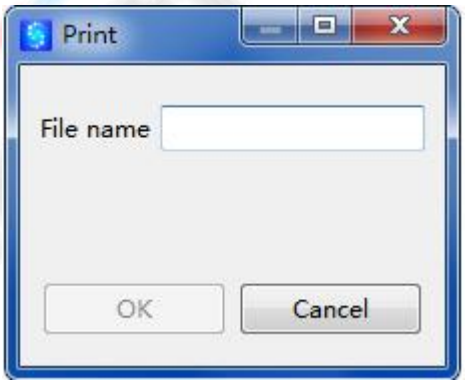


Figure 9.17 Dialog for saving printing template

9.4 Printing

In the print interface, click on the menu bar **File** to select “Print”, or click on the button in the toolbar to open the print dialog box as shown in Figure 9.18.

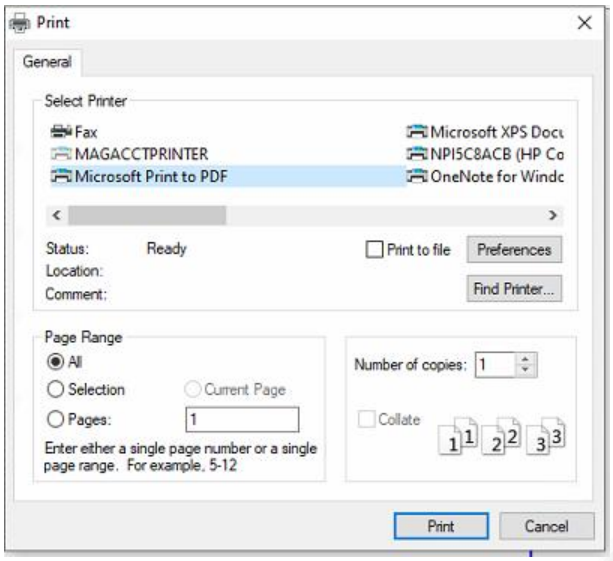


Figure 9.18 Print dialog box

Users can choose printers according to their own needs, page range settings, number of copies settings, etc. After setting up, click on “Print”.

If the printer is not installed, you can also select “Microsoft Print to PDF” in Figure 9.18 to export the pdf format file, as shown in Figure 9.19.

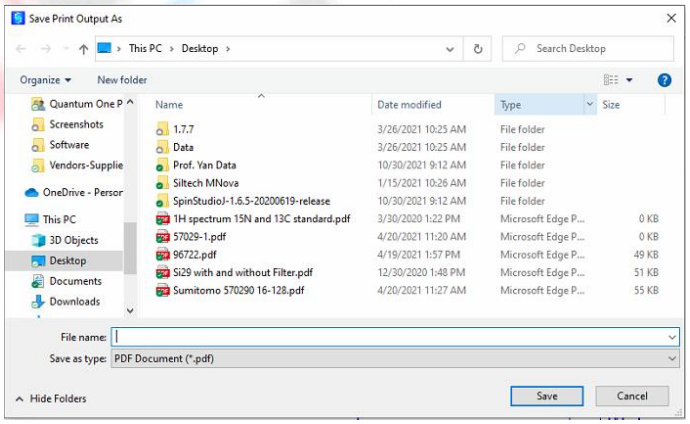


Figure 9.19 Export .pdf file