

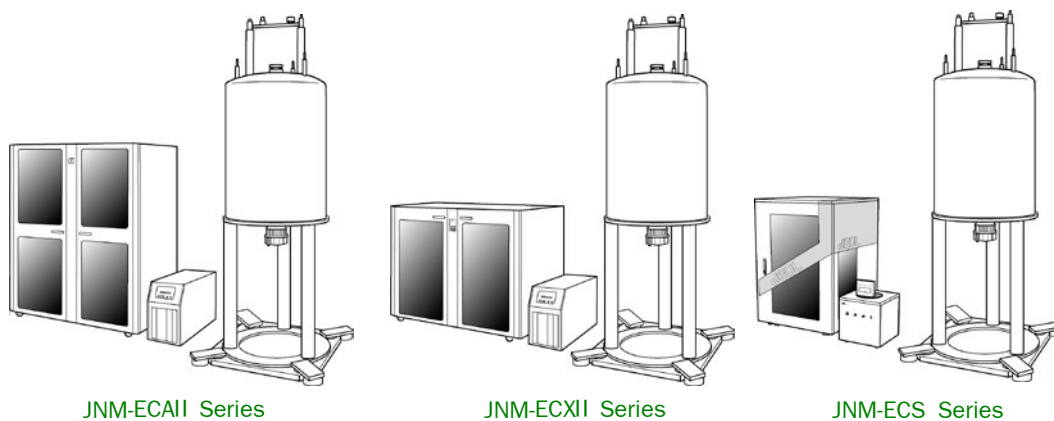
JNM-ECA II Series JNM-ECX II Series JNM-ECS Series

TUTORIAL MANUAL

For the proper use of the instrument, be sure to read this instruction manual. Even after you read it, please keep the manual on hand so that you can consult it whenever necessary.

JNM-ECA II Series JNM-ECX II Series JNM-ECS Series

TUTORIAL MANUAL



This manual is designed for new users of the JNM-ECA II, JNM-ECX II or JNM-ECS Series FT NMR system who want to perform 1D or 2D NMR measurement. It explains the basic procedures for operating the system. For details of individual operations, refer to the on-line "User's Manual".

Please be sure to read this instruction manual carefully, and fully understand its contents prior to the operation or maintenance for the proper use of the instrument.

NOTICE

- This instrument generates, uses, and can radiate the energy of radio frequency and, if not installed and used in accordance with the instruction manual, may cause harmful interference to the environment, especially radio communications.
- The following actions must be avoided without prior written permission from JEOL RESONANCE Inc. or its subsidiary company responsible for the subject (hereinafter referred to as "JEOL RESONANCE"): modifying the instrument; attaching products other than those supplied by JEOL RESONANCE; repairing the instrument, components and parts that have failed, such as replacing pipes in the cooling water system, without consulting your JEOL RESONANCE service office; and adjusting the specified parts that only field service technicians employed or authorized by JEOL RESONANCE are allowed to adjust, such as bolts or regulators which need to be tightened with appropriate torque. Doing any of the above might result in instrument failure and/or a serious accident. If any such modification, attachment, replacement or adjustment is made, all the stipulated warranties and preventative maintenances and/or services contracted by JEOL RESONANCE or its affiliated company or authorized representative will be void.
- Replacement parts for maintenance of the instrument functionality and performance are retained and available for seven years from the date of installation. Thereafter, some of those parts may be available for a certain period of time, and in this case, an extra service charge may be applied for servicing with those parts. Please contact your JEOL RESONANCE service office for details before the period of retention has passed.
- In order to ensure safety in the use of this instrument, the customer is advised to attend to daily maintenance and inspection. In addition, JEOL RESONANCE strongly recommends that the customer have the instrument thoroughly checked up by field service technicians employed or authorized by JEOL RESONANCE, on the occasion of replacement of expendable parts, or at the proper time and interval for preventative maintenance of the instrument. Please note that JEOL RESONANCE will not be held responsible for any instrument failure and/or serious accident occurred with the instrument inappropriately controlled or managed for the maintenance.
- After installation or delivery of the instrument, if the instrument is required for the relocation whether it is within the facility, transportation, resale whether it is involved with the relocation, or disposition, please be sure to contact your JEOL RESONANCE service office. If the instrument is disassembled, moved or transported without the supervision of the personnel authorized by JEOL RESONANCE, JEOL RESONANCE will not be held responsible for any loss, damage, accident or problem with the instrument. Operating the improperly installed instrument might cause accidents such as water leakage, fire, and electric shock.
- The information described in this manual, and the specifications and contents of the software described in this manual are subject to change without prior notice due to the ongoing improvements made in the instrument.
- Every effort has been made to ensure that the contents of this instruction manual provide all necessary information on the basic operation of the instrument and are correct. However, if you find any missing information or errors on the information described in this manual, please advise it to your JEOL RESONANCE service office.
- In no event shall JEOL RESONANCE be liable for any direct, indirect, special, incidental or consequential damages, or any other damages of any kind, including but not limited to loss of use, loss of profits, or loss of data arising out of or in any way connected with the use of the information contained in this manual or the software described in this manual. Some countries do not allow the exclusion or limitation of incidental or consequential damages, so the above may not apply to you.
- This manual and the software described in this manual are copyrighted, all rights reserved by JEOL RESONANCE and/or third-party licensors. Except as stated herein, none of the materials may be copied, reproduced, distributed, republished, displayed, posted or transmitted in any form or by any means, including, but not limited to, electronic, mechanical, photocopying, recording, or otherwise, without the prior written permission of JEOL RESONANCE or the respective copyright owner.
- When this manual or the software described in this manual is furnished under a license agreement, it may only be used or copied in accordance with the terms of such license agreement.

Copyright © 2013 JEOL RESONANCE Inc.

- In some cases, this instrument, the software, and the instruction manual are controlled under the "Foreign Exchange and Foreign Trade Control Law" of Japan in compliance with international security export control. If you intend to export any of these items, please consult JEOL RESONANCE. Procedures are required to obtain the export license from Japan's government.

TRADEMARK

- Windows is a trademark of Microsoft Corporation.
- All other company and product names are trademarks or registered trademarks of their respective companies.

MANUFACTURER

JEOL RESONANCE Inc. 1-2, Musashino 3-chome, Akishima, Tokyo 196-8558 Japan
Telephone: +81-42-542-2234 URL: <http://j-resonance.com/>

Note: For servicing and inquiries, please contact your service office.

CONTENTS

SAFETY PRECAUTIONS

1 PREPARATION FOR MEASUREMENT

1.1	STARTING AND EXITING THE DELTA PROGRAM	1-1
1.2	CONNECTING TO AND DISCONNECTING FROM A SPECTROMETER	1-2
1.3	SAMPLE PREPARATION	1-4
1.3.1	Check Prior to Sample Insertion	1-4
1.3.2	Registration of a Sample to the Delta.....	1-7
1.3.3	Sample Loading, NMR Lock, and Automatic Resolution Adjustment	1-8

2 PRACTICE OF AUTOMATIC MEASUREMENT

2.1	STANDARD METHOD OF AUTOMATIC MEASUREMENT	2-1
2.2	PREPARING FOR MEASUREMENT AND EXECUTION	2-2
2.2.1	Registration of a New Sample and Deletion	2-2
2.2.2	Adding, Deleting and Editing the Spectrometer Parameters	2-3
2.2.3	Creating and Deleting a Job	2-4
2.3	PRECAUTIONS	2-7

3 1D NMR MEASUREMENT

3.1	SETTING 1D NMR MEASUREMENT CONDITIONS	3-1
3.1.1	Setting 1H Measurement Conditions	3-1
3.1.2	Setting 13C Measurement Conditions.....	3-3
3.2	STARTING AND ENDING MEASUREMENT	3-5
3.3	DATA TRANSFER FROM THE SPECTROMETER.....	3-6
3.4	1D NMR DATA PROCESSING.....	3-8
3.4.1	Automatic Baseline Correction	3-9
3.4.2	Automatic Peak Detection and Automatic Integration	3-10
3.4.3	Plotting.....	3-10
3.4.4	Changing the Display Range.....	3-11

4 2D NMR MEASUREMENT

4.1	SETTING 2D NMR MEASUREMENT CONDITIONS	4-1
4.1.1	HH COSY Measurement.....	4-1
4.1.2	HMQC Measurement	4-4
4.1.3	Setting the Observation Range Through the 1D Data	4-5
4.2	STARTING AND ENDING MEASUREMENT	4-8
4.3	DATA PROCESSING OF 2D NMR.....	4-9
4.3.1	Displaying Data Using 2D Viewer.....	4-10
4.3.2	Setting the Conditions for Displaying Contour Lines	4-11
4.3.3	Pasting 1D NMR Data.....	4-12
4.3.4	Plotting.....	4-14
4.3.5	Changing the Display Ranges	4-14



5 QUEUING

5.1	COMMAND FOR STARTING MEASUREMENT	5-1
5.2	QUEUING	5-2

6 SAVING AND LOADING DATA

6.1	SAVING NMR DATA.....	6-1
6.1.1	Saving the measured data (FID).....	6-1
6.1.2	Saving Processed Data.....	6-1
6.2	LOADING NMR DATA	6-4



WARNING for Installation



- Do not attempt to install the instruments by yourself.
Installation work requires professional expertise and JEOL is responsible for the installation of the instruments and related attachments purchased from JEOL.
Consult your JEOL service office.



WARNING



- Be sure to wear protective gloves, protective glasses and masks when handling a sample tube containing a poisonous sample.
If the sample tube breaks, the use of gloves allows you to avoid contact with the sample.

1

PREPARATION FOR MEASUREMENT

1.1	STARTING AND EXITING THE DELTA PROGRAM	1-1
1.2	CONNECTING TO AND DISCONNECTING FROM A SPECTROMETER.....	1-2
1.3	SAMPLE PREPARATION	1-4
1.3.1	Check Prior to Sample Insertion	1-4
1.3.2	Registration of a Sample to the Delta.....	1-7
1.3.3	Sample Loading, NMR Lock, and Automatic Resolution Adjustment	1-8



1.1 STARTING AND EXITING THE DELTA PROGRAM

This section assumes that the data system has started normally, and that the login operation is complete.

☞ For starting the data system and login, refer to the separate manual on the workstation.

■ Starting the Delta program

- ◆ Double-click the Delta  icon on the screen.
The Delta program starts, and the **Delta Console** window opens.

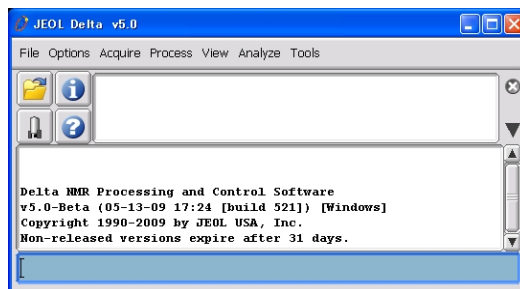


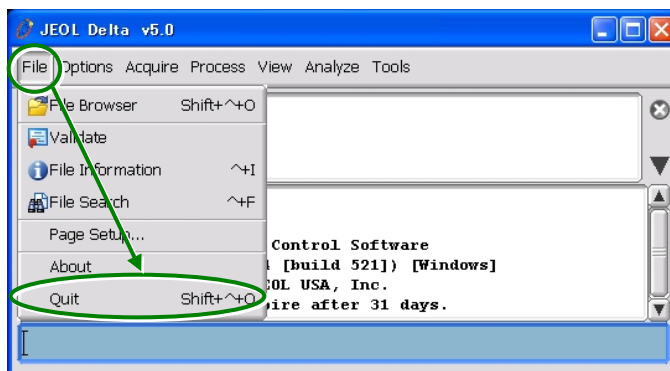
Fig. 1.1 Delta Console window

■ Ending the Delta program

After measurement or NMR data processing is complete, terminate the Delta program as follows.

1. Select **File – Quit** in the **Delta Console** window.

First, point to **File** in the menu bar in the **Delta Console** window, and press the left mouse button. A pull-down menu appears. Then, move the mouse to highlight **Quit** in the pull-down menu, and click the left mouse button there.



The **Confirm** dialog box opens.

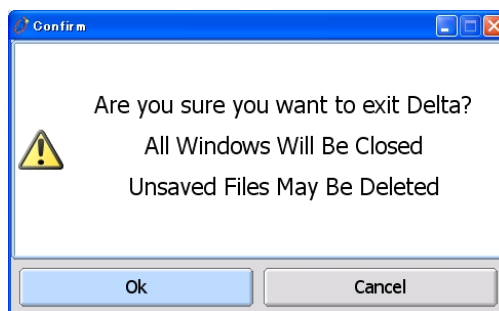


Fig. 1.2 Confirm dialog box

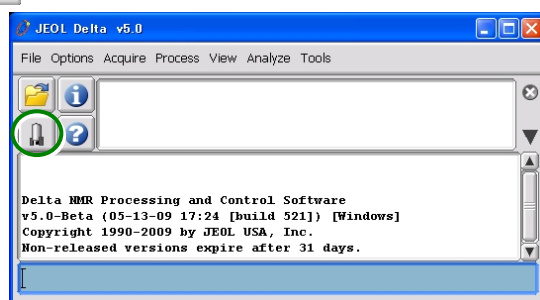
2. Click the **OK** button.
The Delta program ends.

1.2 CONNECTING TO AND DISCONNECTING FROM A SPECTROMETER

The Delta program can be connected with two or more spectrometers through the network in the JNM-ECAII/ECXII/ECS series FT NMR instrument. For this reason, before performing an NMR measurement, it is necessary to connect the Delta program with the spectrometer you will use. After the NMR measurement, disconnect the NMR spectrometer from the Delta program. Here, we will explain how to connect and disconnect the Delta program and the NMR spectrometer.

■ Connecting to the spectrometer

1. Click the  button in the Delta Console window.



The **Spectrometer Control** window opens, and a list of the currently available spectrometers appear in the list box (Fig. 1.3).

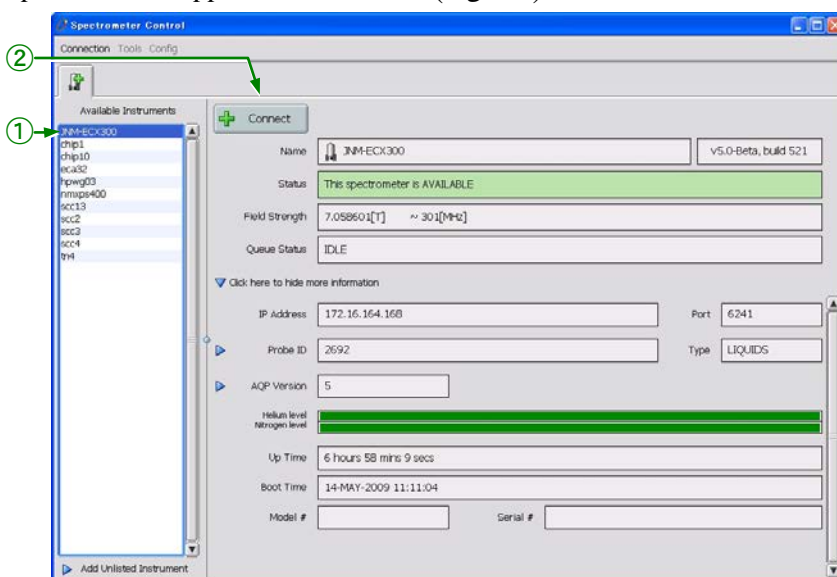
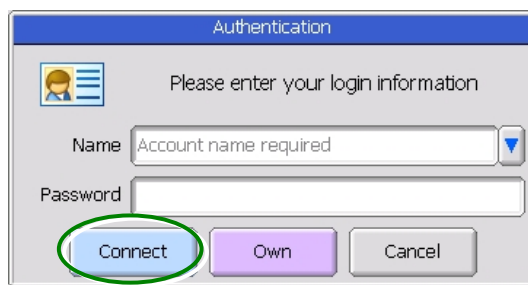


Fig. 1.3 Spectrometer Control window

 The numbers ① and ② are referred to in the following steps.

2. Using the mouse, select the spectrometer you connect to by clicking its name in the list in the Spectrometer Control window (Fig. 1.3 ①).
After clicking a spectrometer in the **Available Instruments** list, if **This spectrometer is AVAILABLE** appears in the **Status** box, the spectrometer is available for connection. If **This spectrometer is BUSY** appears, the spectrometer is currently being used by other users.
3. Click the Connect button (Fig. 1.3 ②).
4. The Authentication dialog box appears as shown below. Enter the user name and the password, and click Connect.



If the connection is carried out correctly, the window is displayed as shown in Fig. 1.4.

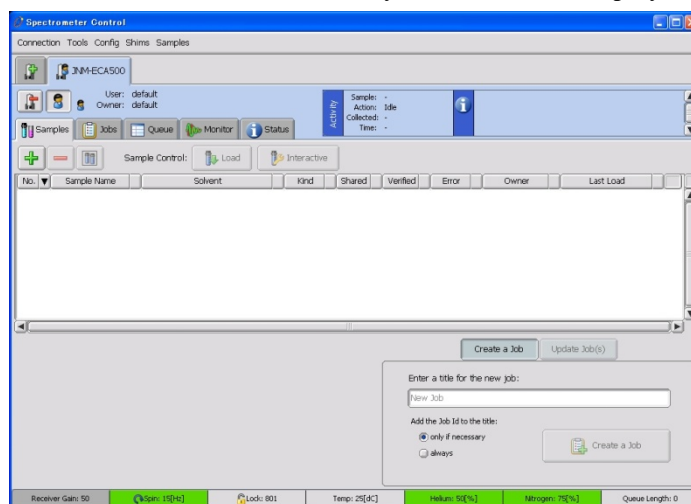


Fig. 1.4 Spectrometer Control window after the spectrometer is connected

■ Disconnecting from the spectrometer

- ◆ After clicking the Disconnect button in the Spectrometer Control window, the confirm message will appear. Click the Yes button.

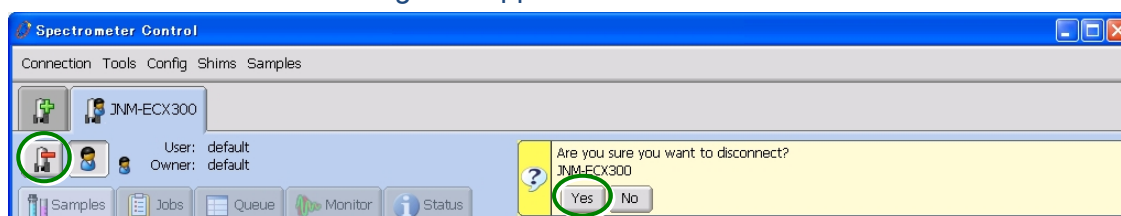


Fig. 1.5 Spectrometer Control window: Spectrometer is connected

When the spectrometer is disconnected, the **Spectrometer Control** window returns to the display shown in Fig. 1.3.

1.3 SAMPLE PREPARATION

This section explains how to insert the sample into the superconducting magnet.

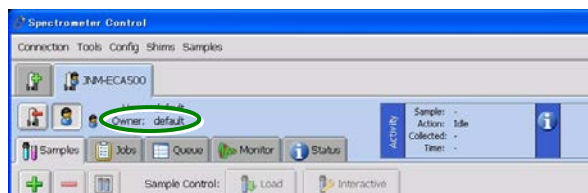
1.3.1 Check Prior to Sample Insertion

Make sure that no other sample is inserted in the superconducting magnet as follows.

1. Connect to the spectrometer according to the procedure of Section 1.2.
2. Click the Owner  button in the Spectrometer Control window.



By this operation, you can have the operating authority. The following display appears to show that you have the operating authority.



3. Click the Samples  tab to open the Samples tab.

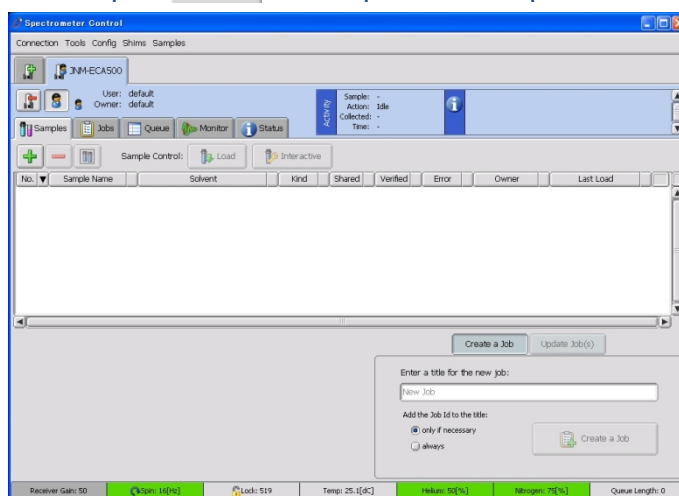


Fig. 1.6 Samples tab

If the sample is not defined as shown in Fig. 1.6, or if the **Sample Load/Eject** button is  or , proceed to ■ **Setting up the sample**. If the **Sample Load/Eject** button is  or , indicating that the sample has been loaded, remove the sample according to the procedure in ■ **Ejecting the sample**.

■ Ejecting the sample

● When an auto sample changer is not installed

If a sample is inserted in the superconducting magnet, remove it by using the following steps.

1. Click the Samples tab in the Spectrometer Control window to open the Samples tab.

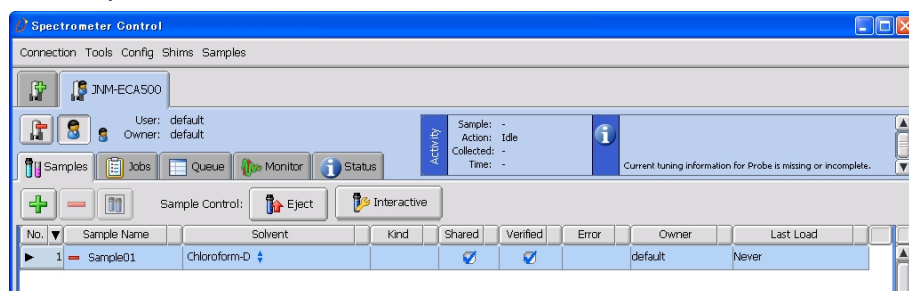
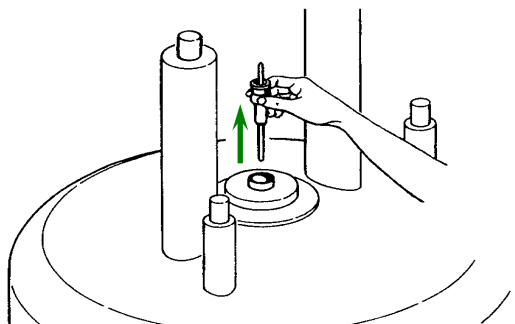


Fig. 1.7 Samples tab

2. Selecting the sample you remove enables you to click the Eject  button. Click the Eject  button.
The sample in the superconducting magnet is ejected.
3. Remove the sample from the insertion port on the superconducting magnet.



● When an auto sample changer is installed

When a sample is inserted into the superconducting magnet, take out the sample according to the following procedure:

1. Open the Samples tab. Selecting the sample you remove enables you to click the Store  button. Click the Store  button.
2. Remove the sample from the slot in the auto sample changer.

■ Setting up the sample

⚠ WARNING



- **Be sure to wear protective gloves, protective glasses and masks when handling a sample tube containing a poisonous sample.**

If the sample tube breaks, the use of gloves allows you to avoid contact with the sample.

1. Set the sample tube containing the sample into the holder.

Since the resolution varies according to the sample volume (height), it is recommended that you standardize the sample volume. The suggested sample height is approximately 4 cm (3.8 to 4.2 cm).

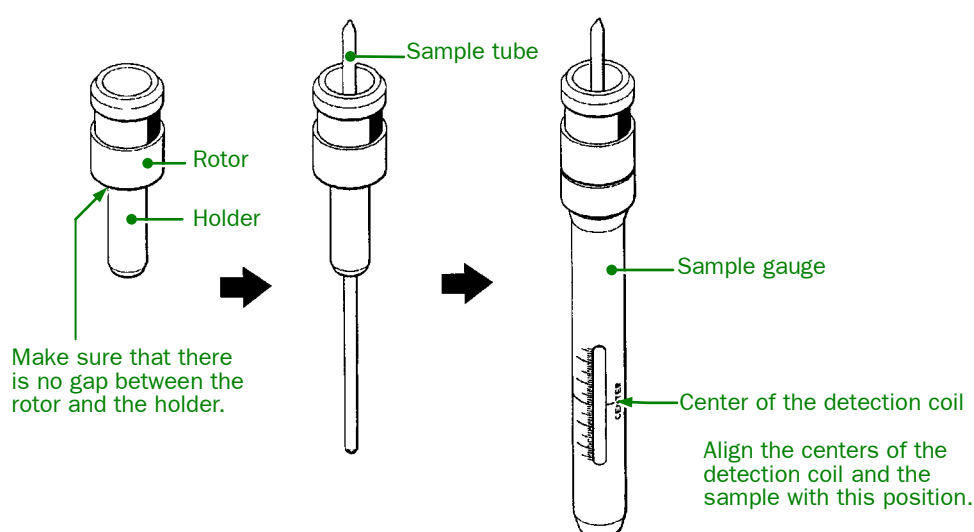


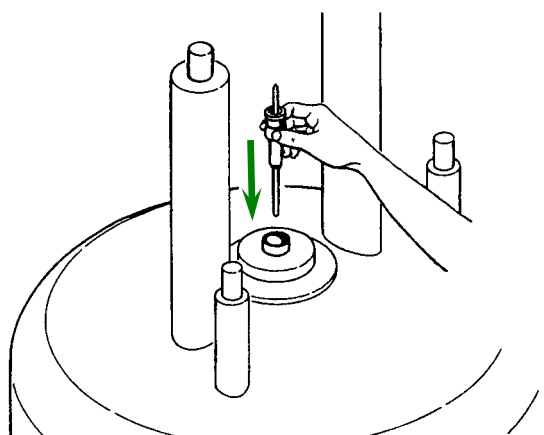
Fig. 1.8 Setting up the holder

2. Make sure that the air is flowing by holding your hand over the sample-insertion port of the superconducting magnet.

✋ If air is not flowing, never insert the sample tube into the insertion port of the superconducting magnet. Another sample may already have been inserted or there might be a problem with the air system.

3. Put the holder with the sample tube into the sample-insertion port of the superconducting magnet and gently release it.

The sample levitates with the compressed air.



1.3.2 Registration of a Sample to the Delta

Define the sample in the **Samples** tab.

■ Opening the Samples tab

- ◆ Just after connecting the spectrometer, the Samples tab is displayed as shown below.

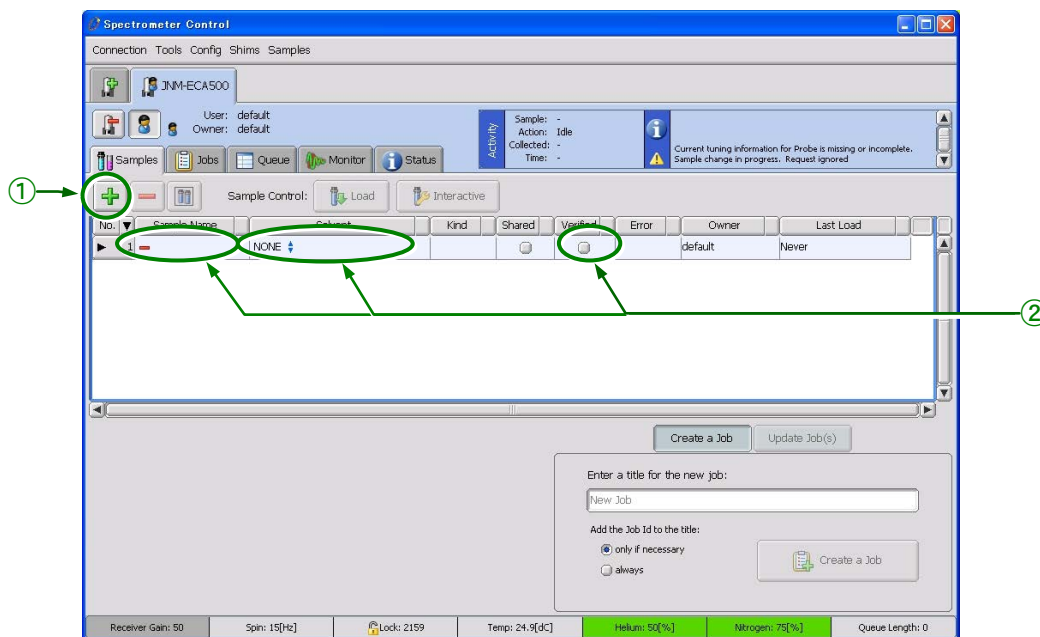


Fig. 1.9 Samples tab

■ Registration of the sample

Register information on the measured sample to the Delta.

1. Click the  button located at the top left in the Samples tab (Fig. 1.9 ①).
2. Enter the sample name in the Sample Name box. After selecting the deuterated solvent that is presently used from the Solvent box, select Verified (Fig. 1.9 ②).

-  If you do not perform this setting, you cannot measure the sample.
-  When the auto sample changer is installed, you also need to specify the number of the **slot**.

1.3.3 Sample Loading, NMR Lock, and Automatic Resolution Adjustment

If you select **No.** in the **Samples** tab and click the **Use Interactive sample controls** button, you can perform sample loading, spinning, NMR lock, and automatic resolution adjustment.

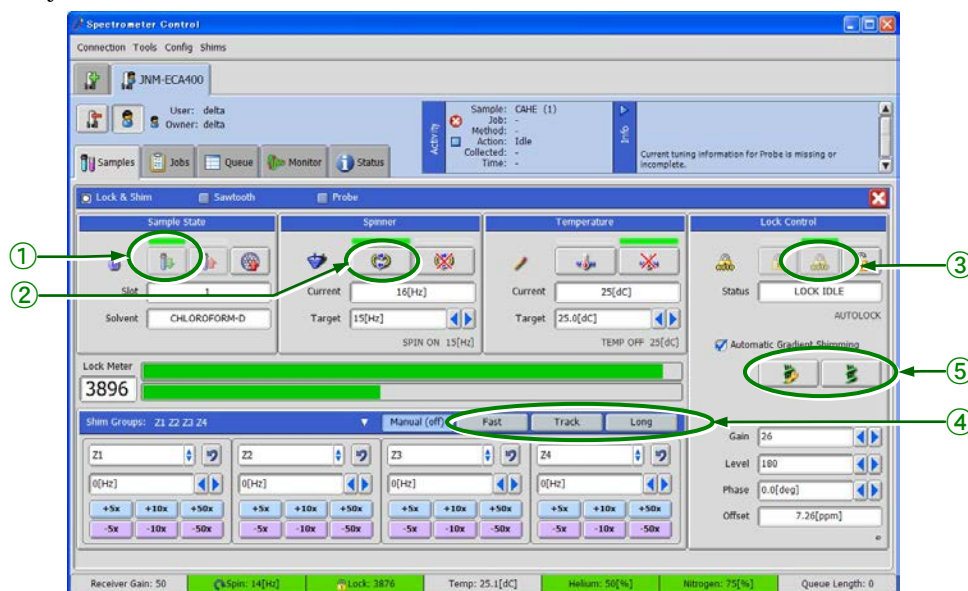


Fig. 1.10 Manual Sample Pane

■ Loading the sample

● When an auto sample changer is not installed

Insert into the superconducting magnet the sample that was set in the sample-insertion port of the superconducting magnet.

- ◆ Click the  button in Manual Sample Pane (Fig. 1.10 ①).

When sample loading is complete, the **Sample State** icon changes as shown below.



● When an auto sample changer is installed

1. Set a sample to the slot of the auto sample changer, and register it.
2. Click the **Samples** tab and select the sample that you want to insert, then click the **Load Sample**  button (Fig. 1.11 ①).

After the sample is carried to the top of the superconducting magnet, it is inserted into the superconducting magnet. When sample loading is complete, the button display changes to , and the color of the **Slot** where the sample was placed changes in the screen.

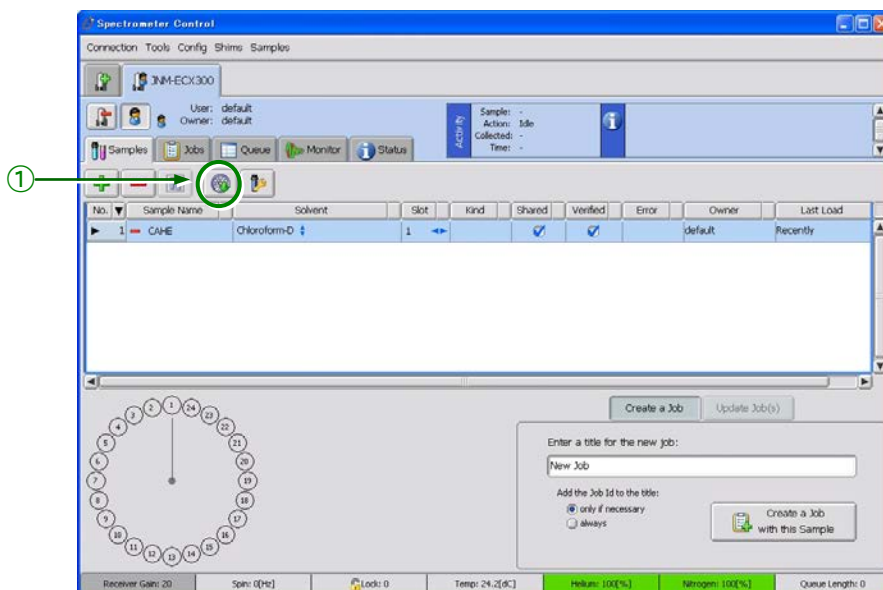


Fig. 1.11 Samples tab (when an auto sample changer is installed)

■ Spinning the sample

Usually, you measure the sample while spinning it. If you measure the sample without spinning, proceed to **■ NMR lock**.

- ◆ Click the  button (Fig. 1.10 ②).

When the sample starts spinning, the toppled top will be upright as shown below.



■ NMR lock

Select the solvent to be used for NMR locking and execute the NMR lock command.

1. Click the  button (Fig. 1.10 ③).

When the NMR lock operates, the message **LOCK IDLE** is displayed in the Status box below the  button.

2. Select Shims—Optimize Lock Phase from the menu at the top of the Spectrometer Control window.

The following message appears in the **Info** dialog box, and optimization of the Lock Phase will be performed.

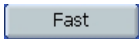
When optimization starts: Optimizing LOCK_PHASE within 60 deg. window

When optimization finishes: Finishing optimizing LOCK_PHASE.

■ Automatic resolution adjustment

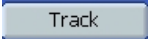
For automatic resolution adjustment, you can select the Automatic shimming (Fig. 1.10 ④) or the Automatic gradient shimming (Fig. 1.10 ⑤).

● Auto shimming

1. Specify a Shim Groups.
2. Clicking either the high-speed auto-shimming  button, or the Auto-shimming  button starts the shim adjustment.

● Automatic gradient shimming

Clicking either the **Gradient Shim Tool**  button, or the **Gradient Shim**  button starts the shim adjustment.

-  For the Gradient Shim, refer to the “Liquid Measurement ” in the separate volume of user’s manuals.
-  Clicking the  button continues to adjust the shim values by tracking them after the specified shims adjustment finishes.

2

PRACTICE OF AUTOMATIC MEASUREMENT

2.1	STANDARD METHOD OF AUTOMATIC MEASUREMENT	2-1
2.2	PREPARING FOR MEASUREMENT AND EXECUTION.....	2-2
2.2.1	Registration of a New Sample and Deletion	2-2
2.2.2	Adding, Deleting and Editing the Spectrometer Parameters	2-3
2.2.3	Creating and Deleting a Job	2-4
2.3	PRECAUTIONS	2-7



2.1 STANDARD METHOD OF AUTOMATIC MEASUREMENT

The Delta program provides the following standard methods for automatic measurement.

Table. 2.1 Automatic measurement methods and measurements

Method	Measurement	Sequence name	Template
Proton	1D ^1H normal measurement	proton.jxp	Standard
Carbon	1D ^{13}C normal measurement	carbon.jxp	Standard
DEPT	Automatic determination of ^{13}C atomic group by means of DEPT	dept.jxp	Standard
COSY	^1H observed homonuclear shift correlation	cosy.jxp	Standard
TOCSY	^1H observed homonuclear shift correlation	tocsy.jxp	Standard
NOESY	^1H observed homonuclear NOE correlation	noesy.jxp	Standard
ROESY	^1H observed homonuclear ROE correlation	roesy.jxp	Standard
HMQC	^1H observed heteronuclear shift correlation	hmqc.jxp	Standard
HSQC	^1H observed heteronuclear shift correlation	hsqc.jxp	Standard
HMBC	^1H observed longrange heteronuclear shift correlation	hmhc.jxp	Standard

2.2 PREPARING FOR MEASUREMENT AND EXECUTION

The spectrometer parameters used to execute automatic measurement can be set for each sample and are treated as a part of the sample parameters (attributes). This section first explains how to set the spectrometer parameters for each sample.

2.2.1 Registration of a New Sample and Deletion

■ Registration of a new sample

For executing automatic measurement, registration of a sample is required the same as it is for a normal measurement.

1. Click the  button above the **Sample** tab.
A blank column for the sample is created.
2. Specify the sample name, solvent, slot number (when using the Auto sample changer) and select the **shared** box if desired. Then, place a check mark into the **Verified** box.

■ Deleting the sample

1. Select the unnecessary sample.
2. Click the  button above the **Sample** tab.

The sample row is deleted.

 If multiple samples are selected using the **Shift**, **Ctrl** keys, you can delete the samples as a batch. Also, you can delete the sample by clicking the  button on the left side of each sample name.

 When the sample is in use by the **Job** in the **Job** tab or the **Queue**, you cannot delete it. In this case, the  mark is displayed on the left side of the sample name.

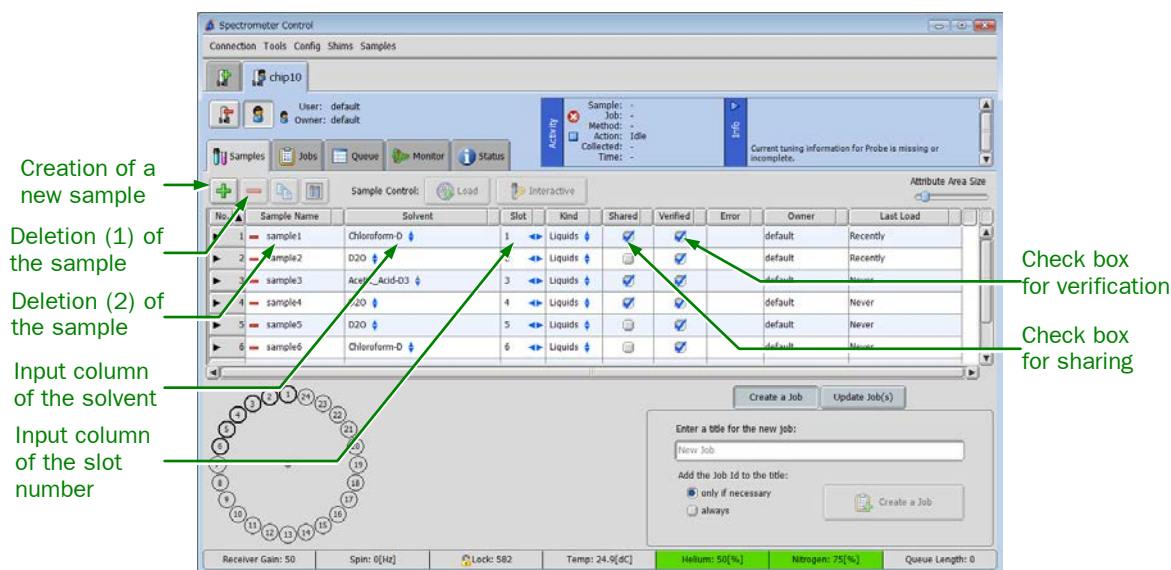


Fig. 2.1 Name of each part in the Samples tab

2.2.2 Adding, Deleting and Editing the Spectrometer Parameters

You can add, delete and edit the spectrometer parameters in the **Samples** tab. Click the ► button on the left side of the sample name to open and close the column for editing parameters.

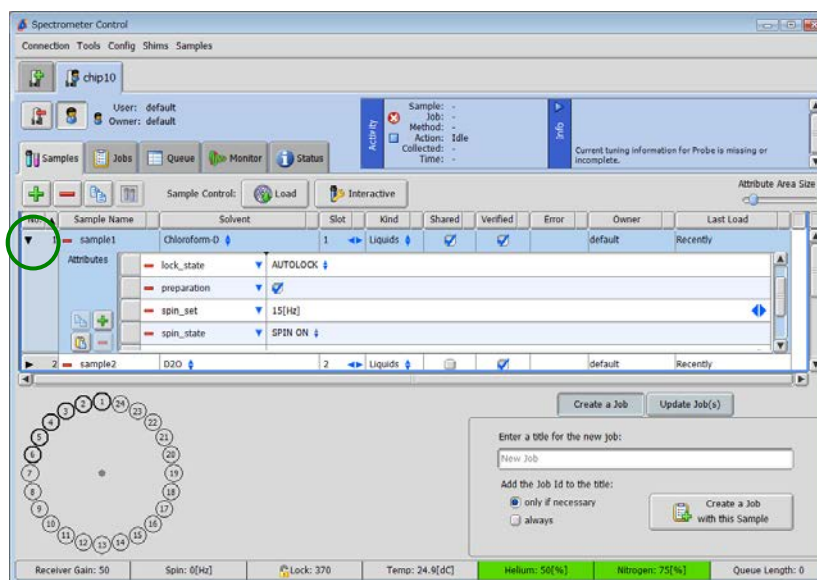


Fig. 2.2 Column for editing the parameters in the Sample tab

◆ Editing the default sample parameter.

The sample parameters have the defaults for each kind of sample (**Kind**). The following table shows the parameters and defaults that are to be set when the **Kind** is **Liquid**. In addition, the software sets the **Kind** automatically.

✍ Do not change the **Kind** of the sample. If changed, the automatic measurement may operate abnormally.

Parameter	Description	Default
gradient shim	Execution of the automatic gradient shimming	TRUE
Lock_state	State of the NMR lock	Autolock
spin_set	Spinning speed	15 Hz
spin_state	Spinning ON/OFF	SPIN ON
temp_set	VT target temperature	25 dC
temp_state	VT ON/OFF	TEMP OFF
preparation	With/without presetting before measurement	TRUE

By editing the defaults parameter shown above, you can change the spectrometer parameters that will be set to execute the automatic measurement.

✍ Do not delete the defaults of the parameters as unexpected operations may be caused in some cases. If you delete it accidentally, remake the sample registration or add the deleted parameter by supplementing it as described later.

2.2.3 Creating and Deleting a Job

1. Select samples.

Select the desired sample and click the **Create a Job with these Samples** button to create a job. At this time, you can select two or more samples by sequential selection with **ctrl** + left-click or selection of the range specified with **shift** + left-click.

The highlighted area in blue has been already selected.

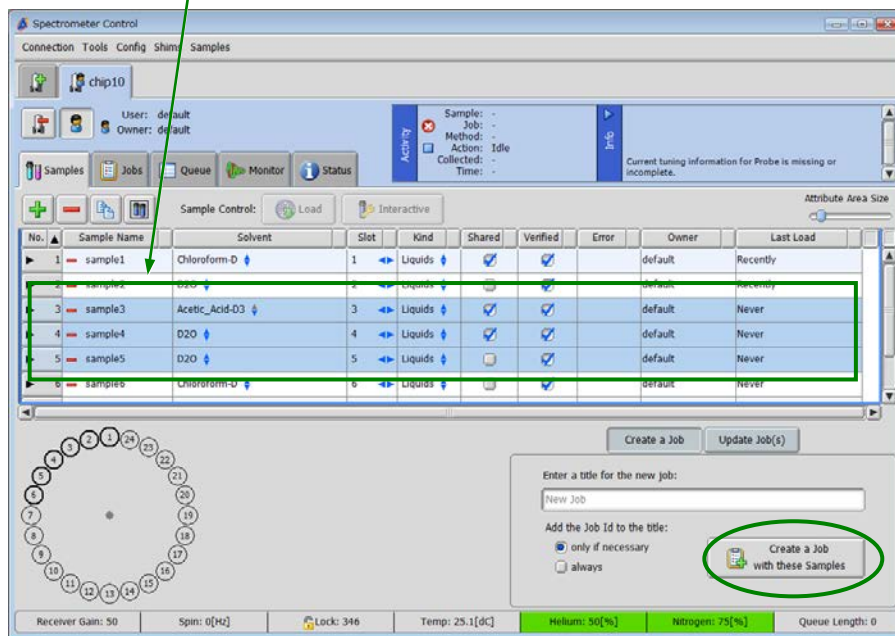


Fig. 2.3 Selecting samples to create a job

When creating a Job, you can name it by entering the Job name into the input box. If you do not enter anything, the name is automatically created. You can also change the Job name by double-clicking the Job in the Job list in the **Jobs** tab after creating the Job.

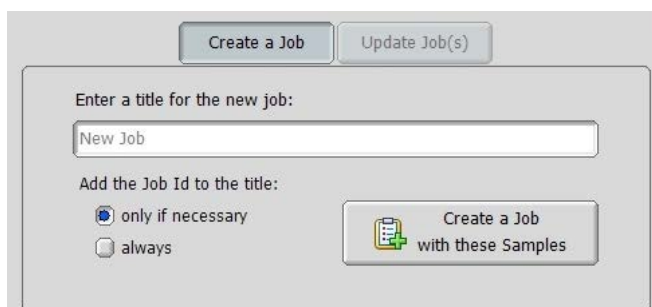


Fig. 2.4 Input box of a Job name

You can delete unnecessary Jobs or Jobs that have been created by mistake by using the  button below the Job list. You can also delete multiple Jobs selected by using the **Shift** and **Ctrl** keys as a batch.

2. Adding a method to the job.

Clicking the **Create a Job with these Samples** button opens the **Jobs** tab automatically. The newly created Job appears in the **Job List** at the left edge of the **Jobs** tab. Also the sample that was selected in the previous section is displayed in the **sample box**.

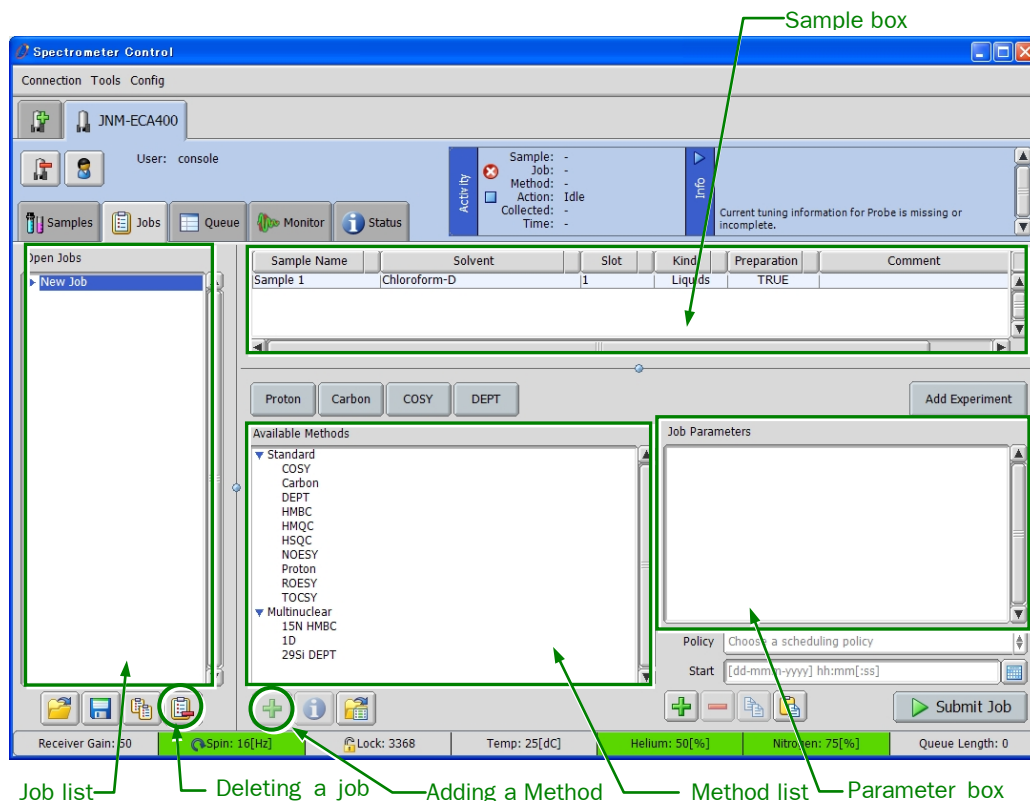


Fig. 2.5 Name and layout of each part in the Jobs tab

For adding a Method to a Job, while selecting the desired Job (highlighted in blue) in the **Open Jobs** List, select the desired item in the **Available Methods** list to highlight in blue, and click the **+** button below the **Available Methods** list.

You can add multiple Methods to a Job. In this case, the added Methods are executed one after another to the sample that is assigned to the Job (displayed in the Sample box when selecting the Job).

Also, if two or more samples are assigned to a Job, after sequential execution of all the methods is carried out for a certain sample, these executions are repeated in the following sample.

3. Setting Method parameters.

When selecting the Method (highlighted in blue) included in the Job in the **Job** list, the parameters of the Method appear in the parameter box. By changing these parameters, you can change the Method parameters.

☞ For the editable parameters, refer to Section 3.2 “How to use each template” in the user’s manual “Automatic Measurement”.

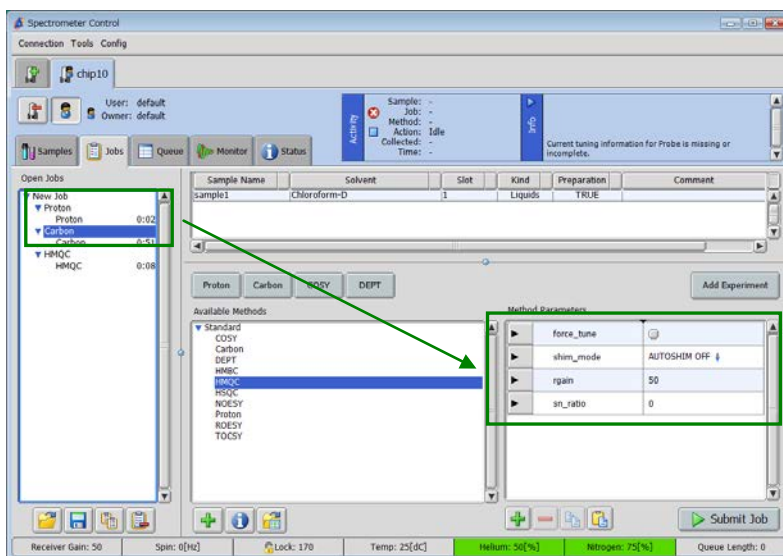


Fig. 2.6 Setting of the Method parameters

4. Setting the measurement parameters.

Selecting the measurement (highlighted in blue) included in the Job in the **Job** list changes the area of **Available Methods** and **Method Parameters** in the window to the area of the **Experiment** tool. Here, you can change the details of the measurement parameters.

☞ For details of how to use the **Experiment** tool, refer to the user's manual "Liquid Measurement".

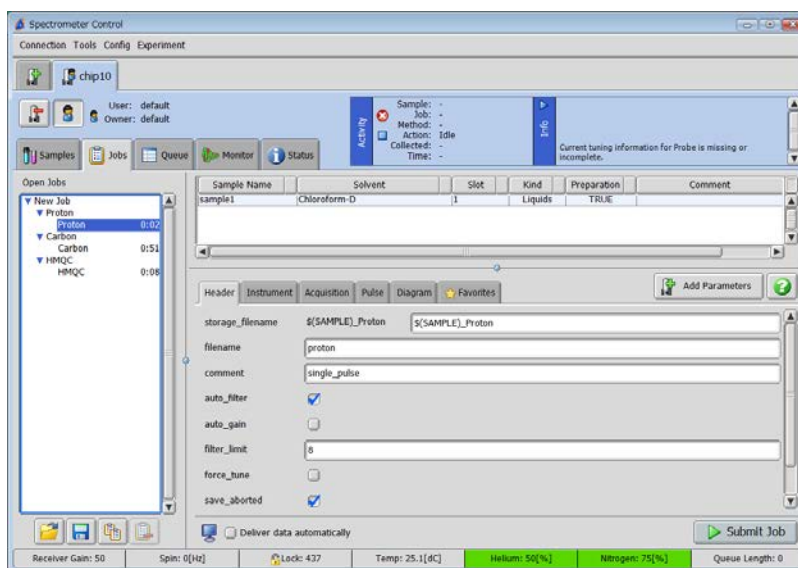
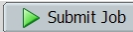


Fig. 2.7 Window for setting measurement parameters

5. Starting measurement.

After setting the Method Parameters for all the Methods, click the  button to register the measurement to the queue.

6. Management of the measurement queue.

The operation such as stopping and canceling the measurement after starting the automatic measurement are the same as that for a normal measurement.

2.3 PRECAUTIONS

● Specifying an NMR lock solvent

The error that occurs most frequently in automatic measurements is an NMR lock error. To prevent NMR lock errors, make sure that the solvent name is entered correctly.

● Automatic resolution adjustment

If the volumes of the samples to be measured are greatly different from the standard volume, or solid substances are present in the sample, the automatic resolution adjustment takes a long time.

Under these conditions, the appropriate resolution may not be achieved.

To prevent these problems, prepare the samples so they are about 4 cm, and align the bottom of the sample tube with that of the sample gauge.

● Probe tuning

The probe tuning varies, depending on measurement parameters such as solvent and temperature. Be sure to tune the probe, especially before performing a measurement such as DEPT that is sensitive to pulse width or when measuring a small amount of a sample that requires improvement of the S/N ratio.

● Measurement conditions

The measurement conditions for general organic-compound samples are used for automatic measurement. If you measure the sample under special conditions, perform the measurement using individual operations.

● Hard-disk capacity and the data file name

Make sure that there is free space to save data before performing automatic measurement. The results of an automatic measurement are saved in a file with a specified file name to which the method name is added. If the filename already exists, a new file with the version number higher by one will be created.

● Processing condition

In automatic measurement, parameters for data processing are prespecified. If you perform your own specific data processing, perform it later using the data that have been saved in the file.

● Plotting

In automatic measurement, data will be plotted automatically. Therefore, before starting automatic measurement, make sure that chart paper is set in the printer correctly, and that the printer is online.

3

1D NMR MEASUREMENT

Chapter 3 explains the procedures for the basic 1D NMR measurement in the order of measurement, data processing, and plotting.

It is assumed that the Delta system has been connected to the spectrometer, and that the sample setup, sample loading, NMR lock, resolution adjustment and other operations have already been performed prior to measurement. If you are not ready to start measurement, prepare for measurement according to Chapter 1, “Preparation for measurement”.

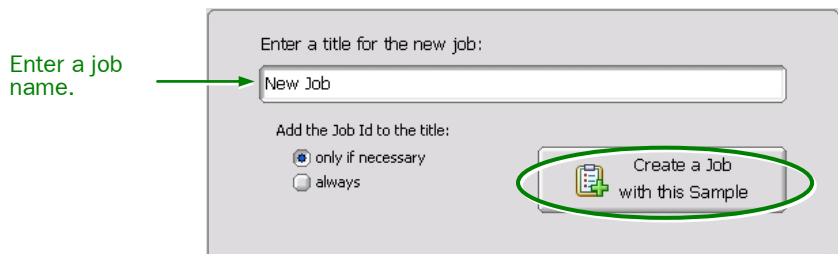
3.1	SETTING 1D NMR MEASUREMENT CONDITIONS.....	3-1
3.1.1	Setting 1H Measurement Conditions	3-1
3.1.2	Setting 13C Measurement Conditions.....	3-3
3.2	STARTING AND ENDING MEASUREMENT	3-5
3.3	DATA TRANSFER FROM THE SPECTROMETER.....	3-6
3.4	1D NMR DATA PROCESSING	3-8
3.4.1	Automatic Baseline Correction	3-9
3.4.2	Automatic Peak Detection and Automatic Integration	3-10
3.4.3	Plotting.....	3-10
3.4.4	Changing the Display Range.....	3-11



3.1 SETTING 1D NMR MEASUREMENT CONDITIONS

3.1.1 Setting 1H Measurement Conditions

1. Enter a job name in the Enter a title for the new job box at the bottom right of the Samples tab in the Spectrometer Control window, and click the Create a Job with this Sample button.



The **Jobs** tab opens automatically.

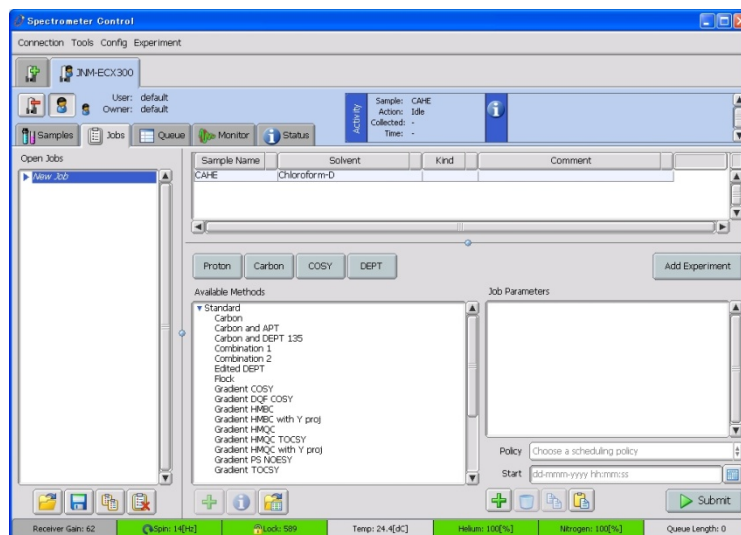


Fig. 3.1 Jobs tab

2. Click the Add Experiment button on the middle right edge of the window. The **Open Experiment** window opens.

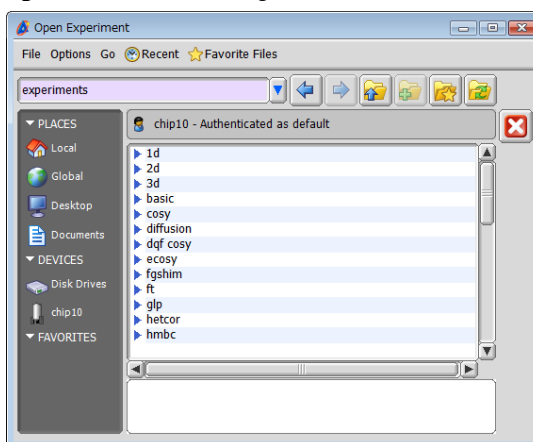


Fig. 3.2 Open Experiment window

3. Click the  button in the Open Experiment window.
A list of the Experiment files* in the Global directory** is displayed.
4. After double-clicking basic in the Open Experiment window, select the measurement mode proton.jsp to highlight it, and click the  button.
This operation opens **Experiment Tool** (Fig. 3.3) under the **Jobs** tab. This **Experiment Tool** window consists of the six sections: **Header**, **Instrument**, **Acquisition**, **Pulse**, **Diagram** and **Favorites**.

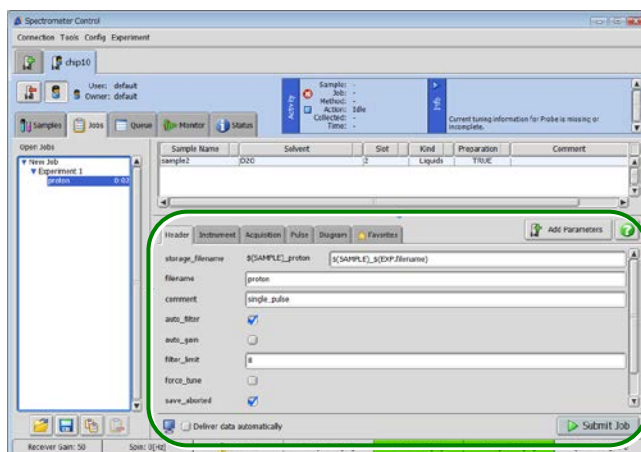


Fig. 3.3 Experiment Tool

5. Set the necessary parameters.
The following parameters are usually used.
 -  For information about the other parameters, refer to the separate volume of user's manual, "Liquid Measurement".
 -  Parameters can be displayed by clicking **Header**, **Instrument**, **Acquisition** or **Pulse**.

Filename:	Data name that is saved (The saved file name is created as sample name_filename .)
Comment:	Comment on measurement data
auto_gain:	If you want the instrument to adjust the receiver gain automatically, select the check box. The results that were acquired using the automatic gain will be displayed on the Delta Console window and the Spectrometer Control window.
recvr_gain:	Receiver gain, if automatic gain is not selected.
x_points:	Number of data points to measure in each scan
scans:	Number of scans to accumulate

* The Experiment files contain the measurement modes, standard measurement conditions, and steps for processing data. The file type is .jsp.

** The Global directory is the directory in which the standard files supplied from JEOL are stored. The files in this directory cannot be changed.

6. Make sure that other necessary parameters are set appropriately.

 Parameters can be displayed by clicking **Header**, **Instrument**, **Acquisition** or **Pulse**.

x_domain:	Observation nucleus:	Proton
x_offset:	Observation center frequency:	5 ppm
x_sweep:	Observation range:	15 ppm
x_pulse:	Pulse width to be used for measurement	
relaxation_delay:	Pulse delay time	

 In the **Favorites** tab, you can specify frequently used parameters in advance. So, if you arrange necessary parameters in the **Favorites** tab, you can specify or confirm the parameters without switching tabs.

3.1.2 Setting ¹³C Measurement Conditions

1. Enter a job name in the Enter a title for the new job box at the bottom right of the Samples tab in the Spectrometer Control window, and click the Create a Job button.

The **Jobs** tab opens automatically.

2. Click the Add Experiment button on the right edge of the window.

The **Open Experiment** window opens.

3. Click the  button in the Open Experiment window.

A list of the Experiment files in the Global directory is displayed.

4. After double-clicking basic in the Open Experiment window, select the measurement mode carbon.jxp to highlight it, and click the  button.

This operation opens **Experiment Tool**.

5. Set the necessary parameters.

The following parameters are usually used.

 For information about the other parameters, refer to the separate volume of user's manual, "Liquid Measurement".

 Parameters can be displayed by clicking **Header**, **Instrument**, **Acquisition** or **Pulse**.

Filename:	Data name that is saved (The saved file name is created as sample name_filename .)
Comment:	Comment on measurement data
auto_gain:	when you adjust the receiver gain automatically, select the check box.
recvr_gain:	Receiver gain, if automatic gain is not selected.
irr_noise:	Noise source for irradiation. Normally, WALTZ is used.
x_points:	Number of data points to measure in each scan
scans:	Number of scans to accumulate
irr_domain:	Irradiation nucleus: Proton
irr_offset:	Irradiation position.

6. Make sure that other necessary parameters are set appropriately.

 Parameters can be displayed by clicking **Header**, **Instrument**, **Acquisition** or **Pulse**.

x_domain:	Observation nucleus:	Carbon13
x_offset:	Observation center frequency:	100 ppm.
x_sweep:	Observation range:	250 ppm
x_pulse:	Pulse width to be used for measurement	
relaxation_delay:	Pulse delay time.	

 In the **Favorites** tab, you can specify frequently used parameters in advance. So, if you arrange necessary parameters in the **Favorites** tab, you can specify or confirm the parameters without switching tabs.

3.2 STARTING AND ENDING MEASUREMENT

This section explains how to start and end 1D NMR measurement and how to confirm the measurement status.

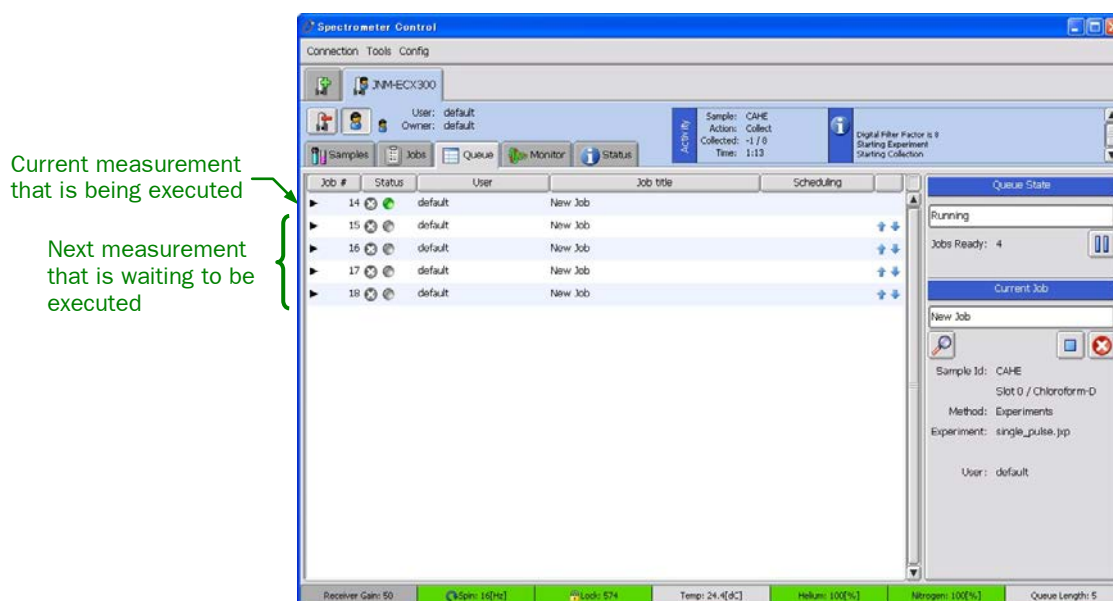
■ Starting measurement and displaying the status

- ◆ Click the Submit button at the bottom right in the Experiment Tool window. The measurement is entered into the spectrometer-control computer's queue. The spectrometer executes measurement on a first-in, first-out basis.



Commands for starting measurements can be issued repeatedly, even if another measurement is being performed. After one measurement finishes, the next measurement will start under the stored measurement conditions.

If you want to deliver the data automatically after completion of the measurement, select the **Deliver data automatically** check box.



■ Ending measurement

If the **Deliver data automatically** check box is selected, the data is displayed in the **1D Processor** window after completion of the measurement.

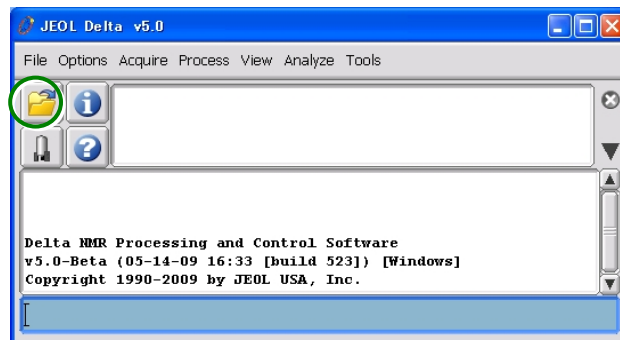
👉 To stop the measurement that is being executed, refer to Chapter 5, “Queuing.”

3.3 DATA TRANSFER FROM THE SPECTROMETER

This section explains how to transfer the data after measurement is complete.

■ Data transfer

1. Click the Open file and choose tool button at the top left in the Delta Console window.



The **File Browser** window opens.

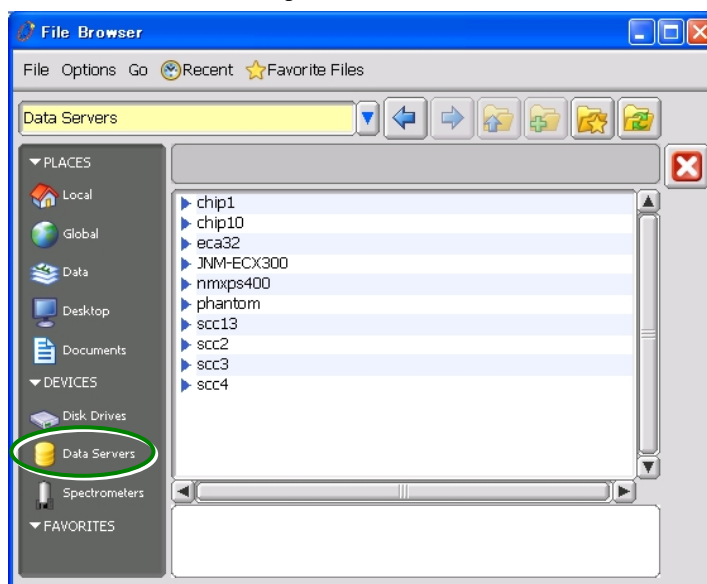
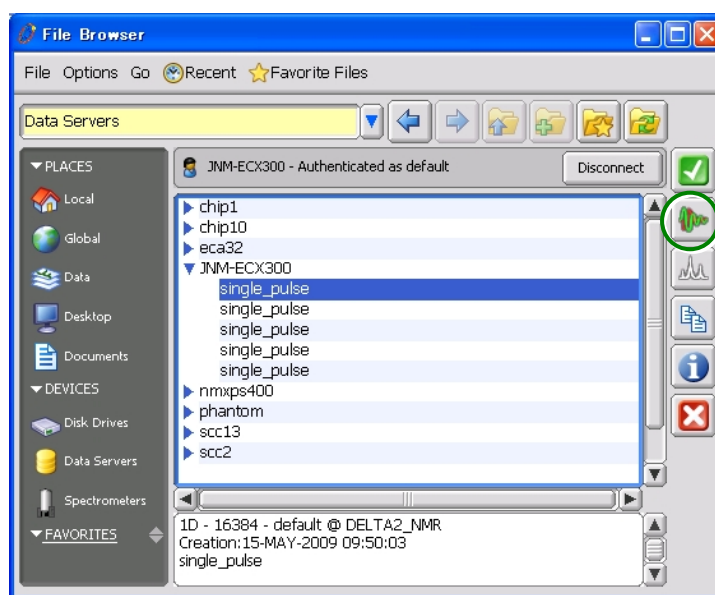


Fig. 3.4 File Browser window

2. Click **Data Servers**  **Data Servers** at the lower left in the File Browser window. The connectable spectrometers are displayed.
3. In the spectrometer list, double-click the spectrometer in which the data is stored. The data list stored in the spectrometer appears.
4. When you select the data that you transfer, a number of buttons will be displayed. Click the Open the selected file for processing  button. The data stored in the spectrometer are transferred, and the specified processing is executed automatically.



3.4 1D NMR DATA PROCESSING

This section explains data processing after measurement was completed and the data were forwarded from the spectrometer.

After the measurement and data transfer, the following processing is automatically executed.

- Correction of the DC components of FID (Free Induction Decay)
- Multiplication by window functions
- FFT (Fast Fourier Transformation)
- Automatic phase correction

Here, data processing following phase correction will be explained.

When measurement is completed, the **1D Processor** window opens.

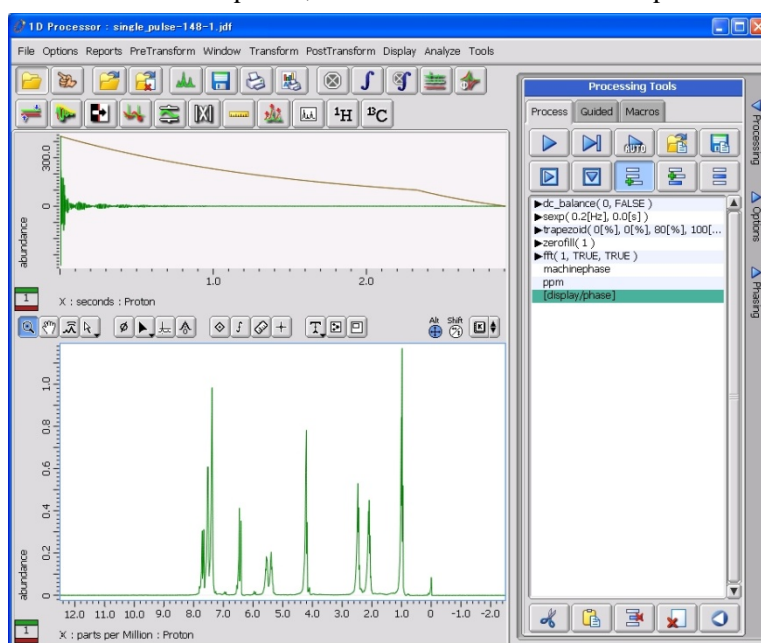
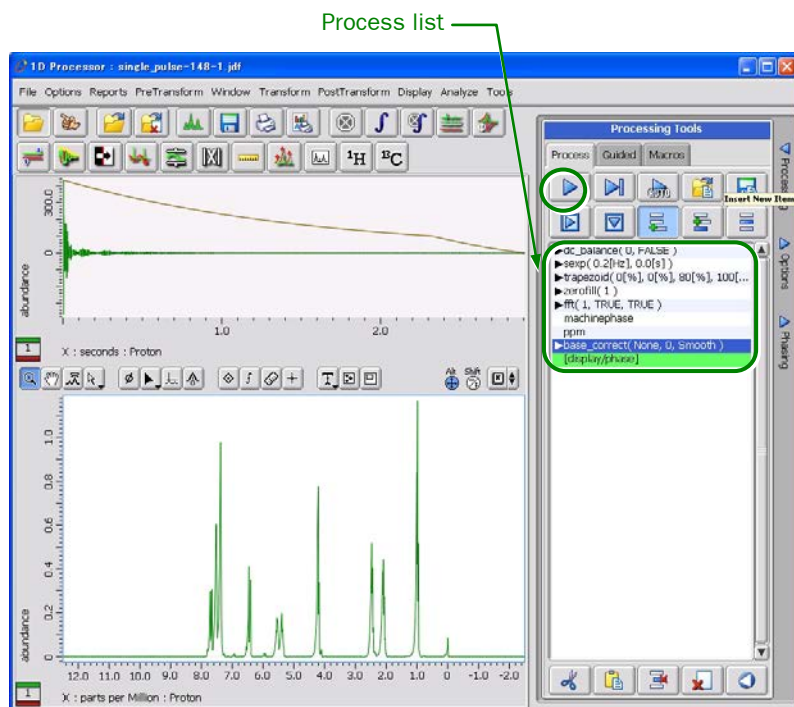


Fig. 3.5 1D Processor window

3.4.1 Automatic Baseline Correction

1. Click the  button in the 1D Processor window.
An item, **Base Correct**, is added to the process list.
2. Click the Process  button.
The spectrum with the baseline corrected is displayed in the lower spectral display area.



3.4.4 Changing the Display Range

■ Usage of Pointer bar

The Pointer bar is used to expand or reduce spectra. This section explains basic usage of the Pointer bar.



Fig. 3.6 Normally displayed Pointer bar

● To switch the Pointer mode

- ◆ Move the mouse pointer to the mode button, and press the left mouse button. The following fifteen mode buttons are displayed.

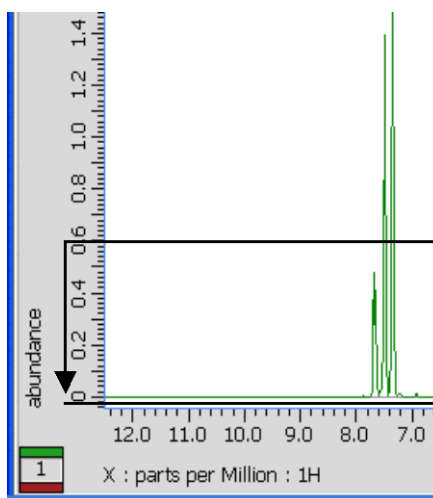
	Zoom:	To expand, reduce, or move spectra.
	Pan View:	To move the area after expanding spectra.
	Amplitude gain:	To adjust the amplitude of spectra.
	Select:	To select data or a geometry.
	Phase correct:	To correct the phase of spectra.
	Copy position:	To copy any area of spectra.
	Peak threshold:	To set the position of the baseline of spectra.
	Reference:	To set a chemical shift reference axis marker.
	Peak:	To execute a peak pick.
	Integral:	To perform integration.
	Measure:	To measure distance between peaks
	Cursor:	To display the horizontal and vertical lines.
	Annotation:	To display an annotation in the geometry.
	Molecule:	To display a structural formula and molecular formula in the geometry.
	PiP:	To display a selected part of the geometry within the present geometry.

- ☞ This chapter explains only the Zoom mode. For more information about the other modes, refer to the separate volume of user's manual, "Data processing".

■ Expanding and resetting spectra in the 1D Processor window

● To expand a spectrum vertically

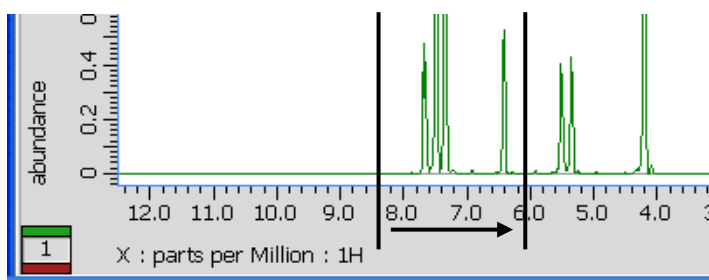
1. Select the Zoom mode from the Pointer bar or press the **F1** key on the keyboard.
The Pointer mode becomes **Zoom**, and the mouse pointer on the spectral screen changes to .
2. Move the mouse pointer to the position that will become the upper end of the expanded Y-axis.
3. Drag the left mouse button to the position that will become the lower end of the expanded Y-axis.
4. Release the left mouse button.



The spectrum is expanded vertically.

● To expand a spectrum horizontally

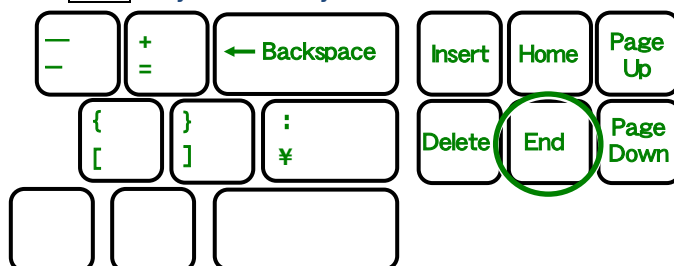
1. Select the Zoom mode from the Pointer bar or press the **F1** key on the keyboard.
The Pointer mode becomes **Zoom**.
2. Move the mouse pointer to the position that will become the left end of the expanded X-axis.
3. Drag the left mouse button to the position that will become the right end of the expanded X-axis.
4. Release the left mouse button.



The spectrum is expanded horizontally.

● To adjust the display to the intensity of the highest peak displayed

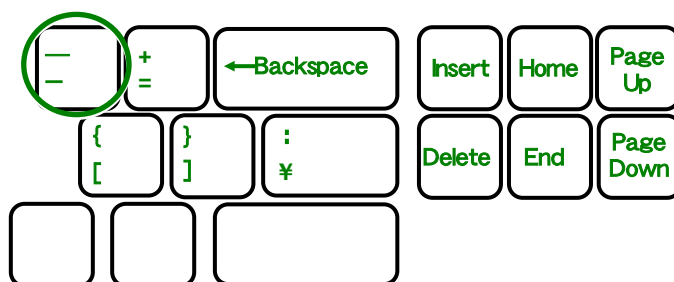
- ◆ Press the **End** key on the keyboard.



The highest peak in the spectral region displayed at present is expanded or reduced to fit within the screen.

● To return the spectrum display to its previous conditions

- ◆ Press the **—** key on the keyboard to return to the previous conditions.



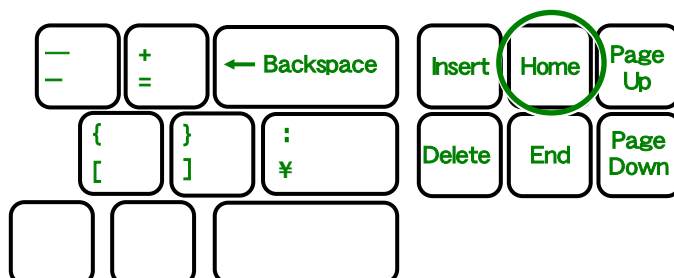
✍ When you press the **—** key, both vertical and horizontal expansions return to their previous values.

You cannot selectively return only the vertical or horizontal size to its previous value.

● To return the spectrum display to its initial conditions

- ◆ Press the **Home** key on the keyboard.

All horizontal and vertical expansions return to their initial values.



4

2D NMR MEASUREMENT

4.1	SETTING 2D NMR MEASUREMENT CONDITIONS.....	4-1
4.1.1	HH COSY Measurement.....	4-1
4.1.2	HMQC Measurement	4-4
4.1.3	Setting the Observation Range Through the 1D Data	4-5
4.2	STARTING AND ENDING MEASUREMENT	4-8
4.3	DATA PROCESSING OF 2D NMR	4-9
4.3.1	Displaying Data Using 2D Viewer.....	4-10
4.3.2	Setting the Conditions for Displaying Contour Lines	4-11
4.3.3	Pasting 1D NMR Data.....	4-12
4.3.4	Plotting	4-14
4.3.5	Changing the Display Ranges	4-14

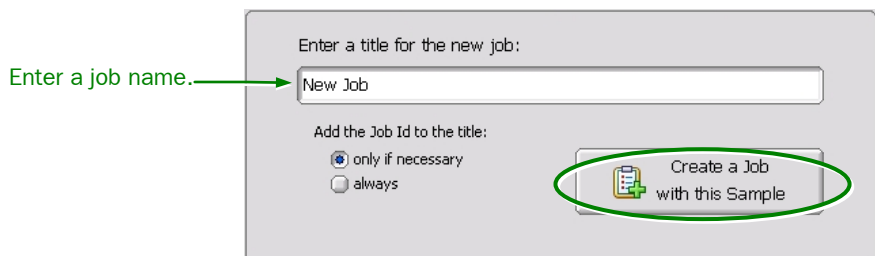


4.1 SETTING 2D NMR MEASUREMENT CONDITIONS

This section explains how to set the conditions for measuring ^1H - ^1H homonuclear 2D NMR (HH COSY) and ^{13}C detection heteronuclear 2D NMR (HETCOR).

4.1.1 HH COSY Measurement

1. Enter a job name in the Enter a title for the new job box at the bottom right of the Samples tab in the Spectrometer Control window, and click the Create a Job with this Sample button.



The **Jobs** tab opens automatically.

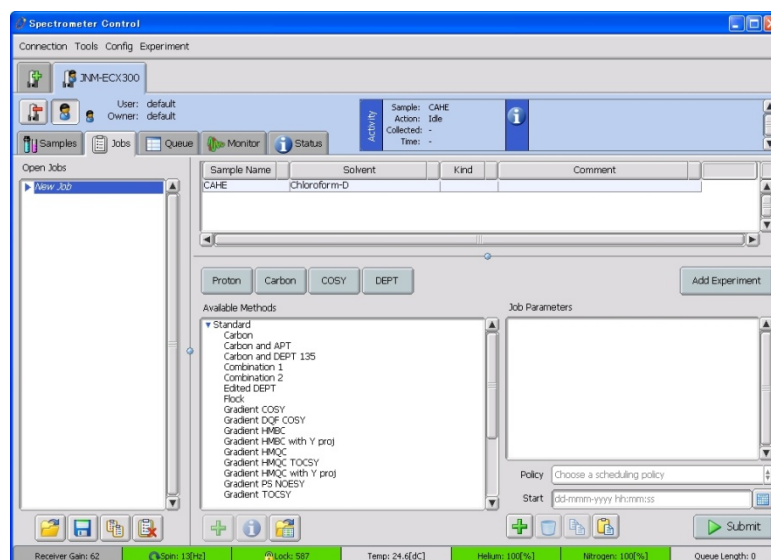


Fig. 4.1 Jobs tab

2. Click the Add Experiment button on the middle right edge of the screen.
The **Open Experiment** window opens.

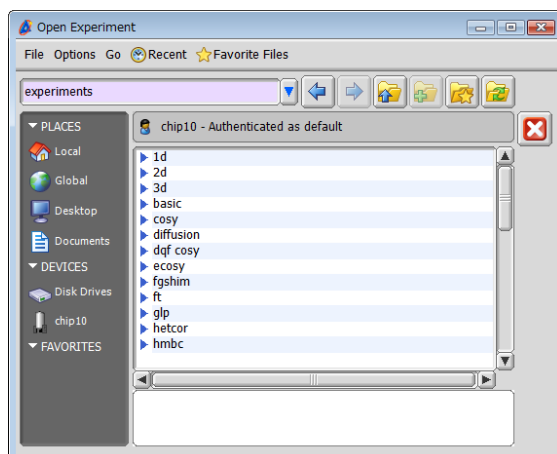


Fig. 4.2 Open Experiment window

3. Click the  button in the Open Experiment window.
A list of the Experiment files* in the Global directory** is displayed.
4. After double-clicking basic in the Open Experiment window, select the measurement mode cosy.jsp to highlight it, and click the  button.
This operation opens **Experiment Tool** (Fig. 4.3) in the lower portion of the **Job** tab.
This **Experiment Tool** window consists of the six sections: **Header**, **Instrument**, **Acquisition**, **Pulse**, **Diagram** and **Favorites**.

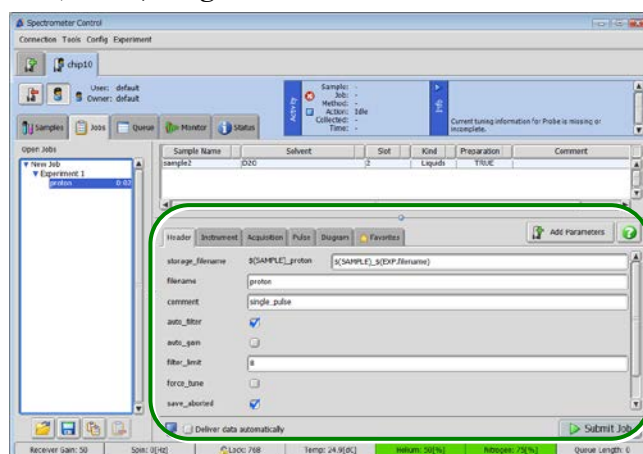


Fig. 4.3 Experiment Tool window

* The Experiment files contain the measurement modes, standard measurement conditions, and steps for processing data.
The file type is .jxp.

** The Global directory is the directory in which the standard files supplied from JEOL are stored. The files in this directory cannot be changed.

5. Set the necessary parameters.

The parameters that are usually used are explained below.

 For information about other parameters, refer to the separate volume of user's manual, "Liquid Measurement".

 Parameters can be displayed by clicking **Header**, **Instrument**, **Acquisition** or **Pulse**.

filename	Data name that is saved (The saved file name is created as sample name_filename .)
comment	Comments on measurement data
auto_gain	If you want the instrument to adjust the receiver gain automatically, select the button to mark it with a check mark. The results that were acquired using the automatic gain will be displayed on the Delta Console window and the Spectrometer Control window.
recvr_gain	Receiver gain, if automatic gain is not selected.
x_points	Number of data points on the f_2 axis
scans	Number of scans to accumulate
y_points	Number of data points on the f_1 axis
pulse_angle_1	Flip angle. 90 [deg] in COSY.
pulse_angle_2	Flip angle. 90 [deg] or 45 [deg] in COSY.
relaxation_delay	Pulse delay time

6. Verify the necessary parameters.

 Parameters can be displayed by clicking **Header**, **Instrument**, **Acquisition** or **Pulse**.

x_domain	Observation nucleus: Proton
x_offset	Observation center frequency (ppm)
x_sweep	Observation range (ppm)
x_90_width	90° pulse width
pulse_1	First pulse width to be used for measurement
pulse_2	Second Pulse width to be used for measurement

 In the **Favorites** tab, you can specify frequently used parameters in advance. So, if you arrange necessary parameters in the **Favorites** tab, you can specify or confirm the parameters without switching tabs.

4.1.2 HMQC Measurement

1. Enter a job name in the Enter a title for the new job box at the bottom right of the Samples tab in the Spectrometer Control window, and click the Create a Job button.

The **Jobs** tab opens automatically.

2. Click the Add Experiment button at the middle right of the screen to open the Open Experiment window.

3. Click the  button in the Open Experiment window.

The contents of the experiment file in the global directory are displayed.

4. After double-clicking basic in the Open Experiment window, select the measurement mode HMQC.jsp to highlight it, and click the  button.

This operation opens **Experiment Tool**.

5. Set the necessary parameters.

The parameters that are usually used are explained below.

 For information about other parameters, refer to the separate volume of user's manual, "Liquid Measurement".

 Parameters can be displayed by clicking **Header, Instrument, Acquisition or Pulse**.

filename	Data name that is saved (The saved file name is created as sample name_filename .)
comment	Comments on measurement data
auto_gain	Normally, do not check this box for 2D measurement.
recvr_gain	Set 50.
x_points	Number of data points on the f_2 axis
scans	Number of scans to accumulate
y_points	Number of data points on the f_1 axis
j_constant	J value
relaxation_delay	Pulse delay time

6. Verify the necessary parameters.

 Parameters can be displayed by clicking **Header, Instrument, Acquisition or Pulse**.

x_domain	Observation nucleus: Proton
x_offset	Observation center frequency (ppm)
x_sweep	Observation range (ppm)
y_domain	Irradiation nucleus: Carbon
y_offset	Irradiation center frequency (ppm)
y_sweep	Irradiation range (ppm)
x_pulse	^1H pulse width to be used for measurement
y_pulse	^{13}C pulse width to be used for measurement

 In the **Favorites** tab, you can specify frequently used parameters in advance. So, if you arrange necessary parameters in the **Favorites** tab, you can specify or confirm the parameters without switching tabs.

4.1.3 Setting the Observation Range Through the 1D Data

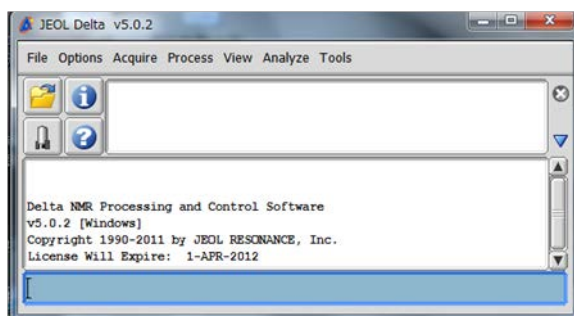
For the measurement of ^1H - ^1H homonuclear 2D NMR, the ^1H 1D data is essential. Also, for the measurement of ^1H detection heteronuclear 2D NMR or ^{13}C detection heteronuclear 2D NMR, ^1H 1D data and ^{13}C 1D data are essential.

If these 1D data are not displayed on the **1D Processor** window or the **Data Slate** window, load these data to the **1D Processor** window from the files.

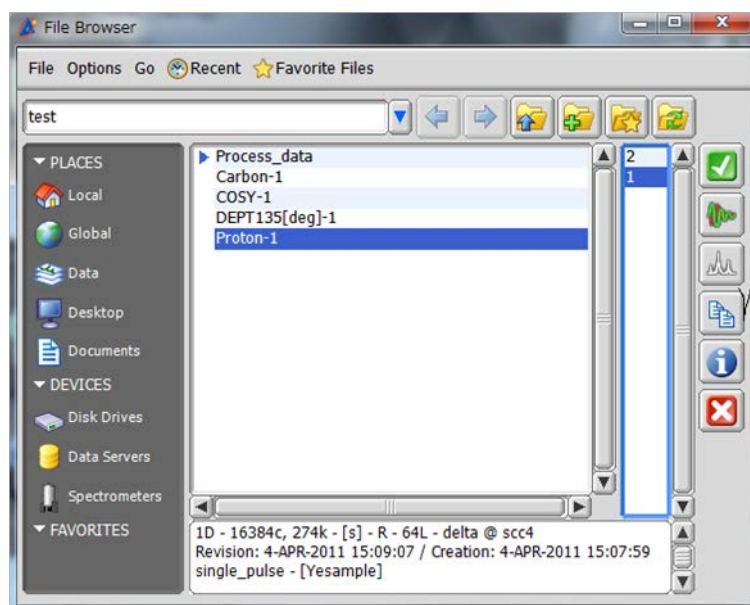
If the 1D data has already been loaded on the **1D Processor** window, proceed to “**Setting the observation range using the 1D data**”.

■ Loading the 1D Data

1. Click the  button in the **Delta Console** window.



The File Browser window opens.



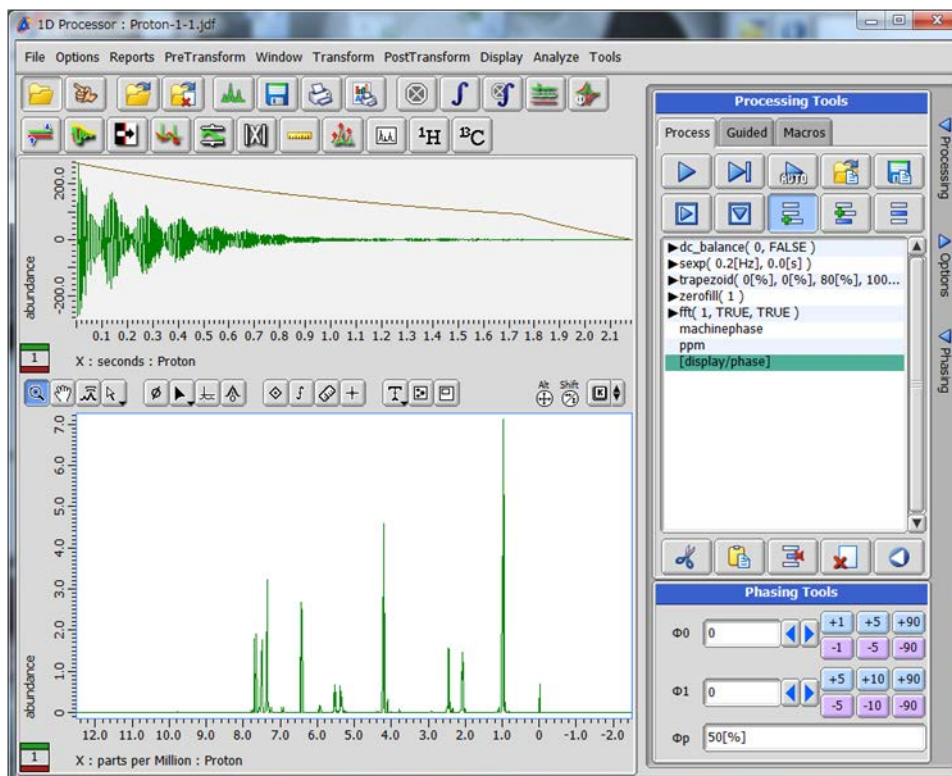
File Browser window

2. Click the name of the data file you want to load in the list box.

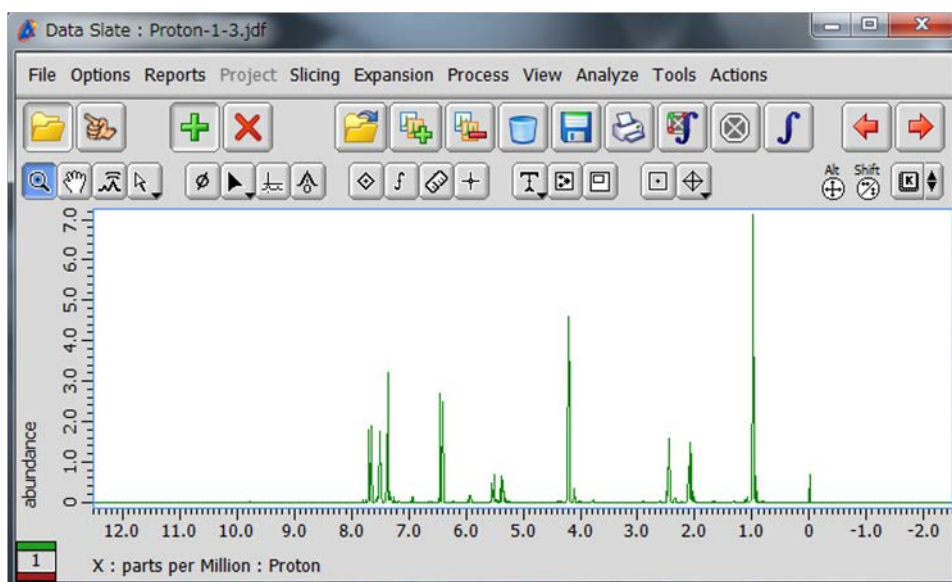
3. Click the button.

The most recent version of the data is displayed in the **1D Processor** window.

If the selected data is the data to which the Fourier Transform has been performed, the data is displayed in the **Data Slate** window.



1D Processor window



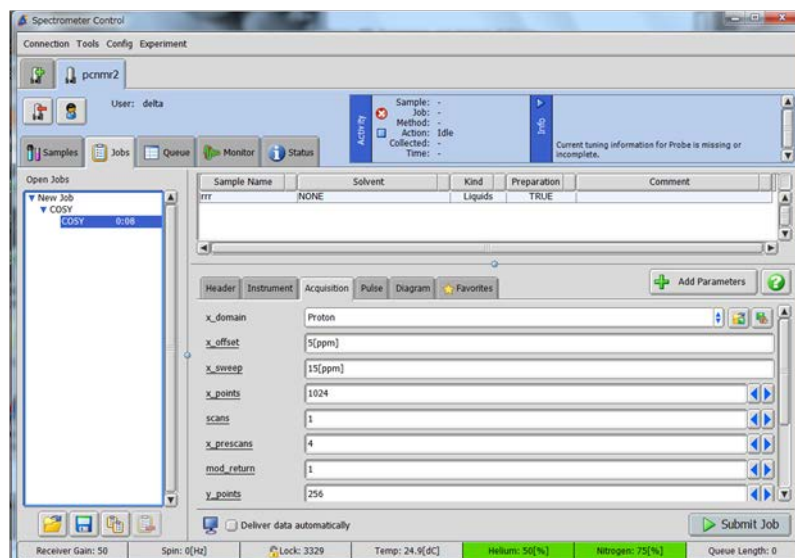
Data Slate window

4. If the 1D NMR data that has been loaded is FID data, perform processing, such as FFT, phase correction, and reference setting, referring to Sect. 3.4 “1D NMR DATA PROCESSING”.

If these processes have already been completed, proceed to the next step.

■ Setting the observation range using the 1D data (HH COSY)

1. Expand the 1D NMR data that has already been processed to the range where the 2D NMR measurement will be performed.
2. Click the  button (located at the right side of the X_domain box) in the **Acquisition** tab of the **Experiment Tool** window.



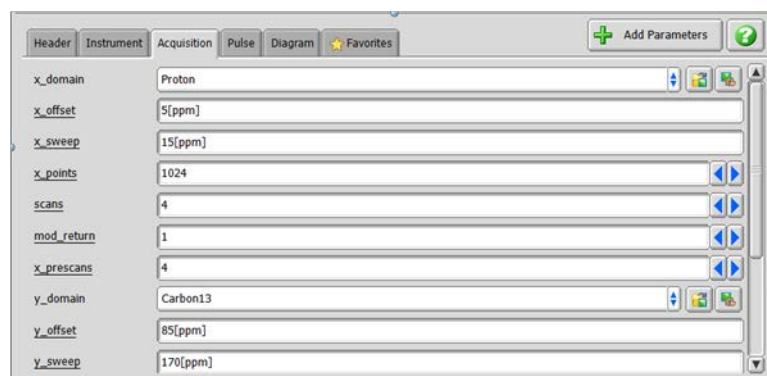
The mouse pointer changes to  after this operation.

3. Move the mouse pointer into the spectrum of the 1D data, and left-click the mouse.

The spectrum range and the offset value of the 1D data that is displayed are set to the **x_sweep** and **x_offset**.

■ Setting the observation range using the 1D data (HMQC and HMBC)

1. Expand the 1D NMR data that has already been processed to the range where the 2D NMR measurement will be performed.
2. Click the  button (located at the right side of the X_domain box) in the **Acquisition** tab of the **Experiment Tool** window.



The mouse pointer changes to  after this operation.

3. Move the mouse pointer into the spectrum of the 1D data (in this case, ^1H spectrum), and left-click the mouse.

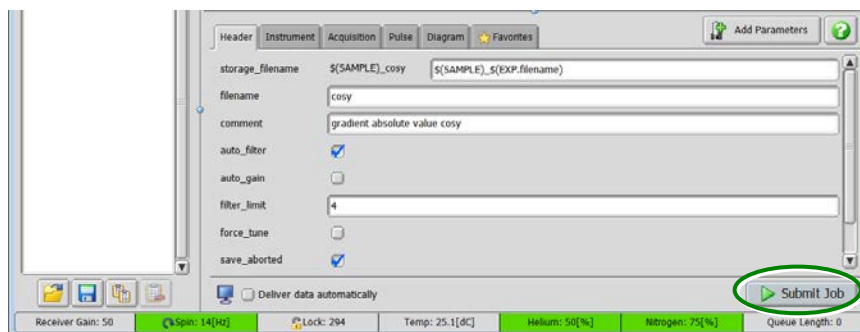
The spectrum range and the offset value of the 1D data that is displayed are set to the **x_sweep** and **x_offset**.

4. Click the  button (located at the right side of the Y_domain box) in the **Acquisition** tab of the **Experiment Tool** window.
5. In the same way as the X-axis setting, move the mouse pointer into the spectrum of the 1D data (in this case, ^{13}C spectrum), and left-click the mouse. The spectrum range and the offset value of the 1D data that is displayed are set to the **y_sweep** and **y_offset**.

4.2 STARTING AND ENDING MEASUREMENT

■ Starting measurement and displaying the present status

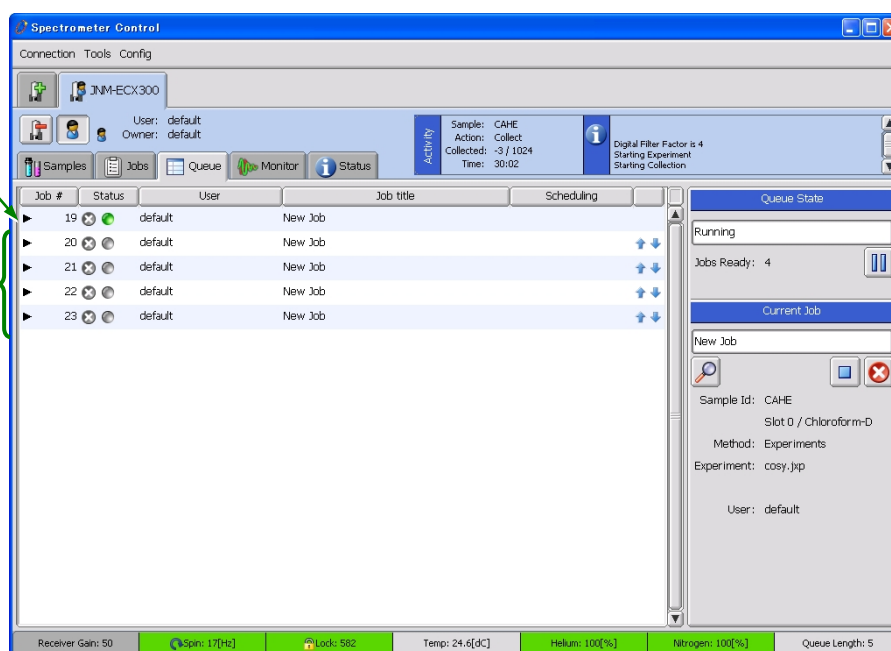
- ◆ Click the Submit button at the bottom right in the Experiment Tool window. The measurement is entered into the spectrometer-control computer's queue. The spectrometer executes measurement on a first-in, first-out basis.



The command for starting measurement can be issued repeatedly, even if another measurement is being performed. After one measurement finishes, the next measurement will start under the stored measurement conditions.

Current measurement that is being executed

Next measurement that is waiting to be executed



Ending measurement

If the **Deliver data automatically** check box was selected, the **nD Processor** window opens after completion of the measurement.

☞ To stop the measurement that is being executed, refer to Chapter 5, “Queuing”.

4.3 DATA PROCESSING OF 2D NMR

This section explains data processing immediately after completion of NMR measurement. When measurement is complete, data will be sent from the spectrometer to the processing computer, and the **nD Processor** window will open.

Data will be processed according to the process list in the pulse sequence.

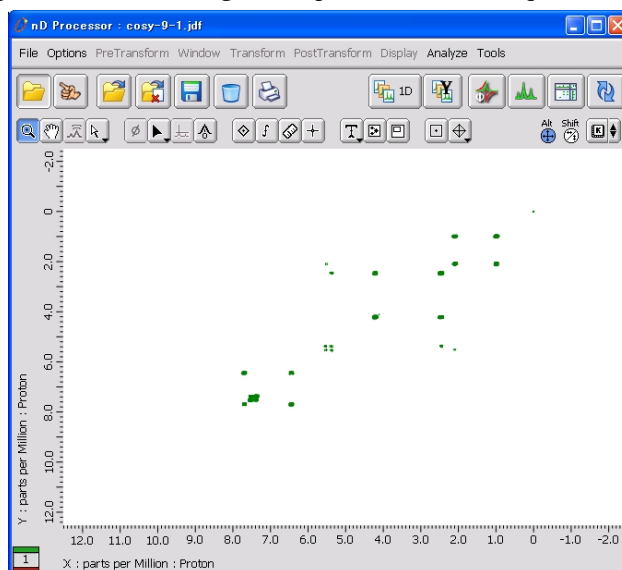


Fig. 4.4 nD Processor window

If you change the processing conditions, click the  button to display the process list. If you click the  button again after changing the process list, processing will be executed again according to the changed conditions.

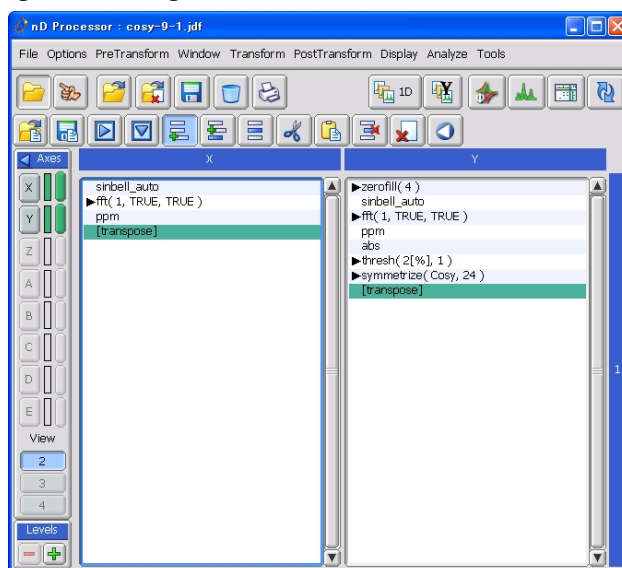
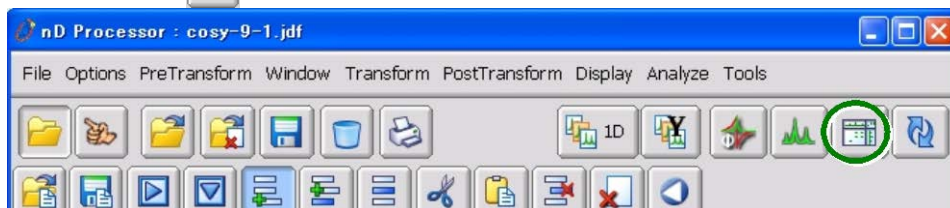


Fig. 4.5 nD Processor window (Process list display screen)

4.3.1 Displaying Data Using 2D Viewer

- ◆ Click the  button in the nD Processor window.



A new **2D Viewer** opens, and 2D data with projections are displayed.

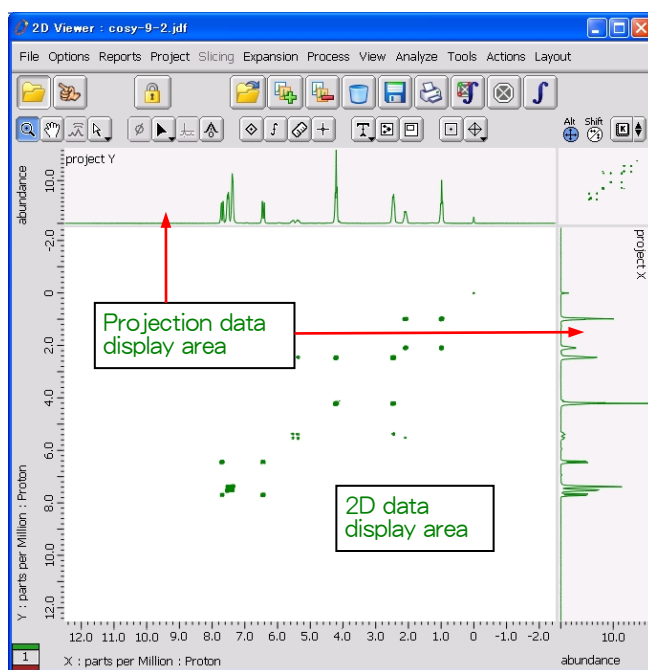


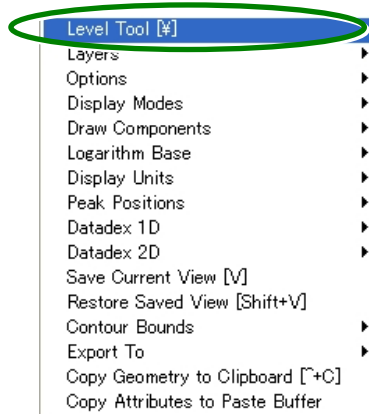
Fig. 4.6 2D Viewer window

4.3.2 Setting the Conditions for Displaying Contour Lines

The **2D Viewer** window is used to set the levels of the contour lines.

The **Level Tool** window is used to change the levels of the contour lines.

1. Click the right mouse button to display the pop-up menu, and select Level Tool.



The **Level Tool** window opens.

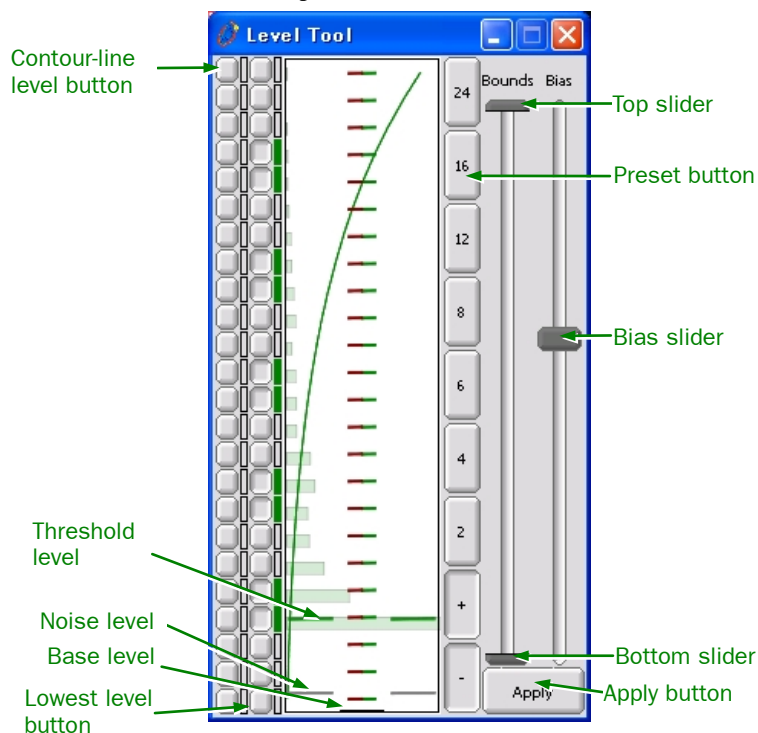


Fig. 4.7 Level Tool window

- Preset button:** Allows you to set the number of contour lines by selecting the 2, 4, 6, 8, 12, or 24 preset button.
- Contour-line level button:** The level of contour can be set manually by clicking each button to highlight it.
- Lowest level button:** It is a button that shows the lowest strength level.
- Top slider:** Determines the maximum intensity of the contour lines.
- Bottom slider:** Determines the minimum intensity of the contour lines.
- Bias slider:** Determines the signal intensity between the ones specified by the top slider and the bottom slider. Moving the bias slider changes the slope of the middle curve shown in Fig.

Noise level:	4.7. Moving it upward results in drawing more contour lines at the lower level of the signal intensities.
Threshold level:	Automatically set after data processing is completed.
Base level:	Automatically set after data processing is completed, and used for peak picking in 2D processing.
Apply button:	Zero level of the signal intensity. Set it to the lowest level in the Level tool window.
	Clicking this button redraws the contour lines at the set levels.

2. Adjust the levels of the contour lines in the Level Tool window.

Set the minimum intensity based on the noise level. Set the number and intensity of the contour lines to be drawn between the minimum intensity and the maximum intensity in the **Level Tool** window.

 Usually, few or no contour lines are set to levels below the noise level. Setting the contour level below the noise level displays the contour lines of noise, and needs a large amount of memory in the system, slowing down the processing of the program.

Set the levels according to the following procedures:

- a. Click the lowest level button of the contour-line level buttons.
- b. Determine the bottom level of the contour lines using the bottom slider.
- c. Determine the top level of the contour lines using the top slider.
- d. Select the number of contour lines you want to display from the preset buttons.
- e. Click the Apply button.

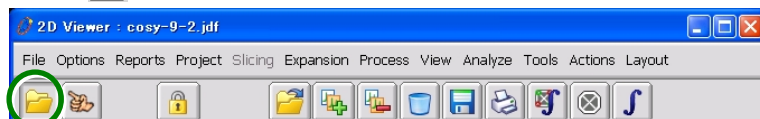
4.3.3 Pasting 1D NMR Data

This section explains how to paste 1D NMR data onto the slice or projection data display area in the **2D Viewer** window.

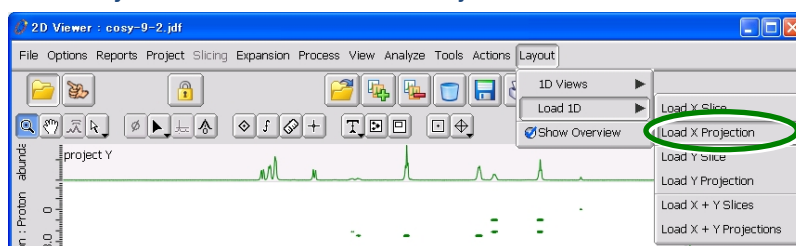
It is assumed that 1D NMR data on which data processing such as FFT was already performed are stored on the hard disk or is displayed in the **1D Processor** window.

■ When 1D NMR data is stored on the hard disk

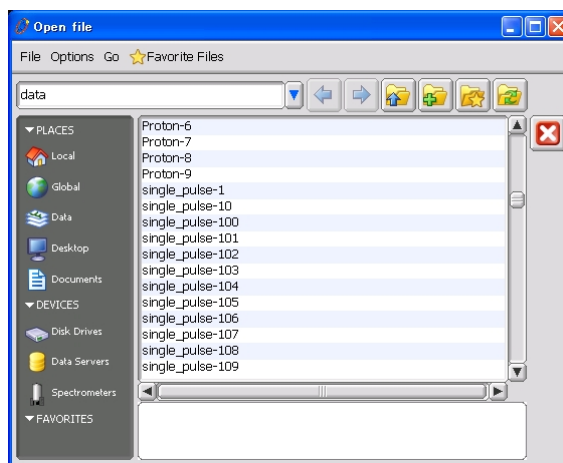
1. Click the  button.



2. Paste 1D data onto the X-axis.
 - a. Select Layout—Load 1D—Load X Projection.



The **Open file** window opens.



- b.** Select a file.

1D NMR data are pasted in the projection display area of the X-axis.

- 3.** Paste 1D data onto the Y-axis.

- a.** Select Layout–Load 1D–Load Y Projection.

- b.** Select a file.

1D NMR data are pasted onto the projection display area of the Y-axis.

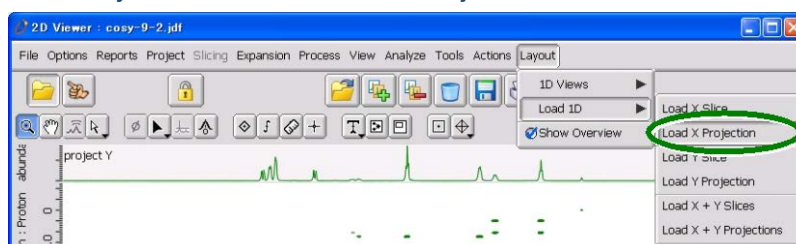
■ When 1D NMR data is displayed on the 1D Processor window

- 1.** Click the  button.



- 2.** Paste 1D data onto the X-axis.

- a.** Select Layout–Load 1D–Load X Projection.



The mouse pointer changes to .

- b.** Move the mouse pointer onto the displayed 1D NMR data and click it.

The 1D NMR data are pasted onto the slice or projection display area of the X-axis.

- 3.** Paste 1D data onto the Y-axis.

- a.** Select Layout–Load 1D–Load Y Projection.

The mouse pointer will be changed to .

- b.** Move the mouse pointer on the displayed 1D data and click.

1D NMR data are pasted onto the projection display area of the Y-axis.

4.3.4 Plotting

This section explains how to perform the standard plotting.

☞ For the details about the settings, refer to the separate volume, “Data Processing” user’s manual.

- ◆ Click the  button in the 2D Viewer window.



The default plotting is performed.

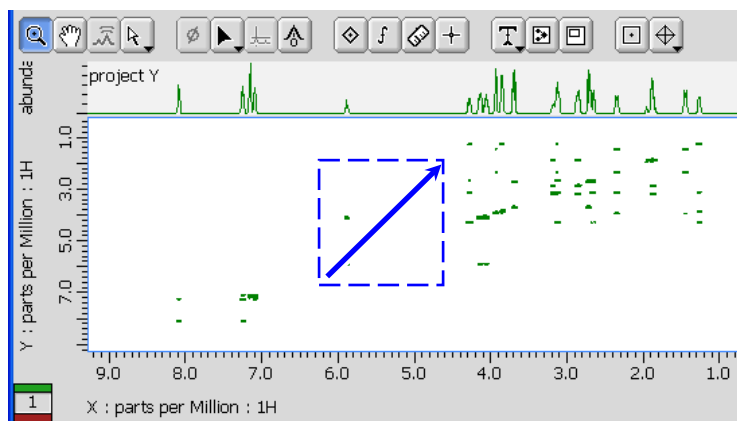
4.3.5 Changing the Display Ranges

The pointer bar is used to expand and reduce spectra.

☞ For how to display the pointer, refer to Section 3.4.4. For the details of the pointer, refer to the separate volume, “Data Processing” user’s manual.

■ To expand a spectrum vertically and horizontally at the same time

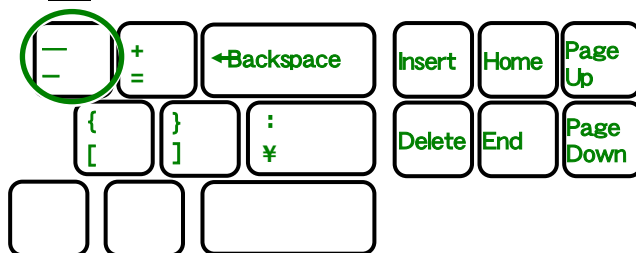
1. Select the Zoom mode from the pointer bar or press the **[F1]** key on the keyboard.
The pointer mode becomes **Zoom**.
2. Move the mouse pointer to the position that will become the lower left corner of expansion in the display area.
3. Press and hold down the left mouse button, and move the mouse pointer to the position that will become the upper right corner of expansion in the display area.



4. Release the left mouse button.
The spectrum is expanded vertically and horizontally at the same time.

■ To return the spectrum display to its previous conditions

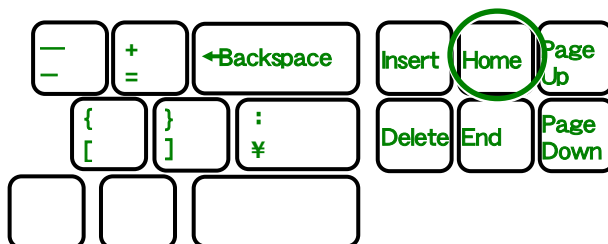
- ◆ Press the  key on the keyboard to return to the previous conditions.



-  When you press the  key, both vertical and horizontal expansions return to their previous values.
You cannot selectively return only the vertical or horizontal size to its previous value.

■ To return the spectrum display to its initial conditions

- ◆ Press the  key on the keyboard.
All horizontal and vertical expansions return to their initial values.



5

QUEUEING

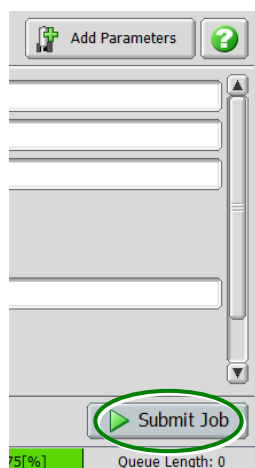
5.1	COMMAND FOR STARTING MEASUREMENT	5-1
5.2	QUEUEING	5-2



5.1 COMMAND FOR STARTING MEASUREMENT

■ Starting measurement and displaying the present status

Clicking the **Submit** button in the **Experiment Tool** window enters the measurement into the spectrometer-control computer's queue, and the spectrometer executes measurement on a first-in, first-out basis.



The command for starting measurement can be issued repeatedly, even if another measurement is being performed. After one measurement finishes, the next measurement will be started under the stored measurement conditions.

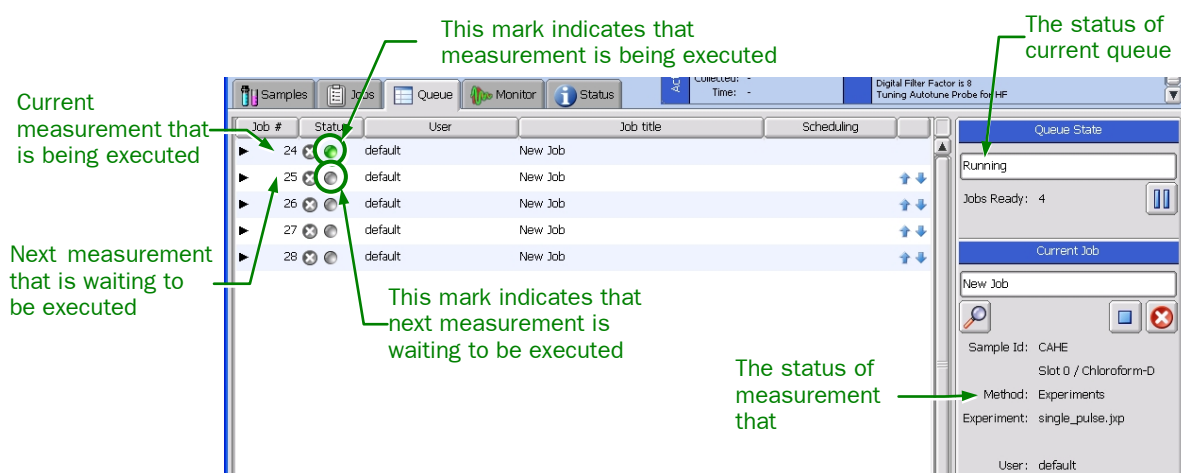


Fig. 5.1 Displaying the status of the queue

5.2 QUEUING

■ Deleting measurement

You can delete measurements in the queue using the following procedures.

● How to delete specified experiment (Job)

1. Click the  button of the measurement to delete in the Queue tab.
The confirmation dialog box appears to verify deletion.

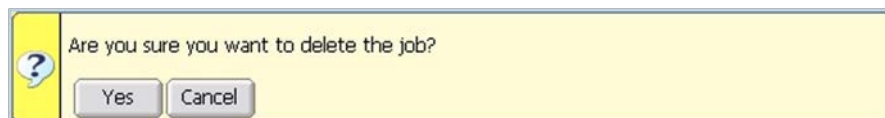


Fig. 5.2 The dialog box that verifies the deletion.

2. To delete the queue, click the Yes button.

■ Viewing information about the jobs in the measurement queue

You can view the details of the job in the measurement queue as follows.

1. Click the  button on the extreme left of the measurement whose information you want to view in the Jobs tab.
The **Job Info** window opens.
2. To finish viewing, click the  button.
The **Job Info** window is closed.



SAVING AND LOADING DATA

6.1	SAVING NMR DATA.....	6-1
6.1.1	Saving the measured data (FID).....	6-1
6.1.2	Saving Processed Data	6-1
6.2	LOADING NMR DATA.....	6-4



6.1 SAVING NMR DATA

6.1.1 Saving the measured data (FID)

The measured data (FID) are saved automatically. A filename for the saved data is specified as in the following procedure.

1. Specify the filename for the data you want to save in the `storage_filename` of the Experiment Tool window.
 If not specified, the default filename is entered. The name shown on the right side of `storage_filename` is the default filename.

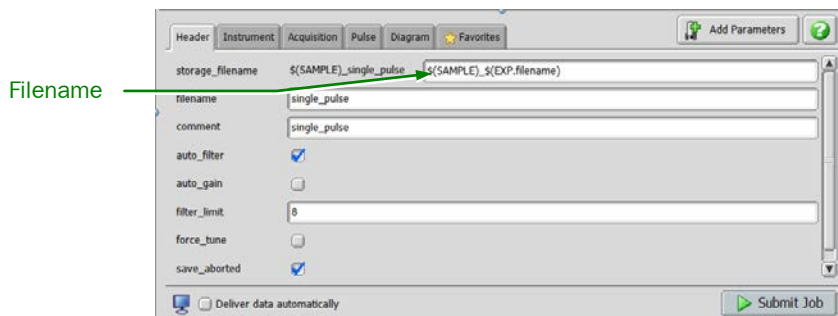


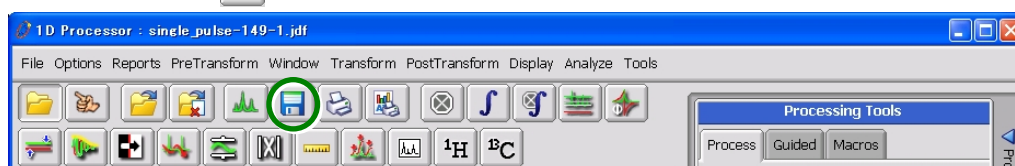
Fig. 6.1 Input box for a filename

2. Start measurement.
 When measurement is complete, the data are saved with the specified name.

6.1.2 Saving Processed Data

- To save 1D processed data in the 1D Processor window under the same filename

- ◆ Click the  button in the 1D Processor window.



The 1D processed data are saved. Then, the version number increases automatically under the same filename.

■ To save 1D processed data in the 1D Processor window under a new filename

1. Select **File – Save As** from the menu bar.
The **Save Data File** window opens.

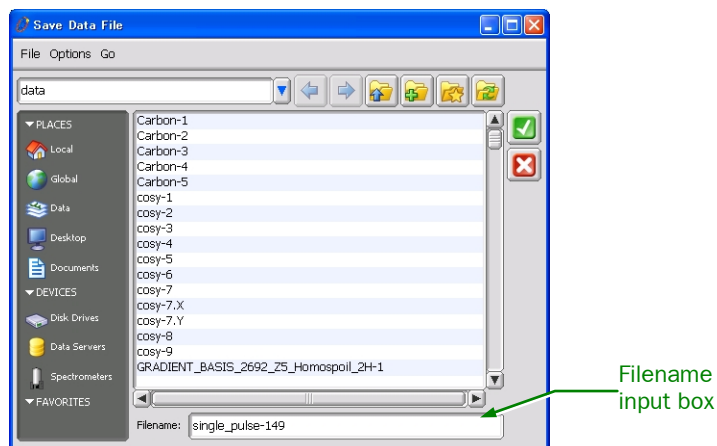
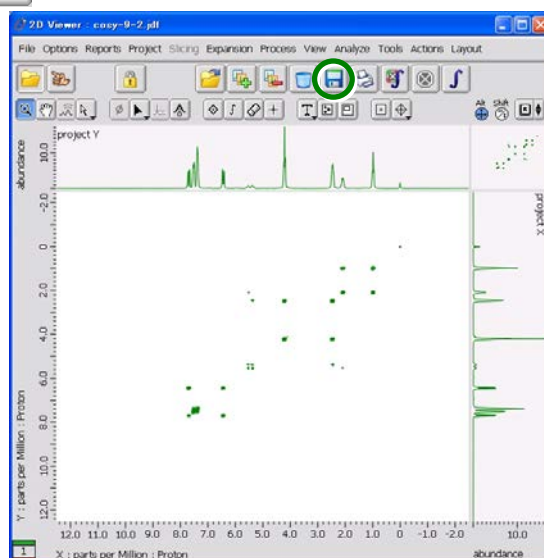


Fig. 6.2 Save Data File window

2. Type any filename in the filename input box.
When creating a new directory, after clicking the  button, type the directory name in the **Create** box and click the **OK** button.
3. Click the  button.
The 1D processed data are saved under the specified filename.

■ To save 2D processed data in the 2D Viewer window under the same filename

- ◆ Click the  button.



The 2D processed data are saved. Then, the version number increases automatically under the same filename.

■ To save 2D processed data in the 2D Viewer window under a new filename

1. Select **File – Save – Save As** from the menu bar.

The **Save File for Data Slate** window opens.

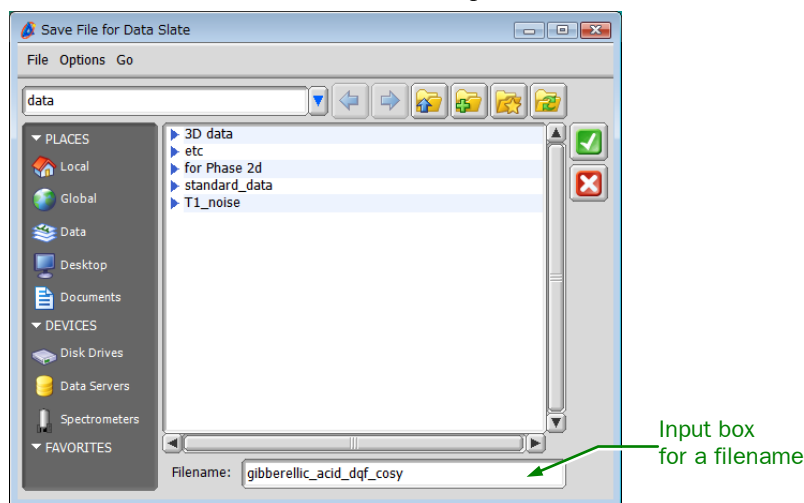
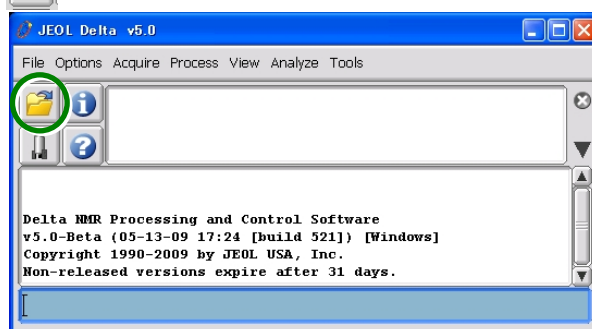


Fig. 6.3 Save File for Data Slate window

2. Type any filename in the **Filename** input box.
 When creating a new directory, after clicking the  button, type the directory name in the **Create** box and click the **OK** button.
3. Click the  button.
 The 2D processed data are saved under the specified filename.

6.2 LOADING NMR DATA

1. Click the  button in the Delta Console window.



The **File Browser** window opens.

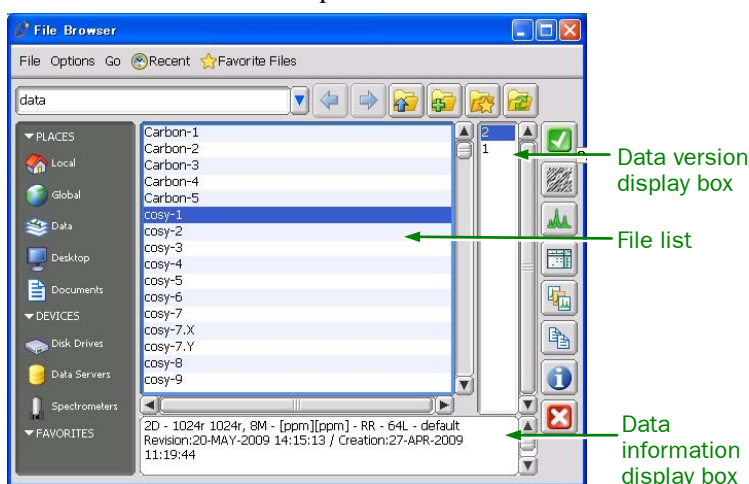
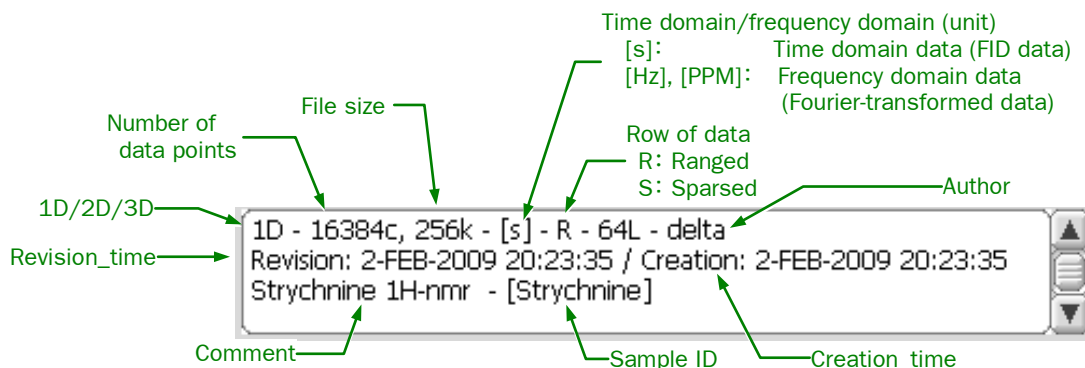


Fig. 6.4 File Browser window

2. Click the name of the data file you want to load in the list box.
3. Click the .

The most recent version of the data is displayed in the **1D Processor** window or **nD Processor** window.

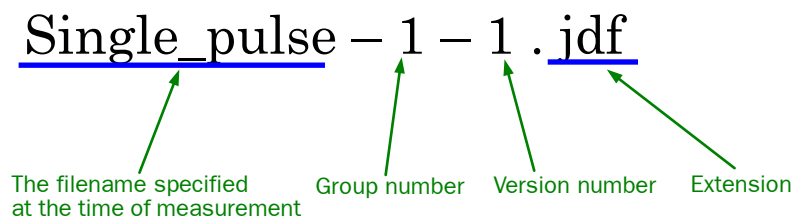
 The following information on the most recent version of the data is displayed in the data information display box.



 If you display earlier data, select the version number, referring to information in the data information display box, and click the .

■ Version number

In the Delta program, when loading a data from the data server, the group number for avoiding a duplication of a file name and a version number are appended to each data file name. Also, before data are processed, a new file is automatically created under the same file name with a version number higher than that of the original data. The original data are not processed, preventing corruption of the original FID data. If you save data under the existing file name, the data are given a version number higher by one. Copied data are sometimes deleted after they are processed. Therefore, some of the version numbers in the file list are usually omitted.



■ Directory

You can specify two directories in the **File Browser** window using the buttons. One is a local directory. The other is a global directory. Normally, the user uses the local directory.

● Local directory

Clicking the  button displays a list of the files in the local directory at the middle of the **Open Data for 1D Processor** window.

 The local directory is specified in **Directory–Data** in **Preferences**.

● Global directory

Clicking the  button displays a list of the files in the global directory at the middle of the **Open Data for 1D Processor** window. Because the global directory cannot be used for writing, it is not used to store data.

 The global directory is specified in **Directory–Global Data** in **Preferences**.

INDEX

1

1D NMR DATA PROCESSING	3-8
1D Processor.....	3-8

2

2D Viewer	4-10
-----------------	------

A

Amplitude gain	3-11
Annotation	3-11
Apply button.....	4-12
Auto shimming	1-10
Automatic Baseline Correction.....	3-9
Automatic gradient shimming	1-10
Automatic Integration.....	3-10
Automatic Peak Detection	3-10
Automatic resolution adjustment.....	1-10

B

Base level	4-12
Bias slider	4-11
Bottom slider	4-11

C

Carbon	2-1
Changing the Display Range	3-11, 4-14
Connecting to the spectrometer	1-2
Contour-line level button.....	4-11
Copy position	3-11
COSY	2-1
Cursor	3-11

D

DATA PROCESSING OF 2D NMR	4-9
Data Servers	3-6
Data transfer	3-6
Deleting measurement.....	5-2
Deleting the sample	2-2
Delta Console window	1-1
DEPT	2-1
Directory.....	6-5
Disconnecting from the spectrometer.....	1-3
displaying the present status.....	4-8

E

Ejecting the sample	1-5
Ending measurement	3-5, 4-9
Ending the Delta program	1-1
Expanding and resetting spectra in the 1D Processor window	3-12
Experiment files	3-2, 3-3
Experiment Tool	3-2

F

File Browser	3-6
File Browser window	4-5

G

Global directory.....	3-2, 3-3, 6-5
gradient shim.....	2-3

H

HMBC.....	2-1
HMQC.....	2-1
HSQC	2-1

I

Integral	3-11
----------------	------

L

Level Tool.....	4-11
LOADING DATA.....	6-4
Loading the sample	1-8
Local directory	6-5
lock_state.....	2-3
Lowest level button	4-11

M

Manual Sample Pane	1-8
Measure	3-11
Molecule.....	3-11

N

nD Processor window.....	4-9
NMR lock.....	1-9
NOESY	2-1

Noise level4-12

P

Pan View3-11
 Pasting 1D NMR Data4-12
 Peak3-11
 Peak threshold.....3-11
 Phase correct.....3-11
 PiP.....3-11
 Plotting 3-10, 4-14
 Pointer bar.....3-11
 preparation2-3
 Preset button4-11
 Process button.....3-9
 Proton.....2-1

R

Reference3-11
 Registration of a new sample2-2
 Registration of the sample1-7
 ROESY2-1

S

Sample State1-8
 SAVING NMR DATA6-1
 Saving Processed Data.....6-1
 Saving the measured data (FID)6-1
 Select3-11
 Setting 13C Measurement Conditions3-3
 Setting the Conditions for Displaying
 Contour Lines4-11
 Setting up the holder.....1-6
 Setting up the sample.....1-6
 Spectrometer Control.....1-2
 spin_set.....2-3
 spin_state2-3
 Spinning the sample.....1-9

Starting measurement 3-5, 4-8, 5-1
 Starting the Delta program1-1

T

temp_set.....2-3
 temp_state.....2-3
 Threshold level4-12
 To adjust the display to the intensity of
 the highest peak displayed.....3-13
 To expand a spectrum vertically and
 horizontally at the same time.....4-14
 To return the spectrum display to its
 initial conditions3-13, 4-15
 To return the spectrum display to its
 previous conditions.....3-13, 4-15
 To save 1D processed data in the 1D
 Processor window under a new
 filename6-2
 To save 1D processed data in the 1D
 Processor window under the same
 filename6-1
 To save 2D processed data in the 2D
 Viewer window under a new
 filename6-3
 To save 2D processed data in the 2D
 Viewer window under the same
 filename6-2
 TOCSY2-1
 Top slider4-11

V

Version number.....6-5
 Viewing information about the jobs in
 the measurement queue5-2

Z

Zoom3-11