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

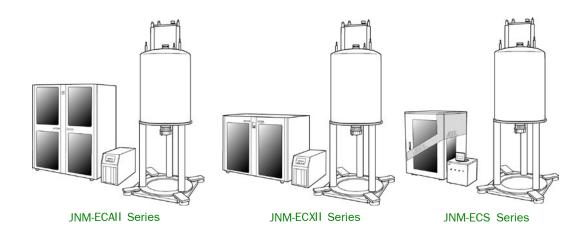
**TUTORIAL MANUAL** 

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.

INMECAXS II\_V50-TU-1 APR2013-08110347 Printed in Japan

# JNM-ECAII Series JNM-ECXII 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.



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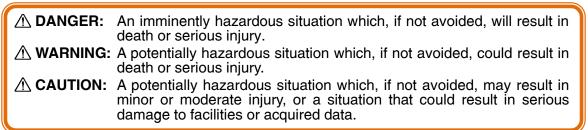
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# SAFETY PRECAUTIONS 💠 💠 💠

Although this instrument is protected with safety device which prevents the occurrence of accident that could result in an injury, harm, and damage to the users or instrument itself, the safety feature may not work properly if you use the instrument for the purpose of use not intended or in an improper usage. For the proper use of the instrument, please be sure to read all of the instructions, descriptions, notices, and precautions contained in this manual carefully to understand them fully prior to the operation or maintenance. This section, "Safety Precautions," contains important information related to safety for using of the instrument.

The safety indications and their meanings are as follows:



Labels bearing the following symbols are attached to dangerous locations on the instrument. Do not touch any of these locations with your hands or anything else.













Examples of symbols

- Use the instrument properly within the scope of the purpose and usage described in its brochures and manuals.
  - Never open/remove protective parts (exterior panels) and parts that can't be opened/removed without use of tool (including key), or disconnect/ connect the cables/connectors that are not described in this manual.
  - Never attempt to do any works of disassembling/assembling the instrument other than those described in this manual.
  - Never make modifications that include installing substitute parts and disabling safety devices or other safety features.
  - Never disconnect the grounding wire or move it from the prescribed position. Failure to follow this instruction could result in electric shock.
- 0 Ç

- The AC power cord provided with this system is supplied for the particular device so that never use it for any other equipment.
- To avoid falling, do not climb onto the operation table and console during daily operation or during maintenance or inspection.



• When you dispose of the instrument or liquid or other waste, follow all applicable laws and regulations, and dispose of it in a proper manner without polluting the environment.



• Be sure to read the "Safety Precautions" section of the manuals for the accessories attached to or built into the instrument.



• If anything is unclear, please contact your JEOL service office.



### MARNING 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.

### 



• 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.



# PREPARATION FOR MEASUREMENT

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### **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.

🤣 JEOL Delta v5.0	
File Options Acquire Process View Analyze Tools	
	0
	▼
Delta NMR Processing and Control Software v5.0-Beta (05-13-09 17:24 [build 521]) [Windows]	=
Copyright 1990-2009 by JEOL USA, Inc. Non-released versions expire after 31 days.	
T	
L	

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.

🔗 JEOL Delta 🕫 J								
File Options Acquire	File Options Acquire Process View Analyze Tools							
🚰 Fle Browser	Shift+^+0		0					
<b>∏</b> Val\tate								
File Information	~+I	]	Y					
File Seatch	~+F	2						
Page Setuy		Control Software						
About		[build 521]) [Windows]						
Quit	Shift+^+0	OL USA, Inc. ire after 31 days.	╡					
Г		·	÷					
Ļ			-					

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

🔗 JEOL Delta 🕫 🔲 🔲	
File Options Acquire Process View Analyze Tools	
	8
Delta NMR Processing and Control Software v5.0-Beta (05-13-09 17:24 [build 52]] [Windows] Copyright 1990-2009 by JEOL USA, Inc. Non-released versions expire after 31 days.	
I	

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

Spectrometer Cont	rol	
Connection Tools Conf	9	
3		
Available Instrument	s ()	
INM-ECX300	S Connect	
chip1 chip10	Name 🔒 JMM-ECX300	v5.0-Beta, build 521
eca32 hpwg03	Status This spectrometer is AVAILABLE	
nmxps400 scc13		
scc2 scc3	Field Strength 7.058601[T] ~ 301[MHz]	
6004 1014	Queue Status IDLE	
	✓ Click here to hide more information	
	IP Address 172.16.164.168	Port 6241
	Probe ID 2692	Type LIQUIDS
	AQP Version 5	
	Holun level	
	Up Time 6 hours 58 mins 9 secs	
	Boot Time 14-MAY-2009 11:11:04	
	Model # Serial #	

Fig. 1.3 Spectrometer Control window

 $\swarrow$  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.

	Authentication				
Please enter your login information					
Name 🛛	ccount name required				
Password					
Conne	ect Own Cancel				

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

Ø Spectrometer Control								
Connection Tools Config Shims Si	amples							
MH-ECA500								
User: default Owner: default		Sample: Action: Collected: Trans	Ide 🚺					
🗓 Samples 📋 Jobs 📃 Que	ue 🕼 Monitor 🕤 Status	Time:				T		
👍 😑 🛐 Sample C	ontrol: 🚺 Load 🚺 🎼 Is	nteractive						
No. V Sample Name	Solvent	Kind Shared	Verified Error	Owner	Last	Load		
			(	Create a Job	Update Job(s)			
Enter a title for the new job:								
New Job								
Add the Job Id to the title:								
			<ul> <li>only if nece</li> <li>always</li> </ul>	ssary	Cre	sate a Job		
Receiver Gain: 50 OSpin:	15[Hz] 🔓Lode: 801	Temp: 25[dC]	Holium: 50(	%] Nitrog	en: 75[%]	Queue Length: 0		

Fig. 1.4 Spectrometer Control window after the spectrometer is connected

### Disconnecting from the spectrometer

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

💋 Spe	strometer Control	
Conne	tion Tools Config Shims Samples	
	User: default Owner: default	Are you sure you want to disconnect?
<b>i i</b> sa	imples 📔 Jobs 🔲 🔲 Queue 🛛 🧄 Monitor 🛛 🧃	Status Ves No

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 S 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.

Spectrometer Control	
Connection Tools Config Shims Samples	
TMHECA500	Sançie: -
	Status
Sample Control:	De Interactive

**3.** Click the Samples **[]** samples tab to open the Samples tab.

Spectrometer Control							
Connection Tools Config Shims Samples							
1 JNM-ECA500							
User: default Owner: default		Sample: Action: Collected: Time:	Idle 🚺			Â	
🚹 Samples 📋 Jobs 🔲 Queue 🥼	Monitor 🕤 Status	Time:				T	
Sample Control:	🖡 Load 🚺 🐉 Inter	active					
No. V Sample Name So	lvent Ki	nd Shared	Verified Error	Owner	Last	Load	
						<b>v</b>	
				Create a Job	Update Job(s)		
Enter a title for the new job:							
New Job							
Add the Job Id to the tible: (6) only / necessary (7) Create a Job							
			问 always		Cre.	ate a JOD	
Receiver Gain: 50 (Spin: 16[Hz]	CLock: 519	Temp: 25.1[dC]	Helium: 50[%	6) Nitrog			

### 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 por , 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

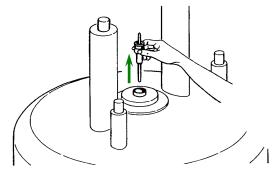
If a sample is inserted in the superconducting magnet, remove it by using the following steps.

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

Spectrometer Control						
Connection Tools Config Shims Samples						
1 IP JNM-ECA500						
User: default Owner: default JUSamples 🗒 Jobs 📰 Queue 🌗 Monitor 👔 Statu	JS	Sample: Action: Collected: Time:	Idle -	Í,	Current tuning information	n for Probe is missing or incomplete.
Sample Control: 👔 Eject	Interactive	]				
No. V Sample Name Solvent	Kind	Shared	Verified	Error	Owner	Last Load
▶ 1 = Sample01 Chloroform-D 🛊		<b>Ø</b>	1		default	Never

Fig. 1.7 Samples tab

- 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

### M 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).

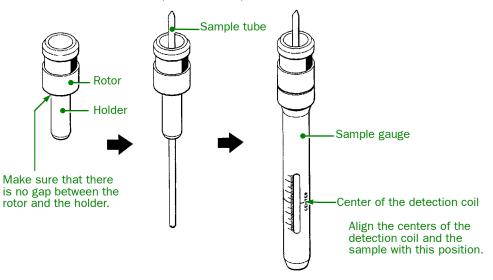
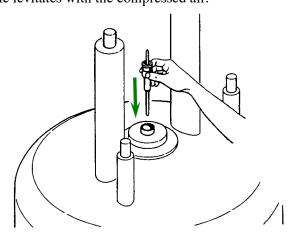


Fig. 1.8 Setting up the holder

- 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.
- 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.
 Spectrometer Control

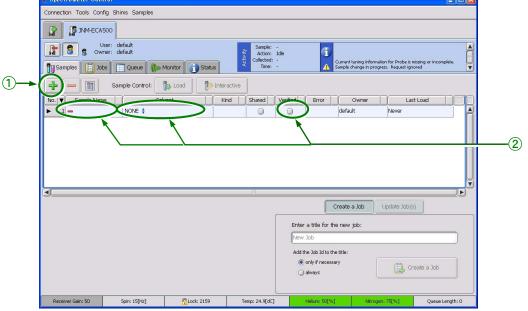


Fig. 1.9 Samples tab

### 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).
- 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).

*K* 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.

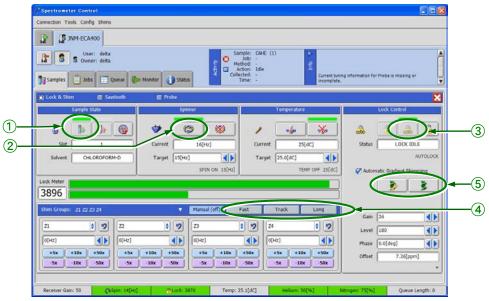


Fig. 1.10 Manual Sample Pane

### 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.

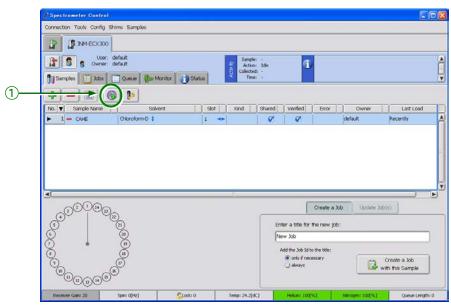


Fig. 1.11 Samples tab (when an auto sample changer is installed)

### Spinning the sample

Usually, you measure the sample while spinning it. If you measure the sample without spinning, proceed to **INMR 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.

- 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.
- 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 Track button continues to adjust the shim values by tracking them after the specified shims adjustment finishes.

. . . . .

# PRACTICE OF AUTOMATIC MEASUREMENT

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Standard

Standard

Standard

Standard

Standard

### 2.1 STANDARD METHOD OF AUTOMATIC MEASUREMENT

<sup>1</sup>H observed homonuclear NOE

<sup>1</sup>H observed homonuclear ROE

<sup>1</sup>H observed heteronuclear shift

<sup>1</sup>H observed heteronuclear shift

heteronuclear shift correlation

<sup>1</sup>H observed longrange

correlation

correlation

correlation

correlation

correlation

NOESY

ROESY

HMQC

HSQC

HMBC

Table. 2.1 Automatic measurement methods and measurements						
Method	Measurement	Sequence name	Template			
Proton	1D <sup>1</sup> H normal measurement	proton.jxp	Standard			
Carbon	1D <sup>13</sup> C normal measurement	carbon.jxp	Standard			
DEPT	Automatic determination of <sup>13</sup> C atomic group by means of DEPT	dept.jxp	Standard			
COSY	<sup>1</sup> H observed homonuclear shift correlation	cosy.jxp	Standard			
TOCSY	<sup>1</sup> H observed homonuclear shift	tocsy.jxp	Standard			

noesy.jxp

roesy.jxp

hmqc.jxp

hsqc.jxp

hmbc.jxp

The Delta program provides the following standard methods for automatic measurement.
Table. 2.1 Automatic measurement methods and measurements



# 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 **X** mark is displayed on the left side of the sample name.

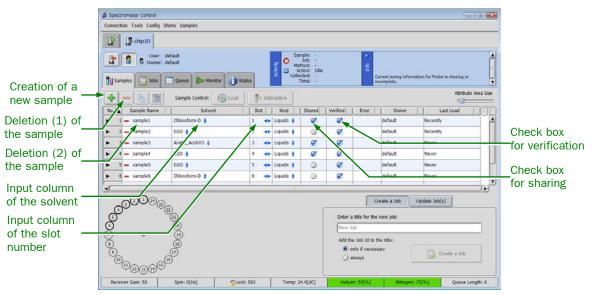


Fig. 2.1 Name of each part in the Samples tab

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### 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.

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

Parameter	Description	Defalt
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.

💰 Spectrometer Control								10	
Connection Tools Config	Shim: Samples								
C chip10									
	default default			mple: - Job: - thod: - ction: Idle		► gu			Î
Jobs 📋 Jobs	Queue () Monitor	r 🕕 Status		cted: - Time: -			Current tuning inform incomplete.	ation for Probe is missing or	
4-60	Sample Control:	🔂 Load 🛛 🐉 Inte	ractive					Attribute	Area Size
No. A Sample Name	Solvent	Slot	Kind	Shared	Verified	Error	Owner	Last Load	
1 — sample1	Chloroform-D	1 🔹	Liquids 🔷	ø	ø		default	Recently	A
r z - rampiez	020 🛊	2 🕶	Crigaids 🛊	0	1		default	Recently	7 11
- 3 - sample3	Acetic_Acid-D3 \$	3 📣	Liquids 🛊	Ø	Ø		default	Never	
+ 4 = sample4	020 🛊	4 🐢	Liquids 🛊	ø	Ø		default	Never	
+ 5 = sample5	D20 🛊	5 📣	Liquids 🛊	0	ø		default	Never	1 1
🕨 o 🛶 sampleo	Chiorororm-D 💡	0 41-	Liquids 💡	9	V		derault	Never	-
									1.
0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0						6	reate a Job	Update Job(s)	
de la	(22)				Enter a	title for the	new job:		
()	(20)				New 3	do			_
•	(1)				Add th	e Job Id to I	the titles		-
(3)	(18)					nly if neces		Create a Job	
(9)	(17)					lways	-r (	with these Samples	
(1)(1)(1)(1)(1)(1)(1)(1)(1)(1)(1)(1)(1)(									
Receiver Gain: 50	Spin: 0[Hz]	GLock: 346	Temp: 2	25.1[dC]	Heliur	n: 50[%]	Nitrogen	75[%] Queue Le	ngth: 0

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.

	Create a Job	Update Job(s)
inter a title for t	the new job:	
New Job		
Add the Job Id t	to the title:	

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 solution 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**.

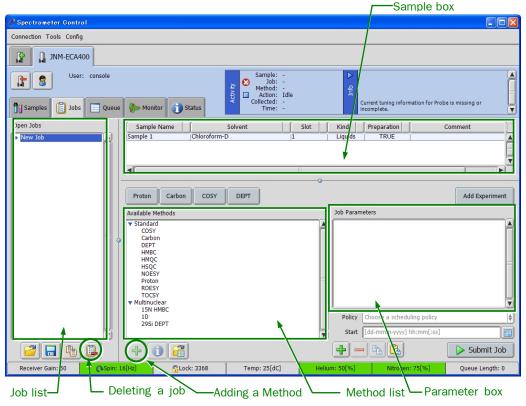


Fig. 2.5 Name and layout of each part in the Jobs tab

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".



Spectrometer Control								
ionnection Tools Config								
😭 🚺 chip10								
User: defau Owner: defau	t	nitor Status	€ S J Meth Acti Collect	on: Idle	1		ition for Probe is missing or	ĺ
Open Jobs		le Name Chiorof	Solvent	Slot	Kind	Preparation	Comment	
Proton     Proton     O:02     Carbon     Carbon     Carbon     O:51     HMQC							1	
HMQC 0:08	Proton		TTEID Y		Metho	1 Parameters	Add Exper	riment
	V Standa	ard				force_tune	10	1
	Carl DEP	bon T				shim_mode	AUTOSHIM OFF	
	HME	ic X		_	•	rgain	50	
	HSC NOT	SY			Þ	sn_ratio	0	
	TOC	SY SY				- 6 3	Submi	t loh
	4	CLock: 170			4		Supuri	1 100
Receiver Gain: 50 S	pin: 0[Hz]		Temp: 25		elium: 50[9	] Nitrogen:	75[%] Queue Len	

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 ".

Connection Tools Config Experiment			
🚱 🎼 chip10			
User: default Owner: default	s Dan Monitor	Status	g or
Open Jobs	Sample Name	Solvent Slot Kind Preparation Comment	
▼ New Job ▼ Proton (Proton	Header Instrumen	Chloroform-D (1 Liquids TRUE)	eters 2
	storage_filename	\$(SAMPLE)_Proton S(SAMPLE)_Proton	
	filename	proton	
	comment	single_pulse	-
	auto_filter	Ø	
	auto_gain	0	
	filter_limit	8	
	force_tune	0	
	save aborted	Ø	
T	save_aporced		

Fig. 2.7 Window for setting measurement parameters

**5.** Starting measurement.

After setting the Method Parameters for all the Methods, click the **Submit Job** 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.



# **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	SETT	ING 1D NMR MEASUREMENT CONDITIONS	3-1
3.1	.1 Se	etting 1H Measurement Conditions	
3.1	.2 Se	etting 13C Measurement Conditions	
3.2	STAR	TING AND ENDING MEASUREMENT	3-5
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3.4		hanging the Display Range	



### **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.

Enter a job	Enter a title for the new job:
name.	Add the Job Id to the title:
	Only if necessary     always

The **Jobs** tab opens automatically.

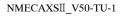
🤣 Spectrometer Control					
Connection Tools Config Experiment					
1 JNM-ECX300					
User: default Us	Monitor 🕤 Status	Sample: CAN Action: Idle Collected: - Time: -			
Open 3dbs	Sample Name CAHE Chloroform	Solvent D	Kind	Comment	
	•				
	Proton Carbon COS	IY DEPT			Add Experiment
	Available Methods  Standard		Job Para	meters	
≓ ¢	Carbon Carbon and APT Carbon and APT 155 Combination 1 Combination 2 Edited DEPT Rock Gradient COSY Gradient LOGY Gradient HMBC Gradient HMBC Gradient HMBC TOCSY Gradient HMOC TOCSY				
	Gradient HMQC with Y proj Gradient PS NOESY Gradient TOCSY		Policy Start		‡ 
	+ 🛭 🚰		+		Submit
Receiver Gain: 62 CSpin: 14[H:	]	Temp: 24.4[dC]	Helium: 100[%]	Ntrogen: 100[%]	Queue Length: 0

Fig. 3.1 Jobs tab

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

👌 Open Experimen	nt 💿 🖻	
File Options Go	🛞 Recent 🦙 Favorite Files	
experiments		]
✓ PLACES	S chip10 - Authenticated as default	
🍖 Local	▶ 1d	$\square$
👔 Global	> 2d > 3d	
Desktop	basic cosy	
Documents	diffusion     dqf cosy	
▼ DEVICES	▶ ecosy ▶ fgshim	
<sub> </sub>	▶ft	
chip10	> glp > hetcor	
▼ FAVORITES	▶ hmbc	

Fig. 3.2 Open Experiment window



- 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.jxp 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.

Spectrometer Control ormection Taols Config Experim	ent				
P D chip10					
User: defaut Corner: defaut	нов (Для Mantor ) () )		ample: - Job: - lethod: - Active: Jalle letted: - Time: -	Current tuning information concerned	tion for Probe is missing or
aper Jobs	Sample Name	Selvent	Siot	Kind Preparation	Comment
	Header Instrument	t Acquisition Pulse D	Logian Cavorties		Add Parameters
	storage_filename	\$(SAMPLE)_proton	S(SAPPLE)_S(EXP./ivrus	ne)	A ADE PARAMEERES
	storoge_filename filename	\$(SAMPLE]_proton	Contrast of Contrastores of	ne)	
e.	slorage_filename filename comment	\$(SAMPLE)_proton [proton [single_pulse	Contrast of Contrastores of	na) : .	
	storoge_filename filename	\$(SAMPLE]_proton	Contrast of Contrastores of	ted)	U AGE PARAMETERS
	storage_filename filename cerrment auto_filter	\$(SAMPLE)_proton proton single_pulse	Contrast of Contrastores of	ne)	U AGE PARAMETERS
	storoge_filename filename cerrment auto_filter auto_gen	\$(SAMPLE)_proton [proton [single_pulse ] ]	Contrast of Contrastores of	te)	1 And Parameters
	storage /fename filerans comment avo_biter avo_biter filer_binit force_bine	\$(SAAPLE)_proton proton single_puble	Contrast of Contrastores of	tre)	1 And Paramotes

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 <b>sample name_filename</b> .)				
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 <b>Delta Console</b> window and the <b>Spectrometer</b> <b>Control</b> 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 .jxp.

<sup>\*\*</sup> 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 meas	surement
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

 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.
- 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 **Selection**. 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 <b>sample name_filename</b> .)
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 measured	surement
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.

	au	o_gan	0				
	filte	r_limit	8				
	ford	:e_tune	0				
	v sav	e_aborted	ø				
20		Deliver data	i automatica	lly		(	Submi
Receiver Gain: 32	(Spn: 15[Hz]	PLod:	ee	Temp: 23.6[dC]	A Helum: 0(%)	A Nitrogen: 0(%)	Oueue Length: 0

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.

Ø JEOL Delta v5.0	
File Options Acquire Process View Analyze Tools	
	<ul> <li>▼</li> </ul>
Delta NMR Processing and Control Software v5.0-Beta (05-14-09 16:33 [build 523]) [Windows] Copyright 1990-2009 by JEOL USA, Inc.	
1	

The File Browser window opens.

🧳 File Browser	
File Options Go (	🔋 Recent 🦙 Favorite Files
Data Servers	
▼PLACES	
🍖 Local	▶ chip1
🌍 Global	chip10     eca32
🍣 Data	JNM-ECX300 nmxps400
Desktop	▶ phantom
	▶ scc13 ▶ scc2
Documents	▶ scc3
- DEVICES	▶ scc4
🚗 Disk Drives	
Data Servers	Ļ
Spectrometers	
▼FAVORITES	

### Fig. 3.4 File Brower window

- 2. Click Data Servers Data Servers at the lower left in the File Brower 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.



🧷 File Browser		
File Options Go 🌾	🔊 Recent 🔶 Favorite Files	
Data Servers		
▼PLACES	S JNM-ECX300 - Authenticated as default	Disconnect
🍖 Local	▶ chip1	
🌍 Global	<ul> <li>chip10</li> <li>eca32</li> </ul>	
😂 Data	▼ JNM-ECX300 single_pulse	
Desktop	single_pulse single_pulse	= 🔁
Documents	single_pulse single_pulse	
▼DEVICES	▶ nmxps400	
🧼 Disk Drives	<ul> <li>phantom</li> <li>scc13</li> </ul>	
🥃 Data Servers	▶ scc2	<b>T</b>
Spectrometers		
▼ <u>FAVORITES</u>	1D - 16384 - default @ DELTA2_NMR Creation:15-MAY-2009 09:50:03 single_pulse	



### **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.

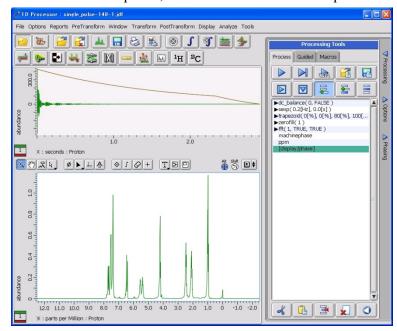


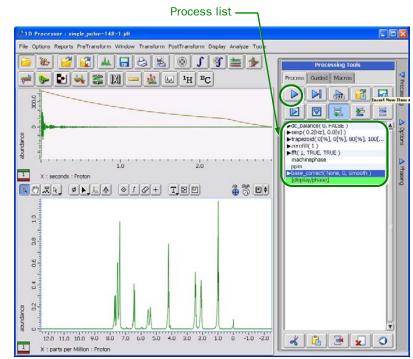
Fig. 3.5 1D Processor window



### **3.4.1** Automatic Baseline Correction

- 1. Click the solution in the 1D Processor window. An item, **Base Correct**, is added to the process list.
- **2.** Click the Process **b** button.

The spectrum with the baseline corrected is displayed in the lower spectral display area.

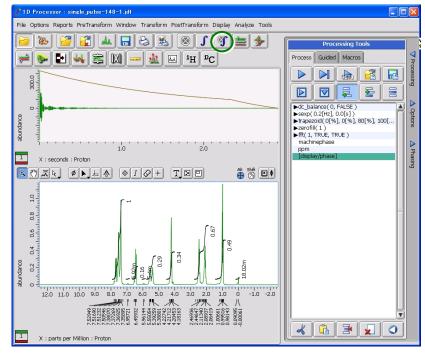




#### 3.4.2 Automatic Peak Detection and Automatic Integration

#### Click the Statton in the 1D Processor window.

Automatic peak detection and automatic integration are carried out, and the results are displayed in the lower spectral display area in the **1D Processor** window.



#### 3.4.3 Plotting

The standard method of plotting is as follows.

Click the 👌 button in the 1D Processor window.

1D Processor : single_pulse=148=1.jdf		
File Options Reports PreTransform Window Transform PostTransform Display Analyze Tools		
► 🖉 🖾 👞 🗟 🖳 💩 🖬 🖋 🛁	Processing Tools	
🤿 🕪 🛃 🇮 🕅 🚥 🎪 🗔 <sup>1</sup> H <sup>13</sup> C	Process Guided Macros	pen Processing I

The processed spectrum in the lower display area is plotted on paper.

#### 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.

<u> 4 7. (% 2</u>	Ø► ₩	+ & F &	Alt Shift 🔳 🖨

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.

\_ \_ \_ \_ \_ \_ \_ \_

Q	Zoom:	To expand, reduce, or move spectra.
ংশ	Pan View:	To move the area after expanding spectra.
ĩ	Amplitude gain:	To adjust the amplitude of spectra.
A,	Select:	To select data or a geometry.
ø	Phase correct:	To correct the phase of spectra.
	Copy position:	To copy any area of spectra.
Æ	Peak threshold:	To set the position of the baseline of spectra.
Ŷ	<b>Reference:</b>	To set a chemical shift reference axis marker.
\$	Peak:	To execute a peak pick.
f	Integral:	To perform integration.
	Measure:	To measure distance between peaks
+	Cursor:	To display the horizontal and vertical lines.
Ŧ,	Annotation:	To display an annotation in the geometry.
D	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".

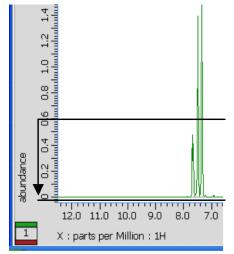
#### Expanding and resetting spectra in the 1D Processor window

#### 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 **Q**.

- 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.



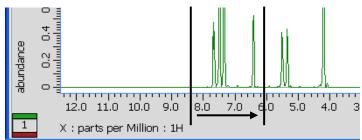
The spectrum is expanded vertically.

#### • 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.

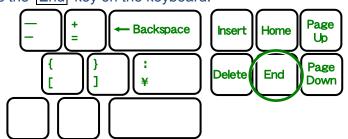


The spectrum is expanded horizontally.



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

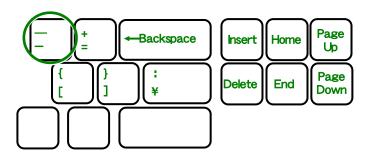
Press the End key on the keyboard.



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.



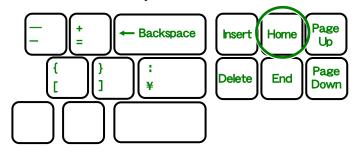
When you press the \_\_\_\_\_ key, both vertical and horizontal expansions return to their previous values.

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.





# **2D NMR MEASUREMENT**

4.1 SI	ETTING 2D NMR MEASUREMENT CONDITIONS.	
4.1.1	HH COSY Measurement	
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4.1.3	Setting the Observation Range Through the 1D Data	
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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 <sup>1</sup>H–<sup>1</sup>H homonuclear 2D NMR (HH COSY) and <sup>13</sup>C detection heteronuclear 2D NMR (HETCOR).

#### 4.1.1 HH COSY Measurement

 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.

	Enter a title for the new job:	
Enter a job name	New Job	
	Add the Job Id to the title:	

The **Jobs** tab opens automatically.

Spectrometer Control		
Connection Tools Config Experiment		
1 JNM-ECX300		
User: default Owner: default	Sample: CAVE Action: Ide Cheltesta - Tret: -	Á
Open Jobs		
New Job	Sample Name Solvent Kind Comment	
	•	
	Proton Carbon COSY DEPT	Add Experiment
	Available Methods Job Parameters	
≓ o 	Standad     Gabon and APT     Cabon and APT	
		Submit
Receiver Gain: 62 OSpin: 13[H	z]Lock: 587 Temp: 24.6[dC] Helium: 100[%] Nitrogen: 100[%]	Queue Length: 0

#### Fig. 4.1 Jobs tab

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

4-2

\land Open Experimen	t – O 💌
File Options Go	🛞 Recent 🦙 Favorite Files
experiments	
▼ PLACES	S chip10 - Authenticated as default
🍖 Local	▶ 1d
🌍 Global	> 2d
Desktop	basic cosy
Documents	diffusion     dgf cosy
▼ DEVICES	ecosy     fgshim
<sub> </sub>	▶ ft
chip 10	▶ glp ▶ hetcor
▼ FAVORITES	▶ hmbc

Fig. 4.2 Open Experiment window

- 3. Click the state button in the Open Experiment window.
  - A list of the Experiment files<sup>\*</sup> in the Global directory<sup>\*\*</sup> is displayed.
- 4. After double-clicking basic in the Open Experiment window, select the measurement mode cosy.jxp to highlight it, and click the Jutton. 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.

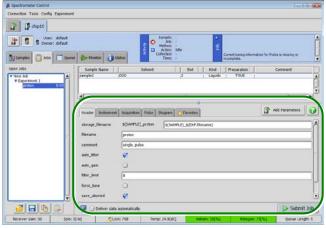


Fig. 4.3 Experiment Tool window

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



<sup>&</sup>lt;sup>\*</sup> The Experiment files contain the measurement modes, standard measurement conditions, and steps for processing data. The file type is **.jxp**.

#### 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

I uise.	
filename	Data name that is saved (The saved file name is created
	as <b>sample name_filename</b> .)
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

 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 **Solution** 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.jxp to highlight it, and click the **Solution**. 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 <b>sample name_filename</b> .)
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) Observation range (ppm) x sweep Irradiation nucleus: Carbon y\_domain y\_offset Irradiation center frequency (ppm) y\_sweep Irradiation range (ppm) x\_pulse <sup>1</sup>H pulse width to be used for measurement y\_pulse <sup>13</sup>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.

🔊 JEOL RESONANCE

#### 4.1.3 Setting the Observation Range Through the 1D Data

For the measurement of  ${}^{1}H{-}{}^{1}H$  homonuclear 2D NMR, the  ${}^{1}H$  1D data is essential. Also, for the measurement of  ${}^{1}H$  detection heteronuclear 2D NMR or  ${}^{13}C$  detection heteronuclear 2D NMR,  ${}^{1}H$  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 "**I** Setting the observation range using the 1D data".

#### Loading the 1D Data

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

File Options Acquire Process View Analyze Tools	
20	e
	A
Delta NMR Processing and Control Software v5.0.2 [Windows]	
Copyright 1990-2011 by JEDL RESONANCE, Inc. License Will Expire: 1-APR-2012	

The File Browser window opens.

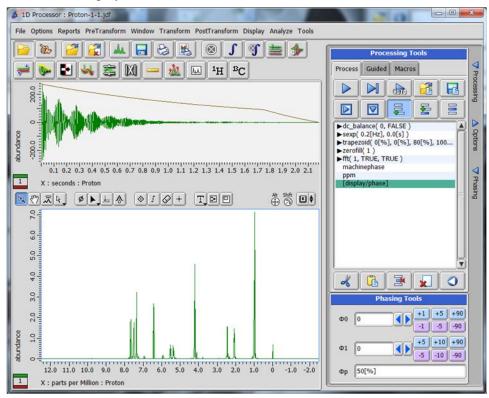
File Opuolis Go (	Recent ☆ Favorite Files	
test		
<ul> <li>PLACES</li> <li>              Local          </li> <li>Global         </li> <li>Data         </li> <li>Desktop         </li> <li>Desktop         </li> <li>Documents         </li> <li>Decuments         </li> <li>Decuments         </li> <li>Documents         <ul> <li>Desktop</li> <li>Documents</li> </ul> </li> <li>Desktop         <ul> <li>Desktop</li> <li>Desktop</li> <li>Desktop</li> <li>Desktop</li> <li>Desktop</li> <li>Desktop</li> <li>Spectrometers</li> <li>FAVORITES</li> </ul> </li> </ul>	Process_data Carbon-1 COSY-1 DEPT135[deg]-1 Proton-1           ID         16384c, 274k - [s] - R - 64L - delta Revision: 4-APR-2011 15:09:07 / Creation single_pulse - [Yesample]	

#### File Browser window

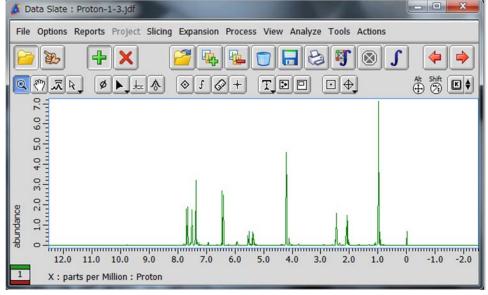
2. Click the name of the data file you want to load in the list box.

#### 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 solution (located at the right side of the X\_domain box) in the **Acquisition** tab of the **Experiment Tool** window.

Spectrometer Control Connection Tools Config Experiment	1					- 0 - *
P D pcnmr2						
User: delta	e 🕼 Monitor 🚺	Status	Sample: - Job: - Method: - Action: Idle Collected: - Time: -		2	on for Probe is missing or
Open Jobs	Sample Name	NONE	livent	Kind Prepar Liquids TF	ation	Comment
▼ New 3ob ▼ COSY COSY 0:06						č
	Header Instrume	ent Acquisition	Pulse Diagram	Favorites		Add Parameters
	x_domain	Proton				1 🖬 🕷
	x_offset	[5[ppm]				
	x_sweep	[15[ppm]				
	x_points	1024				
	scans	1				
	x_prescans	4				
	mod_return	1				
Ŧ	y_points	256				
	Deliver da	ita automatically				Submit Job
Receiver Gain: 50 Spin:	0(Hz)	ock: 3329	Temp: 24.9[dC]	Helium: 50[*	6] Nitrogen: 75	Queue Length: 0

The mouse pointer changes to 8 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.

Header Instrumen	t Acquisition Pulse Diagram Cravorites	Add Parameters
x_domain	Proton	🛊 🗃 🐻 📥
x_offset	[5[ppm]	
x_sweep	[15[ppm]	
x_points	1024	
scans	4	
mod_return	1	
x_prescans	4	
y_domain	Carbon13	t) 🐻 🐻
y_offset	[85[ppm]	
y_sweep	[170[ppm]	V

The mouse pointer changes to 8 after this operation.

**3.** Move the mouse pointer into the spectrum of the 1D data (in this case, <sup>1</sup>H 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 solution (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, <sup>13</sup>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.

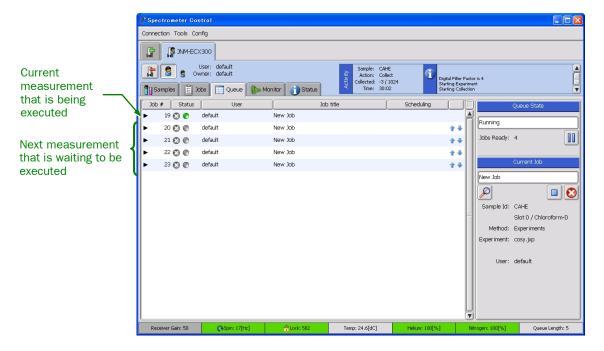
## 4.2 STARTING AND ENDING MEASUREMENT

#### 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.

	3	Header Instrument	Acquisition Pulse	Diagram 🔅 Favo		meters
		storage_filename	\$(SAMPLE)_cosy	\$(SAMPLE)_\$(EXI	?.filename)	
		filename	cosy			
	1 Y	comment	gradient absolute va	alue cosy		
		auto_filter	ø			
		auto_gain	0			
		filter_limit	4			
		force_tune	0			
	-	save_aborted	Ø			
2 8 6 3	1	📮 🗍 Deliver data a	utomatically			Submit Jo

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.





#### Ending measurement

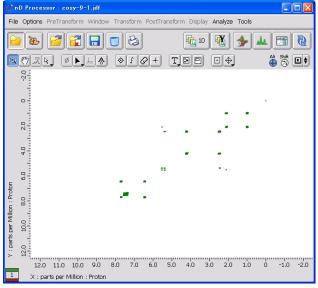
If the **Deliver data automatically** check box was selected, the **nD Processor** window opens after completion of the measurement.

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 0 button to display the process list. If you click the 0 button again after changing the process list, processing will be executed again according to the changed conditions.

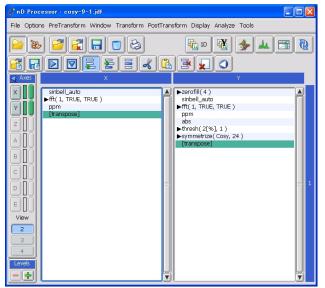


Fig. 4.5 nD Processor window (Process list display screen)



4-10

4.3.1	Displaying Data Using 2D Viewer							
	<ul> <li>Click the model</li> <li>button in the nD Processor window.</li> </ul>							
	🔗 nD Processor : cosy-9-1.jdf 📃 🗖 🔀							
	File Options PreTransform Window Transform PostTransform Display Analyze Tools							

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

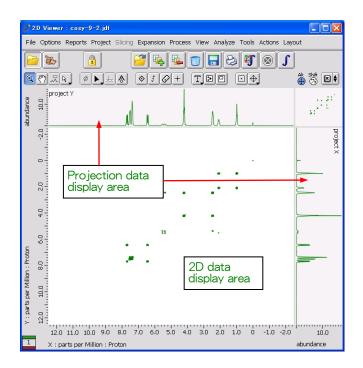


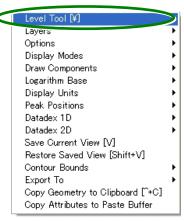
Fig. 4.6 2D Viewer window

#### 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.

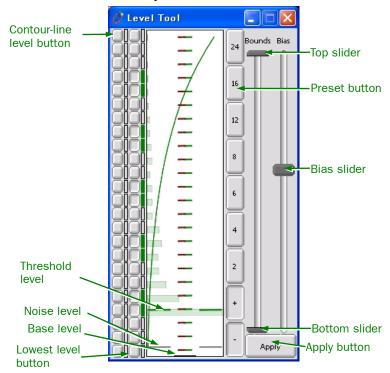


Fig. 4.7 Level Tool window

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: Top slider: Bottom slider: Bias slider: It is a button that shows the lowest strength level. Determines the maximum intensity of the contour lines. Determines the minimum intensity of the contour lines. 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.



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

Solution 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.

🕻 2D Viewer : cosy-9-2.jdf					
File Options Reports Project	t Slicing Expansion Pro	cess View Analyze	Tools Actions	Layout	
	3		1	1D Views	•
				Load 1D 🕨	Load X Slice
<u>a (7 ze</u> ) / <u>1 ze</u>	- & I & +			Show Overview	Load X Projection
project Y	.1			1	Load Y Silce
	N/\N	/			Load Y Projection
					Load X + Y Slices
		·	- 12	2	Load X + Y Projections

The **Open file** window opens.

#### 4 2D NMR MEASUREMENT

🧳 Open file		
File Options Go	☆Favorite Files	
data		
✓ PLACES	Proton-6	
~	Proton-7	
🍖 Local	Proton-8	
A	Proton-9	
🌍 Global	single_pulse-1	
😂 Data	single_pulse-10	
📚 Data	single_pulse-100	
Desktop	single_pulse-101	
Pesktop	single_pulse-102	
🖹 Documents	single_pulse-103	
E Docaments	single_pulse-104	
- DEVICES	single_pulse-105	
	single_pulse-106	
🌎 Disk Drives	single_pulse-107	
😑 Data Servers	single_pulse-108	
Data Servers	single_pulse-109	▼1
Spectrometers		
▼ FAVORITES		

**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.

🥬 2D Viewer	: cosy-9-2.jdf						
File Options	Reports Project	Slicing Expansion	Process View	v Analyze	Tools	Actions Layout	
<b>E B</b>		2		8	g	S 1	

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

File Options Reports Project	Slicing Expansion Proce	ss View Analyze Ti	ools Actions Layout	]	
🔁 👪 🛛				Views 🕨	•
			Loa	ad 1D 🔰	Load X Slice
			∫⊕_ Øsh	ow Overview	Load X Projection
project Y	.1	1			Load Y Since
4				-	Load Y Projection
					Load X + Y Slices
1					Load X + Y Projections

The mouse pointer changes to 8.

- **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 **1**.
- **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.

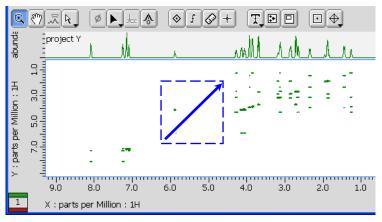
For how to display the pointer, refer to Section 3.4.4. For the details of the pointer, refer to the separate volume, "Data Processing" user's manual.

#### 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.



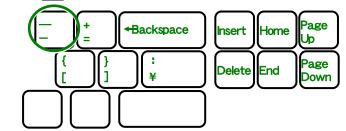
**4.** Release the left mouse button.

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.



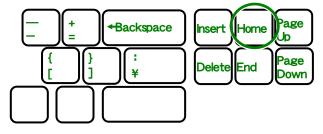
When you press the \_\_\_\_\_ key, both vertical and horizontal expansions return to their previous values.

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 Home key on the keyboard.

All horizontal and vertical expansions return to their initial values.







5.1	COMMAND FOR STARTING MEASUREMENT 5-1	
5.2	QUEUING	



# 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.

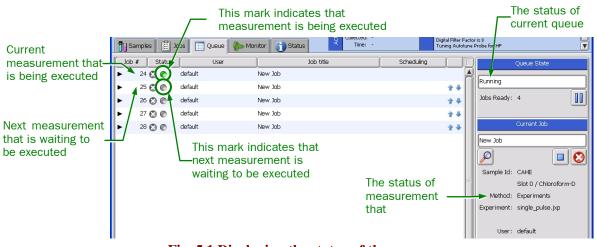


Fig. 5.1 Displaying the status of the queue



# 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.

?	Are you su	are you want to delete the job?	
	Yes	Cancel	



**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.

- 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.

🔊 JEOL RESONANCE

# SAVING AND LOADING DATA

6.1	SAVING NMR DATA	
6.1.	1 Saving the measured data (FID)	
6.1.	2 Saving Processed Data	
6.2	LOADING NMR DATA	



# 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.

K If not specified, the default filename is entered. The name shown on the right side of **storage\_filename** is the default filename.

		APRALEM PL could a class a fateration of a terms for a s	
Filename -	storage_filename	\$(SAMPLE)_single_pulse\$(SAMPLE)_\$(EXP.filename)	I
	filename	single_pulse	
	comment	single_pulse	
	auto_filter	ø	
	auto_gain	0	
	filter_limit	8	
	force_tune	0	Ļ
	save_aborted	Ø	

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	N.
--	----

🔗 1 D. Processor : single_pulse=149=1.jdf		
File Options Reports PreTransform Window Transform PostTransform Display Analyze Tools		
→ ► ► ↓ ≈ M M = + PC	Processing Tools Process Guided Macros	<b>▽</b> Pro

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

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

File Options Go		
data		
✓ PLACES	Carbon-1	
	Carbon-2	
🍖 Local	Carbon-3	
🍻 Global	Carbon-4	
Giobal	Carbon-5	
<table-cell-columns> Data</table-cell-columns>	cosy-1	
~	cosy-2 cosy-3	
Desktop	cosy-4	
-	cosy-5	
Documents	cosy-6	
✓ DEVICES	cosy-7	
▼ DEvices	cosy-7.X	
isk Drives	cosy-7.Y	
~	cosy-8	
🥃 Data Servers	cosy-9	
	GRADIENT_BASIS_2692_Z5_Homospoil_2H-1	Filename
Spectrometers	V	
		input box

Fig. 6.2 Save Data File window

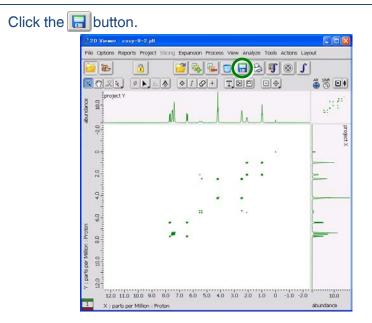
**2.** Type any filename in the filename input box.

When creating a new directory, after clicking the putton, type the directory name in the **Create** box and click the **OK** button.

**3.** Click the **S** 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



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

🔊 JEOL RESONANCE

#### ■ 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.

💰 Save File for Data Slate	e	
File Options Go		
data		
<ul> <li>Cocal</li> <li>Global</li> <li>Global</li> <li>Desktop</li> <li>Desktop</li> <li>Documents</li> <li>DEVICES</li> <li>Disk Drives</li> <li>Data Servers</li> <li>Spectrometers</li> <li>FAVORITES</li> </ul>	3D data etc for Phase 2d standard_data T1_noise	Input box for a filename

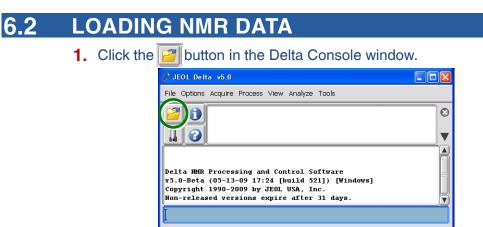
Fig. 6.3 Save File for Data Slate window

**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 Subutton.

The 2D processed data are saved under the specified filename.



The File Browser window opens.

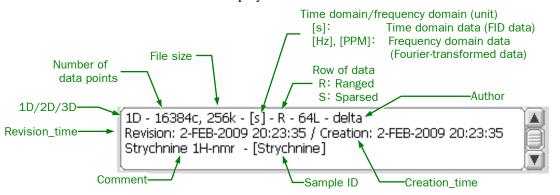
🧬 File Browser		
File Options Go	🛞 Recent 👷 Favorite Files	
data		
VELACES     Coll     Col	Carbon-1 Carbon-2 Carbon-3 Carbon-3 Carbon-4 Carbon-5 Cosy-1 Cosy-2 Cosy-4 Cosy-5 Cosy-6 Cosy-6 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-7 Cosy-9 Cosy-8 Cosy-9 Cosy-1 Cosy-1 Cosy-2 Cosy-4 Cosy-5 Cosy-6 Cosy-7 Cosy-8 Cosy-9 Cosy-1 Cosy-8 Cosy-6 Cosy-9 Cosy-8 Cosy-7 Cosy-9 Cosy-1 Cosy-8 Cosy-7 Cosy-9 Cosy-6 Cosy-6 Cosy-7 Co	<ul> <li>Data version display box</li> <li>File list</li> <li>Data information display box</li> </ul>

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 Volution.

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.

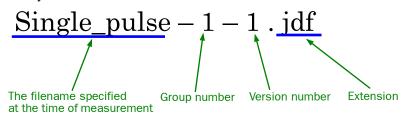


If you display earlier data, select the version number, referring to information in the data information display box, and click the version.



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

.....

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

#### Global directory

Clicking the **Open Data for 1D Processor** window. Because the global directory cannot be used for writing, it is not used to store data.

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

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