# JEOL-Delta Ver.5

# NMR MEASUREMENT USER'S MANUAL

Web page: http://chem-analysis.chem.sci.osaka-u.ac.jp/NMR/index.html Tel: 6787

# 0 Log into a workstation

- Turn on the monitor.
- Press Ctrl+Alt+Del, and then input username and password.

(username : delta password : delta)

# 1.1 Starting up Delta

- Double-click the [Delta] icon.
  - $\rightarrow$ The Delta program starts and the Delta Console appears.





# 1.2 Opening the Spectrometer Control Window

- 1. Click the button in the Delta Console window. The Spectrometer Control window opens. The connectable spectrometers are listed in the Spectrometer Control window.
- 2. Select a spectrometer from the list in the Spectrometer Control window.
- 3. Click the Connect button.



[Spectrometer Control] window

#### 4. Login

The following Authentication dialog box appears. Chose the user name and type the password, and then click the [Own] button.

	Authentication	
	Please enter your login information	
Name	Aoshima ERATO	
Password	Fukase Harada	Authentication
Con	Inaba Ishikawa Kajihara Konno Kubo Mizutani Murata Nakazawa Norisue Ogawa Onitsuka Sato	Please enter your login information Name nmr Password *** Connect Own Cancel
	Suzuki Yamanari console datum	
	, ,	click

If connecting is successful, following window appears.

	🥖 Spectrometer Control										×
	Connection Tools Config Sh	ims Samples									
	JNM-ECS400	← spectrom	neter								
disconnect 🗡	User: ni	mr 🔶 usernar	me	Activity M Col	ample: - Job: - ethod: - Action: Idle lected: -		Duri Cu	rrent tuning inform	ation for Probe is r	nissing or	
	Create a new Sample de Hold Shift to add 8 new	efinition Sample definitions	1) Status		Time: -		inc	omplete.		-	
		Sample Control:	.oad 🦻 Ir	teractive						Attribute Are	ea Size
	No. 🛦 Sample Name	Solvent	Slot	Kind	Shared	Verified	Error	Owner	Last	Load	DDQ
	$\bigcirc \bigcirc $							eate a lob	Lindate Job(s)	1	
		2)(1) (1) (1) (1) (1) (1) (1) (1) (1) (1)				Enter a New Jo Add the O of a	title for the r ob e Job Id to th nly if necessa Iways	new job: e title: ary	Creat	e a Job	
	Receiver Gain: 50	Spin: 0[Hz]	GLock: 1	Temp:	21.3[dC]	Heliun	n: 99[%]	Nitrogen:	71[%]	Queue Lengt	:h: 0

# 1.3 Setting up the Sample

#### 1. Standard Sample Volume

Make the standard sample height (L) about 4 cm, for the 5 mm diameter sample tube. If the sample height is less than 4 cm, resolution adjustment based on the standard shim values set for the sample height of 4 cm or more becomes difficult.

#### 2. Prepare the holder.

Pull the holder so that there is no gap between the rotor and the holder.

Turn the holder left and right a few times to remove any twist of the o-ring while pulling the holder.



3. Make sure that the inside of the probe is empty before inserting the sample into the probe, and that the air used for floating the sample is flowing.

The EMPTY lamp (In the JNM-ECS Series, it is an EJECTED lamp) of the head amplifier chassis indicates that the probe is empty. The flowing air can be confirmed by checking if the sample is floating in the air when inserting the sample tube into the sample insertion port of the Superconducting magnet



# 1.4 Sample Definition



- ① Entering the sample information
- 2 Sample Name
- ③ Name of deuterated solvent
- (d) Slot number (you need to specify this when the spectrometer has the Auto sample changer)
- (5) Verified : Whether you allow to share the sample or not
- 6 Select a sample in the list
- ⑦ Manual Sample Control

### 1.5 Manual Sample Control



- 8 Loading a sample
- (9) Spinning ON/OFF
- 1 Sample temperature : r.t.~30 °C
- 1 Reading the system shim file
- 12 Auto Lock

cf. Manual Gradient Shim (Gradient Shim Tool 🏂 )

System Type : Homosopoil
Nucleus : 2H
Solvent : inputted automatically
Shim Set : Z1~Z4
Tip Angle : 90deg
Scans : 4
X Offset : Select the Calculate and Twice check boxes.
Recvr Gain : Select Calculate. Receiver gain is adjusted automatically.
Relax Delay : 2s
Iterations : Select Auto Converge check box.
Range : Depends on the NMR model.
e.g. ECS400: 30%~70%

ECA500: 28%~73%

Click the Start Shimming button.

🧳 Gradient Shi	mming - JNM-ECS400
Shimming St	catus
System Type	Homospoil_Selective
Nucleus	1H 🔷
Solvent	NONE
	Shim
	✓ Z1
	72
Shim Set	
	₹ 24
	₹ Z5
Tip Angle	90[deg]
Scans	4
X Offset	7.26[ppm] 🧭 Calculate 📋 Twice
Recvr Gain	20 Calculate
Relax Delay	5[s]
Iterations	2 Auto Converge
Range 🝦	25[%] <b>()</b> 73[%] <b>()</b> Calculate
	Preserve Shim Results

- 1 Manual shim
  - $1\,.\,$  Adjust Z1 and Z2.
  - ${\bf 2}$  . Adjust Z1 and Z3. Then Adjust Z1 and Z2.
  - ${\bf 3}\,.\,$  Adjust Z2 and Z4. Then Adjust Z1 and Z2.

## 1.6 Create a Job

💰 Spectrometer Control	
Connection Tools Config Shims Samples	
P INM-ECS400	
User: nmr B S Owner: nmr Method Method Method Method Collected Collected Status	e <b>₽</b>
Sample Control: 🛞 Load 📴 Interactive	
No. Sample Name Solvent Slot Kind Shared	Verified Error Owner Last Load
	Create a Job Update Job(s) Enter a tile for the new job: New Job Add the Job Id to the title:      only if necessary     always     Create a Job     dit is Sample
Receiver Gain: 50 Spin: 0[Hz]	Helium: 97[%] Nitrogen: 88[%] Queue Length: 2
Spectrometer Control Connection Tools Config	
User: nmr Sample: - Sower: nmr Sube: - Method: - Action: If Collected: -	ile Current tuning information for Probe is missing or

IP         IP         JNM-EC5400	
Image: Stample:       Sample:       -	
Open Jobs Sample Name Solvent Slot Kind Preparation Comment	
New Job 1 test Chloroform-D 1 Liquids TRUE	
Proton Carbon COSY DEPT Add Experiment	← 3
Available Methods Job Parameters	
v Standard COSY Coten	
Carbon DEPT HMBC HMQC HSQC NOESY Proton ROESY TOCSY	
Policy Choose a scheduling policy 🕴	
Start [dd-mmm-yyyy] hh:mm[:ss]	
Receiver Gain: 50         Spin: 0[Hz]         GLock: 1         Temp: 21[dC]         Helium: 99[%]         Nitrogen: 71[%]         Queue Length: 0	

① Click the Create a Job with this Sample button, the Jobs tab opens.

Enter your directory for saving your experiment like the following example. cf. nmr/inazumi

 $\Rightarrow$  Form: "The user name as chosed at the time of login authentication dialog box" / "your name"

③ Select a pulse sequence.

2

### 2 1D NMR measurement

1. 1D Proton measurement Select Global / basic / proton.jxp Set up experimental parameters.

• Header

[auto gain] : Select auto gain box [force tune] : Select force tune box

Acquisition
[x\_domain] : [Proton]
[x\_offset] : spectral center [5 ppm]
[x\_sweep] : spectral range [15 ppm]
[x\_points] : Depends on the NMR model.

e.g. ECS400: ~32000 ECA500: ~65000

[scans] : Should be multiple of 4

And click the Submit botton.

1D Carbon measurement Select Global / basic / carbon.jxp
 Set up experimental parameters, as the front paragraph "1. Proton" explained, and click the Submit botton.

Acquisition
[x\_domain] : [Carbon13]
[x\_offset] : spectral center [100 ppm]
[x\_sweep] : spectral range [250 ppm]
[x\_points] : Depends on the NMR model.
e.g. ECS400: ~32000 ECA500: ~65000

• Pulse [irr\_domain] : [Proton] [irr\_noise] : Usually, choose [WALTZ]



#### 3 1D Data Processing

#### 1. 1D processor window

	Title bar	Menu bar	Tool bar/tool bu	tton Process-switc	h tabs				Pointe	r bar
	Hie Options Reports PreTransform	Window Transform PostTra	istorm Displat Analyze Tools				Group	Button	Mode	Function
	<u>≥ ≥ 2 2 4</u> ≓ ▶ 2 4 3 ¤	- 2 ± ⊗ 1 12 ⊡ 13		Processing roots Process Guided Macros		-		Q	Zoom	Expands/reduces the spectrum.
					ocessing		Object/View	<u></u>	Pan	Moves the spectrum.
					A A Pai Sw	anel-display vitch buttons	operation	T.	Gain	Increases/reduces the intensity of the spec- trum peaks.
Input data-	E .	Input	udin dan fan fan dan da	<ul> <li>appendix of a global college for a glo</li></ul>				R.	Select	Selects the object.
	1 X : seconds : 1H			[dspay/prase]	Fhasing			ø	Phase	Corrects the phase.
Output data-					H		Data opera-		Copy Position	Reads the position of the peak.
	0000				list	ocess	tion	L.	Threshold	Sets a threshold level.
	0004 0							\$	Reference	Sets the chemical-shift reference for the axis marker.
	200.0 300 addressed		r II	Phasing Tools	+90	-		$\diamond$	Peak	Picks the peak.
	1000 to the test				Phi	nasing Tools		f	Integral	Integration.
	-4         12.0         11.0         10.0         9.0         8.0           1         X : parts per Million : 1H	7.0 6.0 5.0 4.0 3.	0 2.0 1.0 0 -1.0 -2.0	¢p [50[%]				62	Measure	Displays a measure on the data.
			1				Object crea-	+	Cursor	Corrects the baseline or displays the dif- ference between two points.
$\mathbf{D}$	ata processir	ng for 1D	NMR data	a is roughly o	livided	l into	101	T,	Annotation	Types a comment in the geometry.
foll	lowing five st	ceps.							Molecule	Displays the structural/molecular formula in the geometry.
•	Pre processi	ng before	e FFT						PiP	Creates a partial spectrum from the whole spectrum range, and displays it in the same

- Pre processing before FFT
- Multiplication using window function
- FFT
- Post processing after FFT
- Data display

The processing is stored into the five menus in the 1D processor window: PreTransform, Window, Transform, PostTransform, and Display. You can process the data using the menus.

2. Phase Correction

Expand the phasing panel. Click the  $\checkmark$  button. Then adjust  $\varphi 0$  and  $\varphi 1$ .

#### 3. Setting a Reference

In the Option panel, type the reference value in the X Ref box using the keyboard.

Select the sutton and click the top of the peak to set it as the reference peak.

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	1 X : parts per Million	: 1H				

eometry.

#### 4. Peak detection

Select the 💹 mode, and hold down the mouse button and drag it to set the position of the peak threshold. Click the 🔊 button.

#### 5. Integration

Select the  $\square$  mode. In the spectrum, move the pointer to the left side of the integration range where you want to add an integration curve, and hold down the mouse button. While holding down the mouse button, drag the line to the right side of the range, and release the mouse button.

Select the 🖳 button. Then select the integration curve whose integration you want to set to the normal value.

Open the Options panel, and type the normal value in the Normal box using the keyboard.

#### 6. Printing

Display the contents you want to print (peak detection, integration) on the output data area.

Notes: The contents of this area are printed. Click the [Print Processed Data] button of Tool bar. Clicking the [Delta] tab of the [Print] window will set up the detailed printing. (above fig.: for Windows, following fig.: for Mac)

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# 4 Close the Delta program

