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.

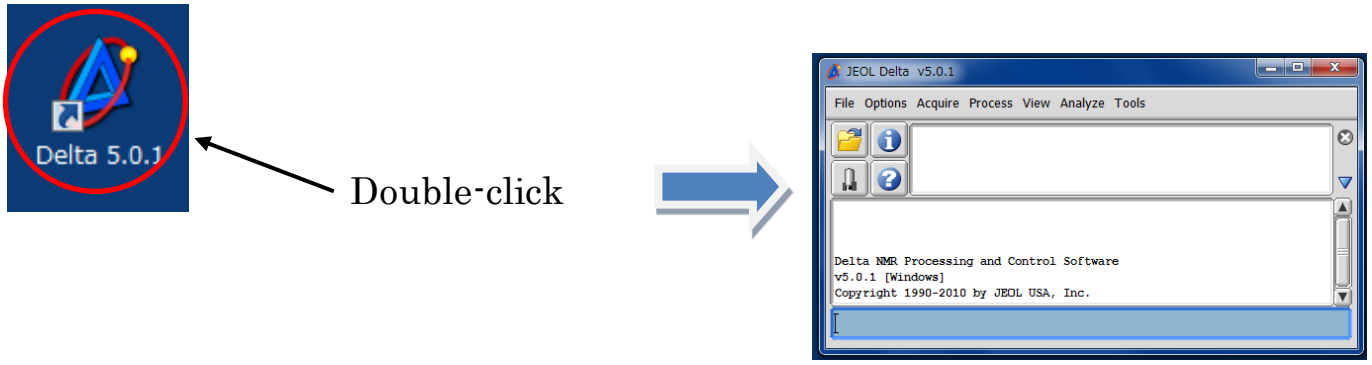
・Press Ctrl+Alt+Del, and then input username and password.

(username : delta　　password : delta)

1.1 Starting up Delta

・Double-click the [Delta] icon.

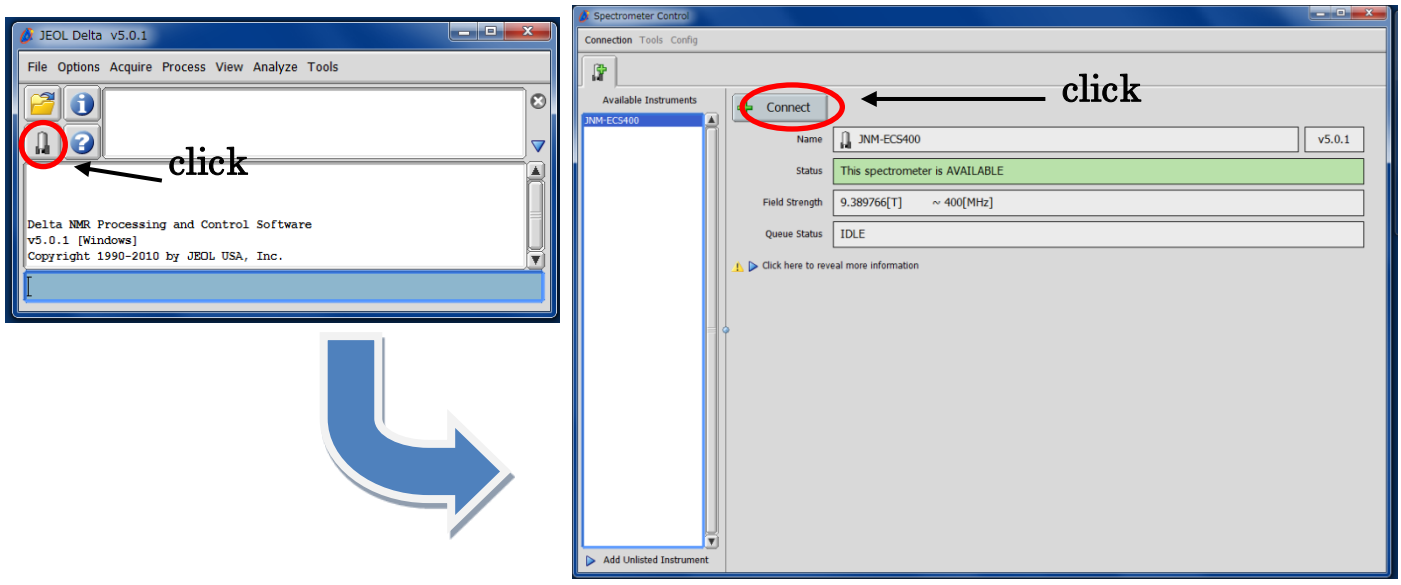
→The Delta program starts and the Delta Console appears.



[Delta Console] window

1.2 Opening the Spectrometer Control Window

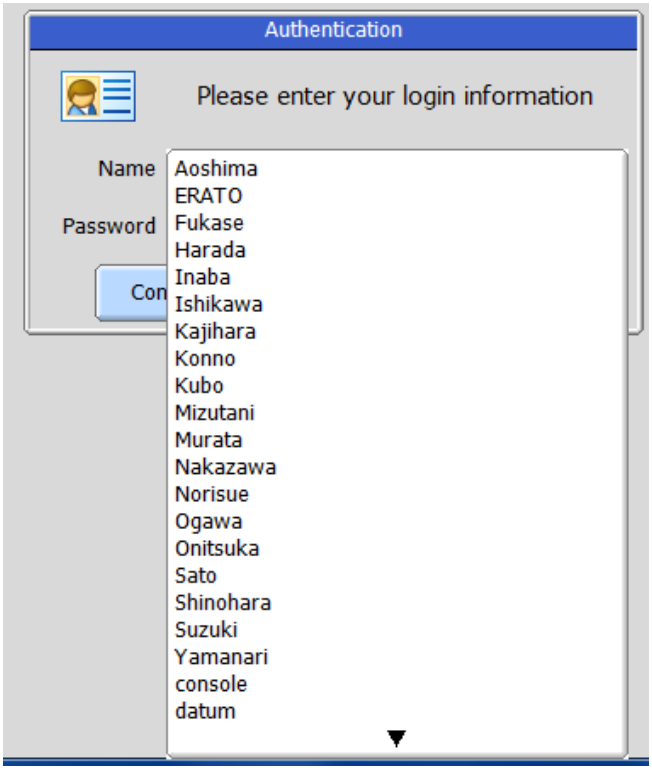
1. Click the button in the Delta Console window. The Spectrometer Control window opens. The connectable spectrometers are listed in the Spectrometer Control window.
2. Select a spectrometer from the list in the Spectrometer Control window.
3. Click the Connect button.

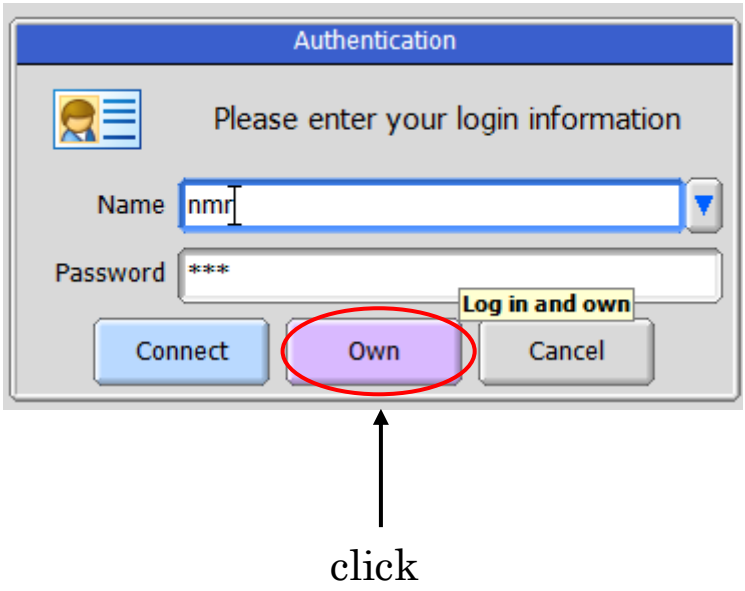


[Spectrometer Control] window

4. Login

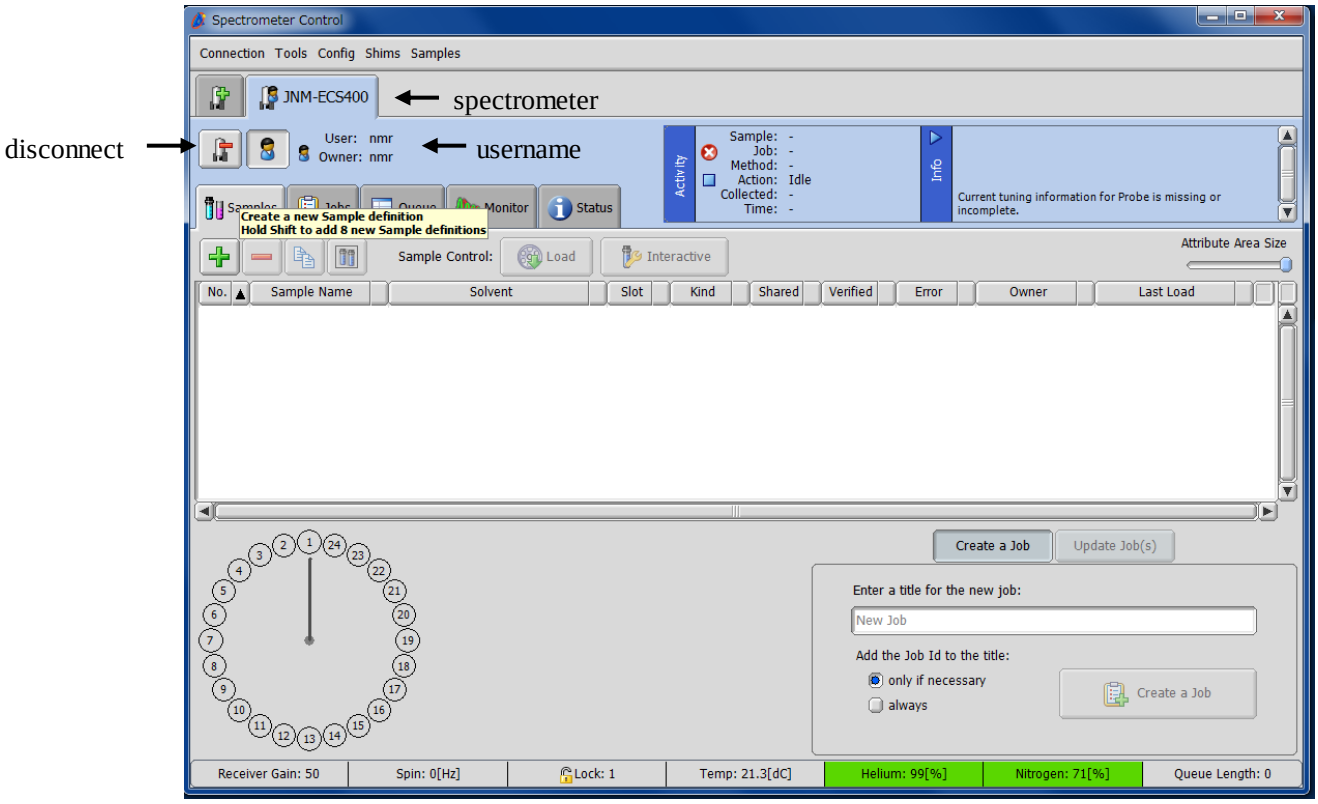
The following Authentication dialog box appears. Chose the user name and type the password, and then click the [Own] button.





click

If connecting is successful, following window appears.



1.3 Setting up the Sample

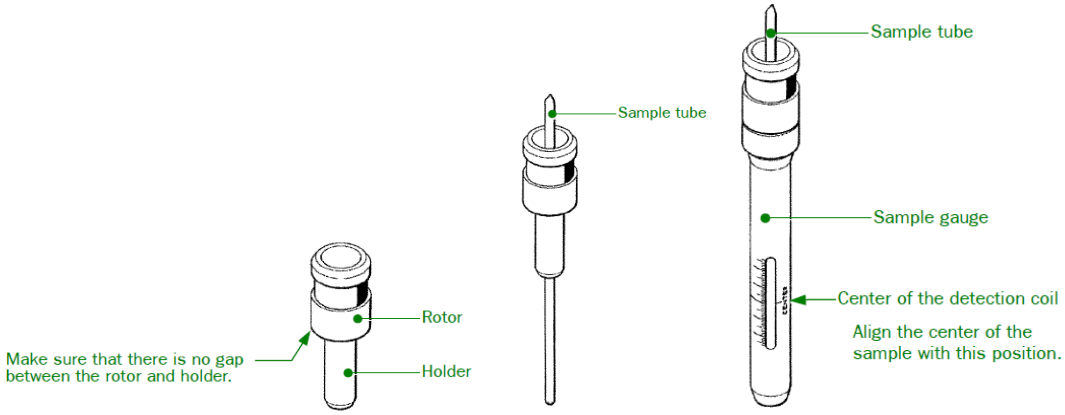
1. Standard Sample Volume

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.



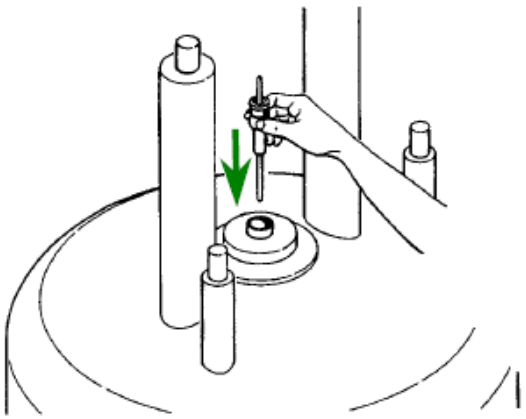
⚠ WARNING

Be sure to wear protective gloves when handling a sample tube containing a poisonous sample.

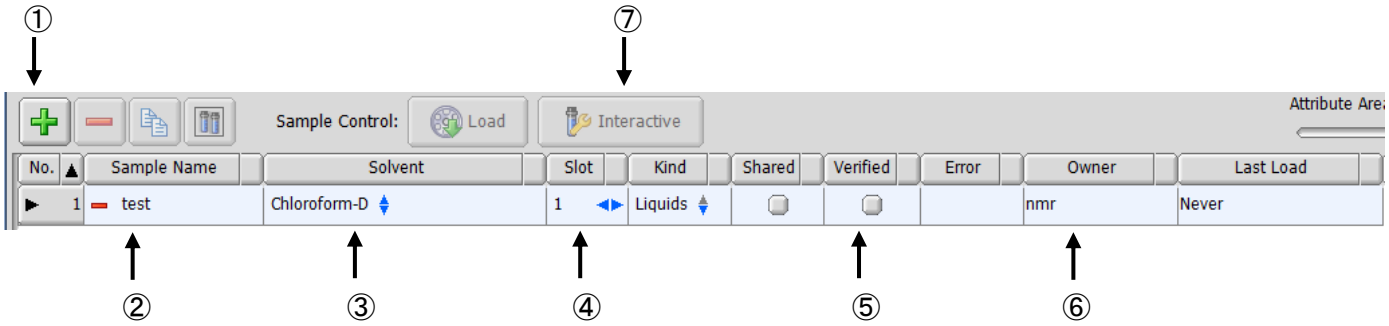
If the sample tube breaks, the use of gloves allows you to avoid contact with the sample.

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

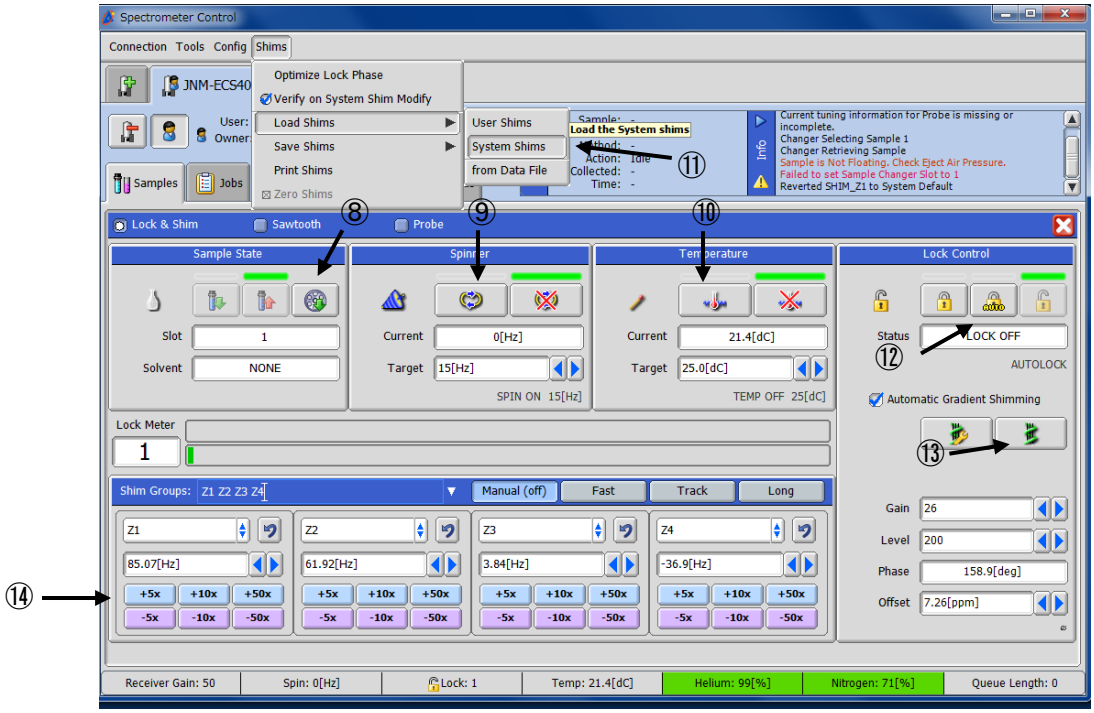


1.4 Sample Definition



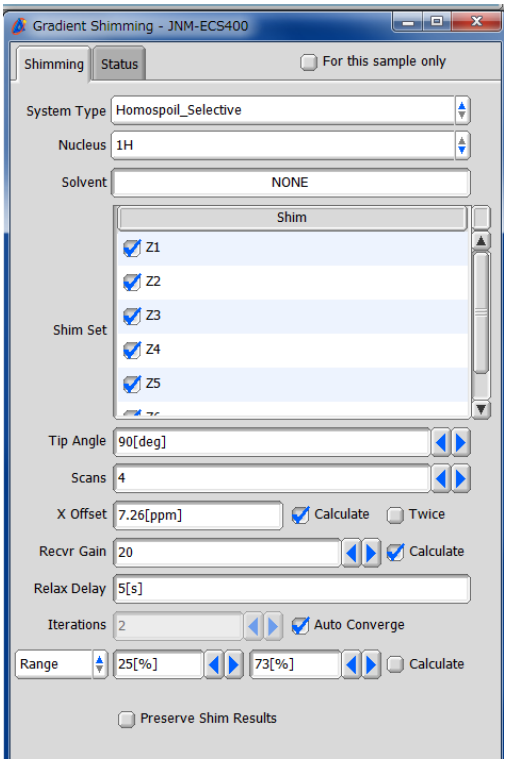
① Entering the sample information
② Sample Name
③ Name of deuterated solvent
④ Slot number (you need to specify this when the spectrometer has the Auto sample changer)
⑤ Verified : Whether you allow to share the sample or not
⑥ Select a sample in the list
⑦ Manual Sample Control

1.5 Manual Sample Control



⑧ Loading a sample
⑨ Spinning ON/OFF
⑩ Sample temperature : r.t.～30 °C
⑪ Reading the system shim file
⑫ Auto Lock

⑬ Auto Gradient Shim



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%

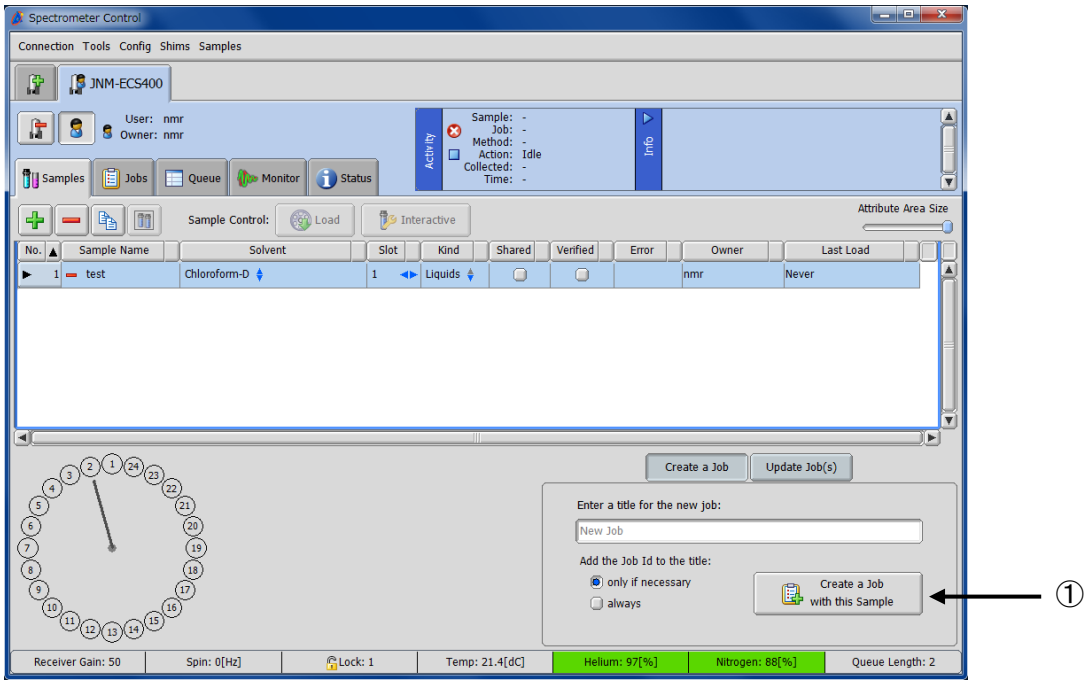
ECA500: 28%～73%

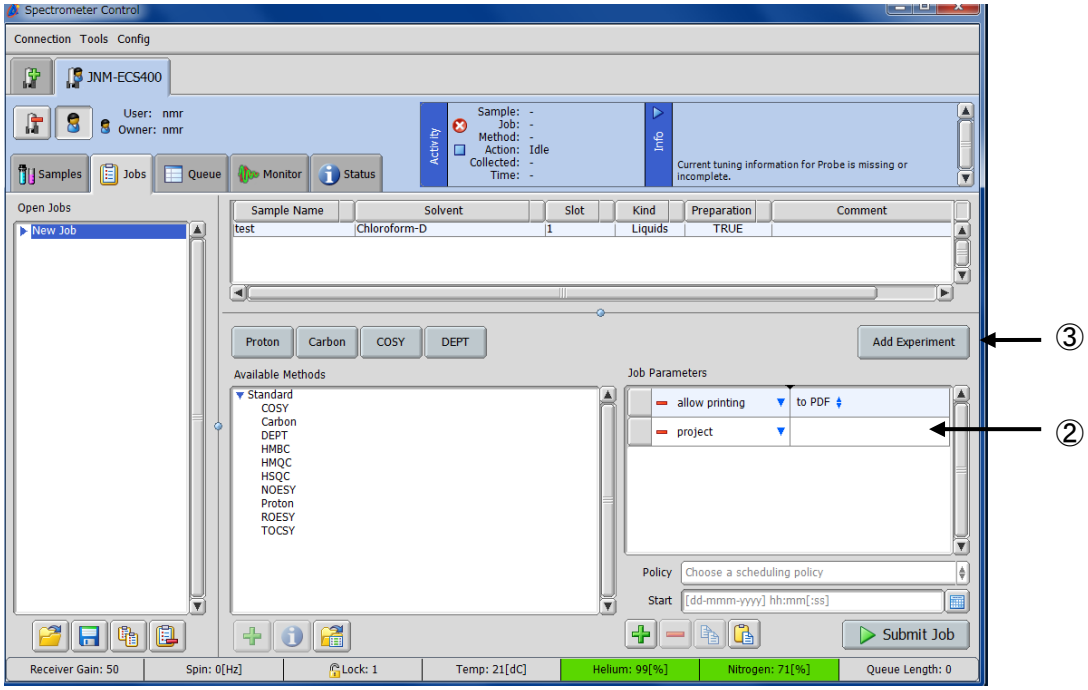
Click the Start Shimming button.

⑭ Manual shim

1. Adjust Z1 and Z2.
2. Adjust Z1 and Z3. Then Adjust Z1 and Z2.
3. Adjust Z2 and Z4. Then Adjust Z1 and Z2.

1.6 Create a Job





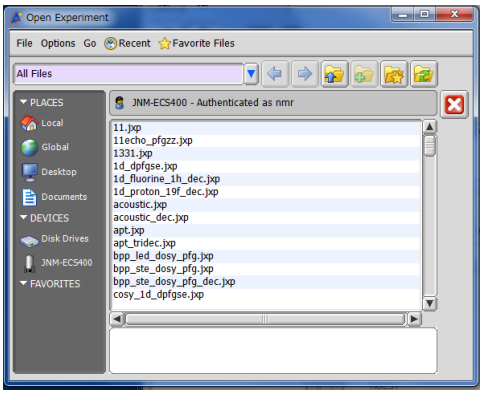
① Click the Create a Job with this Sample button, the Jobs tab opens.

② Enter your directory for saving your experiment like the following example. *cf.* nmr/inazumi

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

③ Select a pulse sequence.

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.

2.　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**

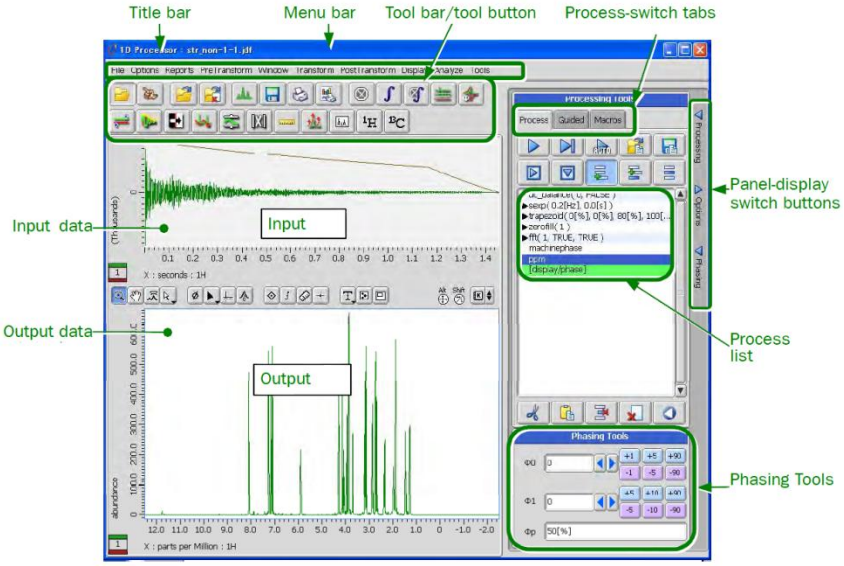
・Pulse

[irr_domain] : [Proton]

[irr_noise] : Usually, choose [WALTZ]

3 1D Data Processing

1. 1D processor window



Pointer bar

Group	Button	Mode	Function
Object/View operation		Zoom	Expands/reduces the spectrum.
		Pan	Moves the spectrum.
		Gain	Increases/reduces the intensity of the spectrum peaks.
		Select	Selects the object.
Data operation		Phase	Corrects the phase.
		Copy Position	Reads the position of the peak.
		Threshold	Sets a threshold level.
		Reference	Sets the chemical-shift reference for the axis marker.
Object creation		Peak	Picks the peak.
		Integral	Integration.
		Measure	Displays a measure on the data.
		Cursor	Corrects the baseline or displays the difference between two points.
		Annotation	Types a comment in the geometry.
		Molecule	Displays the structural/molecular formula in the geometry.
		PiP	Creates a partial spectrum from the whole spectrum range, and displays it in the same geometry.

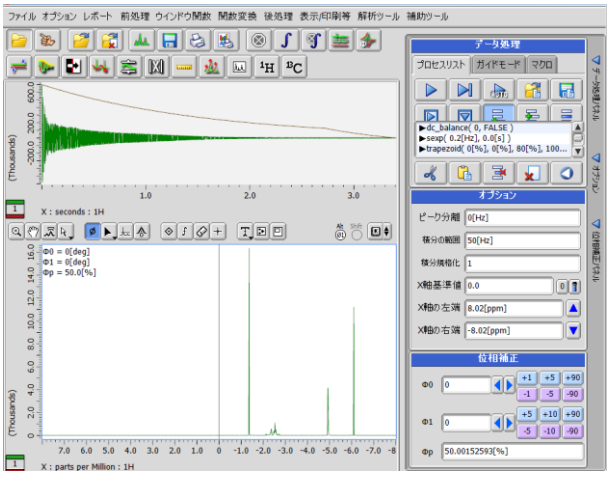
Data processing for 1D NMR data is roughly divided into following five steps.

- 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 button. Then adjust φ0 and φ1.



3. Setting a Reference

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

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

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 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.

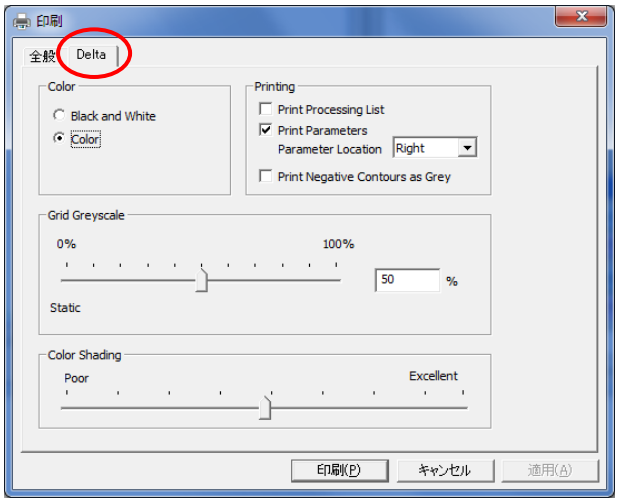
6. Printing

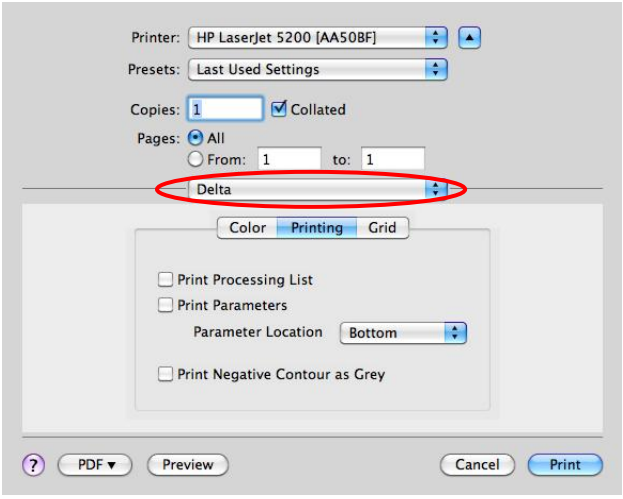
Display the contents you want to print (peak detection, integration) on the output data area.

Notes: The contents of this area are printed.

Click the [Print Processed Data] button of Tool bar.

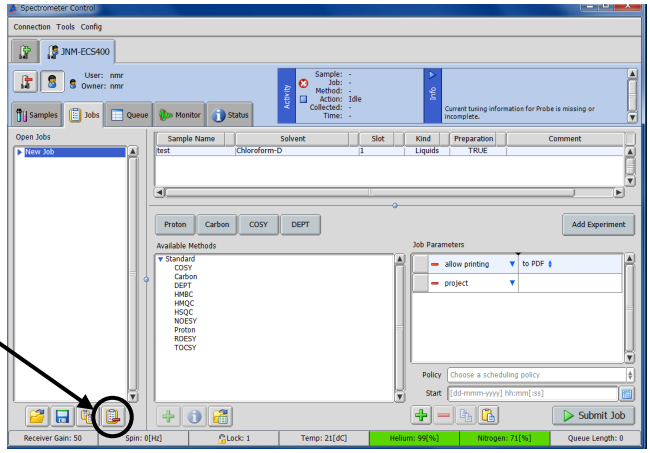
Clicking the [Delta] tab of the [Print] window will set up the detailed printing. (above fig.: for Windows, following fig.: for Mac)



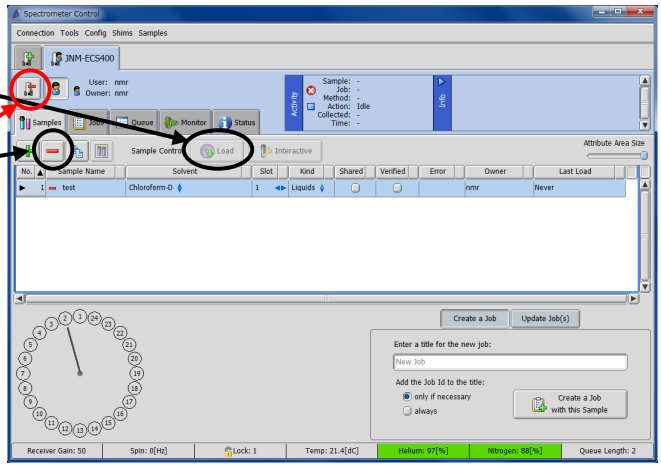


4 Close the Delta program

1. Delete all jobs



2. Eject the sample tube
3. Delete the sample list
4. Click the Unlink button



5. Close the Delta program