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1. PURPOSE

The purpose of this procedure established is to provide clear instructions for steps or orders on how to run sample analysis using JEOL NMR ECX 400MHz SYSTEM JOEL equipment.

2. SCOPE

Applied for user from Chemistry Department.

3. INSTRUCTIONS PROCEDURE

3.1 Delta STARTUP AND CONNECTION

- a) Air Compressor ON mode.
- b) Type Ctrl + Alt + Delete at a time.

Username: delta

Password: delta (displayed as asterisks (*****)).

- c) Double-click the Delta icon on the desktop.
- d) The Delta console window opens.



Delta console window

e) Click the circled button

- indicated in the figure below.
- f) The "Spectrometer control" window opens.

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SPECTROMETER CONTROL WINDOW

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g) Select a spectrometer displayed in the "Spectrometer Control "window.

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per .	Status	This spectrometer is AVAILABLE	and a second
	Field Strength	9.389766[T] ~ 400[MHz]	
	Queue Status	IDLE	
-		Land and the second	
	1. Click here to rev	eal more information	
	0		

h) Clicking the connection button

switches to the "User Authentication" window.

	Authentication					
Please enter your login information						
Name	Account name required					
Password	Password					
Cor	nect Own Cancel					

User authentication window

- i) Type the user name and the password.
 - Name delta
 - Password: delta (displayed as asterisks (*****)

Kata kunci: **** (karakter yang dimasukkan berada dalam paparan 'asterisks')

- j) Click the Own button.
- k) The window view changes as shown below.

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After completion of user authentication

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3.2 SAMPLE PREPARATION

a) Mount the sample tube on the sample rotor and the holder.



Sample mounting

b) Set the sample tube set in the sample rotor and holder so that it floats in the SCM.#When using the auto-sample changer, place the tube in the slot.



Setting the sample tube unit on the SCM

IMPORTANT NOTICE :

- Before making the sample float in the SCM, check that floating air emerges.
- Check that no sample is inside the SCM.
- Do not insert a bare glass tube or the empty rotor and holder.

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3.3 PREPARATION FOR MEASUREMENT

(a) Creating Sample Definitions

A sample definition is: Conditions of a sample to be measured.

1. Click the 🕒 button to create a new sample definition (see the figure below).



Creating sample definitions

Details of the parameters displayed in the sample definitions:

+ - 1	Sample Control: 🛞 Load	toteractive						Attribut	e Area S
No. A Sample Name	Solvent	Slot	Kind	Shared	Verified	Error	Owner	Last Load	TT
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Item	l			Des	cription				
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Sample r	name	Arbitr	ary (us	ed as a	a part of	f a save	ed name)	
Solve	nt Selea	et a solvent. S	Select a	sampl inte	e slot (grated)	if an au	uto samp	le changer i	is
Slot	Se	lect a sample	slot (if	an au	to same	le chai	nger is ir	tegrated).	

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(b) Creating a Job

A job is:

A process of configuring conditions for performing an NMR measurement.

1. Click the utto a Job button displayed in the Spectrometer Control Window.



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2. The window automatically switches to the Job tab.



Jobs tab

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3.4 1H MEASUREMENT

a) Click the

Add Experiment button to select a pulse sequence. 3 1 In ishka 1 S S Over: delta 1) Samples 📋 Sebs 🗇 Queses 🌸 Slot | Pri Preton Carbon COSY DEPT Catter DEPT HMBC HMDC HSQC NOES 0 2 -Submit Job

Selecting a pulse sequence

b) Select basic / proton.jxp. (If not found, search by double clicking **Global**).

ent 🦷

c) Select the pulse sequence to use, and then click the 🚺 button.

The measurement is started by clicking the Submit Job button. d)

Note:

The sample preparation, sample definition creation, job creation and measurement have been

• • • • ~ 1 20

iment File ories: basic, 1d, liquids, singlo_pulse



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described.

These are basic procedures for performing measurements using Delta V5.

This procedure should be practiced in order to achieve a smooth measurement process.

3.5 1H DATA PROCESSING

When the NMR measurement is completed, the measurement result is displayed on the screen.

a) Peak Picking

1. Select the 4 button in the toolbar to determine the threshold.



i) Automatic detection

Peak picking is performed by clicking the 🔞 button.





2. Integration

i. Automatic detection

1. An integral curve can be automatically detected by clicking the **I** button.

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	31	⊗(S	5	=	*
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ii. Adding and deleting a selected integral curve

1. To draw an integral curve separately, select the **f** button in the toolbar.

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2. An integral curve can be created by dragging a selected part on the X axis.



Dragging right and left

iii. Saving processed data

- 1. Select File from the pull-down in the 1D Processor window.
- 2. The data can be saved by using **Save as.**

3.6 PRINTING DATA

a) Clicking the 🛃 button opens the printing option window.

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Status Location: Convert	Friend for the Base Promotocol - Fried Printeer-
Pase Failer C. All C. Solitation C. Plager C. Plager (C. Plag	Number of gapties 1

3.7 13C MEASUREMENT

a) Measurement

- i. Create a new job or add parameters to an existing job.
 - a. Click the Add Experiment button to select basic / carbon.jxp.

b. (If not found, search by double clicking Global).

- ii. Change the parameters as needed.
- iii. The measurements is started by clicking the Submit Job button.

b) Data processing

Perform the processing for the acquired spectrum.

Follow the same procedure as 1H.

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- Phase Correction
- Reference setting
- Peak picking

3.8 DEPT MEASUREMENT

a) Measurement

- i. Create a new job or add parameters to an existing job.
 - a. Click the Add Boortmont button to select basic / dept.jxp.

b. (If not found, search by double clicking **Global**).

ii. Change the parameters as needed.

[Pulse] tab: selection_angle=135[deg]

The parameter above can be changed to 45[deg], 90[deg], or 135[deg].

iii. The measurement is started by clicking the Submit Job button.

3.9 DATA SAVE LOCATION

a) Opening Past Data

i. Click the *iii* button in the [delta] console window.

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		File Opbons Ge 🔗 Fasorite Files		
		feats	T 4 - 4 - 6	
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Delta console window and file browser window

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- ii. The File Browser window appears.
- iii. Click the State button.
- iv. The selected data is opened by clicking the [button.

When the saving location for a certain data is changed, specify the location separately. The data once displayed on the screen is saved in the folder.

4. RELATED PROCEDURES

Instrument Usage/Service Application Procedure GP-I-001

5. REFERENCE CHECKING

REVISION	PREPARED BY	CHECKED BY	DATE	REMARK
1	Sugakumar A/L Varuthan Dara Fiona Mohamad	Fateh Ngaliman	31.07.2018	1 st issue