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| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 1 of 10 |

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.

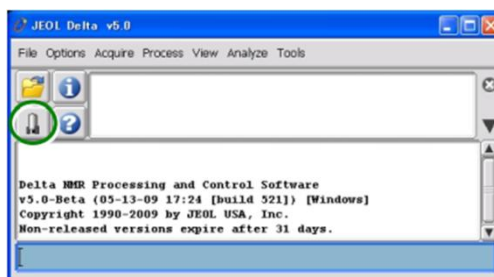
2. SCOPE

Applied for user from Chemistry Department.

3. INSTRUCTIONS PROCEDURE

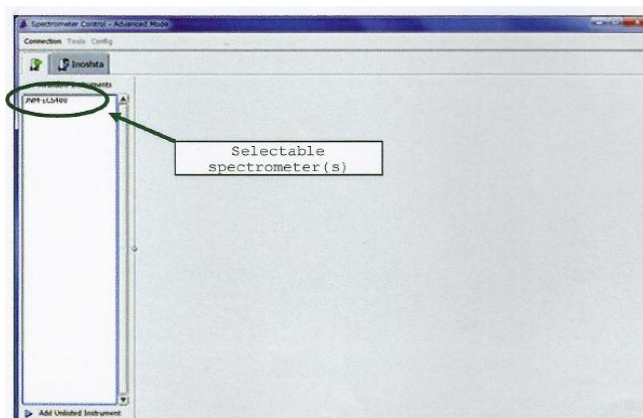
3.1 Delta STARTUP AND CONNECTION

- 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

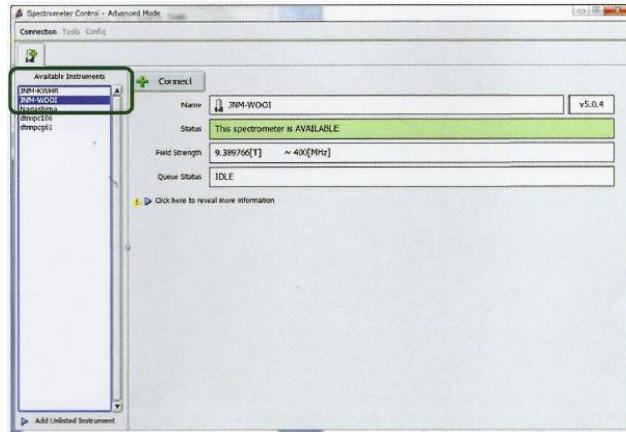
- e) Click the circled button  indicated in the figure below.
- f) The “Spectrometer control” window opens.



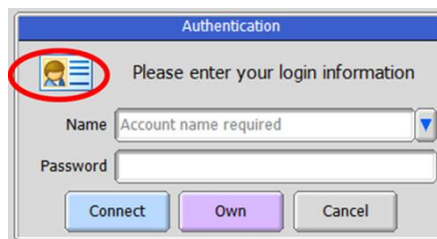
SPECTROMETER CONTROL WINDOW

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|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 2 of 10 |

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



h) Clicking the connection button  switches to the “User Authentication” window.



User authentication window

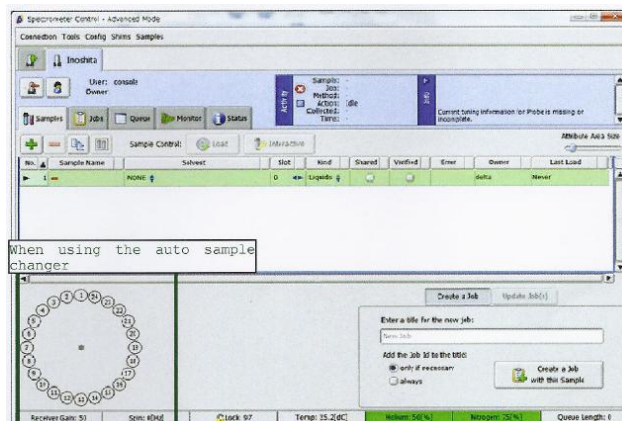
i) Type the user name and the password.

- Name delta
- Password: delta (displayed as asterisks (*****)

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

j) Click the  button.

k) The window view changes as shown below.

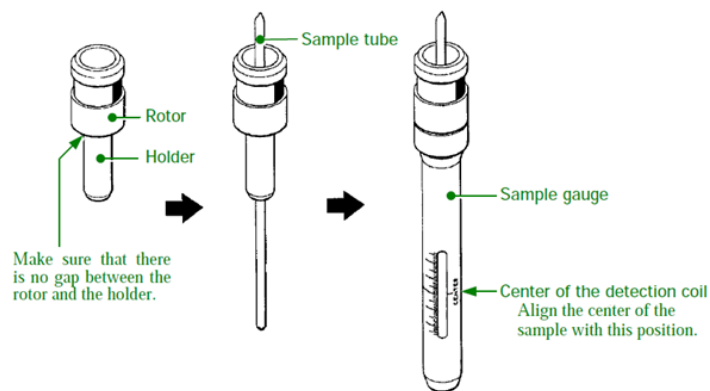


After completion of user authentication

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|-----------|---|-----------------|------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 3 of 10 |

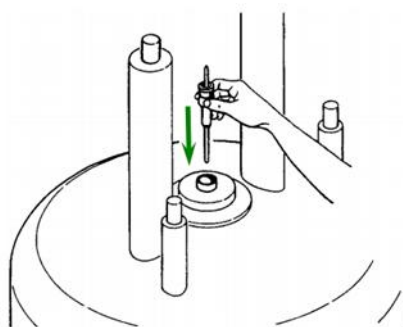
3.2 SAMPLE PREPARATION

- 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.


| | | | |
|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 4 of 10 |

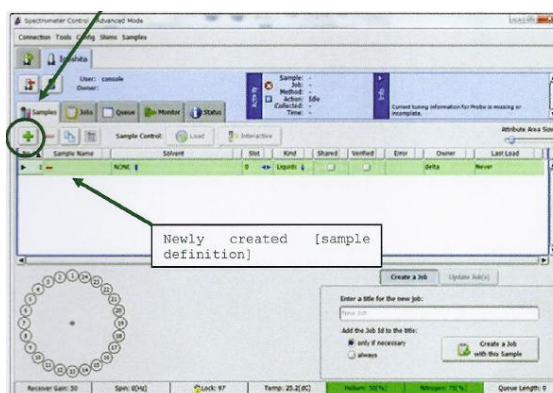
3.3 PREPARATION FOR MEASUREMENT

(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:

| No. | Sample Name | Solvent | Slot | Kind | Shared | Verified | Error | Owner | Last Load |
|-----|-------------|---------|------|---------|--------|----------|-------|-------|-----------|
| 1 | | NONE | 0 | Liquids | | | | delta | Never |

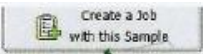
| Item | Description |
|---|---|
|  | Add a sample definition |
|  | Delete a sample definition |
| Sample name | Arbitrary (used as a part of a saved name) |
| Solvent | Select a solvent. Select a sample slot (if an auto sample changer is integrated). |
| Slot | Select a sample slot (if an auto sample changer is integrated). |

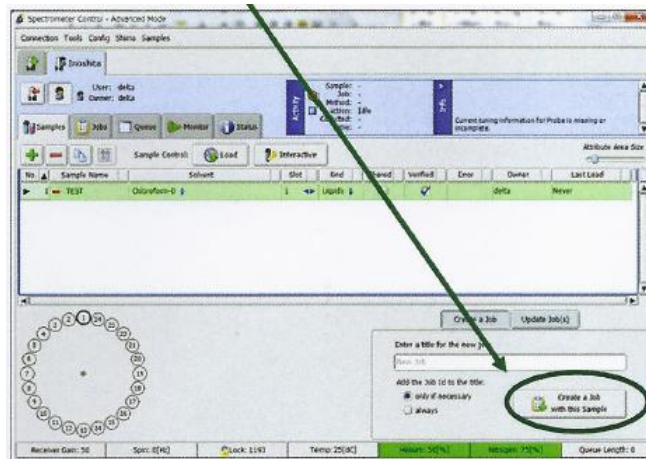
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|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 5 of 10 |

(b) Creating a Job

A job is:

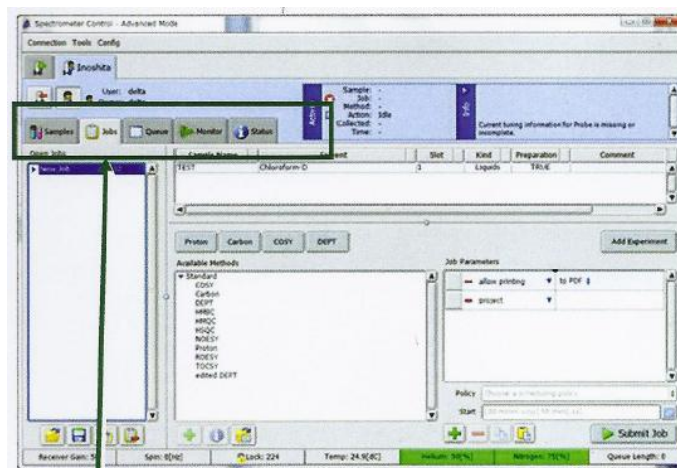
A process of configuring conditions for performing an NMR measurement.

1. Click the  button displayed in the Spectrometer Control Window.



SPECTROMETER CONTROL WINDOW

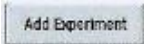
2. The window automatically switches to the Job tab.

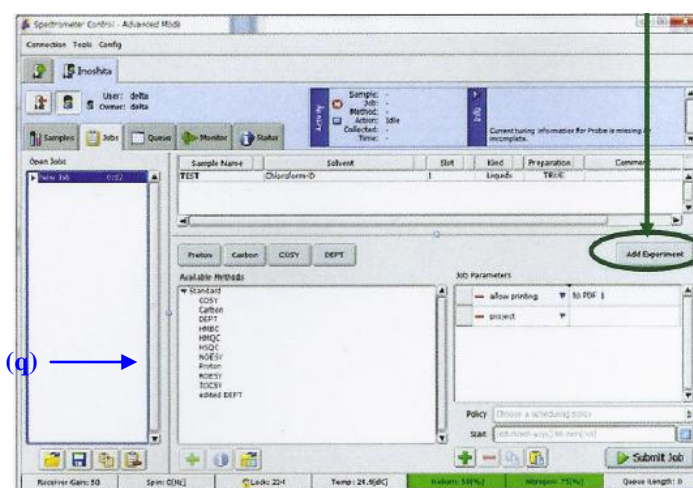


Jobs tab


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|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 6 of 10 |

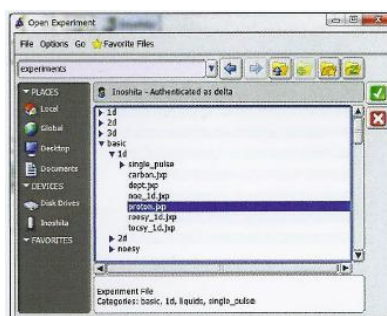
3.4 1H MEASUREMENT


a) Click the  button to select a pulse sequence.




Selecting a pulse sequence

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



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

d) The measurement is started by clicking the  button.

Note:

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

| | | | |
|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 7 of 10 |

described.


These are basic procedures for performing measurements using Delta V5.

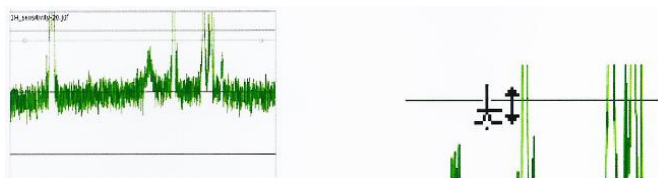
This procedure should be practiced in order to achieve a smooth measurement process.

3.5 ¹H DATA PROCESSING


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

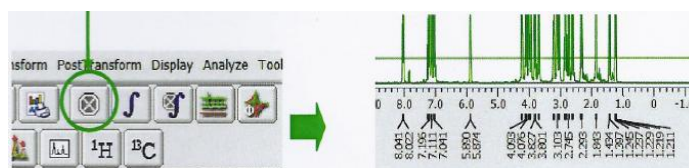
a) Peak Picking

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



i) Automatic detection


Peak picking is performed by clicking the  button.

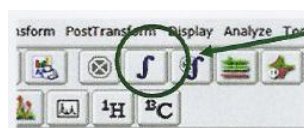


After peak picking


2. Integration

i. Automatic detection

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

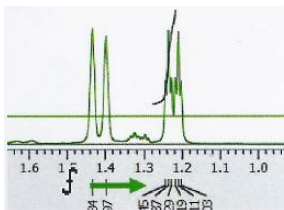


ii. Adding and deleting a selected integral curve

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

| | | | |
|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 8 of 10 |

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**.

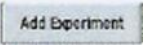


3.6 PRINTING DATA

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



3.7 ¹³C MEASUREMENT

a) Measurement

- i. Create a new job or add parameters to an existing job.
 - a. Click the  button to select basic / carbon.jxp.
 - b. (If not found, search by double clicking ).
- ii. Change the parameters as needed.
- iii. The measurements is started by clicking the  button.

b) Data processing

Perform the processing for the acquired spectrum.



Follow the same procedure as 1H.

| | | | |
|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 9 of 10 |


- 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  button to select basic / dept.jxp.
 - b. (If not found, search by double clicking ).
- ii. Change the parameters as needed.

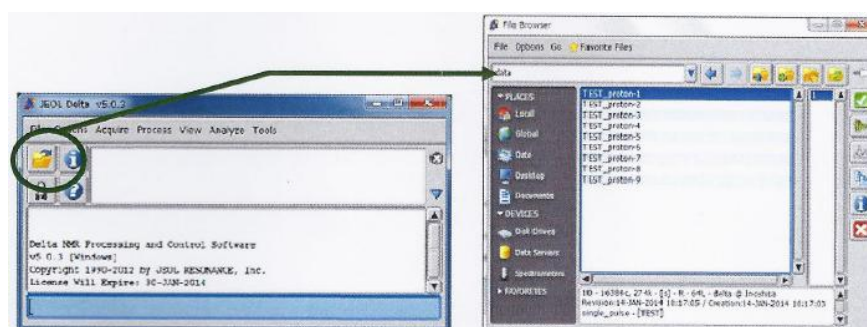
[Pulse] tab: selection_angle=135[deg]

The parameter above can be changed to 45[deg], 90[deg], or 135[deg].
- iii. The measurement is started by clicking the  button.

3.9 DATA SAVE LOCATION

a) Opening Past Data

- i. Click the  button in the [delta] console window.



Delta console window and file browser window

| | | | |
|-----------|---|------------------------|-------------------------|
| UM | CHEMISTRY DEPARTMENT | Category | Work Instruction |
| | PROSEDURE FOR EQUIPMENT HANDLING FT-NMR ECX 400MHz SYSTEM JOEL FOR SAMPLE ANALYSIS | Document No. | AK/JK.P/C/01-BI |
| | | Date Revision | 31.07.2018 1 |
| | | No. of pages: | 10 of 10 |

ii. The File Browser window appears.

iii. Click the  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 |