1. **TUJUAN**

   Tujuan prosedur ini ditubuhkan adalah untuk memberikan arahan yang jelas bagi langkah atau tatacara bagaimana menjalankan analisis sampel menggunakan peralatan JEOL NMR ECX 400MHz SYSTEM JOEL.

2. **SKOP**

   Terpakai untuk pengguna dari Jabatan Kimia.

3. **PROSEDUR ARAHAN**

   **3.1  Dari log masuk ke 'connection'.**

   Hidupkan bekalan sumber kuasa di stesen kerja.
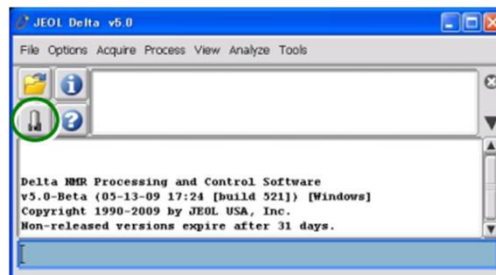
   a)  Taip Ctrl+Alt+Del di log masuk 'window'.

       Username: delta

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

   b)  Klik dua kali pada ikon Delta [ikon] setelah selesai log masuk.
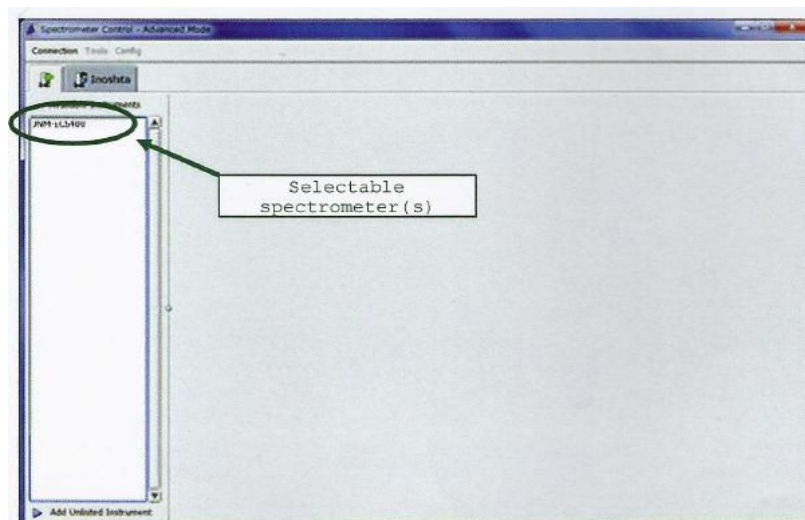
   c)  Delta 'Console window' akan terbuka.



**Delta console window**

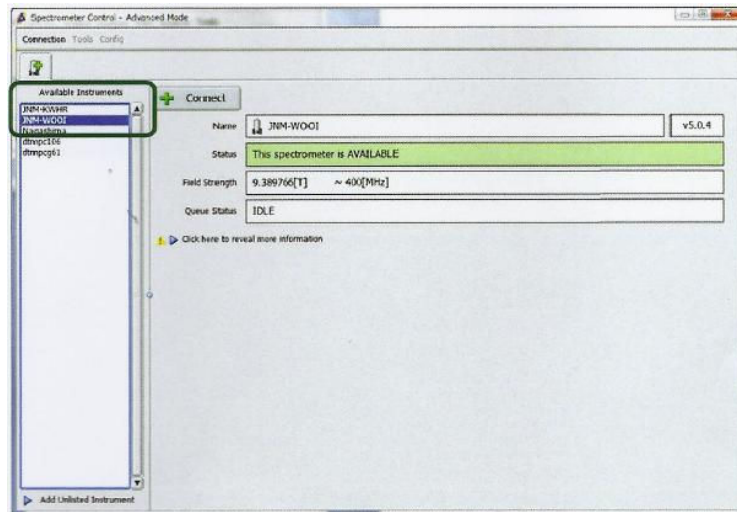   d)  Klik butang yang dibulatkan [ikon] seperti dalam rajah di atas.

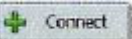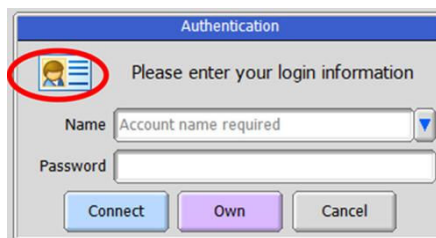   e)  'Window Spectrometer Control' akan terbuka.



**SPECTROMETER CONTROL WINDOW**

f) Pilih paparan 'spectrometer' pada 'Spectrometer control window'.



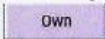g) Klik butang 'connection' [Connect] dan tukar kepada 'authentication window' pengguna.
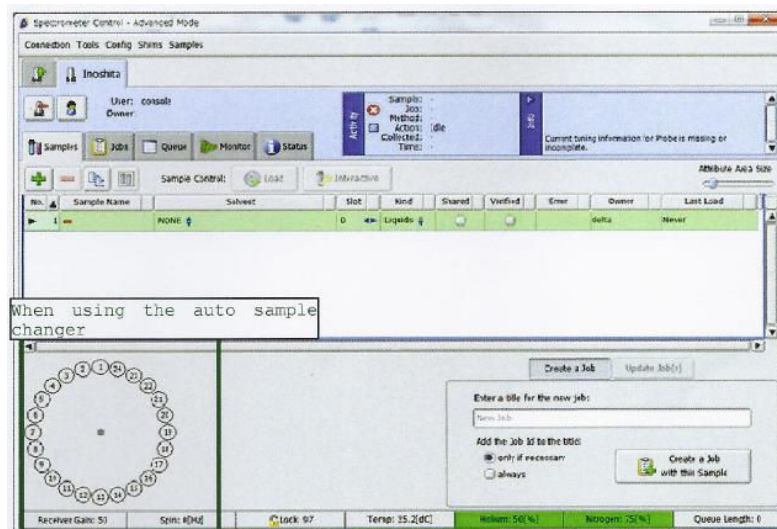


**User authentication window**

h) Taip nama pengguna dan kata kunci.

Nama pengguna: delta

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

i) Klik butang [Own]
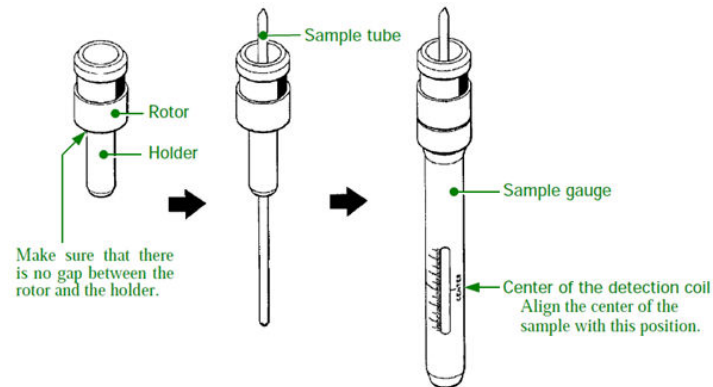
j) 'Window' paparan akan bertukar seperti di bawah.
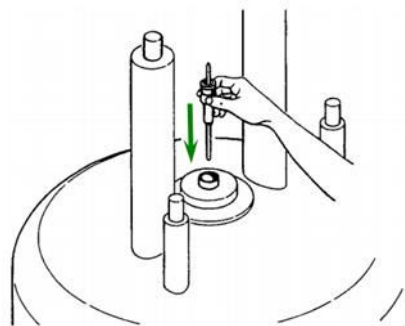


**After completion of user authentication**

### 3.2 Penyediaan sampel

a) Masukkan tiub sampel pada pemegang dan rotor sampel.



**Sample mounting**

b) Letak set tiub sampel pada pemegang dan rotor sampel supaya tiub boleh terapung di dalam SCM.

Ketika menggunakan 'auto-sample changer', sila masukkan tiub ke dalam slot.



**Setting the sample tube unit on the SCM**

**Notis penting:**

- Sebelum membuat sampel terapung di dalam SCM, pastikan udara terapung keluar.
- Pastikan tiada sampel di dalam SCM.
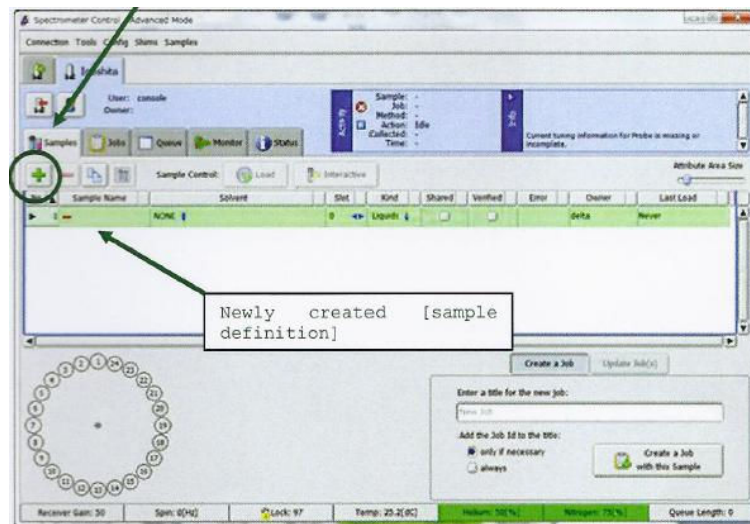- Dilarang memasukkan tiub kaca atau pemegang dan rotor yang kosong.

### 3.3  Persiapan untuk pengukuran

(a)  Cipta definisi sampel

Definisi sampel adalah:

Kondisi sampel yang hendak diukur.

1. Klik butang ![+] untuk mencipta definisi sampel yang baru. (lihat gambar rajah dibawah).



**Creating sample definitions**
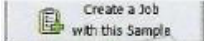
Butiran parameter terpapar di dalam definisi sampel.



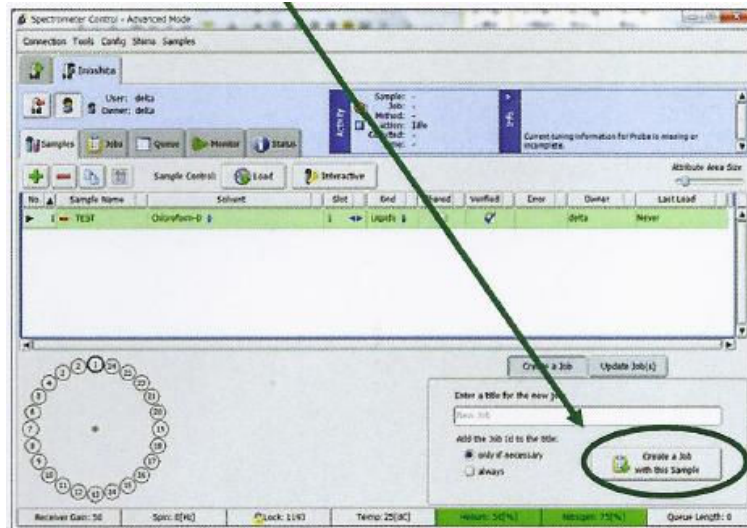| Item | Huraian |
|---|---|
| ![+] | Tambah definisi sampel |
| ![-] | Buang definisi sampel |
| **Nama sampel** | Arbitrari (digunakan sebagai nama yang akan disimpan) |
| **'Solvent'** | Pilih 'solvent'. Pilih slot sampel (jika pengubah sampel auto digunakan) |
| **Slot** | Pilih slot sampel. (jika pengubah sampel auto digunakan) |

(b) Cipta 'job'

'Job' adalah:

Satu proses mengenal pasti kondisi untuk menjalankan ukuran NMR.

1. Klik butang [Create a Job with this Sample] yang terpapar di 'Spectrometer control window'.



**SPECTROMETER CONTROL WINDOW**
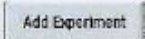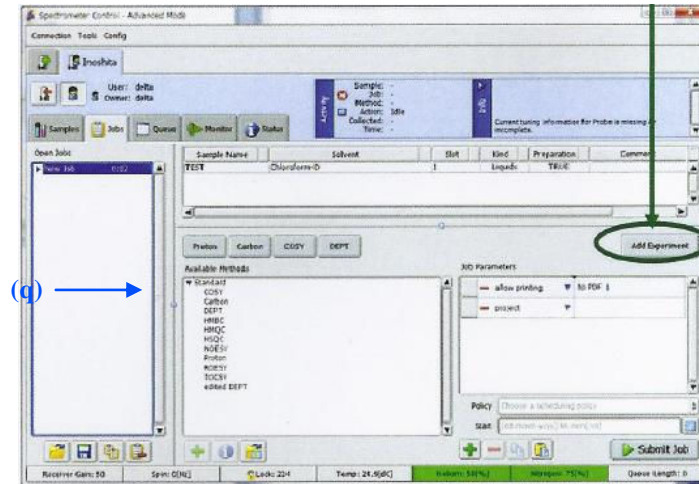
2. 'Window' akan bertukar kepada tab 'Job.



**Jobs tab**

### 3.4 Pengukuran 1H

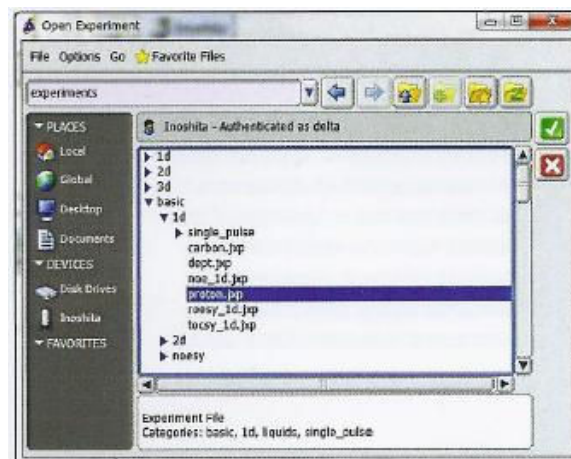a) Klik butang [Add Experiment] untuk memilih 'pulse sequence'.



**Selecting a pulse sequence**

b) Pilih 'basic/proton.jxp.' (sekiranya tidak dijumpai, cari dengan klik dua kali butang [Global]).



c) Pengukuran akan dimulakan dengan klik butang [Submit Job]

Nota:

Penyediaan sampel, penciptaan definisi sample, penciptaan kerja and pengukuran telah dijelaskan. Tersebut merupakan prosedur asas bagi pengukuran menggunakan Delta V5. Prosedur ini hendaklah digunakan untuk melancarkan proses pengukuran.
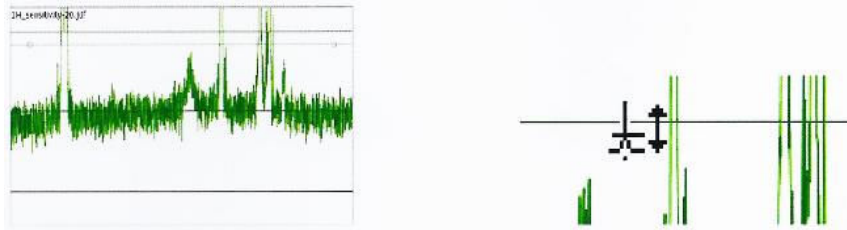
### 3.5  1H data processing

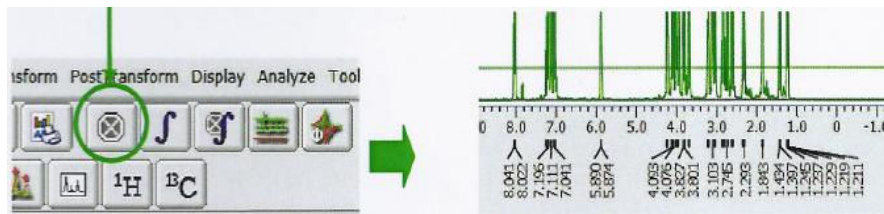Apabila pengukuran NMR telah selesai, keputusan pengukuran akan dipamerkan pada skrin.

a) Pemilihan 'peak'

1. Pilih butang ⊥ pada 'toolbar' untuk menentukan 'threshold'



i.     Pengesanan automatik
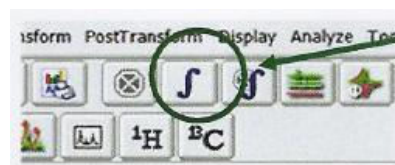
Pemilihan 'peak' dilakukan dengan klik butang ⊗



**After peak picking**

2. Integrasi

i.     Pengesanan automatik

1. Lengkung integral boleh didapati secara automatik dengan klik butang ∫
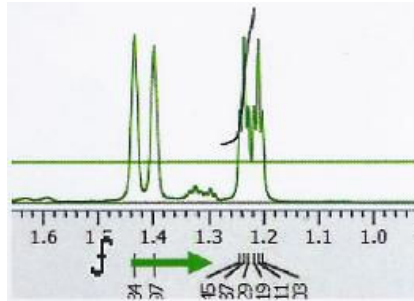
ii. Menambah dan memadam lengkung integral pilihan

    1. Untuk melukis lengkungan integral secara berasingan, pilih butang $\int$ pada 'toolbar'

    2. Lengkungan integral boleh dibuat dengan menarik bahagian pilihan pada paksi X.



**Dragging right and left**

iii. Menyimpan data yang telah diproses

    1. Pilih '**File**' dari 'pull-down' pada 'window' pemproses 1D.

    2. Data boleh disimpan dengan menggunakan **'Save as'**.

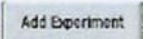### 3.6 Mencetak data

Klik butang 🖨 untuk membuka 'printing option window'.



### 3.7 Pengukuran 13C

a) Pengukuran

    i. Cipta 'job' baru atau tambah parameter pada 'job' yang telah tersedia.

        a. Klik butang [Add Experiment] untuk memilih 'basic/carbon.jxp.'

        b. Sekiranya tidak dijumpai, cari dengan klik dua kali butang [Global]

    ii. Tukar parameter sebagaimana yang dikehendaki

    iii. Pengukuran akan dimulakan dengan klik butang [▷ Submit Job]

b) Pemprosesan data

Lakukan pemprosesan bagi spektrum yang diperoleh.

Ikut prosedur yang sama sebagaimana 1H:

- Pembetulan fasa

- Seting rujukan

- Pemilihan 'peak'

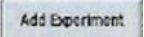### 3.8 Pengukuran DEPT

a) Pengukuran

   i. Cipta 'job' baru atau tambah parameter pada 'job' yang telah tersedia.

   a. Klik butang [Add Experiment] untuk memilih 'basic/dept.jxp.'

   b. Sekiranya tidak dijumpai, cari dengan klik dua kali butang [Global]

   ii. Tukar parameter sebagaimana yang dikehendaki.
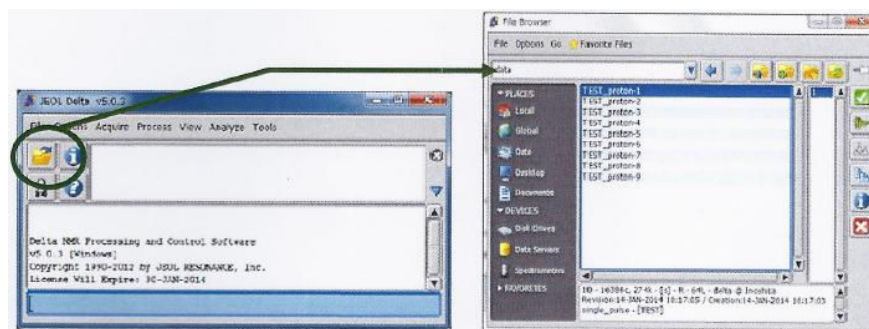
   [Pulse] tab: selection_angle=135[deg]

   Parameter diatas boleh diubah kepada 45[deg], 90[deg], atau 135[deg].

   iii. Pengukuran akan dimulakan dengan klik butang [Submit Job]

### 3.9 Lokasi simpan data

a) Membuka data terdahulu

   i. Klik butang 📂 pada 'console window' [delta].



**Delta console window and file browser window**

ii.    'Window' pelayar fail akan muncul.

iii.   Klik butang 

iv.    Data yang dipilih akan dibuka dengan klik butang 

Apabila lokasi menyimpan untuk data tertentu diubah, nyatakan lokasi berlainan. Data yang dipamerkan pada skrin akan disimpan di fail

## 4.   PROSEDUR BERKAITAN

Instrument Usage/Service Application Procedure GP-I-001

## 5.   RUJUKAN SEMAKAN

| SEMAKAN | DISEDIAKAN | DISEMAK | TARIKH | CATATAN |
|---|---|---|---|---|
| 1 | Dara Fiona Mohamad<br><br>Sugakumar A/L Varuthan | Fateh Ngaliman | 27.07.2018 | Isu kali pertama |

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.
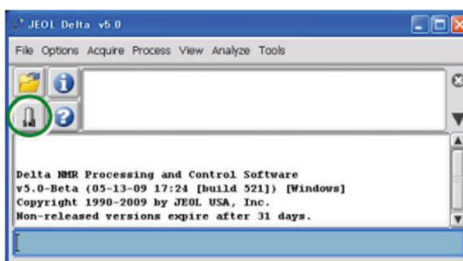
2. **SCOPE**

   Applied for user from Chemistry Department.

3. **INSTRUCTIONS PROCEDURE**

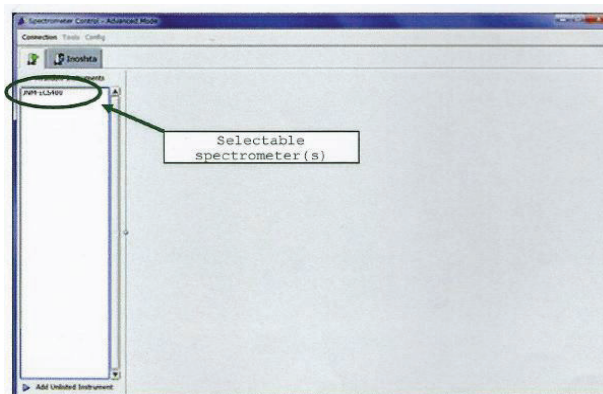   **3.1 Delta STARTUP AND CONNECTION**

   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 [icon] on the desktop.

   d) The Delta console window opens.



**Delta console window**
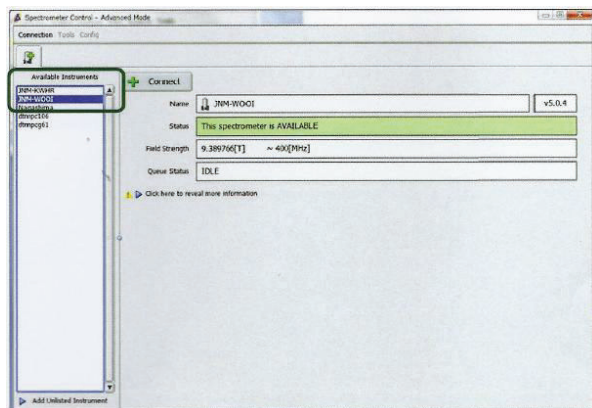
   e) Click the circled button [icon] indicated in the figure below.

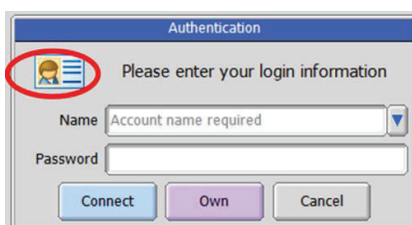   f) The "Spectrometer control" window opens.



**SPECTROMETER CONTROL WINDOW**

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



h) Clicking the connection button [Connect] switches to the " User Authentication" window.
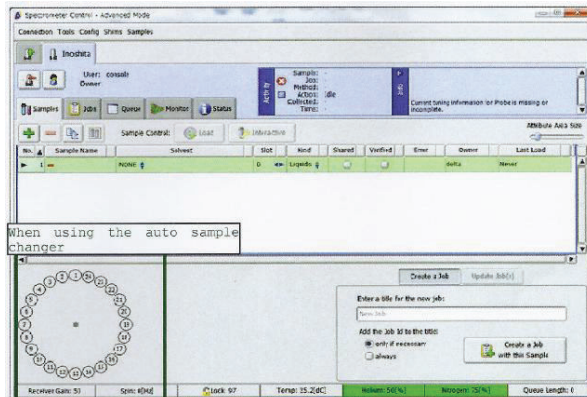


**User authentication window**

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 [Own] button.
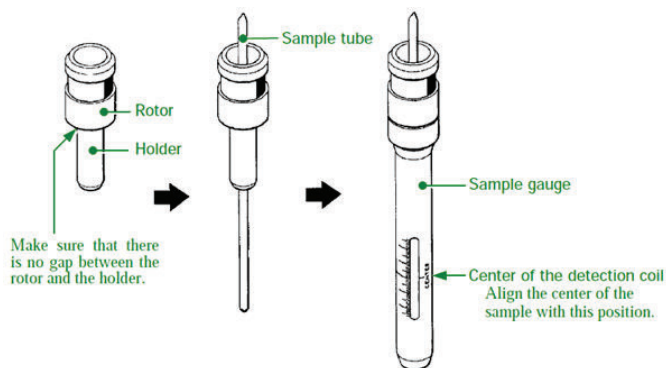
k) The window view changes as shown below.
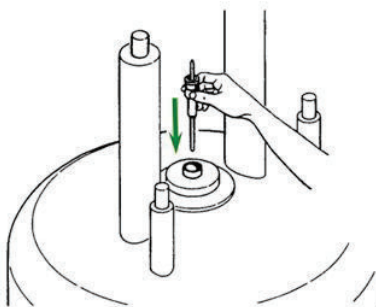


**After completion of user authentication**

### 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.**
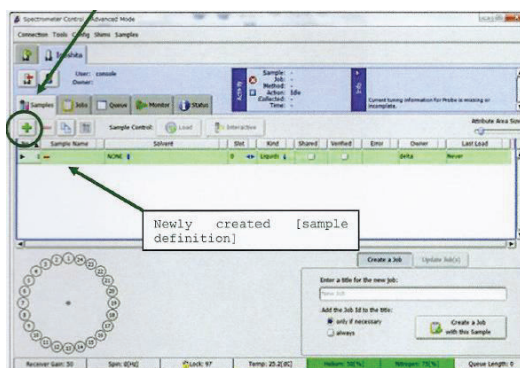- **Do not insert a bare glass tube or the empty rotor and holder.**

## 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:



| Item | Description |
|---|---|
| ➕ | Add a sample definition |
| ➖ | Delete a sample definiton |
| **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 ). |

(b) Creating a Job

**A job is:**

**A process of configuring conditions for performing an NMR measurement.**

1. Click the [Create a Job with this Sample] button displayed in the Spectrometer Control Window.
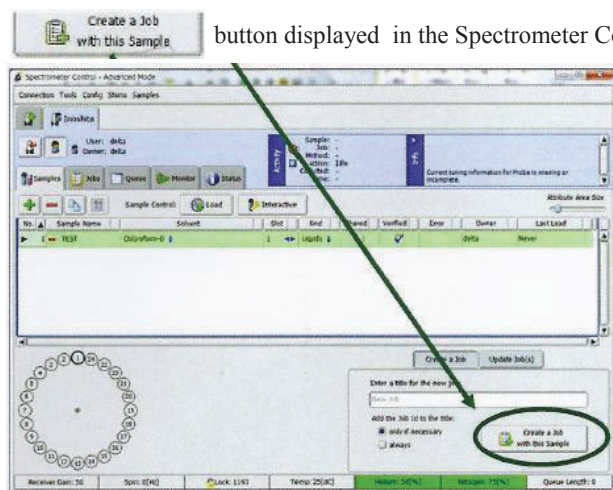


**SPECTROMETER CONTROL WINDOW**
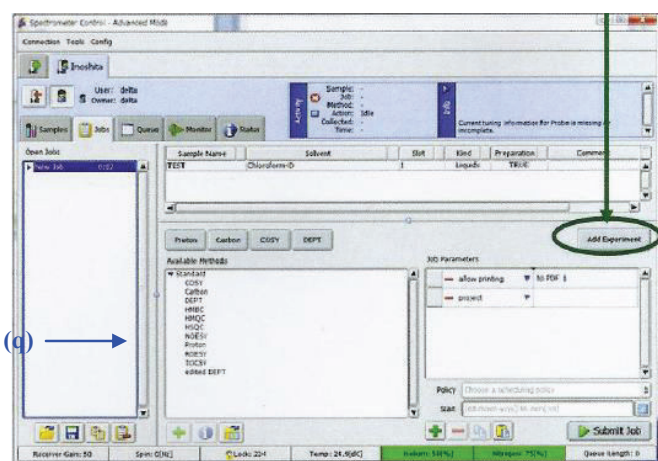
2. The window automatically switches to the Job tab.
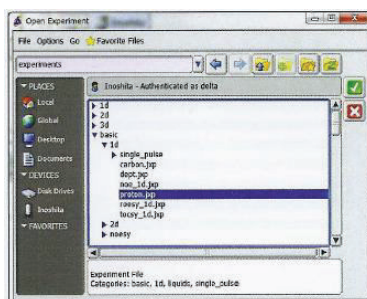


**Jobs tab**

### 3.4  1H MEASUREMENT

a)  Click the [Add Experiment] button to select a pulse sequence.



**Selecting a pulse sequence**

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



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

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

*Note:*

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

**described.**

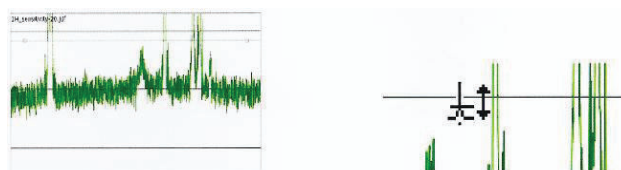**These are basic procedures for performing measurements using Delta V5.**

**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.
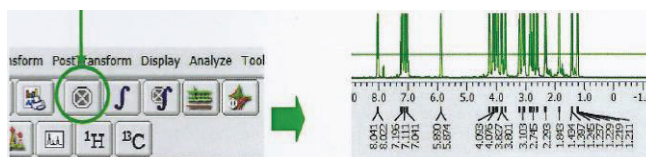
a) **Peak Picking**

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**

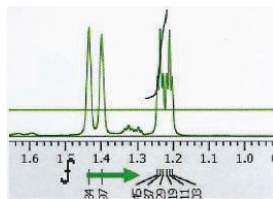1. An integral curve can be automatically detected  by clicking the ∫ button.



ii. **Adding and deleting a selected integral curve**

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

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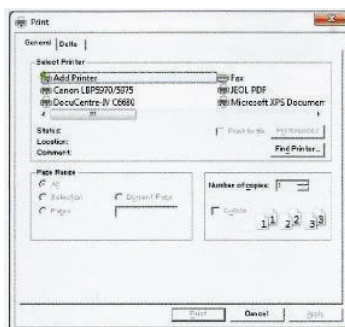


**Dragging right and left**

### iii.    Saving processed data

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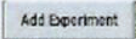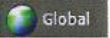2. The data can be saved by using **Save as.**

## 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 [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**
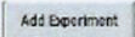
Perform the processing for the acquired spectrum.

**Follow the same procedure as 1H.**

- **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 Experiment   button to select basic / dept.jxp.

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

    ii.    Change the parameters  as needed.

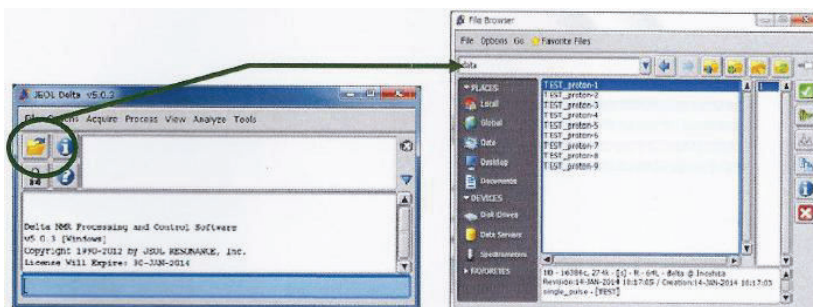            [Pulse] tab: selection_angle=135[deg]

            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    button in the  [delta] console window.



**Delta console window and file browser window**

ii.   The File Browser window appears.

iii.  Click the   Data   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   Data   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<br><br>Dara Fiona Mohamad | Fateh Ngaliman | 31.07.2018 | 1st issue |