JABATAN KIMIA	Kategori	Arahan Kerja
PROSEDUR PENGENDALIAN	Dokumen No.	AK/JK.P/C/02
PERALATAN FT-NMR ECX 400MHz System IOEL BAGI ANALISIS	Tarikh   Semakan	27.07.2018   1
SAMPEL	Mukasurat:	1 daripada 10

# 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 setelah selesai log masuk.
- c) Delta 'Console window' akan terbuka.

💕 JEOL Delta v5.0	
File Options Acquire Process View Analyze Tools	
20	Ø
	V
	A
Delta NMR Processing and Control Software v5.0-Beta (05-13-09 17:24 [build 521]) [Windows]	
Copyright 1990-2009 by JEOL USA, Inc. Non-released versions expire after 31 days.	V.
1	

# Delta console window

- d) Klik butang yang dibulatkan 🔒 seperti dalam rajah di atas.
- e) 'Window Spectrometer Control' akan terbuka.



## SPECTROMETER CONTROL WINDOW

	JABATAN KIMIA	Kategori	Arahan Kerja
TINA	PROSEDUR PENGENDALIAN	Dokumen No.	AK/JK.P/C/02
UIVI	PERALATAN FT-NMR ECX 400MHz System loel bagi analisis	Tarikh   Semakan	27.07.2018   1
	SAMPEL	Mukasurat:	2 daripada 10

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

pectrometer Control -	Advanced Mode		0.8
nection Tools Confid	And the second second		
	-		
Available Instruments	Cornect		
-wool	Name	A JNM-WOOI	v5.0.4
pc106 pcg61	Status	This spectrometer is AVAILABLE	
	Field Strength	9.389766[T] ~ 400[MHz]	
	Queur Status	IDLE	
	C management		
	1 D Click here to rev	eal more information	
	1		

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

		Authenticatio	n
	Please	enter your	login information
Name	Account	name require	d 🔻
Password			
Con	nect	Own	Cancel

User authentication window

h) Taip nama pengguna dan kata kunci.

Nama pengguna: delta

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

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

Connection Tools Config Shims Samples		-	-						
Inoshita									
Dier: cossie Domer	Status	Sample: Jos: Method: Action: I Collected: Tarve:	idle		Current to	ning informa	son for Pio	be is missing o	. (
4 - 1 In Sampe Contral: O Load	2 Interactive	1						Athibi	74 AJQ3 SIX
No. A Sample Name Solvest	Slot	Nind	Stared	Verfied	Erner	Ont	er   {	Last Load	T
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hen using the auto sam hanger	ple				-				
hen using the auto sam hanger 1 (1 <sup>332</sup> )	ple			[	Create a	Jab	lipdate Ist	65	
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hen using the auto sam nanger J (0 <sup>000</sup> ) (0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	ple			Ster a tile for	Draste a the new je	Jak b:	lipet.ite Job	46	
hen using the auto sam hanger	ple			lister a tille for tern Job Kidd file Job Jä Sonhr if re Jahnays	Droste a the new je to the bitici cessary	Job b:	lipitate Job	(1) Creats a Jub	

After completion of user authentication

# 3.2 Penyediaan sampel

a) Masukkan tiub sampel pada pemegang dan rotor sampel.



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.

+	Sample Control:	is is	deractiv						Attribute Area	5
No. 🛦 Sample Name	Solvent	TI	Slot	Kind	Shared	Verified	Error	Owner	Last Load	Ĩ
► 1=	NONE \$	1	0 -	Liquids \$	0	0		delta	Never	Ĩ

Item	Huraian
4	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)

JABATAN KIMIA	Kategori	Arahan Kerja
PROSEDUR PENGENDALIAN	Dokumen No.	AK/JK.P/C/02
PERALATAN FT-NMR ECX 400MHz SYSTEM JOEL BAGI ANALISIS	Tarikh   Semakan	27.07.2018   1
SAMPEL	Mukasurat:	5 daripada 10

(b) Cipta 'job'

'Job' adalah:

Satu proses mengenal pasti kondisi untuk menjalankan ukuran NMR.

Spectrometer Control - Advanced Mode			[oite	
Connection Tools Canilig Shana Samples	1			25.1
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+ - 1 IT Sample Control ( Load	20 Interactive		Attribute Area	a Size
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th   a) Sample Norma   Solvert ► I == TEST ObserfameD \$	I die   dee	Core a 20 Core a 20 Deter a tille for the new ye	Diver Lattend ddfa Nover 6 Update Sob(s)	

SPECTROMETER CONTROL WINDOW

2. 'Window' akan bertukar kepada tab 'Job.

Spectrometer Control - Advanced	Mode	LGR/BR MR
annection Tools Canfig	in the second statement of the	
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- Uner deta	Sample -	•
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	edited DEPT	
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38150	+ 0 2	🔶 — 🔥 🚯 🕨 Submit Job
second design of the second		

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



the second state of the se	
V 🗇 🔿	
Inoshita - Authenticated as delta          1d         2d         3d         visit         vingle_pulse         carbon.pp         dopt.pp         nos_id.pp         pattors.pp         torsy_id.pp         torsy_id.pp         b nossy	
	Thoshika - Authrebicated as delta  Id  Id  Id  Id  Id  Id  Id  Id  Id  I

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'

94,5e840628.07		
paper language and the land land	<mark>_‡‡</mark>	
	<b>1</b>	

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 🧊





JABATAN KIMIA	Kategori	Arahan Kerja
PROSEDUR PENGENDALIAN	Dokumen No.	AK/JK.P/C/02
PERALATAN FT-NMR ECX 400MHz System IOEL BAGI ANALISIS	Tarikh   Semakan	27.07.2018   1
SAMPEL	Mukasurat:	8 daripada 10

- ii. Menambah dan memadam lengkung integral pilihan
  - 1. Untuk melukis lengkungan integral secara berasingan, pilih butang 👔 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'.

Canon LBP5970/5975	Microsoft XPS Documen
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<b>race Rente</b> G. All C. Selector C. Dynami Piete C. Piger	Number of gaples:
	1 1 2 2 3 3

# 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

Submit Job

- ii. Tukar parameter sebagaimana yang dikehendaki
- iii. Pengukuran akan dimulakan dengan klik butang

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].

		& File Browser		
		File Options Go 🔗 Favorite Files		
		- Cata	V 4 3	
D JEOL Delts V5.0.2		* FLACES	TEST_proton-1 TEST_proton-2 TEST_proton-3 TEST_proton-4	
	0	Date Ozsitep	TIST_protons TIST_protons TIST_protons TIST_protons TIST_protons	
Delia NSE Frommaning and Control Software		Decements     OFVEES     Oak Drives     Dats Services		
<pre>05.0.3 [kthole] Copyright 1990-2012 by JSUL RENEWAYLE, Inc. License Vill Expire: 30-308-2014</pre>	7	Speakamenna + Revoluties	<ul> <li>●).</li> <li>10 - 16384c, 274k - 53) - R - 64t, - 84ta ⊕ In Revuedo 14-3M-2514 (1:1745 / Creation 14)</li> </ul>	oshta -301-2014 (41,7:03

Delta console window and file browser window

JABATAN KIMIA	Kategori	Arahan Kerja
PROSEDUR PENGENDALIAN	Dokumen No.	AK/JK.P/C/02
PERALATAN FT-NMR ECX 400MHz System IOEL BAGI ANALISIS	Tarikh   Semakan	27.07.2018   1
SAMPEL	Mukasurat:	10 daripada 10

- ii. 'Window' pelayar fail akan muncul.
- iii. Klik butang 😂 Data
- 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 Sugakumar A/L Varuthan	Fateh Ngaliman	27.07.2018	Isu kali pertama

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

- 🗆 🖬
O
4
-

# Delta console window

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

Controller Control - Advanced		A DESCRIPTION OF THE OWNER OF THE
strection Tanks Carily		Constanting of the second second
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M-1C5400		
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SPECTROMETER CONTROL WINDOW

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

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

Spectrometer Control - Advor	soed Mode		(a)@
meeten Tools Config			
2			
Available Instruments	Cornect		
-wooi	Name	A JIM-WOOT	v5.0.4
pc106 pcg61	Status	This spectrometer is AVAILABLE	
	Field Strength	9.389766[T] ~ 400[MHz]	
	Queue Status	IDLE	
	A Do Circle have to ma	al more information	
11	L P COUNT O RD		
11			
- 11			
- 11			
11			
11			
11			
U			
T			

h) Clicking the connection button 🕴 connect switches to the "User Authentication" window.

	Authentication
	Please enter your login information
Name	Account name required
Password	
Cor	mect 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.

Spectrometer Control	Advenced Mode	and the second second	Contraction of the local division of the loc						100	
Connection Tools Confi	Shins Samples		1		-	1	-			
1 Inoshita										
Tanpirs Com	cossale	In Monitor	Active P	Sample: Jos: Method: Actor: Collected: Time:	idie	i	Current to	uning information	for Probe is making or	Ĺ
	Sample Con	ant: @ 1994	1 Interactive	1		7.1			ANDUS	e Area Size
No. a Sample Name	11	Selvest	slot	Ned	Shared	Verfied	Erner	Owner	Lest Load	T
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After completion of user authentication

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

### **3.2 SAMPLE PREPARATION**

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



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.

	CHEMISTRY DEPARTMENT	Category	Work Instruction
TINA	PROSEDURE FOR EOUIPMENT	Document No.	AK/JK.P/C/01-BI
UIVI	HANDLING FT-NMR ECX 400MHz	Date   Revision	31.07.2018   1
	SYSTEM JOEL FOR SAMPLE ANALYSIS	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:

+ - 2 10	Sample Control: 🚱 Load	inte 🕄	teractive						Attribute Area S
No. A Sample Name	Solvent		Slot	Kind	Shared	Verified	Error	Owner	Last Load
► 1 =	NONE \$	0	41	Liquids 🖕	0	0		delta	Never
Item					Des	cription	l		
4				Ado	l a sam	nple def	inition		
-				Dele	ete a sa	mple de	efinitor	1	
Sample na	ame	А	rbitra	ary ( us	sed as a	a part of	f a save	ed name )	)
Solven	t Select	a solve	ent. S	elect a	sample	e slot (	if an aı	ito samp	le changer is
					integ	grated)	-		
Slot	Sele	ect a sar	mple	slot ( i	f an au	to samp	le chai	nger is in	tegrated ).

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

### (b) Creating a Job

# A job is:

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



SPECTROMETER CONTROL WINDOW

2. The window automatically switches to the Job tab.



Jobs tab

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

### **3.4 1H MEASUREMENT**

a) Click the Add Experiment button





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

	CHEMISTRY DEPARTMENT	Category	Work Instruction
	PROSEDURE FOR EQUIPMENT	Document No.	AK/JK.P/C/01-BI
UIVI	HANDLING FT-NMR ECX 400MHz	Date   Revision	31.07.2018   1
	SYSTEM JOEL FOR SAMPLE ANALYSIS	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 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 **S** button.

sform	Pos	tTrans		splay	Analy	ze To
		⊗(	S	5	=	*
12	E.	1 <sub>H</sub>	<sup>B</sup> C			

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

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

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

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States Location: Comment	Fing Printer-
Pask Rado G. Ko G. Scholgon C. Dogram ( Page C. Pages	Thumber of gapters 1 = = = = = = = = = = = = = = = = = =

#### 3.7 13C MEASUREMENT

#### a) Measurement

- i. Create a new job or add parameters to an existing job.
  a. Click the Add Dependent 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.

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 Add Decomment 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.

		🗿 Fila Browser	19/8	-12
		File Options Ge 👷 Favorite Files		
		data	y 4 = 🙀 😹 🥌 🧉	-
A JEOL Delte v5.0.2 Di Gron Acquire Process View Analyze Teols		* PLACES	Alterna A	2
	0	Date TEST Date TEST Desities TEST	prezon-s pre	14 18
Delts 1060 Frocussing and Control Software of 0.1 [Windows] Copyright 1940-2012 by JSDL RENAMACE, Inc.		OFVICES     Dak Daves     Dak Serves     Spectrometry		2
License Vill Expire: 30-338-2014		+ FRAMERIES 10 - 10 Revisio single	3984, 2743 - [4] - R - 641, - 8453 @ Incentra 6:14-304-2014 [8:17:05 / Creation:14-304-2014 [8:17:03 pulse - [FEST]	

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 Seats 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 specific 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 <sup>st</sup> issue