ECA 400 FT-NMR SYSTEM

OPERATING PROCEDURE (FOR USER)

1. On the monitor, you will see 3 windows

- a) Delta Console (Fig. 1.0)
- b) Sample: scc (Sample Tool) (Fig. 2.0 at page 2)
- c) Spectrometer control (Fig. 3.0 at page 4)

Ø⊈–# [aabe] Delta	• ×
File Processors Viewers Tools Analysis Acquisition	- Menu bar
	Tool bar
	JEUL USH
Delta NMR Processing and Control Software Copyright 1990-2003 by JEOL USA, Inc. Version: 4.3-Beta (10-16-03 21:55) [Linux] 21 CFR part 11 technical features are enabled	View window



- 2. Introducing Sample To The System
 - i. Go to Sample Tool Window (Fig 2.0, Sample scc)

핥-녀 (aabe) Sample: scc2.nm.	jeol.co.jp	• X		
Options				
Field Strength	Helium Nitrogen			
11.7473579[T]	87[%] 92[%]			
Sample State	Spinner	Temperature		
Probe ID 2692 Slot	Image: Wight of the second s			
Solvent CHLOROFORMED CYCLOHEXANE-D12 D2O DMF-D7 DMSO-D6 METHANOL-D3 CHLOROFORM-D	I Gain 22 Level 180 Phase 202.4[Offset 7.26[p	cock Control		
User Shims System	Shims	Refresh Shims		
Shim Groups Z1 Z2 Z3 Z4	t 592	Anto Shims		
SHIM_Z1 \$ SHIM -165[Hz] 7 •5x •10x •50x -5x -10x •50x	1_Z2 ♦ SHIM_Z3 .77[Hz] -192.5[H •10x +50x -5x -10x -10x -50x -5x -10x	\$ SHIM_Z4 [z] 59.03[Hz] •50x •5x •10x •50x •50x •5x •10x •50x		

Fig 2.0 Sample Tool Window

ii. Enter slot number in Slot box in Sample State (Refer Fig 2.1)



Fig. 2.1: Sample State Icon

iii. After the sample has been loaded, click on to turn on the spinner (Refer to Fig. 2.2)



Fig 2.2: Spinner Icon

iv. Select a solvent, from the list

CHLOROFORM-D	Ĭ.A
CYCLOHEXANE-D12	
D2O	
DMF-D7	
DMSO-D6	- 8
METHANOL-D3	



v. Click on Shim and lock icon (Refer Fig. 2.4)

	Lock (Contr	ol		
Gain	23		8	ď	
Level	180			1	-
Phase	202.5[deg]	D	LOCK	DN	
Offset] 7.26[ppm]	D	IDLE		

B	Gradient Shim&Lock	Automatically locking is performed after performing the gradient shimming.
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Fig 2.4: Lock Control

vi. Please wait for state of NMR lock and state of shim turn green (Refer Fig. 2.5)

Fig. 2.5: Status of NMR Lock and Shim

3. Running The Experiments

i. Go to Spectrometer Control Window (3.0)



Fig. 3.0: Spectrometer Control Window



Fig. 3.1: Spectrometer Control and its function

ii. Click on Expmnt (Refer to Fig. : 3.2)

<u>a</u>	Pri	o Slo	t Jol	o Submit I	'ime ←Ó
		1			
Sample	Expmnt	Auto	Sawth	View	Copy

Fig. 3.2: Button at Spectrometer Control

🏂 🛱 [aabe] Ope	n Experiment			• ×
Path: /usr/	delta/global/exp	periments		
Format:		† Filt	er: [*.ex2]	
Directo	ry	Filenar	ne	Version
- Favorites - Id Id_cosy Id_inadequate	Apple ap	Lex2 ry.ex2 ry.pfg.ex2 ference_noe_1 [_cosy.ex2 [_cosy.pfg.ex4 f_cosy_pfg.ex4 f_cosy_phase. tcor.ex2	ld.ex2 2 ex2	
Ok	Info	Delete	Refresh	Cancel

iii. Open Experiment Window is displayed as below (Refer Fig 3.3)

Fig. 3.3: Open Experiment Window

- *iv.* Select the experiment from the filename column (Fig. 3.3). *For any 1D & 2D measurement, e.g. 13C, Noe-diff etc., please refer to Measurement User's Manual in Chapter 4 & 5. Here, we select single pulse experiment.*
- v. Click Ok
- vi. Experiment Tool window is displayed as below (Refer Fig. 3.4)

🖉-¤ [aabe] Experim	ent Tool: single_pulse.ex2	• 🗆 🗙
File Tools View	Options	
		ubmit
Get Acq. View:	X Y Z A B C D E	
Header Inst	trument Acquisition Pulse	
filename	single_pulse Bro	wse
sample_id	[
comment	single_pulse]	
process	active_global 'std_proton_autophase.list'	lit 📃 🚍
auto_filter	Ø	
auto_gain	0	11721
filter_limit	8	
force_tune	0	Ī
scc2.nm.jeol.co.jp	Total Collection Time: 00:01:03	

Fig. 3.4: Experiment Tool Window

- vii. At Header Section (Fig. 3.4), enter
 - a) File name
 - b) Sample id
 - c) Tick in auto_gain box
 - d) Tick in force_tune box
- viii. Go to Instrument section and check the solvent, refer Fig. 3.5

ø∕r⊮ (aabe) Experin	nent Tool: single_pulse.ex2	• 0 ×
File Tools View	Options	
	Add 🔛 🔡 🛃	Submit
Get Acq. View:	XYZABCDE	
Header	strument Acquisition Pulse	
solvent	CHLOROFORM-D CYCLOHEXANE-D12 D2O DMF-D7 DMSO-D6	
recvr_gain	50	
scc2.nm.jeol.co.jp	Total Collection Time: 00:01:03	

Fig. 3.5: Instrument Section

ix. Go to Acquisition section as shown at Fig. : 3.6You may change parameter that needed for your experiment in the Acquisition section

OPERATING PROCEDURE | ECA 400 NMR SYSTEM

🖉⊣¤ (aabe) Experim	ent Tool: single_pulse.ex2
File Tools View	Options
	Add 😰 Submit
Get Acq. View:	X Y Z A B C D E
Header Ins	trument Acquisition Pulse
x_domain	Proton
x_offset	5[ppm]
x_sweep	15[ppm]
x_points	16384
scans	
x_prescans	
mod_return	
x_acq_time	2.18383[s]
scc2.nm.jeol.co.jp	Total Collection Time: 00:01:03

Fig. 3.6: Acquisition Section

- x. Go to Pulse section, see Fig. 3.7
- xi. You may change parameter that needed in the Pulse section

🏂-∺ (aabe) Experime	ent Tool: single_pulse.ex2
File Tools View	Options
	Submit
Get Acq. View:	YZABCDE
Header Inst	rument Acquisition Pulse
x_angle	45[deg]
x_90_width	17[us] x90]
x_atn	3.5[dB]
x_pulse	8.5[us]
relaxation_delay	5[s]
repetition_time	7.18383[s]
dante_presat	0
presat_time	5[s] relaxation_delay
scc2.nm.jeol.co.jp	Total Collection Time: 00:01:03

Fig. 3.7: Pulse Section

xii. After completing key in all needed parameter, then click "Submit" button, refer Fig. 3.8

år-l≓ (aabe) Exper	iment Tool: single_pulse.ex2	• D X
File Tools Vie	v Options	
Get Acq. View:	X Y Z A B C D	E Submit
x_angle	45[deg]	
x_90_width	17[us] x90]	Ĩ.

Fig. 3.8 Experiment Tool Window (Submit Button Highlighted)

xiii. "Inform" window will be displayed (Fig. 3.9) then click GO button



Fig. 3.9: Inform Window

xiv. On the "spectrometer control" window will show the status of the experiment (Fig. 3.10)

∦dabe]	Spectrometer Contro	1	
Tools Cor	dig Queue Machine	e Options	
Info	Connect Moni	itor Unlink	Frae
	Connect : scc2	.nm.jeoLco.jp	
Queue State : OWNED Selected Job : UNKNOWN			
00_039	Atsushi_Abe S#493561 single_pulse 17-DEC-2003	32 13:42:56	2
<u>oo</u>]	Prio Slot	Job Submit T	ime +ť
Sample	Expmnt Auto	Sawth View	Copy

Fig. 3.10: Spectrometer control shows measuring in progress



4.0 Ejecting the sample

- i. Go to Sample State box
- ii. Enter "0" in the slot column (Fig. 4.0)



Fig. 4.0: Sample state box

iii. Press advance button at ASC (automatic sample changer) until you can take your nmr tube safely.