Operational Procedures of Varian NMR

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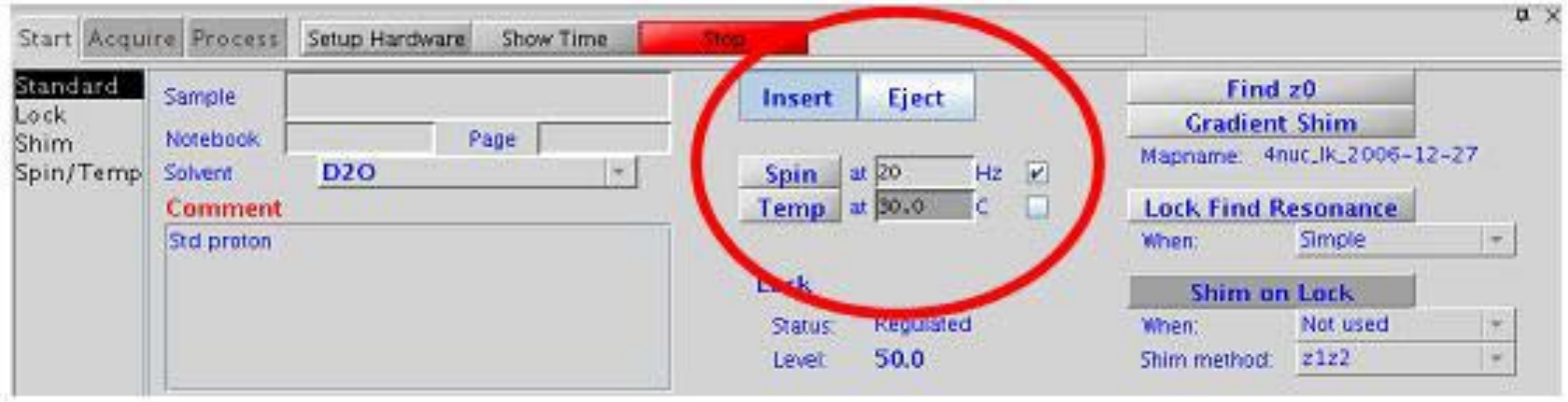
For all experiments

- Sample preparation:
  - For ^{1}H spectrum: 3 ~ 10 mg of sample;
  - For ^{13}C spectrum: 20 ~ 50 mg of sample;
  - For 2D spectrum: the more the better, typically 50 mg.
- Solvent: ~ 0.7 ml

For all experiments

- Insert sample (input “i” in command line or press “Insert” button);
- Write down sample information;

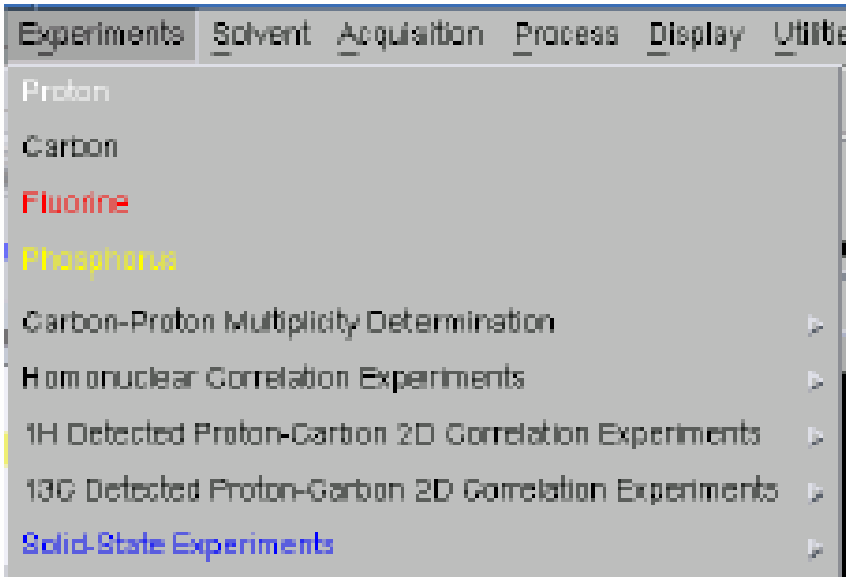


For all experiments

- Find Z0: Lock power and lock gain can be increased if the lock status is “Not regulated” (It may occur when using $CDCl_3$ as solvent, or high concentration).
- Gradient shim.
- Spin control: Only ^{1}H spectrum needs spin.

For all experiments

- Select experiment from “Experiments” menu.



- Save data: Select File → Save or press Ctrl + S.

^{1}H and ^{13}C Spectrum

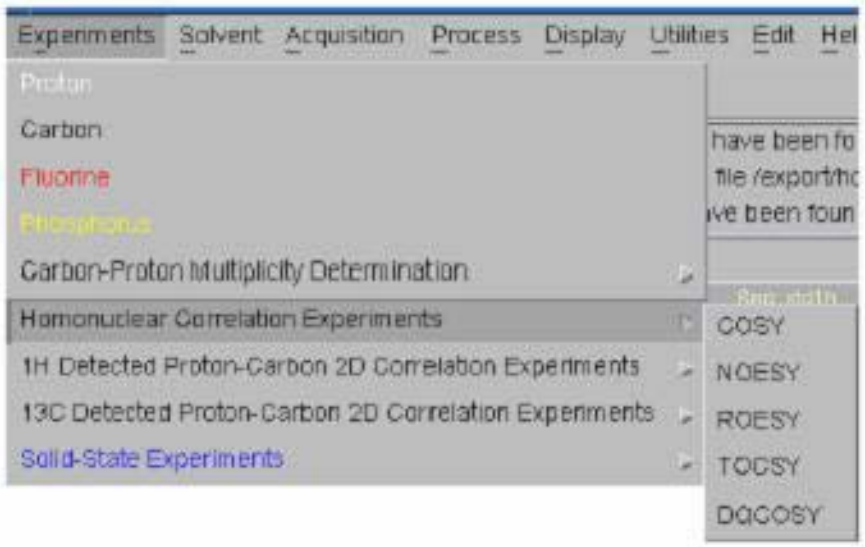
- Enter “nt=scan times bs=block size ga” in command line.

^{1}H and ^{13}C Spectrum

- Data process: Enter “wft aph” to display spectrum and carry out phase correction.
- Set reference and integrate (only for ^{1}H).
- Set peak display threshold: Press  button in toolbar on the right.
- Plot: Enter “phs” (for ^{1}H) or “pcs” (for ^{13}C).

$^{1}H-^{1}H$ correlation

- Scan ^{1}H spectrum or load saved spectrum in one experiment zone at first;
- Jump to another zone using jexp+number command (for example: jexp2 to jump to zone 2);
- Write down sample information, choose the solvent used and select experiment from “Experiments” menu.

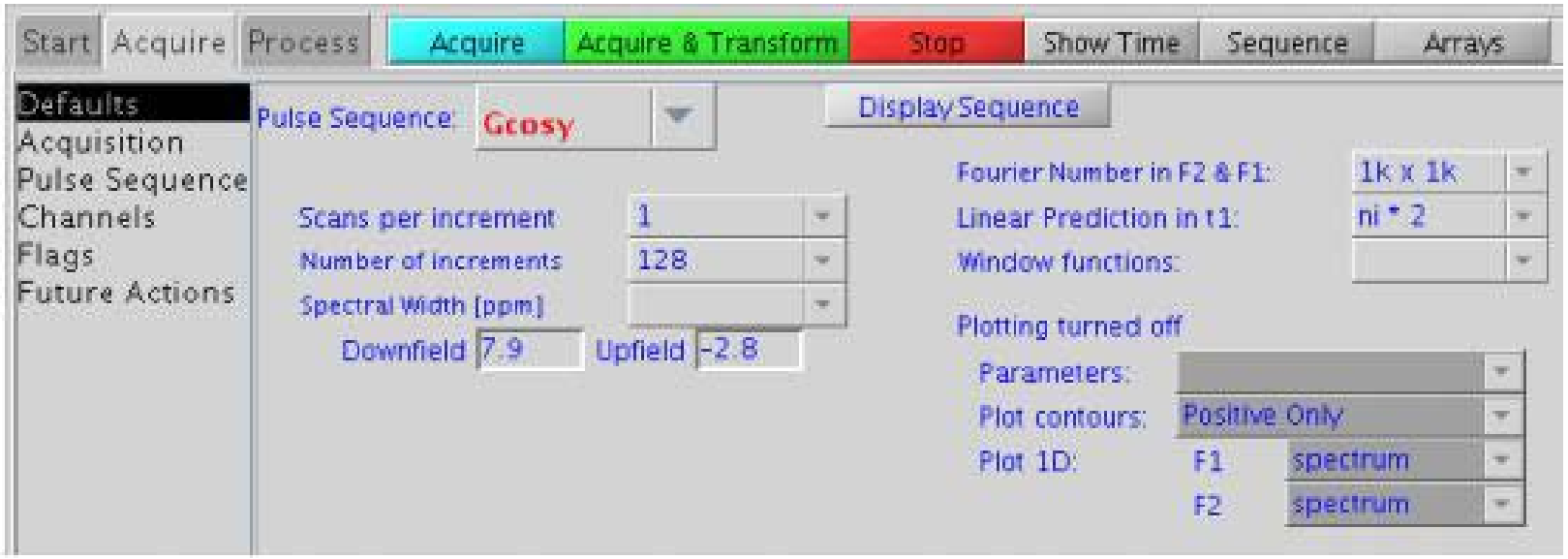


$^1H-^1H$ correlation

- Recommended parameters for $^1H-^1H$ correlation spectrum:

COSY	Scans per increment(nt) Number of increment(ni)	4(min),multiple of 4(max) 128 or more
NOESY	Scans per increment(nt) Number of increment(ni) Mixing time(mix)	8(min),multiple of 32(max) 128 or more
DQCOSY	Scans per increment(nt) Number of increment(ni)	8(min),multiple of 32(max) 128 or more
TOCSY	Scans per increment(nt) Number of increment(ni) Mixing time(mix)	8(min),multiple of 32(max) 128 or more 0.08, range:0.03~0.1
ROESY	Scans per increment(nt) Number of increment(ni) Mixing time(mix)	8(min),multiple of 8(max) 128 or more 0.3, range:0.1~0.8
gCOSY	Scans per increment(nt) Number of increment(ni)	4(min),multiple of 4(max) 128 or more

gCOSY


Start
Acquire
Process
Acquire
Acquire & Transform
Stop
Show Time
Sequence
Arrays
Defaults
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
Pulse Sequence:
Gcosy
Display Sequence
Scans per increment
1
Number of increments
128
Spectral Width [ppm]
Downfield
7.9
Upfield
-2.8
Fourier Number in F2 & F1:
1k x 1k
Linear Prediction in t1:
ni * 2
Window functions:
Plotting turned off
Parameters:
Plot contours:
Positive Only
Plot 1D:
F1
spectrum
F2
spectrum

NOESY


Start
Acquire
Process
Acquire
Acquire & Transform
Stop
Show Time
Sequence
Arrays
Defaults
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
Pulse Sequence: Noesy
Scans per increment 8
Number of increments 128
Mixing time [ms] 400
Relaxation time [s] 1
Spectral Width [ppm] 14 → -2
Downfield 14.0 Upfield -2.0
Zero Quantum Suppression
Fourier Number in F2 & F1: 1k x 1k
Linear Prediction in t1: ni * 2
Window functions:
Plotting turned off
Parameters:
Plot contours:
Plot 1D: F1
F2

TOCSY


Start
Acquire
Process
Acquire
Acquire & Transform
Stop
Show Time
Sequence
Arrays
Defaults
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
Pulse Sequence: Tocsy
Scans per increment 4
Number of increments 128
Spectral Width [ppm] 14 → -2
Downfield 14.0
Upfield -2.0
Mixing time [ms] 80
Z-TOCSY with ZQ suppression
XY-TOCSY
Fourier Number in F2 & F1: 1k x 1k
Linear Prediction in t1: ni * 2
Window functions:
Plotting turned off
Parameters:
Plot contours:
Plot 1D:
F1
F2

Data process for 1H-1H correlation

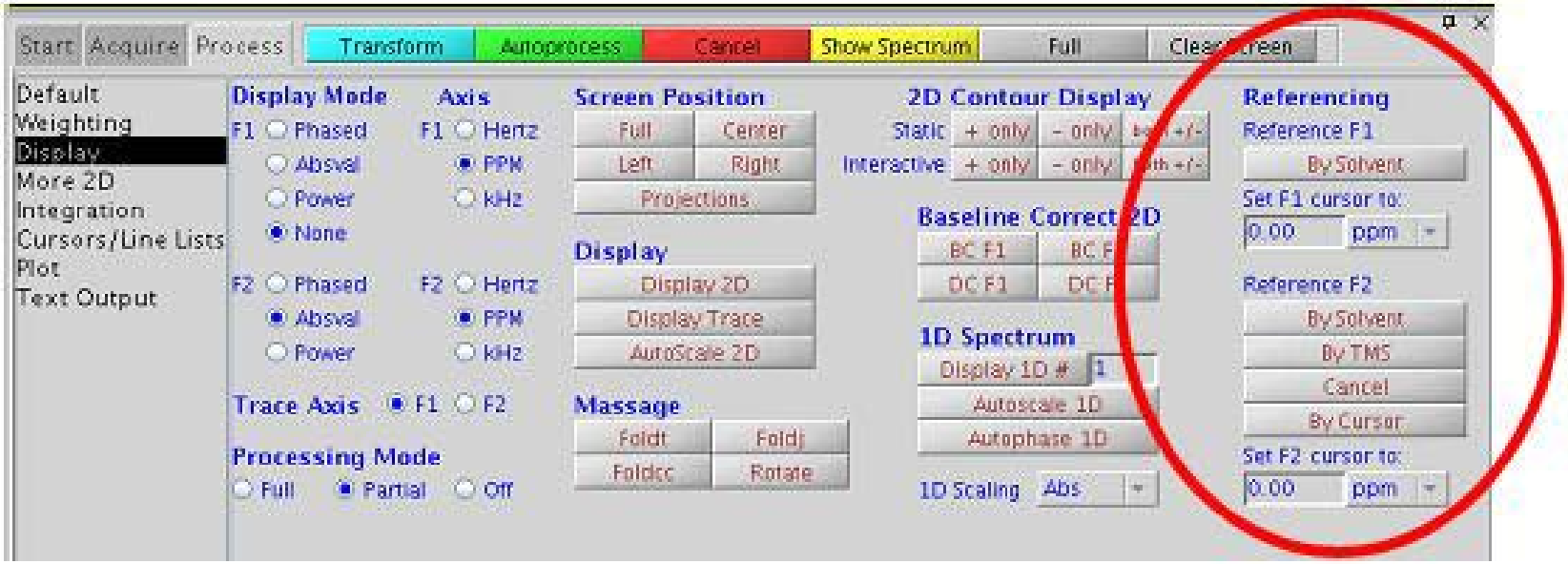
- During the scan: Jump to a third zone, and use “mf (the scan zone, the third zone)” (for example, “mf(2,3)”) to transfer data to the target zone.
- Press “Autoprocess” button to display 2D spectrum.

Data process for 1H-1H correlation

- Phase correction: Enter “aph” for auto phase correction.
- Adjusting vertical height using  or  button in toolbar on the right.
- Display 2D spectrum: Enter “dconi” command.

Data process for 1H-1H correlation

- Referencing:

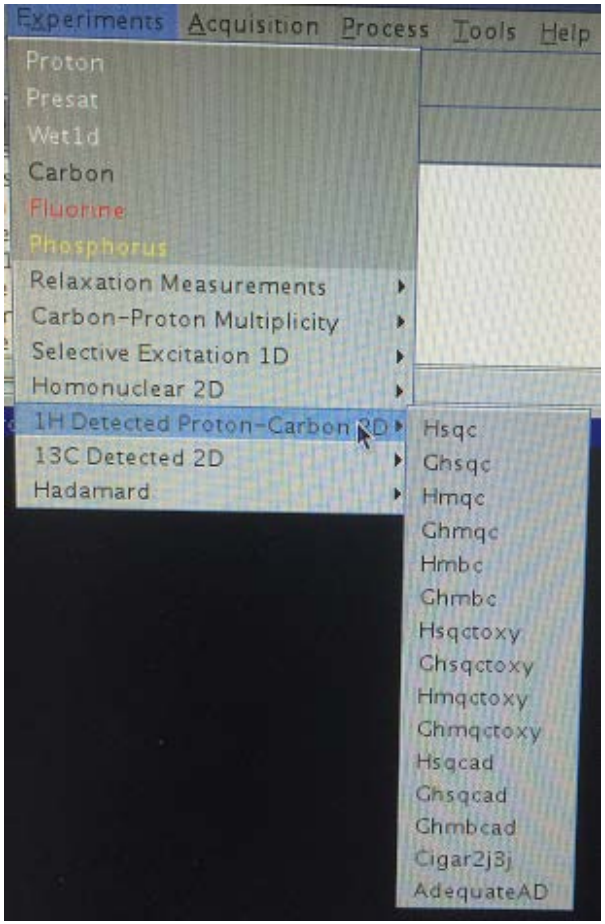


Plot ^{1}H-^{1}H spectrum

- Enter “plcosy(15,1.2,experiment zone of loaded ^{1}H spectrum)” (for example, plcosy(15,1.2,1) if the ^{1}H spectrum is in zone 1) to plot 2D spectrum.

HSQC and HMBC

- Scan ^{1}H and ^{13}C spectrum or load saved spectrum in two experiment zones at first;
- Jump to another zone;
- Write down sample information, choose the solvent used and select experiment from “Experiments” menu.
- Data process of HSQC and HMBC is similar to ^{1}H-^{1}H correlation.



gHSQC and gHMBC

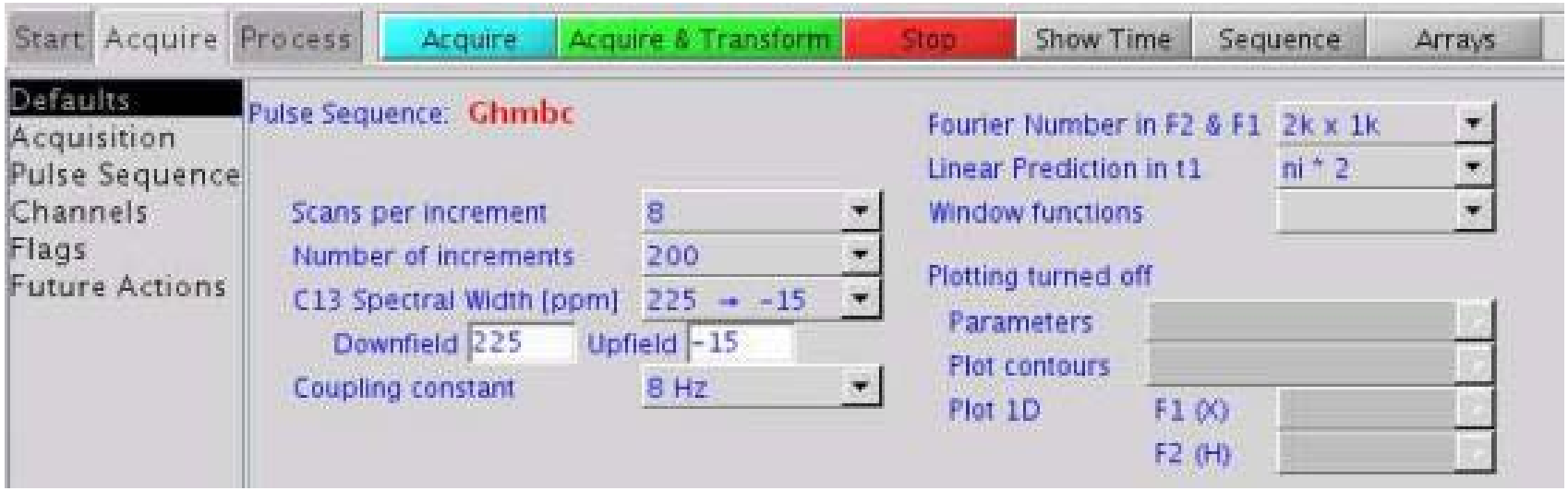
- Recommended parameters for HSQC and HMBC:

gHSQC	Scans per increment(nt)	4(min),multiple of 4(max)
	Number of increment(ni)	128 or more
	C13 Spectral Width［ppm］(sw1)	160ppm~-10ppm, selectable
	C-H Multiplicity Edit(mult)	mult=2(CH,CH3 up, CH2 down)
gHMBC	Scans per increment(nt)	8(min),multiple of 4(max)
	Number of increment(ni)	200 or more
	C13 Spectral Width［ppm］(sw1)	225ppm~-15ppm, selectable
	Coupling constang(jnxh)	8,range 5-10Hz

gHSQC


Start
Acquire
Process
Acquire
Acquire & Transform
Stop
Show Time
Sequence
Arrays
Defaults
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
Pulse Sequence: Ghsqc
Scans per increment 2
Number of increments 128
C13 Spectral Width [ppm] 160 → -10
Downfield 160
Upfield -10
C-H Multiplicity Edit Yes
Fourier Number in F2 & F1 2k x 1k
Linear Prediction in t1 ni * 2
Window functions
Plot when done
Parameters Basic, Top Left
Plot contours Positive & Negative
Plot 1D F1 (X) spectrum
F2 (H) spectrum

gHMBC

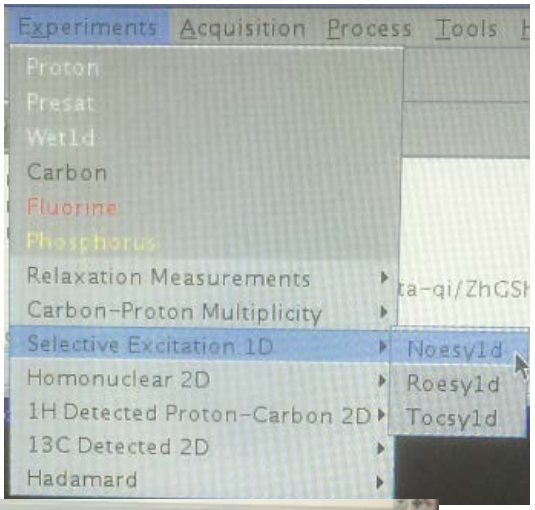

Start
Acquire
Process
Acquire
Acquire & Transform
Stop
Show Time
Sequence
Arrays
Defaults
Acquisition
Pulse Sequence
Channels
Flags
Future Actions
Pulse Sequence: Ghmbc
Scans per increment 8
Number of increments 200
C13 Spectral Width [ppm] 225 → -15
Downfield 225
Upfield -15
Coupling constant 8 Hz
Fourier Number in F2 & F1 2k x 1k
Linear Prediction in t1 ni * 2
Window functions
Plotting turned off
Parameters
Plot contours
Plot 1D
F1 (X)
F2 (H)

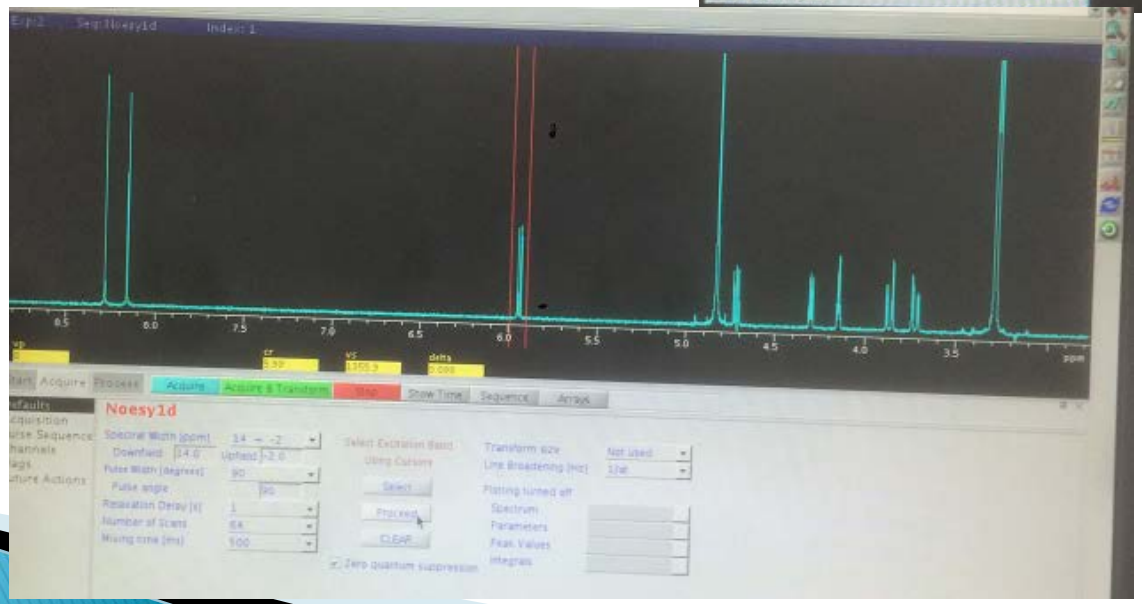
Plot HSQC and HMBC

- Enter “plhxcor(15,1.2,experiment zone of loaded ^{1}H spectrum,experiment zone of loaded ^{13}C spectrum)” (for example, plhxcor(15,1.2,1,2) if the ^{1}H spectrum is in zone 1 and the ^{13}C spectrum is in zone 2) to plot 2D spectrum.

1D NOESY (ROESY and TOCSY)

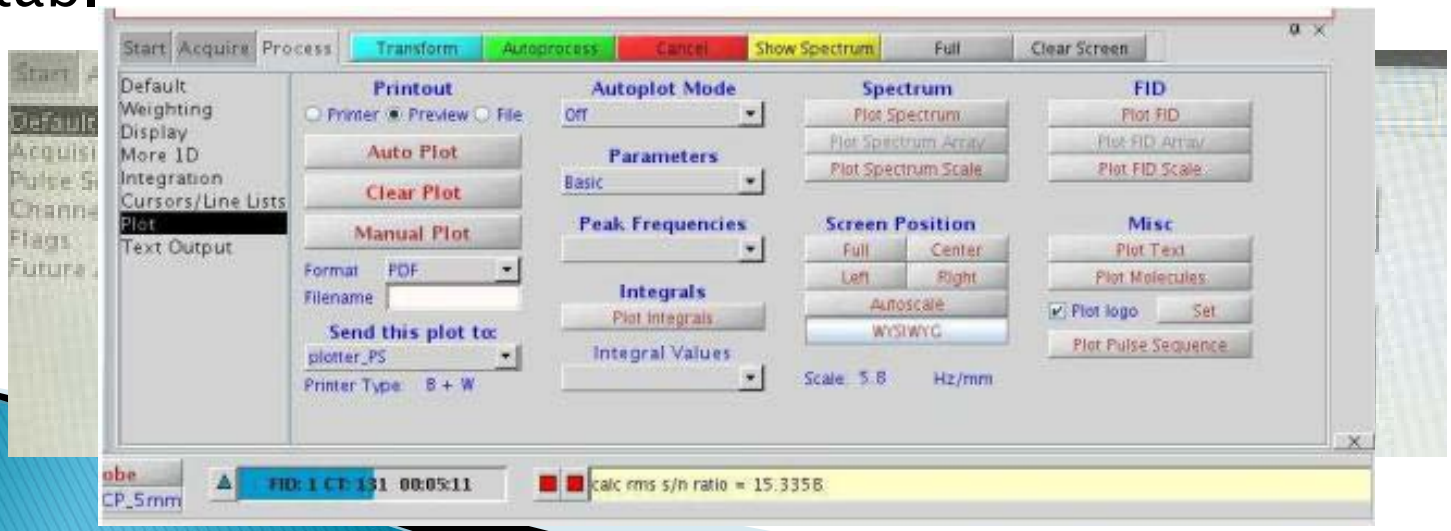
- Select experiment;
- Load a ^{1}H spectrum and conduct referencing at first, then select H in the spectrum to be excited;
- Press “Select” button and “Proceed” button;





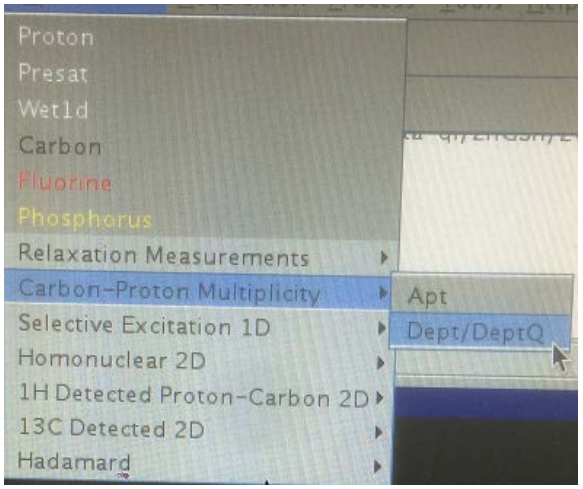
1D NOESY (ROESY and TOCSY)

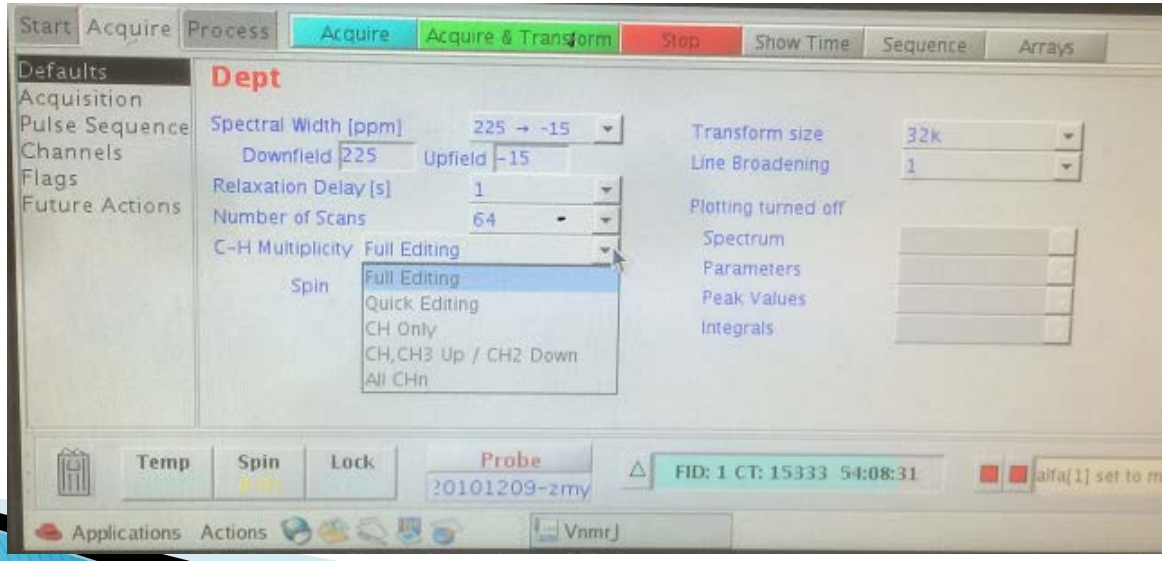
- Type command like ^{1}H spectrum, for example, nt=128 bs=4 ga, or select number of scans in Defaults tab and block size in Flags tab then press the green “Acquire & Transform” button to start scanning.
- Data process is similar to ^{1}H spectrum.
- Press “Auto Plot” button in “Process” menu at “Plot” tab.



DEPT

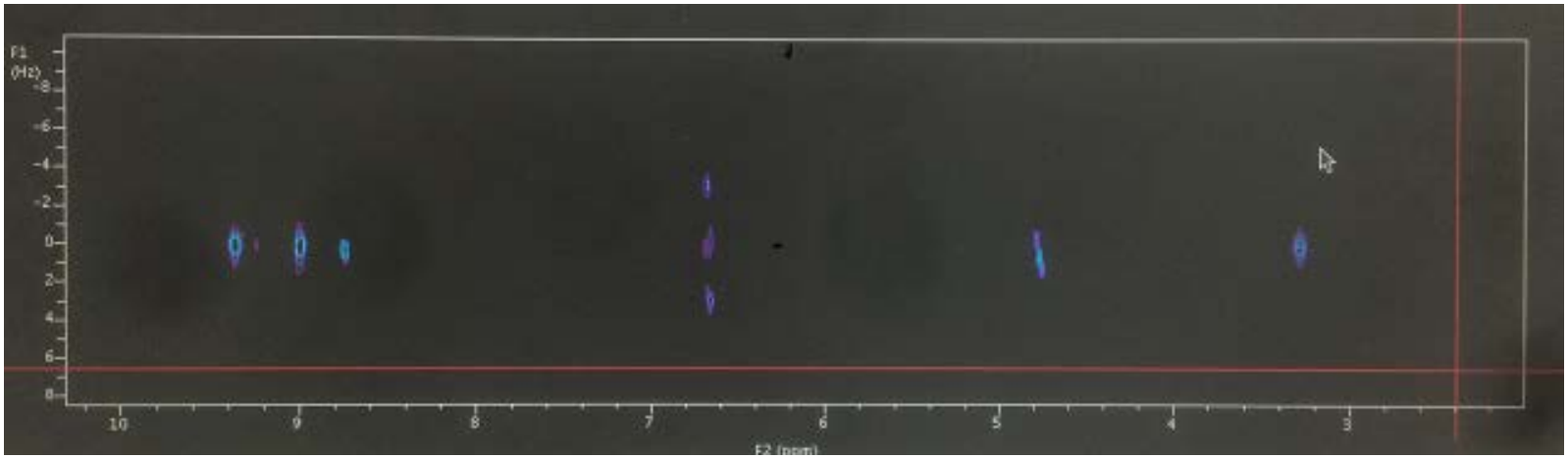
- Select experiment;
- Set parameters and start acquire;
- Data process is similar to ^{13}C spectrum.





Homo 2DJ

- 2D J-resolved Spectroscopy: Chemical shift in one dimension and J-value in another.
- A typical homo 2DJ spectrum as below:



Homo 2DJ

- Set parameters as follows;
- Data process is similar to COSY.