Use of the NMR Spectrometer

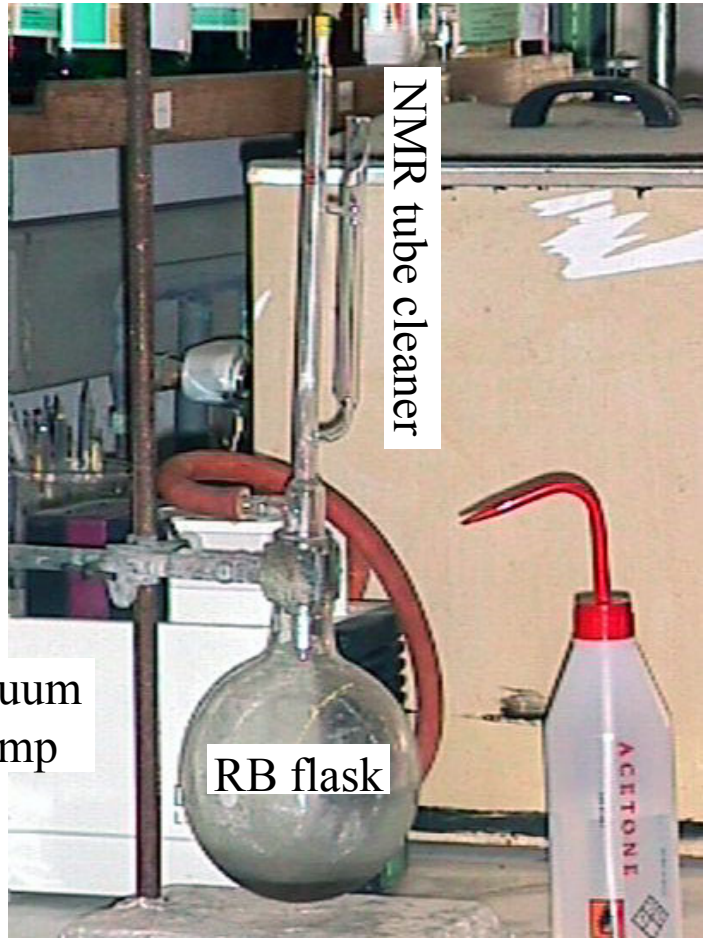
A Practical guide

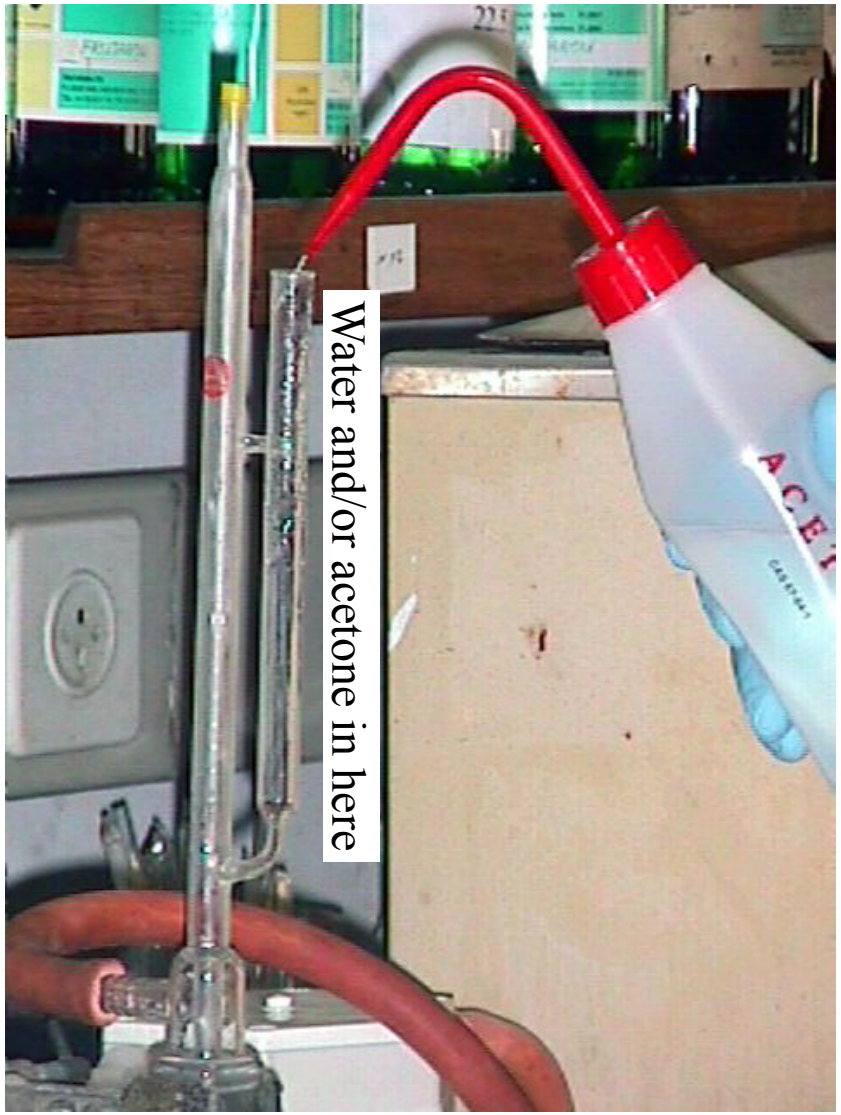
Choose NMR tube

- 300MHz
  - Wilmad 507-PP-7
  - Aldrich Series 300
  - Kontes Grade 3
- 400MHz
  - Wilmad 528-PP-7
  - Aldrich Series 400
  - Kontes Grade 6

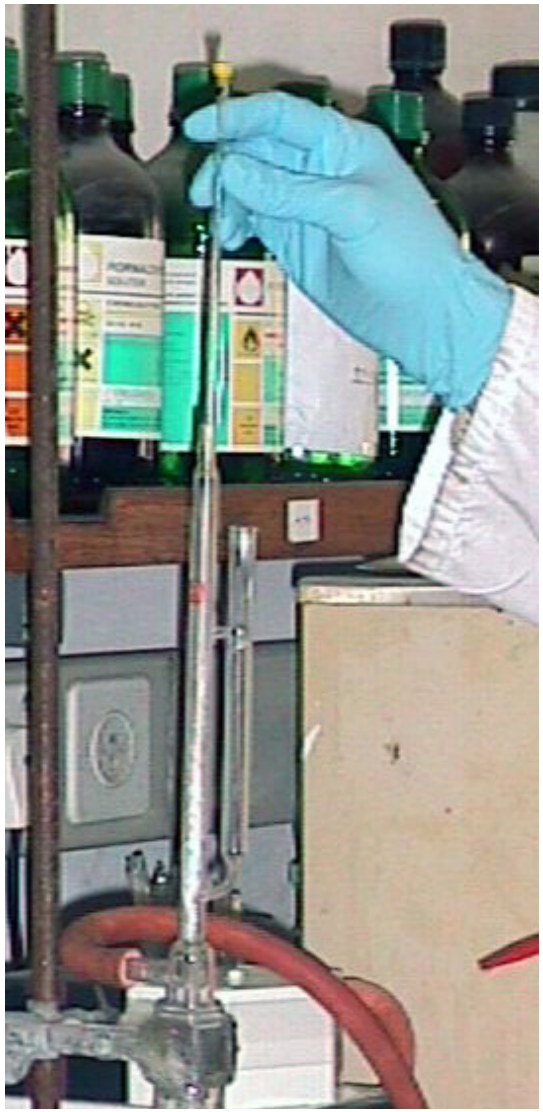
Ensure that the tube is clean

First remove any labels then use an NMR tube cleaner



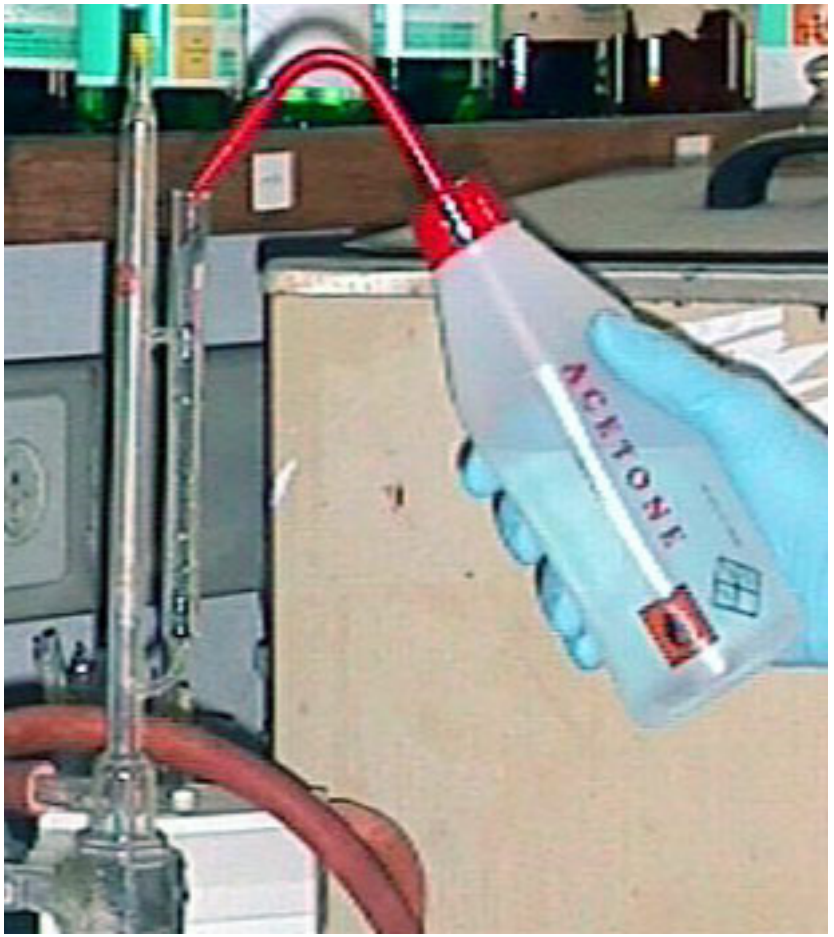


Wash the cap



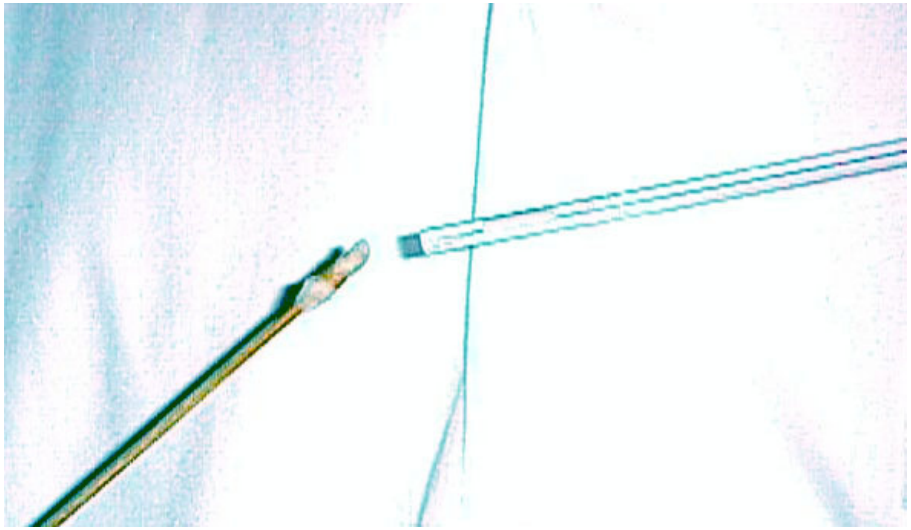
Place cap on the bottom of the NMR tube and insert the tube

Wash the outside of the tube

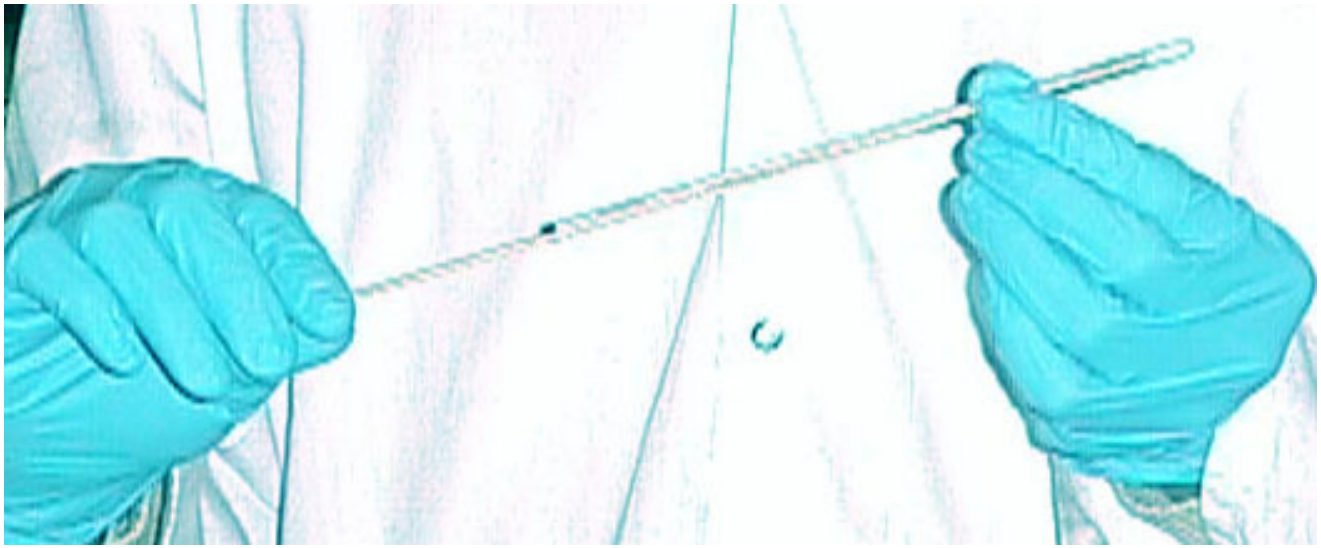


Insert the tube completely and
Wash the inside of the tube

If you are not sure it is now clean put some cotton wool on the end of a stick,



Soak it with acetone and insert it into the tube



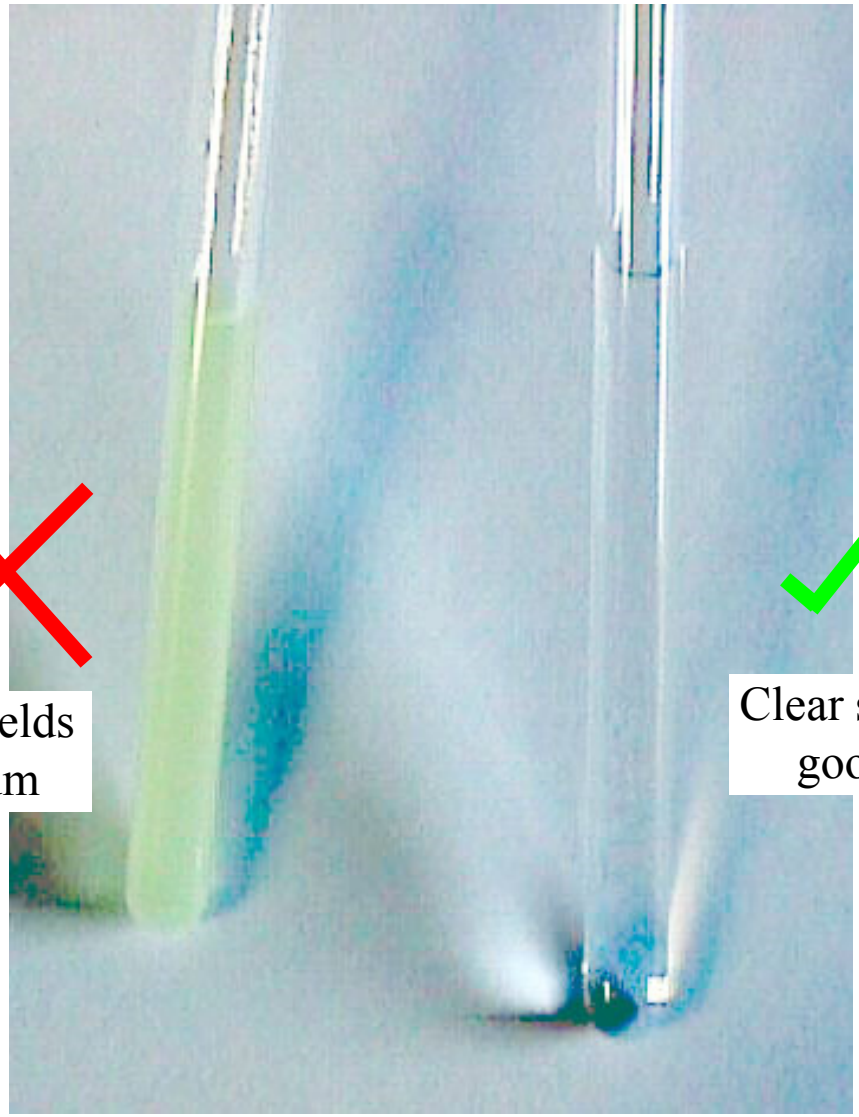
Repeat with fresh cotton wool until it comes out clean

Choose solvent

- Deuterated
  - Solvent signal less likely to swamp sample signal
  - Allows field frequency lock
  - Easier to achieve homogeneity
- Solubility
  - Preferably over 2 mg/mL for ^{1}H
  - Preferably over 10 mg/mL for ^{13}C
- Cost: D_2O and $CDCl_3$ are the cheapest so used most often
- Overlapping signals: If the sample's signals are near the solvent signal consider another solvent

The sample must be clear

or the spectrum will yield broad and misshapen signals



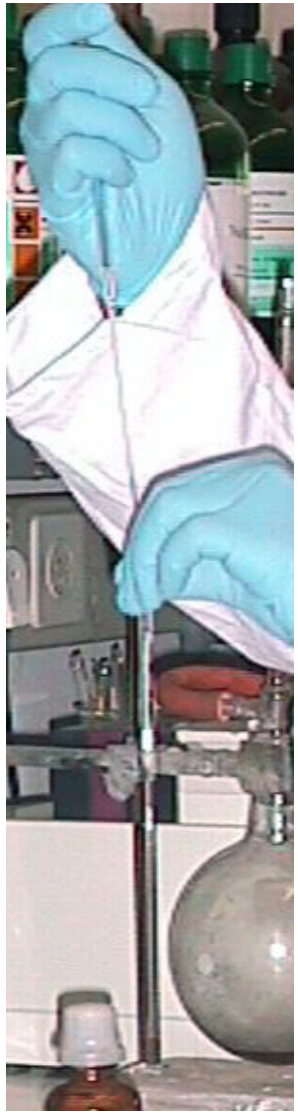
Suspension yields poor spectrum

Clear solution yields good spectrum

Filter it if there is precipitate

Insert a piece of cotton or glass wool into a pipette

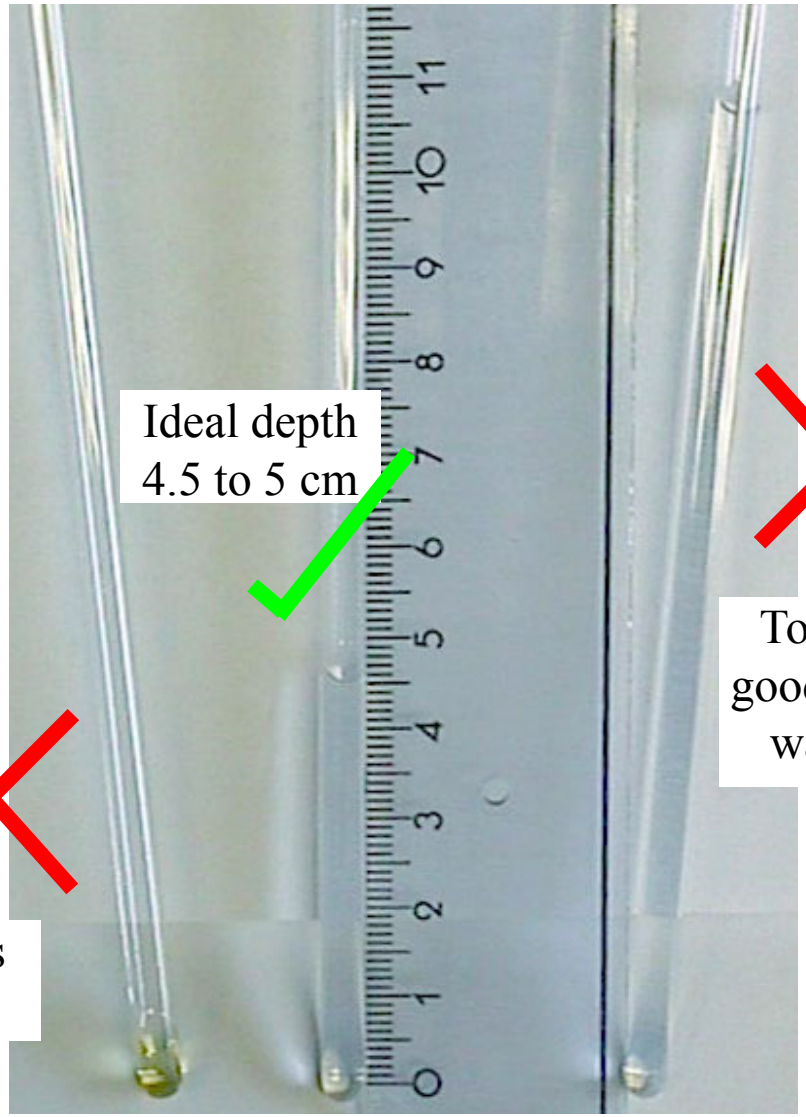
Pack it down

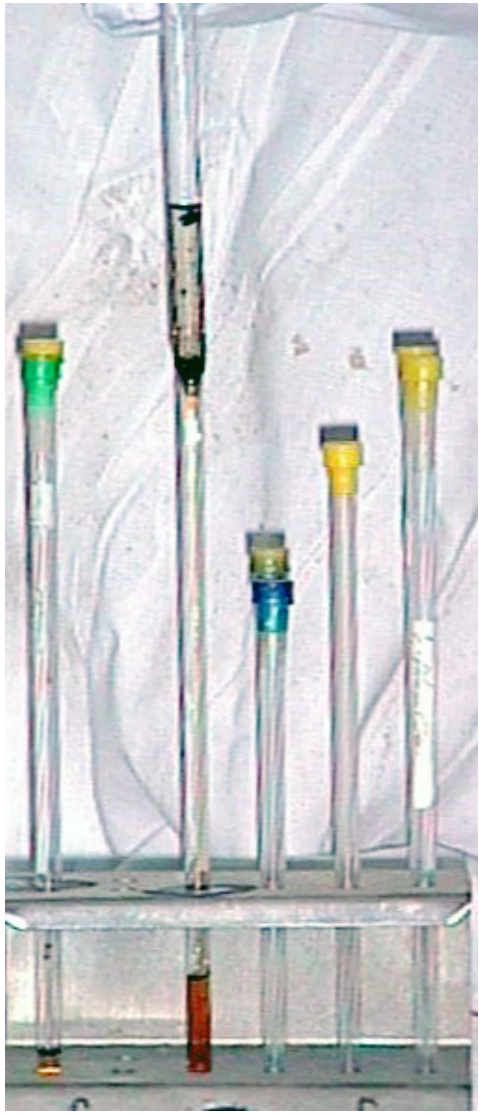


Wash it with a little solvent

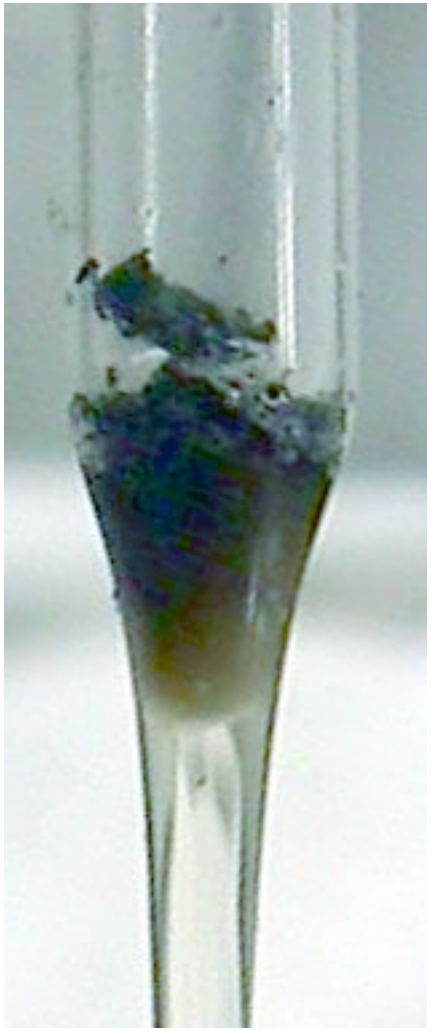
But never return the solvent to the bottle

Solvent depth 4.5 to 5 cm

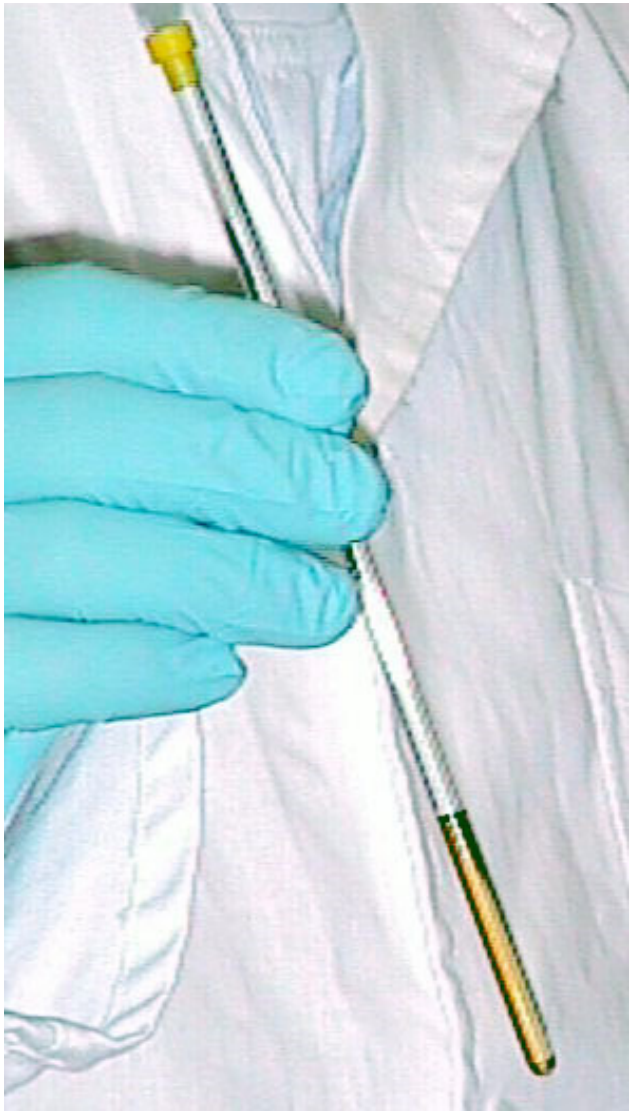




Filter the suspension

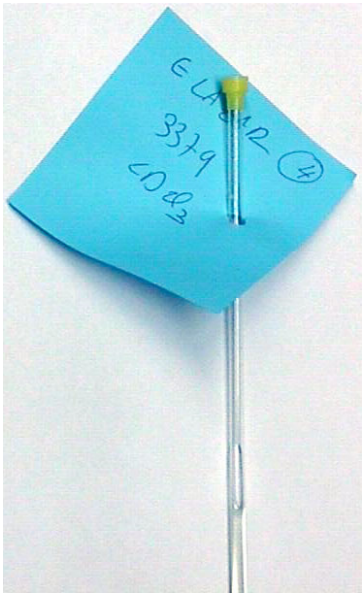


The residue stays
in the filter

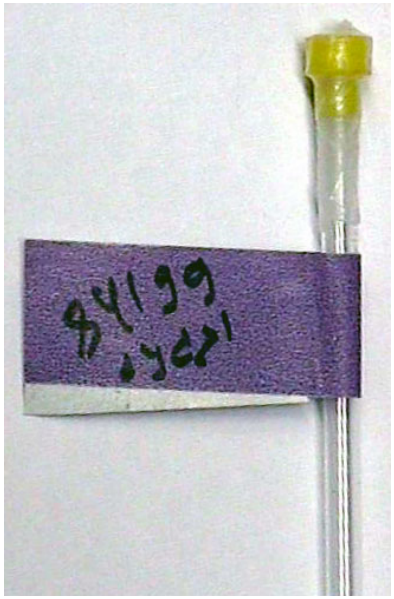


and the sample is clear

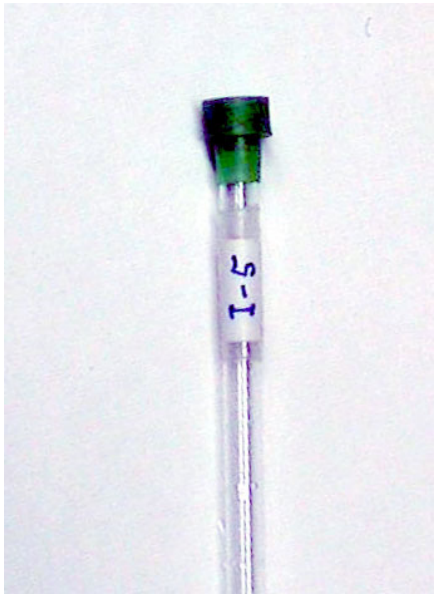
Label it concentrically



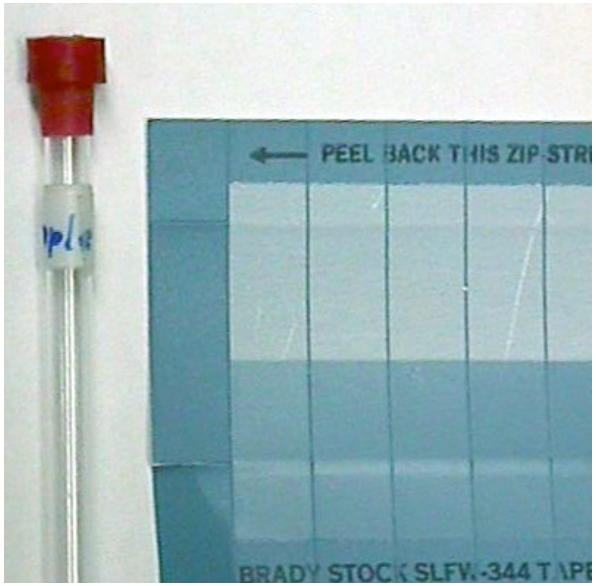
Must be removed to run sample & may get lost



Stuck on & does not fit in the magnet

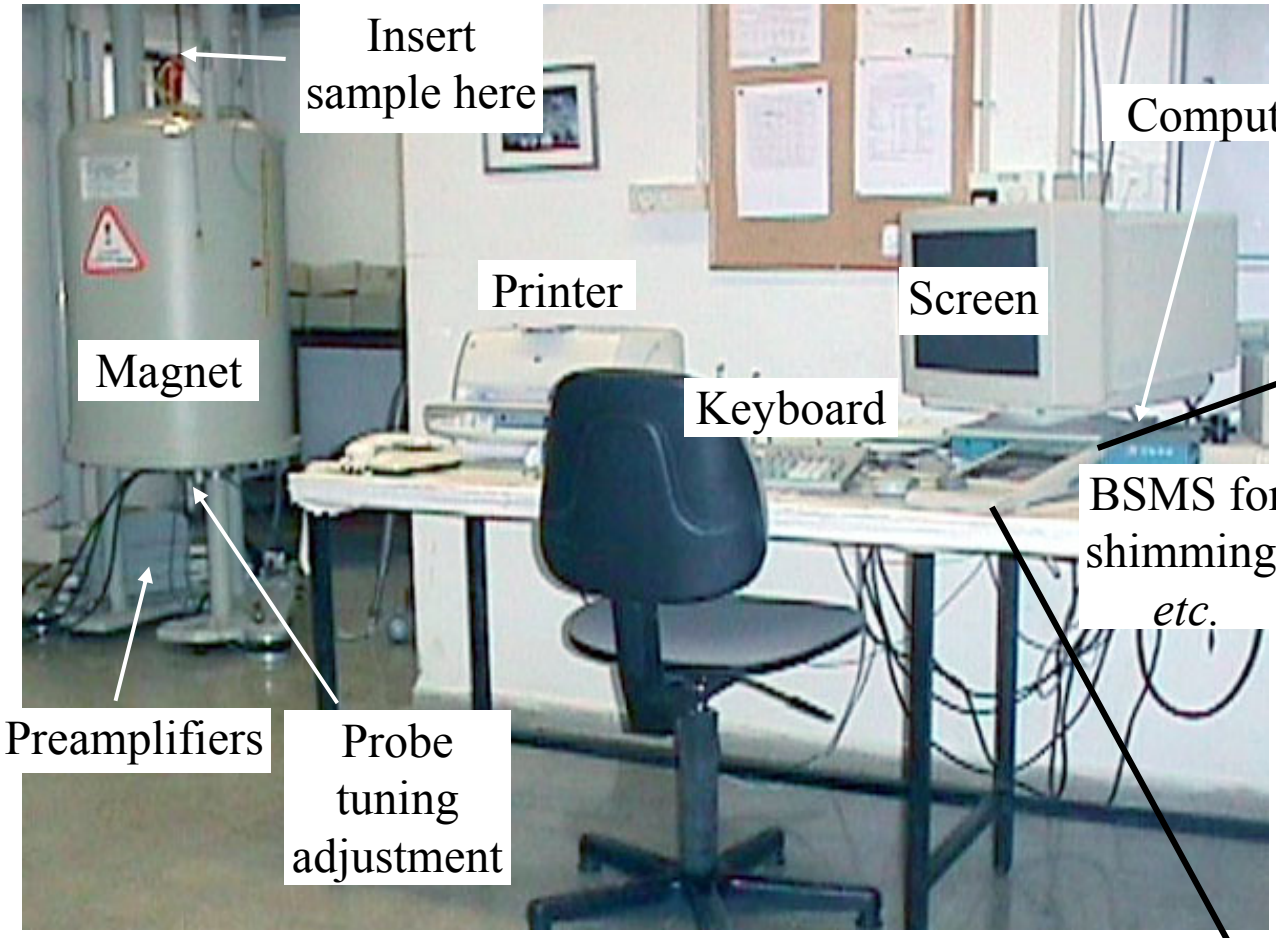


Paper & magic tape



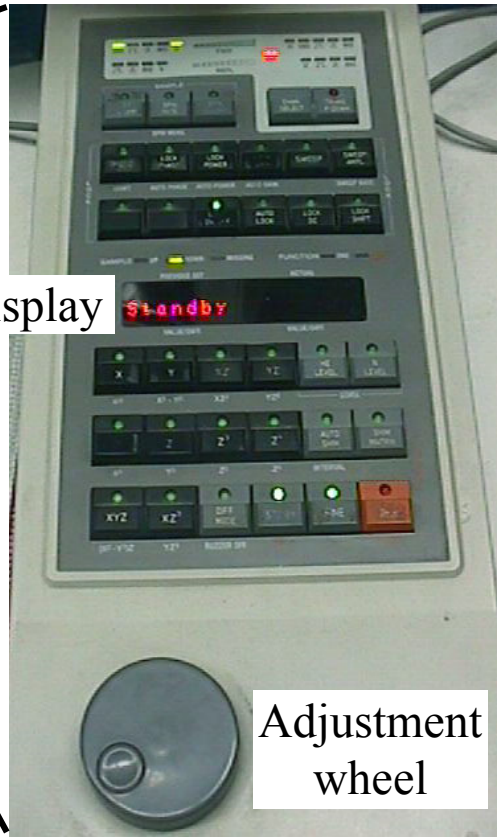
Aldrich NMR tube lable

NMR Spectrometer



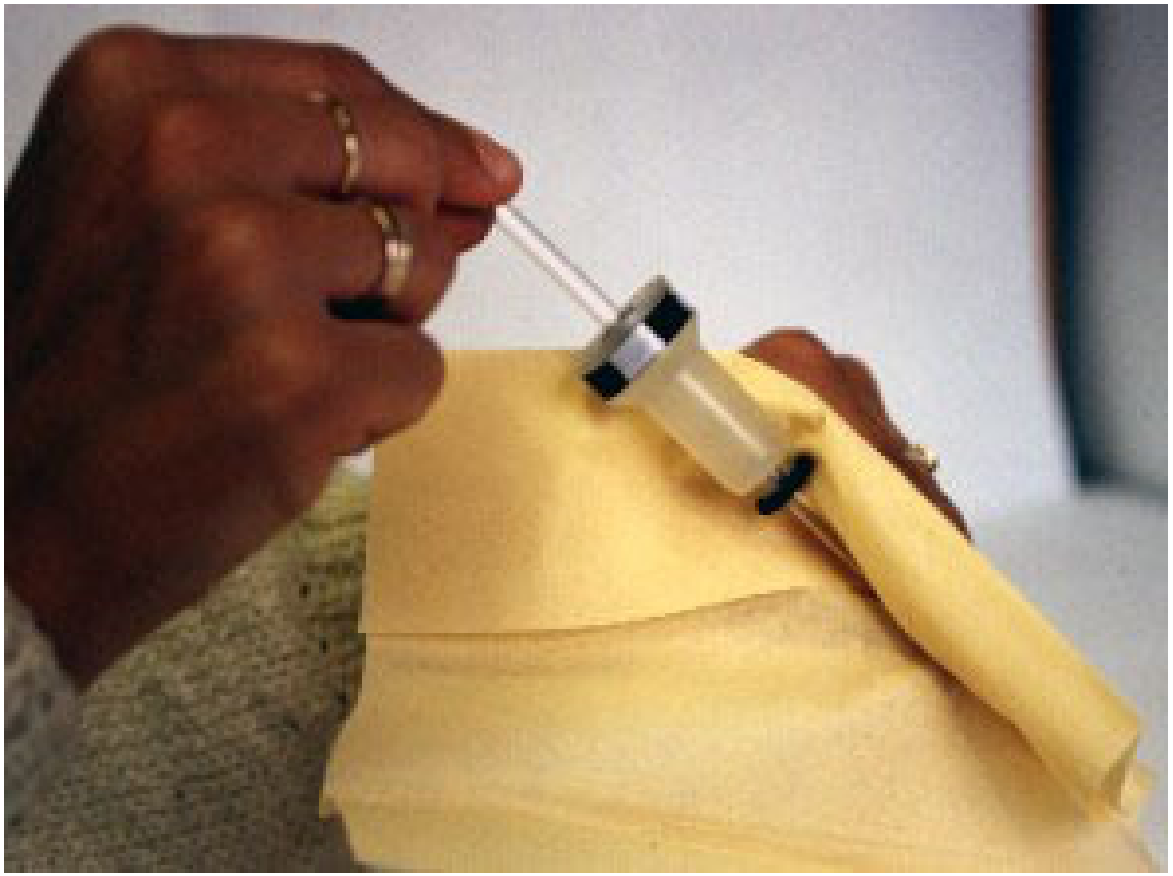
Bruker DRX 400 spectrometer

RF pulse display

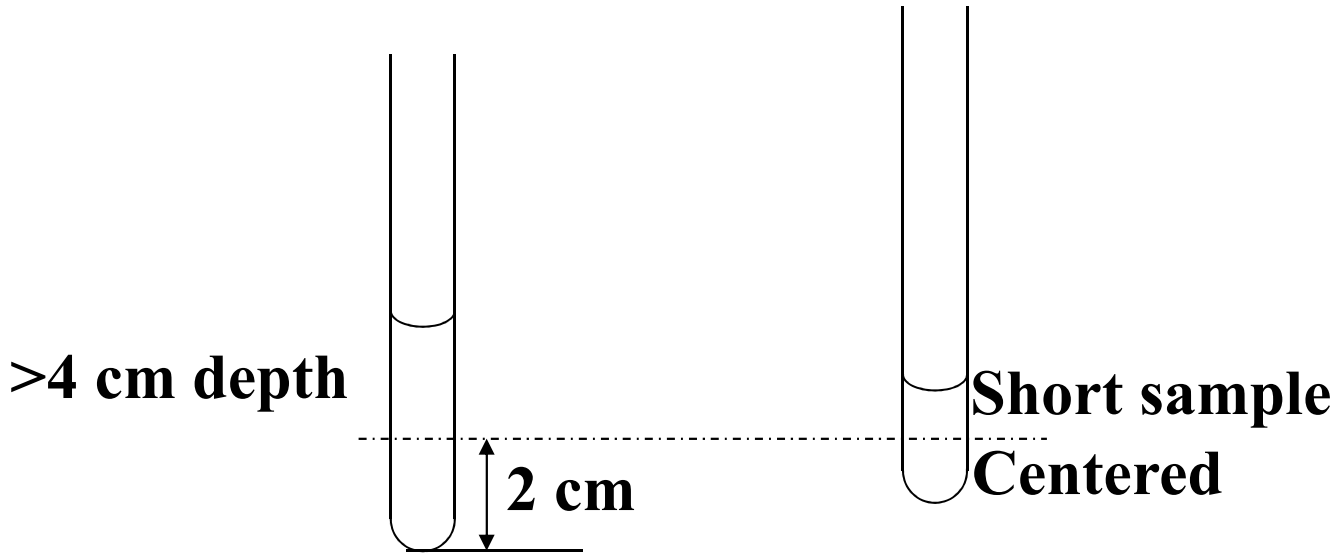


Cabinet not shown

Put sample in spinner and clean with a dry tissue



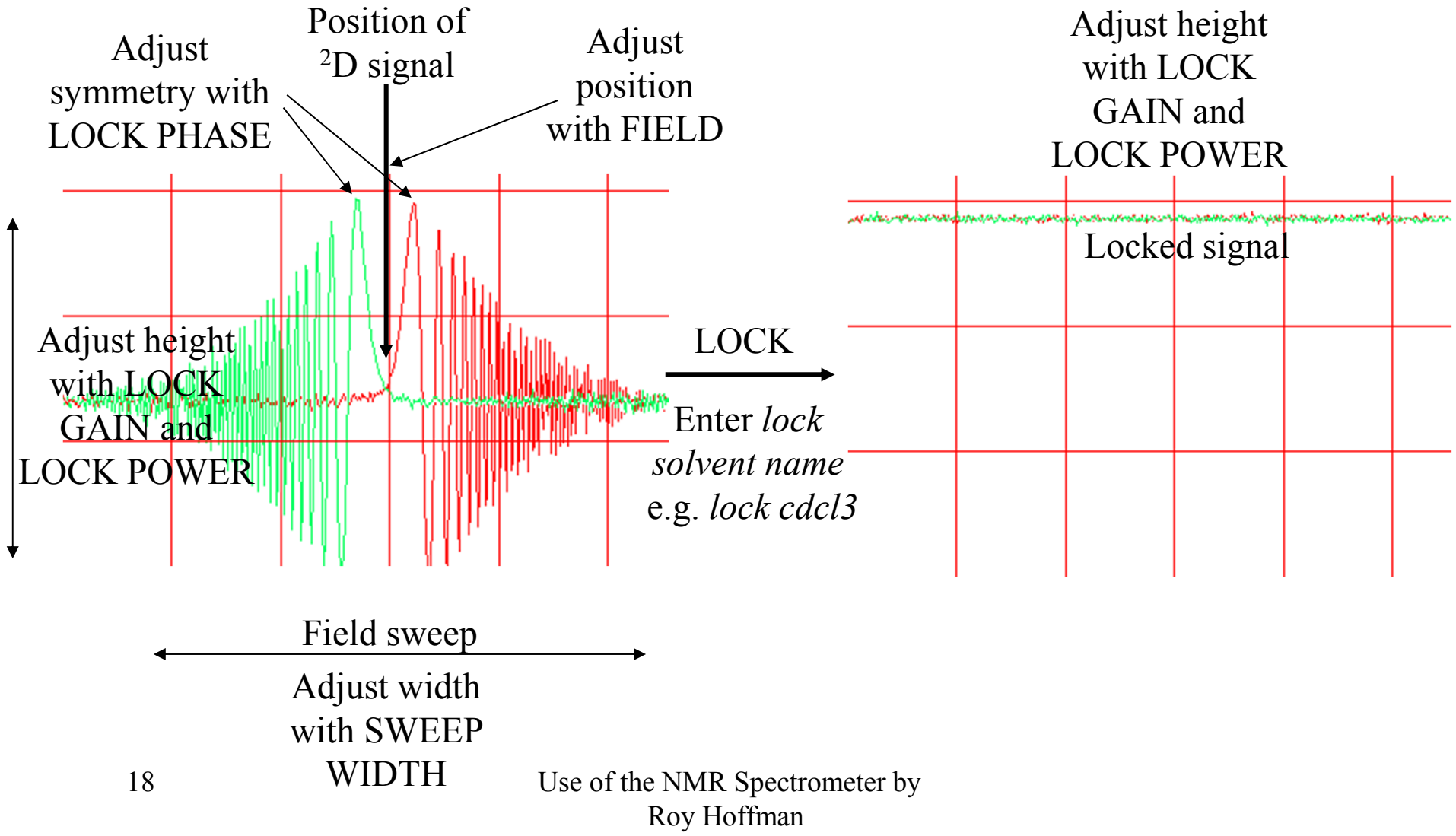
Sample position



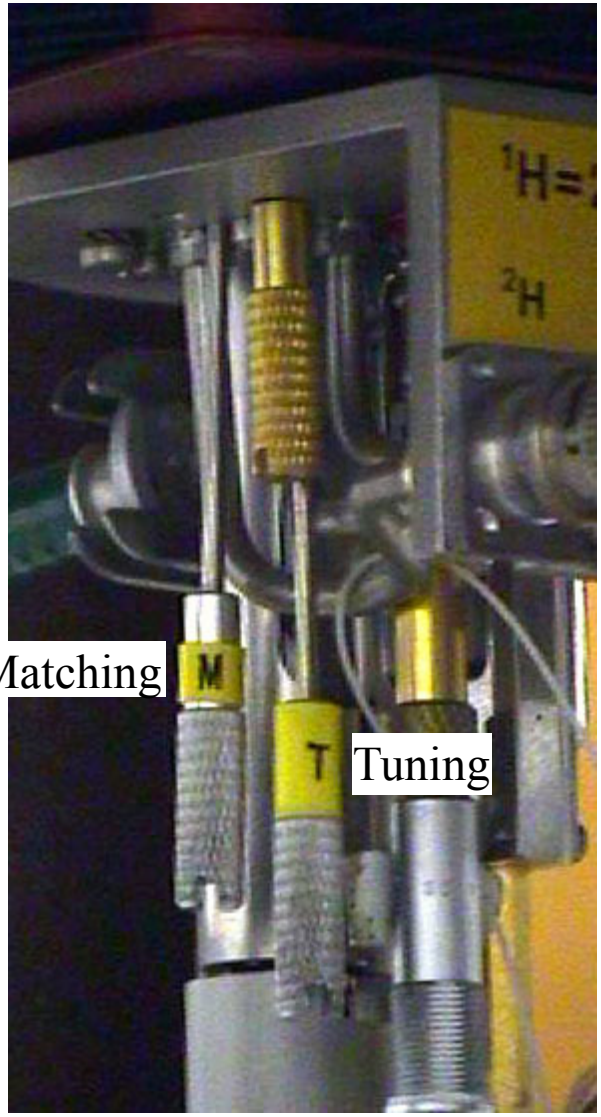
Inserting the sample

Sample in probe

Field-frequency Lock



Probe tuning controls



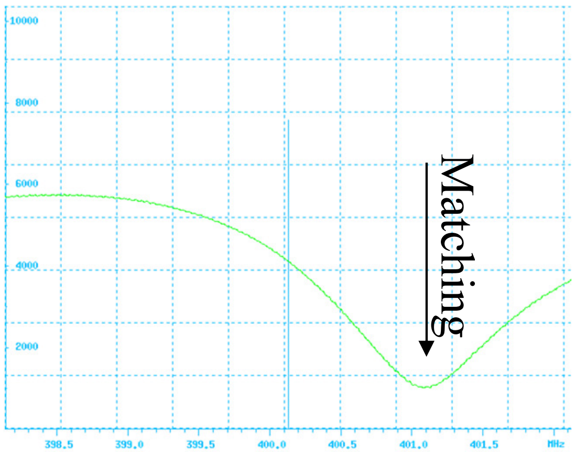
Tuning screws for ^{1}H

Tuning sliders for ^{13}C, *etc.*

How to tune the probe

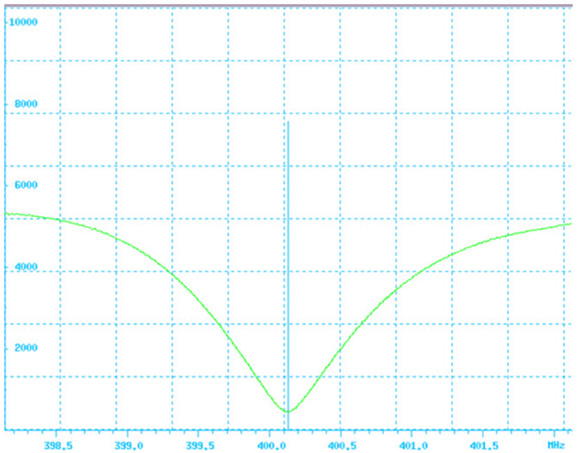
On screen

Start tuning



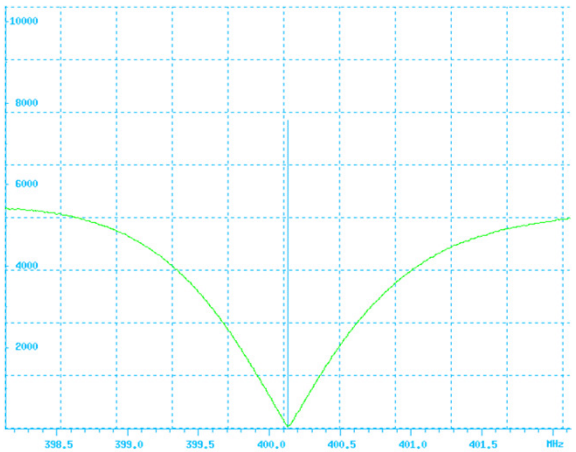
Tuned frequency

Adjust T



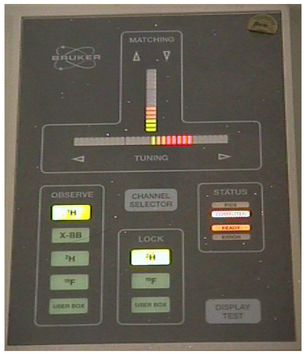
This is tuned

Adjust M while correcting T



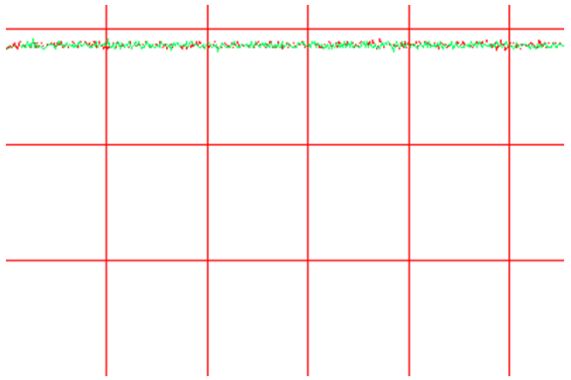
This is tuned & matched

On preamplifiers



Shimming

For basic shimming start adjust Z and Z2 (with sample spinning). For more thourough shimming (non-spinning) adjust X & Y then XZ & YZ (if there is a large change redo X & Y) then adjust XZ2 & YZ2 (if there is a large change redo X, Y, XZ & YZ) then adjust XY & X2-Y2. Spin and readjust Z & Z2, acquire the spectrum and if it looks reasonable adjust Z3 then Z & Z2. If there are problems with the spectrum use the lineshape as a guide



The higher the lock signal the better the shimming

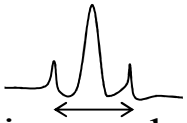
Increase Z2 then adjust Z

Reduce Z2 then adjust Z

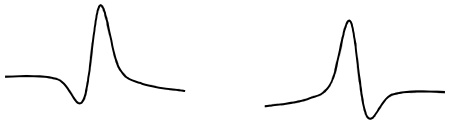
Adjust Z3 then Z & Z2

Adjust Z3 & Z then Z2

Problem with X, Y, *etc.*

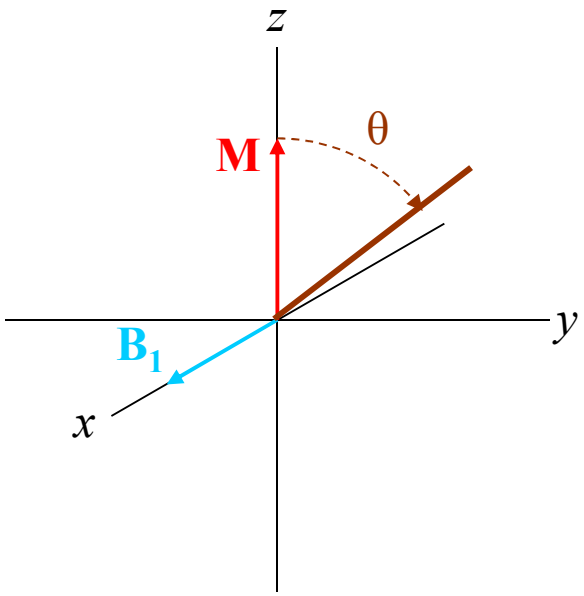


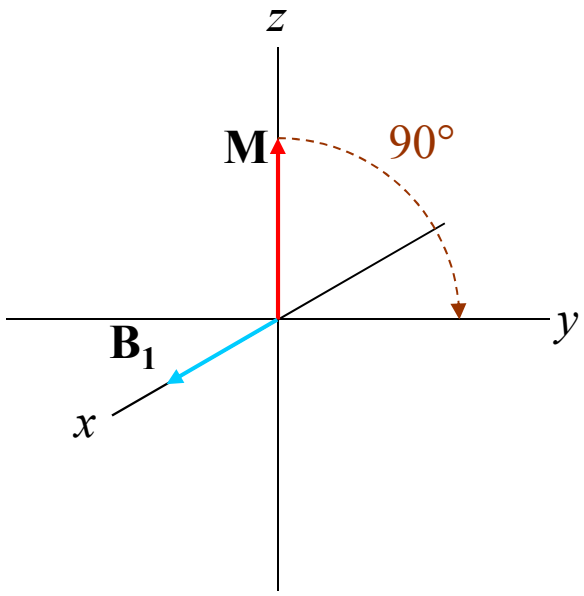
BUT shimming OK here



Just fix the phase

Effect of an rf pulse





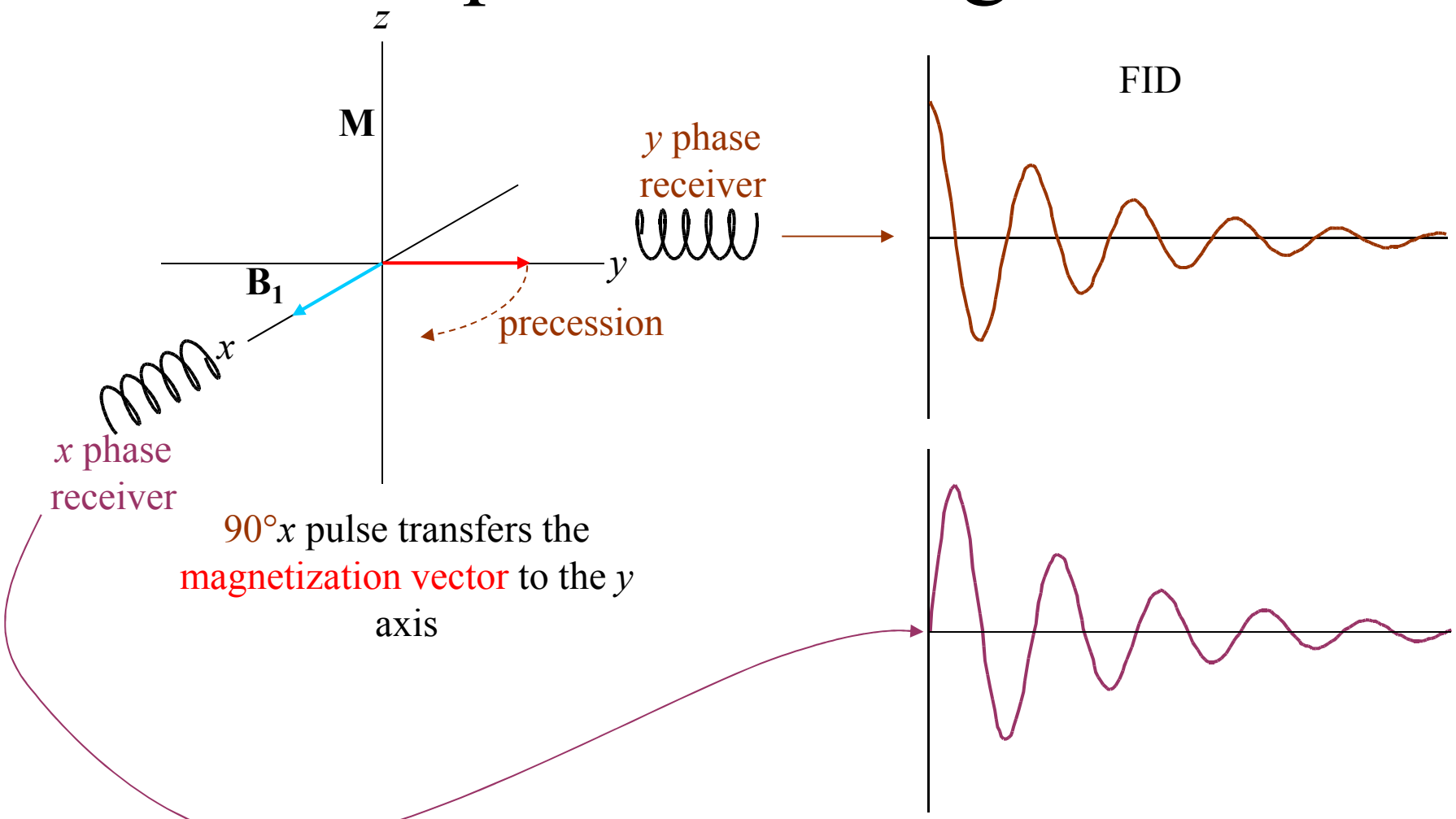
$\theta \propto \log(B_1)Tp$

90°*x* pulse transfers the magnetization vector to the *y* axis

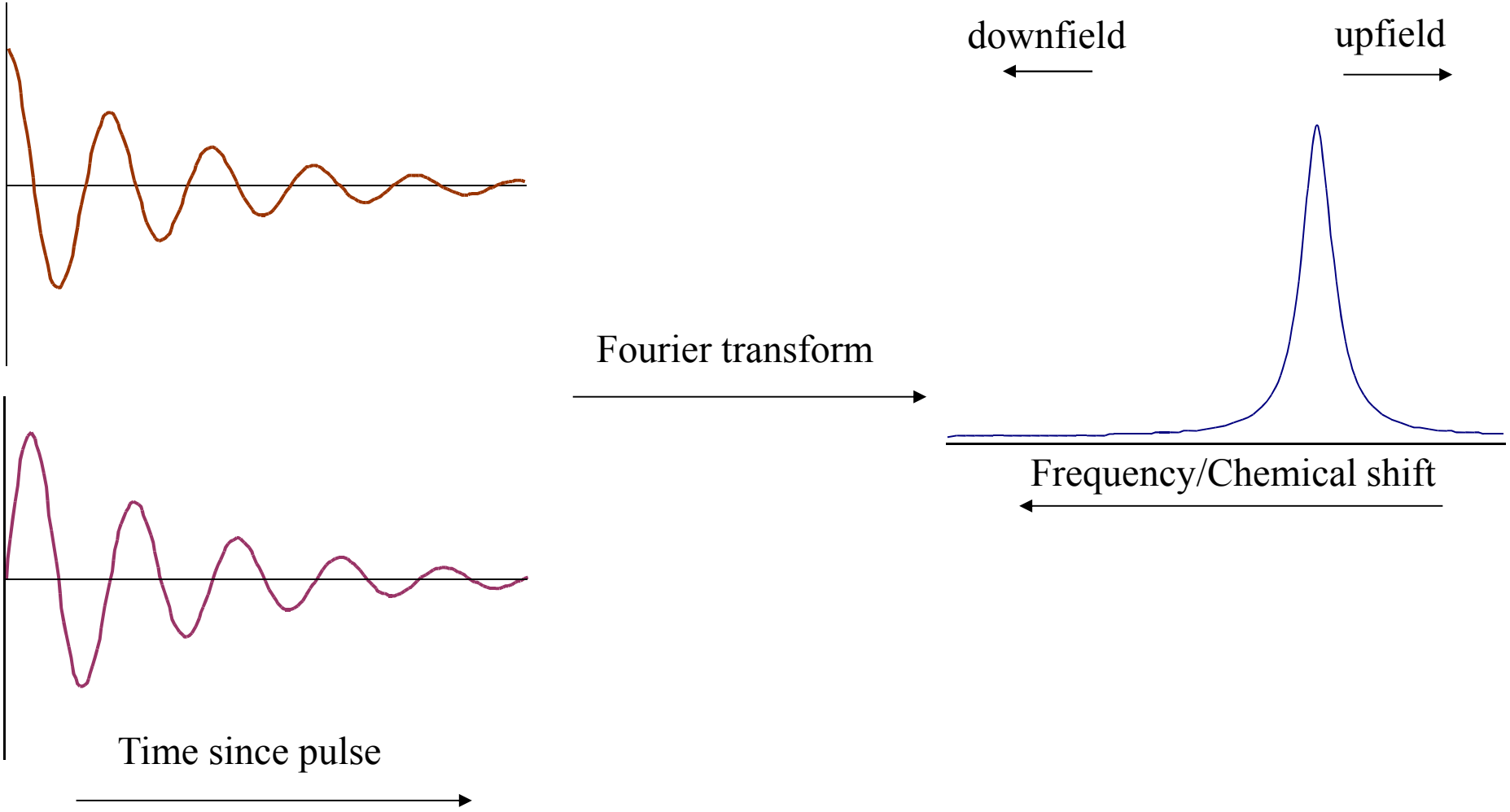
Pulse power, on the spectrometer use the attenuation in dB: pl1, pl2, *etc.*

Pulse duration on the spectrometer in μs: p1, p2, *etc.*

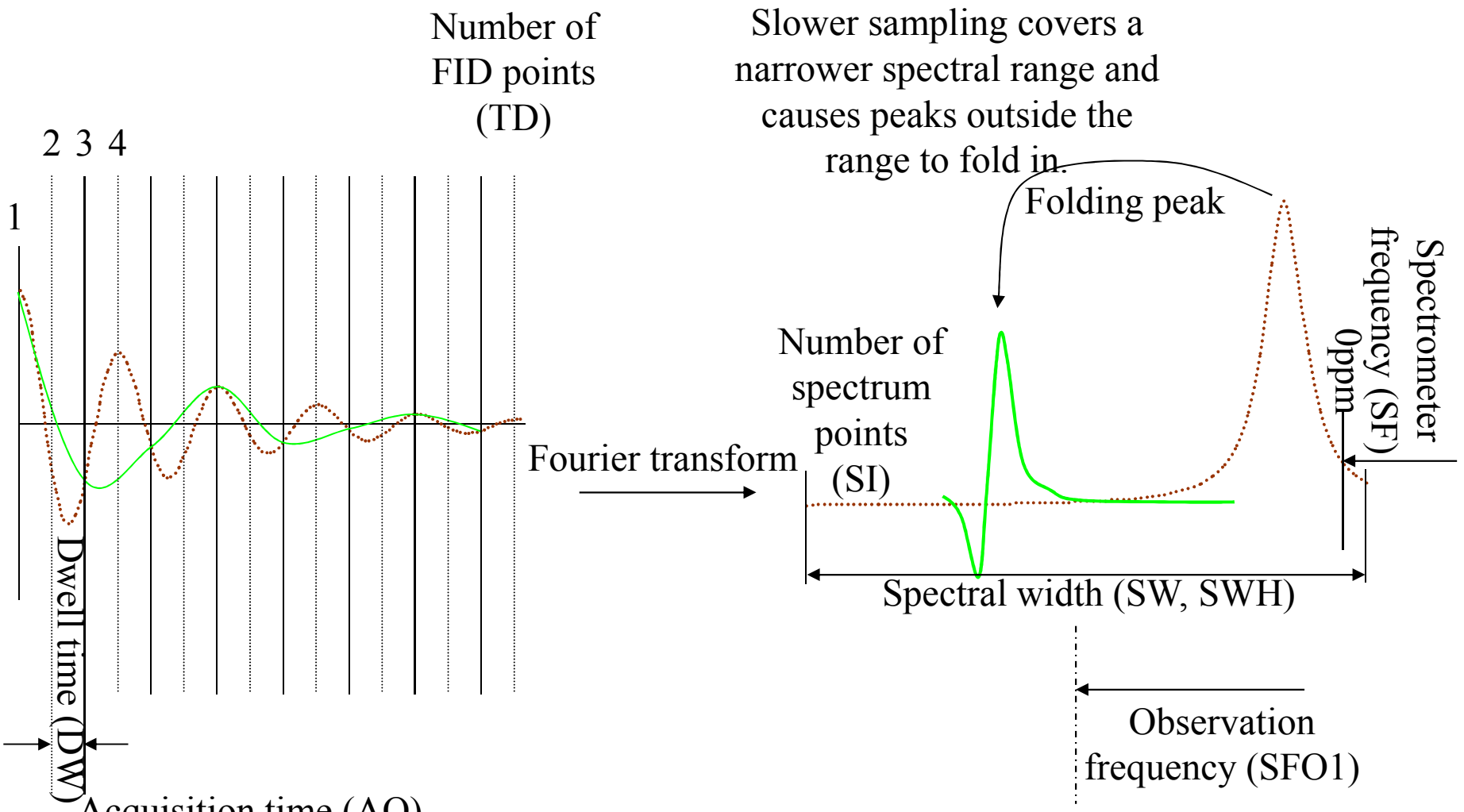
Acquisition of signal



Fourier transform



Sampling and folding

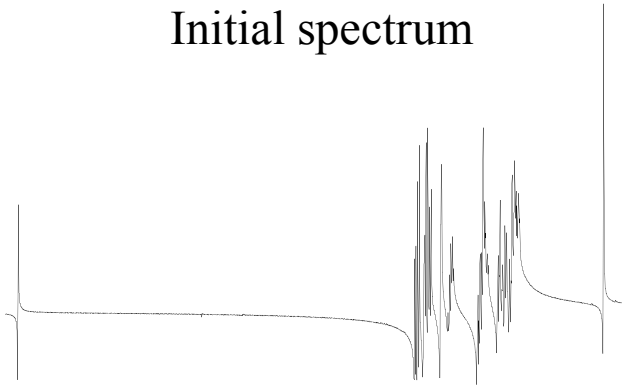


Parameters

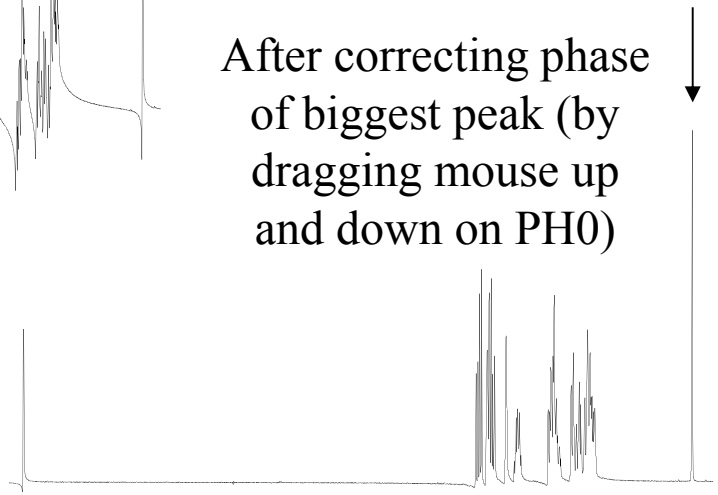
- (Acquisition time) AQ = DW * (TD – 1)
- (Spectral width in Hz) SWH = $^{1}/_{2DW}$
- (Spectral width in ppm) SW = SWH/SF
- Frequency at 0 ppm = SF
- (Intrinsic digital resn.) FIDRES = SWH/TD
- Digital resolution in Hz/point = SWH/SI
- (Observation frequency) SFO1 = BFO1 + O1

Phasing

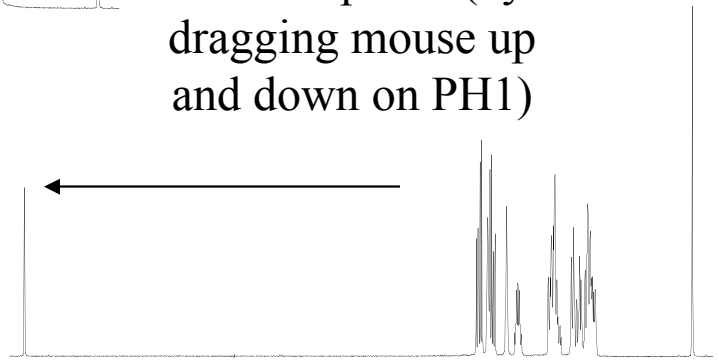
Initial spectrum



After correcting phase of biggest peak (by dragging mouse up and down on PH0)

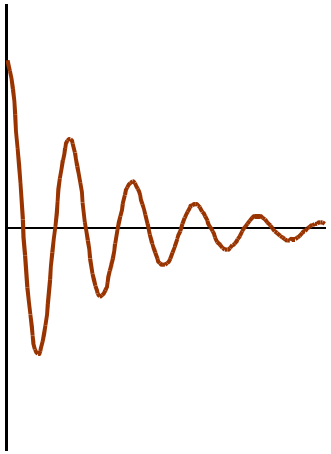


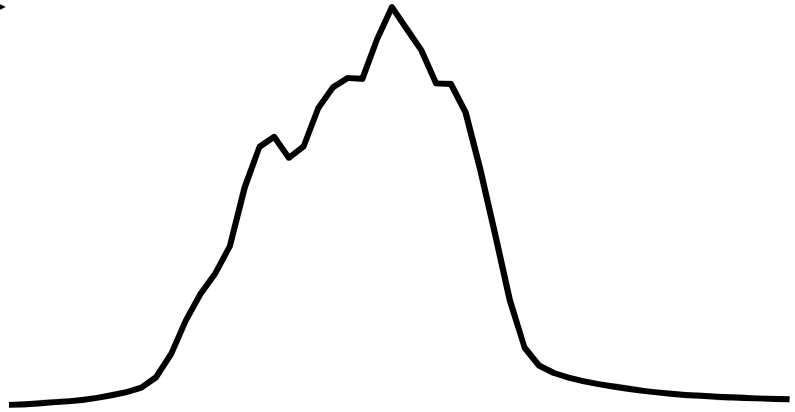
After correcting phase of other peaks (by dragging mouse up and down on PH1)

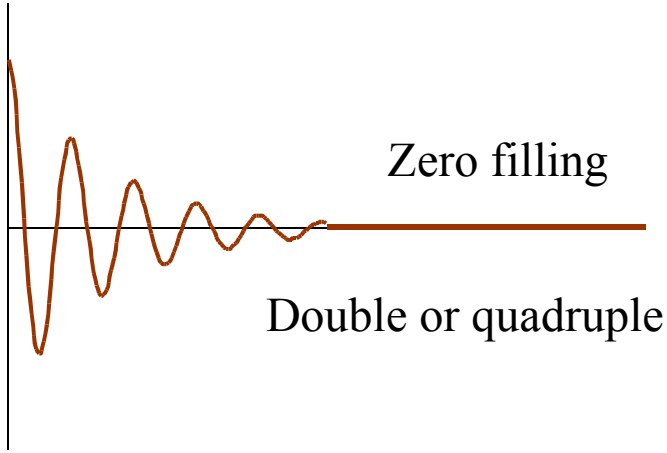


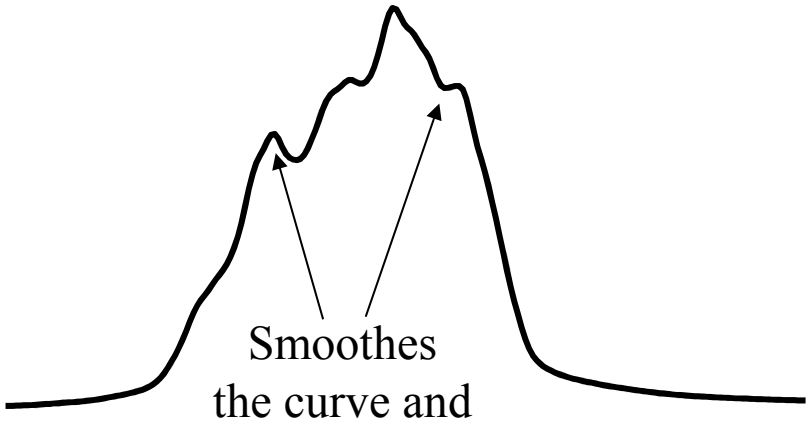
Zero filling

Fourier transform





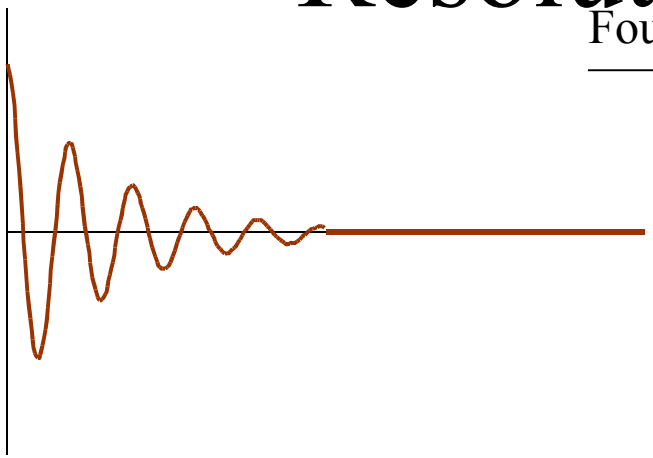


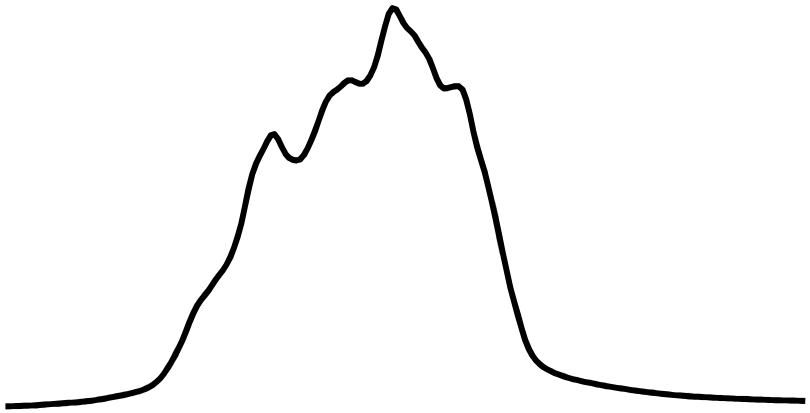


Smoothes the curve and slightly improves resolution

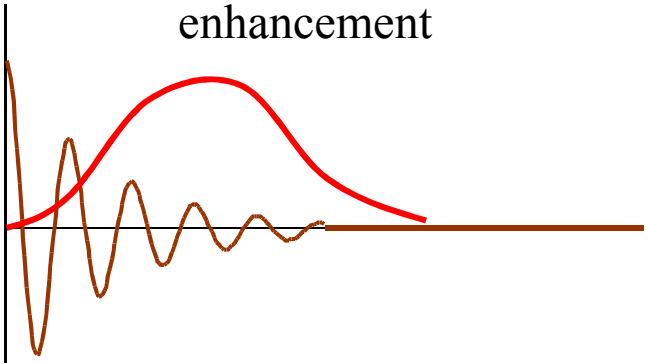
Resolution enhancement

Fourier transform





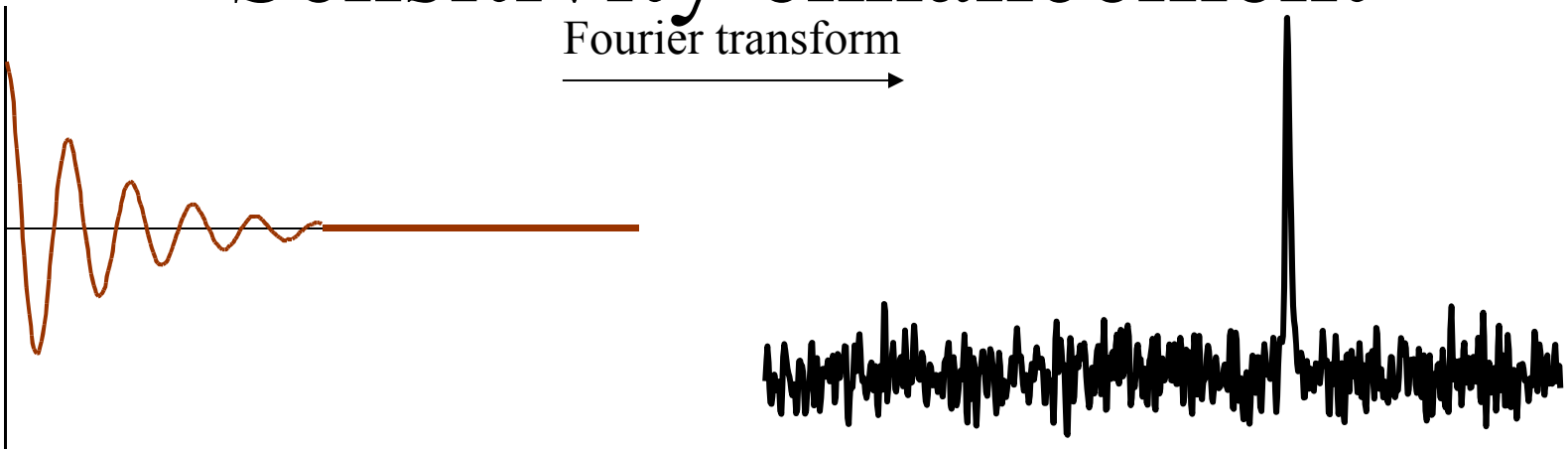
Gaussian resolution enhancement

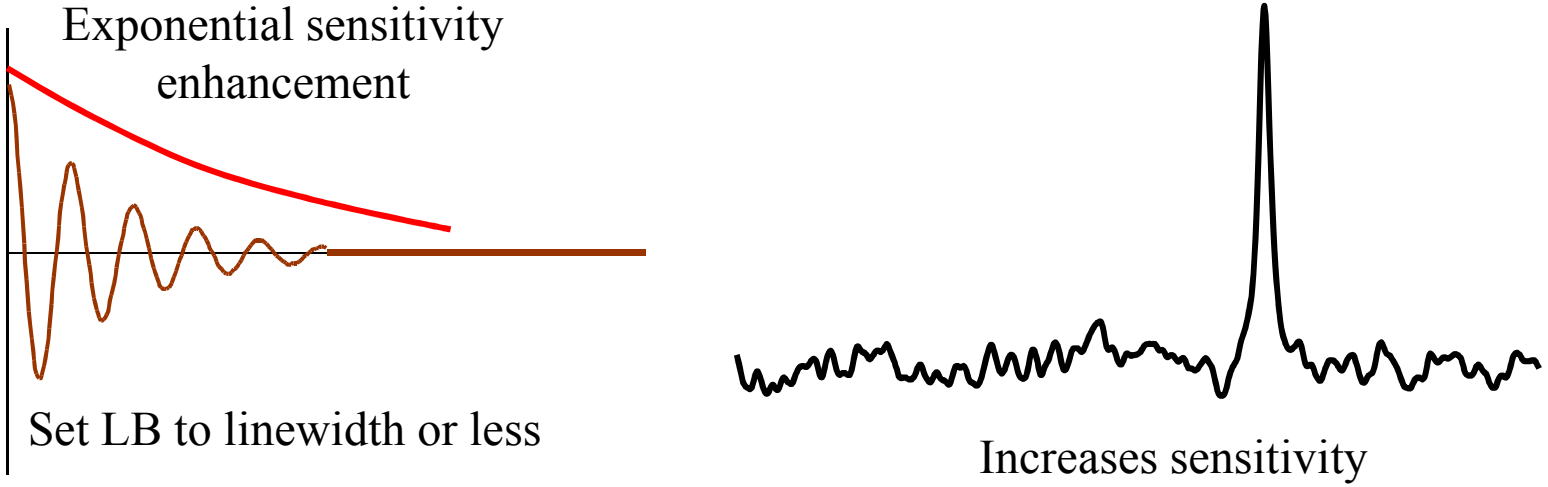


Set LB to negative linewidth and GB between 0 and 0.5 according to noise

Increases resolution but increases noise

Sensitivity enhancement

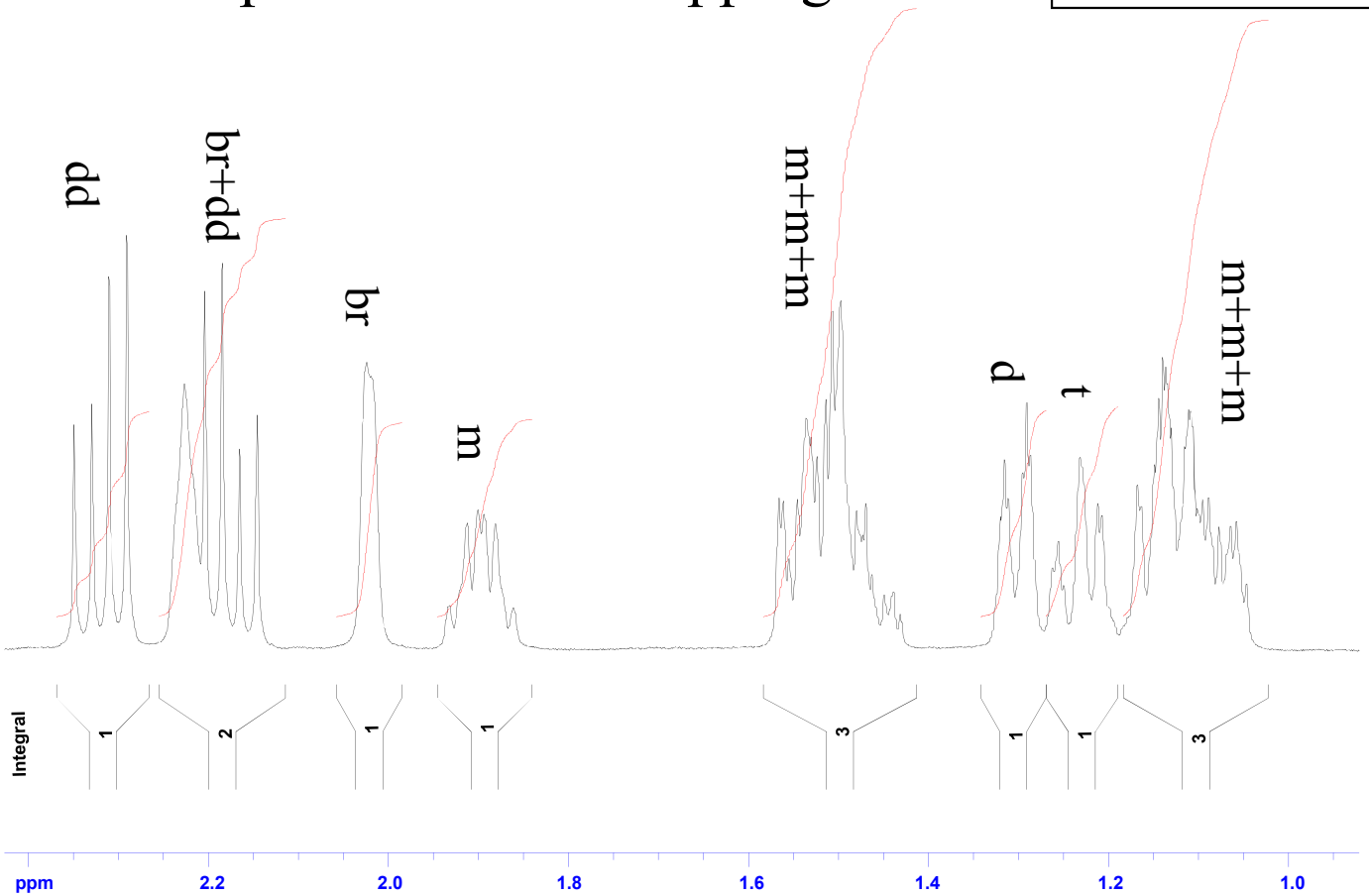




Integration

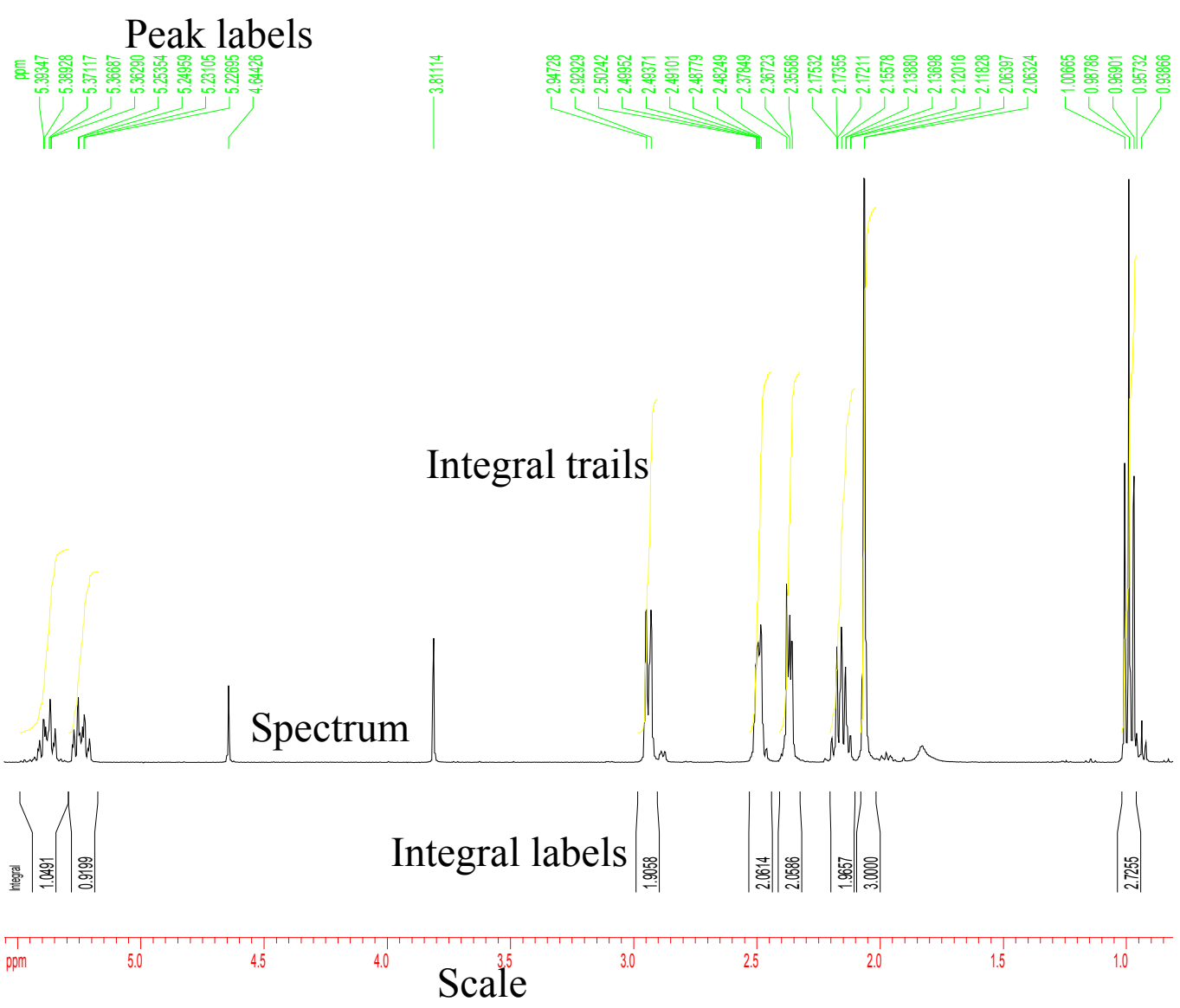
Key	
br	broad
d	doublet
dd	double doublet
m	mutiplet
t	triplet

- Flatten baseline with abs
- Integrate multiplets when separate and groups of multiplets when overlapping



Plot of spectrum

Title 1H-NMR
cis-Jasmone in CDCl3



Parameters

Current Data Parameters
NAME jasmone
EXPNO 1
PROCNO 1

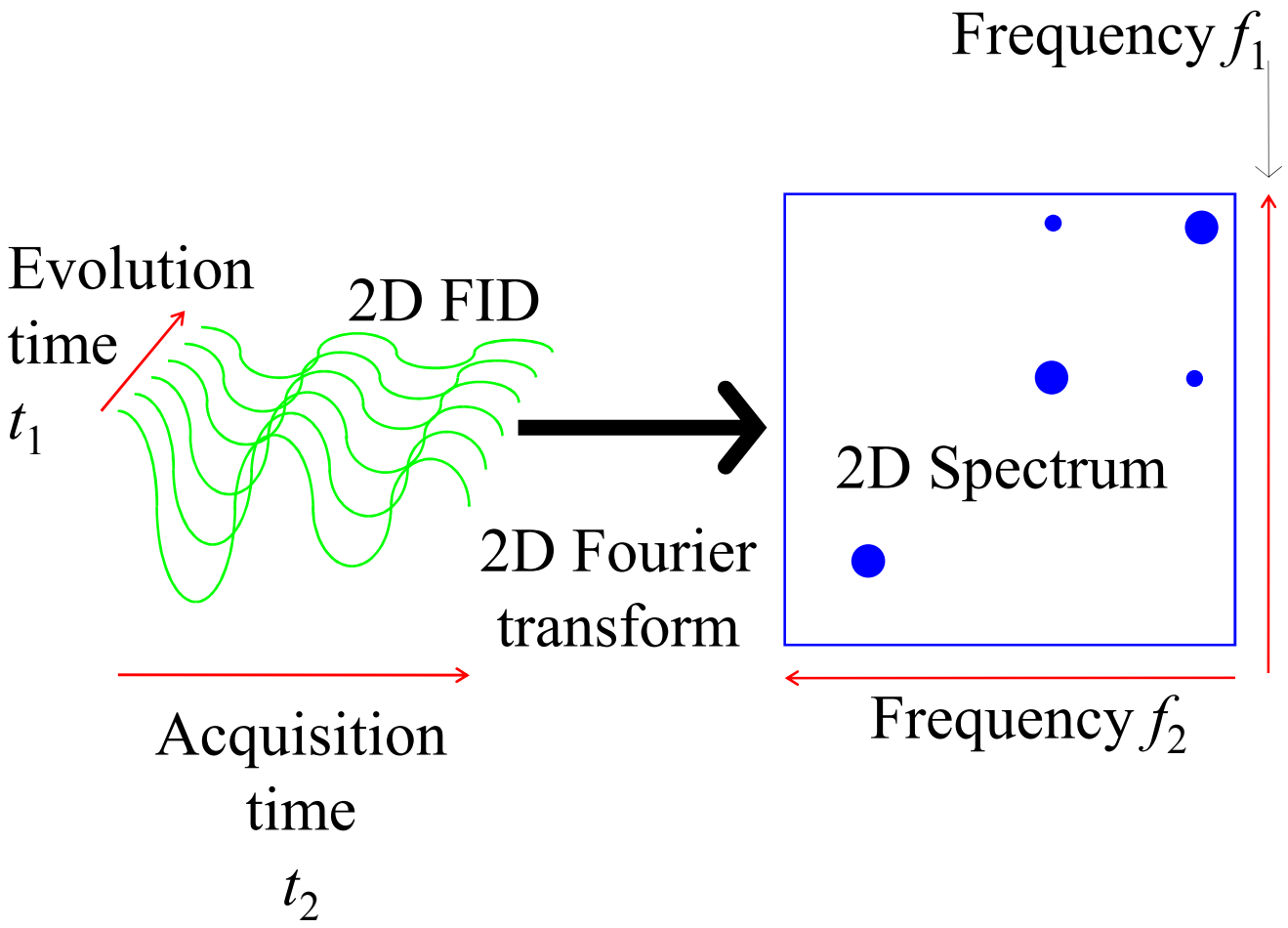
F2 - Acquisition Parameters
Date_ 20021230
Time 11.01
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 90.5
DW 104.000 usec
DE 10.00 usec
TE 300.0 K
D1 2.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 6.10 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 65536
SF 400.1300048 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 5.554 ppm
F1 2222.22 Hz
F2P 0.811 ppm
F2 324.45 Hz
PPMCM 0.23714 ppm/cm
HZCM 94.88867 Hz/cm

2D

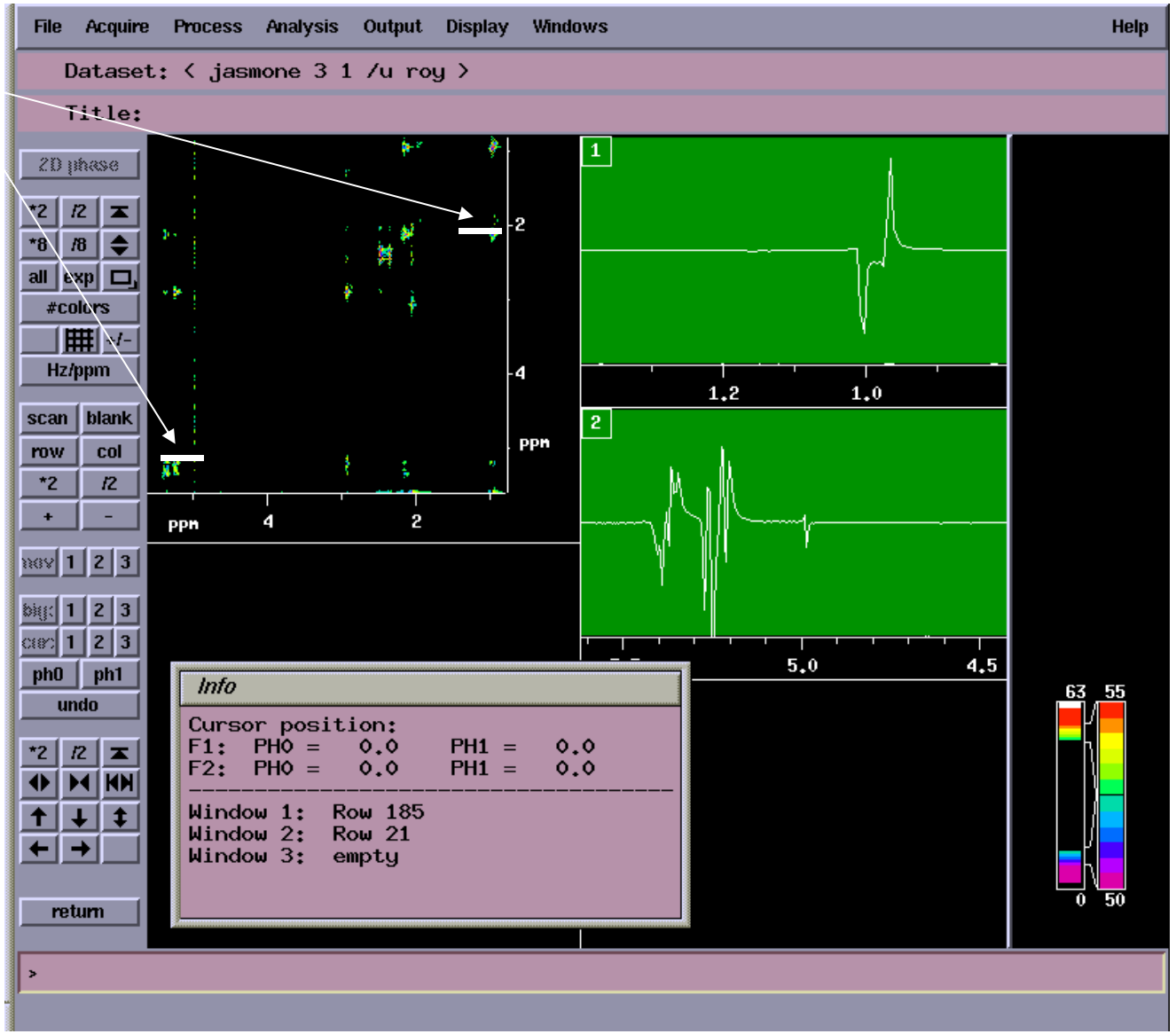

Frequency f_1
Evolution time t_1
2D FID
2D Fourier transform
2D Spectrum
Acquisition time t_2
Frequency f_2

2D Parameters

- (Evolution time) IN0 * (1 TD – 1)
- (Spectral width in Hz) 1 SWH = $^{1}/_{2\text{INO} * \text{ND0}}$
- (Spectral width in ppm) 1 SW = 1 SWH/1 SF
- Frequency at 0 ppm = 1 SF
- (Intrinsic digital resn.) 1 FIDRES = 1 SWH/1 TD
- Digital resolution in Hz/point = 1 SWH/1 SI
- (Observation frequency)1 SFO1(= SFO1 or SFO2)

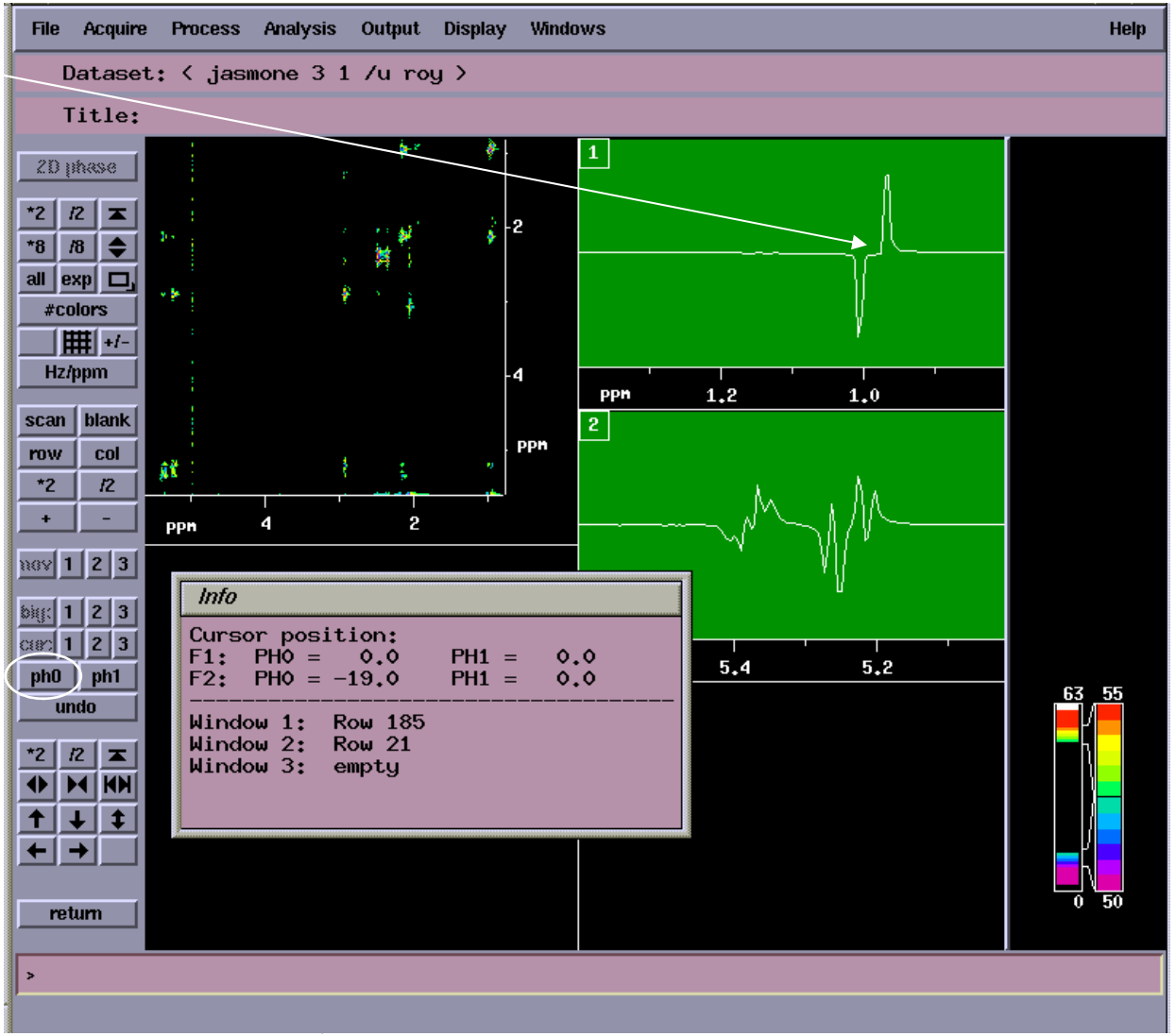
2D Phasing for COSY

Choose two rows



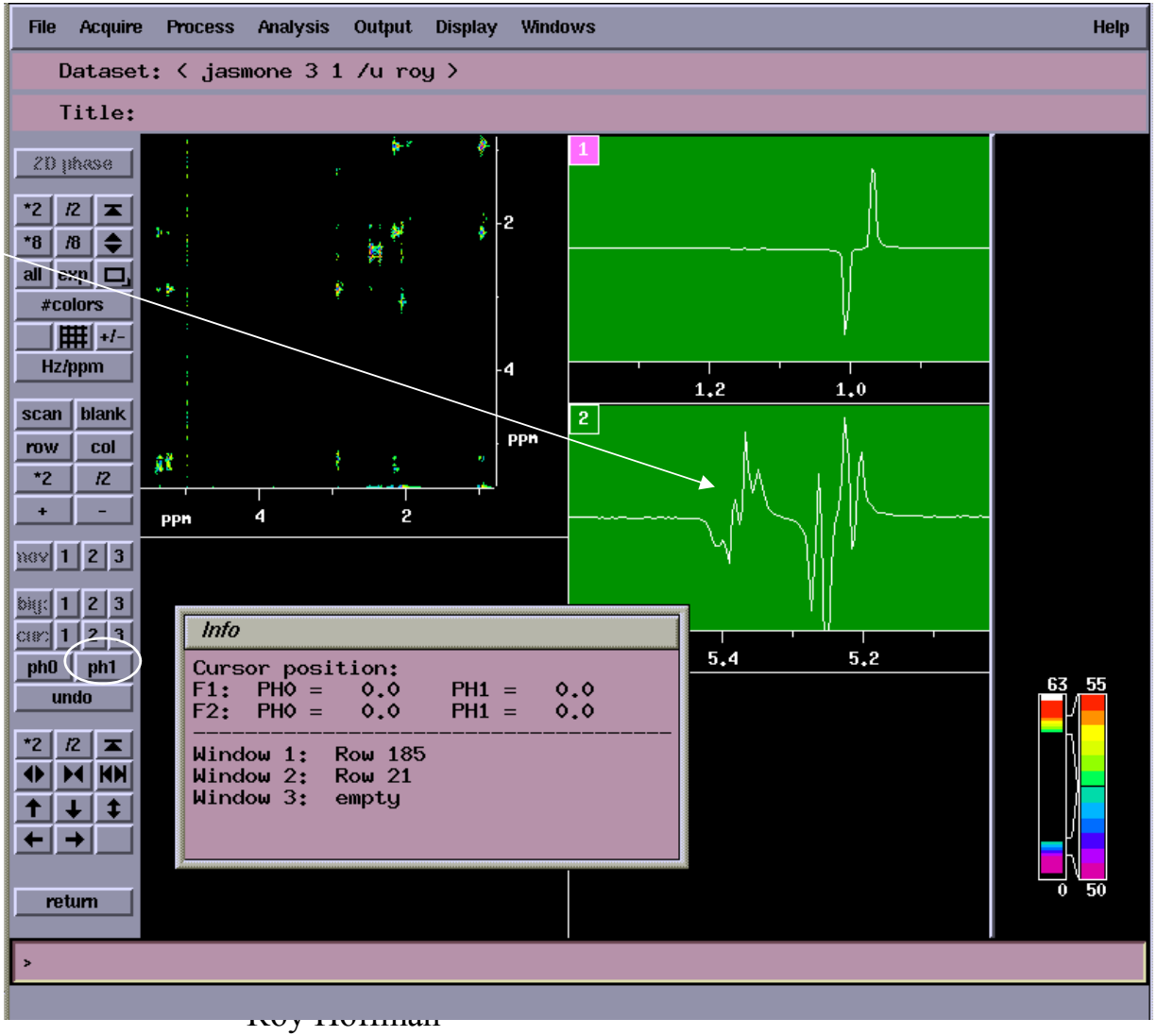
2D phasing for COSY

Phase one peak to anti-phase by dragging the mouse on ph0



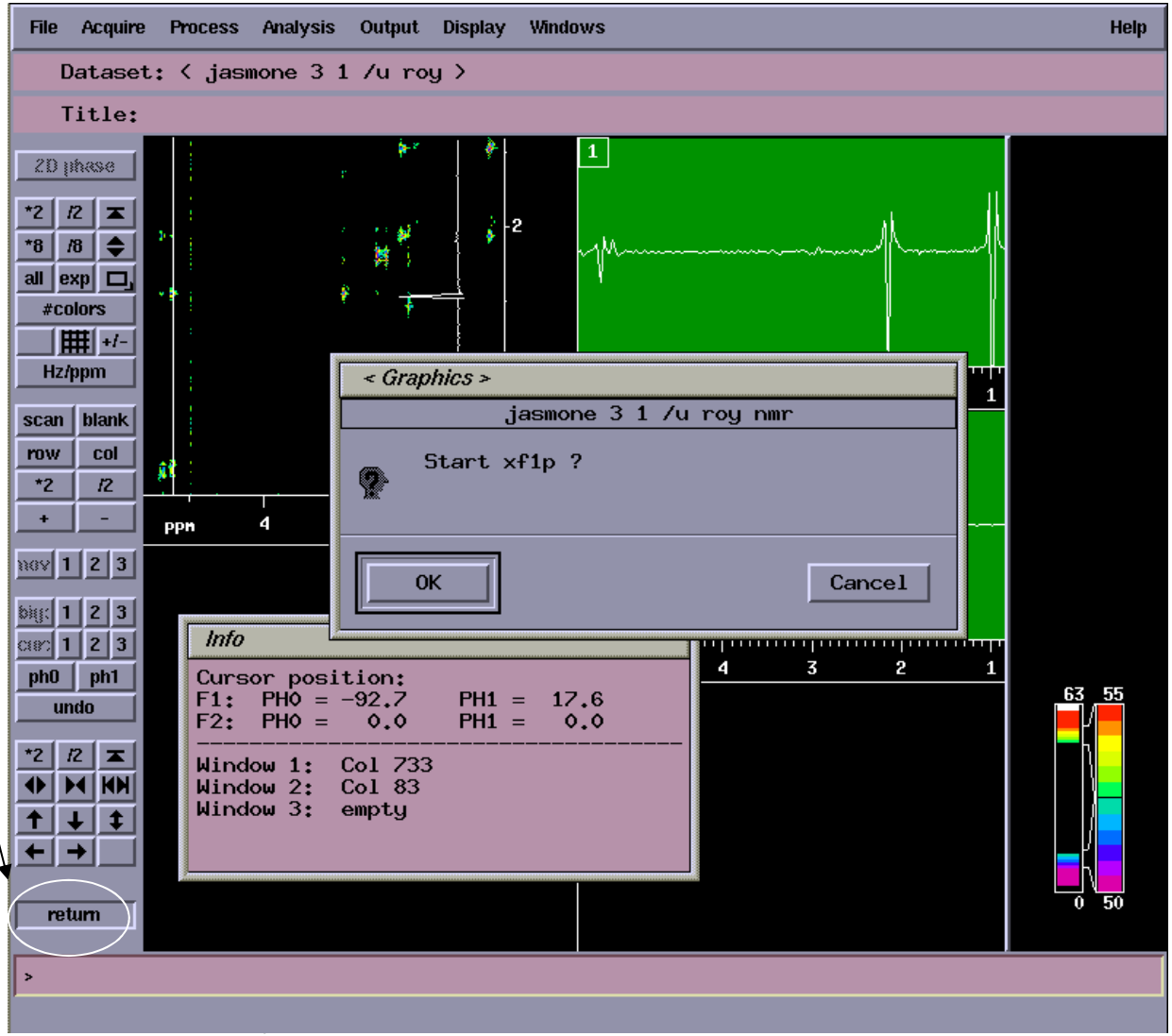
2D Phasing for COSY

Phase the other peaks to anti-phase by dragging the mouse on ph1



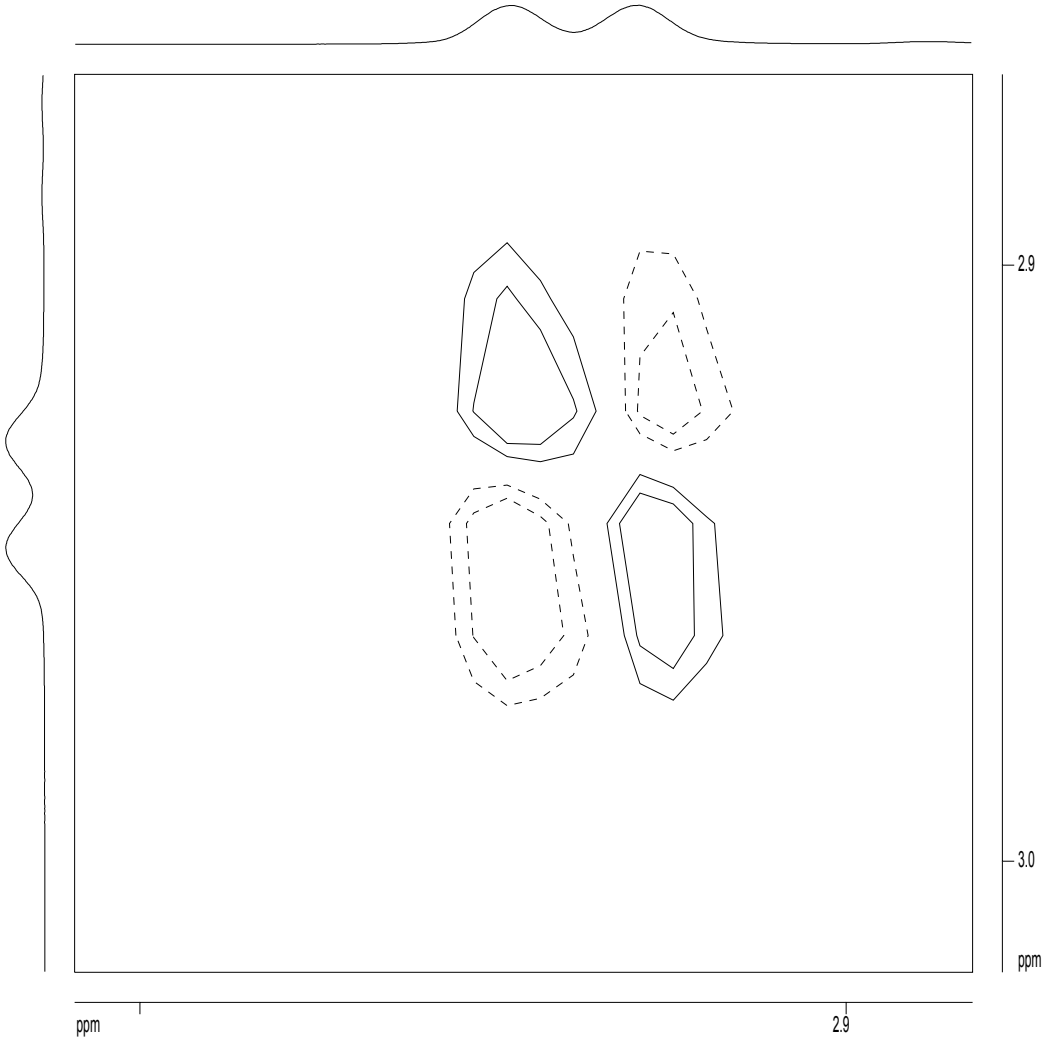
2D Phasing for COSY

Repeat for columns
Click on return to store phase



Antiphase doublet

COSY-DQF
cis-Jasmone in CDCl3



Current Data Parameters
NAME jasmone
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021230
Time 11.19
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG cosygsmftp2
TD 2048
SOLVENT DMSO
NS 1
DS 4
SWH 1929.012 Hz
FIDRES 0.941901 Hz
AQ 0.5308916 sec
RG 2048
DW 259.200 usec
DE 10.00 usec
TE 300.0 K
d0 0.00000300 sec
D1 2.00000000 sec
d13 0.00000300 sec
D16 0.00020000 sec
d20 0.00120300 sec
IN0 0.00025920 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 6.10 usec
p2 11.40 usec
PL1 -6.00 dB
SFO1 400.1312880 MHz

========== GRADIENT CHANNEL =====
P16 1000.00 usec

F1 - Acquisition parameters
ND0 2
TD 256
SFO1 400.1313 MHz
FIDRES 7.535204 Hz
SW 4.821 ppm

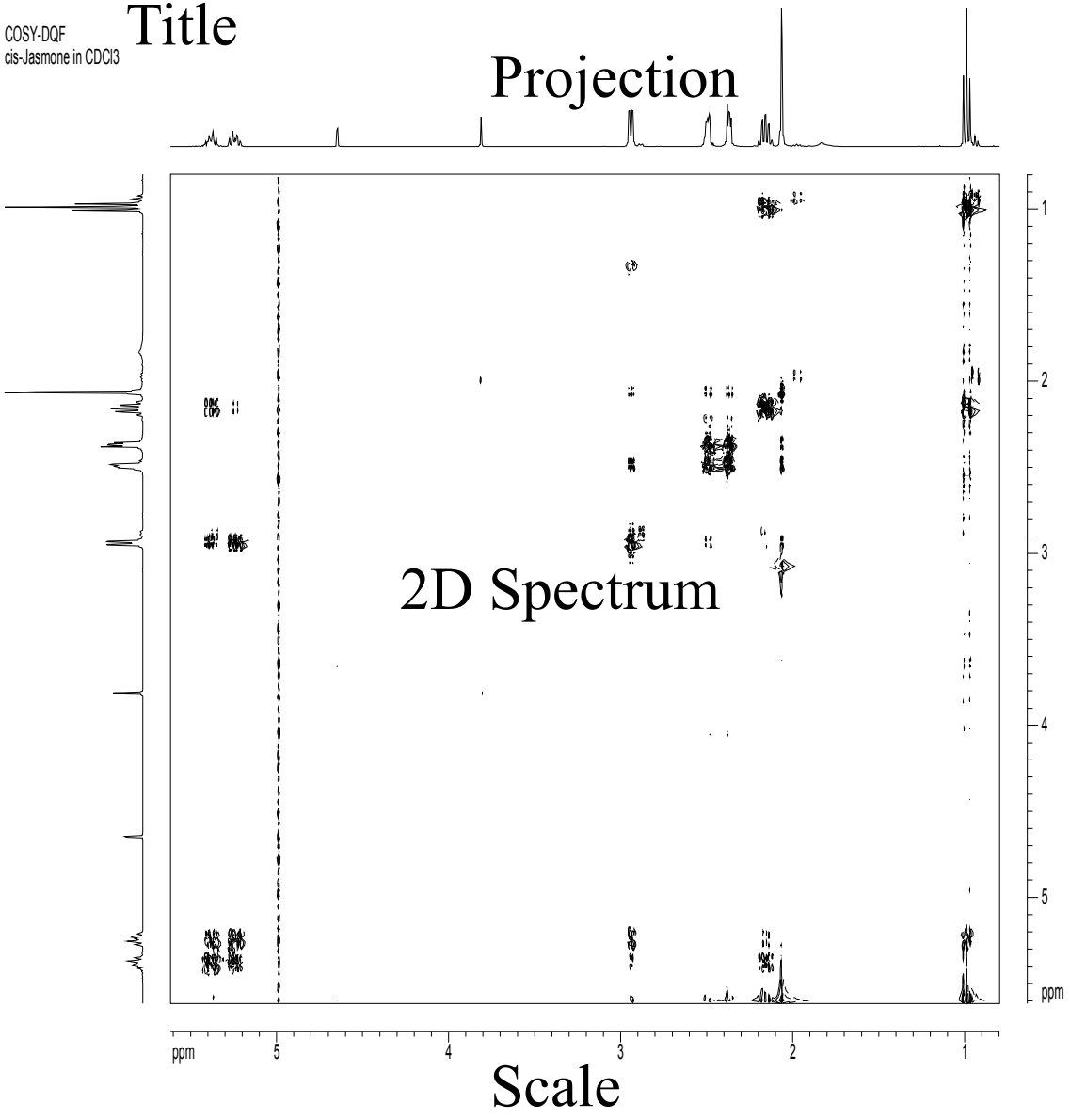
F2 - Processing parameters
SI 1024
SF 400.1300048 MHz
WDW SINE
SSB 2

2D plot

COSY-DQF
cis-Jasmone in CDCl3

Title

Projection



2D Spectrum

Scale

Parameters

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PROCNO 1

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Time 11.19
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG cosygsmftp2
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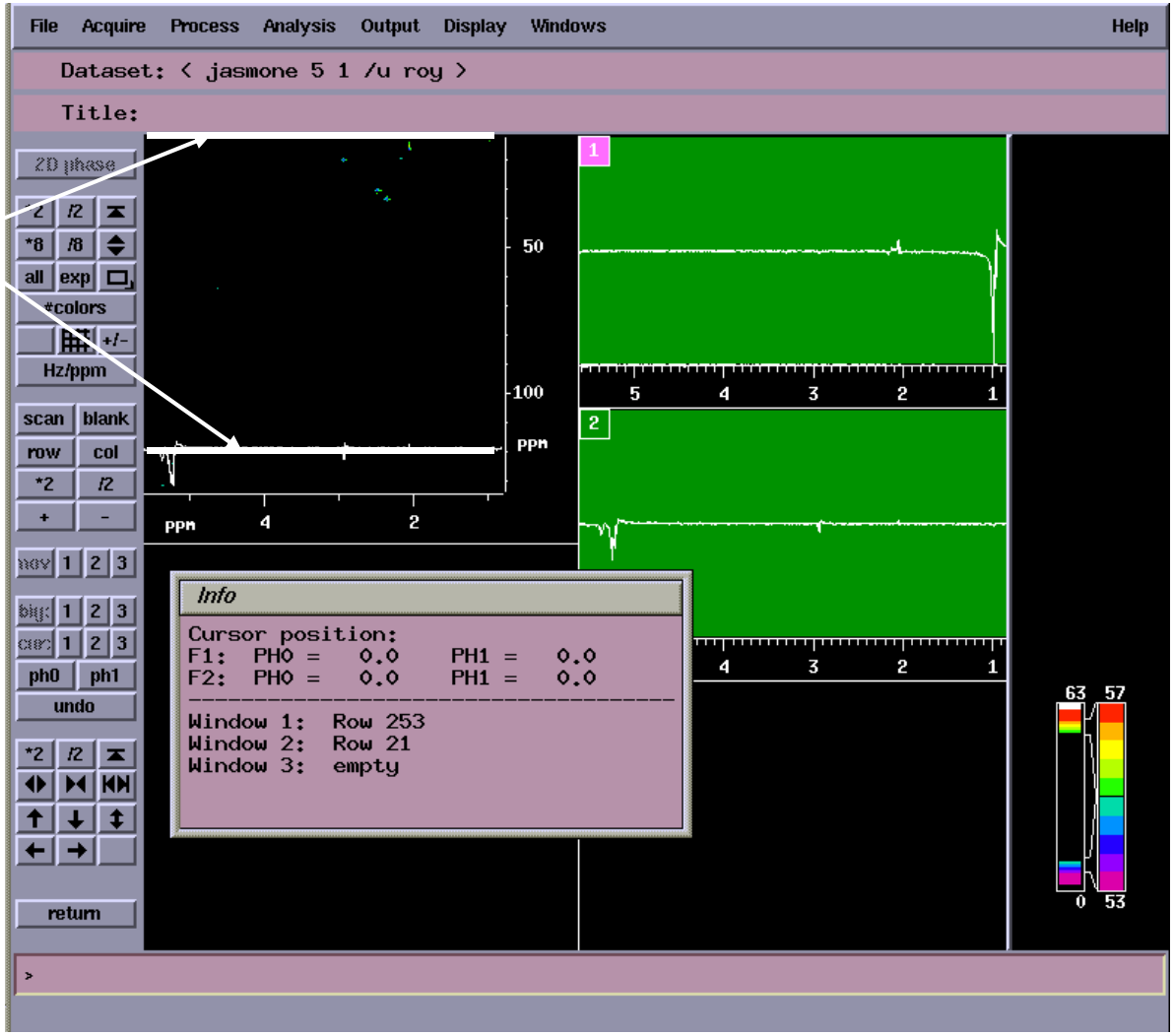
SI 1024
SF 400.1300048 MHz
WDW SINE
SSB 2
LB 0.00 Hz
GB 0

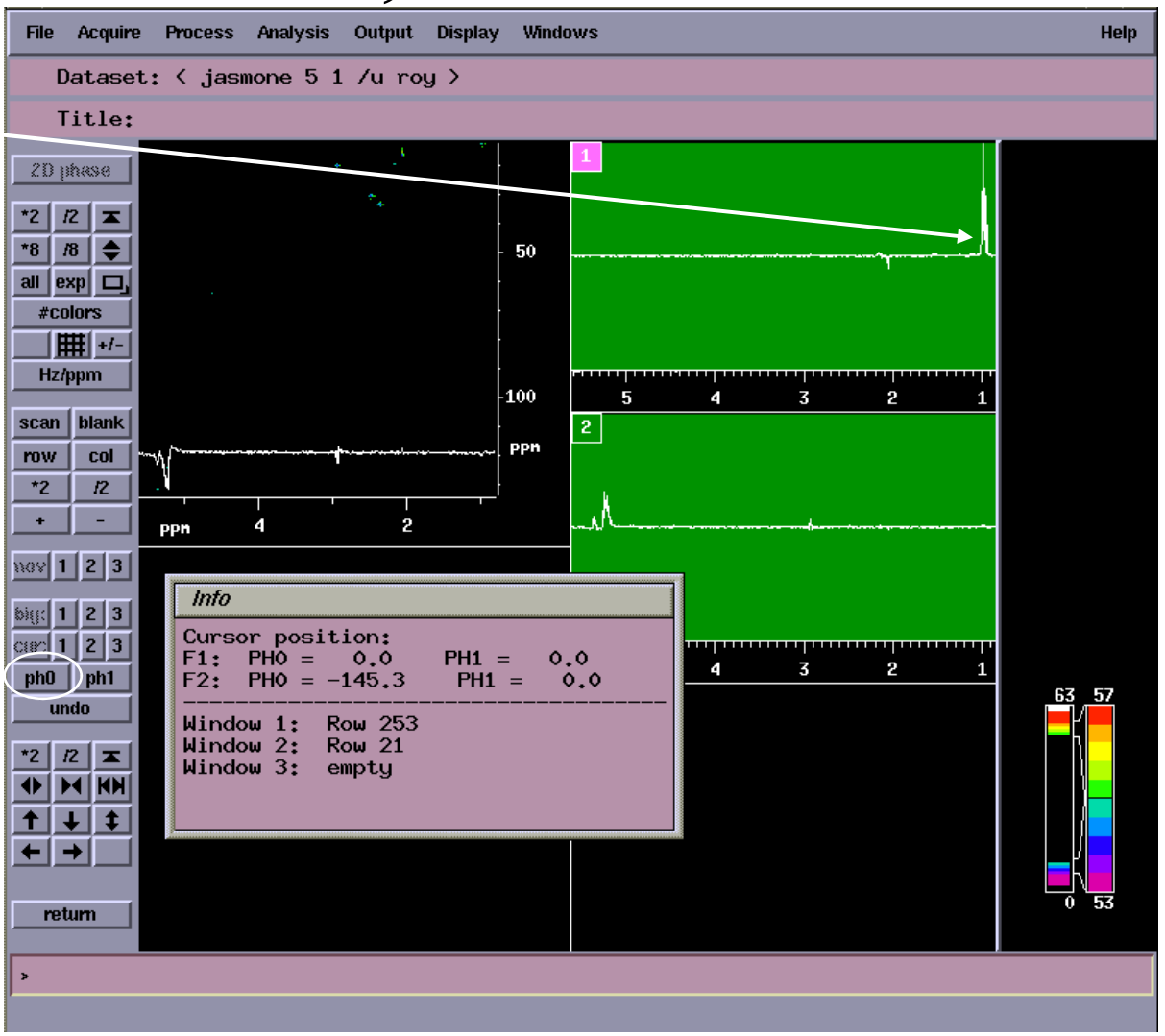
2D phasing – HSQC, NOESY, ROESY, TOCSY

Choose two rows



2D phasing – HSQC, NOESY, ROESY, TOCSY

Phase one peak by dragging the mouse on ph0



2D phasing – HSQC, NOESY, ROESY, TOCSY

Phase the other peaks by dragging the mouse on ph1

Repeat for columns
Click on return to store phase