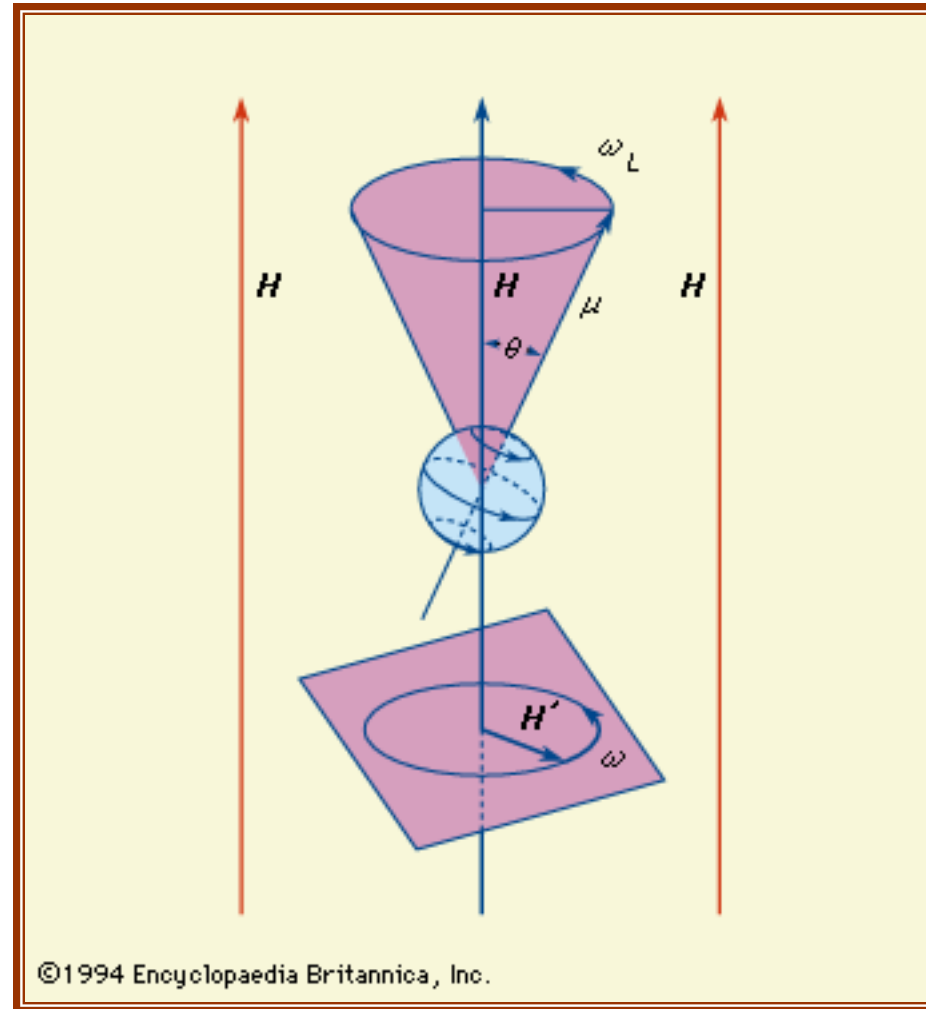


NMR

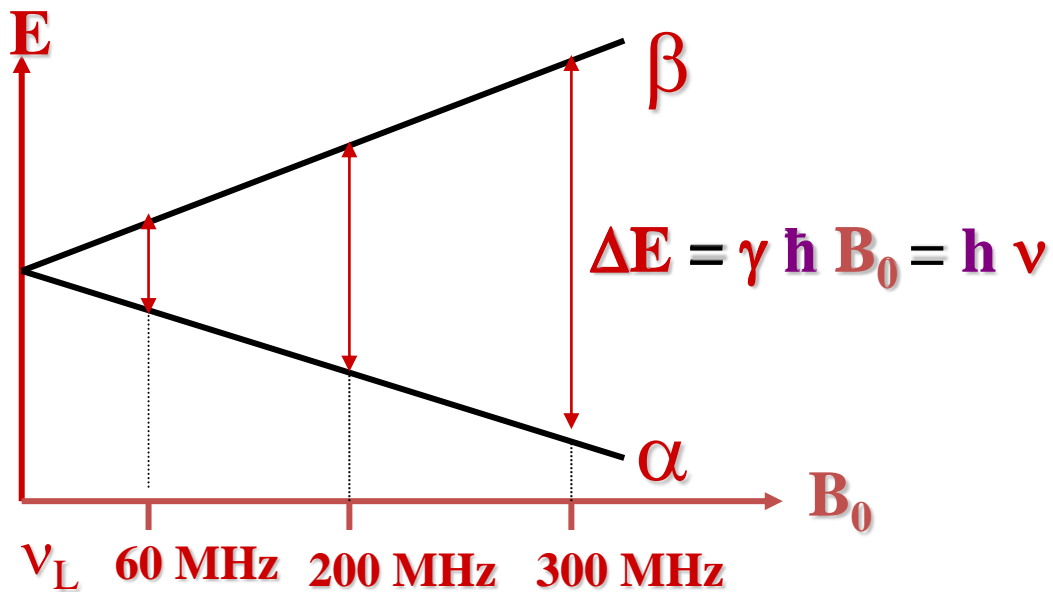
Nuclear
Magnetic
Resonance



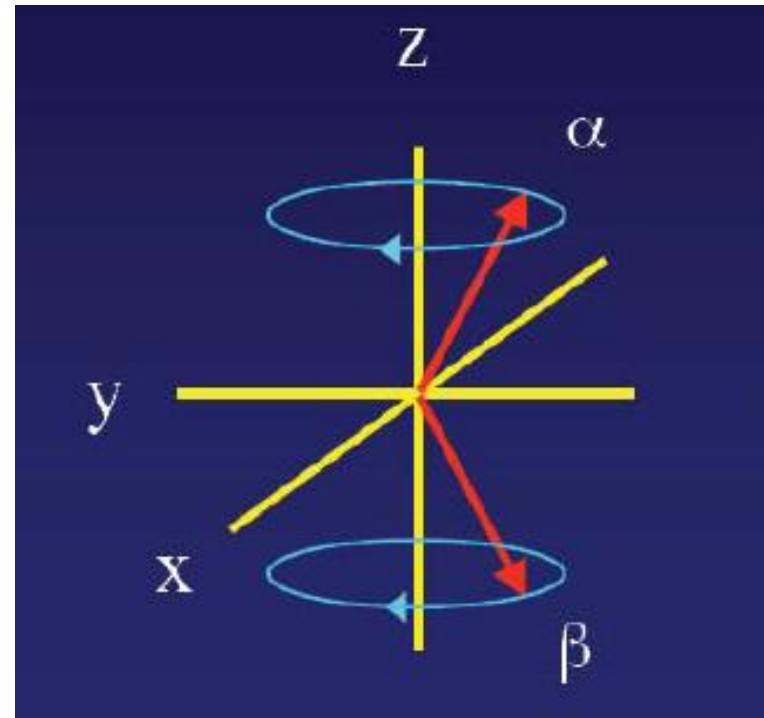
Precession



Precession and energy (spin 1/2)



$$\nu = \gamma B_0 / 2\pi$$



NMR spectrometer

Computer



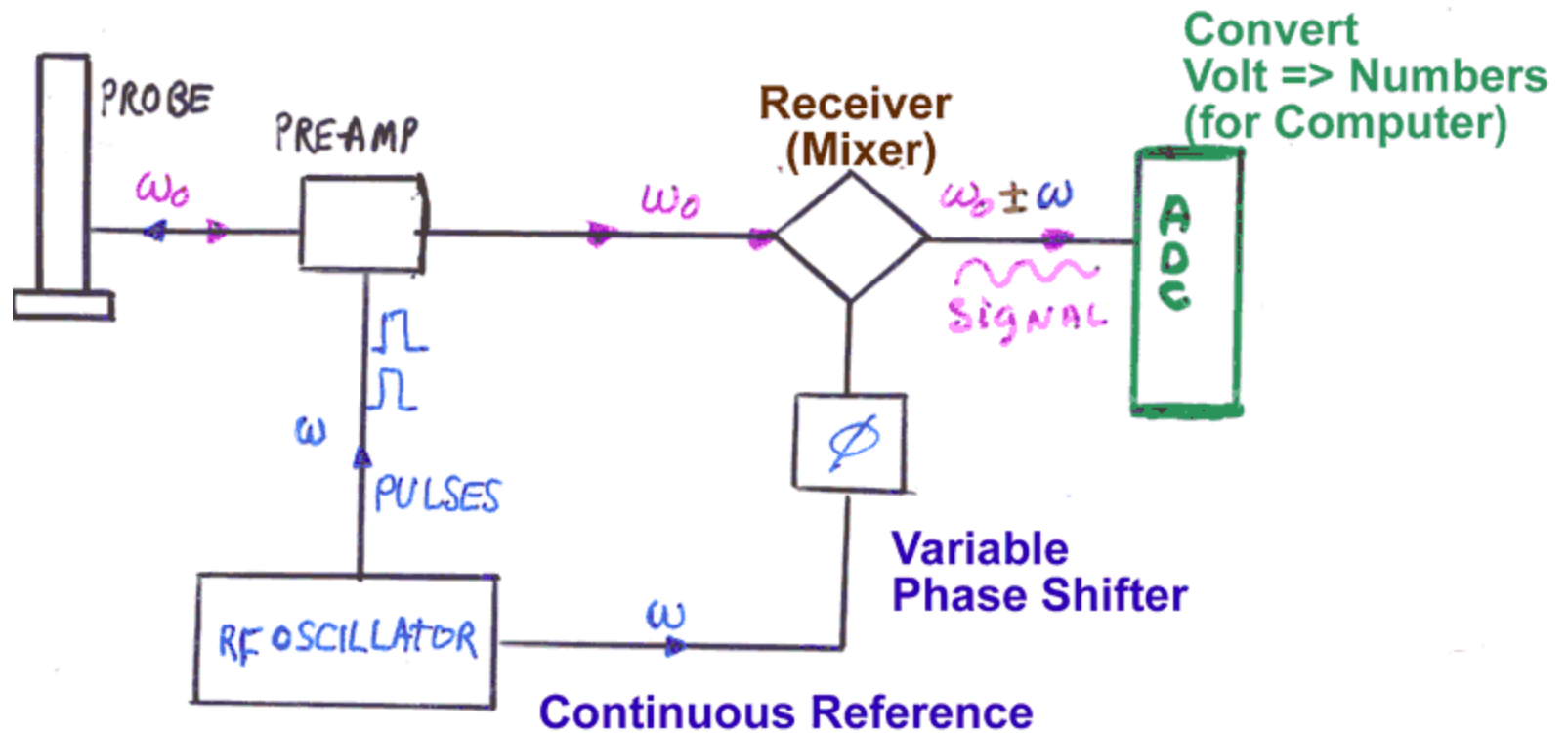
Console



Magnet



Pulse NMR spectrometer



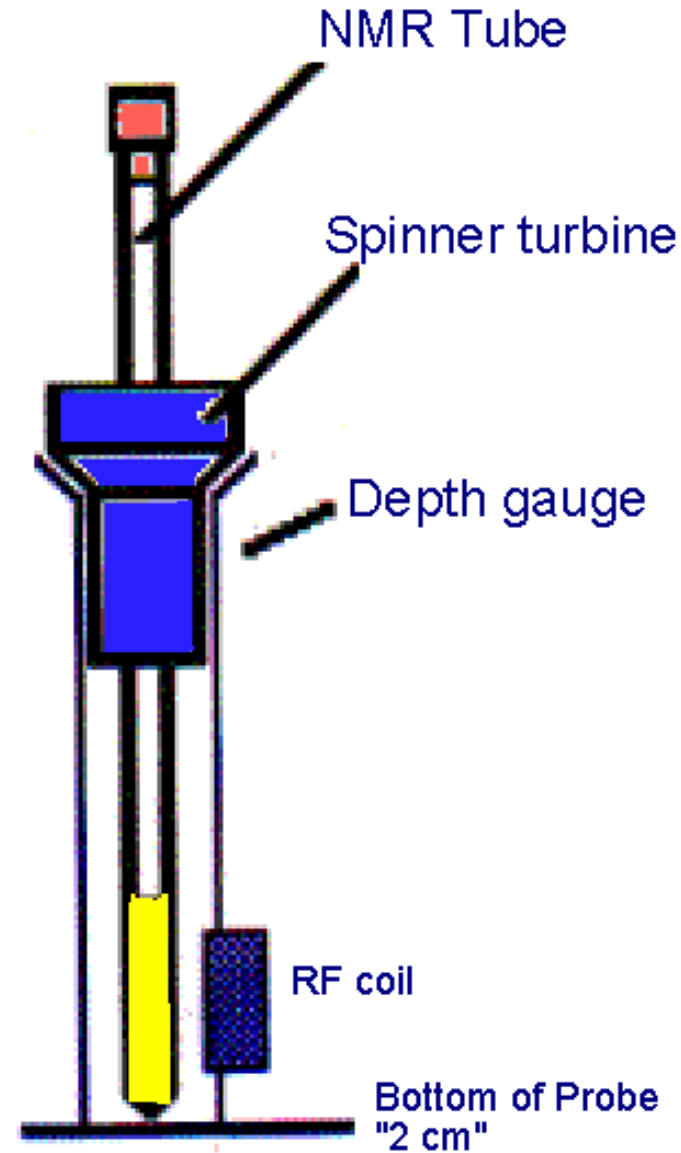
- * Frequency Synthesizer: Produce RF at ω frequency (MHz)
 - Signal divided
 - Ref. Signal (Receiver)
 - High gain Amplifier -> Pulse

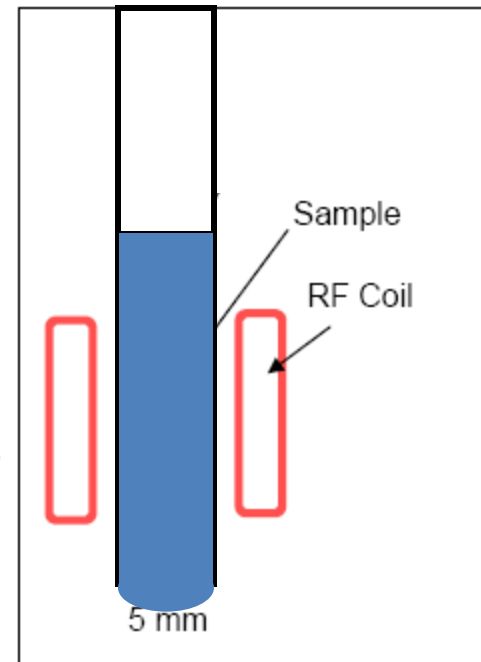
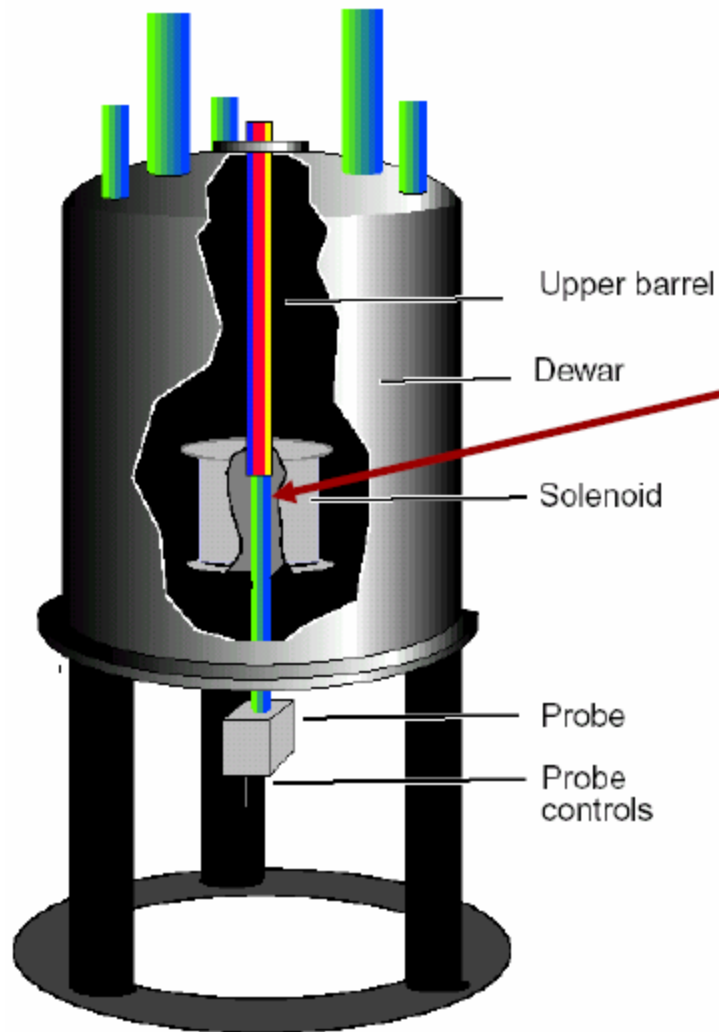
- * Mixer (Demodulator) yields signal (Rotating Frame)

$$|\omega_0 \pm \omega|$$

Sample preparation

- Sample preparation:
 - H1: 5-10 mg
 - C13: 30-50 mg
- In deuterated solvent:
~ 0.7 ml (4 cm)
- Insert sample :
 - ej : eject sample
 - ij : inject sample

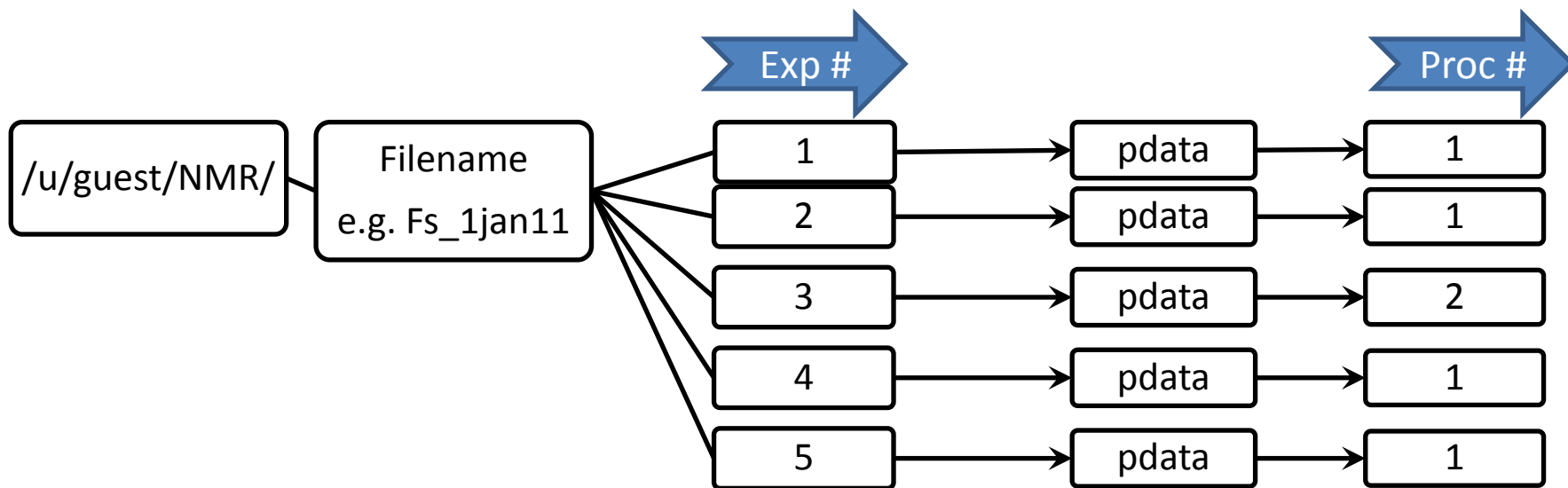




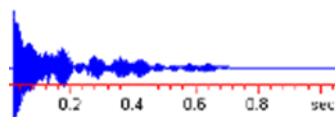
and:

- Radio Transmitter (200-900 MHz)
- Radio Receiver
- Digitizer (A/D converter)
- Pulse Programmer
- Computer

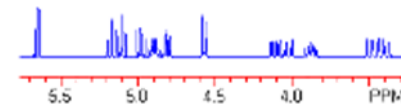
Define a filename



Command:
re (exp#)
Read exp



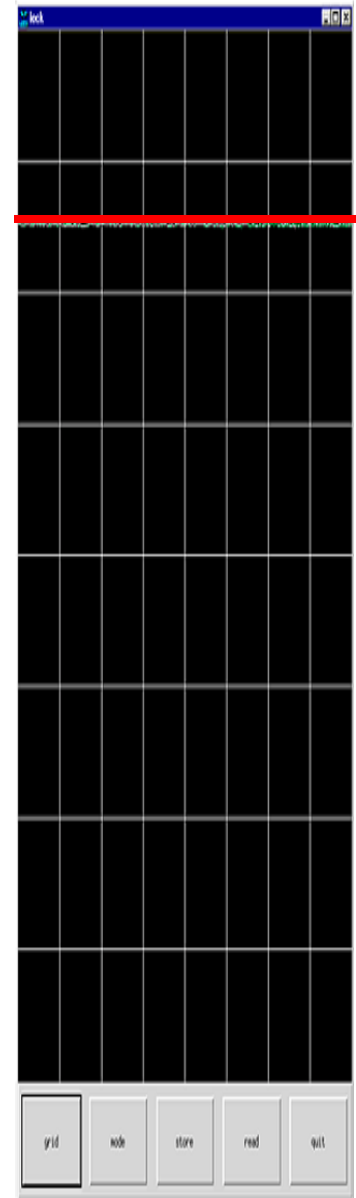
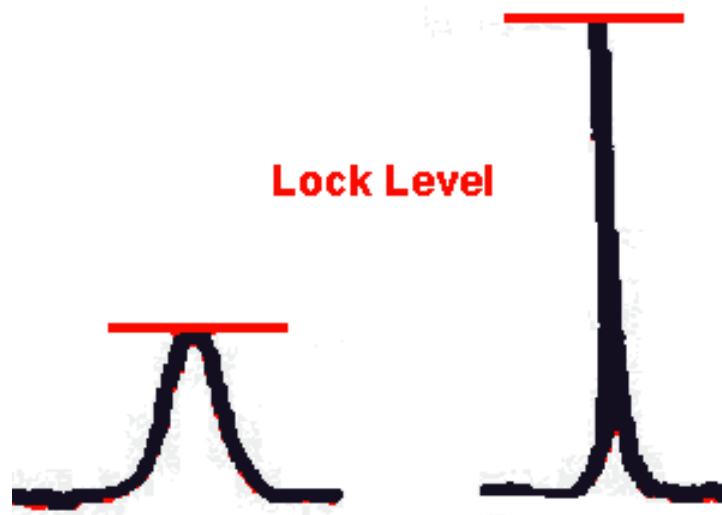
FID,
Parameters
(related to time)



Spectra
Parameters
(related to
Frequency)

Lock on the Deuterated solvent

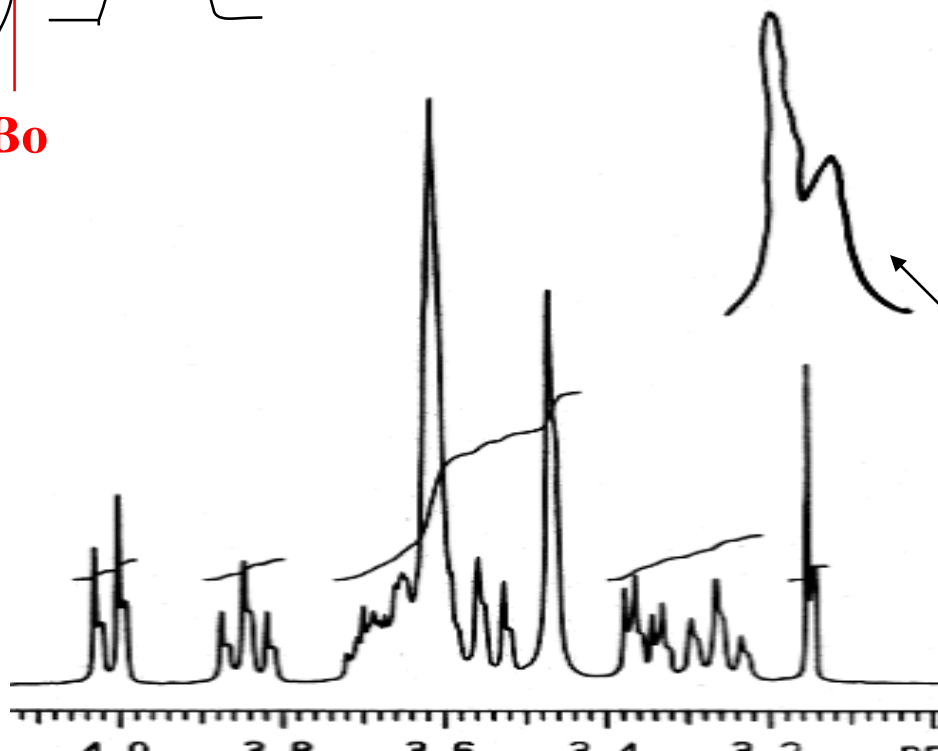
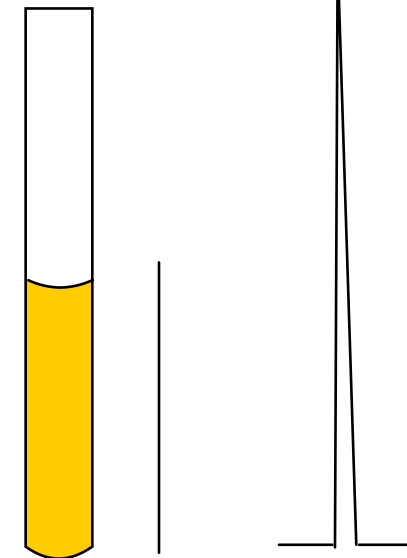
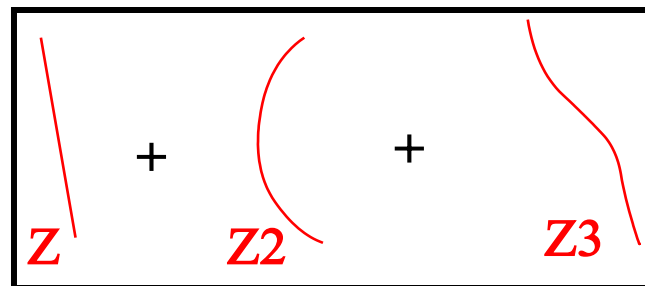
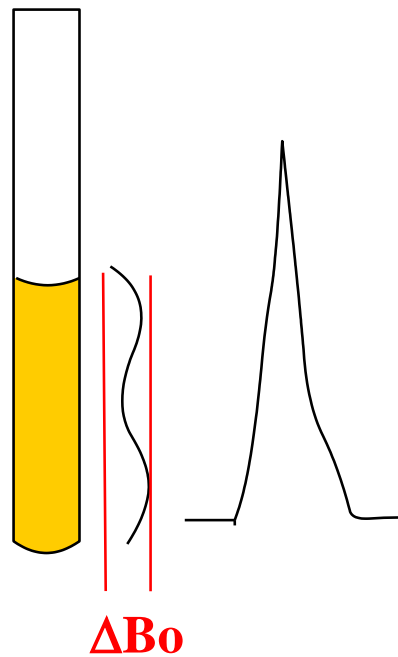
- Choose your deuterated solvent
 - When the lock is engage it looks like →
 - You are then monitoring the height of the solvent peak



Bad shimming

Good shimming

Shim: optimize field



Bad Z2

Choose parameters

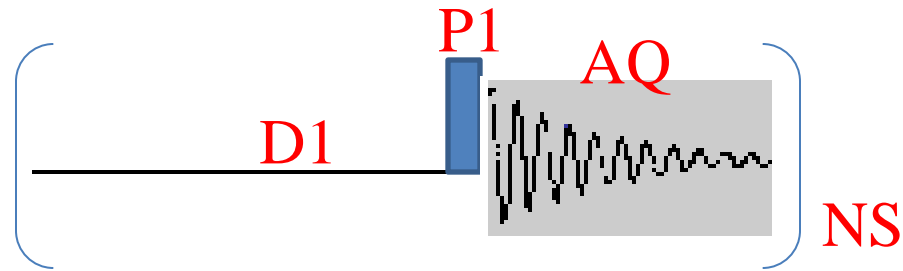
1D-NMR

- 1d_proton
- 1d_carbon
- 1d_carbon-invigated
- 1d_DEPT-135
- 1D-jmod (APT)
- 1d_phosphorous
-

2D-NMR

- 2d_COSY
- 2d_HSQC
- 2d_HMQC
- 2d_HMBC
- 2d_NOESY
- 2d_ROESY
-

Standard experiment and parameters



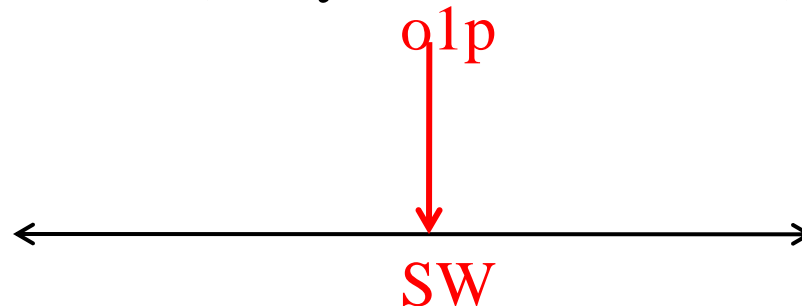
Sequence :

zg : Standard sequence : no decoupling

zgdc : Decoupler ON during D1 and AQ

zgig : Decoupler ON during AQ – decoupled spectra,
Off during D1 to get rid of NOE

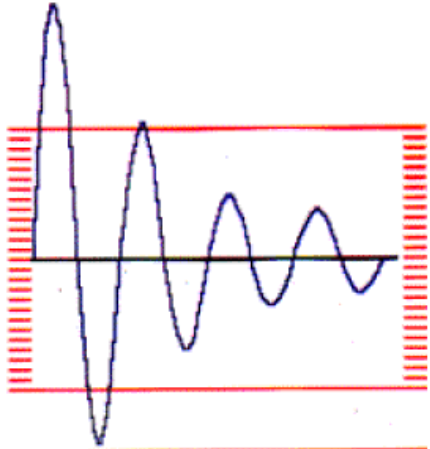
→ quantitative C13 (delay must be 5-10 T1)



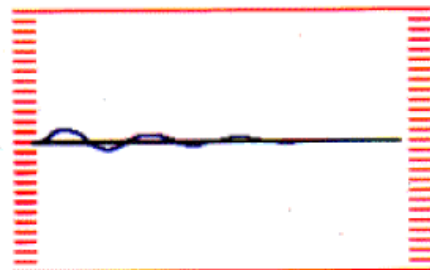
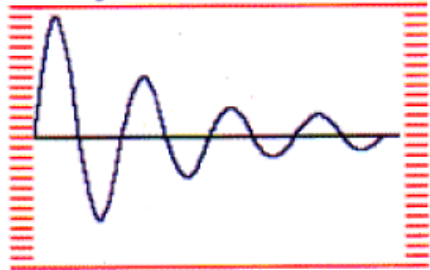
Acquire the data

RGA:

Adjust receiver gain



Too high!



Too low!

ZG

Zero the memory

Go : acquire the data

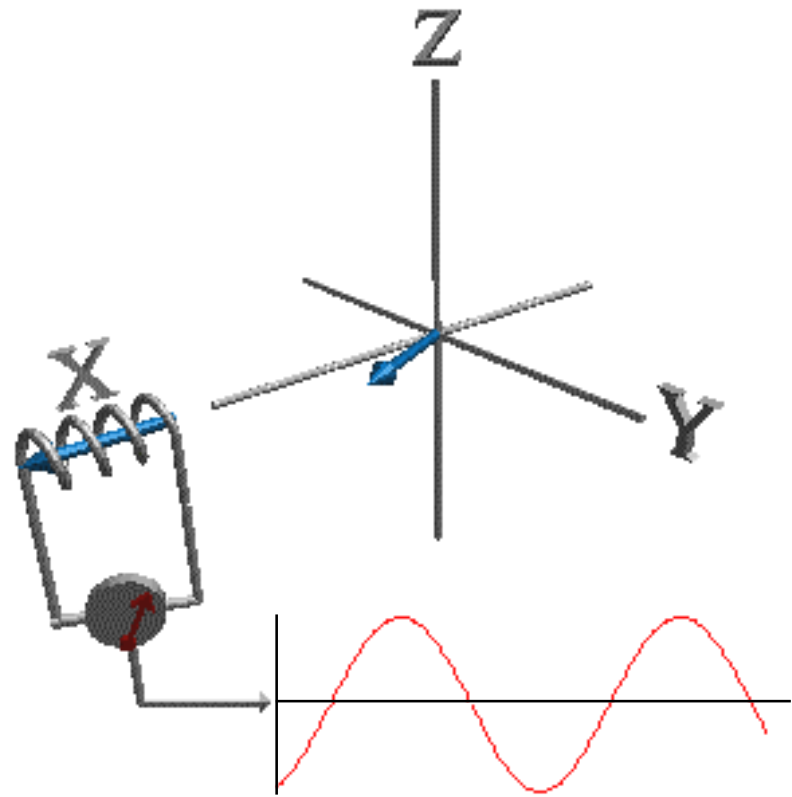
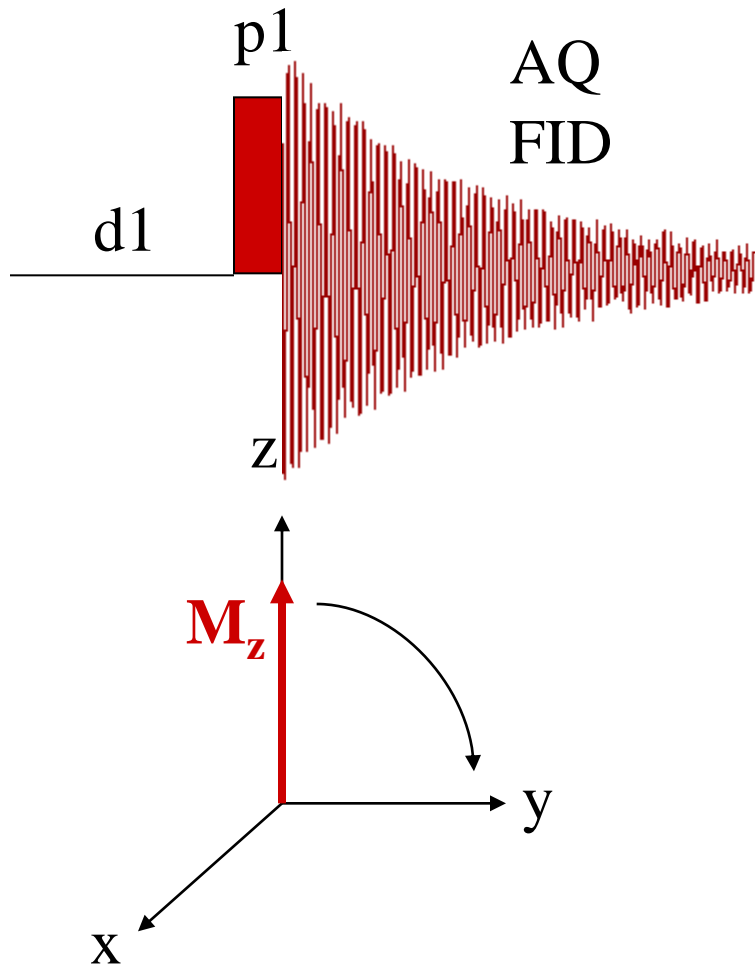
Useful commands:

tr : transfer the data while acquisition is running

go : if the acquisition is finished, this command will continue acquiring

Sampling the NMR signal: FID

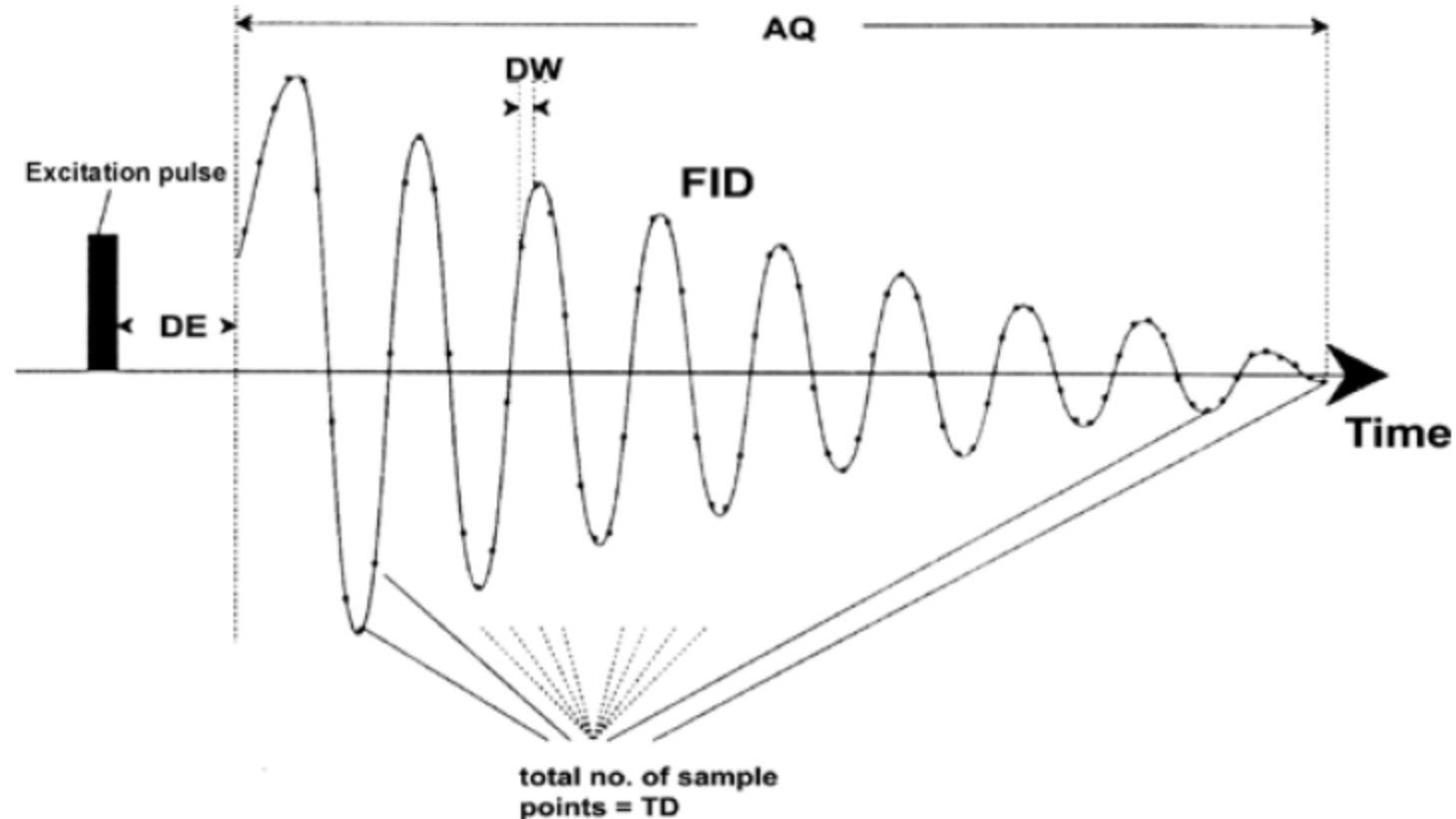
Free Induction Decay



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Free Induction decay : FID

Figure 7.4. Graphical Representation of Some Acquisition Parameters

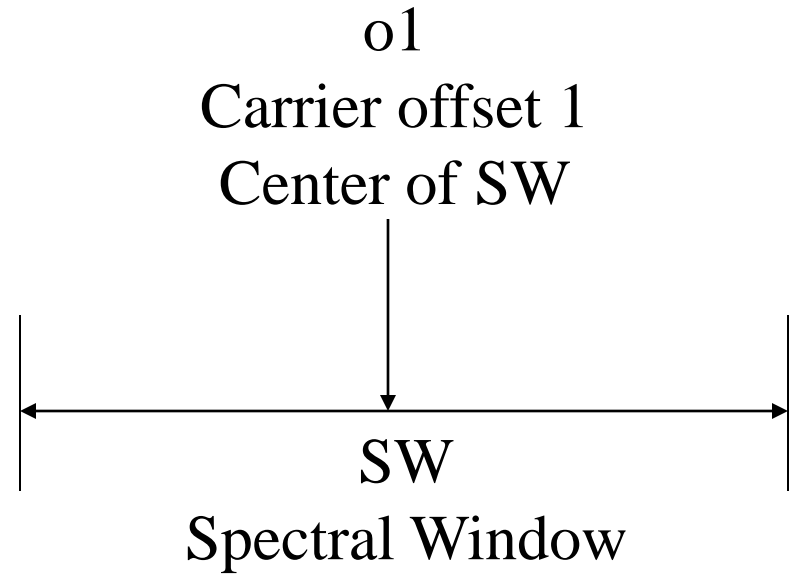
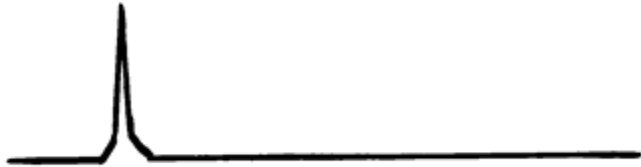
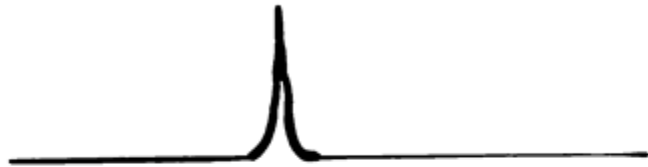


For $SW=2,000$ Hz and $TD=16K$

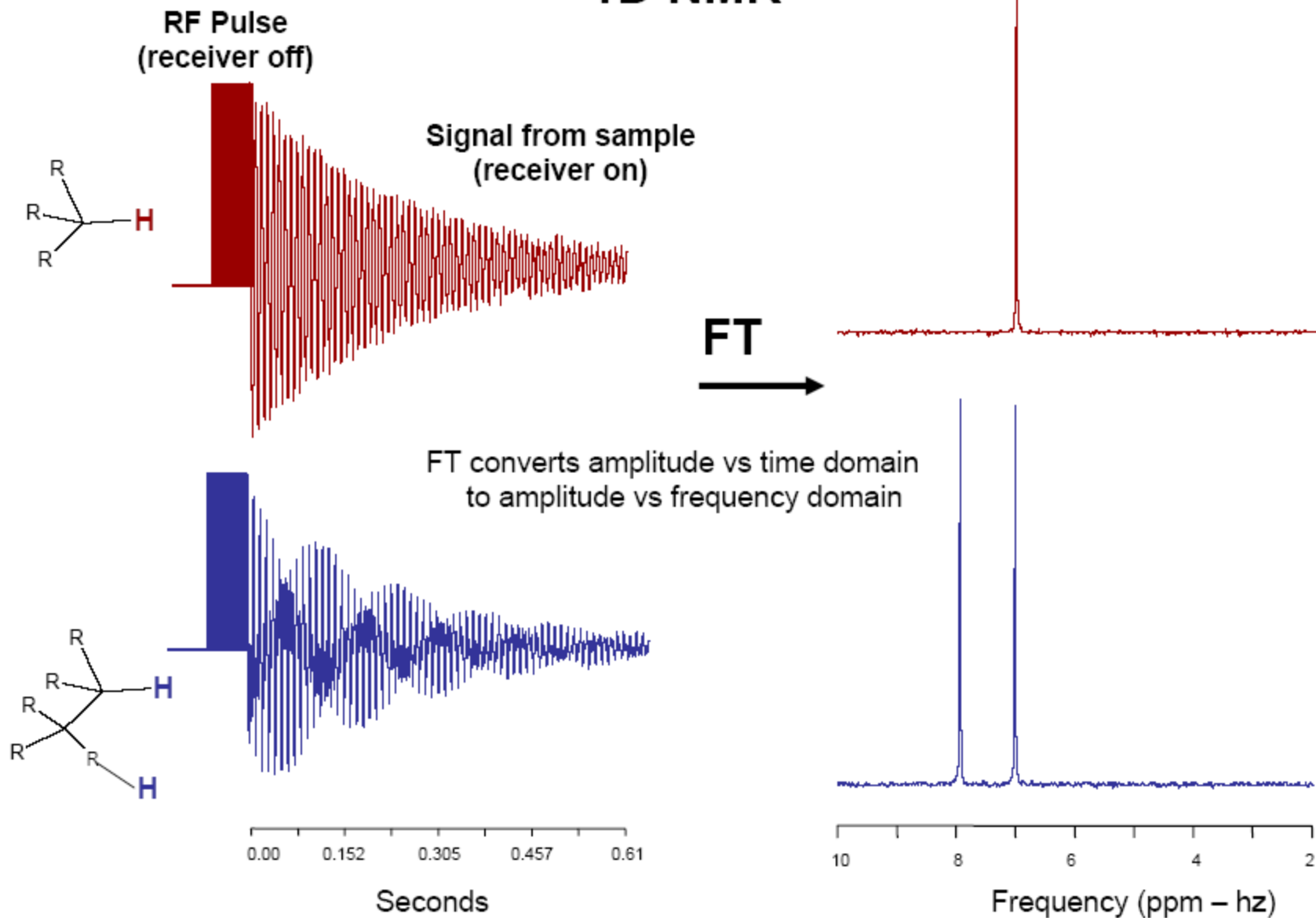
$$D.R. = 1 / AQ = 2 * SW / TD$$

$$AQ = TD * DW = TD / (2 * SW) = 4 \text{ seconds}$$

Fourier Transform

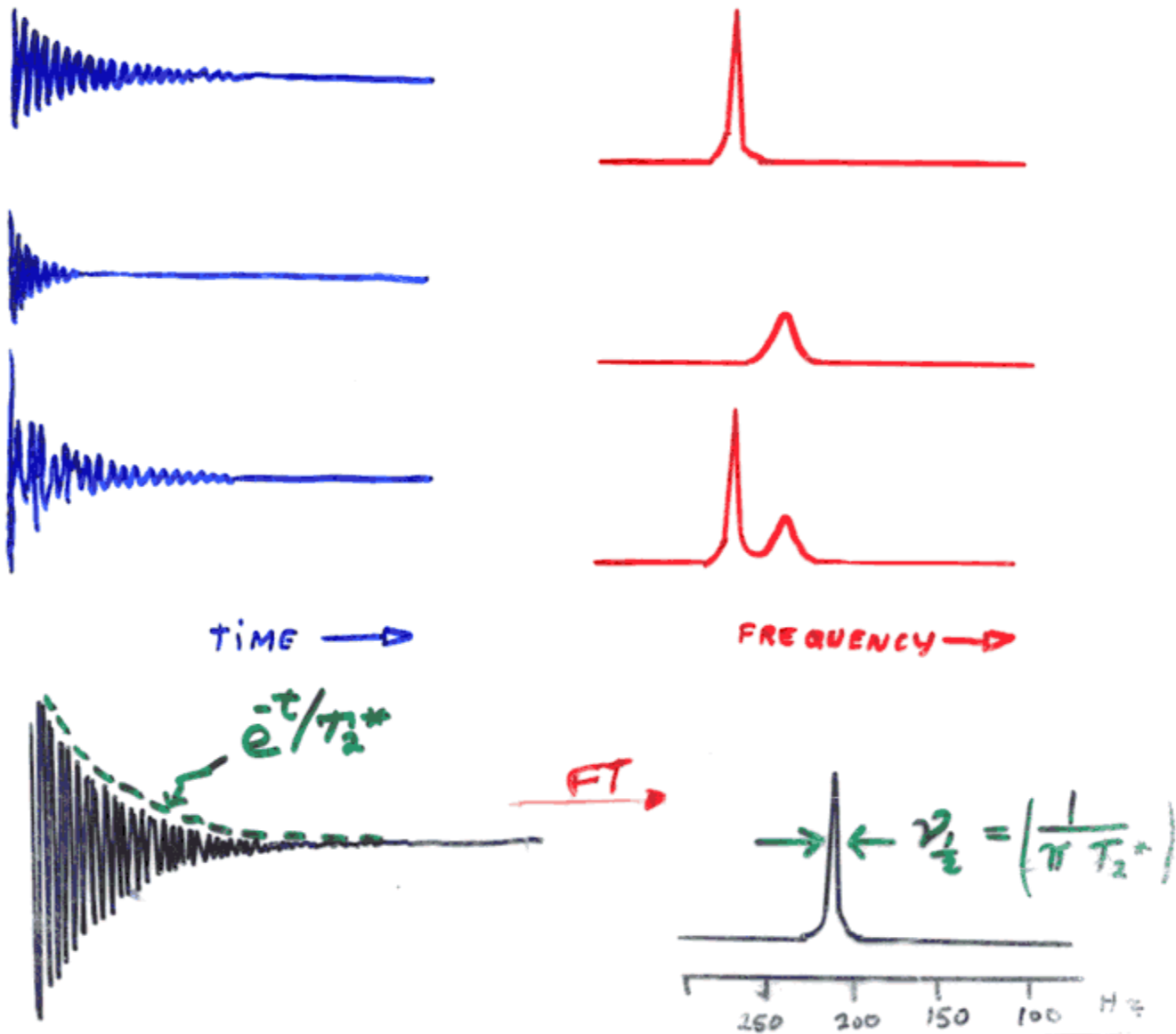


1D NMR



Fourier Transformation

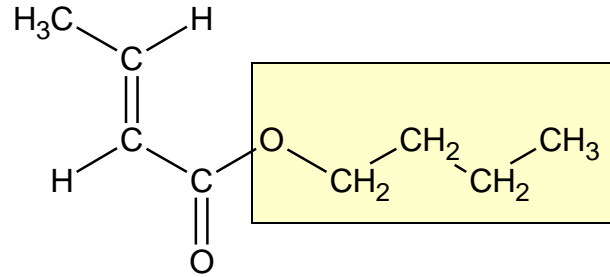
FID Duration and Line Width



Steps to acquire data

- Prepare sample
dissolve in deuterated solvent (CDCl₃, acetone-d₆ ...)
filter solution in NMR tube : fill the tube up to ~4 cm
of solution
- Insert sample in the magnet
- Lock on deuterium signal from the solvent
- Optimize shimming
- Select parameters ¹H, ¹³C, ³¹P,
- Set receiver gain
- Acquire the data
- Process and plot data

$^1\text{H NMR}:\text{C}_8\text{H}_{14}\text{O}_2$



triplet

triplet

Roof effect

#lines = n + 1

quintet

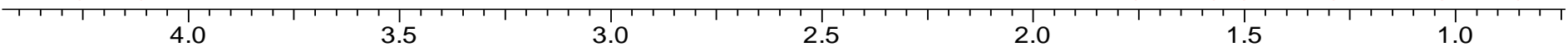
sixtet

2.00

2.05

2.06

2.99



NMR processing: apodization Window

- **Line Broadning (LB) : Exponential Multiplication**
improves the signal to noise ratio (at the expense of resolution)

Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

Function:

f2

Navigation: \leftarrow \rightarrow \uparrow \downarrow \leftarrow \rightarrow

ppm/Hz

fid:

f2

Navigation: \leftarrow \rightarrow \uparrow \downarrow \leftarrow \rightarrow

Action:

gm

qsin

Parameters:

+ - \updownarrow

+ - \updownarrow

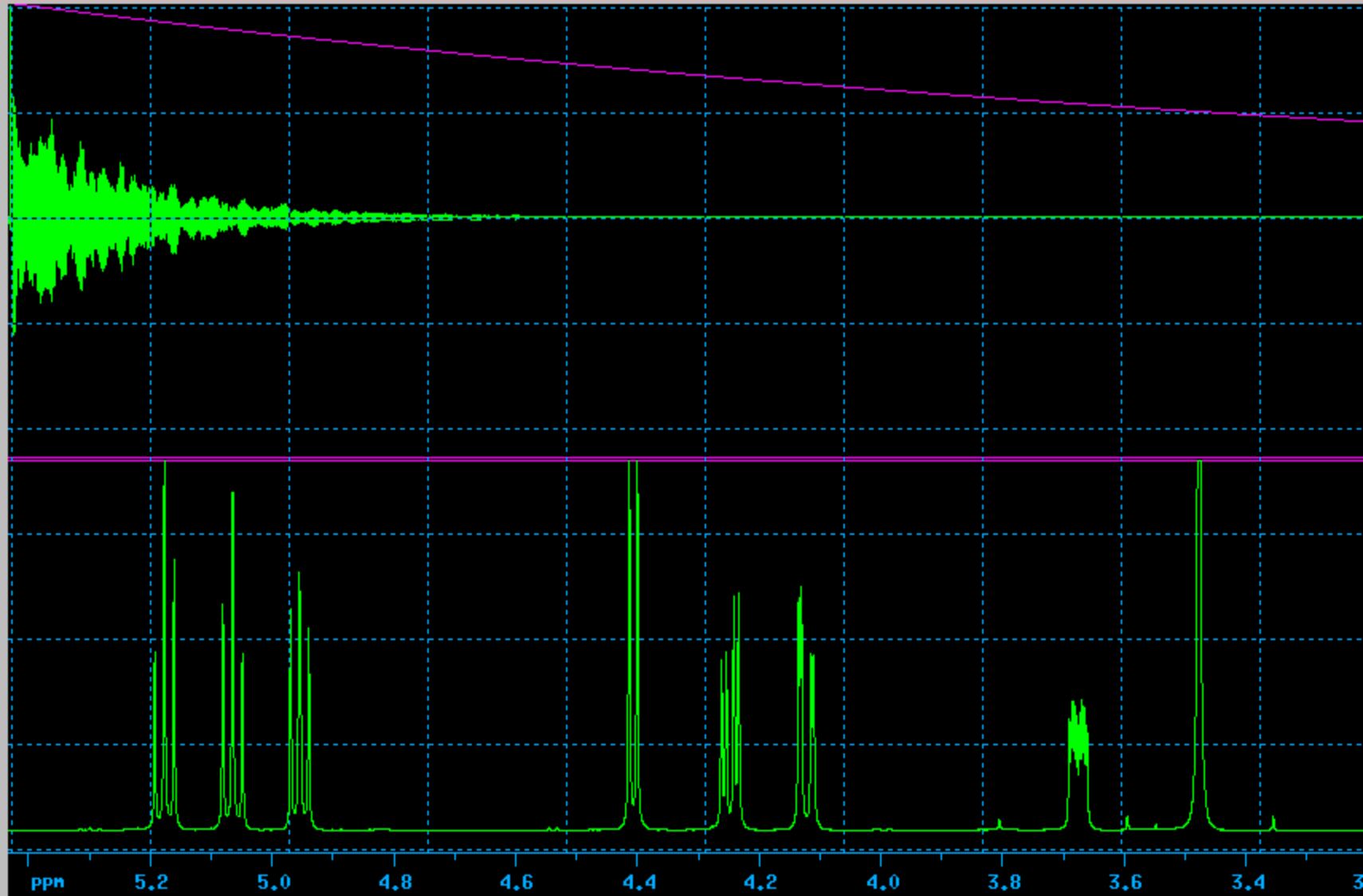
+ - \updownarrow

=1 =2

delta

ph-mod

return



Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

waifunc

f2

ppm/Hz

fid:

function:

gm

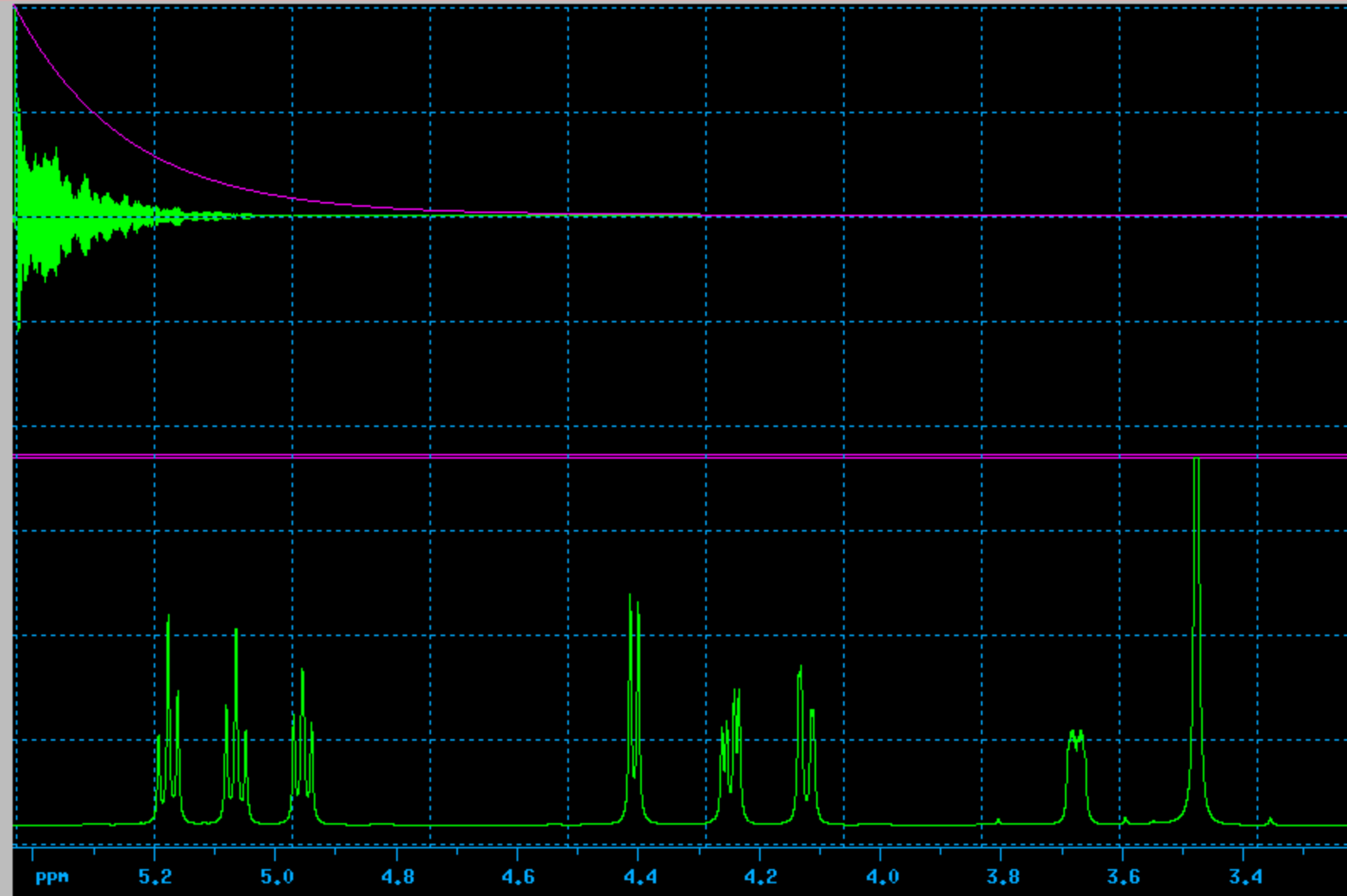
qsin

parameters:

delta

ph-mod

return



NMR processing: apodization Window

- Line broadening : Exponential multiplication
improves the signal to noise ratio (at the expense of resolution)

- Resolution Enhancement
reverse exponential + Gaussian function
also traf function,
also sine function (for 2D – magnitude mode)

Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

Window = GM
PH_mod = pk
LB = -0.70
GB = 0.40

wavfunc

f2

ppm/Hz

fid:

function:

m gm

m qsin

parameters:

+ -

+ -

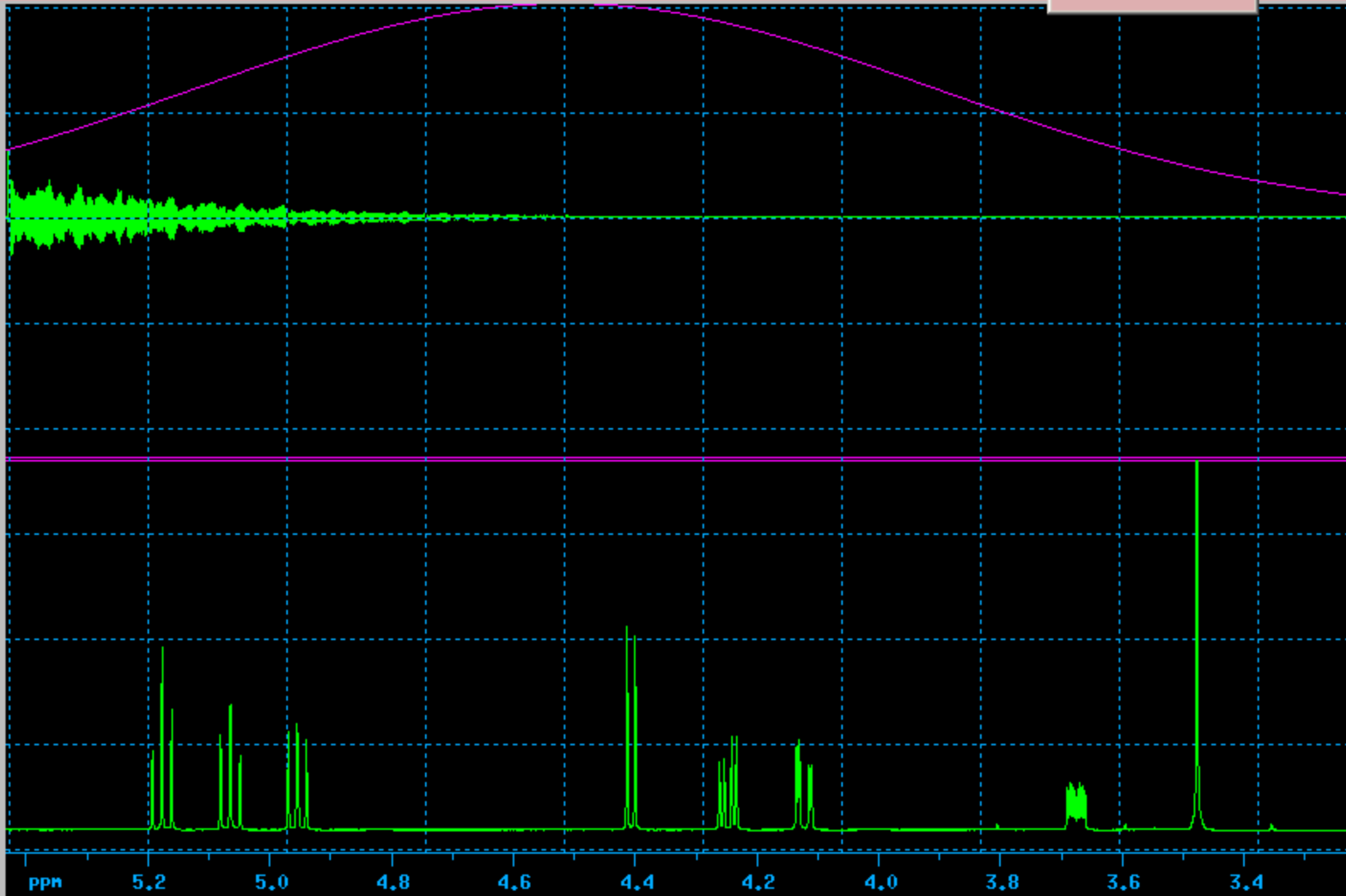
+ -

=1 =2

delta

ph-mod

return



Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

Info

Window = SINM
PH_mod = pk

SSB = 0.00

vairfunc

f2

Navigation: \leftarrow \rightarrow \uparrow \downarrow \leftarrow \rightarrow

ppm/Hz

fid:

f2

Navigation: \leftarrow \rightarrow \uparrow \downarrow \leftarrow \rightarrow

function:

m gm
am qsin

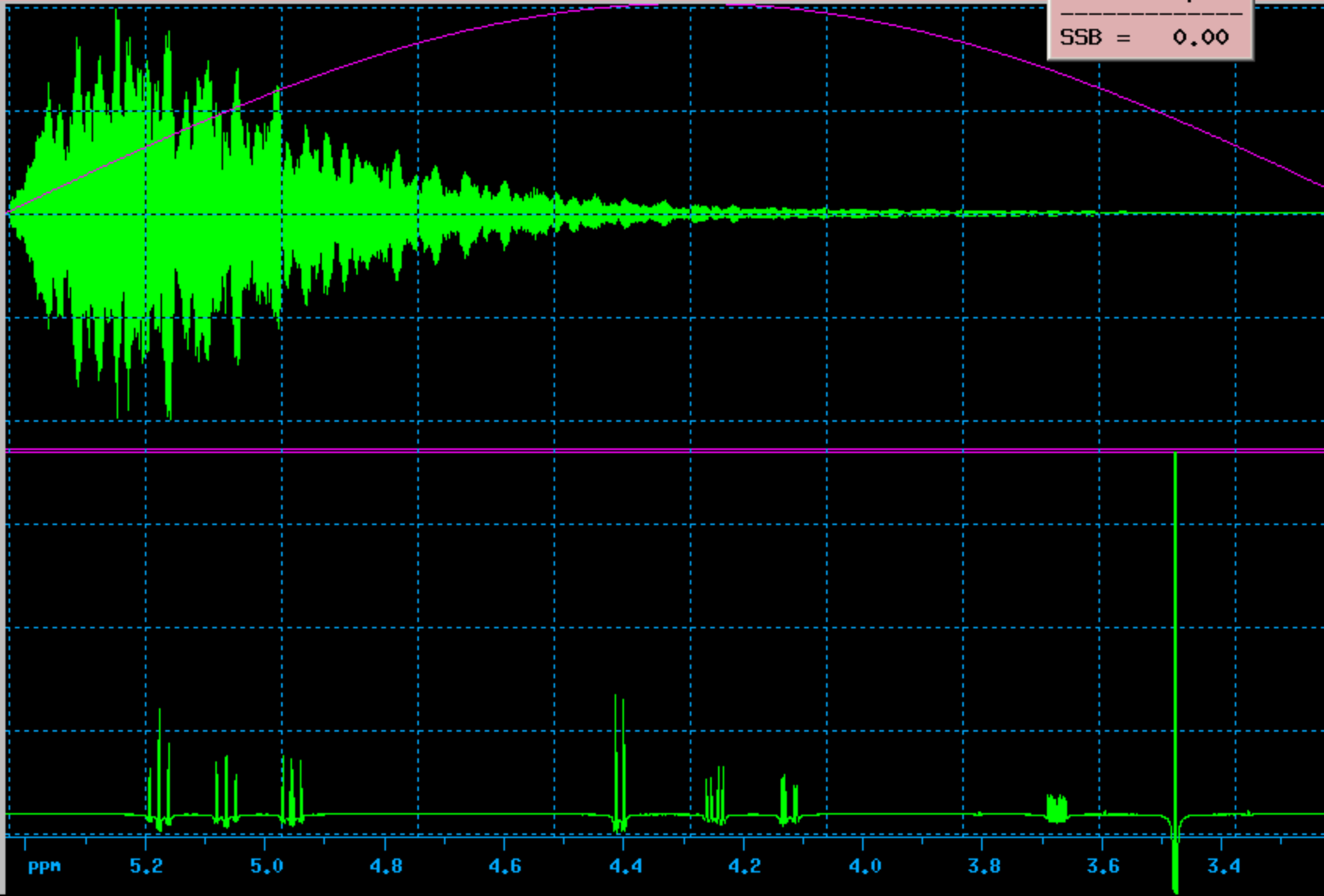
parameters:

+ - \updownarrow
+ - \updownarrow
+ - \updownarrow
=1 =2

delta

ph-mod

return



Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

Info

Window = SINM
PH_mod = mc

SSB = 0.00

wavfunc

f2

Navigation buttons: left, right, double left, double right

Navigation buttons: up, down

Navigation button: right

ppm/Hz

fid:

f2

Navigation buttons: down, left, right

function:

m gm

m qsin

parameters:

+ -

+ -

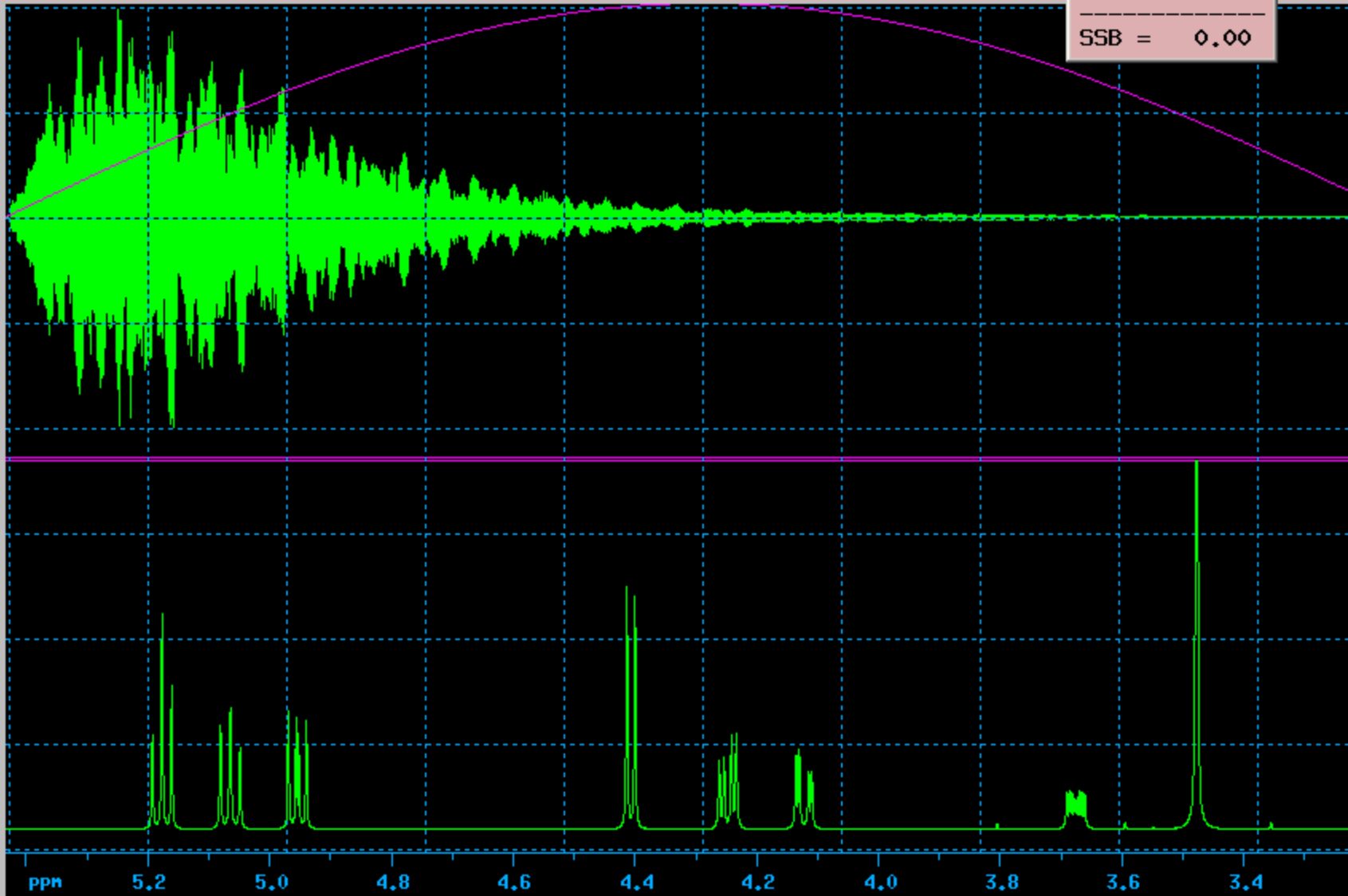
+ -

=1 =2

delta

ph-mod

return



Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

Info

Window = QSIN
PH_mod = ps

SSB = 0.00

varfunc

f2

ppm/Hz

fid:

function:

m gm
m qsin

parameters:

+ -

+ -

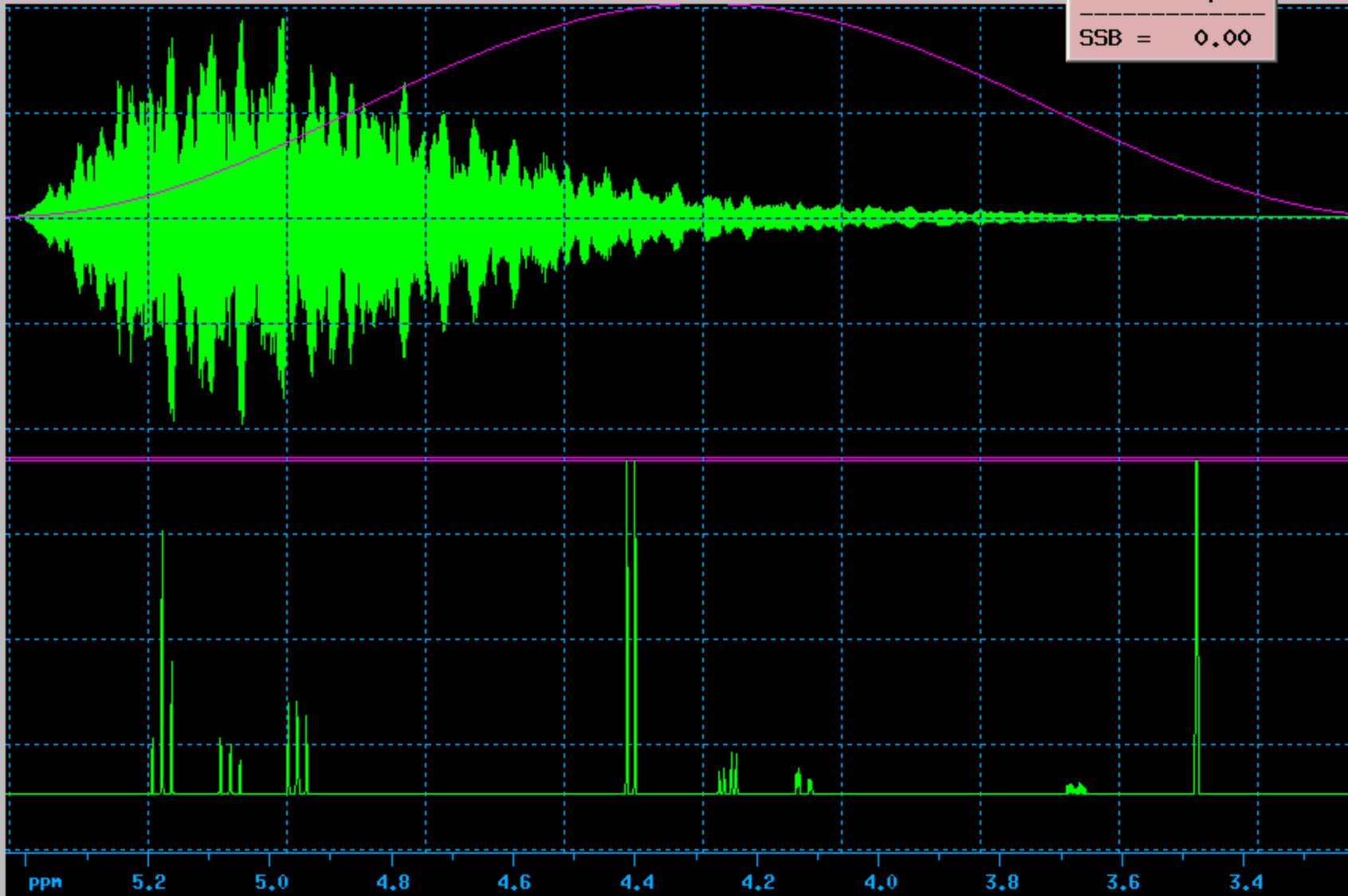
+ -

=1 =2

delta

ph-mod

return



Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

Info

Window = QSIN
PH_mod = pk

SSB = 0.00

varifunc

f2

ppm/Hz

fid:

function:

m gm

m qsin

parameters:

+ -

+ -

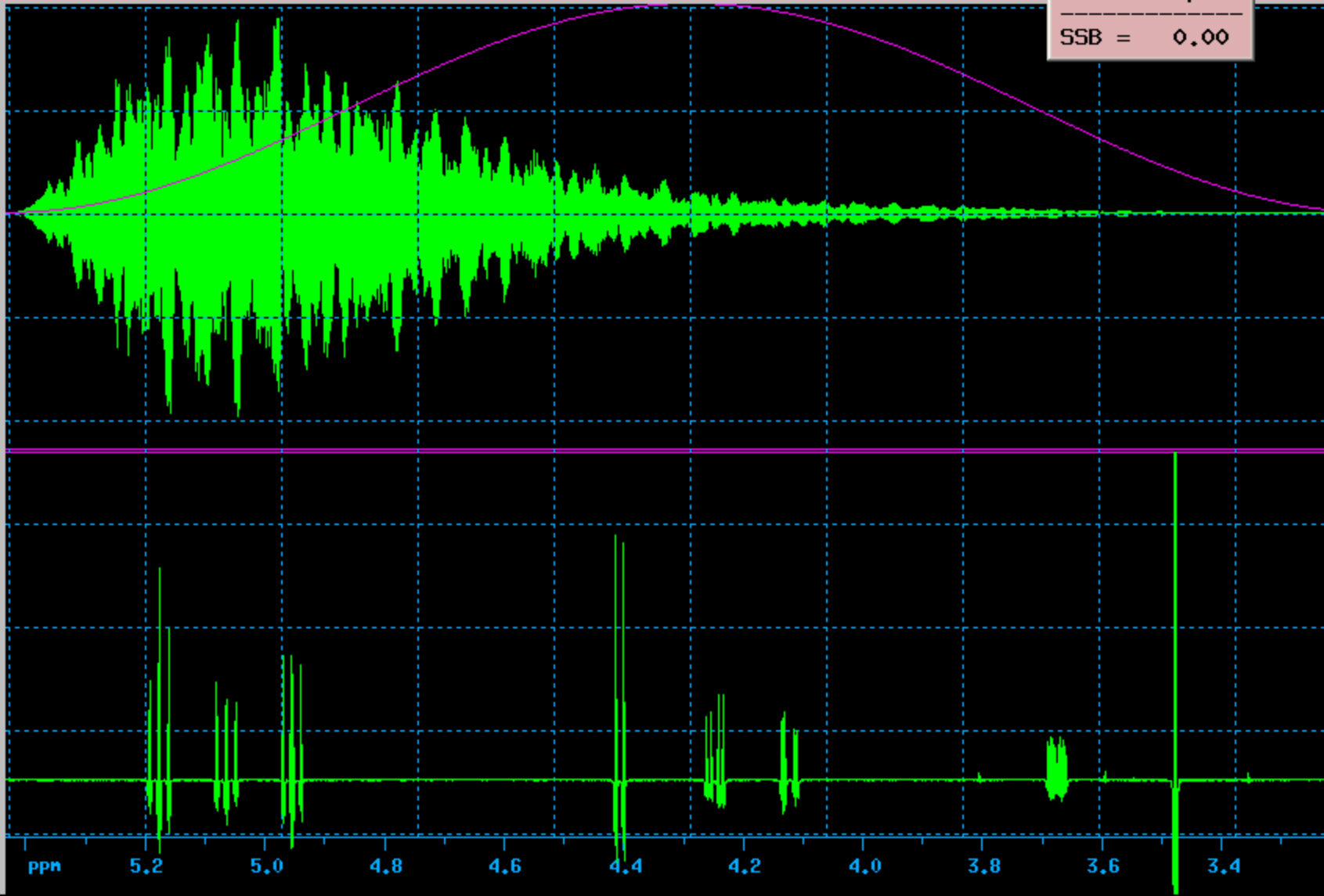
+ -

=1 =2

delta

ph-mod

return



Dataset: < fstest_gluc 1 1 C:/Bruker/XWIN-NMR guest >

Title: Tetraacetyl-Methoxy Glucose H standard 600 MHz

Info

Window = QSIN
PH_mod = mc

SSB = 0.00

varfunc

f2

Navigation: \leftarrow \rightarrow \uparrow \downarrow \leftarrow \rightarrow

ppm/Hz

fid:

f2

Navigation: \leftarrow \rightarrow \uparrow \downarrow \leftarrow \rightarrow

function:

m gm
m qsin

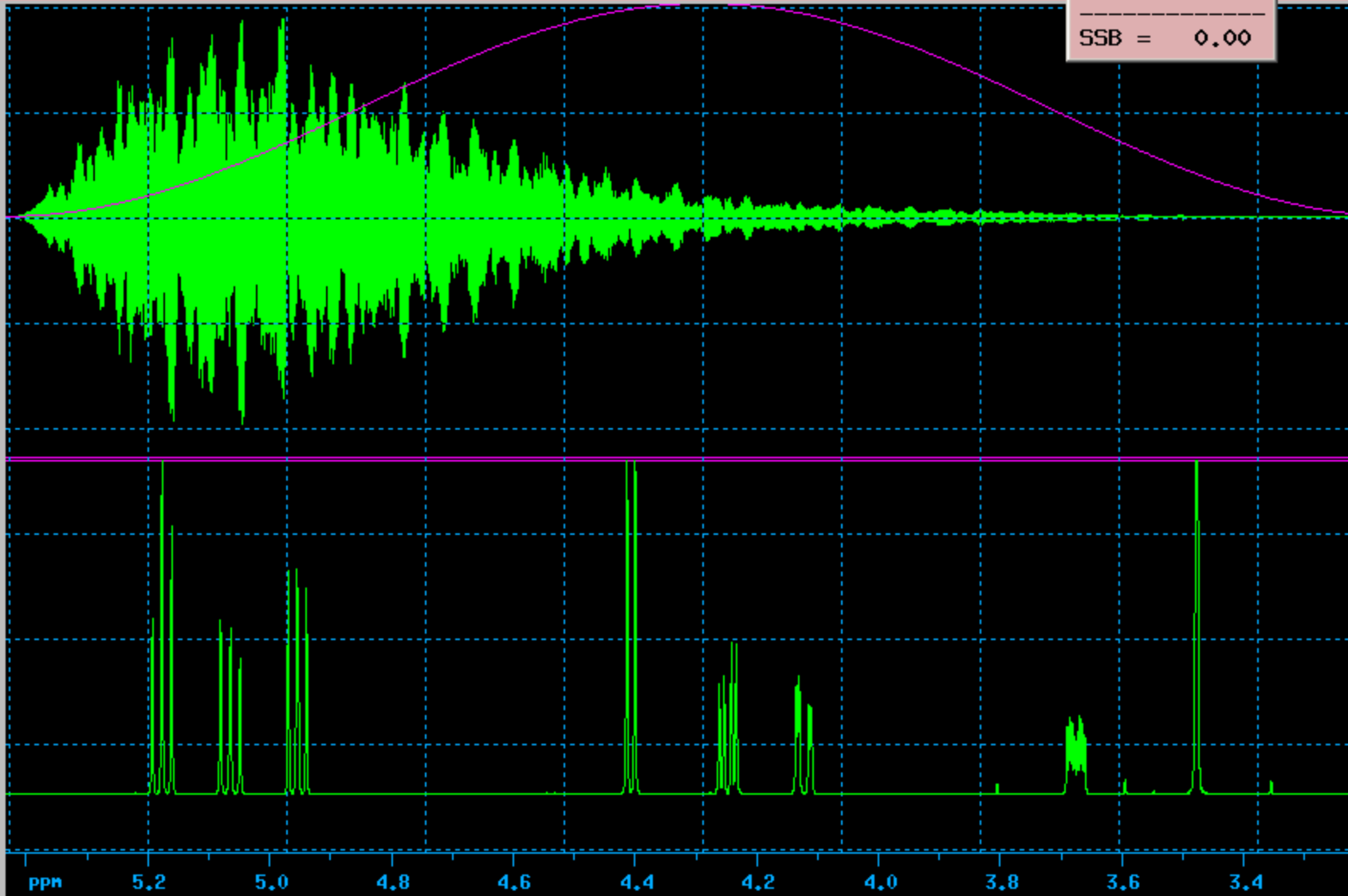
parameters:

+ - \updownarrow
+ - \updownarrow
+ - \updownarrow
=1 =2

delta

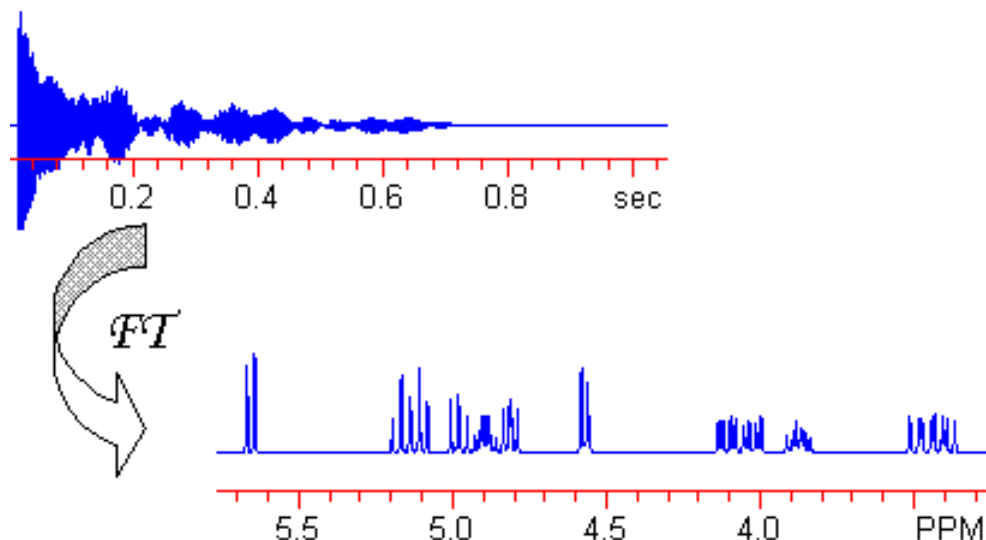
ph-mod

return



ppm 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4

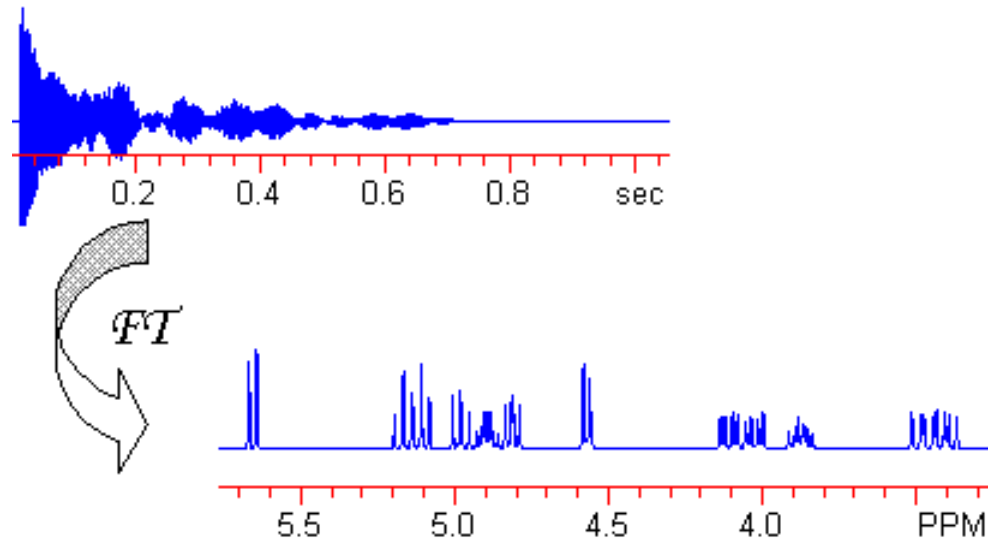
NMR processing



There is a variety of software available for NMR processing Many of them are **Freeware** for student

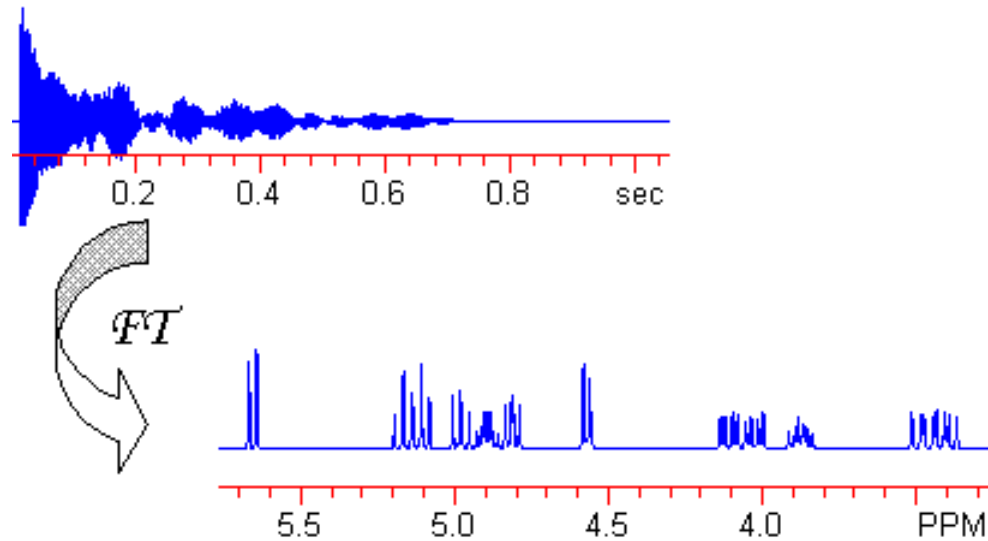
- [ACD-lab NMR processing software](#)
- Spinwork
- NMR notebook

NMR processing Steps



- 1- Open the FID and Apply 'Zero Filling' (more points in the spectra)
- 2- Click on mouse phase
 - left mouse to phase cursor peak
 - Tight mouse for other peaks
- 3- Choose an Apodization function:
 - Line broadening (better S/N)
 - Traf. function for better resolution (worse S/N)

NMR processing steps



- 4- Do Baseline correction (auto → result → accept)
- 5- Do peak picking (select peak level → accept)
- 6- Do integration: Manual → drag the mouse → define number of H
- 7- Do multiplet analysis: Drag around a multiplet