Data Collection using Xwinmr

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Before go into NMR LAB

Which NMR , which probe head to choose?

AV500 in IBMS : 5mm TXI-Z ($^{1}H/^{15}N/^{13}C$, with Z gradient) only

5mm TXI-Z CryoProbe

AV600 in IBMS: 5mm QXI-Z ($^{1}H/^{15}N/^{13}C$ /^{31}P)

5mm TXI-Z CryoProbe

AV600 in CHEM: 5mm BBO & TXI-Z ($^{1}H/^{15}N/^{13}C$, with Z gradient)

DRX600 in IBMS: 5mm TXI-XYZ ($^{1}H/^{15}N/^{13}C$, with XYZ gradient) and others

5mm : ^{1}H , $^{1}H/^{19}F$, BBO, TXI($^{1}H/^{15}N/^{13}C$) , TXI-Z ($^{1}H/^{3}C/^{31}P$)

8mm : TXI ($^{1}H/^{13}C/^{15}N$) 8mm with Z gradient

10mm: ^{1}H , ^{1}H /^{19}F , BBO

AV800 in IBMS : 5mm TXI-Z & CryoProbe (not available yet)

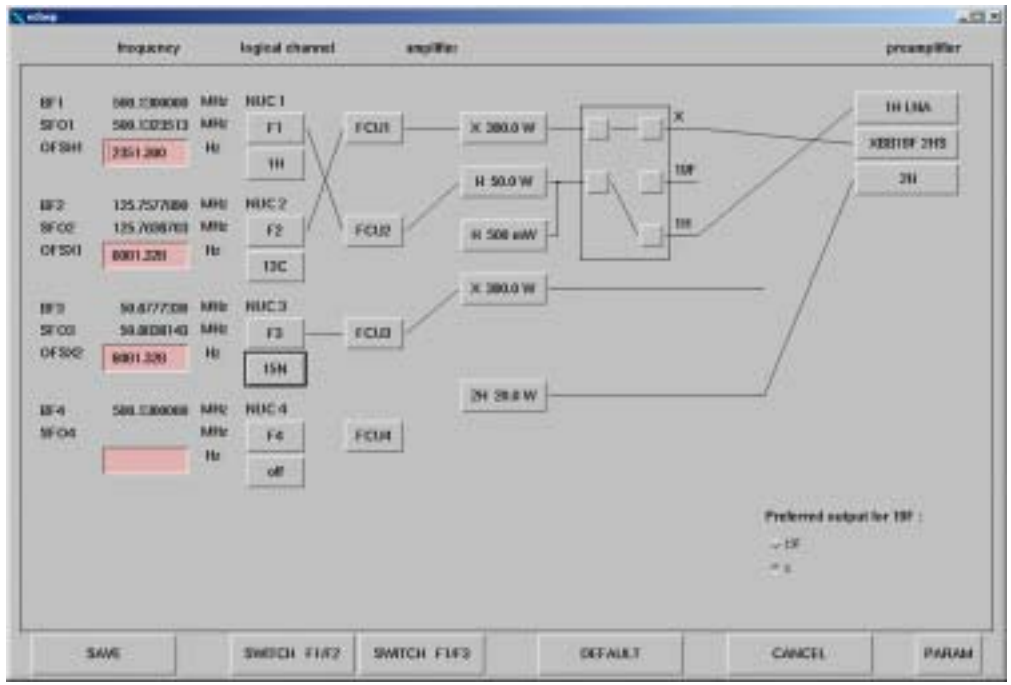
In the NMR LAB

Software Connection

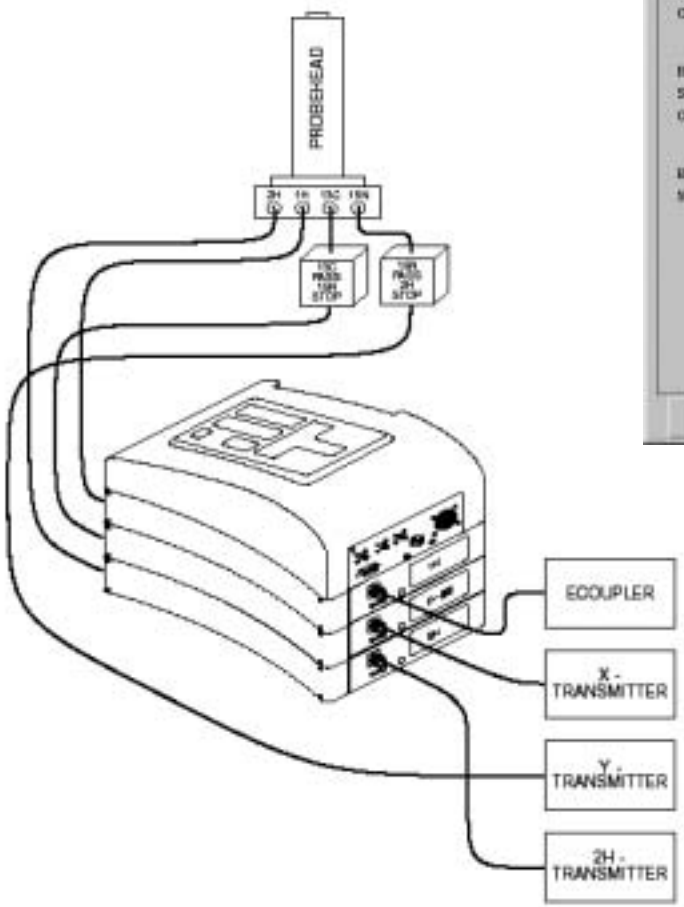
Click on XWIN-NMR 3.5 icon

Hardware Connection

edasp



observe nucleus:1H
decoupling 1:13C
lock:2H
decoupling 2:15N



1H LNA

XBB19F

2H

Loading Sample

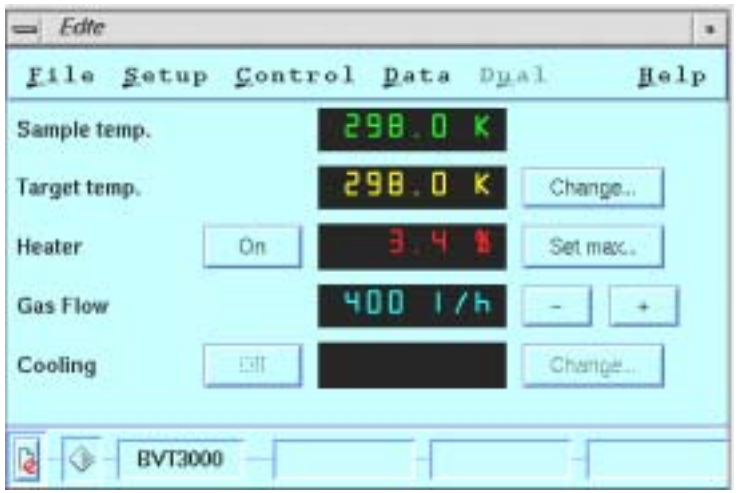
1. The best condition for sample? Probes, Temperature, Sample position

1-1. Selecting appropriate probe :

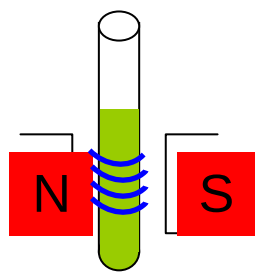
inner coil is observe coil and outer coil is decoupling

Example: better 13C sensitivity should use BBO (13C: inner, 1H: outer), but if for better 1H, “inversed probe” (1H: inner, others: outer) is recommended

1-2. Set up temperature : **edte** (edit temperature)



1-3. Adjust sample position



Loading Sample

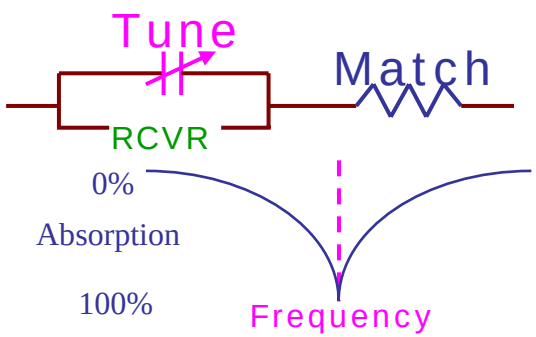
2. Load sample to the magnet

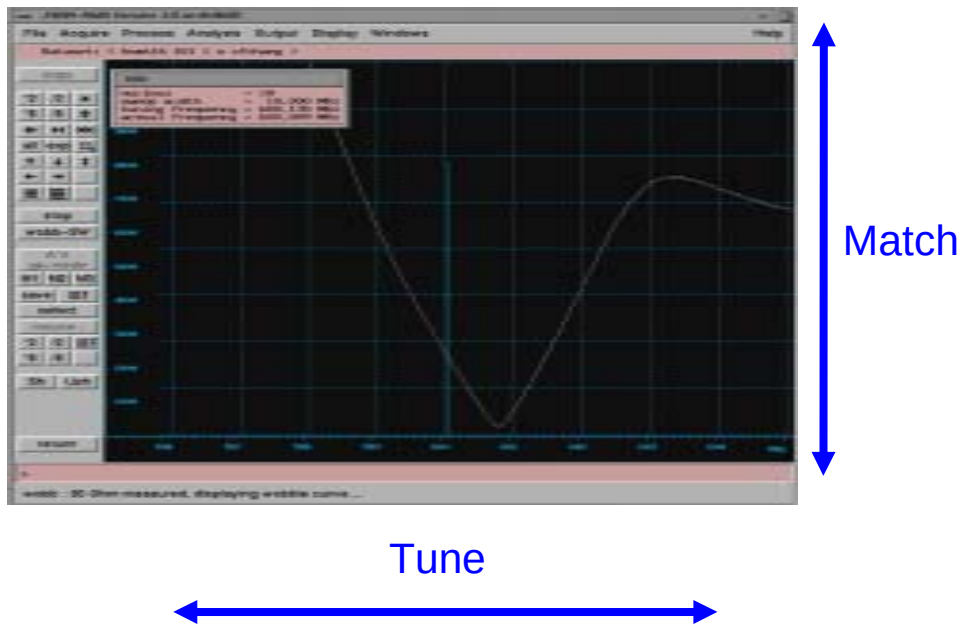
   Use “BSMS” board next to computer

3. The best condition for NMR? → wobble : Tune & Match

   -Tuning is the process of adjusting freq. until it coincides with the desired frequency

   -Matching is the process of adjusting the impedance of the resonant circuit



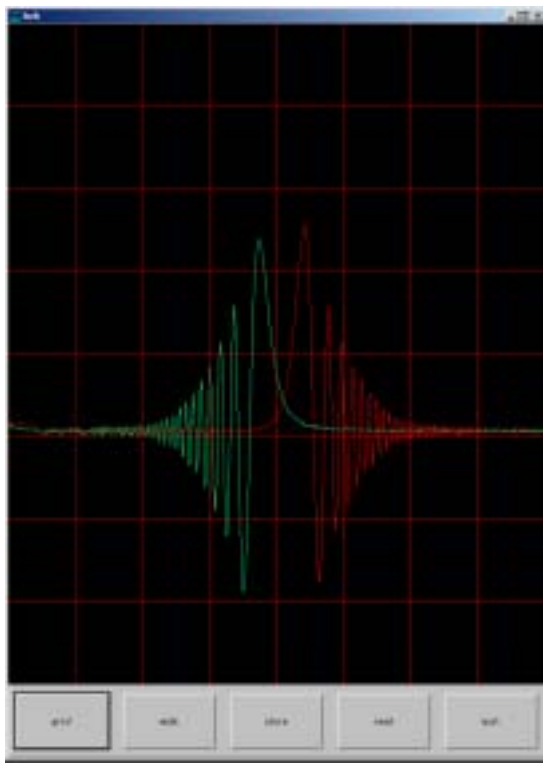


4. The best condition for field? → lock and gradshim or manual shim on "BSMS"

-Deuterium lock means the long term stability of the magnetic field is achieved

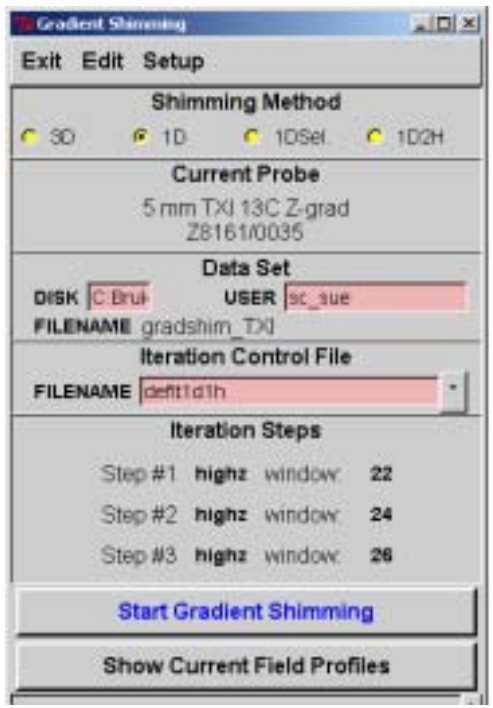
-The shims (coils) are small magnetic fields used to adjust the homogeneity of the field

lockdisp



lock

gradshim : gradient shimming



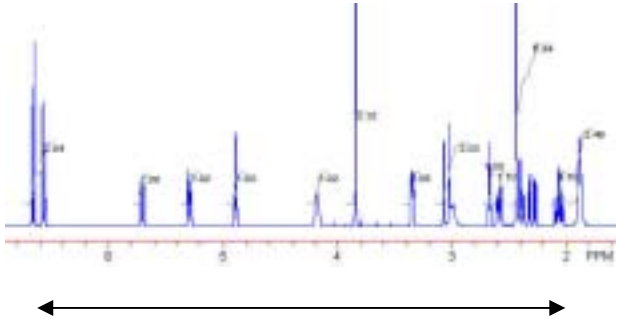
Definition of some AQ Commands

edc,new	edit current data set or generate a new data set
edasp	edit hardware connection
edte	edit temperature
wobb	tune and match
eda.ased	edit AQ parameters (eda: shows all, ased: shows required only)
getprosol	load the existed pulse calibration values for current probe
rga	auto optimize rg value
zg	zero memory, and start to collect FID
go	start to collect FID and add signals to the previous memory
acqu	switch window to observe FID
ft	Fourier transfer for 1D
xfb	Fourier transfer for 2D
stop/halt	stop the active job (currently AQ job)
kill	kill active job (can choose several jobs)

Definition of some AQ Commands & parameters

o1.o2,o3	center frequency of the spectrum for nuclear at f1 channel (ex: 1H), f2 channel (ex:13C), and f3 channel (ex:15N)
sw	spectrum width (1 sw : F1 dimension, 2 sw: F2 dimension…….)
td	number of points for FID collection(1 td: F1 dimension, 2 td: F2 dimension….)
d1	relaxation time (usually > 5* T1)
ns	number of scan
ds	dummy scan
rg	receiver gain (usually use the value calculated by rga)
p0 ~	pulse length in us
pcpd1 ~	pulse length for decoupling pulse in us
pl1~	power level in dB
sp1 ~	power level for shape pulse in dB
d2 ~	delay in sec

1D

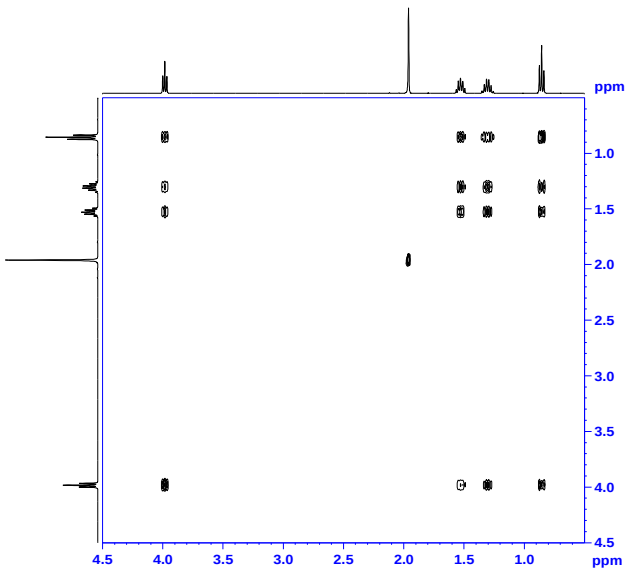


center (o1) at 4.5ppm

sw=6ppm

1.5ppm (=o1-sw/2) to 7.5ppm(=o1+sw/2)

2D/homo



F2 dimension !!!

F1 dimension !!!

F2: H (f1 channel)

O1(f1 channel):2.25ppm

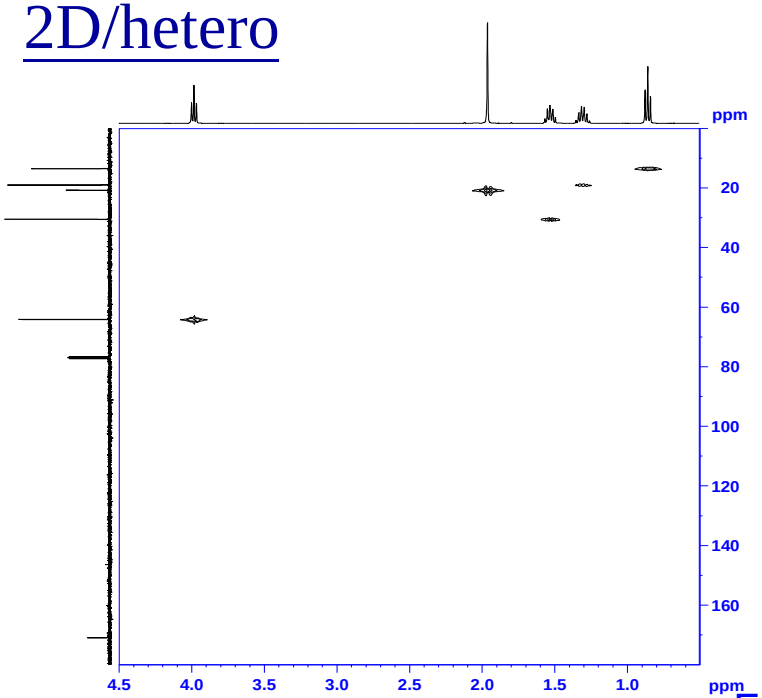
2 sw(F2 dimension): 4.5ppm

F1: H (f1 channel)

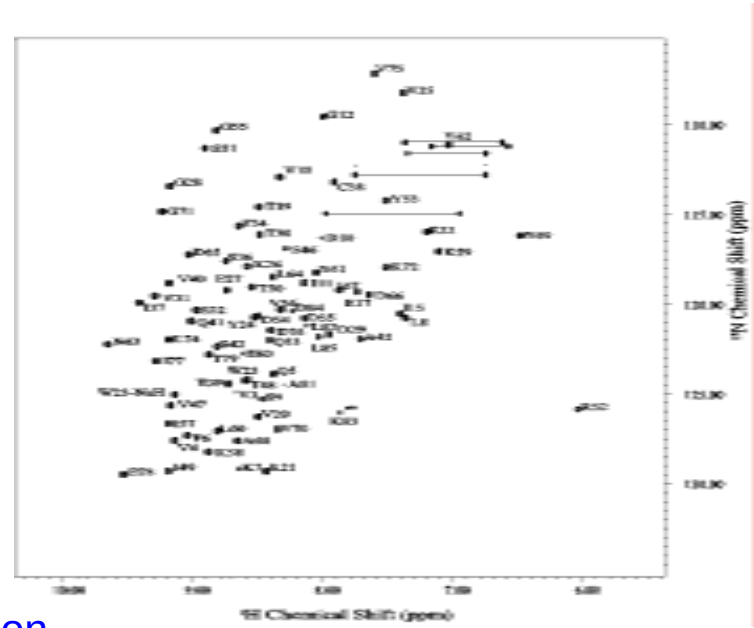
O1(f1 channel): 2.25ppm

1 sw(F1 diemnsion): 4.5ppm

2D/hetero



F1 dimension



F2 dimension

HMQC (13C-1H) ⟷ HSQC (15N-1H)

F2: H (f1 channel)

O1(f1 channel):2.6ppm

2 sw(F2 dimension): 5ppm

F1: C (f2 channel)

O2 (f2channel) :80ppm

1sw (F1 dimension): 200ppm

F2: H (f1 channel)

O1(f1 channel):2.6ppm

2 sw(F2 dimension): 5ppm

F1: N (f3 channel)

O3(f3 channel): 80ppm

1 sw(F1 diemnsion): 40ppm

LAB handout for 09/06/2004 Training Course

Simple Operation Guide for BRUKER AV System

Set up Experiment

Easy steps for beginner (1D):

1. Type " new" <enter> to start a new experiment
2. Type "rpar" <enter> to load an appropriate parameter set
3. Type "getprosol" <enter> to load pulse parameter for probehead
4. Type "ns" <enter> to input number of scan
5. Type "rga" <enter> to find appropriate receiver gain
6. Type "zg" <enter> to collect spectrum
7. Type "ft" <enter> to do Fourier Transfer
8. Click on phase to phase spectrum
9. Click on return, then save to save the spectrum
10. Print out the spectrum or Save your data on floppy

Set up Experiment for Bio-molecules

(1) **Experiment Name: 1D 1H**
Experiment Type: one pulse for 1H

(2) **Experiment Name: 1D 1H H2O suppression**
Experiment Type: H2O suppression using presaturation

(3) **Experiment Name: 2D 1H TOCSY DIPSI**
Experiment Type: H2O suppression using 3-9-19

(4) **Experiment Name: 2D 15N-1H HSQC**
Experiment Type: using echo-antiecho

(5) **Experiment Name: 3D HNCO**
Experiment Type: using echo-antiecho

Set up Experiment for Small molecules

(1) **Experiment Name: 1D 1H**
Experiment Type: one pulse for 1H

(2) **Experiment Name: 2D COSY 45 deg**
Experiment Type: QF mode

(3) **Experiment Name: 1D 13C**
Experiment Type: with 1H decoupling

(4) **Experiment Name: 1D 13C dept90**
Experiment Type: with 90 deg read pulse to give XH only

(5) **Experiment Name: 1D 13C dept135**
Experiment Type: XH, XH3 positive, XH2 negative

(6) **Experiment Name: 2D 13C-1H HMQC**
Experiment Type: hmqcph

- **How to optimize condition? → For users: Follow Experiment Guide**

•Experiment Name: 2D 15N-1H HSQC

•Experiment Type: Using echo-antiecho, f1: H, f3:N

•Standard Parameter Set: std_2D_15N_HSQC_ET

•Pulse Program: hsqcf3gpsi

•AQ parameters to check

1H pulses

pl1 (high power, ex: 0dB), p1 (90o pulse at pl1)

p28 (trim pulse, ex:1m)

Others

cnst4 (J H-N , ex: 90Hz)

d24 (1/4JH-N)

o1 (for 1H)

o3 (for 15N)

1 sw, 1td (for F1 dimension, N)

2 sw, 2 td (for F2 dimension, H)

d1

ns(=1*n)

ds(>=16)

rg

Users need to adjust parameters in "red" (meaning of the parameter in "green")

- **How to optimize condition? → For users: Follow Experiment Guide**

•Experiment Name: 3D HNCO

•Experiment Type: Using Echo/antiecho , f1: H, f2:C, f3:N, F1(CO), F2(N), F3(H)

•Standard Parameter Set: std_3D_HNCO_wg

•Pulse Program: hncogp3dwg

•AQ parameters to check

1H pulses

pl1 (high power, ex: 0db), p1(90deg at pl1), p2(180deg at pl1)

pl19 (low power for dipsi2,pcpd1), p26(90deg at pl19), pcpd1(90deg ,ex: 40-50usec)

sp1 (shape pulse power for Sinc.1000) , p11(pulse length for sp1, ex: 2m)

Others

o1 (for 1H), o2 (for 13CO), o3 (for 15N)

1 sw, 1 td (for F1 dimension, ie: 13C)

2 sw, 2 td (for F2 dimension, ie:15N)

3 sw, 3 td (for F3 dimension, ie:1H)

d1, rg, ns(=8*n), ds (≧ 16)

Users need to adjust parameters in "red" (meaning of the parameter in "green")

Steps for optimize 90 deg pulse:

1. "rpar" <std_1D_1H_ZGPR> , and set O1 to H2O position
   (if not in H2O, just use std_1D_1H_ZG to determine 90 deg pulse)

2. Set pL1= 0db (or 10db or ? db), p1=5u

3. "rga" → "zg"→ "ft" →<phase> → <save>

4. Keep pL1, increase p1= expected 360deg
   (or, keep p1=360 deg pulse length, decrease pL1=expected power level for p1 value)

5. "zg"→ "fp" →check if the spectrum almost become null, if not, repeat step 4

6. Now, you should have a table with 1H pulse information of your sample