



How a basic Spectrum is generated via pulse NMR ?



➤ Theoretical Background: Part One, the Basic

- ❖ Acquisition parameters.
- ❖ Timing sequence and pulse.
- ❖ Basic receiver ---- RG , AQ & TD
- ❖ How all these parameters are stored: Data organization.
- ❖ Multiple scans pre-requisite:
 - ❖ Homogeneity --- lock and shim



Link: Theory-part1.ppx

➤ Practical Procedure: (Demo during Lab sessions)


- ❖ Set Up menu.
- ❖ Optimization menu.

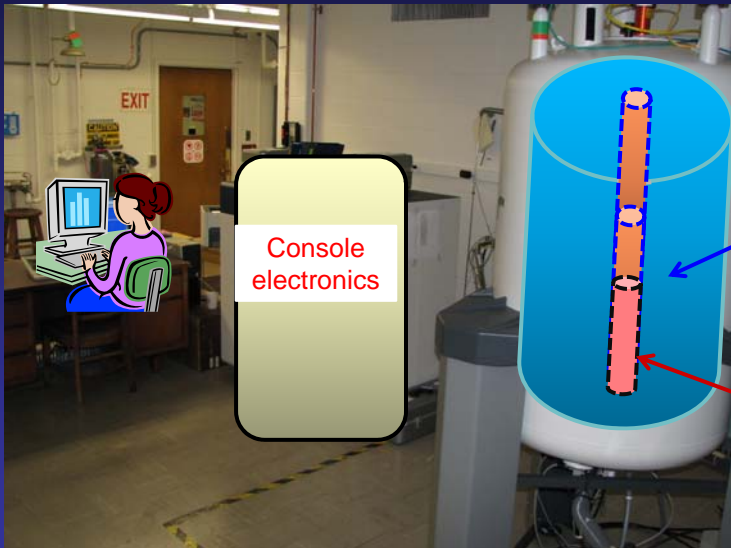
The rest of the slides show the abstract from the Demo. Data acquisition and basic processing.

From: [introduction-chem471-r3](#)

Basic-1d-win2010..Y.F. Lam


A typical Pulse NMR spectrometer





Console
electronics

Dual cylinders:
Liquid Nitrogen
& Helium




Probe

From: [introduction-chem471-r3](#)


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Dec 29 2009 yflam

Adjust NMR tube



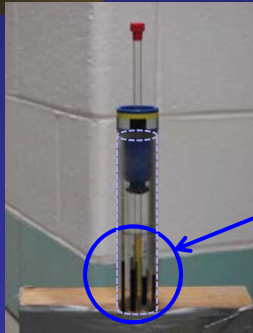
DO Hold sample tube steady while screwing spinner in & up!

DON'T




Fragile!

Gauge for proper position of solution inside the NMR tube




Probe Coil NMR detector




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Load sample into magnet





Ma

Lift On/ Off



Spin on/off




Set Up: via the Control Center PC






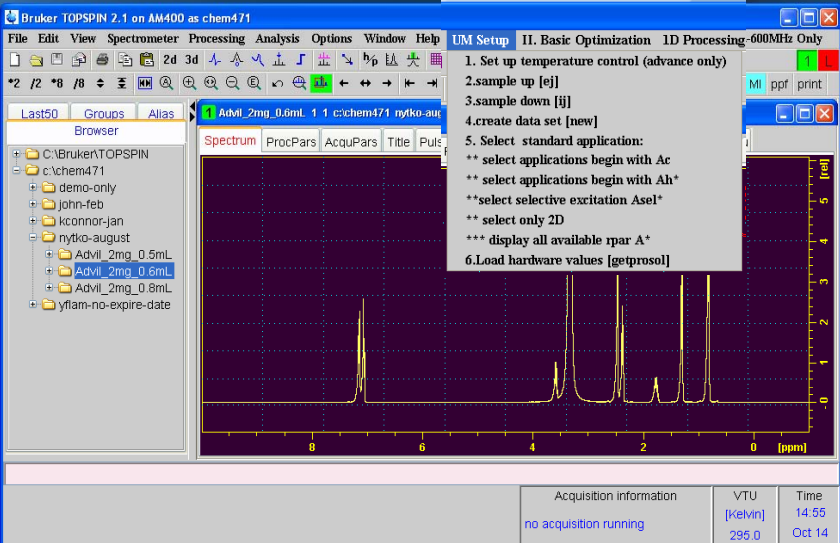
With Bruker NMR
Software TOPSPIN





Bruker NMR software: TOPSPIN





Acquisition Information	VTU	Time
no acquisition running	[kelvin] 295.0	14:55 Oct 14

4. "new" CREAT YOUR WORK SPACE " FILE"

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UM Setup II. Basic Optimization 1D Processing

1. Set up temperature control (advance only)
- 2.sample up [ej]
- 3.sample down [ij]
- 4.create data set [new]
5. Select standard application:
 - ** select applications begin with Ac
 - ** select applications begin with Ah*
 - **select selective excitation Ase1*
 - ** select only 2D
 - *** display all available rpar A*
- 6.Load hardware values [getprosol]

Link: Data File Structure

NAME: your-data-collected-in-march
 EXPNO: 1
 PROCNO: 1
 DIR: C:\sintim
 USER: wjxin-mar
 Solvent: CDCl3
 Experiment: Use current params.
 TITLE:

When you load Experiment in this window, Content of Title will not be replaced with the new experient.

OK Cancel More Info... Help

An experiment is a chapter (example shown # 3) within a work book Data Set, NAME. (example shown "example2")

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Spectrum ProcPars AcqPars Title PulseProg Peaks Integrals Sample Structure Fid Acqu

example2 3 1 c:\chem471 john

Spectrum ProcPars AcqPars Title PulseProg Peaks Integrals Sample Structure Fid Acqu

example2 3 1 c:\chem471 john

Rel Spectrum ProcPars AcqPars Title PulseProg Peaks Integrals Sample Structure Fid Acqu

example2 3 1 c:\chem471 john


Spectrum ProcPars AcqPars Title PulseProg Peaks Integrals Sample Structure Fid Acqu


Short ins: 1.98 ppm
 Index = 31248 - 31301
 Value = -1.25 rel

1.98 ppm
 Index = 31248 - 31301
 Value = -1.25 rel

0.5 1.0 1.5 [s]

Set up the experiment:






5. RPAR (load a specific NMR application into workbook all pages).
6. GETPROSOL (update values of the power settings)


UM Setup	II. Basic Optimization	1D Processing
<ol style="list-style-type: none"> 1. Set up temperature control (advance only) 2. sample up [ej] 3. sample down [ij] 4. create data set [new] 5. Select standard application: <ul style="list-style-type: none"> ** select applications begin with Ac ** select applications begin with Ah* **select selective excitation Ase1* ** select only 2D *** display all available rpar A* 6. Load hardware values [getprosol] 		

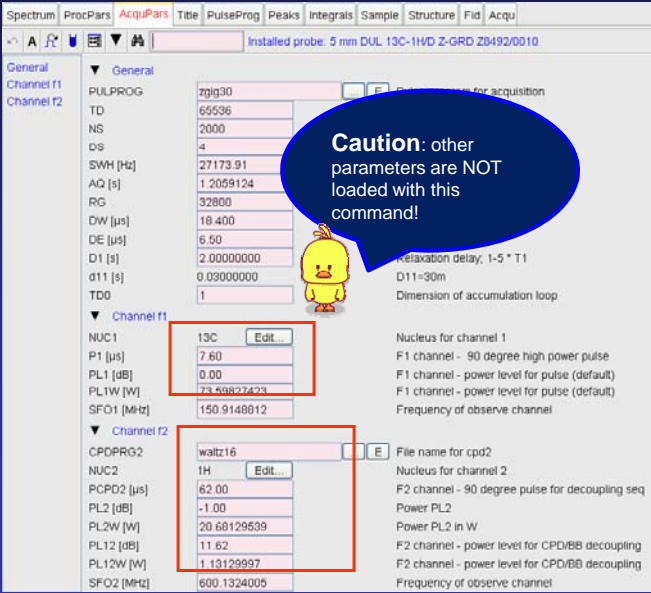
Examples:

- 1D proton: **Ah1**
- 2D measurements: A2d* e.g. A2d_cosy

“getprosol” : load pulses settings







Caution: other parameters are NOT loaded with this command!

Corresponding to the hardware (probe and solvent).

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Optimization and data collection: 1 D NMR

II. Basic Optimization 1D Processing Oct-600

1. Recall standard shims [rsh shim.current]
2. Auto lock to solvent [lock]
3. Use Keypad to Shim
- **600MHz only [topshim]
4. Tune probe* [wobb]
5. Modify parameters as needed [ased]
6. Auto gain adjust [rga]
7. Start measurement [zg]
- ** auto shim -overnight 1D expt.
- ==== optional
- ** duplicate data set [sav]

Console

D-NMR
lock & shim
electronics

rpar
getprosol

Receiver
A/D Converter

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LOCKING to the solvent of your sample!

Setup II. Basic Optimizati III. Basic Data Collect

rpar getprosol rsh lockdisp wobb ased

Solvent	Description
Acetic	acetic acid-d4
Acetone	acetone-d6
C6D6	benzene-d6
CD2Cl2	methylenechloride-d2
CD3CN	acetonitrile-d3
CD3CN_SPE	LC-SPE Solvent (Acetonitrile)
CDCl3	chloroform-d
CH3CN+D2O	HPLC Solvent (Acetonitril/D2O)
CH3OH+D2O	HPLC Solvent (Methanol/D2O)
D2O	deuteriumoxide
DEE	diethylether-d10
Dioxane	dioxane-d8
DME	dimethylether-d6
DMF	dimethylformamide-d7
DMSO	dimethylsulfoxide-d6
EtOD	ethanol-d6
H2O+D2O	90%H2O and 10%D2O
MeOD	methanol-d4
Pyr	pyridine-d5
THF	tetrahydrofuran-d8
Tol	toluene-d8

Acquisition in
no acquisition run

Click to select solvent in your sample

Use of Shim keypad :

II. Basic Optimization 1D Processing Oct-600

1. Recall standard shims [rsh shim.current]
2. Auto lock to solvent [lock]
3. Use Keypad to Shim
- **600MHz only [topshim]
4. Tune probe* [wobb]
5. Modify parameters as needed [ased]
6. Auto gain adjust [rga]
7. Start measurement [zg]

** auto shim -overnight 1D expt.
 ===== optional
 ** duplicate data set [sav]

Refer to Basic NMR manual for basic procedure

Modify acquisition parameters as needed:

II. Basic Optimization 1D Processing Oct-600

1. Recall standard shims [rsh shim.current]
2. Auto lock to solvent [lock]
3. Use Keypad to Shim
- **600MHz only [topshim]
4. Tune probe* [wobb]
5. Modify parameters as needed [ased]
6. Auto gain adjust [rga]
7. Start measurement [zg]

** auto shim -overnight 1D expt.
 ===== optional
 ** duplicate data set [sav]

Number of dummy scans
 Sweep width in Hz
 Acquisition time
 Receiver gain
 Dwell time
 Pre-scan
 F1 channel - high power pulse
 F1 channel - power level for pulse (default)

Example:
change ns according to concentration

Probe Adjustment: WOBB



```

II. Basic Optimization 1D Processing Oct-600
1.Recall standard shims [rsh shim.current]
2.Auto lock to solvent [lock]
3.Use Keypad to Shim
  ** 600MHz only [topshim]
4.Tune probe* [wobb]
5.Modify parameters as needed [ased]
6.Auto gain adjust [rga]
7.Start measurement [zg]
** auto shim -overnight 1D expt.
==== optional
** duplicate data set [sav]
  
```

To minimize the lost of electronic signals to and from the spectrometer to the NMR sample

Link: Tune-probe-win2010

To be discussed in Lecture 2 & Lab 2.

Skip the details for First Lecture & Lab 1.



Finally-- adjust Receiver sensitivity



```

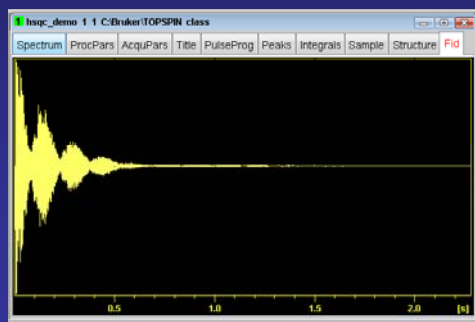
UM Data Collection Quick Auto
Reset shims [rsh]
Lock stabilization [lock]
Adjust shims [shim]
Modify parameters [ased]
Adjust electronic gain [rga]
Start NMR expt [zg]
  
```

•RGA

•Adjust the electronic detector sensitivity (should be done after all parameters are defined)

•ZG

•Zero (overwrites) previous FID page memory (if any) and start new scans (go) for NS.



What if I change my mind?

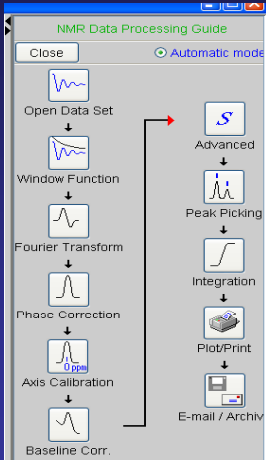
Type "halt"

Data collected will be saved in FID page and available for processing.

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One dimensional NMR data processing


Use either the 1D-processing Pull down menu





1/4/2010

or

Quick Short Cuts



Video demo available in
NMR workstations



Further [reference](#): 1d-data-pro-win2010

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THE END
LECTURE 1

Lab group Assignment and time changes.

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