Unlock Solid-State NMR: TopSolids

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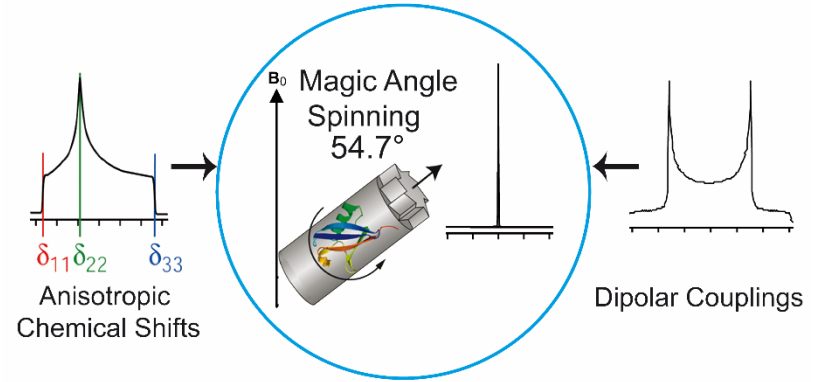
The Analytical Tool Solid-State NMR

- Solid-State NMR is often the only method of choice for solid samples when other analytical techniques cannot provide an answer:
  - Long-range order (X-ray crystallography), IR and Raman Spectroscopy and DTGA and related methods

- Uses Magic Angle Spinning (MAS) of the sample to average unwanted interactions resulting in resolved NMR signals

- Powerful method in many research areas:
  - ✓ Batteries
  - ✓ Polymers
  - ✓ Proteins
  - ✓ Pharmaceuticals



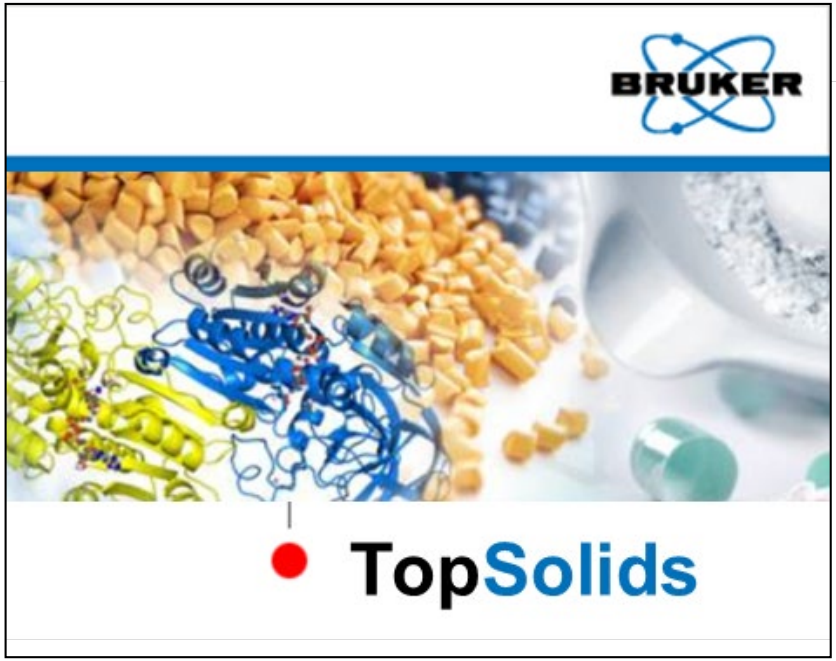
Solid-State NMR – Cliff Hanger.

- Manual probe setup (magic angle-setting, shimming)
- Thinking about, calculating & optimizing suited conditions (Hartmann-Hahn, ^{1}H Decoupling, etc.)
- Complicated parameter handling between different data sets

➢ Knowledge & expertise needed

Is Solid-state NMR - A method for experts only?

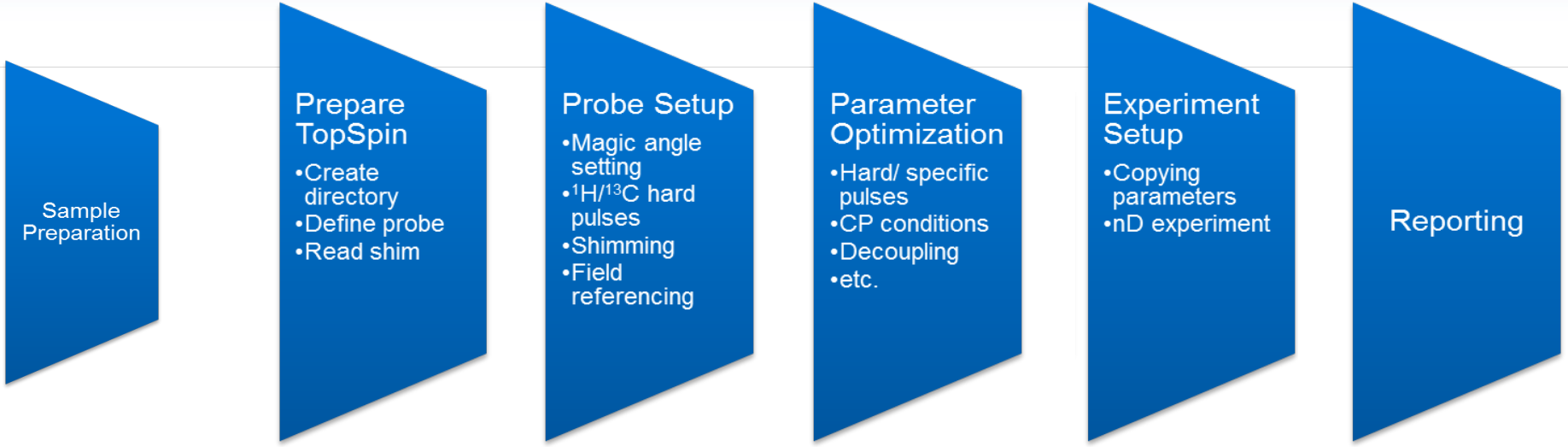
TopSolids: The Solid State NMR Expert



- The delicate setup of solid-state NMR experiments is made easy
- By reliable automated procedures
- Whatever your background and experience may be

- 2nd generation available with TopSpin 3.5.6 and TopSpin 4
- Free of charge
- Compatible with 4 mm and 3.2 mm double/ triple channel SB/ WB probes

TopSolids– Workflow

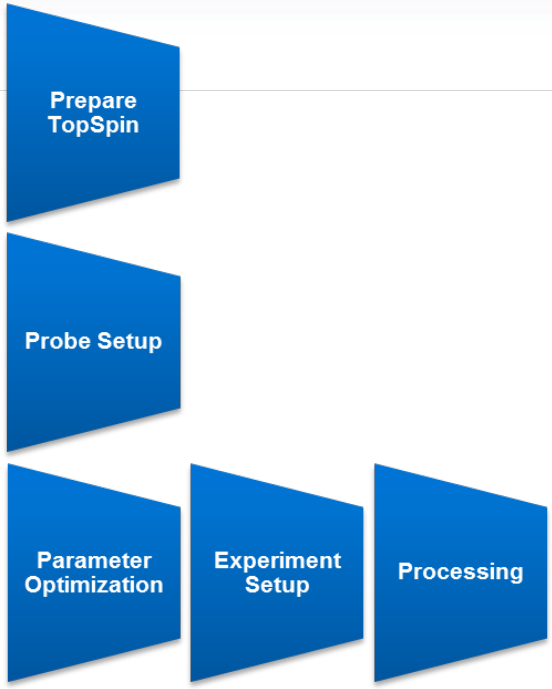


→ **TopSolids is workflow-oriented**

TopSolids – Flow Bar Modules

Different modules in flow bar design to ensure a proper workflow:

- ***Preparation*** (incl. advanced user interface to fetch general information in one step, import of former data possible)
- ***Probe Setup*** (not only for general setup, but for e.g. testing a probe after repair, etc.)
- ***Applications***:
  - ***Proteins*** (incl. setup and optimization, up to 4D ^{13}C-detected experiments)
  - ***Materials*** (1D/ 2D X, HX CP, HetCor and MQMAS experiments)

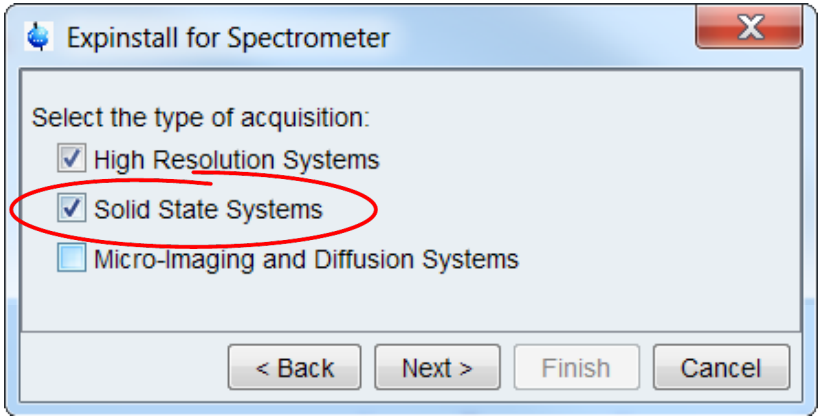


TopSolids – Parameter Handling

TopSolids parameter handling is based on:

1. Parameter sets

Experiments with pre-defined starting values



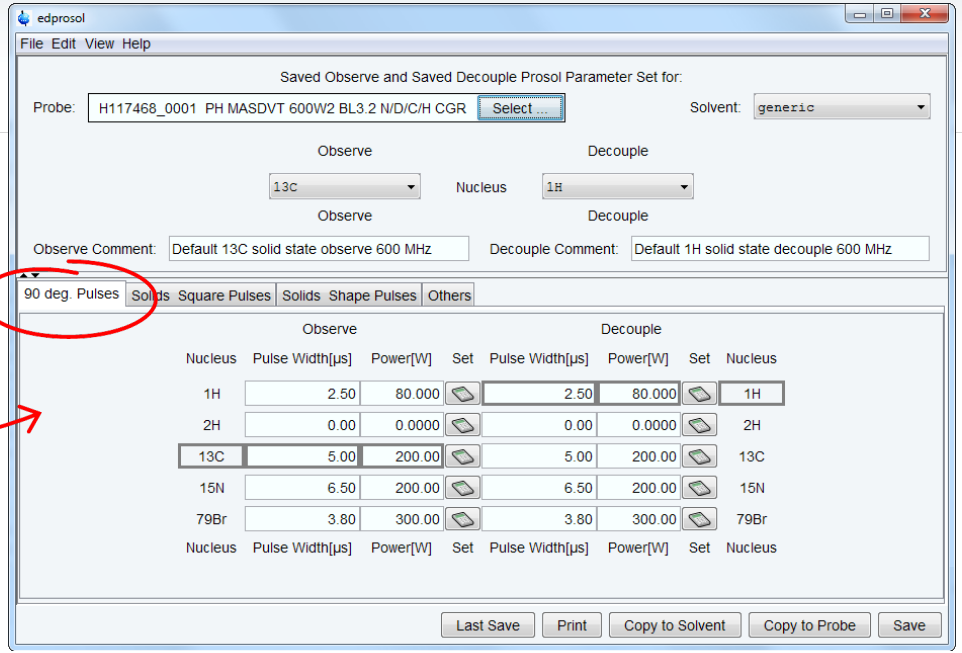
Do not forget to run '*expinstall*" for solid systems once…

TopSolids – Parameter Handling

TopSolids parameter handling is based on:

1. Parameter sets
2. PROSOL table ('edprosol')

Enter 90° pulse parameters for nuclei (^{1}H, ^{13}C, X)

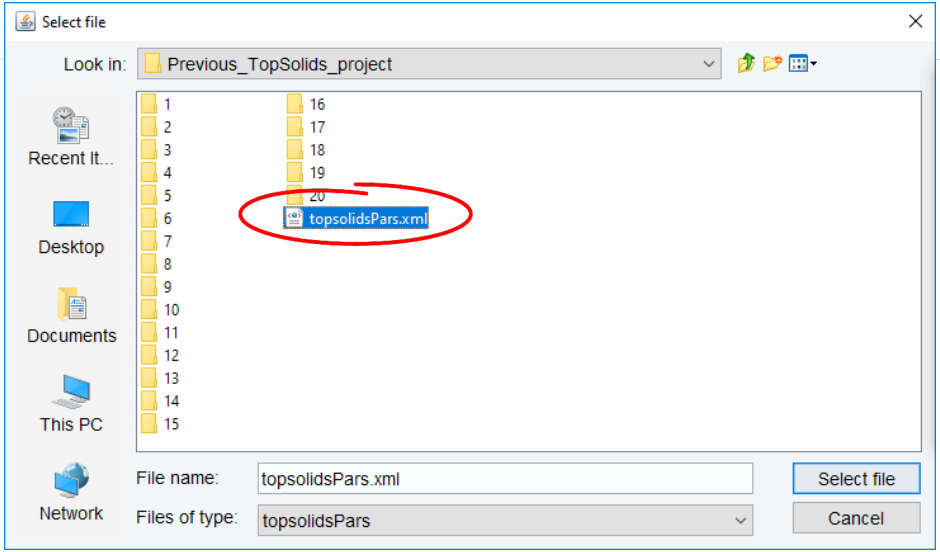


TopSolids – Parameter Handling

TopSolids parameter handling is based on:

1. Parameter sets
2. PROSOL table (‘edprosol’)
3. Project-specific “parameter data bank”

- ‘*topsolidsPars.xml*’
- Automatically created & managed by TopSolids (not meant for manual editing)
- Stored in the project’s directory
- Used to:
  - Store optimized parameters for later use
  - Import parameters from a previous project
  - Create PDF report



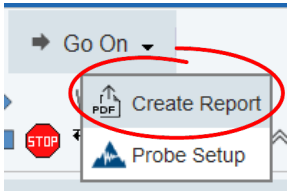
TopSolids – Parameter Handling

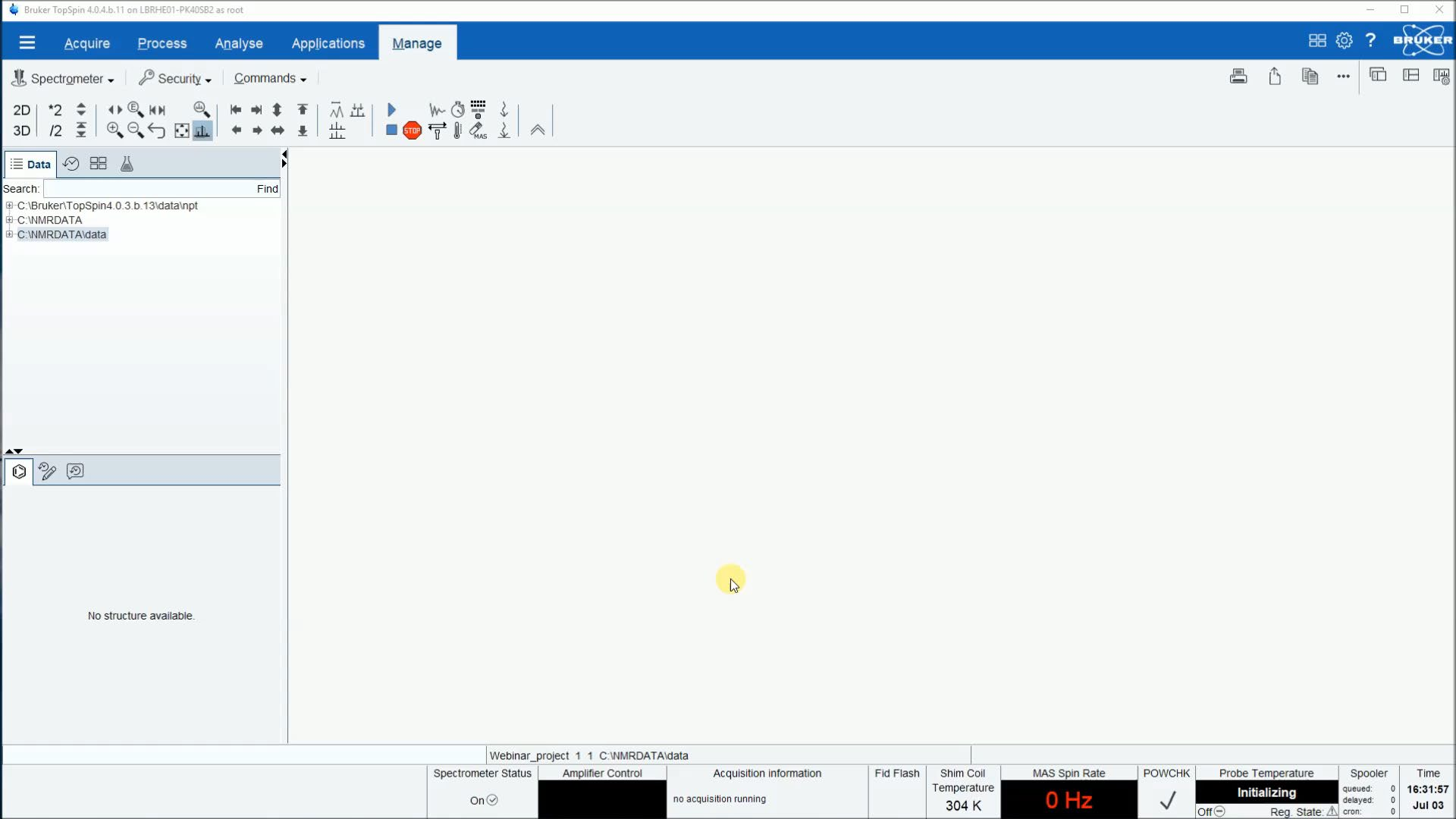
TopSolids parameter handling is based on:

1. Parameter sets
2. PROSOL table (‘edprosol’)
3. Project-specific “parameter data bank”

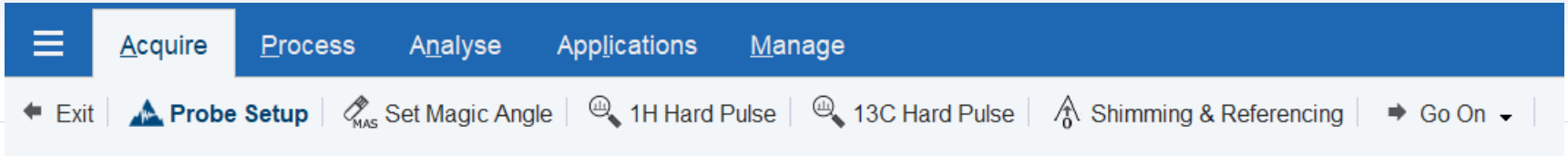
- Ensures reasonable values for parameters at any time – whether optimized or not yet
- Determined parameters are stored in the parameter data bank for further usage
- Parameters can be reviewed in form of a PDF report at any time to keep track and to assist fast publication

→ Let’s get started and create a TopSolids project…



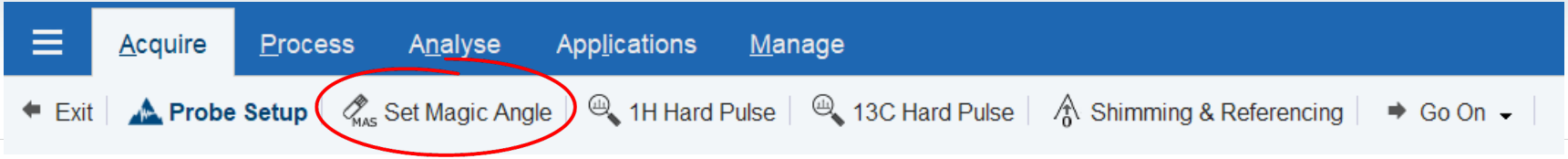

Bruker TopSpin 4.0.4.b.11 on LBRHE01-PK40SB2 as root
Acquire
Process
Analyse
Applications
Manage
Spectrometer
Security
Commands
2D
3D
*2
/2
Data
Search:
Find
C:\Bruker\TopSpin4.0.3.b.13\data\npt
C:\NMRDATA
C:\NMRDATA\data
No structure available.
Webinar_project 1 1 C:\NMRDATA\data
Spectrometer Status
On
Amplifier Control
Acquisition information
no acquisition running
Fid Flash
Shim Coil Temperature
304 K
MAS Spin Rate
0 Hz
POWCHK
Probe Temperature
Initializing
Off
Reg. State:
Spooler
queued: 0
delayed: 0
cron: 0
Time
16:31:57
Jul 03

TopSolids – Probe Setup Flow Bar

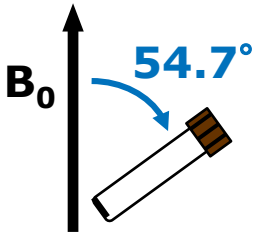


- Probe performance needs to be tested on a regular basis to ensure best results
- Though routine work, it can be time consuming - even for an NMR expert
- The ***Probe Setup*** module offers a high level of convenience to:
  - ✓ Setting the magic angle
  - ✓ Optimizing ^{1}H and ^{13}C hard pulses on standard samples
  - ✓ Shimming the probe and referencing the magnetic field
  - ✓ With least user interaction to overcome the necessity for extensive prior knowledge
  - ✓ Only two samples needed: KBr, adamantane

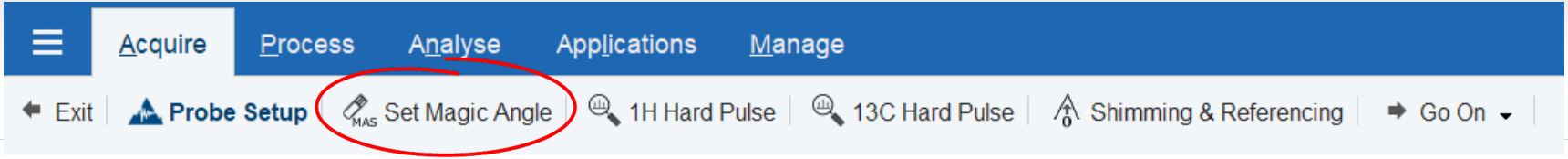
Probe Setup – Set Magic Angle



- On KBr (^{79}Br) at 5 kHz MAS TopSolids:
  - ✓ Checks for fixed frequency H/C/N probes to adjust respective parameter sets automatically
  - ✓ Detects Cover Module 2, which can be used for MA adjustment at the spectrometer directly – if wanted
  - ✓ Records a reference spectrum and evaluates the quality of the MA setting
  - ✓ Performs an angle-stability test for SB probes
  - ✓ Checks final quality

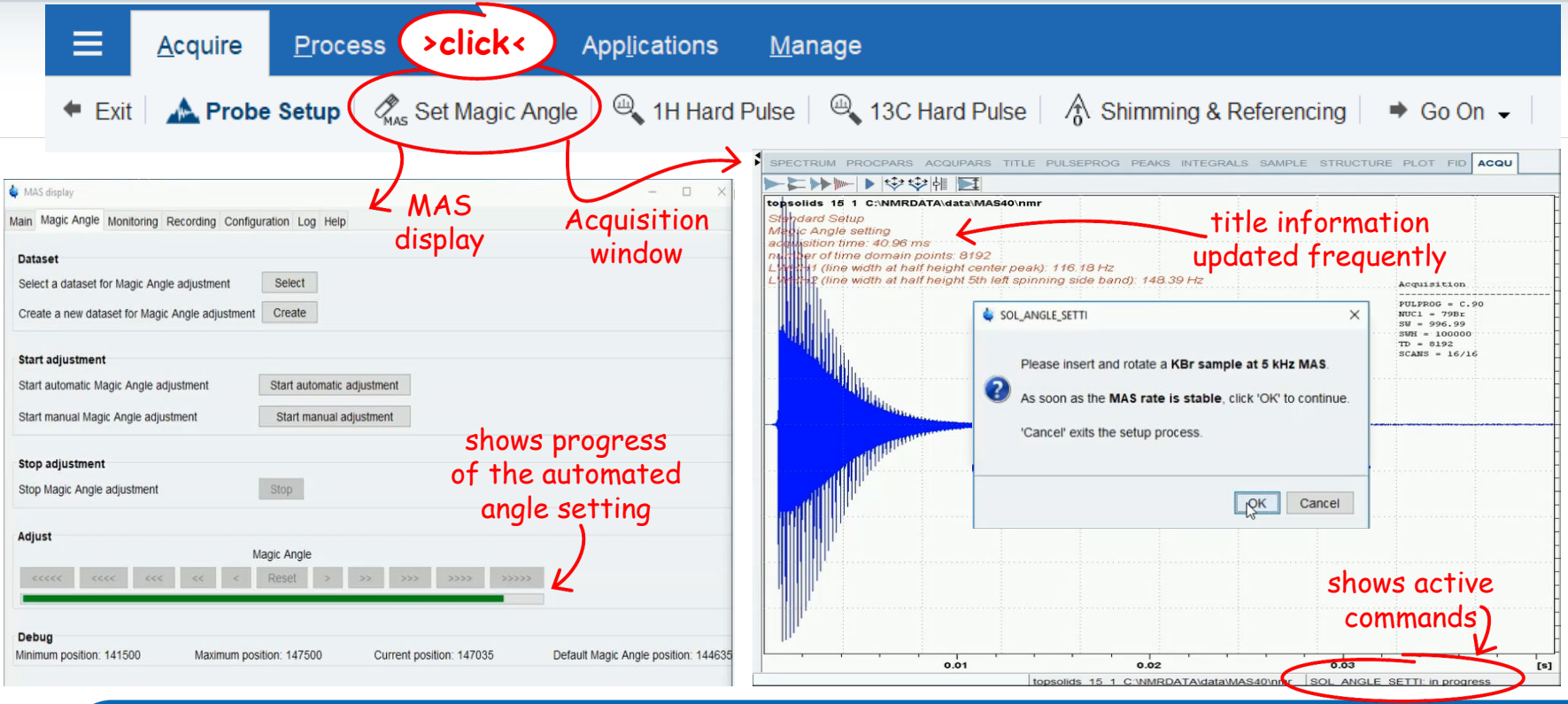


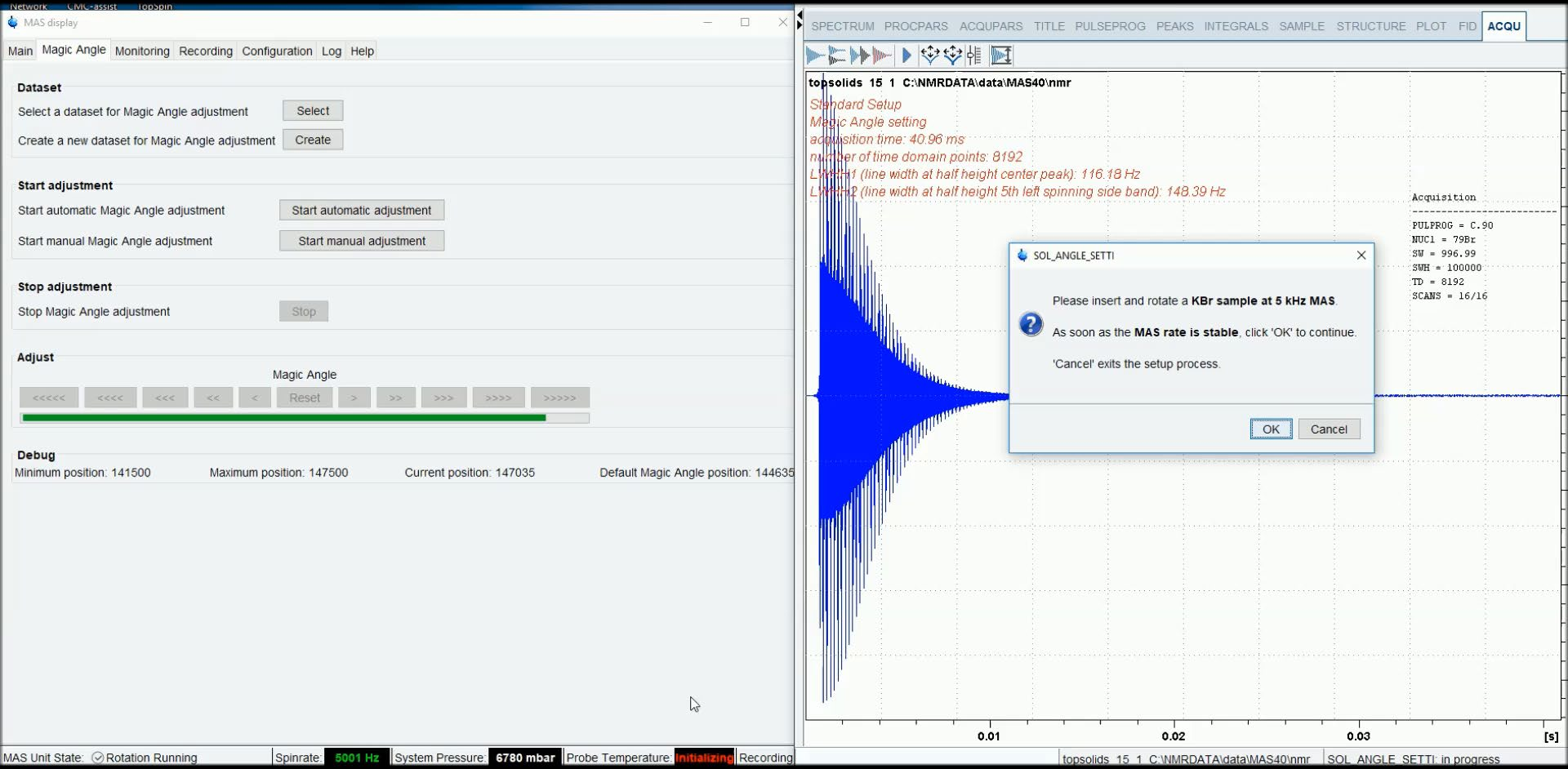
Probe Setup – Set Magic Angle using the *iProbe*



- Using an iProbe, TopSolids:
  - ✓ Automatically tunes & matches the probe
  - ✓ Automatically adjusts the angle setting
  - ✓ Records a reference spectrum and evaluates the quality of the MA setting
  - → No manual interactions required

Probe Setup – Set Magic Angle using the *iProbe*




MAS display
Main
Magic Angle
Monitoring
Recording
Configuration
Log
Help
Dataset
Select a dataset for Magic Angle adjustment
Select
Create a new dataset for Magic Angle adjustment
Create
Start adjustment
Start automatic Magic Angle adjustment
Start automatic adjustment
Start manual Magic Angle adjustment
Start manual adjustment
Stop adjustment
Stop Magic Angle adjustment
Stop
Adjust
Magic Angle
<<<<<
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Reset
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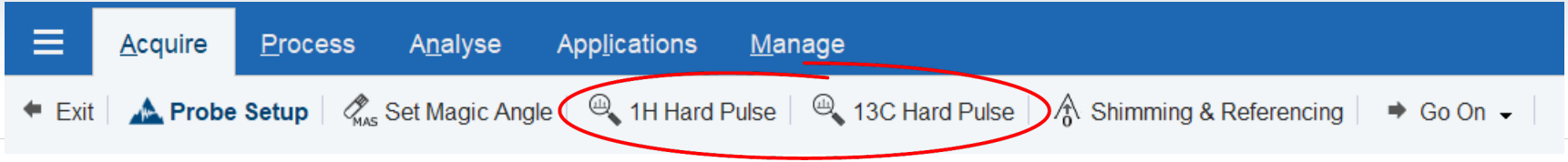
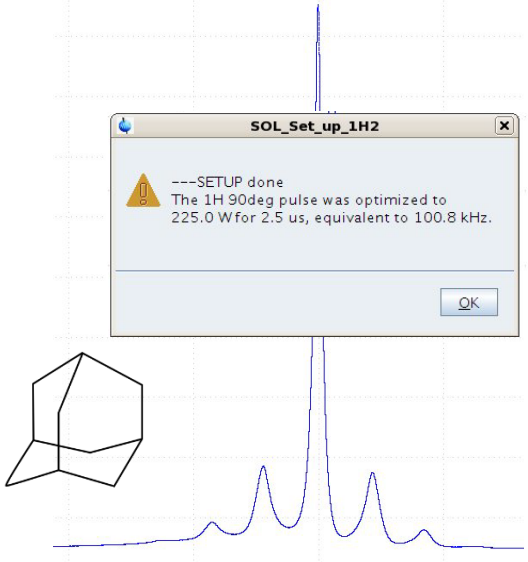
Debug
Minimum position: 141500
Maximum position: 147500
Current position: 147035
Default Magic Angle position: 144635
MAS Unit State:
Rotation Running
Spinrate:
5001 Hz
System Pressure:
6780 mbar
Probe Temperature:
Initializing
Recording
SPECTRUM
PROCPARS
ACQUPARS
TITLE
PULSEPROG
PEAKS
INTEGRALS
SAMPLE
STRUCTURE
PLOT
FID
ACQU
topsolids 15 1 C:\NMRDATA\data\MAS40\nmr
Standard Setup
Magic Angle setting
acquisition time: 40.96 ms
number of time domain points: 8192
LW@-1 (line width at half height center peak): 116.18 Hz
LW@-2 (line width at half height 5th left spinning side band): 148.39 Hz
Acquisition
PULPROG = C.90
NUC1 = 79Br
SW = 996.99
SWH = 100000
TD = 8192
SCANS = 16/16
SOL_ANGLE_SETTI
Please insert and rotate a KBr sample at 5 kHz MAS.
As soon as the MAS rate is stable, click 'OK' to continue.
'Cancel' exits the setup process.
OK
Cancel
0.01
0.02
0.03
[s]
topsolids 15 1 C:\NMRDATA\data\MAS40\nmr
SOL_ANGLE_SETTI: in progress

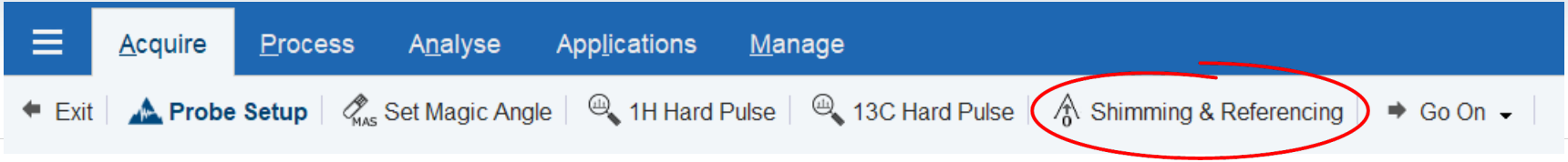
Probe Setup – Hard Pulse Optimization



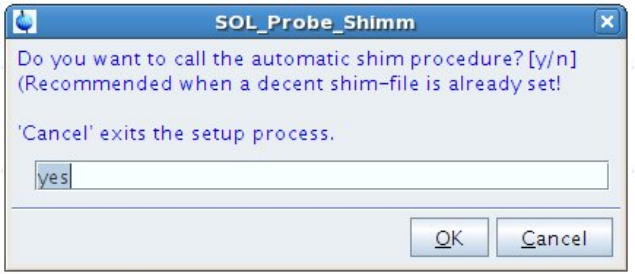
- Determining the 90 degree pulses for ^{1}H & ^{13}C is:
  - ✓ Done automatically
  - ✓ On adamantane at 10 kHz MAS
  - ✓ Using the script routine '*pulsecal*' (known from solution NMR, adjusted for TopSolids)
  - ✓ Using PROSOL values as starting parameters or data bank entries IF present (= imported)
  - ✓ Taking ~2 minutes per nucleus



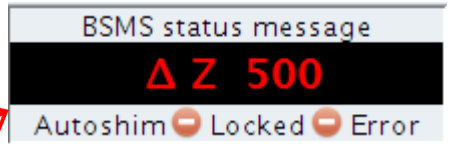
Probe Setup – Shimming & Referencing



- Shimming essential to ensure for well-resolved lines
- Done on adamantane at 10 kHz MAS
- Automated Z shimming routine available (2-5 min depending on starting values)
- Shim quality determined automatically & compared to starting values
- B_0 field is automatically referenced to neat TMS



Note: Z shim values will be updated by the routine



Bruker TopSpin 4.0.4.b.11 on LBRHE01-PK40SB2 as root

Acquire | Process | Analyse | Applications | Manage

Exit | Probe Setup | Set Magic Angle | 1H Hard Pulse | 13C Hard Pulse | Shimming & Referencing | Go On

Spectral calibration is recommended!
An automated *z shimming procedure* can be used to refine spectral resolution. Otherwise, manual shimming is initiated. Shim quality evaluation and spectral calibration (referenced to neat TMS) is done fully automated.

Data

Show Spectra Thumbnails

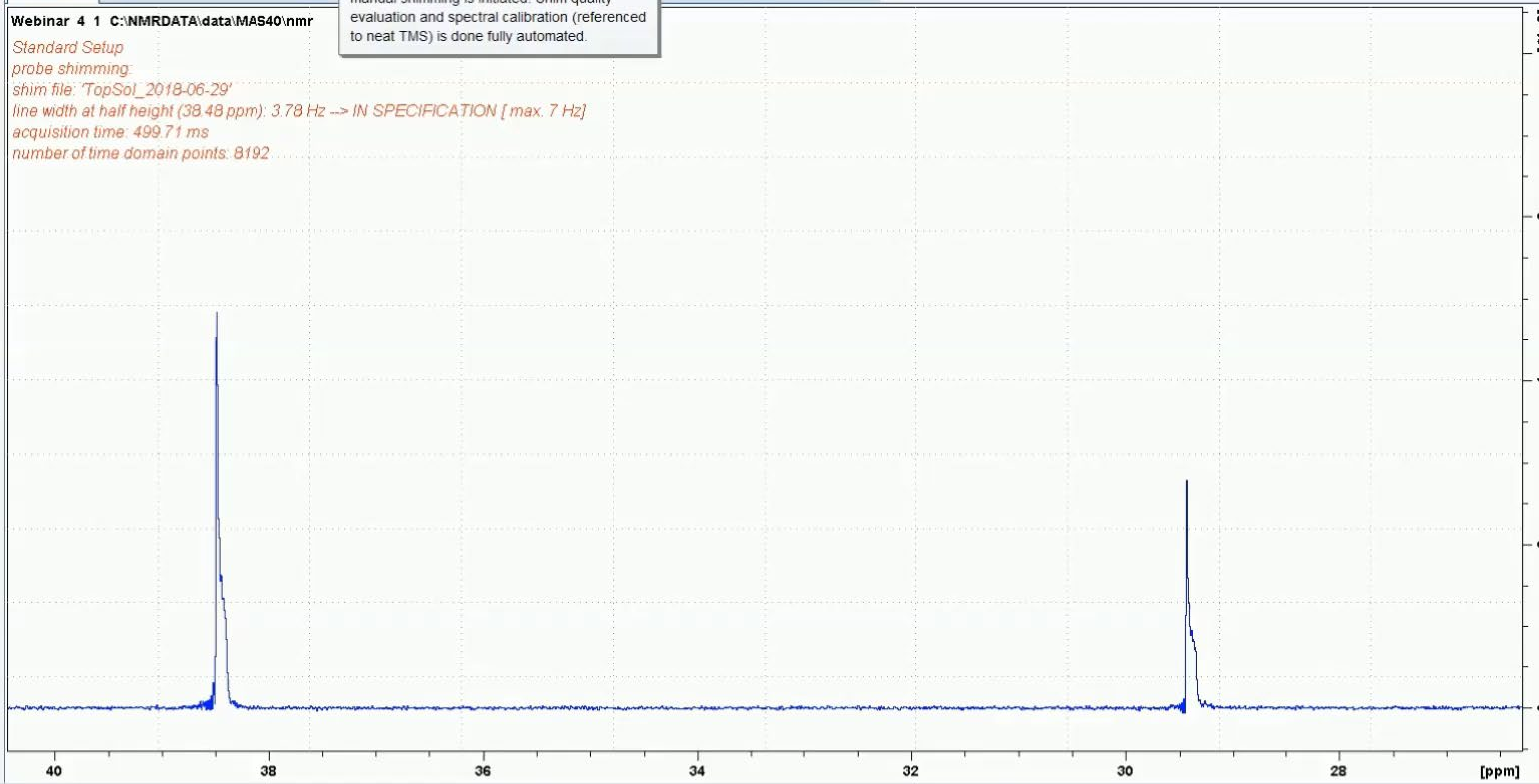
Webinar 4 1 C:\NMRDATA\data\MAS40\nmr - C.dp
Webinar 1 1 C:\NMRDATA\data\MAS40\nmr - Hpul
test 1 1 C:\NMRDATA\data\MAS40\nmr - C.90, Sta
Webinar 2 1 C:\NMRDATA\data\MAS40\nmr - C.dp
Webinar 3 1 C:\NMRDATA\data\MAS40\nmr - C.dp
euromar 1 1 C:\NMRDATA\data\MAS40\nmr - Hpul
topsolids 20 1 C:\NMRDATA\data\MAS40\nmr - Hp
topsolids 19 1 C:\NMRDATA\data\MAS40\nmr - Hp
topsolids 18 1 C:\NMRDATA\data\MAS40\nmr - Hp
topsolids 17 1 C:\NMRDATA\data\MAS40\nmr - C.
topsolids 16 1 C:\NMRDATA\data\MAS40\nmr - C.
topsolids 15 1 C:\NMRDATA\data\MAS40\nmr - C.
topsolids 14 1 C:\NMRDATA\data\MAS40\nmr - C.
topsolids 13 1 C:\NMRDATA\data\MAS40\nmr - C.
topsolids 12 1 C:\NMRDATA\data\MAS40\nmr - C.
topsolids 11 1 C:\NMRDATA\data\MAS40\nmr - C.

No structure available.

SPECTRUM | PROCPARS | ACQUPARS | TITLE | STRUCTURE | PLOT | FID | ACQU

Webinar 4 1 C:\NMRDATA\data\MAS40\nmr

Standard Setup
probe shimming:
shim file: 'TopSol_2018-06-29'
line width at half height (38.48 ppm): 3.78 Hz --> IN SPECIFICATION [max. 7 Hz]
acquisition time: 499.71 ms
number of time domain points: 8192



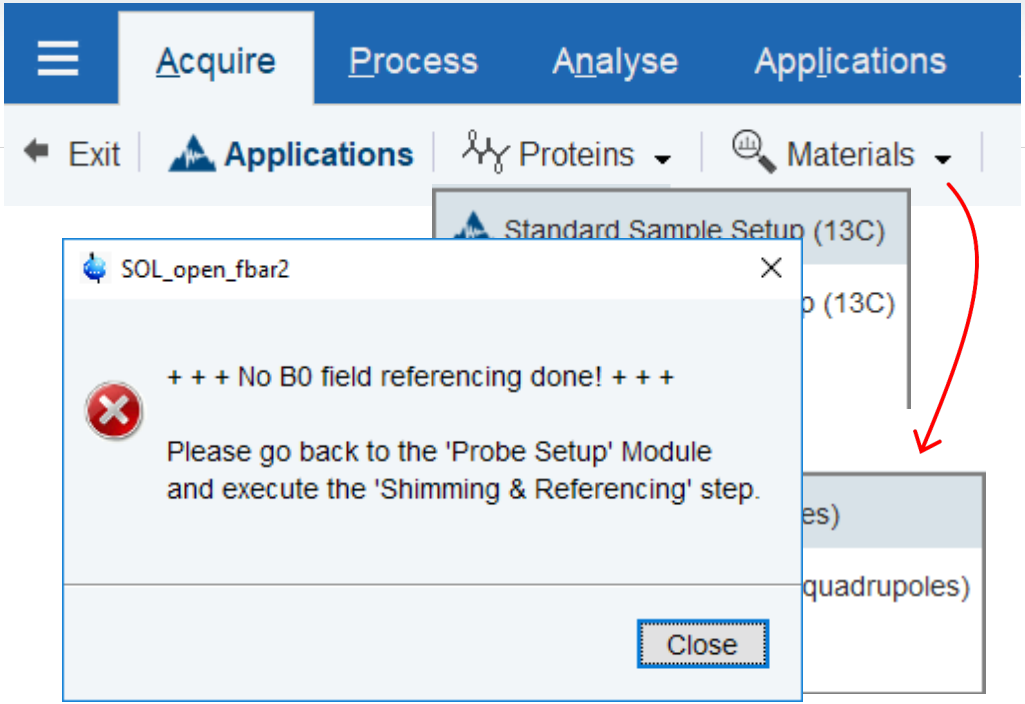
Webinar 4 1 C:\NMRDATA\data\MAS40\nmr | Region saved to parameters: F1P/F2P

Spectrometer Status	Amplifier Control	Acquisition information	Fid Flash	Lock	Sample	Shim Coil Temperature	MAS Spin Rate	POWCHK	Probe Temperature	No Channel selected	Spooler	BSMS status message	Time
On		no acquisition running				302 K	10000 Hz	✓	Initializing	No channel or auxiliary channel selected	queued: 0 delayed: 0	Δ Z 200	15:39:41 Jun 29

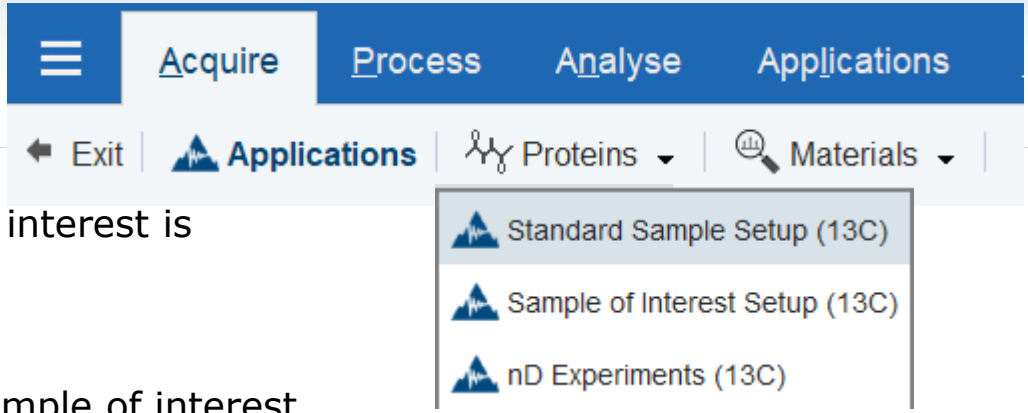
TopSolids – Applications Module

- Can be opened as soon as ‘Shimming & Referencing’ in the ‘Probe Setup’ module has been performed ✓

- Dedicated workflows for:
  - ✓ Protein samples
  - ✓ Non-protein samples (‘Materials’)



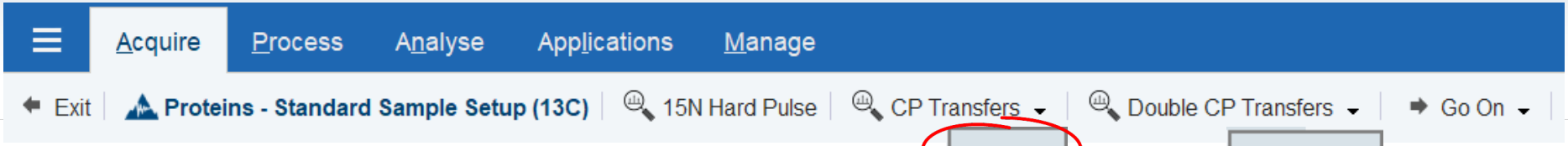
TopSolids – Protein Applications



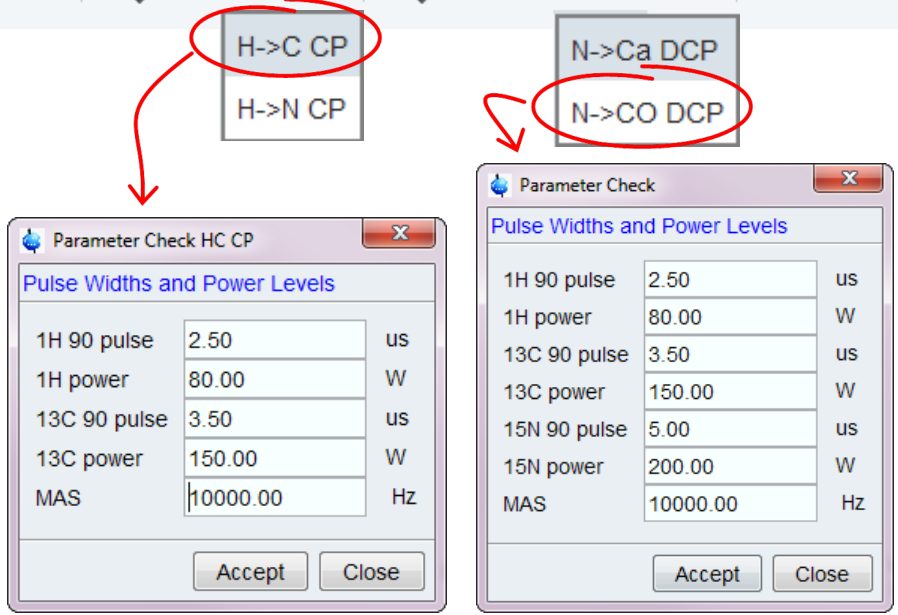
Whole setup divided into three modules:

1. 'Standard Sample Setup'
   - Should be performed if the sample of interest is giving poor signal to noise
2. 'Sample of Interest Setup'
   - (Re-) optimize parameters on your sample of interest
   - Optional optimizations: spectral widths, carrier frequencies, ^{1}H decoupling during acquisition
3. 'nD Experiments'
   - Actual experiments of interest
   - 1-click-optimization possible → does no longer rely on prior optimization

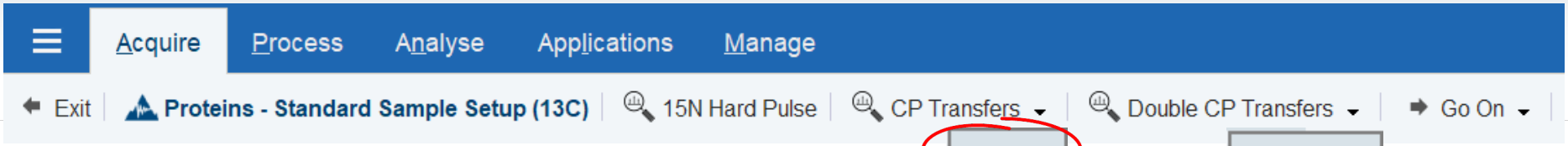
Protein Applications – Standard Sample Setup



- Standard sample: [^{13}C, ^{15}N]-glycine
- For NCO transfer: any [^{13}C, ^{15}N]-peptide (e.g. fMLF) or standard protein (e.g. SH3, GB1, ubiquitin)
- Cross polarization (CP) transfers:
  - 'Parameter Check' based on optimized hard pulses & current MAS rate
    - For MAS III units the rate is detected automatically
    - Else: put in manually

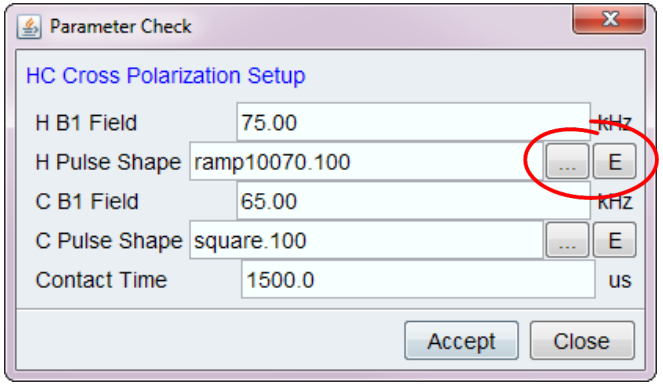


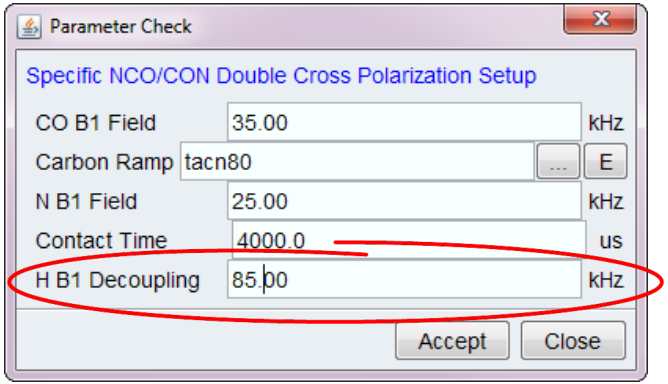
Protein Applications – Standard Sample Setup



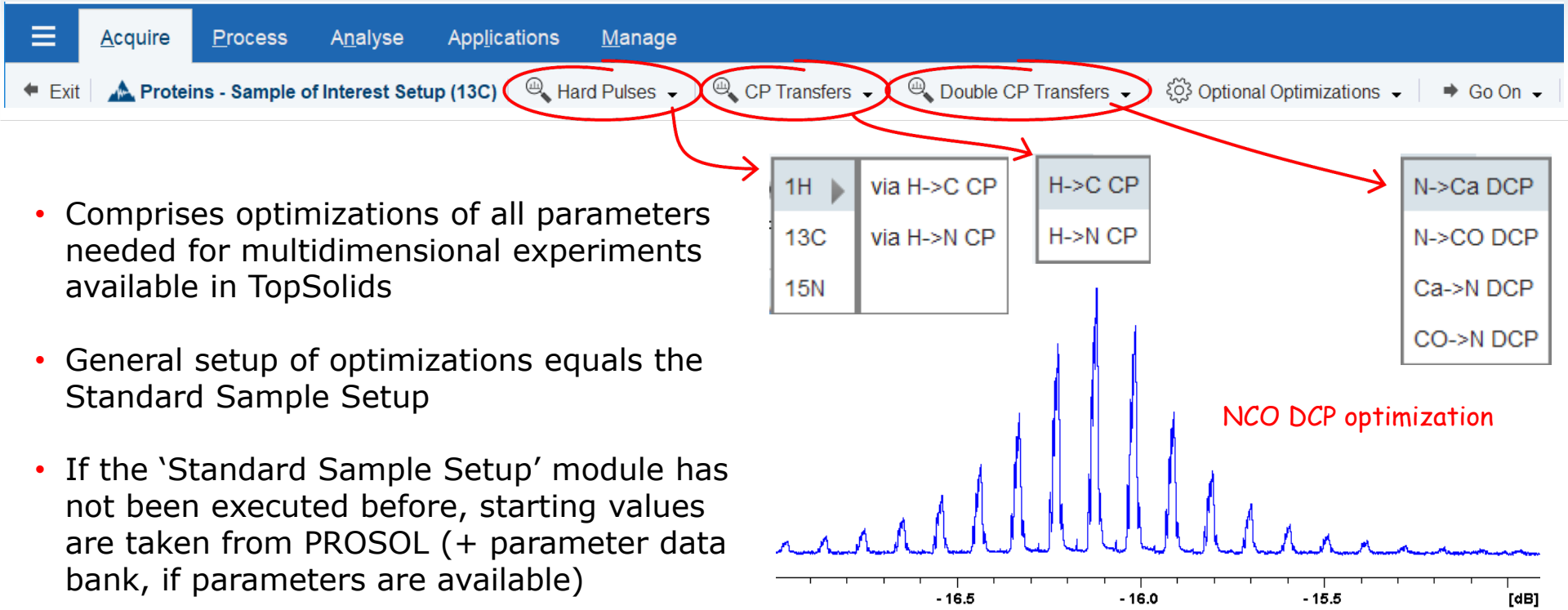
Based on 'Parameter Check' TopSolids suggests a suitable Hartmann-Hahn condition:

- ✓ Checks power limits
- ✓ Checks for unwanted conditions
- ✓ Free choice of shape
- ✓ Takes into account ^{1}H decoupling during double CP



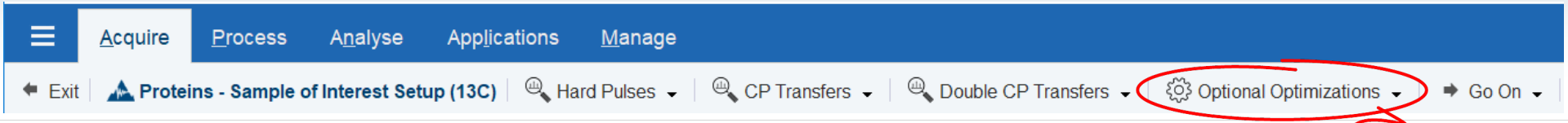


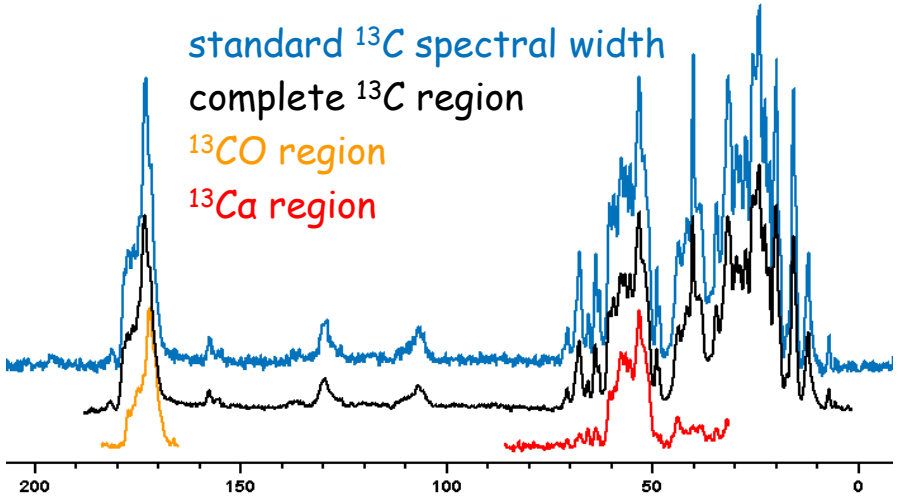
Protein Applications – Sample of Interest Setup

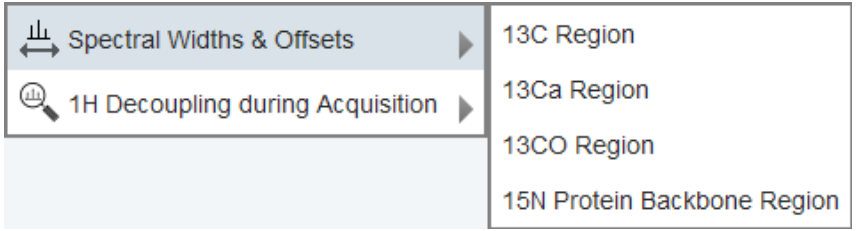


- Comprises optimizations of all parameters needed for multidimensional experiments available in TopSolids
- General setup of optimizations equals the Standard Sample Setup
- If the 'Standard Sample Setup' module has not been executed before, starting values are taken from PROSOL (+ parameter data bank, if parameters are available)

Protein Applications – Sample of Interest Setup

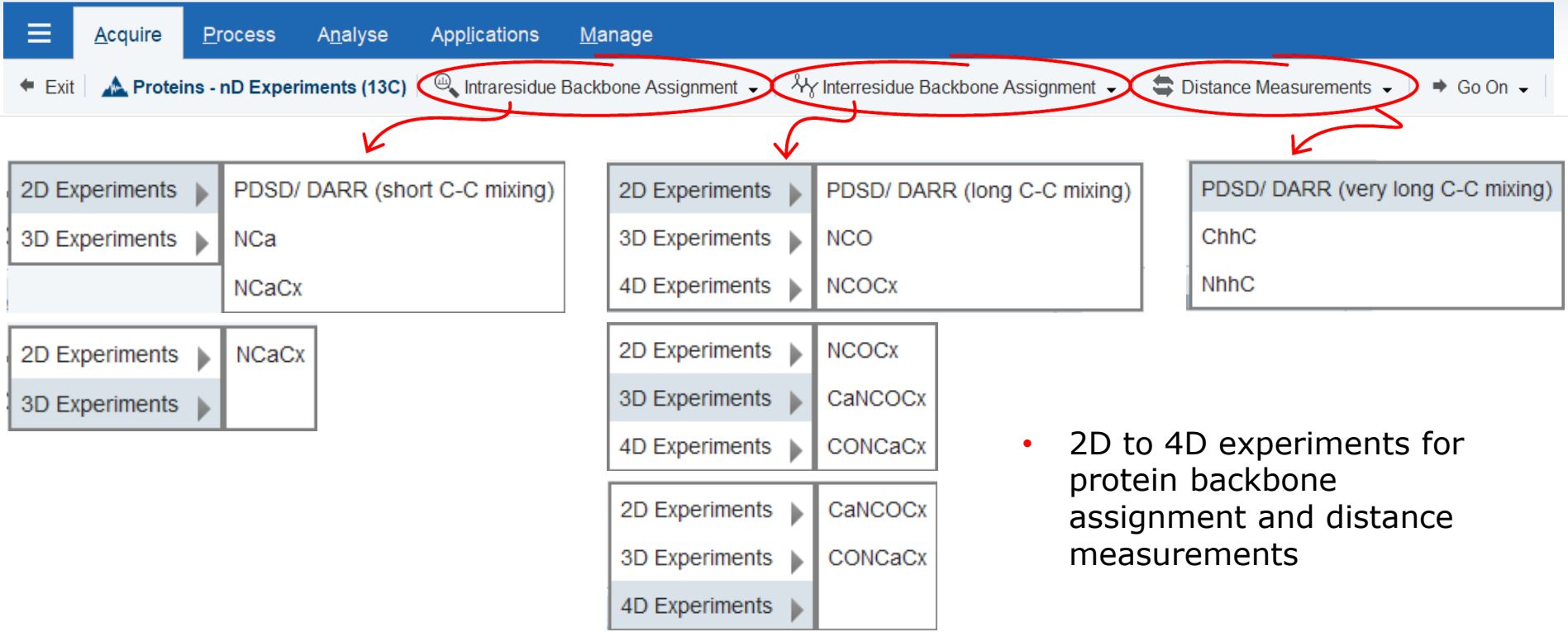






- The smaller a spectral width in an indirect dimension, the shorter the experimental measurement time of an nD experiment
- TopSolids optimizes spectral widths & carrier frequencies fully automated - based on CP experiments

Protein Applications – nD Experiments Module



- 2D to 4D experiments for protein backbone assignment and distance measurements

nD Experiments Module – ‘1-Click-Optimization’

- Do I need to go through all these optimizations if I only want to record one specific nD experiment?
- Which parameters do I need for recording a 3D NCOCx experiment?

✓ Skip the Sample Setup modules & start directly with the ‘nD Experiments’ using ‘1-click-optimization’!

✓ Select an nD experiment of interest in the ‘nD Experiments’ module (e.g. 3D NCOCx)

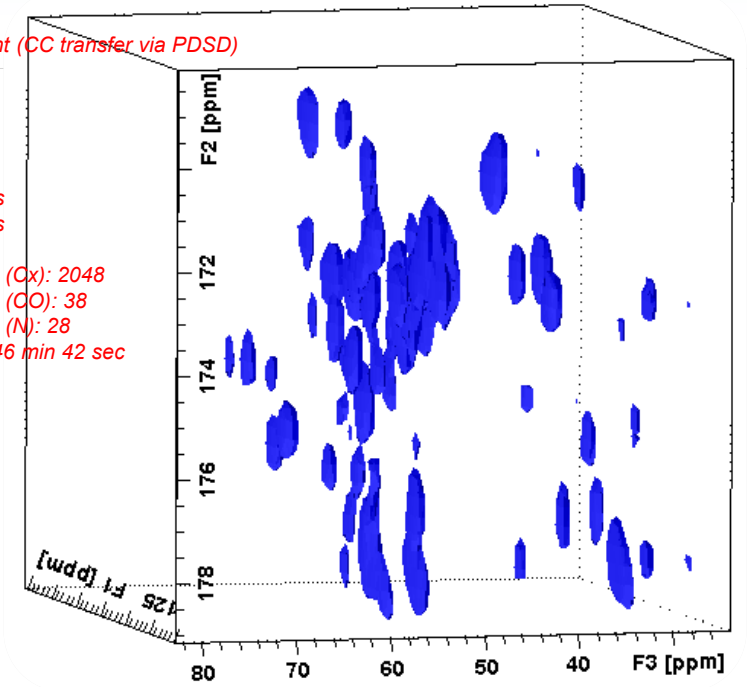
✓ TopSolids automatically detects missing parameters & initiates the respective optimization directly using least user interaction

✓ Once all parameters have been optimized, it will go back to the nD experiment to support its set up

nD Experiments Module – '1-Click-Optimization'

Spectrum | ProcPars | AcquPars | Title | PulseProg | Peaks | Integrals | Sample | Structure | Plot | Fid | Acqu

Backbone Assignment
3D NCOCx correlation experiment (CC transfer via PDSD)
on biological sample of interest
CO-Cx mixing time: 30 ms
MAS rate: 10.0 kHz
number of scans: 48
relaxation rate: 2.0 s
acquisition time F3(Cx): 22.53 ms
acquisition time F2 (CO): 5.70 ms
acquisition time F1 (N): 5.60 ms
number of time domain points F3 (Cx): 2048
number of time domain points F2 (CO): 38
number of time domain points F1 (N): 28
total experimental time: 1 d 5 h 46 min 42 sec
experiment in queue
experiment finished



- 3D NCOCx recorded with 1-click-optimized parameters
- Title includes information about acquisition parameters
- Processing was done fully automated by TopSolids after acquisition was finished
- TopSolids automatically queues experiments in the spooler when acquisition is active

Processing

TopSolids – Materials Applications

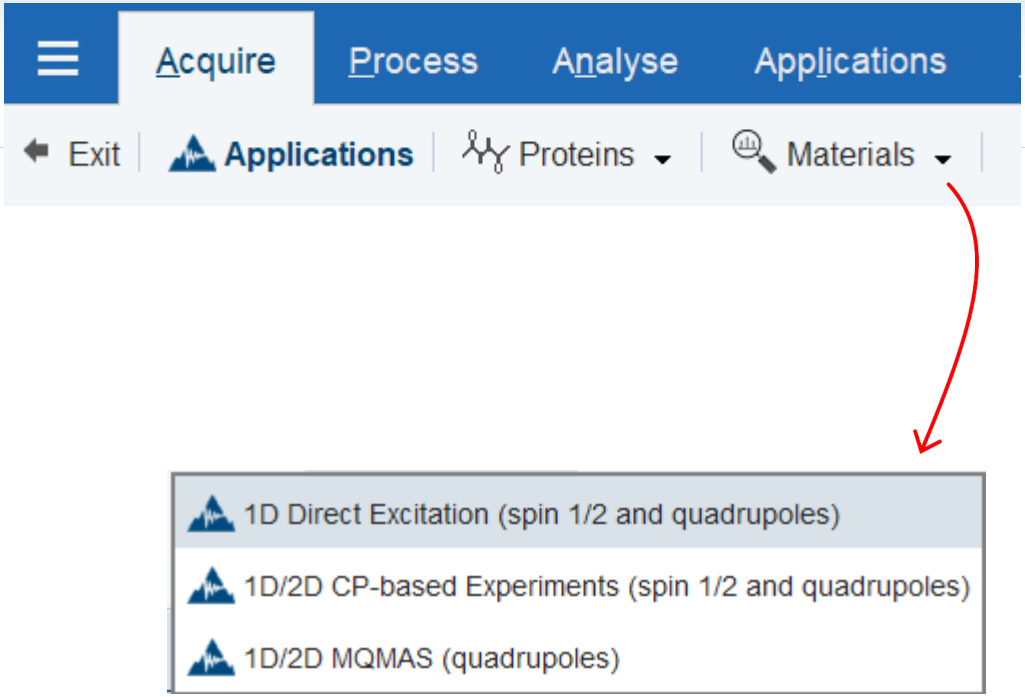
- Any X nucleus of choice the mounted probe can handle

  → TopSolids automatically detects, if the chosen X nucleus is:

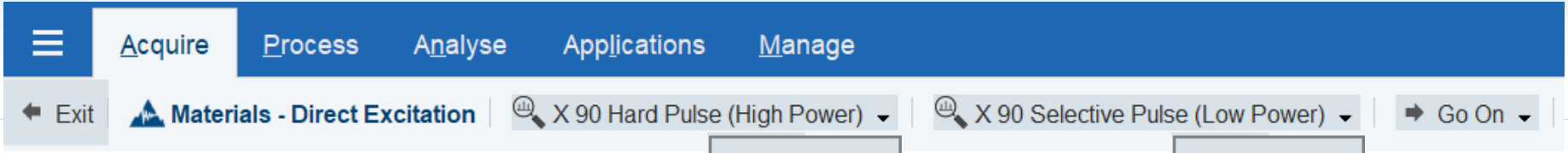
  - A spin ½ nucleus
  - A quadrupolar nucleus
  - Compatible with the chosen probe

- Optimizations available for:

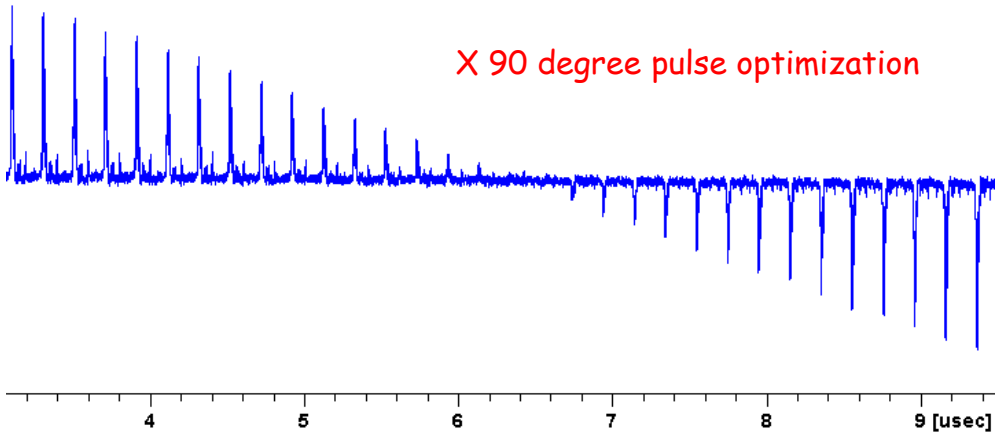
  ✓ X 90 degree pulse
  ✓ HX Cross Polarization (CP)
  ✓ 2D HetCor
  ✓ 1D/ 2D MQMAS



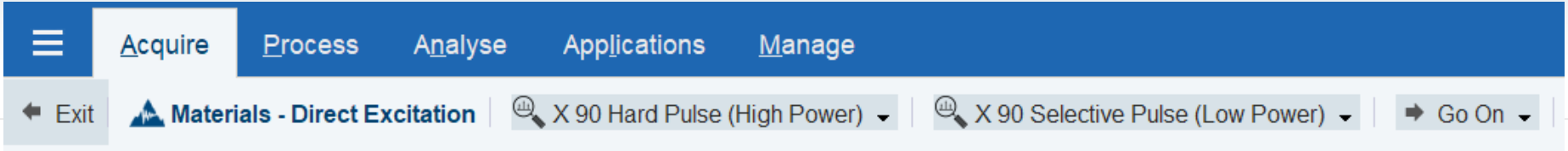
Materials – Direct Excitation Module



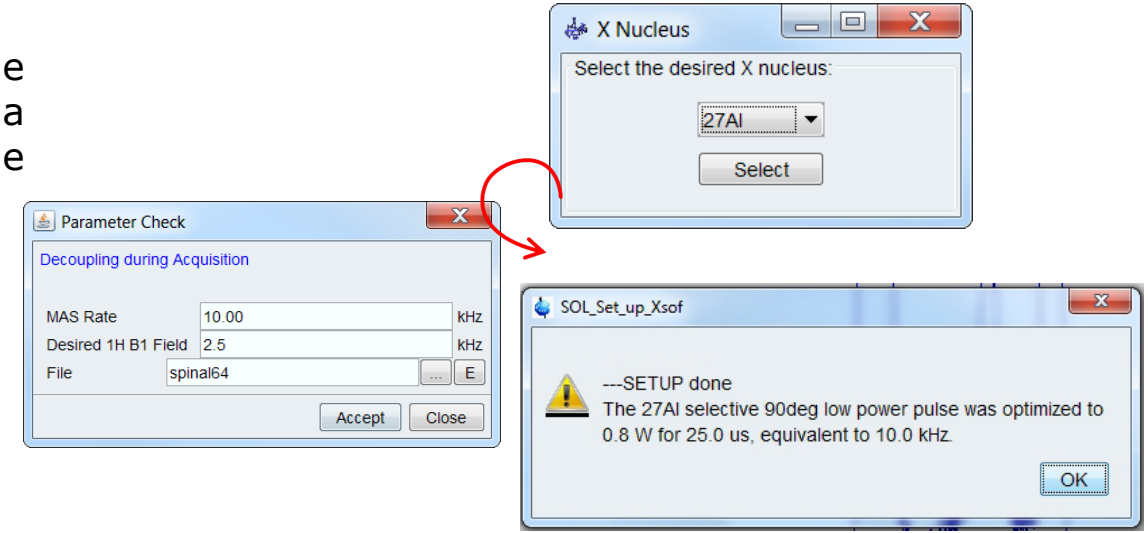
- Guides through basic setup routine of 90 degree pulse calibration on a standard samples and/ or the sample of interest



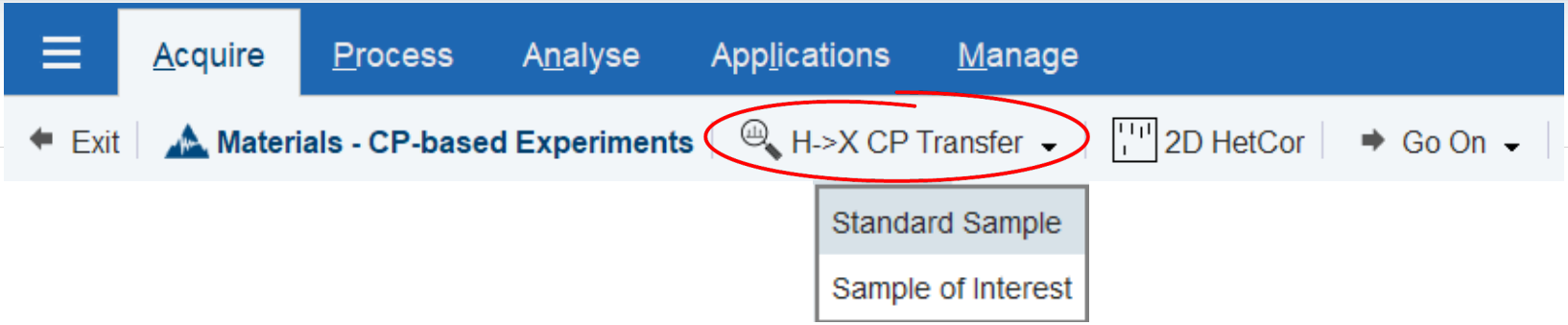
Materials – Direct Excitation Module



- Guides through basic setup routine of 90 degree pulse calibration on a standard samples and/ or the sample of interest
- X 90 degree pulse optimization either as a high or a low power pulse, depending on the X nucleus of choice
- (High/ low power) ^{1}H decoupling optional

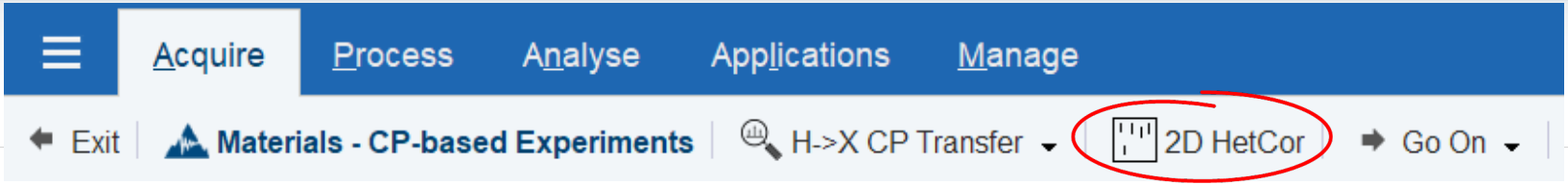


Materials – CP-based Experiments

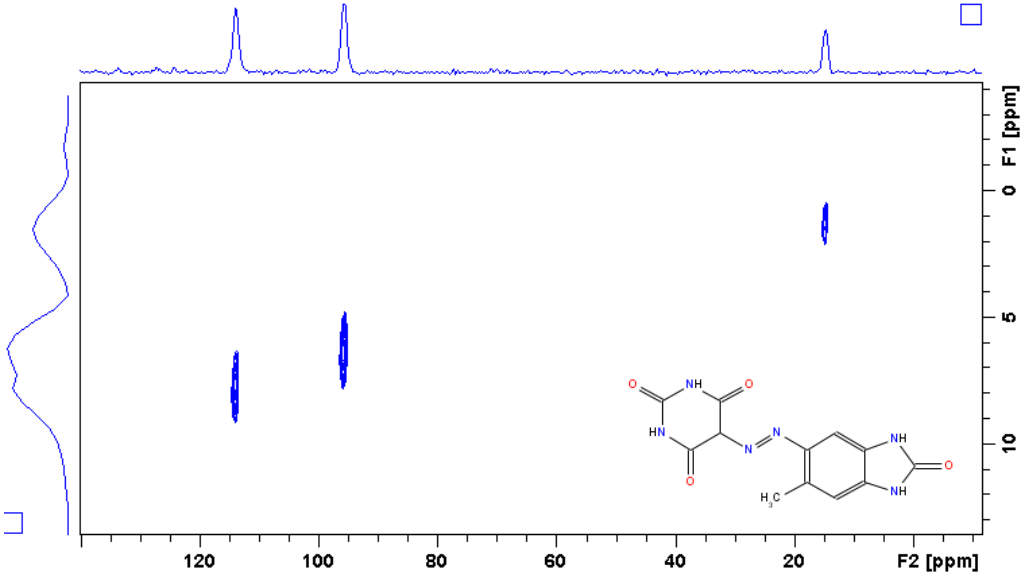


- Based on successful performance of X 90 degree pulse calibration
  - TopSolids automatically starts the '1-click-optimization' if missing
- CP transfer can be optimized on a standard samples and/ or the sample of interest
- CP conditions differ for spin ½ and quadrupolar nuclei
  - TopSolids is taking care to guarantee for best transfer efficiency

Materials – CP-based Experiments

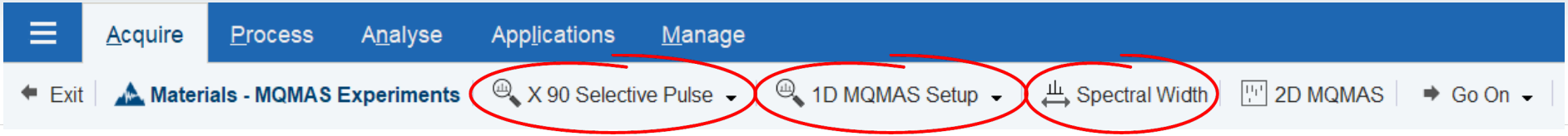


- Based on the H-X CP parameters a 2D H-X HetCor spectrum can be set up and recorded
- Spectrum automatically processed and phased

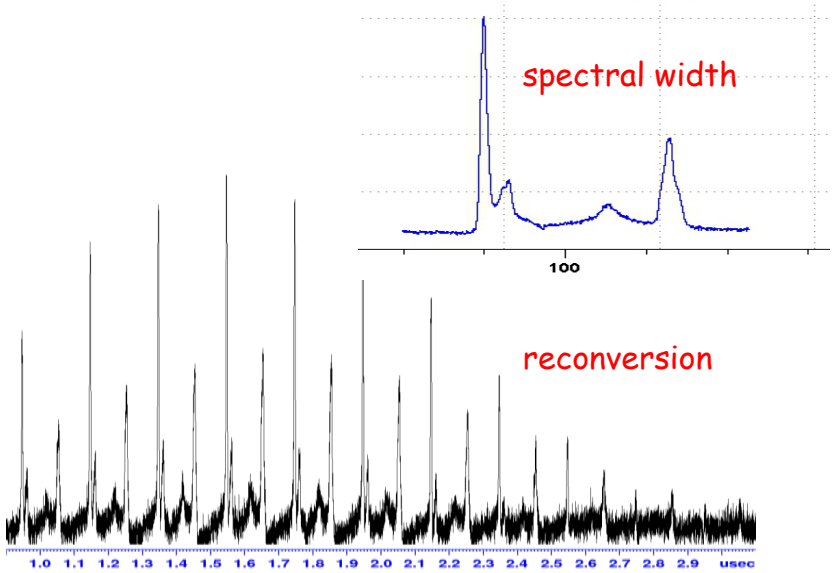


Processing

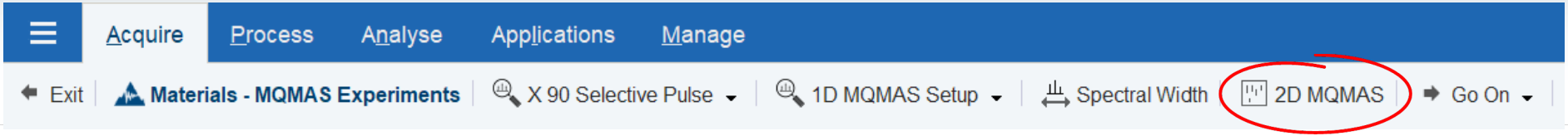
Materials – MQMAS Experiments



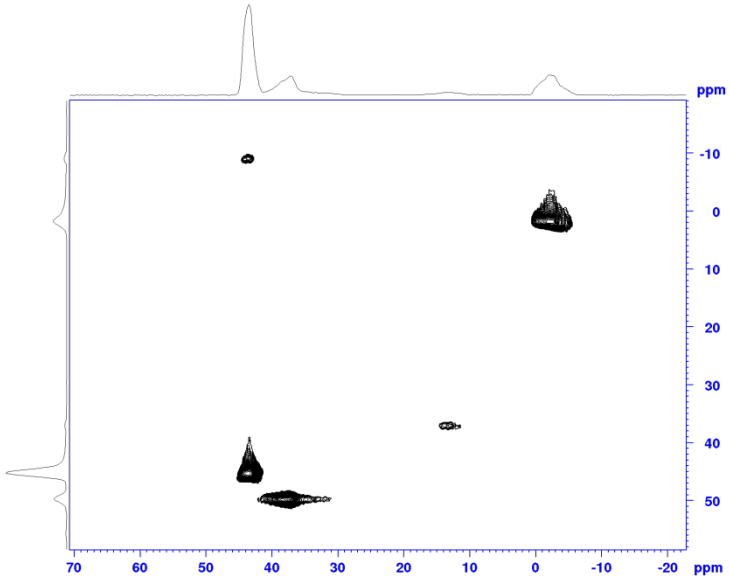
- Stand-alone module in the Materials Applications
- Guides stepwise through the setup to run a 2D MQMAS experiment:
  1. X 90 soft pulse (if not known already) on standard sample and/ or sample of interest
  2. Specific MQMAS excitation & reconversion pulse lengths for optimum sensitivity
3. Minimum spectral width to reduce 2D measurement when used as indirect dimension



Materials – MQMAS Experiments

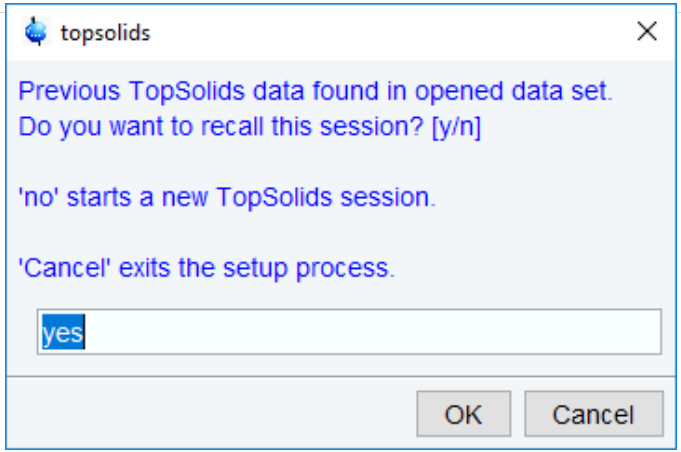


4. Stepwise 2D setup where TopSolids:
   - Takes care that a multiple of the phase cycle is used as number of scans
   - Automatically synchronizes the optimized spectral width with the MAS rate
   - Applies basic processing and shearing
   - User interaction only needed for entering number of scans & relaxation delay



TopSolids – Navigation

- Whenever TopSolids shall be restarted, open an ExpNo of the respective project directory and start TopSolids
- TopSolids recalls the module that has been used latest
- Navigation between the different Application modules is done easily…



Data

Search: Find

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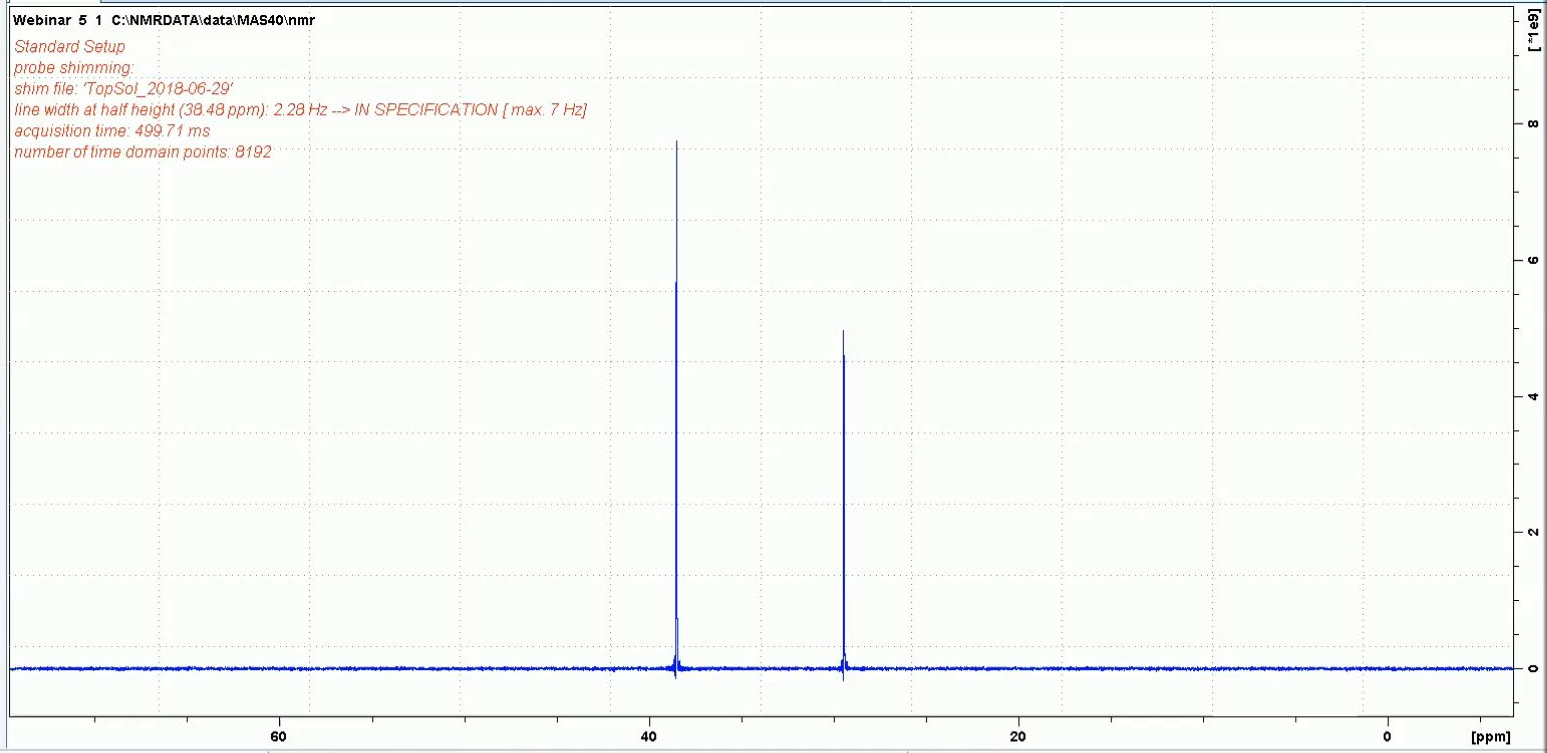
- K3166_0302_40_npt
- K3166_0303_40
- K3166_0304_40_npt
- K3166_0305_40_npt
- K3166_0306_40
- LCZSHIM_TMP
- Webinar
- chjo_test
- euromar
- teststopsolids
- test
- topsolids
- MIC_DIFF
- SEWE
- Training
- C:\NMRDATA\data

No structure available.

SPECTRUM | PROCPARS | ACQUPARS | TITLE | PULSEPROG | PEAKS | INTEGRALS | SAMPLE | STRUCTURE | PLOT | FID | ACQU



Webinar 5 1 C:\NMRDATA\data\MAS40\nmr | topsolids: finished

Spectrometer Status	Amplifier Control	Acquisition information	Fid Flash	Shim Coil Temperature	MAS Spin Rate	POWCHK	Probe Temperature	Spooler	Time
On		no acquisition running		304 K	0 Hz	✓	Initializing	queued: 0 delayed: 0 cron: 0	16:57:33 Jul 03

Off | Reg. State:

TopSolids – Features at a Glance

- Intelligent handling of project and parameters (incl. import/ report)
- Probe setup: within less than 30 minutes possible
  - Automated quality evaluation of magic angle adjustment and shimming
- Protein Applications: state-of-the-art pulse program library up to 4D experiments
  - "1-click-optimization" – TopSolids detects missing parameters to initialize their optimization
  - Automated optional optimizations of spectral widths, carrier frequencies and decoupling parameters
- Materials Applications: suited for any X nucleus the hardware can measure, incl. quadrupolar nuclei
  - High/ low power X 90 degree pulses
  - HX cross polarization (CP) transfer & 2D HetCor
  - 1D/ 2D MQMAS experiments
- Queuing of multidimensional experiments
- Automated processing up to 3D, including phase corrections (& shearing for MQMAS experiments)

TopSolids – Benefits at a Glance

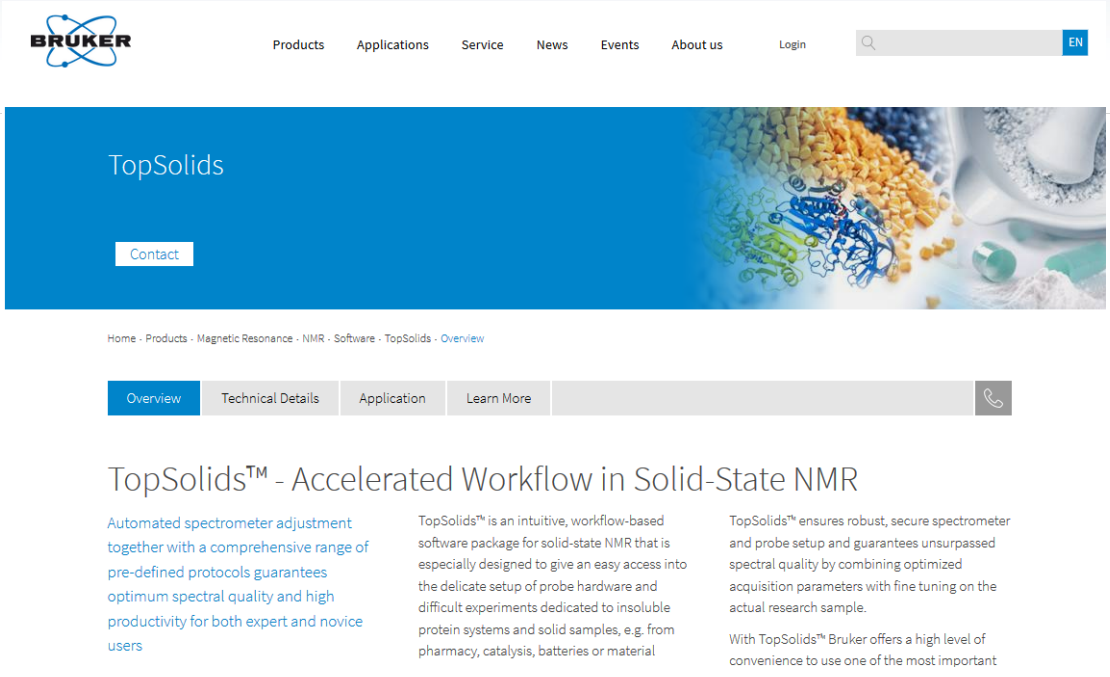
- Quality output
- Minimized risk of damage
- Efficient use of spectrometer time
- Uniform workflows
- Keep track via fast reporting

… whatever your background and experience will be

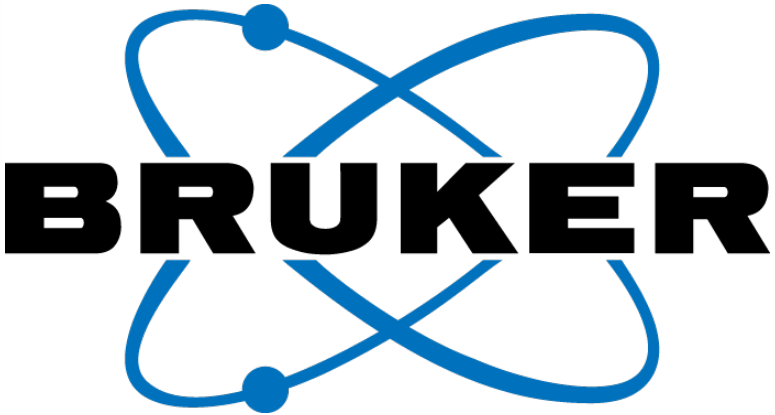
→ TopSolids: Your key to unlock solid-state NMR !

Further Information

- Do you want to get in contact with us?
  - ➢ solids@bruker.com
- Do you want to get further details?
  - ➢ Product page at www.bruker.com



谢谢



Innovation with Integrity

结束