Introduction to TopSpin

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Introduction to TopSpin

- Overview of Topspin interface
- Basic procedure for setting up an acquisition
  - setting up your dataset
  - controlling the spectrometer
- Basic 1D processing
- A little about acquiring and processing 2D’s
- A few other useful features
Starting Topspin

TopSpin 3.5

- If Topspin isn’t already running, click the desktop ICON to start

- It’s yet to be decided whether each user will have an individual Linux login, or if there will be shared group logins...
Topspin layout

- Data Browser
- Command line
- Flowbar menus
- Dataset window
- Tool buttons
- Custom user buttons
- Acquisition status bar
Flowbars – guiding your workflow
Experiment Setup

Prepare for a new experiment by creating a new data set and initializing its NMR parameters according to the selected experiment type. For multi-receiver experiments, several datasets are created. Please define the number of receivers in the Options.

NAME: cyclosporine
EXPNO: 1
PROCNO: 1

- Use current parameters
- Experiment: PROTON

Options:
- Set solvent: C6D6
- Execute 'getprosilo'
- Keep parameters: P1, O1, PLW1
- DIR: /opt/topspin3.5/ecj
- Show new dataset in new window

TITLE: 50mM Cyclosporin in benzene-d6
Experiment Setup

1. Where do you want to store your data

2. What experiment do you want to run?
New experiment dataset will be created in the directory specified by

\[
DIR/NAME/EXPNO/
\]

\[/opt/topspin3.5/ecj/cyclosporine/1/\]

NAME is a directory which can contain several experiments

EXPNO is any positive integer.
Experiment Setup – data storage location

Some recommendations:

- Remember, *DIR* and *NAME* are directories.
- Limit directory names to letters and numbers
- dashes “-” and underscores “_” are OK
- spaces or special characters such as %, #, (, etc. can sometimes cause problems
- Always store data on a locally mounted hard drive
- The software will allow you to specify a network drive, but it’s not recommended!
Experiment Setup – parameter sets

What is a parameter set?

- All of the information necessary to run your experiment:
  - pulse program name
  - nuclei
  - excitation frequencies and sweep widths
  - durations of pulses* and delays
  - pulse power levels*
  - gradient strengths
  - number of scans
  - relevant spectrometer configuration
  - etc…

* pulse durations and power levels will usually be set in a later step

Prepare for a new experiment by creating a new dataset and initializing its NMR parameters according to the selected experiment type. For multi-receiver experiments several datasets are created. Please define the number of receivers in the Options.

NAME  cyclosporine
EXPNO  1
PROCNO  1

Options
- Set solvent: C6D6
- Execute ‘getprosol’
- Keep parameters
- DIR: /opt/topspin3.5/edq
- Show new dataset in new window

Receivers (1,2,…16): 1

50mM Cyclosporin in benzene-d8

TITLE

[Image of Bruker software interface with parameter set options]
Experiment Setup – parameter sets

Standard parameter sets exist for (almost) any experiment you want to run.
Experiment Setup – parameter sets

You can search through parameter set names
“Show Recommended” button gives list of most common small molecule experiments

I’ll discuss these a little more towards the end of this presentation…
Experiment Setup – parameter sets

User modified parameter sets are stored in a separate directory

The command “wpar” is used to write parameters
Experiment Setup – parameter sets

Select parameter set of interest, then click “Set selected item in editor”
After defining data storage location and parameter set name, click “Ok”
Controlling the spectrometer – the Acquire tab

But first…

… a few more details on the Topspin flowbar
some Flowbar details

- Hovering the mouse over a button will open a tool tip
- command line commands are usually shown in parentheses
3 types of Flowbar buttons

1) Some buttons (like “Sample”) open a pull-down menu of options
3 types of Flowbar buttons

2) Some buttons (like “Lock”) execute a single command
3) Some buttons (like “Tune”) have two parts

- clicking on the left part executes a default command
- clicking on the right opens a list of related options
Back to data acquisition…

- click Flowbar buttons from left to right

“Sample” button

- Control the sample changer
- Turn on or off the sample change lift air
- Open the sample temperature control panel
More info on loading samples...

- Use Bruker spinners!
  - Varian/Agilent spinners are slightly different and won’t work

- Use depth gauge to determine how far to insert NMR tube into spinner
  - Don’t adjust position of gauge – it should be at 2cm
"Lock" button

- Select the solvent from list
- All relevant lock parameters are set and the lock is automatically optimized
- "D2O" vs. "H2O+D2O" – this will determine whether automatic shimming will use $^2\text{H}$ or $^1\text{H}$ observe.
Automatic tune and match

“Tune” button – (left part of button)

- Automatically tunes and matches to the nuclei defined in current dataset
If spinning lineshape is significantly better than non-spinning, then X/Y shims should be touched up.
Shimming – topshim

- Default gradient shimming (topshim) usually works quite well from sample to sample
- fully automated
- usually takes less than a minute
- uses info from lock solvent to specify shimming method
Shimming – topshim

topshim’s GUI offers more automatic shimming options
Shimming – topshim

A lot more details in the Topshim manual…
- Prosol = probe and solvent dependent parameters
- reads calibrated pulses for installed into current dataset
- RF pulse power levels are all set to zero in standard parameter sets
Automatic receiver gain

- Sets the receiver gain to optimal value
- Acquired data will use fill dynamic range of the analogue to digital converter in the receiver
Start acquiring data

- The Go button starts the acquisition using all of the parameters in the current dataset.

- **Warning**: you can potentially overwrite data if you click “Go” in a dataset which already contains spectral data.

- User configurable preferences will determine whether you get a warning before overwriting data.
Some info on data acquisition

Clicking the Acqu tab will show you the FID of the acquisition in progress.

If the dataset window the acquisition in progress isn’t open, clicking the “FID” icon will open it and display the “Acqu” tab.
Some info on data acquisition

When looking at the “Acqu” tab during an acquisition in progress, you can toggle between the FID and a real time Fourier transform display.
Some info on data acquisition

- During the acquisition of a 1D spectrum, all of the data is on the console electronics until the last scan is finished.

- After the last scan, data is automatically copied to the dataset directory and can be processed, etc…

- To copy data during an acquisition in progress, click the “save” icon in the “Acqu” tab
Stopping an acquisition in progress

• The red “halt” button first copies the current data in progress to the dataset directory, then stops the acquisition

• “All is OK”

• The red “stop” button stops the acquisition immediately
  • data is not stored
  • command spooler is suspended
  • “Something is wrong that needs to be fixed”
Command Spooler – queuing multiple acquisitions

- all acquisition commands can automatically be sent to a spooler
- once one process finishes, the next will start
- you can queue up multiple experiments as well as shimming, tuning, etc.
- This must be tuned on in the user settings
Command Spooler – queuing multiple acquisitions

- Clicking on “queued” will show a list of the spooled jobs
- Right-clicking on a spooled job will give options for changing or deleting it

List of spooled commands
Datasets used for each spooled command will
Basic data processing...

- The "Proc. Spectrum" provides a 1-click processing
Basic data processing…

• The details of exactly what Proc Spectrum does can be configured
Manipulating spectrum display

- Buttons to adjust intensity of spectrum
- Intensity also adjusted by scrolling middle mouse wheel
Manipulating spectrum display

- Buttons for controlling zoom and position of spectrum
- Can also drag with left mouse button to zoom into region of spectrum

(left-click and drag)
Basic data processing – manual phasing

Start manual phasing mode

To adjust zero-order phase:
- left-click on “0”
- move mouse up and down while continuing to hold down left mouse button

To adjust first-order phase:
- left-click on “1”
- move mouse up and down while continuing to hold down left mouse button

When done, click “save and return” icon
Basic data processing – chemical shift referencing

Default referencing is based on lock frequency. This is often sufficient.

“Calib Axis” allows you to define the exact position of TMS or your solvent peak:

1) Click “Calib Axis” button
2) Click on exact position in spectrum
3) Enter new chemical shift value
Basic data processing – manual peak picking

- When the draw regions button is highlighted, drawing a box with the left mouse button allows you to define peak picking regions.
- Peaks which are within these thresholds (max and min) will be automatically picked.
- Different thresholds can be drawn for different chemical shift ranges.
Basic data processing – manual peak picking

- Drawing a box with the left mouse button allows you to define peak picking regions.
- Peaks which are within these thresholds (max and min) will be automatically picked.
- Different thresholds can be drawn for different chemical shift ranges.

- Click “save and return” to save new peaks and exit peak picking mode.
When the integrate button is highlighted, clicking with the left mouse and dragging across the spectrum defines an integral region.
Right-clicking on an integral gives options, such as calibrating or normalizing their values.
Additional 1D display options

- overlaying spectra

- Icon opens multiple spectrum display mode
- Additional spectra can be drag-and-dropped from the Browser into the dataset window
- Works for both 1D’s and 2D’s
Additional 1D display options

- overlaying spectra
2D experiments...pretty much the same as 1D’s

- Define location for new experiment (new EXPNO used here)
- Define parameter set for new experiment
Additional setup option for 2D’s - Setlimits

- Setlimits provides a graphics tool for setting the spectral regions for 2D experiments (direct and indirect dimensions)
Additional setup option for 2D’s - Setlimits

- New window pops up giving instructions for “setlimits”
- keep this window open for now!
Additional setup option for 2D’s - Setlimits

1) Drag-and drop 1D reference spectrum from Browser into dataset window
Additional setup option for 2D’s - Setlimits

2) Zoom into region of 1D you want to copy to 2D dataset
Additional setup option for 2D’s - Setlimits

3) Click “OK”
Additional setup option for 2D’s - Setlimits

- Relevant sweep widths and excitation frequencies are automatically copied to 2D dataset
- Rest of setup is the same:
  - “Prosol”
  - “Gain”
  - “Go”
Processing 2D’s

- “Proc Spectrum” button does most of what you need…
Processing 2D’s – manual phasing

- Works much like 1D phasing, but you need to define slices through 2D spectrum
- Right click with mouse to define rows
- Click “R” button to display rows
Processing 2D’s – manual phasing

- “0” and “1” buttons adjust zero- and first-order phase correction (just like with 1D’s)
More about the Topspin spectrum display

- Too many things can clutter the spectrum display
- Right-click on spectrum window to select what’s displayed
More about the Topspin spectrum display
Plotting – copying/pasting spectra

- Copy button will copy a screen shot of spectrum as it’s displayed
- Can the Paste into word processor or presentation software
- Send spectrum (as displayed) to printer
- Output spectrum (as displayed) to a PDF or various image formats
Plotting – copying/pasting spectra

- Open Plot Editor
- Can print spectra according to pre-defined Layouts
- Can also create custom layouts
Back to recommended parameter sets…

- Options -> Show Comment will display brief description of parameter sets
• Parameter set (and pulse program) names often have several 2-letter codes

• HSQCEDETGSPISP:
  ED = with multiplicity editing
  ET = echo-Antiecho
  GP = with gradients
  SI = sensitivity improved
  SP = with shaped pulses

• for full list of 2-letter codes, look at the file “Pulprog.info” in the pulse program directory

• “edpul” will display list of pulse programs
Acquisition parameters

The "AcquPars" tab shows the full list of acquisition parameters.

Clicking the "pulse" or "A" icon will toggle between pulse program specific pulses and delays, and all acquisition parameters.
Acquisition parameters

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Some User preferences (for different Linux or Windows login users)

Directories displayed in Browser:
- these correspond to DIR in the new dataset window

Right-click in Browser to add or remove directories from display
Some User preferences

Automatic command spooling
Some User preferences

- Whether to warn before overwriting data when starting acquisition
- Select tools displayed in Status Bar
A couple of useful manuals

Beginner Guide:
- Basic introduction to NMR
- Basic description of spectrometer components
- Some explanation of the various steps of setting up an acquisition

Step-by-Step guides:
Menu for acquiring basic and advanced 1D and 2D experiments
Topspin student licenses

If you want to install Topspin on your own computer…

The Student License

- Can be purchased from the online Bruker store: store.bruker-biospin.com
- Price is $99 for a 3-year license