

Basic 2D NMR Instructions

An efficient way to collect 1D and 2D experiments for a given substance would be to first complete the ^1H experiments first with 20 - 40 mg of sample, then add sample so that the quantity is near 80 mg and collect the ^{13}C spectra. Then you can set up your 2D experiments and use 'multizg' to run them all sequentially.

If you will be collecting a 2D experiment that uses the ^1H or ^{13}C signal as one of the channels, you must first run the 1D experiment and retrieve a couple of parameters. After processing the 1D experiment, zoom in on the region of interest - making sure to leave margins of about 0.5 ppm on the left and right for ^1H , and 5 ppm for ^{13}C . Then type:

“.setsw” sets the sweep width and spectrometer frequency equal to the zoomed region. Record the value for SW and O1 that are displayed.

It is generally recommended that all 2D experiments be run without sample spinning. You must, however, shim Z and Z^2 while the sample is spinning, then you can turn the spinner off prior to beginning acquisition. Just don't forget to turn the spinner back on after you're done!

You can set up multiple experiments to run back-to-back (typically overnight) by using the 'multizg' command (see last page).

^1H - ^1H COrrrelation SpectroscopY (COSY):

ACQUISITION

“new file”	Create a new file
name	Use same name as for your 1D experiment(s)
expno	increment sequentially (1, 2, 3...)
procno	1
experiment	COSY
“sw”	Enter the value for SW as determined from the zoomed ^1H region (.setsw) into the F2 and F1 fields (make sure each value is the same)
“o1”	Enter the value for O1 as determined from the zoomed ^1H region (.setsw)
“rga”	Determine gain (dependent on sample concentration)
“ns”	Number of scans = 8 x n (16 scans (recommended) will result in about a 1 hour acquisition time)
“zg”	Begin acquisition

PROCESSING

“xfb”	process data
“sym”	symmetrize data
“projd”	Enter the filename/expno where the 1D reference spectra is located (F2 and F1 should be the same)

Make sure 2D spectra is calibrated with 1D spectra - if necessary, use calibration icon to adjust diagonal signal (make sure F2 and F1 values are the same).

“print” or “plot” “print” will print what is on the screen (may not be square), while “plot” will load a default template file that forces the data to be square. As with plotting 1D experiments, further adjustments can also be made here.

¹H - ¹H Nuclear Overhauser Effect Spectroscopy (NOESY)

ACQUISITION

"new file"	Create a new file
name	Use same name as for 1D experiment(s)
expno	increment sequentially (1, 2, 3...)
procno	1
experiment	NOESY2
"sw"	Enter the value for SW as determined from the zoomed ¹ H region (.setsw) into the F2 and F1 fields
"o1"	Enter the value for O1 as determined from the zoomed ¹ H region (.setsw)
"rga"	Determine gain (dependent on sample concentration)
"ns"	Number of scans = 8 x n (8 scans (recommended) will result in about a 2 hour acquisition time)
"zg"	Begin acquisition

PROCESSING

Phase in F2:

"rser 1"	Transfer the 1st row of data to a temporary 1D data set
"sinm"	Apply sine-bell windowing function
"ft"	Fourier transform the data
select interactive [phase] icon	adjust PH0 and PH1 - then save for 2D spectrum, return and select [2d]
"xfb"	process data

Phase in F1:

select interactive [phase] icon
choose three diagonal peaks use left mouse button to select three relatively large diagonal peaks - one each at left, middle and right.

Phase [C] columns optimize 0 and 1, then save, return and return again.

When the NOESY spectrum is properly phased, the diagonal peaks will be either all positive or all negative. The cross peaks may be positive or negative. Any anti-phase cross peaks are COSY artifacts.

"projd"	Enter the filename/expno where the 1D reference spectra is located (F2 and F1 should be the same)
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Make sure 2D spectra is calibrated with 1D spectra - if necessary, use calibration icon to adjust diagonal signal (make sure F2 and F1 values are the same).

"print" or "plot"	"print" will print what is on the screen (may not be square), while "plot" will load a default template file that forces the data to be square. As with plotting 1D experiments, further adjustments can also be made here.
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¹³C - ¹H HETeronuclear CORrelation spectroscopy (HETCOR)

ACQUISITION

"new file"	Create a new file
name	Use same name as for 1D experiment(s)
expno	increment sequentially (1, 2, 3...)
procno	1
experiment	CHCORR
"sw"	Enter the value for SW as determined from the zoomed ¹³ C region (.setsw) into the F2 field, and the value for ¹ H in the F1 fields
"o1"	Enter the value for O1 as determined from the zoomed ¹³ C region (.setsw)
"o2"	Enter the value for O1 as determined from the zoomed ¹ H region (.setsw)
"rga"	Determine gain (dependent on sample concentration)
"ns"	Number of scans = 16 x n (16 scans (recommended) will result in about a 1.25 hour acquisition time)
"zg"	Begin acquisition

PROCESSING

"xfb"	process data
"projd"	Enter the filename/expno where the 1D reference spectra is located (F2 is the carbon experiment and F1 is the proton experiment).

Make sure 2D spectra is calibrated with 1D spectra - if necessary, use calibration icon to adjust diagonal.

"print" (not "plot") "print" will print what is on the screen (may not be square).

Multizg Instructions

Create all experiments to be run in consecutive EXPNOs. This includes entering the SW and O1 values from the 1D experiment, setting the number of scans for each experiment and running "rga" to set the receiver gain.

Then go to the first experiment in your consecutive sequence and type "multizg" - then simply enter the number of consecutive experiments that need to be run. Unfortunately there is no automated procedure for processing all of them at once when they are done...