A Guide Obtaining Common NMR Spectra

This pamphlet provides parameters, settings, and the like for relatively simple NMR experiments beyond the standard ^{1}H and ^{13}C. For more complicated experiments, see the handouts in

`https://www.chem.wilkes.edu/~trujillo/NMR/How_To...`

or

`/usr/local/share/doc/Info_Sheets/NMR_Handouts.html` .

Please let me know of any corrections, other kinds of spectra that should be included, etc.

— Professor Trujillo

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1 ^{31}P and other Broadband Nuclei

Spectra of nuclei other than ^{1}H and ^{13}C may be obtained similarly to the procedure for acquiring carbon. (See below for ^{19}F.)

1. `rpar P31 all`
   - "`P31CPD`" for ^{1}H decoupling
   - For other nuclei, substitute *NUmass** for other atoms (eg, "`NA23*`" or "`N15*`") and select from the choices that appear.
2. `getprosol`
   `atma` to tune for the nucleus and match the impedance.
3. `rga`
4. Acquire (`zg`) and process as usual.

2 ^{19}F Spectra

^{19}F spectra are obtained similarly to other broadband nuclei (above), but manual tuning/matching generally works better for it than the automated routine.

1. `rpar F19 all` (`F19CPD` for ^{1}H decoupling)
   `getprosol`
2. `atmm` to manually tune and match.
   Using the arrow keys, try to align the tip of the trough with the red vertical line and the baseline.
3. `rga`
4. Acquire (`zg`) and process as usual.
   Note that some peaks will go up and others down.

3 APT

APT (Attached Proton Test) is an alternative to DEPT. Although APT is less sensitive than DEPT, it shows quaternary carbons, which DEPT does not.

1. `rpar C13APT all`
2. Set `ns` and `ds` as desired.
3. Acquire (`zg`) and process as usual.

4 DEPT

DEPT allows one to determine whether a carbon is a CH, CH_2, or CH_3. Note that quaternary carbons do not show up.

For DEPT-45 and DEPT-90, substitute "C13DEPT45" and "C13DEPT90" in the `rpar` command.

1. `rpar C13DEPT135 all`
   `getprosol`
2. Acquire (`zg`) and process as usual.
   Note that some peaks will go up and others down.

5 INEPT

INEPT is a useful, albeit dated, way to obtain J_{CH} and other heteronuclear coupling constants. It also provides a very nice tool to assign carbon spectra.

For ^{15}N INEPT, use `rpar N15INEPT`.

1. `rpar C13CPD all`
2. Set the following parameters

   `pulprog` = ineptnd
   `cnst2` = 128 (approx $^1J_{CH}$ — adjust if sp or many sp^2 carbons)
3. Acquire (`zg`) and process as usual.
   When phasing, remember that peak shapes will be distorted.

6 Running a Spectrum Unlocked

At times it is necessary to run a sample without locking on the solvent — for example, if one is using a non-deuterated solvent, referencing to an external standard, or acquiring a ^{2}H spectrum.

1. Lock and shim on a sample as close to that of your actual sample as possible — same solvent height, similar solvent but deuterated, etc.
2. Run the shim sample and set the reference line.
3. Turn the lock and lock-sweep off:
   In the *Lock/Level* tab of the BSMS control panel, unclick the lock and sweep buttons.
4. Replace the shim-sample with your actual sample.
5. Run the spectrum (zg) as usual.
6. When done, don't forget to turn the lock-sweep and lock back on in the BSMS panel, and to verify that the PhEt sample locks.

7 Using an External Standard

For many broadband nuclei, either an internal standard (in a capillary tube within your sample) or an external standard (in a separate NMR tube) may be used. An internal standard allows the sample to be run as normal (locked on the solvent, etc). With an external standard, however, the lock mechanism will look for the deuterium signal, causing the field to shift during the spectrum. Thus, one must reference the spectrum with the lock disabled.

The instructions below assume you are obtaining a ^{31}P spectrum, referenced to 85% H_3PO_4, but the principal applies to other external standards as well.

1. Insert your sample, lock on it, and shim, as usual.
2. Turn the lock and lock-sweep off:
   In the *Lock/Level* tab of the `BSMS` control panel, unclick the lock and sweep buttons.
3. Replace your sample with the standard (eg, 85% H_3PO_4).
   Obtain a spectrum of the standard and set the reference line.
4. Remove the standard and put your sample back in.
   If your sample is in deuterated solvent, turn the lock back on.
5. Run the spectrum (`zg`) as usual.
   Don't try to reference the spectrum.
6. When done, don't forget to turn the lock-sweep and lock back on in the `BSMS` panel, and to verify that the PhEt sample locks.

8 Water Suppression

There are many ways to suppress solvent peaks. The `WATERGATE` sequence typically provides splendid results with a minimum of effort.

1. `rpar P3919GP all`
   `getprosol`
2. Acquire (`zg`) and process as usual.

9 COSY

COSY is a workhorse. These instructions are for gradient-filtered COSY spectra, which tend to be cleaner than traditional COSY spectra.

1. Obtain a normal ^{1}H spectrum, with NS set as low as possible to still provide decent S/N. Type `iexpno` to increment the experiment number.
2. Reduce the spectral window (See item **??** below). Note the values for SW and O1 (in Hz).
3. `rpar COSYGPSW all` (no spaces in name)
   `getprosol`
4. Set the following parameters

SW	= value recorded in step 2	
O1	= value recorded in step 2	
TD (F1)	= # F1 slices desired	(typically 128)
NS	= value from step 1	
DS	= 2	

5. `zg` as usual
6. To process:
   `xfb` to FT the data
   `sym` if symmetrization is desired

10 COSY-45

COSY-45 has a 45° read pulse, rather than a 90° pulse. This often leads to sharper cross-peaks, giving better resolution if peaks are very close.

Bruker offers a parameter set for COSY-45 (`rpar COSY45SW`), but the gradient-filtered method described below leads to better results and is often faster.

To obtain a gradient-filtered COSY-45, proceed as per "COSY" above, but also change the value of P0 to half its value (typically ~ 7.75 sec), to produce a 45° read-pulse, rather than a 90° read-pulse.

11 Phase-Sensitive COSY

Phase-Sensitive COSY gives sharper peaks, especially along the diagonal, than does normal (magnitude) acquisition.

1. Obtain a normal ^{1}H spectrum and trim the window.
   Type `iexpno` to increment the experiment number
2. Trim the window (See item **??** below) and note the values for SW and O1 (in Hz).
3. `rpar COSYGPDFPHSW all` (no spaces in name)
   `getprosol`
4. Set/verify the following parameters

SW	=	value from step 2
O1	=	value from step 2
NS, DS	=	reasonable values based on reference spectrum
TD (F1)	=	number of F1 slices

5. `zg`
6. To process:
   `xfb` to Fourier Transform the data.
   `apk2d` to phase
   `syma` if symmetrization is desired

12 HSQC

$^{1}H/^{13}C$ HSQC provides H/C correlations, with gradient filtering.

1. Obtain a ^{1}H spectrum. Trim the window (See item **??** below), and note the SW and O1 values. Type `iexpno` to increment the experiment number.
2. Do likewise for ^{13}C. (Don't forget the `atma`!)
   Type `iexpno` again.
3. `rpar HSQCEDETGP all` (no spaces in name)
   `getprosol`
4. Set the following parameters

   SW, O1 = values obtained in steps 1 and 2.
   F2 = values for ^{1}H
   F1 = values for ^{13}C
   TD (F1) = 128 or 256, depending on desired ^{13}C resolution
5. Verify gradients: Enter `gpnam`

   gpz1 = 80%
   gpz2 = 20.1% for ^{13}C 8.1% for ^{15}N
   gpnam1 = SMSQ10.100
   gpnam2 = SMSQ10.100
6. `zg`
7. To process:

   `apk2d` to phase
   `xfb` to Fourier Transform
   If the scale is backwards, set REVERSE to "True" for F1.

   If things look wrong (missing peaks, etc), try adjusting the weighting functions:

   Type `wm`
   Set wdw for F2 and F1 to "squared sine" and FT the data (`xfb`).
   Repeat with wdw = "sine bell" and ssb = 0, 1, and 2, to see what looks the best.

13 NOESY

NOESY provides a sense of physical distance between protons, rather than through-bond distance the way COSY does.

NOESY spectra are often temperamental. To provide a good spectrum, the sample must be pure, degassed, and relatively dilute — aim for one which will require about 8 scans for a good 1D spectrum. In addition, dummy scans are helpful, a long D1 is essential, and the mixing time (D8) may take adjusting. In short, for good results, NOESY cannot be rushed.

See `http://www2.chem.uic.edu/nmr/downloads/Avance-NOESY-Guide.0702.pdf` and `http://triton.iqfr.csic.es/guide/nmr/manual/13c_noesy.html` for hints and more information.

1. Obtain a normal ^{1}H spectrum.
   Type `iexpno` to increment the experiment number.
2. Reduce the spectral window (See item **??** below). Note the values for SW and O1 (in Hz).
3. `rpar NOESYPHSW all` (no spaces in name)
   `getprosol`
4. Set the following parameters

SW	=	value from in step 2	
O1	=	value from in step 2	
TD (F1)	=	# F1 slices desired	typically 128
NS	=	value from step 1	multiple of 2
DS	=	8	minimum of 4
D1	=	3–5 T1	delay betw cycles; defaults to 4s
D8	=		mixing time. 0.1–0.8s for small mols

5. Acquire as usual (`zg`)
6. To process:
   `xfb` to FT the data
   `apk2d` to phase the data
   `syma` if symmetrization is desired

14 Homonuclear 2D *J*-Resolved Spectra

HOMO2DJ spectra "twist" multiplets so that they appear as singlets along the F2 axis, and all the coupling occurs in the F1 direction. One thus obtains a 2D spectrum showing chemical shift vs. J.

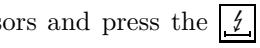
1. Obtain a normal ^{1}H spectrum.
   Type `iexpno` to increment the experiment number.
2. Reduce the spectral window (See item **??** below). Note the values for SW and O1 (in Hz).
   `rpar COSY45SW all`
   `getprosol`
3. Set the following parameters

pulprog	=	jresqf	
sw (F2)	=	value from step 2	
sw (F1)	=	40	(reduce F1 width)
o1 (both)	=	value from step 2	
td (F1)	=	64	(reduce # slices)

4. Set ns and ds as desired, then zg, as usual
5. To process:
   `xfb` to FT
   `tilt` (may be automatic)
   `symj` if symmetrization is desired

15 Reducing the Spectral Window

Reducing the width of the data window will give higher resolution for the same data-set size. You may also want to trim down the data set size (TD).

1. Acquire a spectrum as usual.
2. Bracket the region containing peaks with the cursors and press the  button.
   Be sure all (non-tiny) peaks are between the cursors as you do this.
3. Record the O1 and SW parameters, so you can enter them after recalling a new experiment.

16 Experiment Name Codes

The experiments on the Bruker have names with many embedded codes to describe what each does. Although not always clear and not always consistent, here are some common codes. For example, "COSYPHPR" sets up a phase-sensitive (PH) COSY with presaturation of a solvent resonance (PR).

In general, gradients are preferable over phase cycling.

19	3-9-19 water supprsn	
30	30° tip angle	
CPD	decoupling	(compound pulse decoupling)
D	decoupling	
DC	^{13}C decoupling	
DQF	double quantum filtered	
GP	gradients used	
PH	phase sensitive	
PS	phase sensitive	
WT	water suppression	
ZG	normal spectrum	