Calibration of 90° Pulsewidths

Measurement of pw90 can often be skipped on Bruker spectrometers equipped with automatic tune-and-match (ATM) probes. On manually tuned probes — e.g., the Inova600 and Avance360 — the procedure is highly recommended.

Calibration of 1H 90° pulsewidths in TopSpin:¹

Measuring the length of the standard proton pulse to achieve a 90° nutation² is a primary calibration done for NMR experiments. This pulsewidth on the observe channel, \( p_1 \), (or \( pw90^3 \)) is probe-dependent and is always matched to the power used, \( plw1 \). A series of spectra at increasing pulse length — which increasingly nutate the magnetization — is collected via Bruker’s popt automation routine. The best measurement is made at the 360° null crossing, with \( pw90 = pw360 / 4 \).

(a) rpar PROTON all \( \sim \) ns 1 \( \sim \) ds 0 \( \sim \) pulprog zg \( \sim \)
(b) Do normal lock, atma, getprosol, etc.
(c) Changing PULPROG to zg uses \( p_1 \) directly, rather than \( p_1*0.33 \) as in zg30.
(d) efp \( \sim \) apk \( \sim \) .all \( \sim \) abs n \( \sim \)
(e) Set PH_mod = pk so spectra are phased during popt. Set at cmd line or from the PROC Pars tab.
(f) Expand the spectrum about peaks in the midrange ppms, (don’t expand on solvent or impurity multiplets).
(g) Right-click on the spectrum, and choose: Save Display Region To…

and use the top selection. You must run this command prior to doing a popt experiment.

(i) popt \( \sim \) will open the table to the right. Enter PARAMETER \( p_1 \) and STARTVAL = 4 .
(j) Delete other values existing in the table.
(k) Set INC = 4, NEXP = 16. ENDVAL will automatically update.
(l) Click START OPTIMIZE .
Give the appropriate responses [don’t know what APPEND means; doesn’t seem to matter].

(m) FIRST TIME ISSUE: you may need to make the following changes that, once done, will be remembered by TopSpin (then again, you might prefer the original settings…)

To keep popt from constant changing away from the stacked spectrum presentation:

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¹ A (relatively) new TopSpin command, pulsecal, works fine in samples that have a very strong singlet peak, but fails otherwise. It is therefore a very good alternative to the above for biological samples in 90:10% H₂O:D₂O. It is not recommended for samples in other solvents.

² Nutation in NMR refers to the rotation of magnetization about a rotating-frame axis, e.g. \( x' \). For magnetization initially along the \( z \) axis (parallel to \( B_0 \)), then in a left-hand system, the magnetization nutates \( z \overset{\Delta}{\rightarrow} y'\overset{\Delta}{\rightarrow} z' \overset{\Delta}{\rightarrow} -y' \overset{\Delta}{\rightarrow} z \overset{\Delta}{\rightarrow} … \).

³ I (cgf) often use the Varian symbol \( pw90 \) for the 90° pulsewidth, as it is more directly descriptive; in vnmr/vnmrj the observe channel power is tpwr. \( pw360 \) is not used in vnmr, but the extension for a 360° pulsewidth is obvious.
MANAGE → PREFERENCES → ACQUISITION uncheck “Auto open acquisition window after “zg”

(n) You should now be seeing a horizontal stack plot growing. Click to get it scaled correctly, and to get the stack mid-screen vertically.

Note the sinusoidal behavior: 90° at ~ 15μs, 180° at 30μs, 360° at 60 μs (on the 400; differs elsewhere). The 360° crossover is the best to measure. 90° is too flat; 180° too dependent on repetition rate.

(o) TopSpin automatically moves the spectra into the 999 processing folder. To get back to the acquisition folder, type:

re 1 1 .

where you’ll see the last spectrum acquired, with the parameters used for that spectrum: in this example, the last spectrum has p1=96 μs.

(p) Run popt again, narrowing in on the 360° region. A good choice for the above example is: STARTVAL = 53, INC = 0.4, NEXP = 16.

(q) p1 = pw360 /4. To invoke the new pulselength throughout all calculated pulses (including shaped pulses), enter the following in each new experiment:

getprosol 1H <pw90> <power>

where <power> is stated in attenuation, not watts (e.g., -13.94 dBW, not 24.8 watts).

If pw90 = 8.15 @ -13.94 , then after reading in a new experiment such as noesy1d, you would enter:

getprosol 1H 8.15 -13.94 .

This command will recalculate the shaped pulse based on the new pulselength.