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 ^{1}H 90° pulsewidths in TopSpin:[1]

Measuring the length of the standard proton pulse to achieve a 90° nutation[2] is a primary calibration done for NMR experiments. This pulsewidth on the observe channel, **p1**, (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** ↵ **ns 1** ↵ **ds 0** ↵ **pulprog zg** ↵

(b) Do normal **lock**, **atma**, **getprosol**, etc.

(c) Changing PULPROG to **zg** uses **p1** directly, rather than **p1***0.33 as in **zg30**.

(d) **efp** ↵ **apk** ↵ **.all** ↵ **abs n** ↵

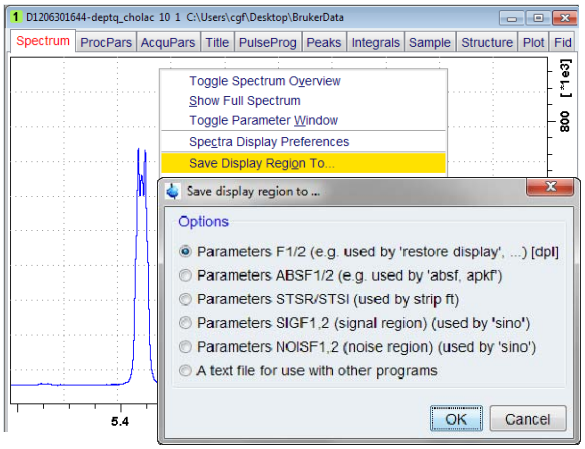
(e) Set **PH_mod = pk** so spectra are phased during **popt**. Set at cmnd line or from the PROCPARS tab.

(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.*

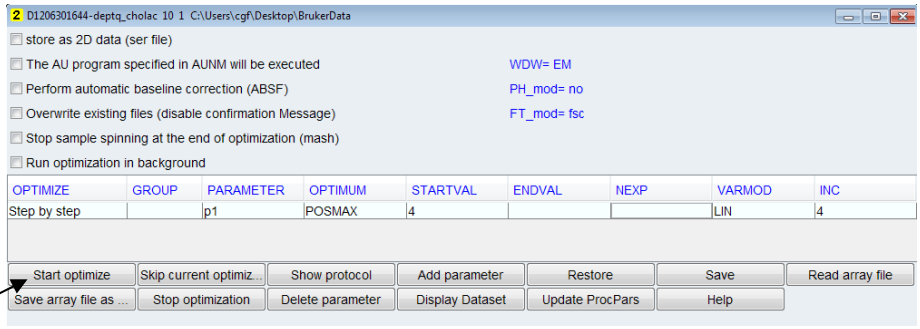


(i) **popt** ↵ will open the table to the right. Enter PARAMETER **p1** and STARTVAL = **4** .

(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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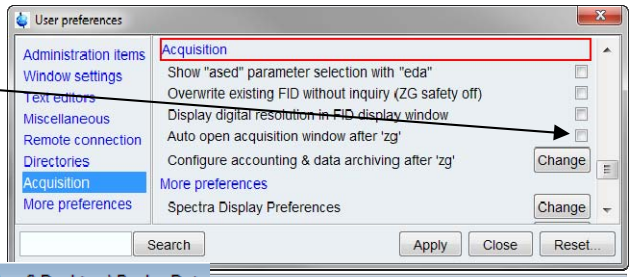
[1] A (relatively) new TopSpin command, **pulsecal**, works fine in samples that have a very strong singlet peak, but fails otherwise. It is therefore a very good alternative to the above for biological samples in 90:10% H_2O:D_2O. It is not recommended for samples in other solvents.

[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 $\mathbf{B_0}$), then in a left-hand system, the magnetization nutates $z \rightarrow y' \rightarrow -z \rightarrow -y' \rightarrow z \rightarrow \ldots$.

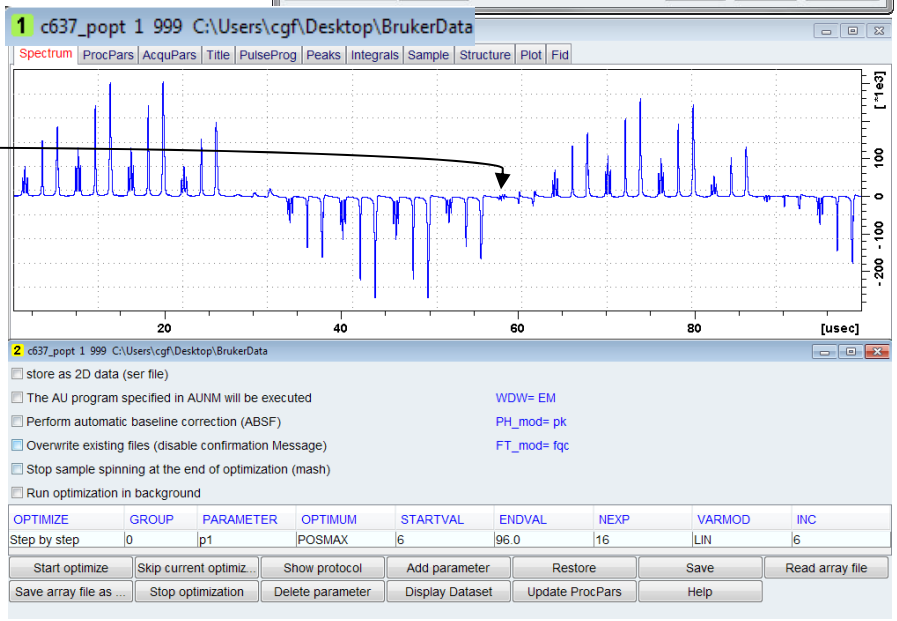
[3] I (cgf) often use the Varian symbol **pw90** for the 90° pulsewidth, as it is more directly descriptive; in vnmr/vnmrj the observe channel power is **tpwr** . **pw360** is not used in vnmr, but the extension for a 360° pulsewidth is obvious.

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.