

Instructions for Measurement of Self Diffusion Coefficients

IU NMR Facility - September, 2016

- 1) Use I500 or I600 for diffusion measurement.
- 2) Calibrate instrument temperature for most accurate results.
- 3) Gradient strength can be calibrated using the Varian Acceptance Test Procedure with the **setgcal** macro and a doped H₂O/D₂O sample, a physical phantom, or reverse calculated using a reference sample of H₂O/D₂O (diffusion constant=1.90 x 10⁻⁹ m²/s). This has been done by NMR staff for each probe. A gcal (gradient strength in G/cm per DAC unit) value was stored in each probe file. The gcal value will be retrieved with the **diffusion** macro when users set up a diffusion experiment. The table below lists the gcal values and maximum gradient strengths (= gcal * 32767).

	Probe	gcal	G _{max} (Gauss/cm)
I500	PFGBB	0.00178	58.3
	HCN	0.00215	70.5
	INV	0.00130	42.6
I600	HCN	0.00183	60.0

- 4) Type **diffusion** to call in DgcsteSL_cc parameters. This is a descendant of other pulse sequences and features self compensating gradients and convection compensation. It is much better than BPP pulse sequences like Oneshot.
- 5) Establish acquisition parameters including 90 degree pulse width, gain, appropriate d1 relaxation delay and transients (nt, must be 4*N) needed to get useable S/N. Do not spin the sample!
- 6) Set up a linear array of gzlv11 values. I typically use 16 from 2000, 3000, 4000 etc. to 17000. Values of up to 32767 can be used (for larger molecules). The macros can also support exponential arrays.
- 7) When the experiment is finished, phase and baseline correct the spectra. Integrate the peak(s) of interest. Multiple regions can be checked one at a time.
- 8) The macro **D_process** will calculate the diffusion coefficient and display the fit for the integrated region(s). 10% H₂O in 90% D₂O is about 1.9 x 10⁻⁹ m²/s for reference -- if in doubt run the experiment on this sample. Confirm that DAC_to_G is set to the correct gcal value (*note: DAC_to_G replacing gcal is used for data processing*).

This experiment was initially set up by Doug Brown with modifications to the "decre" package in the Varian User Library. Non-exponential behavior is often due to convection, especially with low viscosity solvents. Typically lowering air flow will help unless the problem is sample heating from high gradients. Spurious results are sometimes due to interaction of gzlv12 and gzlv13 in the DgcsteSL_cc experiment. Try changing them.

For DOSY see: <http://mestrelab.com/resources/dosy/> and <http://personalpages.manchester.ac.uk/staff/mathias.nilsson/software.htm>.