Instructions for Measurement of Self Diffusion Coefficients

IU NMR Facility - *November, 2017*

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 H_2O/D_2O sample, a physical phantom, or reverse calculated using a reference sample of H_2O/D_2O (diffusion constant=1.90 x 10^{-9} m^2/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.

	Probe	gcal	G_{max} (Gauss/cm)
I500	PFGBB	0.00344	56.4
	HCN	0.00415	68.1
	INV	0.00251	41.2
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 gzlvl1 values. On I600, typically use 16 from 2000, 3000, 4000 etc. to 17000. Values of up to 32767 can be used (for larger molecules). I500 is equipped with a 20A gradient amplifier, the maximum gzlvl1 (as well as gzlvl2, gzlvl3) value is limited to 16400 (Using higher values than 16400 on I500 could damage the probes!!). Typically, 16 values from 800, 1600, 2400 etc. to 12800 are used on I500. 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% H_2O in 90% D_2O is about 1.9 x 10^{-9} m^2/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*).

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 gzlvl2 and gzlvl3 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.