Diffusion analysis in ssNake

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1 Introduction

Is this tutorial, we will analyse a diffusion measurement of a liquid state sample in ssNake. Diffusion is a powerful tool to distinguish multiple molecules in complicated spectra, especially when other 2D methods cannot readily identify the different components. The diffusion experiment consists of a spin-echo experiment, with gradients present during the echo time. In case of no diffusion, each nucleus experiences the same B_0 field during the first part of the echo as during the last, and perfect echo refocussing occurs. In case of displacements in this time (i.e. diffusion), the B_0 field are different during part 1 and part 2 of the echo, and attenuation of the signal occurs. If the strength and durations of the gradients are known, the diffusion constant can be extracted from such a measurement.

2 Data

The data for this tutorial was recorded at 300 MHz. The sample was a tube of pure water. The maximum gradient strength was 1.5 T/m. The relevant times were: $\delta = 1$ ms, and $\Delta = 20$ ms.

3 Fitting a diffusion curve

Start by loading the Bruker type data:

- Load the 'ser' file that was delivered with this tutorial

Now, we want to zero-fill the data, as well as correct for the Bruker time delay (i.e. digital filter). It is best to do the zero-filling first:

- Use 'Matrix ⟶ Sizing', and set the size to 65536
- Correct the digital filter by using 'Tools ⟶ Correct digital filter'
- Fourier transform

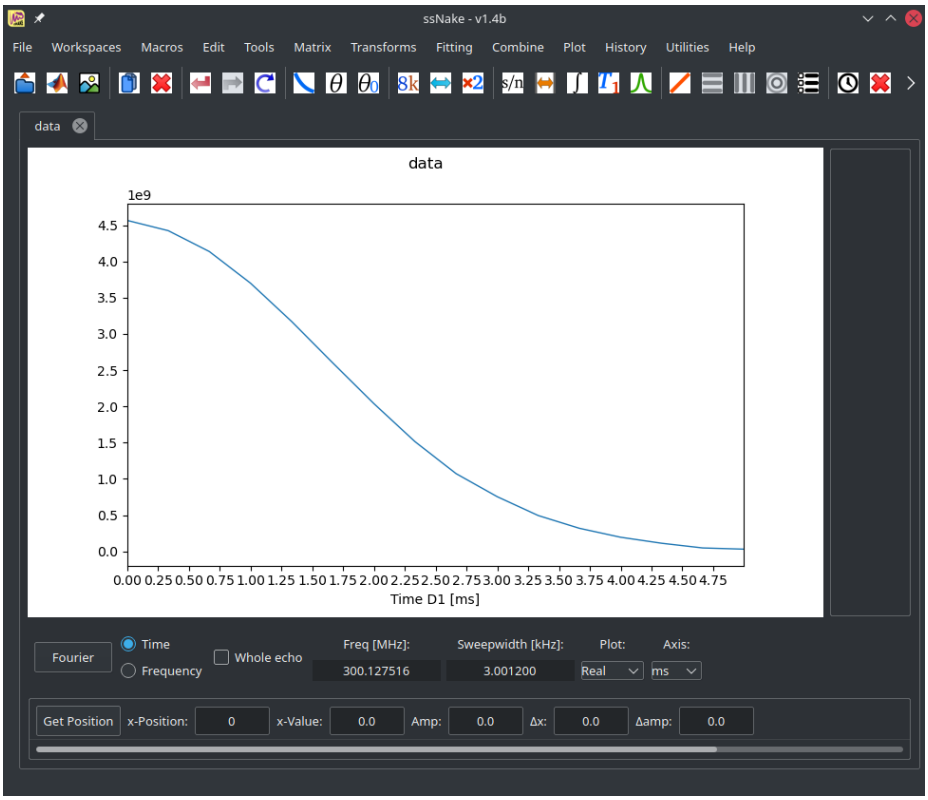
Correct for the phasing:

- Phase using ‘Tools ⟶ Phasing’ with -118.470 zero order.

Now, we will analyse the intensity variation of this peak. In order to do this, we will integrate this region.

- Use ‘Matrix ⟶ Region ⟶’, and integrate between point 31393 and 33638. (Or left click on the left and right side of the relevant peak in the spectrum.)

This should show:



which shows a nice Gaussian decay due to the diffusion.

Now, we want to fit this decay, which means that we must supply ssNake with the relevant x-axis. In this case, we have measured multiple spectra with a different gradient strengths. The strengths were approximately linear, with real values: 4.995005e-03, 7.132867e-02, 1.376623e-01, 2.039960e-01, 2.703297e-01, 3.366633e-01, 4.029970e-01, 4.693307e-01, 5.356643e-01, 6.019980e-01, 6.683317e-01, 7.346653e-01, 8.009990e-01, 8.673327e-01, 9.336663e-01, 1.000000e+00.

These are the values passed to the pulse sequence. 1 stands for the maximum gradient strength of the machine. In this case this is 1.5 T/m. Additionally, the puls esequence itself caps the gradient at 90% for safety reasons.

With these values, we must change the x-axis in ssNake:

- Use ‘Plot ⟶ User x-axis’ and fill in: array([4.995005e-03, 7.132867e-02, 1.376623e-01, 2.039960e-01, 2.703297e-01, 3.366633e-01, 4.029970e-01, 4.693307e-01, 5.356643e-01,

6.019980e-01, 6.683317e-01, 7.346653e-01, 8.009990e-01, 8.673327e-01, 9.336663e-01, 1.000000e+00])*1.5*0.9

- Set the Axis unit to ‘s’

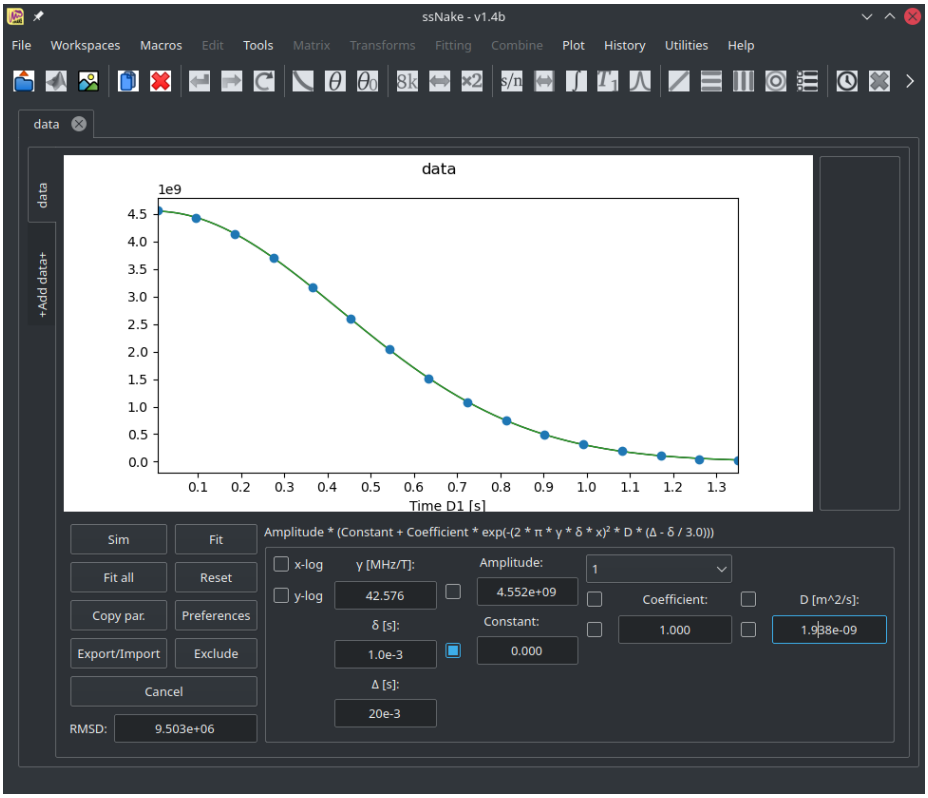
Here, we make use of ssNake’s calculation capability to multiply the array by some values. Note that the axis still shows ‘Time D1 [s]’, while actually it is a gradient strength T/m (you need to remember this for yourself!).

The final step is to fit this curve using the Diffusion fit method of ssNake.

- Use ‘Fitting ⟶ Diffusion Curve’.
- Set δ to 1e-3.
- Set Δ to 20e-3.
- Fix the Coefficient by ticking the box next to it.
- Push ‘Fit’. (Might need a second push to do a second fit run.)

This should lead to a D value of 1.938e-09 m^2/s, which is a typical value for pure water at room temperature.

The fit looks like:



which is an excellent fit.

4 Pitfalls

It can sometime be difficult to establish the true gradients used by the spectrometer. At least in my experience, Bruker Topspin handles this a bit weirdly. While other parameters (pulse lengths, delays etc.) are saved in the parameter files, the gradient list is in an external file. As shown above, some recalculation is needed to get the gradient values in T/m from these. When setting op these measurements, it might be wise to do a check on a sample of pure water, to verify if your methods are correct.

The diffusion equation used in ssNake is applicable for the most common diffusion sequences (spin echo and stimulated echo). When using a stimulated echo with bi-polar gradients, note that the length δ signifies the total time of both gradient lobes combined.

When the gradient pulses are not rectangular (e.g. trapezoidal), the time δ needs to be corrected for this before fitting.