Nutation data analysis using ssNake

17th May 2017

1 Introduction

The following will explain how a standard nutation experiment can be processed in ssNake. The tutorial delivered with the ssNake program is considered as prior knowledge. If you have not yet studied this, please do so before continuing with this example.

A nutation experiment is nothing more than a series of single pulse NMR experiment with an increasing pulse length (see Figure 1. In this way, the modulation of the signal during the pulse can be examined. Usually, the effects of chemical shift can be ignored during the pulse, which leads to a sine modulation of the signal intensity with a frequency ν_1: the nutation frequency. Knowledge of the nutation frequency (i.e. the RF field strength) can be used for setting up decoupling sequences, cross-polarization, and so on.

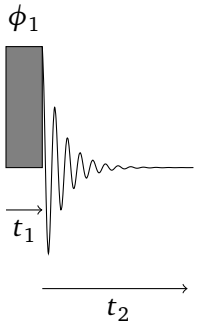


Figure 1: Pulse sequence of a single pulse nutation experiment.

2 Used data

For this example, we will use a Varian data set recorded at 14.1 T. The sample was a saturated solution of $KMnO_4$ in water, and the ^{55}Mn signal is being measured. In this data set, an array of experiment is performed, with the pulse length starting at 6 μs, and increasing with 6 μs with a total of 128 experiments. For the analysis, it is essential that the step size and initial value are equal.

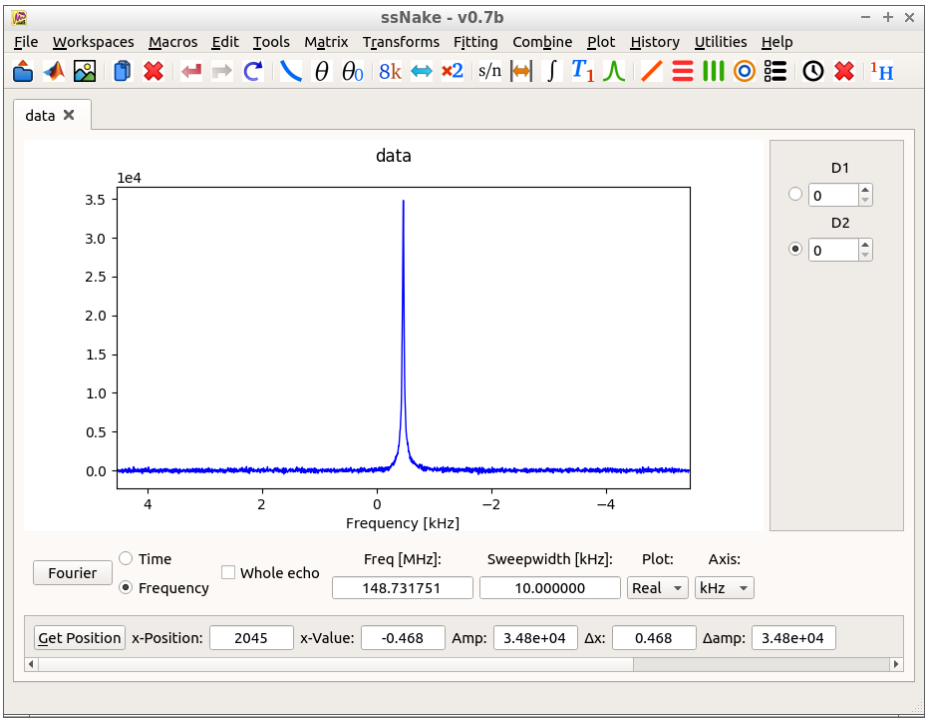
3 Processing

- Open the data file by using File ⟶ Open.
- Zero fill the data to 4k points (4096 points) by using Matrix ⟶ Sizing, and filling in the value in the Size box.
- Fourier transform via the 'Fourier' button in the bottom frame.

This shows us the spectrum of the first experiment (with a 6 µs pulse width).

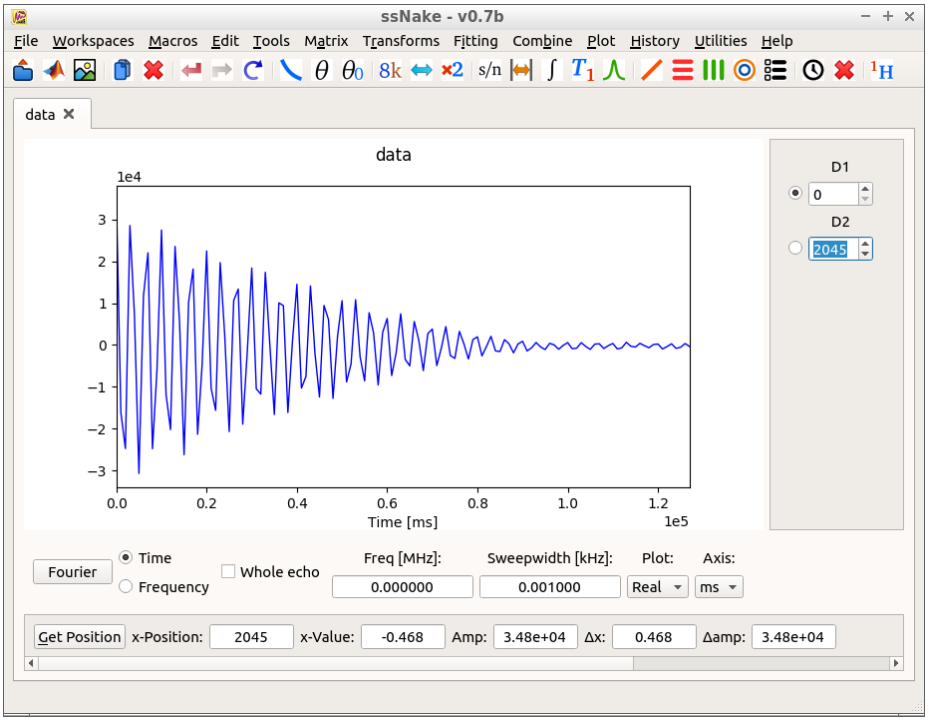
- Phase this spectrum to get a nice positive peak.

This gives us the following figure:



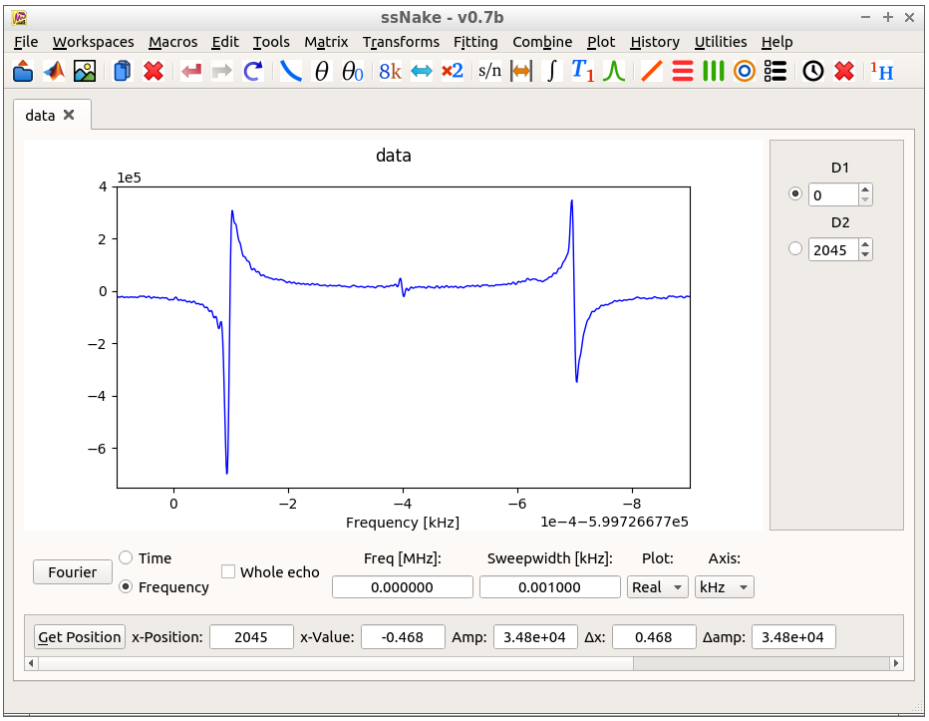
Now we can view the other experiments by increasing the value of the D1 box in the side frame. Note that we want to continue to view the D2 dimension, and therefore keep the bullet next to D2 as the active dimension.

Now we want to examine the change of the peak height along the D1 dimension. We therefore need to get the position of the peak (in data points). Push the 'get position' button in the bottom frame, and click on the highest part of the peak (zoom in the be extra accurate). This should print an x-position of approximately 2045. To view this D2 point along D1, copy this value to the D2 box, and push press enter. This gives the following Figure, and shows a sine oscillation with increasing pulse width:



- Zero fill along this D1 by using Matrix ⟶ Sizing and fill in 1024 points.
- Right shift the data with 1 point using Matrix ⟶ Shift Data, and pushing the right button once.

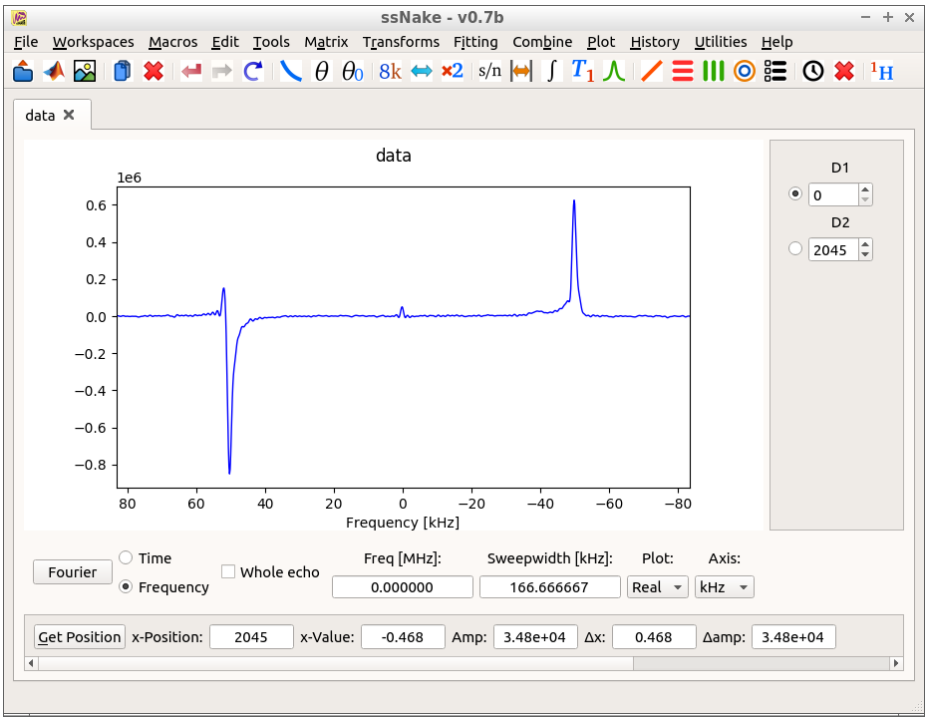
This gives the following Figure:



This last step is removing a data point from the end (in this case an added zero) and puts a zero at the front. This zero represents the spectrum with a pulse width of 0, which we did not measure (but we are sure it gives no signal). This way, we artificially made our oscillation start at $t = 0$.

- Phase with -90° to get the in-phase spectrum.
- Clear the reference frequency (none is needed) via Plot ⟶ Reference ⟶ Clear Current Reference.

We now must set the sweepwidth to the right value, as ssNake does not automatically understand what the D1 dimension means. In our case, the dwell time of the experiment is the stepsize of the pulse width: 6 µs. We can therefore type in the sweepwidth box in the bottomframe: '1000/6e-6'. '1/6e-6' gives us the sweepwidth in Hz, and the 1000 is added for conversion to kHz. Alternatively, '1/6e-3' leads to the same result in a shorter way. This leads to the following Figure, which is the final spectrum. It shows an (nearly) anti-symmetric spectrum with a peak at 50 kHz, so $\nu_1 = 50$ kHz in this case.

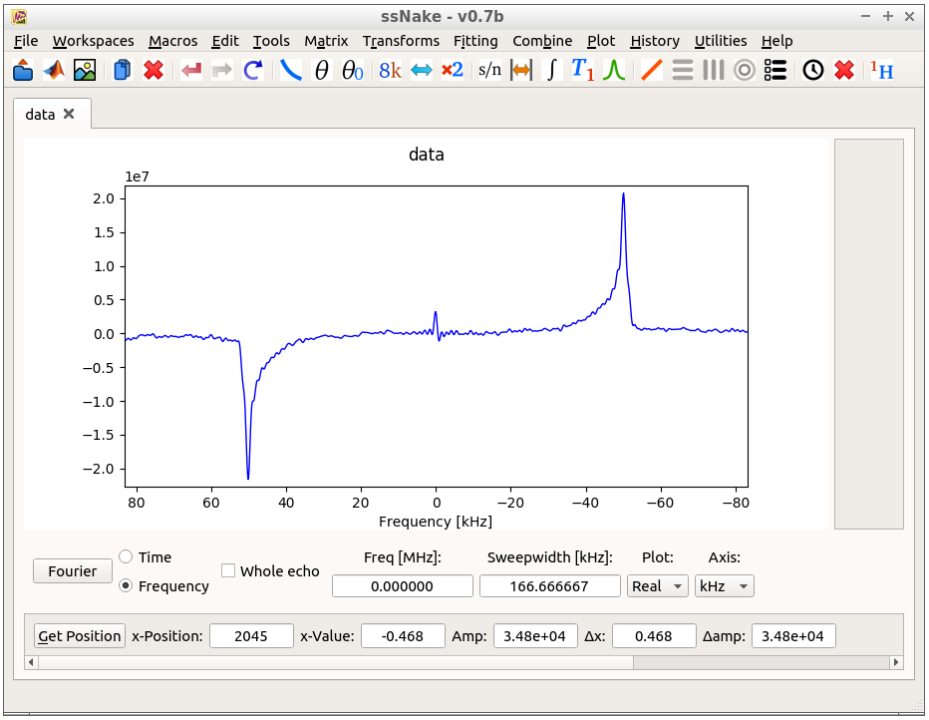


4 Alternatives

4.1 Integrate D2

When having phased the first spectrum in D2, Matrix ⟶ Region ⟶ Integrate can be used to integrate the peak, and get more convenient data. Use the tool by left clicking on the left and right of the peak and press OK. Continue the steps described above, but notice that the old D2

dimension has now been removed, and only the D1 remains. As there is only one dimension now, the dimension selector in the side frame has disappeared. Integrating usually gives a better representation of the nutation spectrum than the via the peak height. This gives the following Figure (after 41.5° 1st order phasing):



Note that looks quite different form the 'normal' processed spectrum. This probably means that the nutation frequency is not identical over the peak. This is probably caused by bad shimming, which gives a correlation between the NMR frequency (D2) and nutation frequency (D1).

4.2 Force symmetry

Ideally, the nutation spectrum has inversion symmetry (the left is -1 times the right). Sometimes it is convenient to force this. This can be done by performing Tools ⟶ Real (removing the imaginary part) before the Fourier transform in the D1 domain.

4.3 Correct DC offset

In some cases, the nutation signal does not decay to zero, but to some other value. This will give a peak at 0 Hz in the nutation spectrum. This can be avoided by subtracting the average of the last part of the nutation time signal from signal. This can be done by using Tools ⟶ Subtract Averages. Ideally, this is done before the zero filling in the D1 domain.

4.4 2D nutation

When following the regular method to get the nutation spectrum, the spectrum can be viewed for every point along the D2 dimension, as it is a 2D spectrum. Viewing this can also be done in

a contour plot via Plot ⟶ Contour.