REDOR processing in ssNake

30th July 2018

1 Introduction

The following will explain how rotational-echo double-resonance (REDOR) NMR data 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.

In a REDOR experiment, echo spectra are recorded with an increase in echo time τ. As the experiment is recorded in spinning fashion, the echo time must be multiple of the rotor period $1/\nu_\mathrm{r}$. In the experiment, the spinning cause the dipolar couplings to be less effective. The goal of a REDOR experiment is to get information on the dipolar couplings between two nuclei of a different type, while still having the resolution enhancing MAS active. This is done by performing the echo experiments twice: once as is, and other time using 180 degree pulses on the other nuclei. The goal of these pulses is to reintroduce (recouple) the dipolar coupling, causing a strong dephasing during the echo time. Essentially, this is a measure of the effective T_2^* polarization loss in the presence or absence of the dipolar coupling.

Measuring REDOR data leads to two data sets: one with, and one without the recoupling pulses. Both these data sets are an array of 1D spectra, for increasing echo times (number of rotor periods). For each echo time, we determine the integral (or peak height) of a peak. We get two integrals of the relevant peak: without pulse (S_wo) and with pulses (S_wi). The relevant information we need, is the relative difference between these:

$$\Delta S = \frac{S_\mathrm{wo} - S_\mathrm{wi}}{S_\mathrm{wo}}$$

A REDOR plot feature this ΔS as a function of echo time (or rotor periods).

2 Data

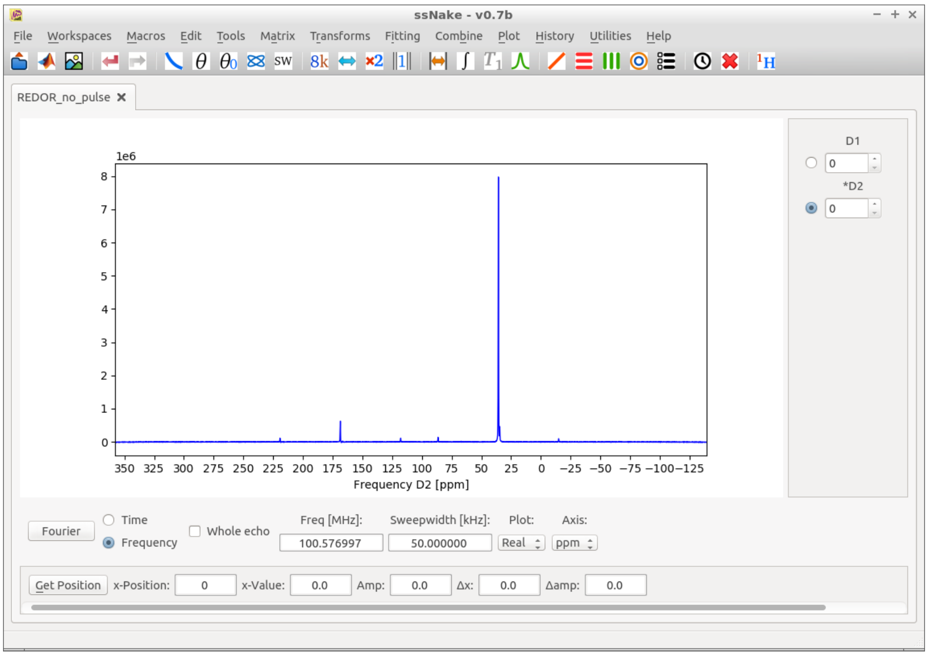
The data in this tutorial was recorded on a Varian 400 MHz machine on glycine using 5 kHz MAS. The data were recorded with 2 to 40 rotor periods per echo delay (with steps of 2).

3 Processing

Processing REDOR data is not that hard. Only a couple of steps are needed.

- Open the REDOR_no_pulse.fid data using File ⟶ Open
- Zerofill to 16384 points (Matrix ⟶ Sizing)
- Fourier Transform
- Phase with −6.0 degrees 0 order phasing (Tools ⟶ Phasing)

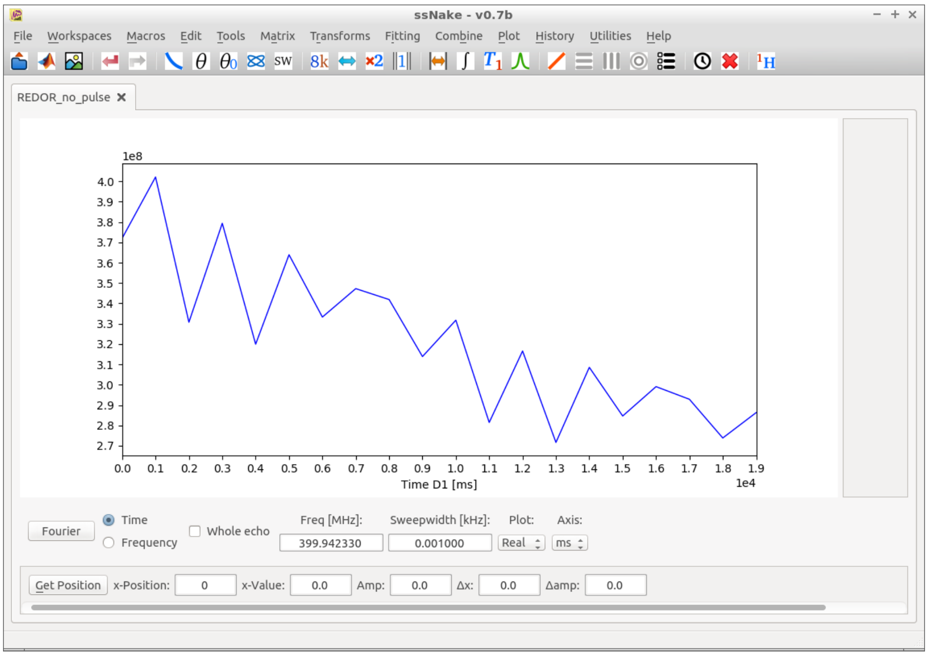
This gives the following:



In this case, we are interested in the right peak (CH_2). We want a plot of the integral of this line over all the 11 spectra. For this, we integrate the peak. Note that ssNake has two types of integral: via Fitting ⟶ Integrals, which can be seen as a way to ask ssNake 'what is the integral of this peak'. Or via Matrix ⟶ Region ⟶ Integrate, which integrates a region across all traces and changes the data to lose the integrated dimension. In this case, we need the latter.

- Use Matrix ⟶ Region ⟶ Integrate and click on the left and right side of the CH2 peak (ignore the tiny peak next to it for the moment). In my case, these are positions 5679 and 5861.

This should give:

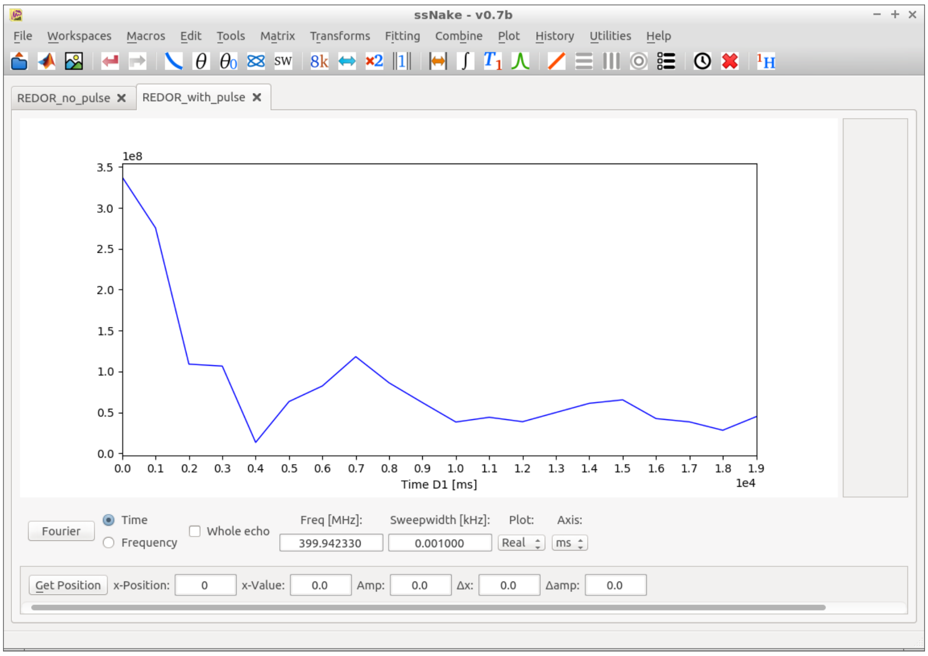


This shows how the integral decays in the experiment without the pulses (dipolar coupling disabled).

Now, we need to do the same for the data with the pulses.

- Open the REDOR_with_pulse.fid data using File ⟶ Open

Do the same processing as for the other data, but now with −5.0 degrees phasing. After integrating, this should give:



Now, there are multiple ways to continue, either you can export both data sets via File ⟶

Export ⟶ ASCII, and do further processing in your favourite software, or we can continue within ssNake. Just for the sake of it, we will continue in ssNake.

We want a plot of:

$$\Delta S = \frac{S_{\mathrm{wo}} - S_{\mathrm{wi}}}{S_{\mathrm{wo}}}$$

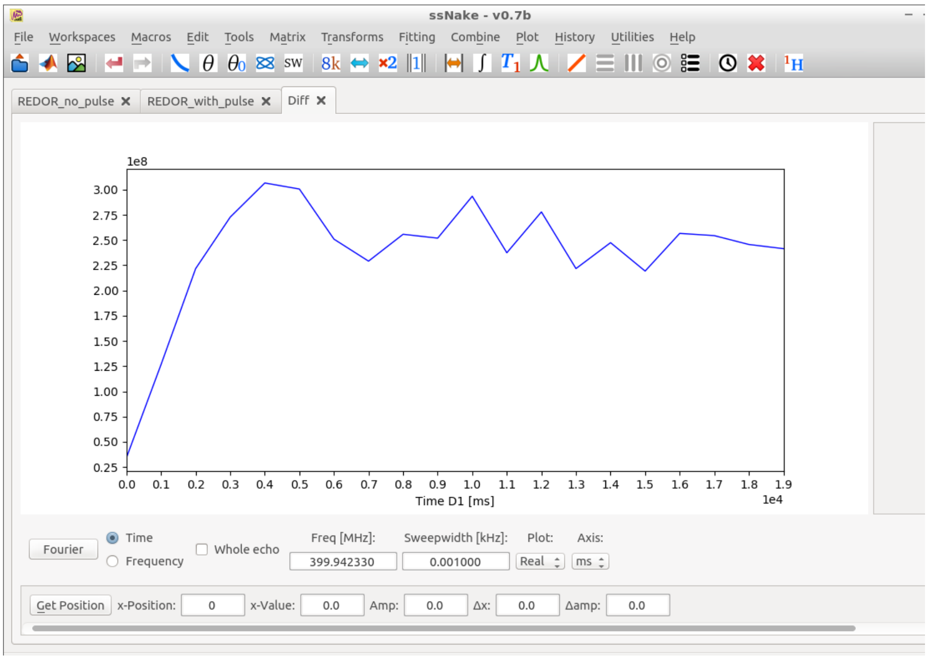
for each point. To do this, we make a copy of the no_pulse data set:

- When on the 'REDOR_no_pulse' data, use Workspaces ⟶ Duplicate (and name it 'Diff').

Now, we must subtract the data with the pulses. While in the 'Diff' workspace:

- Combine ⟶ Subtract (and select the 'REDOR_with_pulse' data)

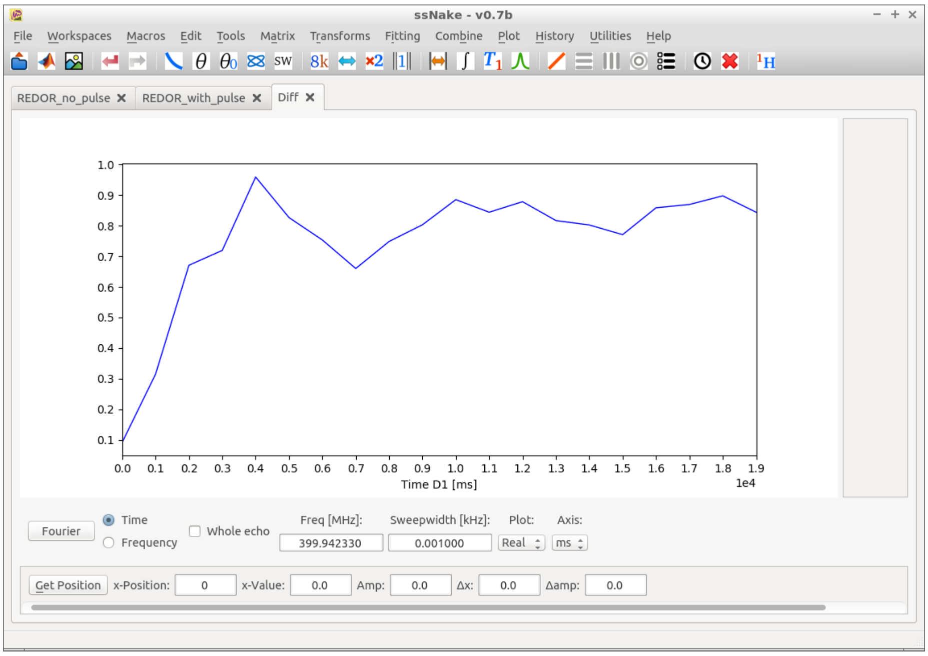
This should give:



Now, we must divide the data by S_{wo}. However, we must realize that, when dividing data, we do not want to do a complex divide (we have complex numbers). Therefore, we must first take the real values of the data:

- In workspace 'Diff' do Tools ⟶ Real
- In workspace 'REDOR_no_pulse' do Tools ⟶ Real
- In workspace 'Diff' do Combine ⟶ Divide, and select 'REDOR_no_pulse'

This gives:

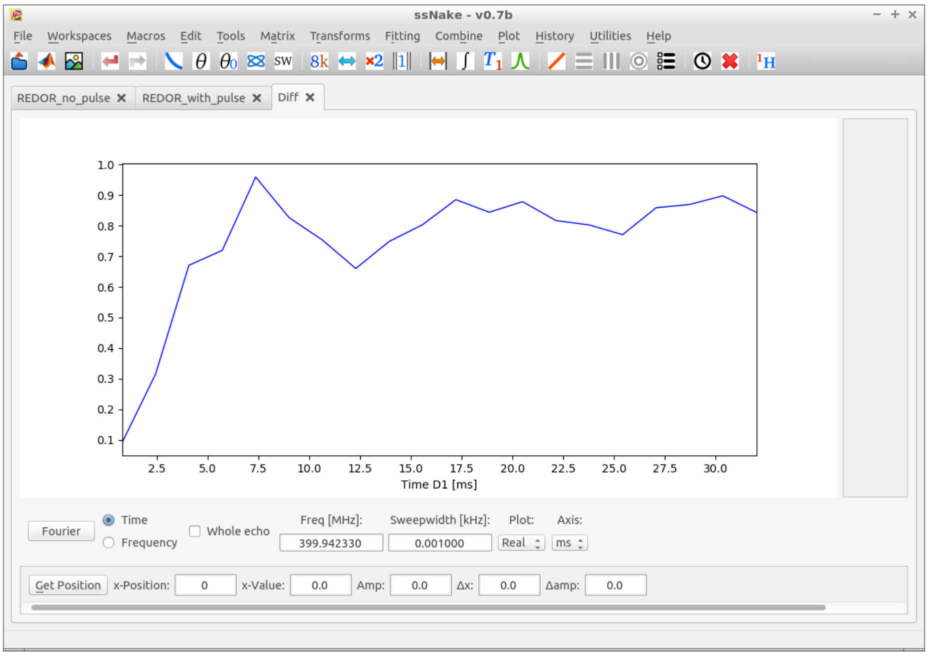


Which is clearly between 0 and 1 (as it should be).

Now, we can set the axis. The first point was 2 rotor periods, the last 40. Note that this means 4 and 80 for the total echo time. The spinning speed was 5 kHz, so a period is 0.2 ms. This means that the axis should go from 0.8 to 32 ms:

- In workspace 'Diff' use Plot ⟶ User X-axis (Type: Linear, Start: 0.8e-3, Stop: 32e-3)

This gives the final figure:



Which can be saved via File ⟶ Export ⟶ Figure. Also note that this user axis is reset every time the axis needs to be re-calculated (in case of a Fourier transform for example). This data is saved as ‘Final.mat’ and is delivered with this tutorial.