Whole echo acquisition data processing using ssNake

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1 Introduction

In this tutorial, we will explain how to processed a whole echo acquired data set. The tutorial provided with the ssNake program is considered as prior knowledge. If you have not yet studied this, please do so before continuing with this example.

Whole echo acquisition is a nice trick to make powder patterns easier (and thus more accurate) to phase. NMR signals from powders have the tendency to decay quite rapidly due to destructive interference. Using an echo, the start of the signal can be 'delayed ' in such a way that it does not fall within the dead time, and allows for a proper recording of the signal. Whole echo acquisition means that the recording of the signal is started well before the echo maximum, in such a way that both the rising and decay of the echo is recorded. When processed correctly, the imaginary part of the spectrum goes to zero, and phasing is straightforward.

2 Used data

For this example, we will use a data set recorded on 14.1 T Varian spectrometer. The sample was a fine powder of lanthanum fluoride (LaF_3), and the ^{139}La signal was recorded. The settings were such that the echo maximum is in the centre of the acquisition window.

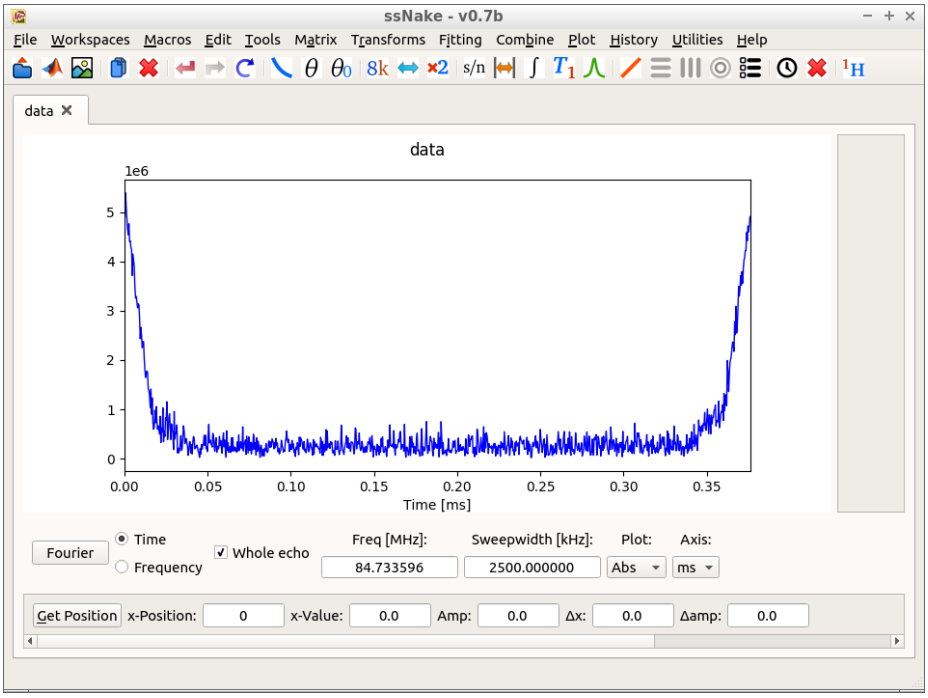
3 Processing

- Load the data file by using File ⟶ Open (or drag and drop).
- Put the display on absolute (Abs) via the bottom frame under 'Plot:'

Viewing in absolute mode helps to locate the echo maximum. Now we need to swap the echo and move one part at the start, and the other to the end of the FID. As the signal is symmetric around the echo maximum, this will force the imaginary part of the spectrum to zero.

- Swap the echo via 'Tools ⟶ Swap Echo' and left-click on the echo top to set the index (about 468), and press OK.

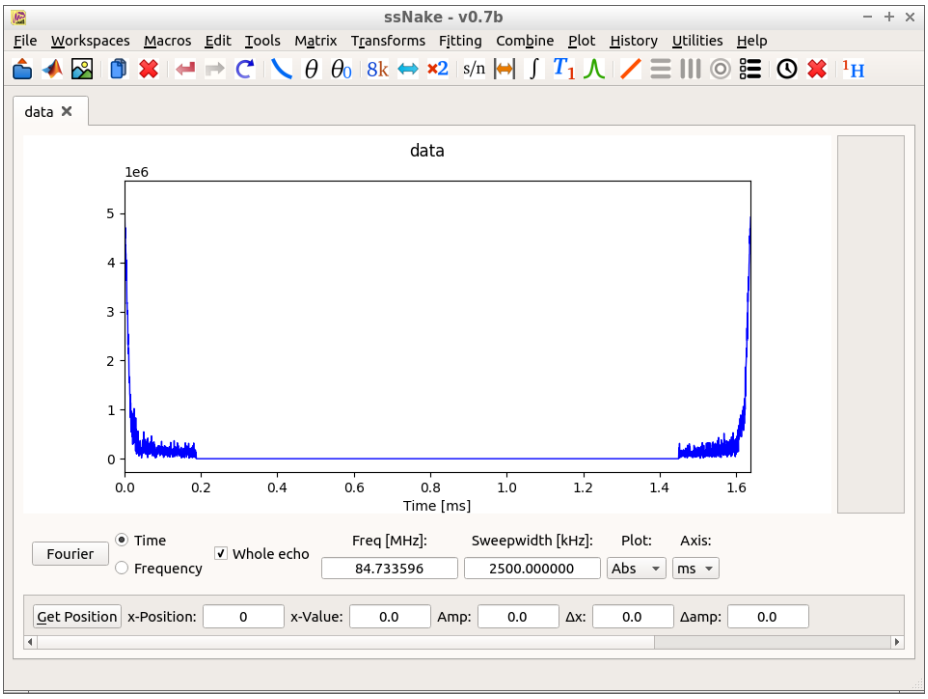
This will make the FID look like this:



Note that the 'Whole Echo' check box in the bottom frame has now been activated. This makes sure that zerofilling is now done at the centre instead of at the end of the signal. Also, apodizing is done in a symmetric way.

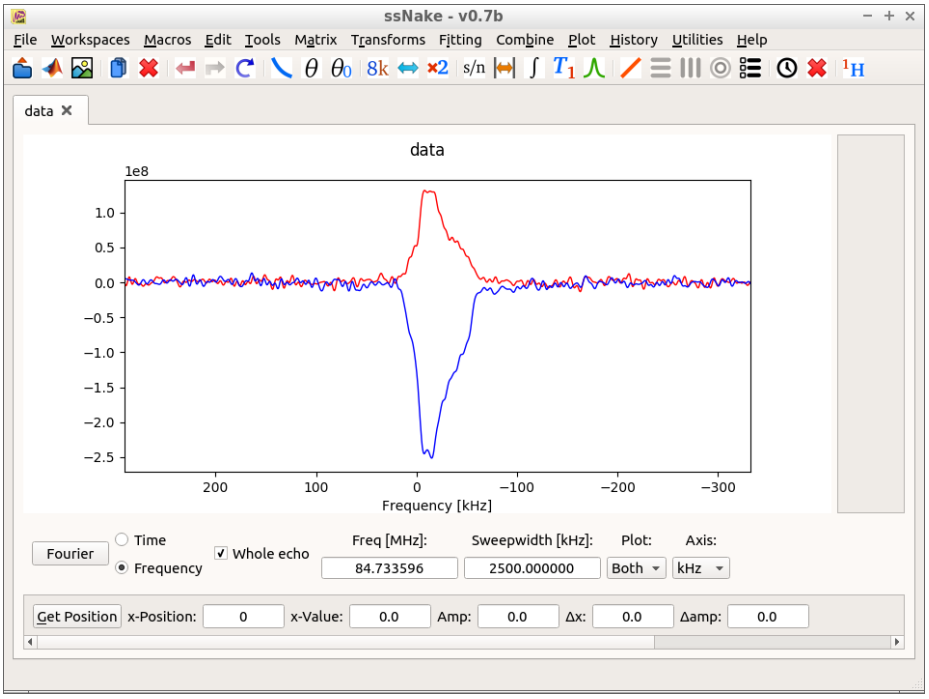
- Zerofill to '4k' points via 'Matrix ⟶ Sizing'
- Apodize using 1500 Hz Lorentzian broadening ('Tools ⟶ Apodize')

This should show an FID similar to this:



- Fourier transform via the ‘Fourier’ button
- Put the display at ‘Both’ via the bottom frame, to show the real and imaginary part of the spectrum

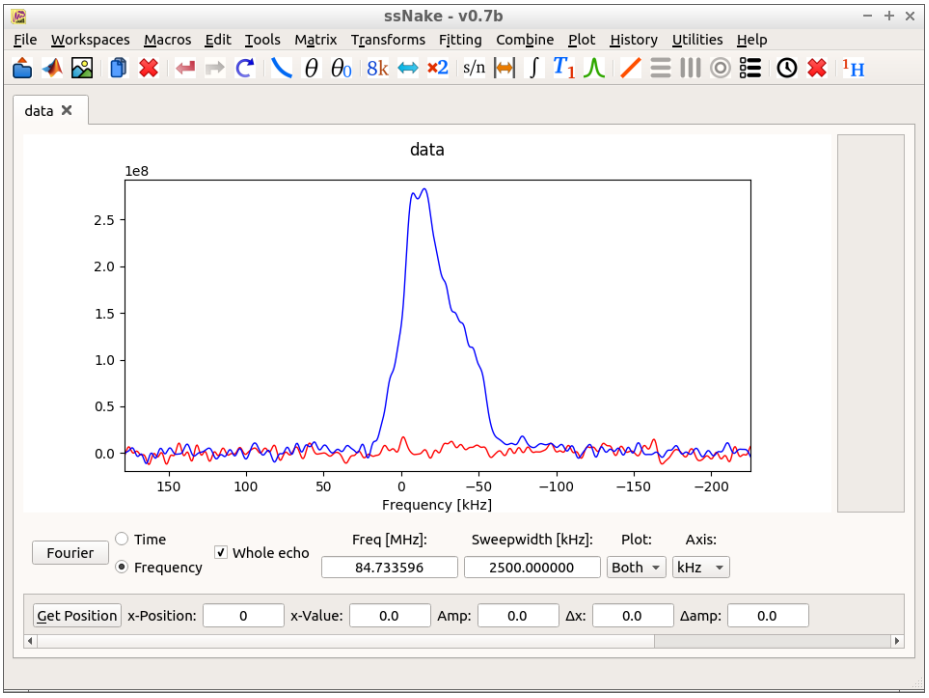
Because of the ‘Whole Echo’ setting the first data point of the FID is also no longer multiplied by 1/2 before the Fourier transform. The resulting spectrum should look like this (zoomed in):



To obtain the desired spectrum, we now need to phase it to make the imaginary part zero, and the real part positive:

- Phase with -152° zero order order phasing (‘Tools ⟶ Phasing’)

This results in a final spectrum:

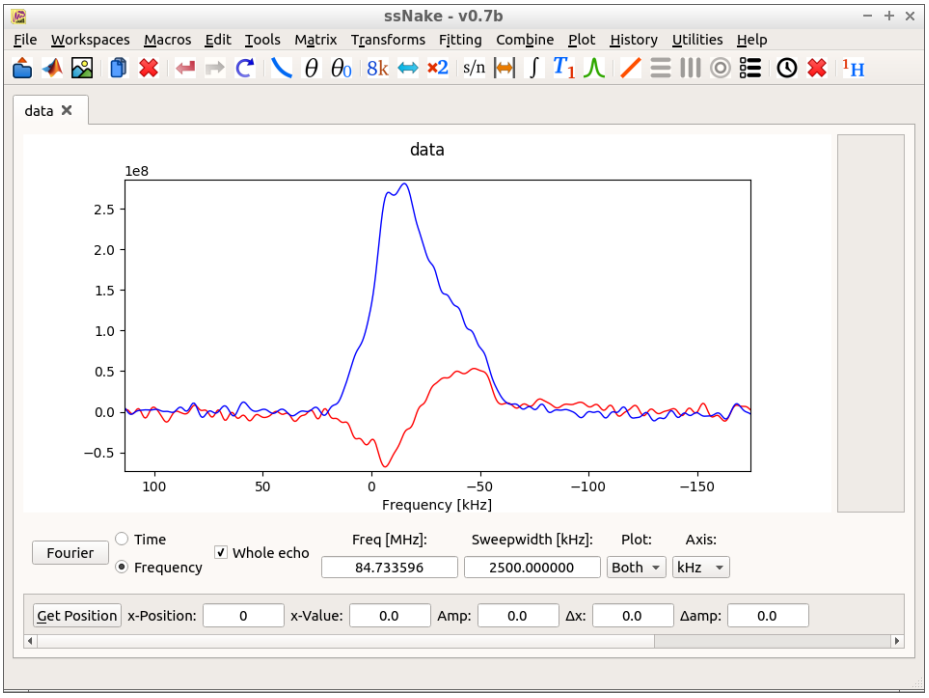


Optionally, you can now fit this 2nd order quadrupolar lineshape via 'Fitting ⟶ Quadrupole'. This should result in $C_Q = 15.6$ MHz, and $\eta = 0.78$. Do remember that ^{139}La has a spin quantum number of 7/2.

4 Alternatives

4.1 Wrong echo top

In some cases, the echo maximum can be difficult to locate. When the echo is split in the wrong location, a first order phasing is introduced to the spectrum, and zero order phasing will still lead to some imaginary component. When the echo is swapped at index 461, the following zero order phased (-154.7°) spectrum is found:



This can be fixed by using first order phasing. After first order phasing (2332° at pivot point -20317) the properly phased spectrum is recovered: