Supporting Information

Ultra-high Resolution in Low Field Tabletop NMR Spectrometers

Experimental details

J-res

Relaxation delay: 1s
Number of scans: 8
Number of data points: 1024 (F2) and 128 (F1)
Spectral width: 1250 Hz (F2) and 50 Hz (F1)
Pulse length: 7 μs
Experimental time: 1 hr 5 min

COSY

Relaxation delay: 1s
Number of scans: 8
Number of data points: 1024 (F2) and 512 (F1)
Spectral width: 1250 Hz in both the dimensions
Pulse length: 7 μs
Experimental time: 2 hr 20 min

TOCSY

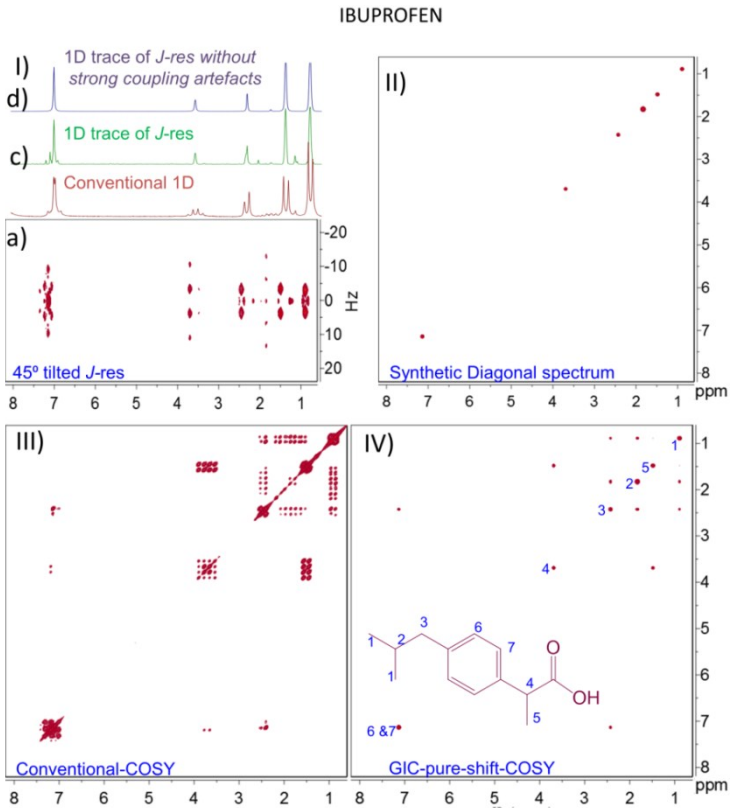
Relaxation delay: 1s
Number of scans: 8
Number of data points: 1024 in both the dimensions
Spectral width: 1250 Hz in both the dimensions
Pulse length: 7 μs
TOCSY mixing time: 100 ms (achieved with the MLEV-17 composite pulse sequence, wherein 40 μs of low power pulses are used)
Experimental time: 4 hr 40 min

Processing details

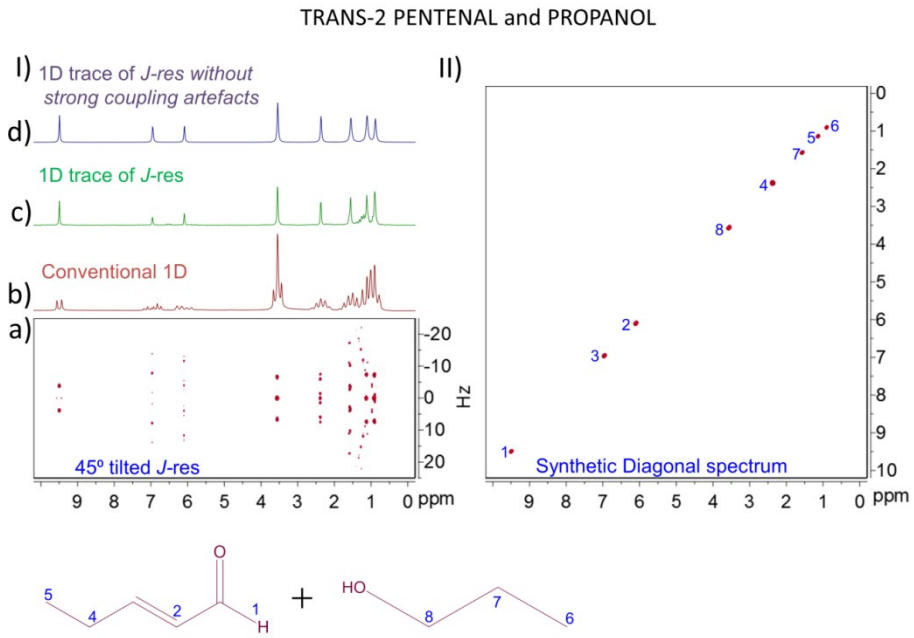
All the data sets are zero-filled to 1024 points in both the dimensions. For COSY and TOCSY, sine-square apodization is used at 0^{o}.

Pure Shift GIC Processing

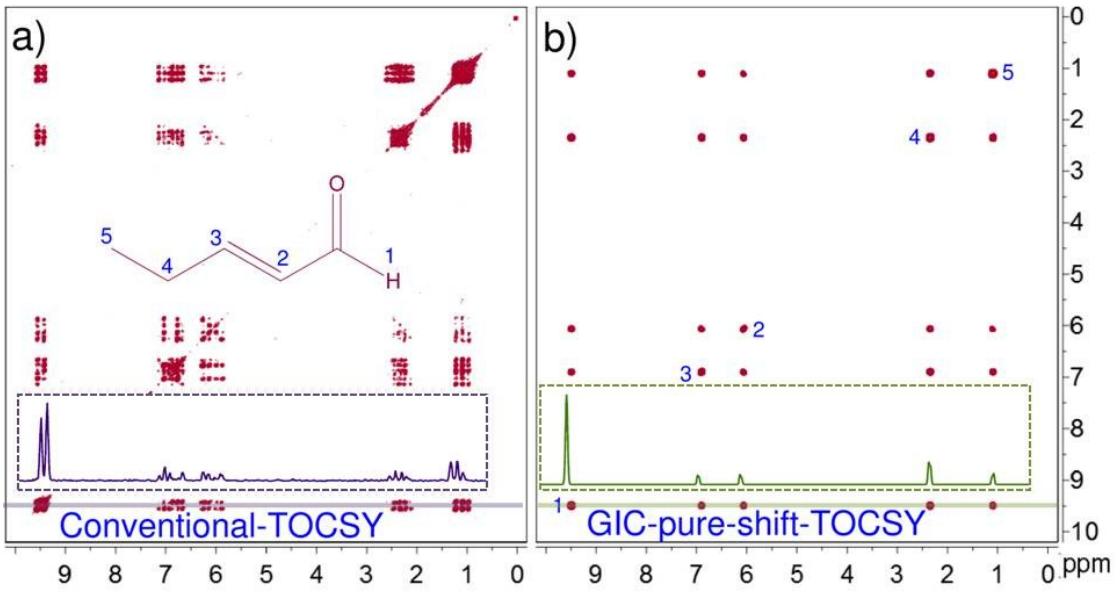
On the 60 MHz tabletop spectrometers, pure shift spectra were obtained from the following procedure. In this method, first, the 45º tilted 1D-homodecoupled trace of *J*-Res serves is used as a base for the pure shift spectrum. Subsequently, advanced line fitting in the Mnova software helps in selecting only the pure shift resonances (without any strong coupling artefacts and noise in the spectrum), which enables obtaining clean synthetic diagonal spectrum from the make2D script. This spectrum acts as a filter in combination with the conventional 2D homonuclear experiments by the GIC formalism. However, the complex coupling information still present along the indirect dimension can be suppressed with the aid of the direct covariance processing, and that helps in obtaining the F1 and F2 decoupled spectra with enhanced resolution.



Supporting Figure 1: Illustrates the GIC processing procedure involved in obtaining the pure shift NMR spectrum at 60MHz on Ibuprofen sample. Herein, the required 1D pure shift spectrum can be obtained from the 45^0 tilted 1D trace of *J*-res (I(c))(*J*-res spectrum is shown in(I(a)). Here, the 1D trace of *J*-res has nicely resolved resonances (without any scalar coupling information) when compared with the conventional-1D (1(b)).However, it still has some strong coupling artefacts, which can be eliminated by using the advance line fitting options in the Mnova and the obtained1D pure shift spectrum is very clean (I(d)) and that helps in generating the 2D diagonal spectrum(II) with the aid of make2D script processing option. Subsequently, this synthetic diagonal spectrum is combined to the conventional 2D-COSY (III) with the GIC processing formalism followed by using the direct covariance, which resulted in a spectrum with enhanced resolution (GIC pure shift COSY spectrum (IV)).



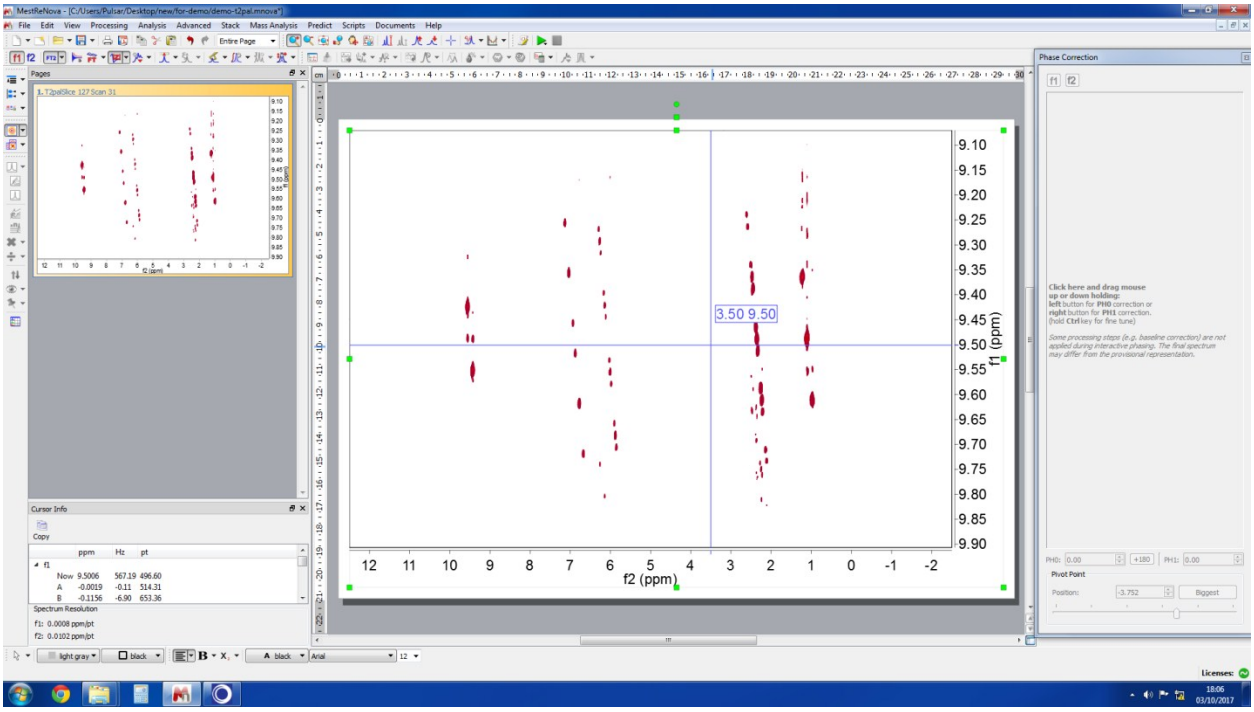
Supporting Figure 2: Illustrates the processing procedure to obtain the 2D diagonal spectrum, which is a basic requirement for the GIC formalism. This process has been demonstrated on another sample, mixture of trans-2-pentenal and propanol. Herein, J-res 1D trace (I(c)) is considered from the 45^0 projection of J-res (I (a)) experiment. This spectrum has nicely resolved singlets compared to the conventional 1D (1(b)). Further, strong coupling artefacts have been removed to generate clean 1D (1(d)), which serves as a base to obtain the 2D diagonal spectrum (II).



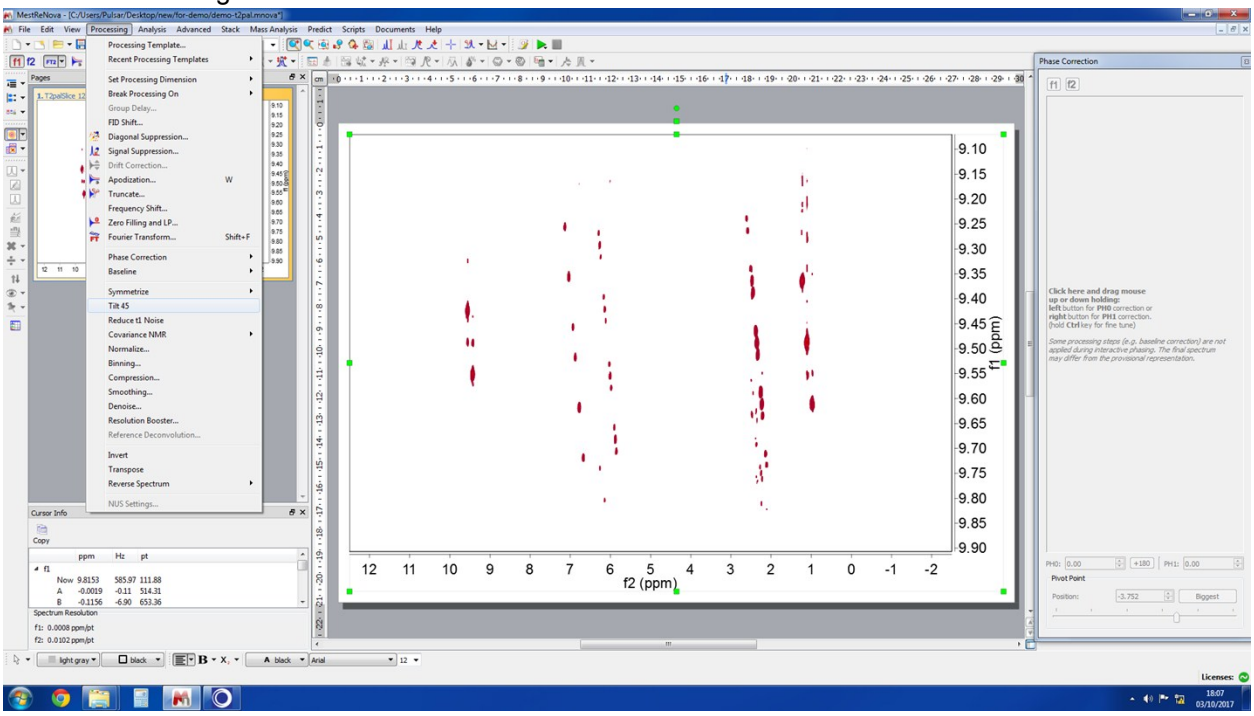
Supporting Figure 3: Comparison of internal traces obtained from the conventional TOCSY (a) and the respective pure shift GIC TOCSY (b).

Step by step implementation of pure shift GIC processing

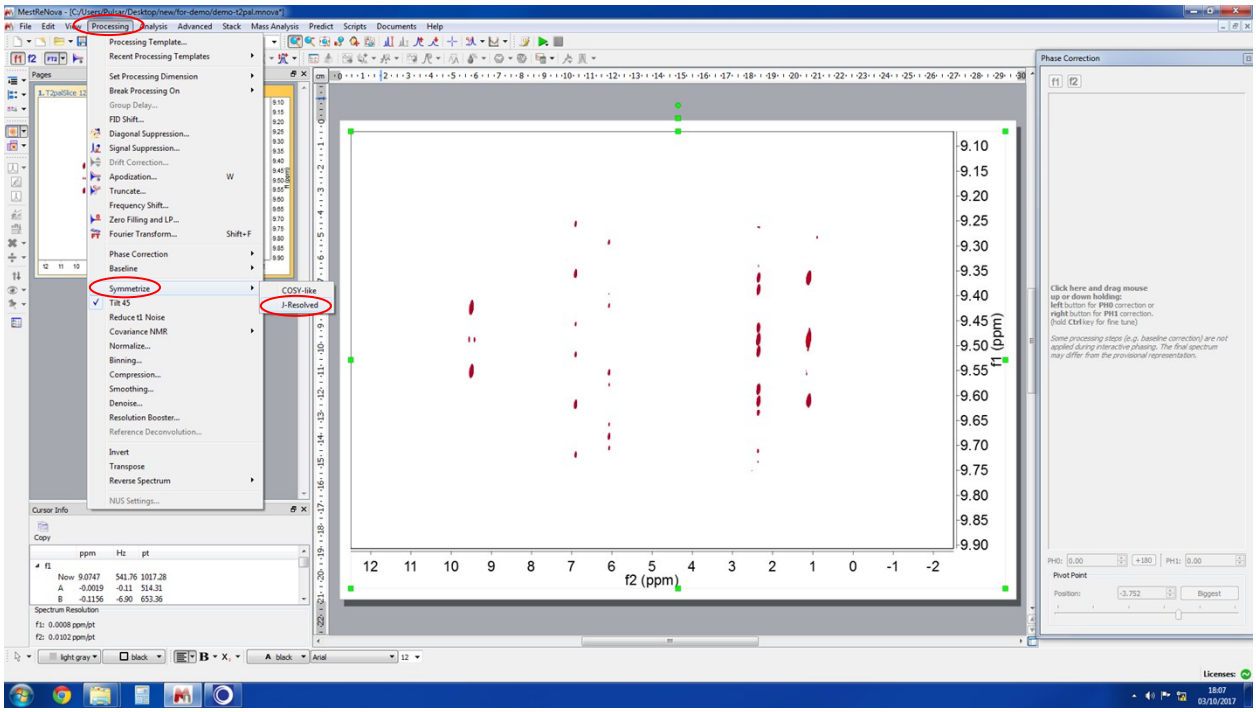
1. Import the J-res spectrum into the Mnova software



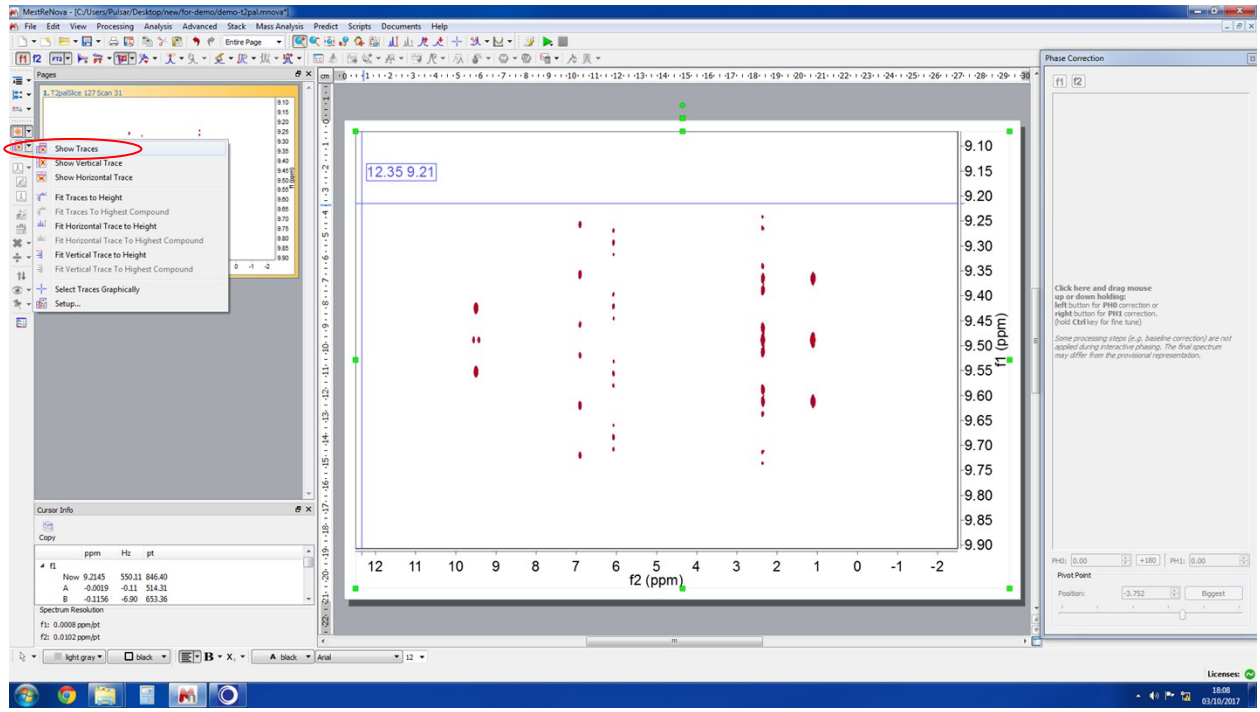
2. Processing tab → Tilt 45º



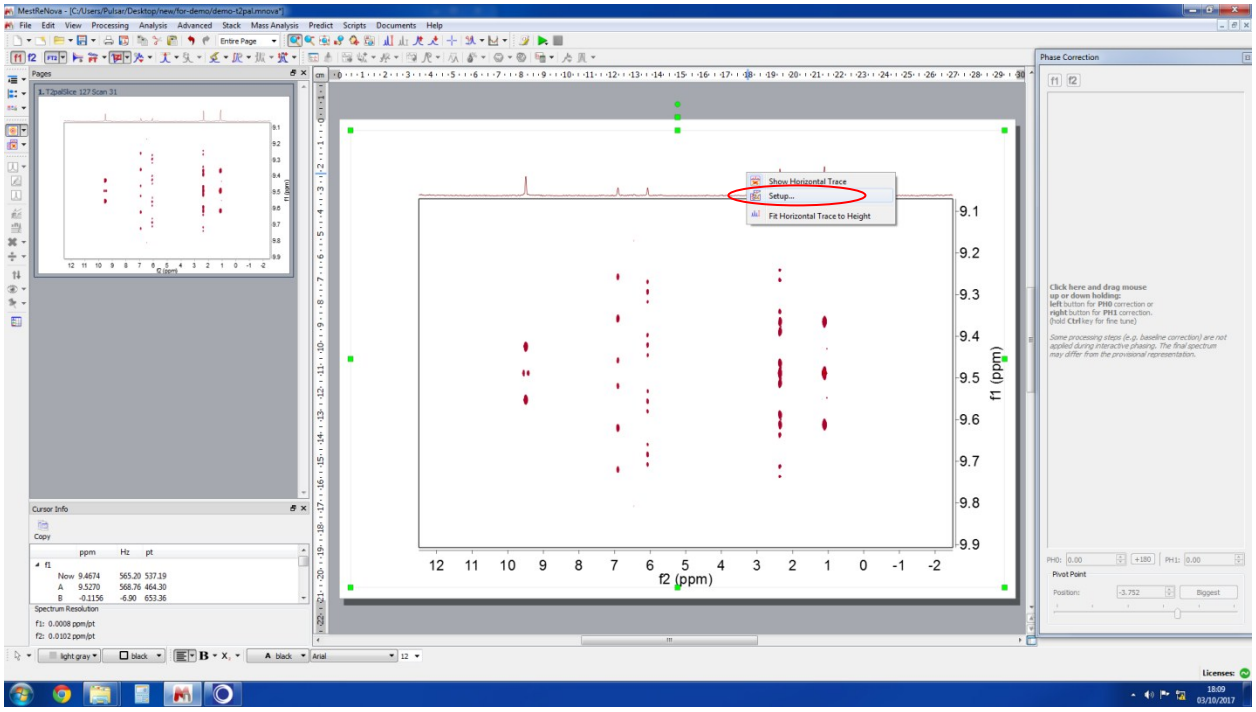
3. Processing tab →Symmetrize → J-Resolved



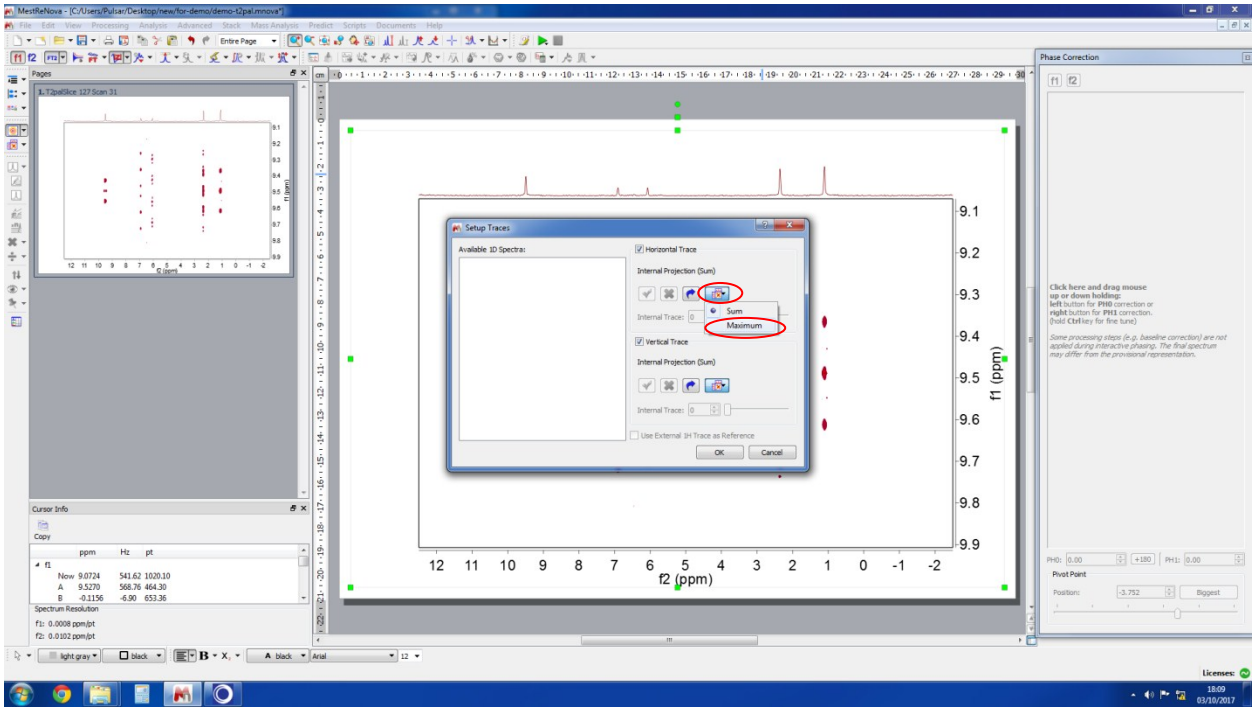
4. Show traces



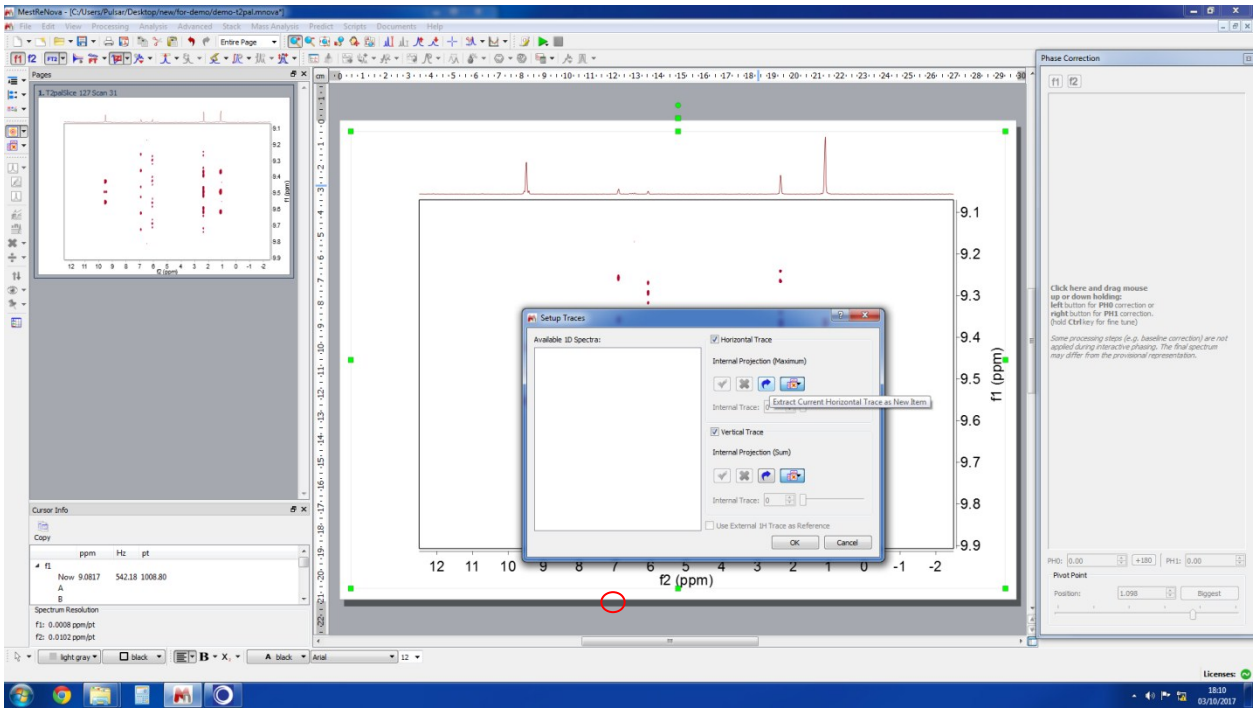
5. Horizontal projection → Right click → Setup



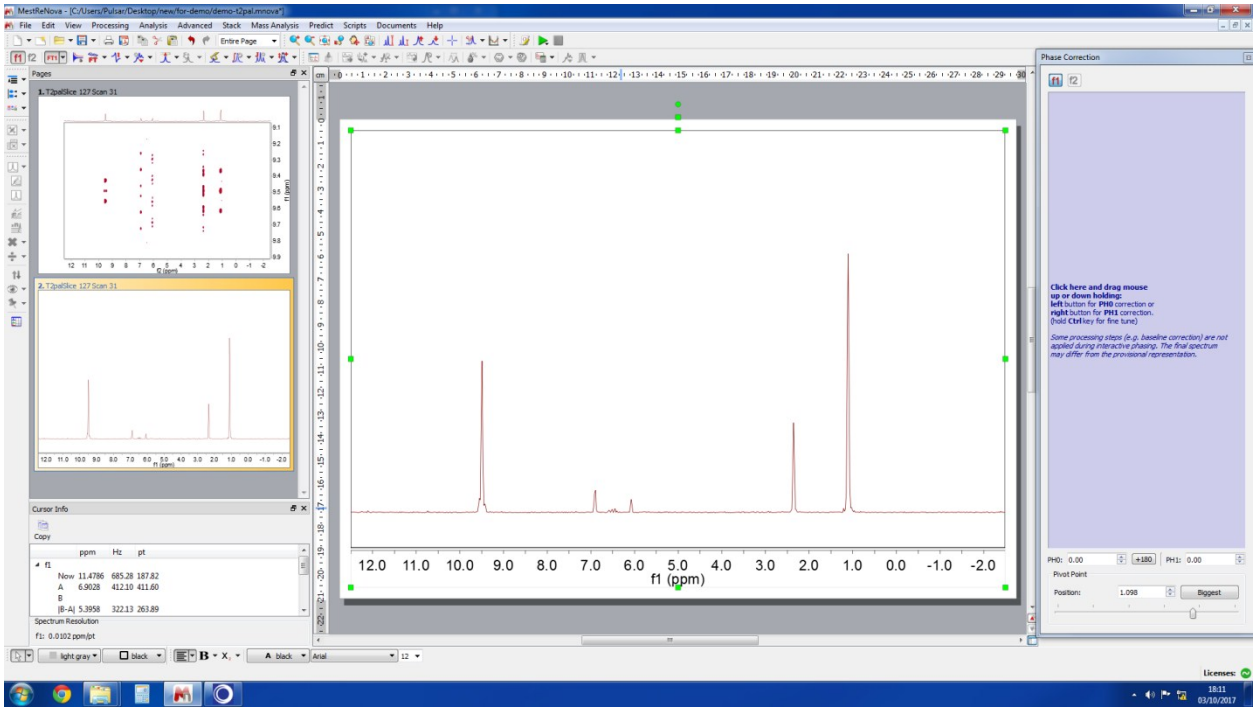
6. Trace Maximum



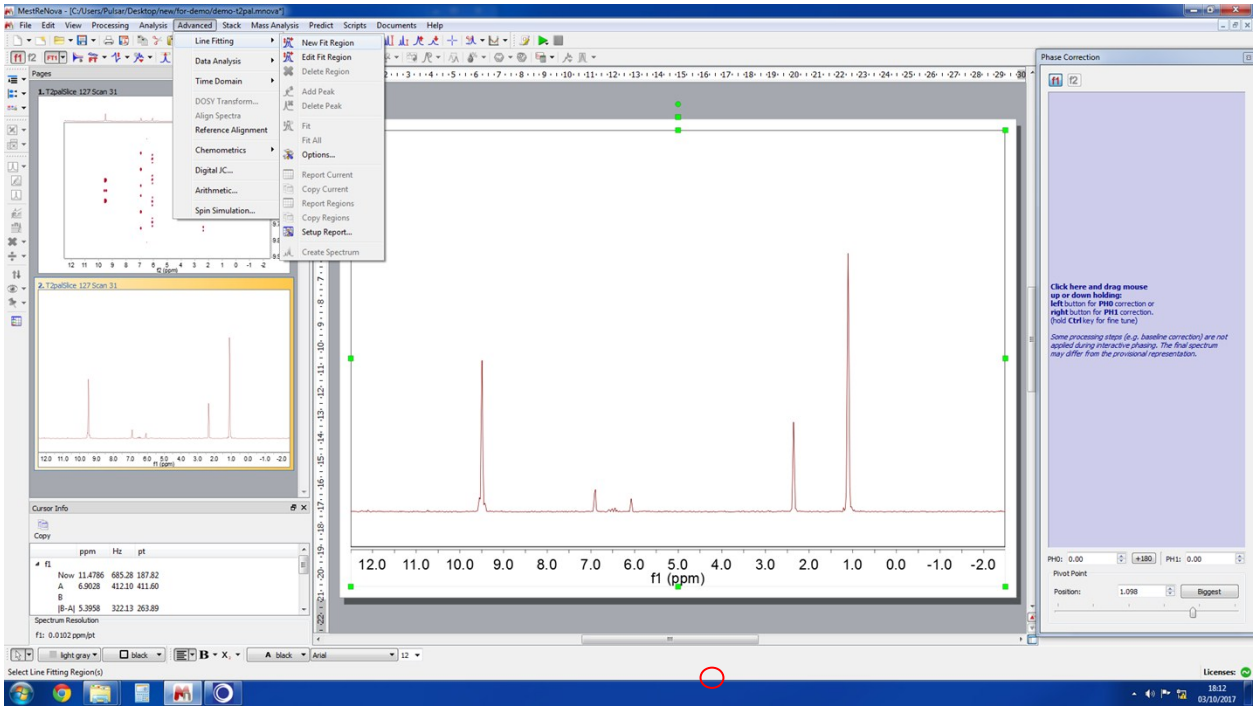
7. Extract Current Horizontal Trace as New Item



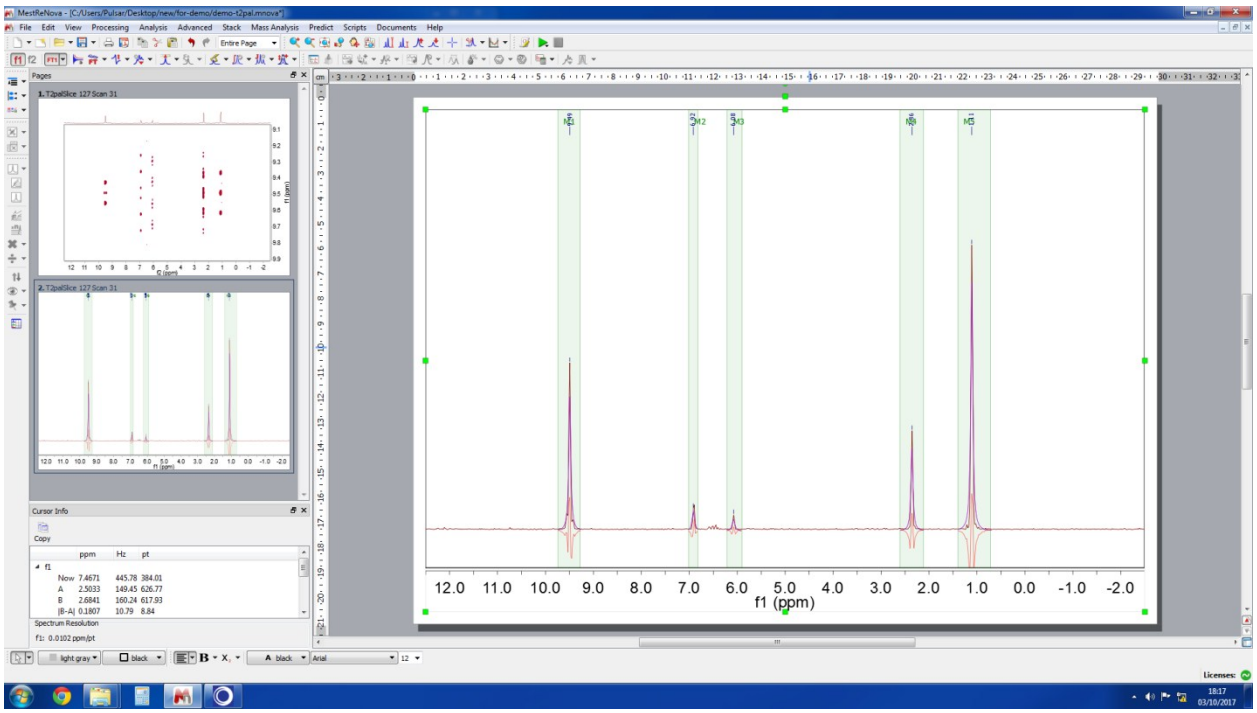
8. Around 6.5 ppm, it has strong coupling artefacts and they have to be removed



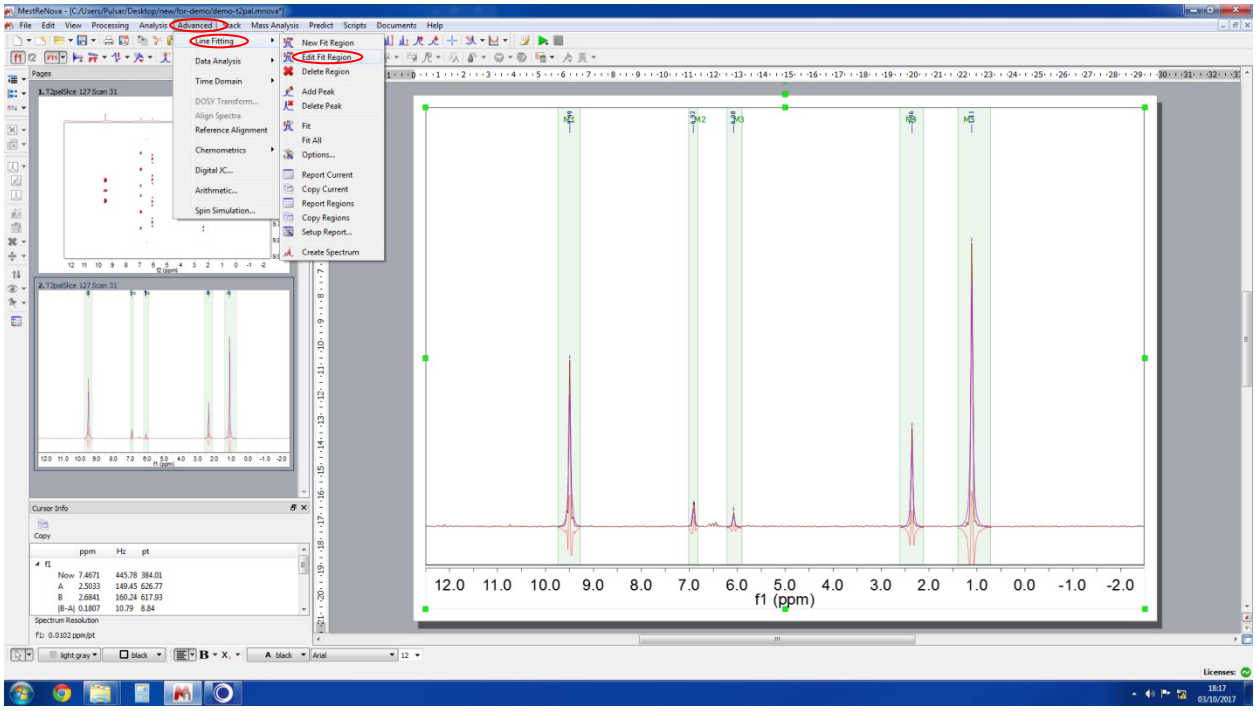
9. Analysis → Line Fitting → New Fit Region



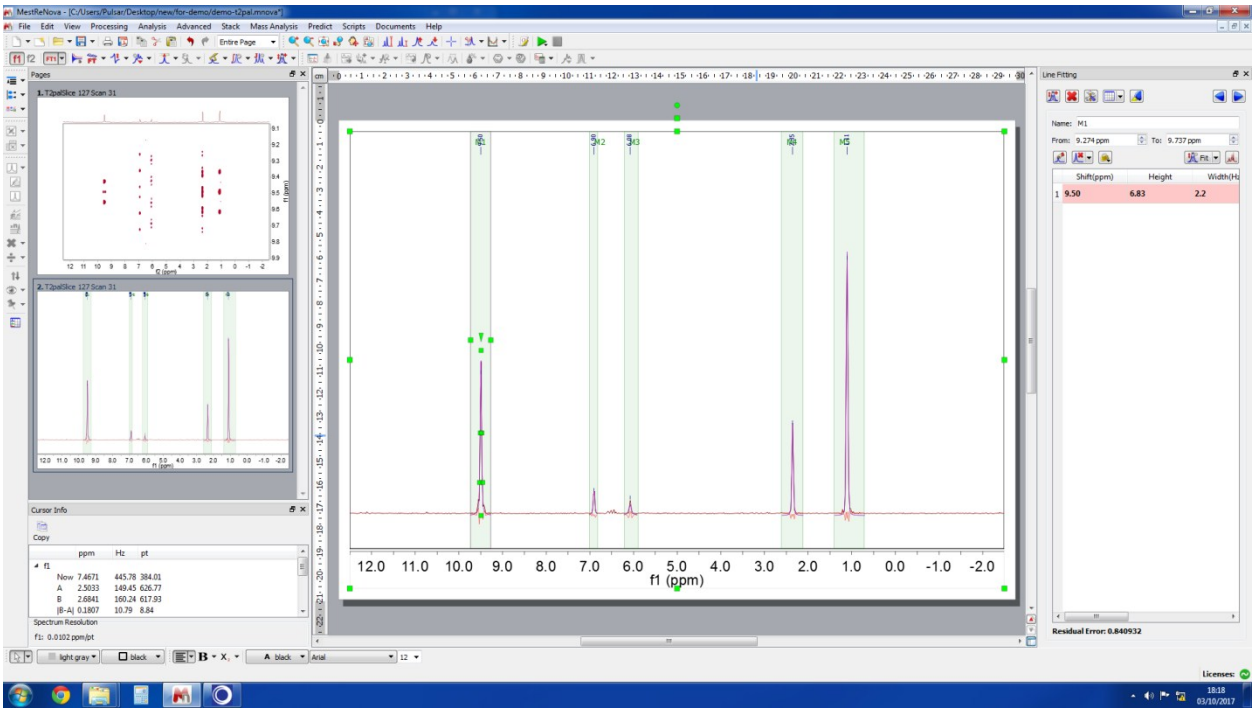
10. Select all the peaks and omit the strong coupling artefacts



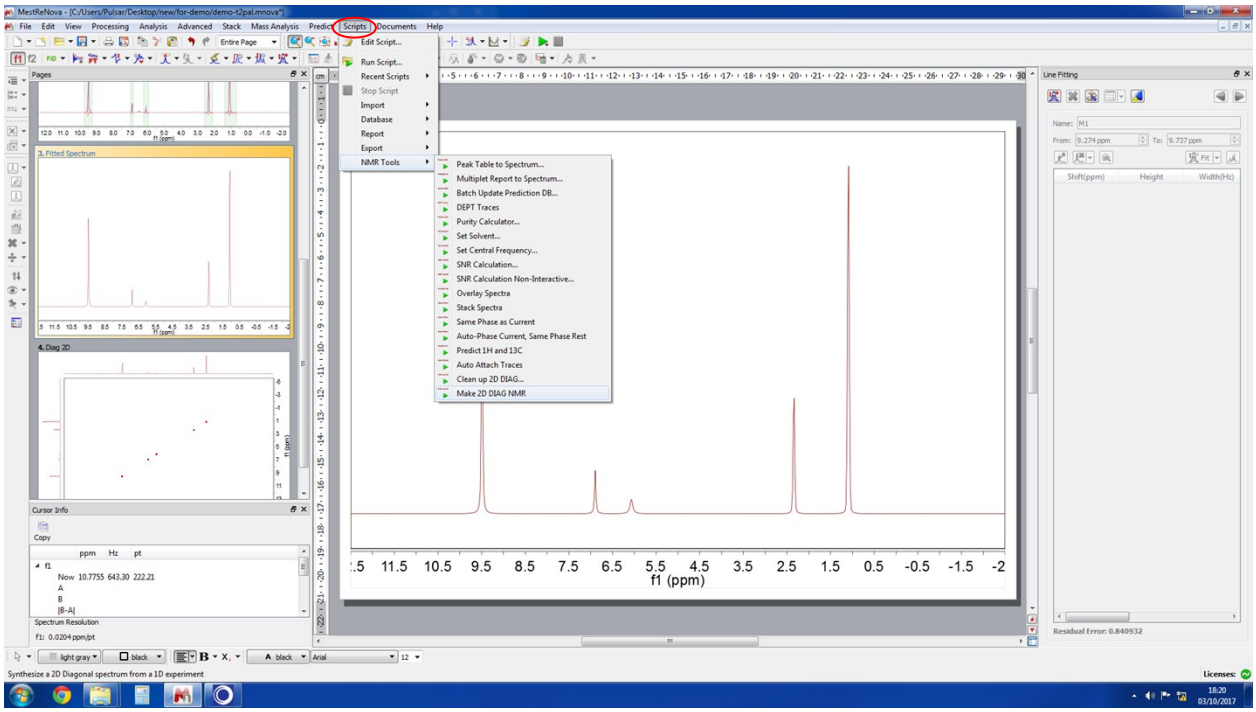
11. Advanced → Line Fitting → Edit Fit Region

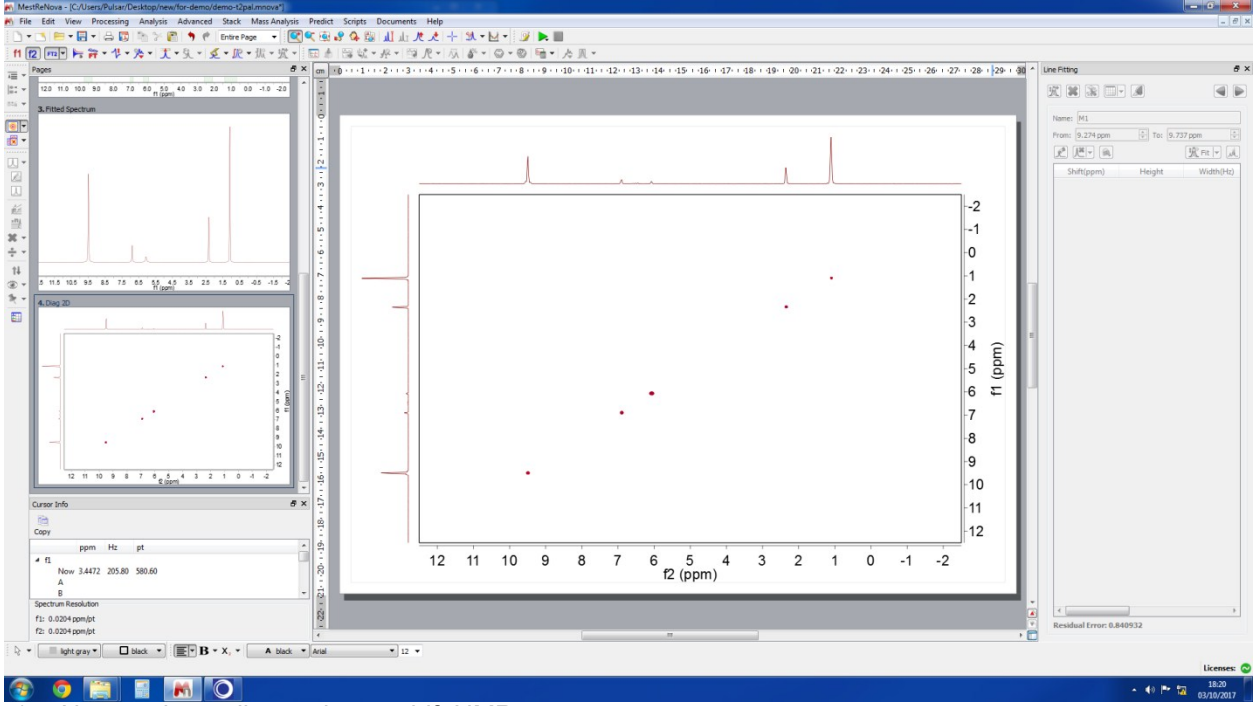


12. Fit → Fit All → Create Spectrum (Now we have artefact and noise free pure shift spectrum)



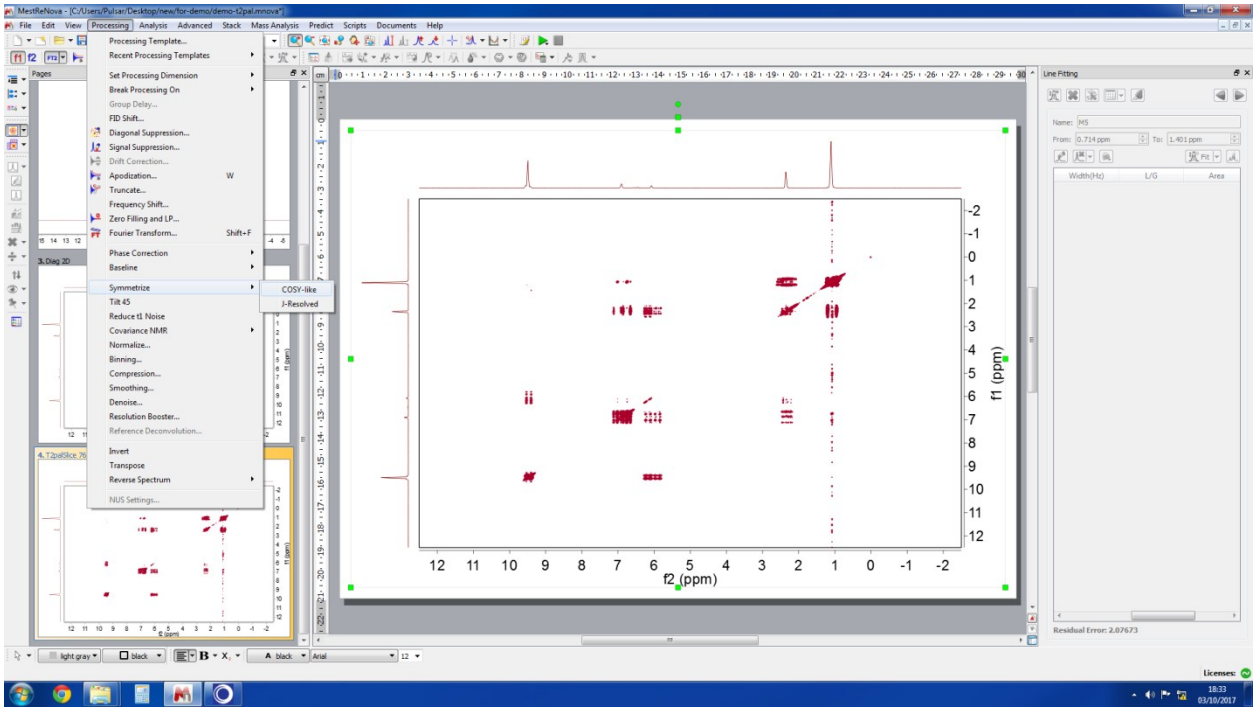
13. Scripts → NMR Tools → Make 2D DIAG NMR



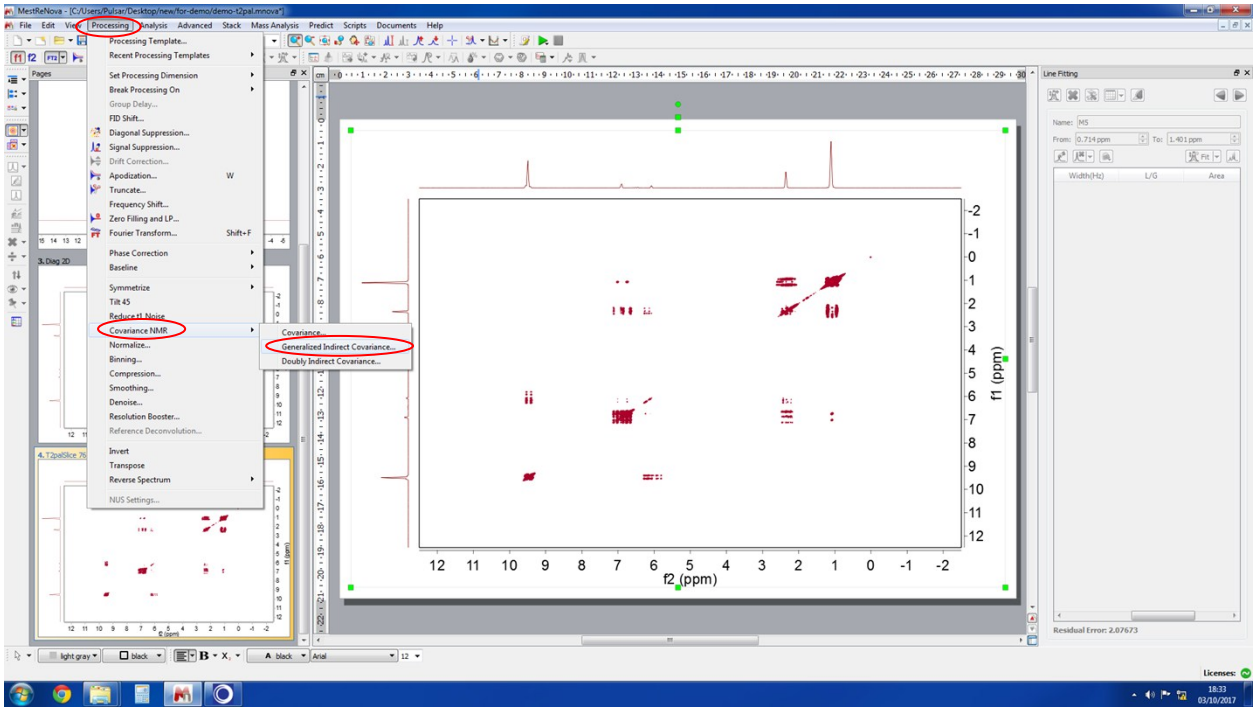


14. Now we have diagonal pure shift NMR

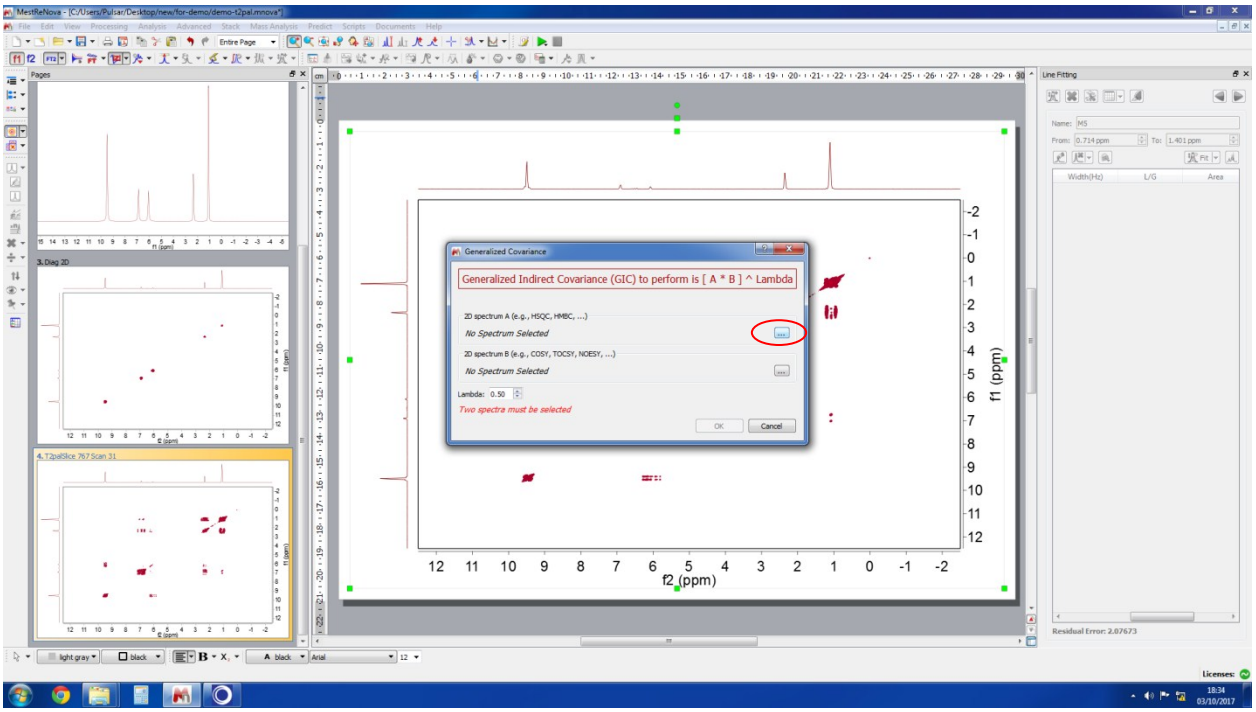
15. Import COSY → Symmetrize → COSY-like (it is the time to perform GIC pure shift processing)



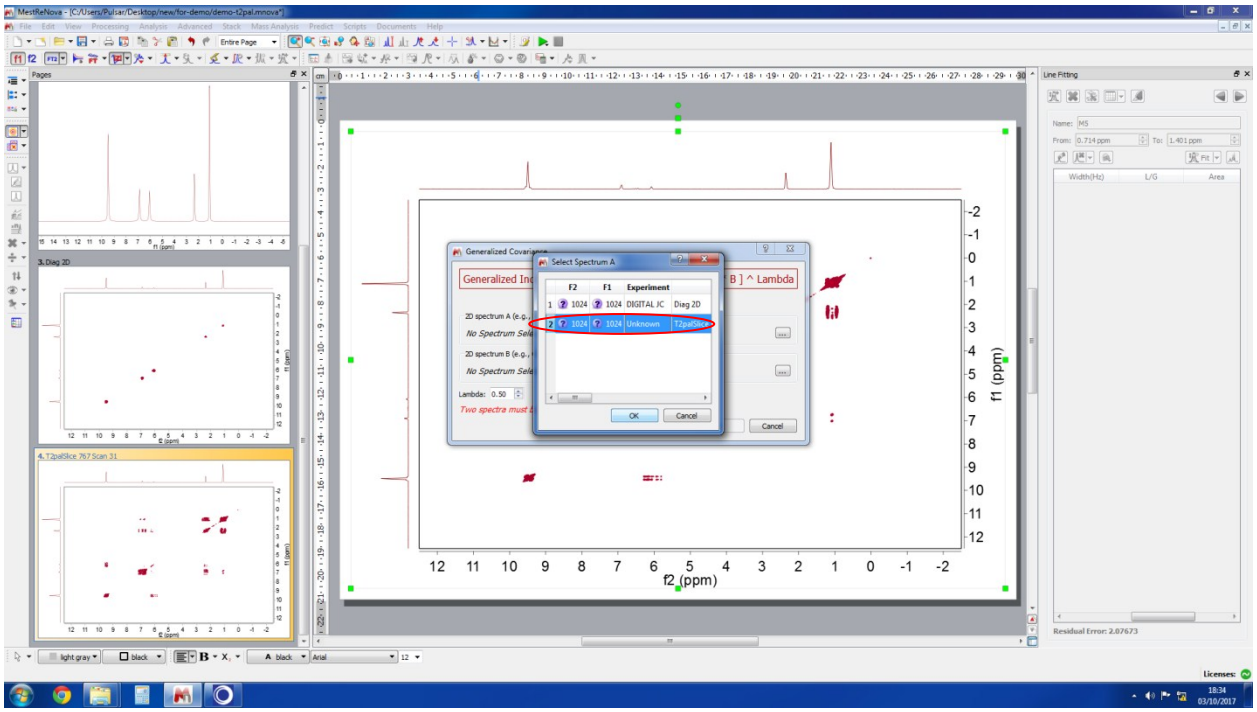
16. Processing → Covariance NMR → Generalizes Indirect Covariance



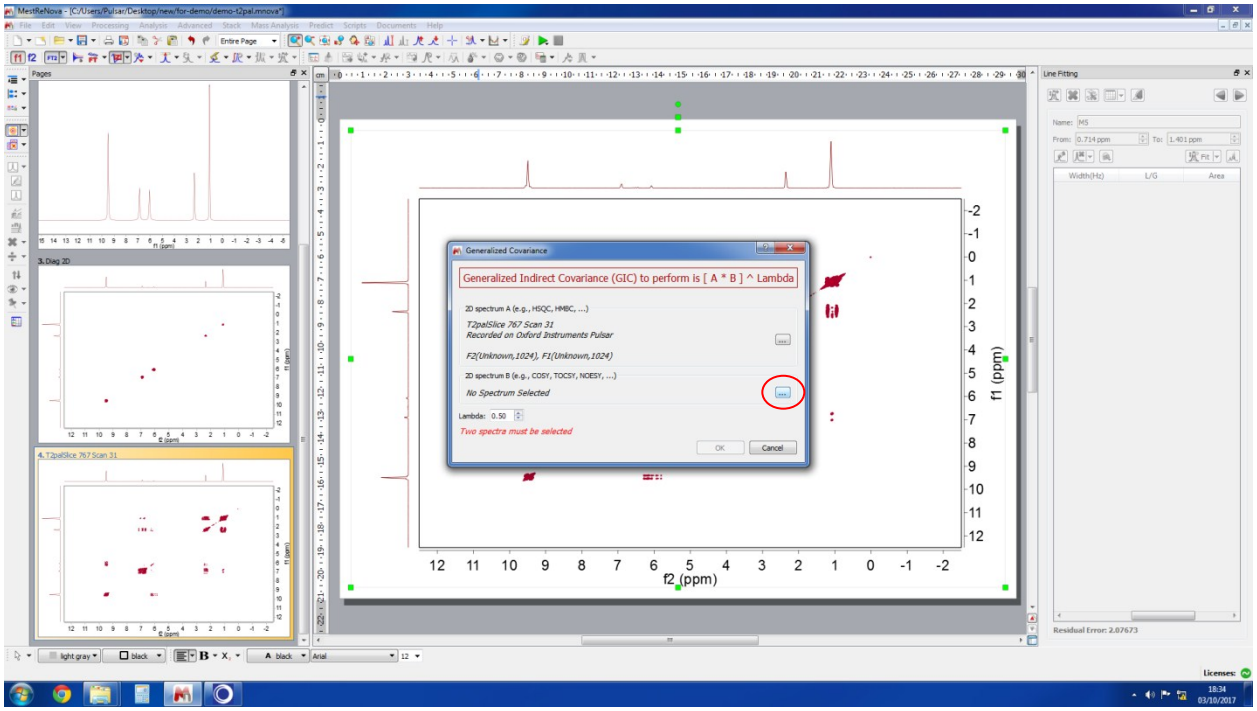
17. To select COSY spectrum



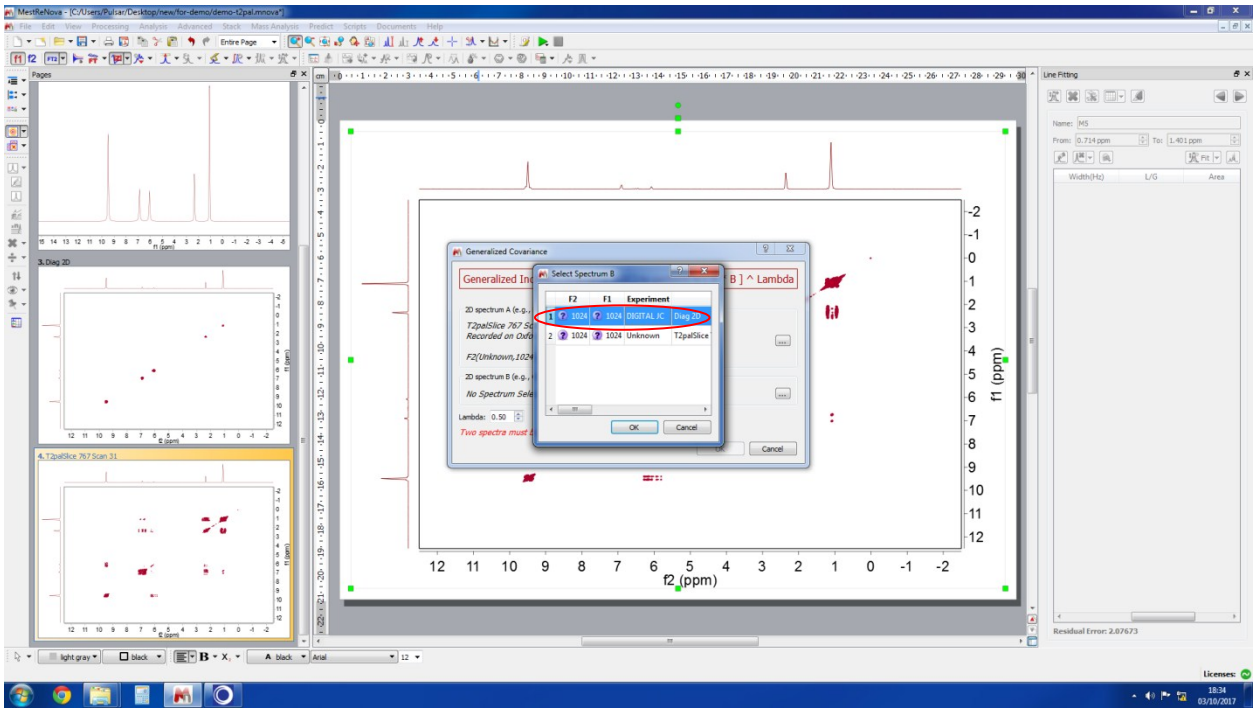
18. Select COSY spectrum



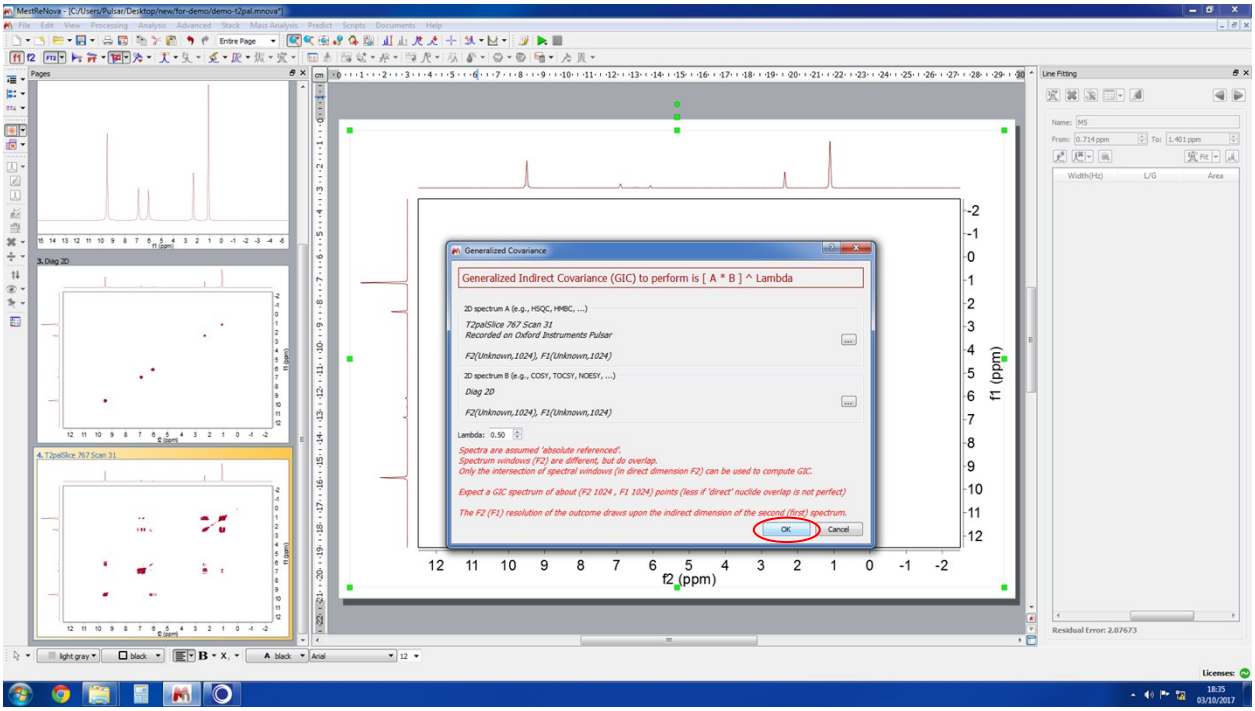
19. To select DIAG spectrum



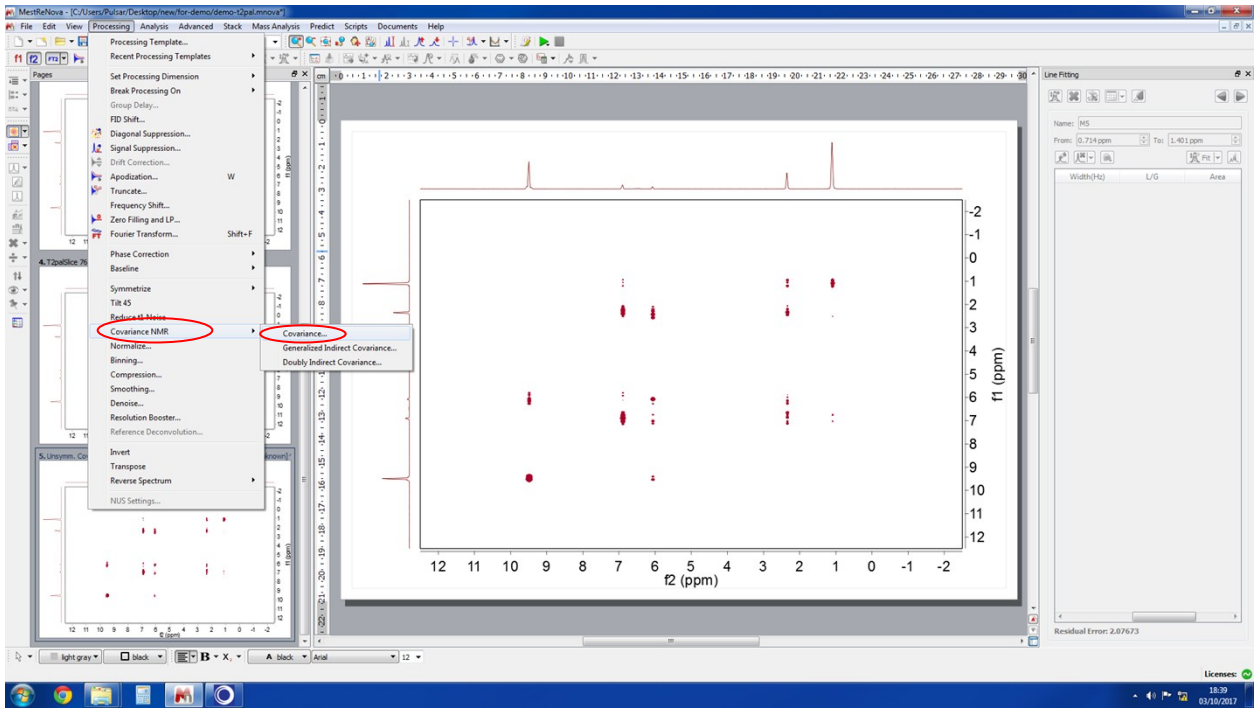
20. Select DIAG spectrum



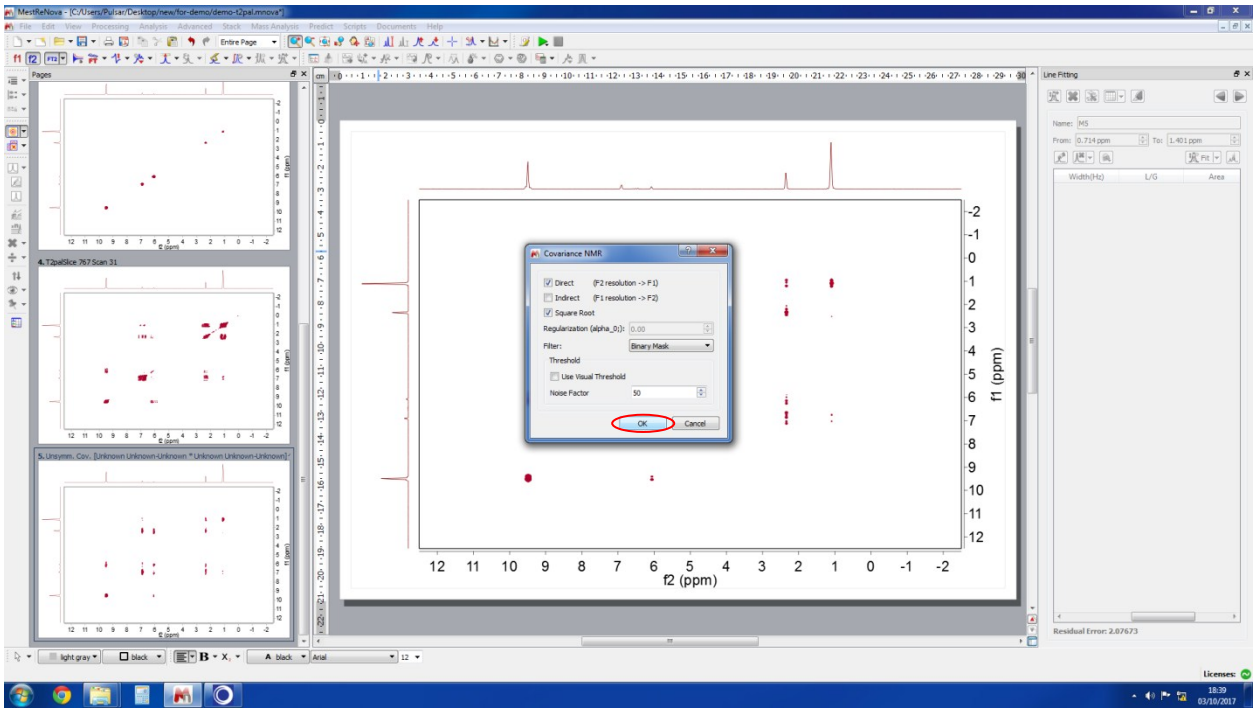
21. To perform GIC processing → OK



22. Now we have decoupled COSY (along the F2 dimension)
    To perform Direct Covariance→ Processing → Covariance NMR → Covariance



23. To Execute Direct Covariance Processing → OK



24. Finally we have F1 and F2 decoupled GIC processed pure shift COSY NMR spectrum.