

Delta-CRAFT

Introduction and documentation Draft

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What is Delta-CRAFT

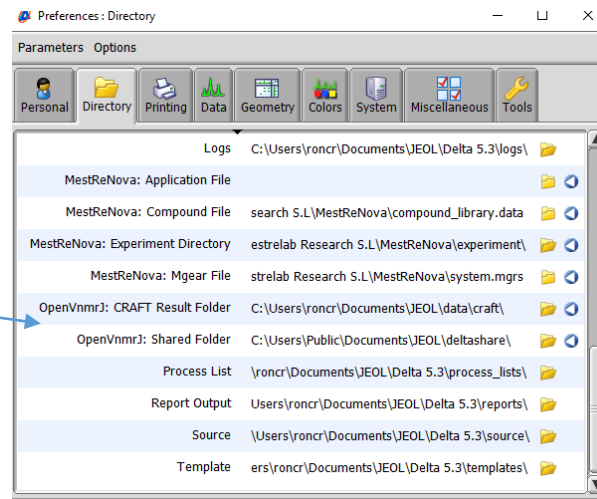
- Delta-CRAFT is a software package which integrates time-domain analysis of NMR data conducted in a LINUX virtual machine with JEOL Delta software.
- Delta provides a user interface to allow the user to use the frequency-domain NMR data as a convenient guide to define the submission for the CRAFT time-domain analysis.
- Upon completion CRAFT returns the result as a simple text file table which completely defines the requested portions of the data along with a synthesized frequency-domain result for easy visualization of the quality of the result.
- It is important to keep in mind that the answer is in the table and the visualization is for simple fast evaluation. As experience is gained with Delta-CRAFT the fact that the table provides a direct “FID to spreadsheet” will become very powerful and as confidence is gained the visualization will decrease in interest.
- Reference for CRAFT: CRAFT (complete reduction to amplitude frequency table) – robust and time-efficient Bayesian approach for quantitative mixture analysis by NMR. Krish Krishnamurthy, Magn. Reson. Chem., 2015 51 821-9, <https://doi.org/10.1002/mrc.4022>

Installing Craft for Delta

- See the Install CRAFT for Delta.pdf file from complete instructions to download and install Craft for Delta.

Directory Preferences.

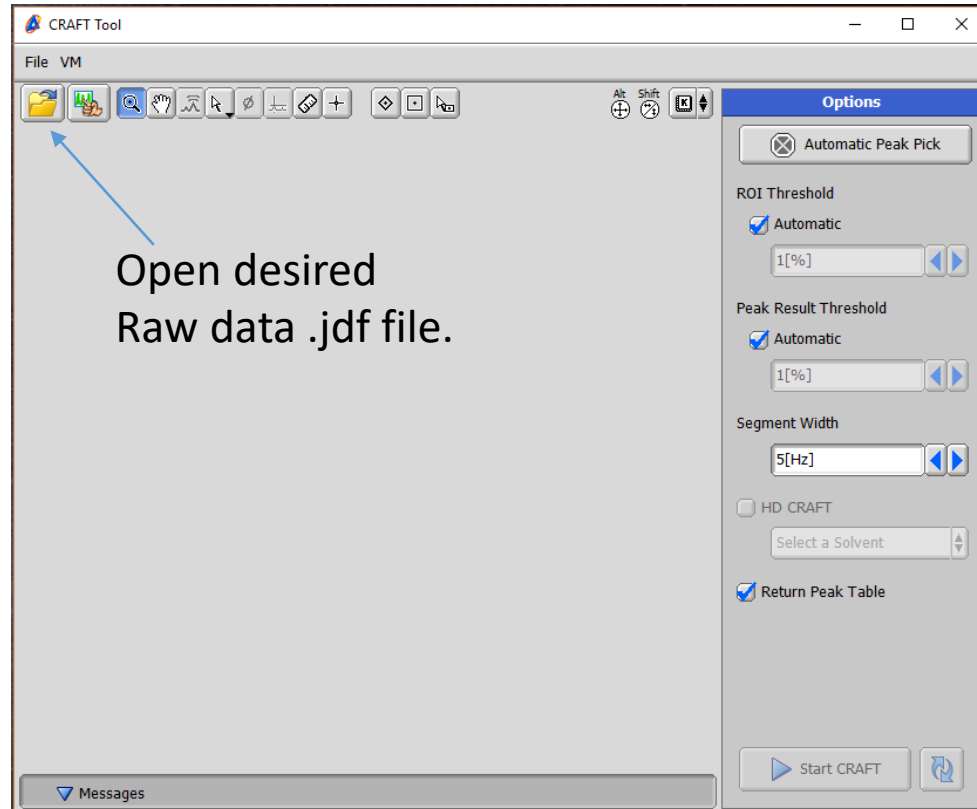
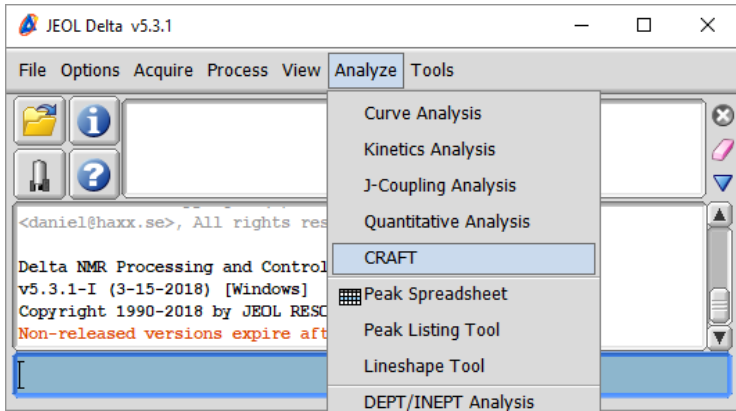
- “OpenVnmrj: CRAFT Result folder” => Set to the path desired for all returning CRAFT results to be saved if it is desired to do so. A choice could be your local “Documents\JEOL\Data\craft”. Delta will save the results to the data directory if left blank. It is required to first create the folder in Windows before selection in Delta Preferences.
- “OpenVnmrJ: Shared Folder”. This should be checked and set for CRAFT to function. Left blank Delta will automatically default to the path shown here. At installation the default windows path is “C:\Users\Public\Documents\JEOL\deltashare”. Placing the shared folder on the windows fast-access tab is convenient.



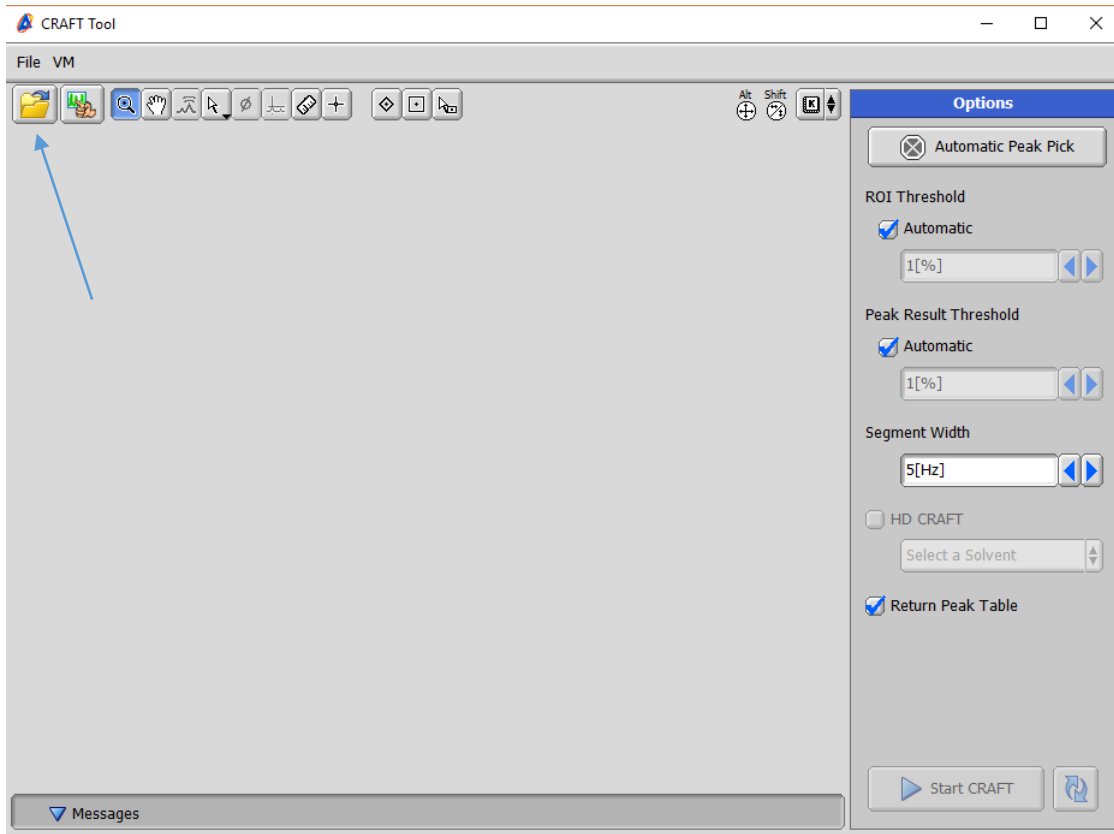
Tools to Control CRAFT Process Described in this document

- How to open file and perform CRAFT on a 1D data set and what the resulting three spectra represent.
- How to select regions for CRAFT, by specifying a threshold, optionally with a "box" and other combined features to identify peaks of interest.
- How to include "solvent" information for the CRAFT process.
- How to re-process a CRAFT result to generate a different line list.
- How to CRAFT a 2D data set, including the option of providing a 1D data set from which to extract the F2 ROIs. Also the availability of a 1024 point extended y axis projection is described.
- How to monitor progress of the CRAFT process.
- How to abort a CRAFT process.
- A little Virtual Box information for the user.

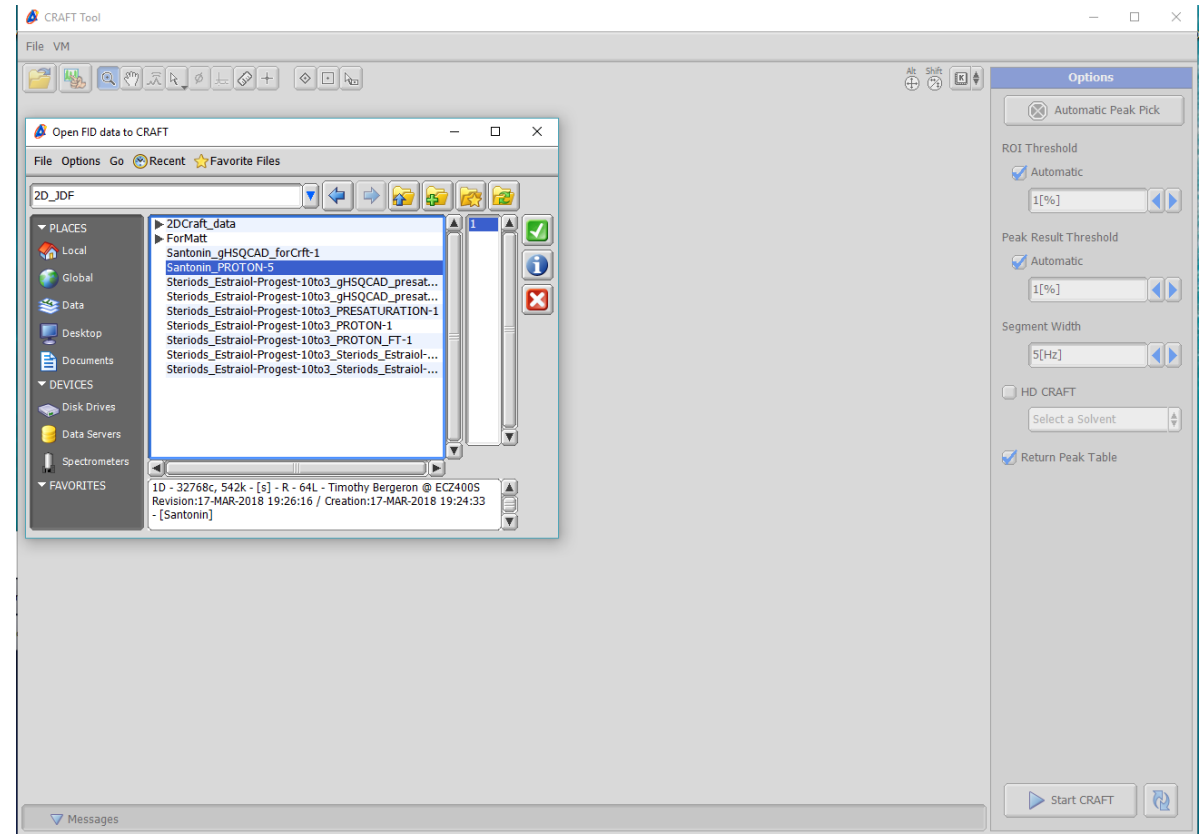
Launching Craft Tool



1. Open File for Crafting

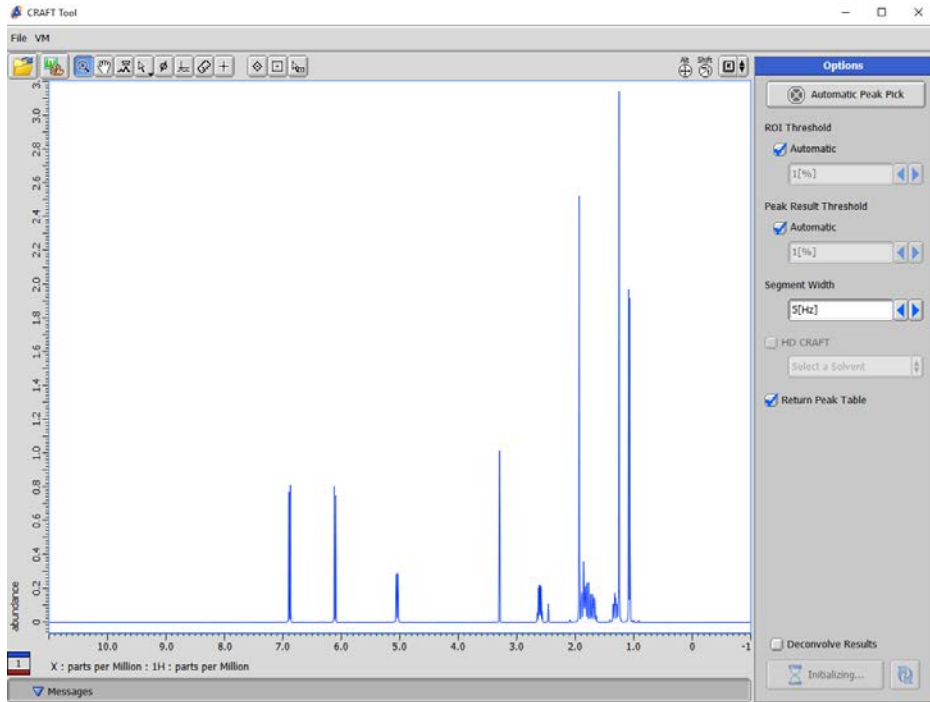


Choose File open utility in CraftTool

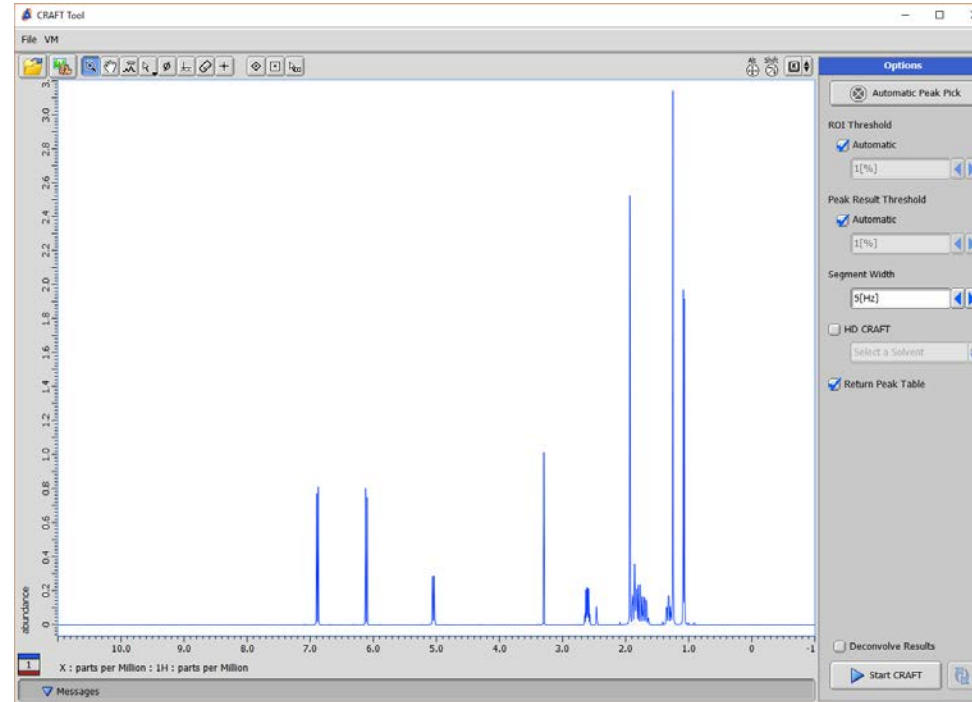


Select desired jdf file for analysis

1. Basic Submission to CRAFT

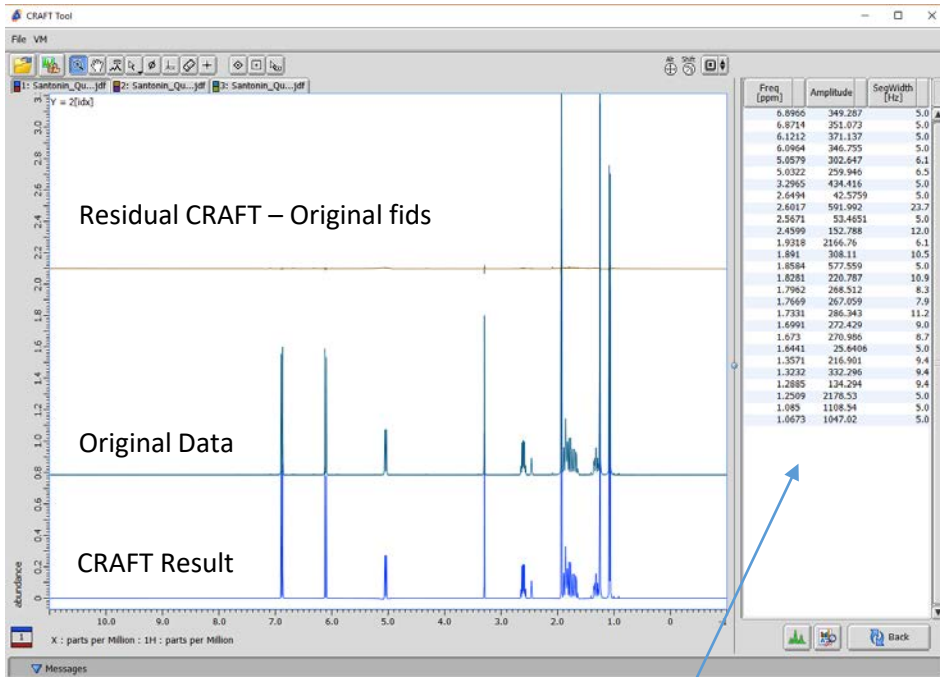


When file is opened Virtual machine starts up. Submission button shows “Initializing”.

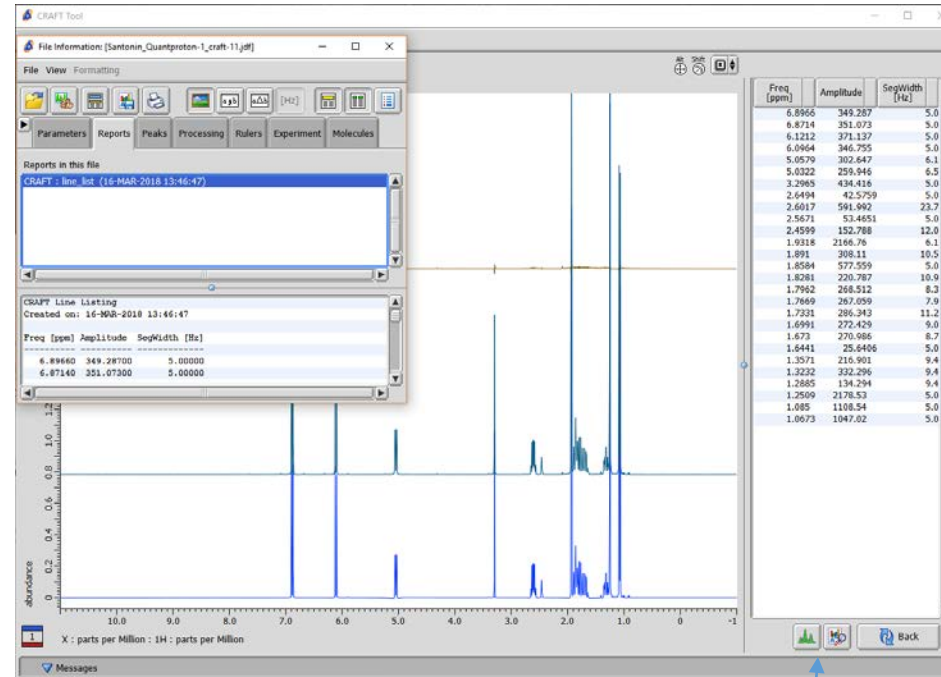


When Start CRAFT appears simply push button!

1. Basic automatic CRAFT Result

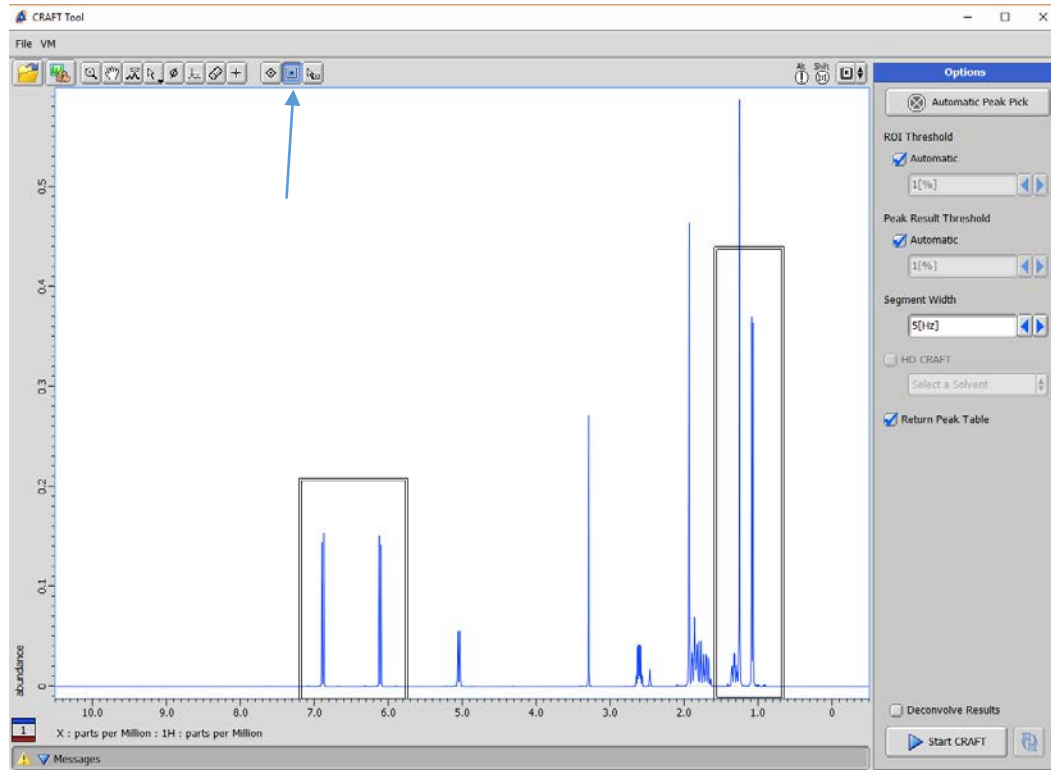


Spectrum->Spreadsheet Results

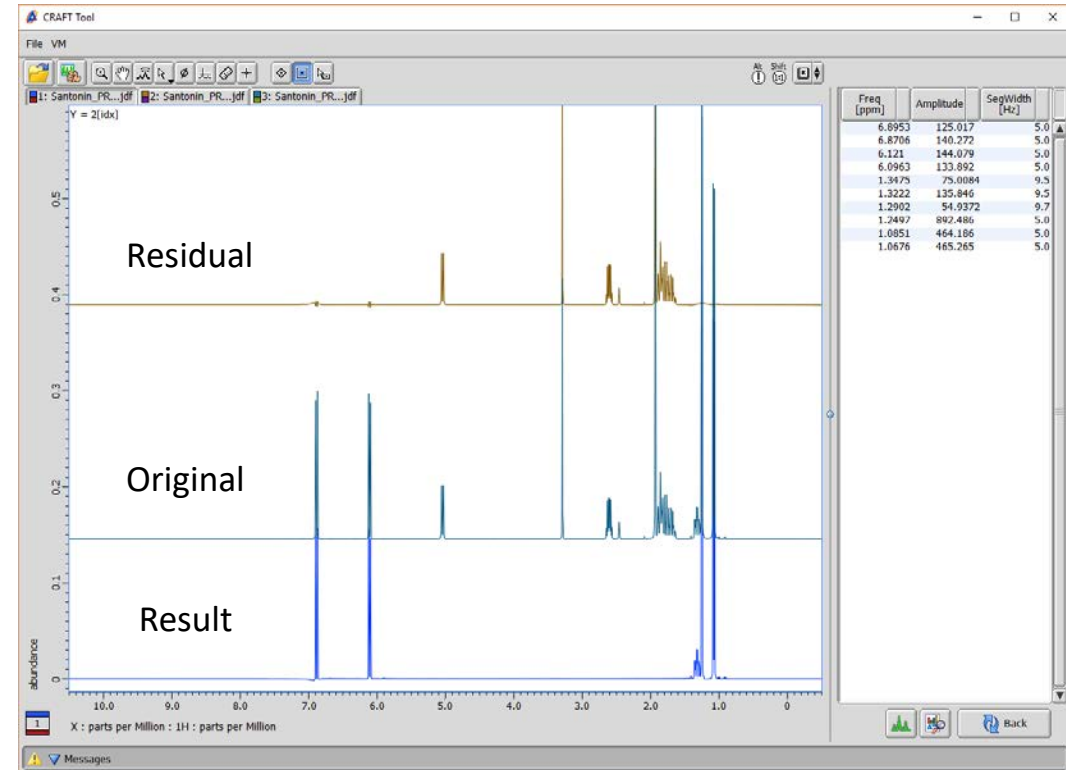


Saved CRAFT line list result can be viewed in saved CRAFT result jdf file, or data exported to data slate. It is important to understand that the “Amplitudes” reflect the total area of each reported frequency not peak height.

2. Peak Selections Basics – Region of Interest Box

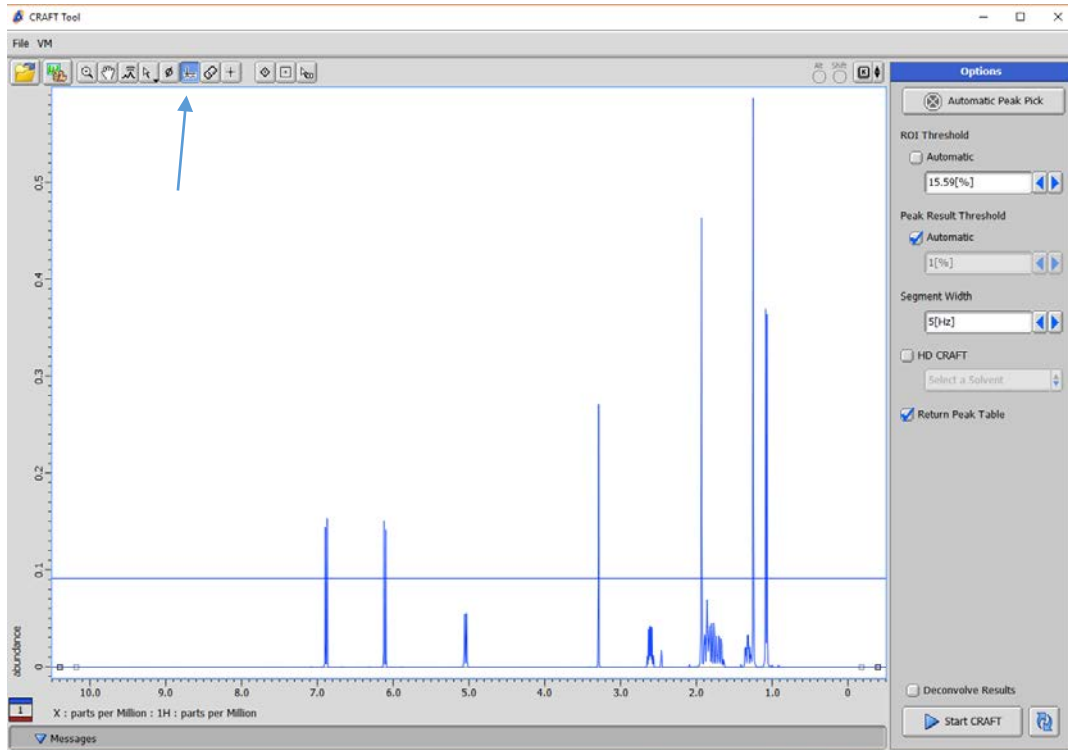


Use Box tool to draw desired regions.
Vertical height has no meaning

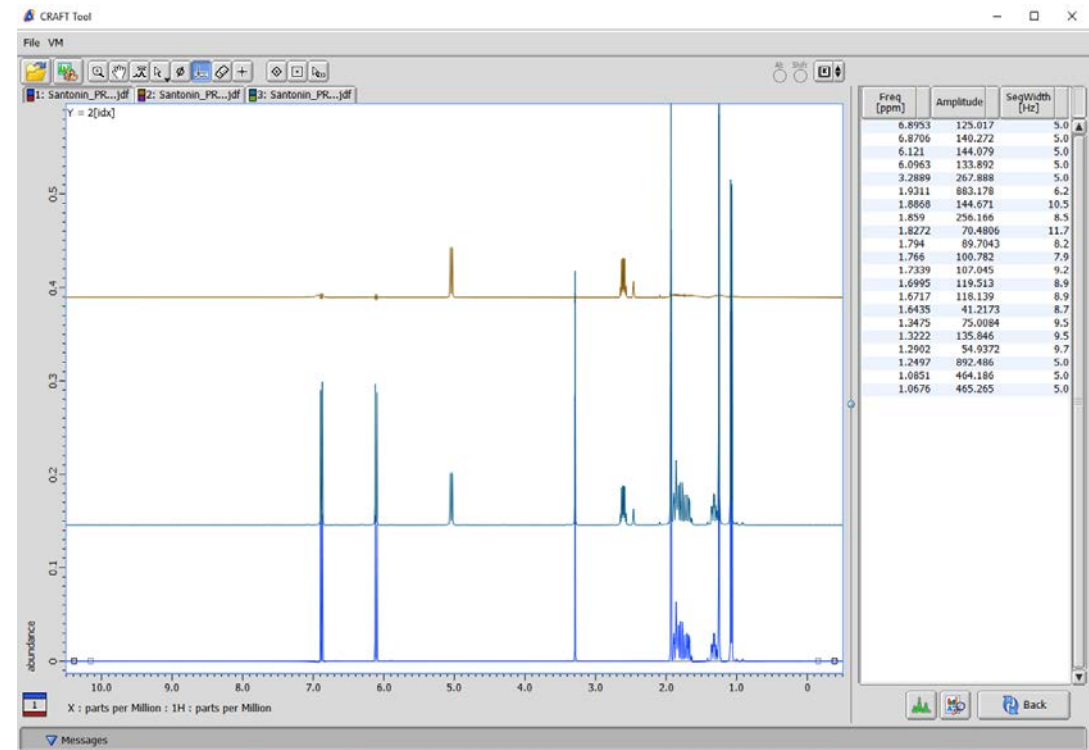


CRAFT result from box selection example.
Note the residual has the un-crafted data.

2. Peak Selection Basics – Threshold.

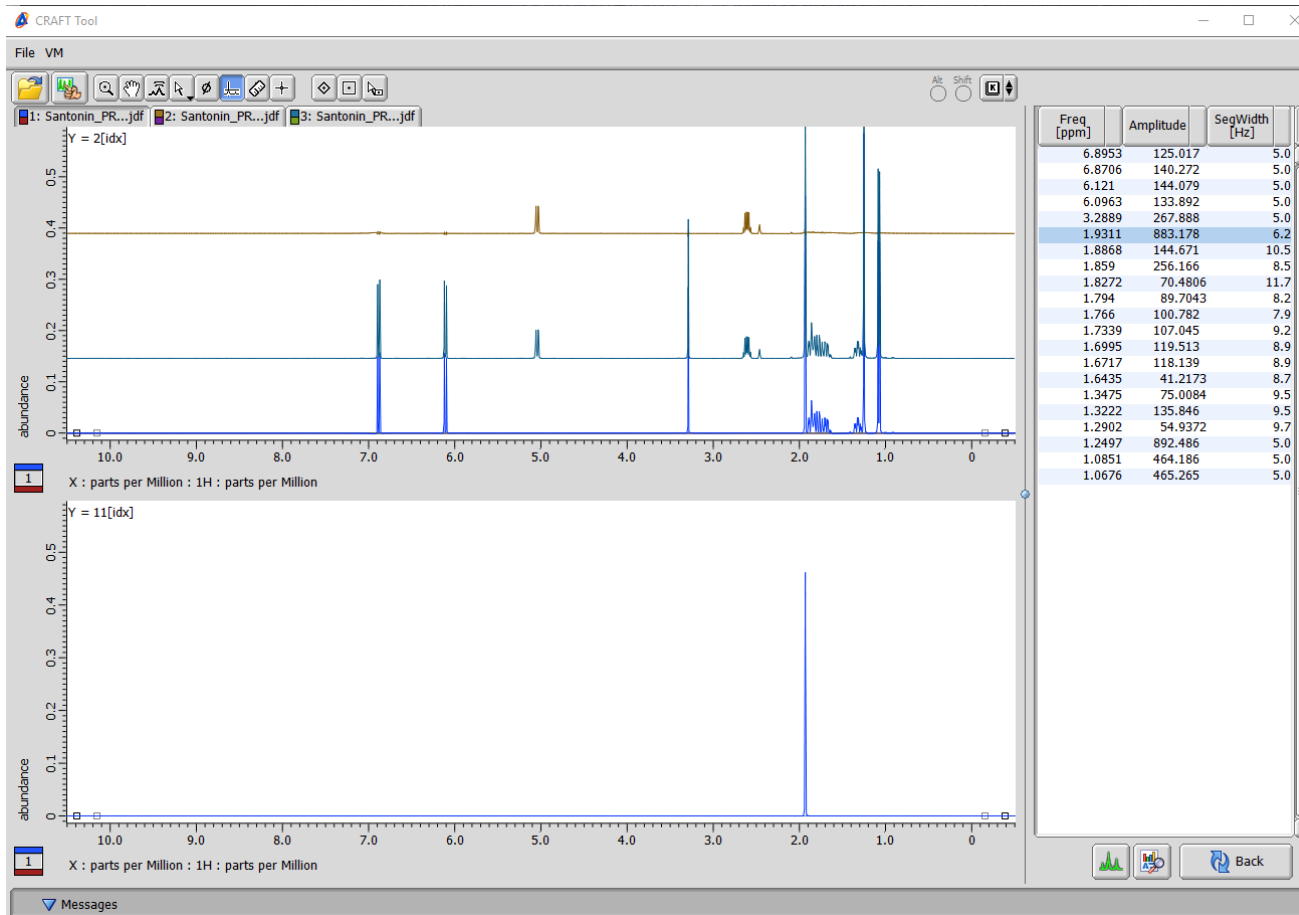


Either use threshold cursor or type in a value
For ROI threshold.



Note that the CRAFT result includes the complex multiplets near 1.8ppm because internally the singlet at 1.9311ppm is included in a region with those.

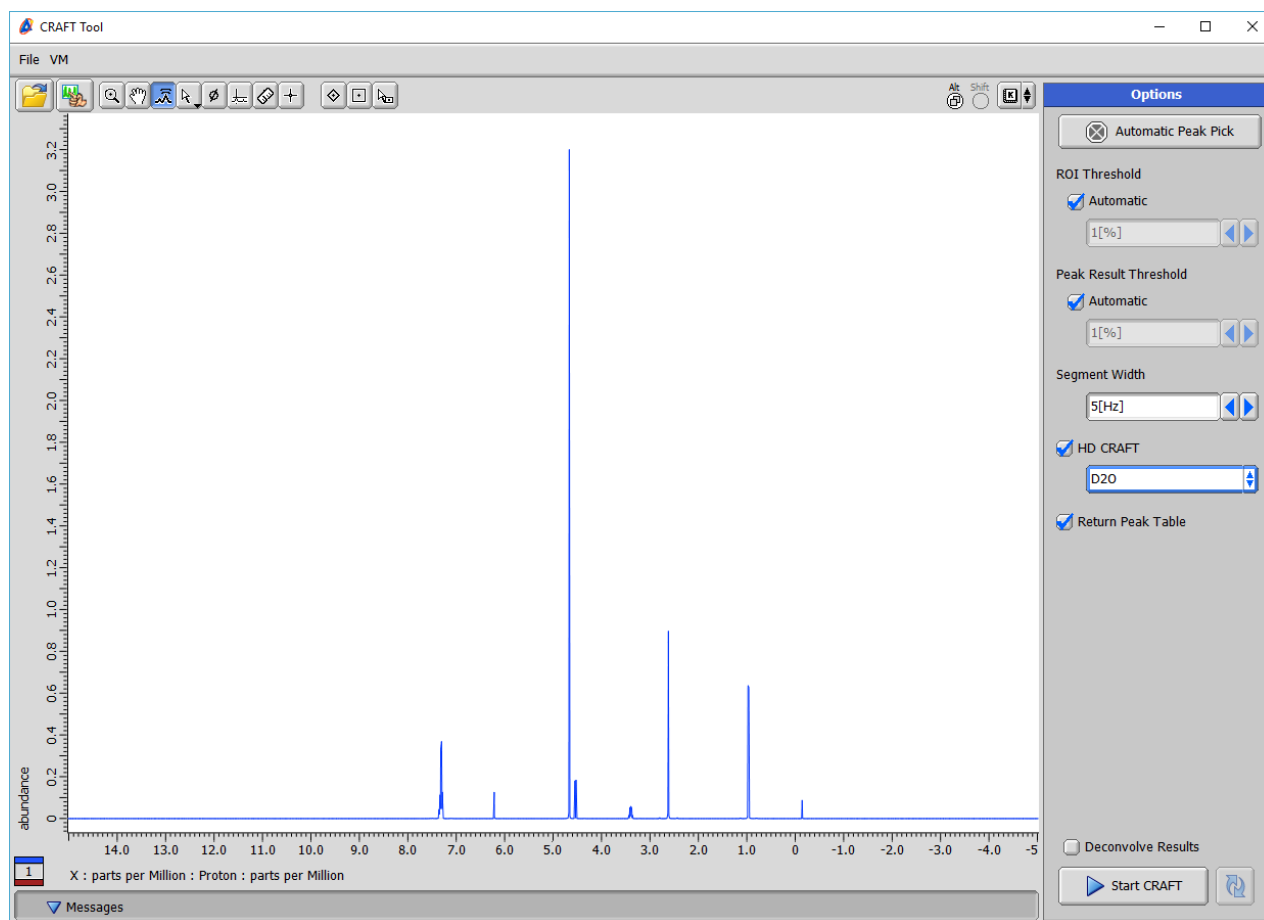
Showing CRAFT Model from Linelist



Note that by selecting a value in the line list table with the a mouse click (darker blue line) it's associated CRAFT model is displayed. The user can choose as many peaks as desired and normal shift-click, control-click options are respected.

To remove the display of selected models control-click the selected lines in line list report to unselect.

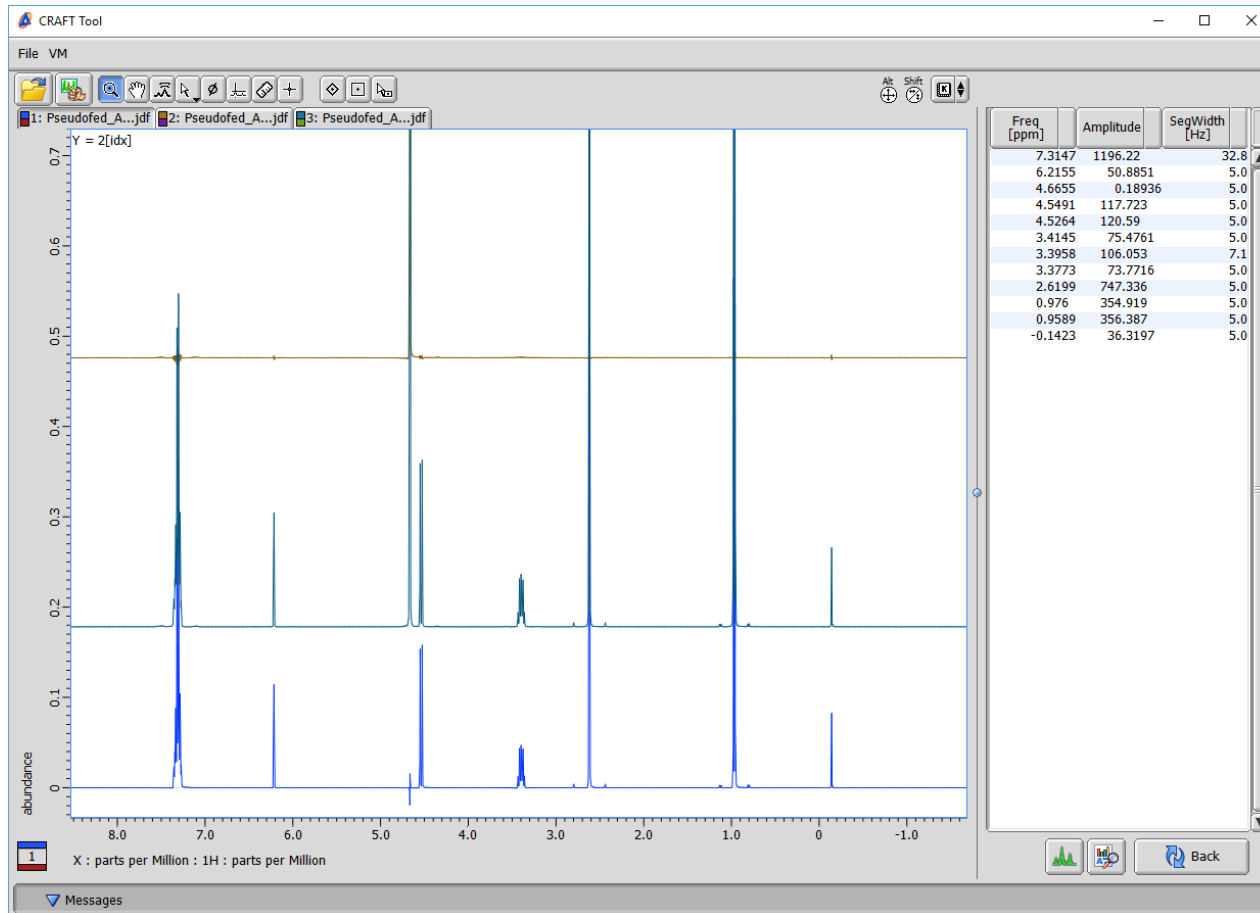
HD - CRAFT



The HD CRAFT option allows a selection of peaks to be picked from menu for suppression in the CRAFT result. In most instances the menu items would be common solvents but they could also point to a file with a peak list of a compound desired to be filtered out of the result.

In this example the solvent D₂O has been selected.

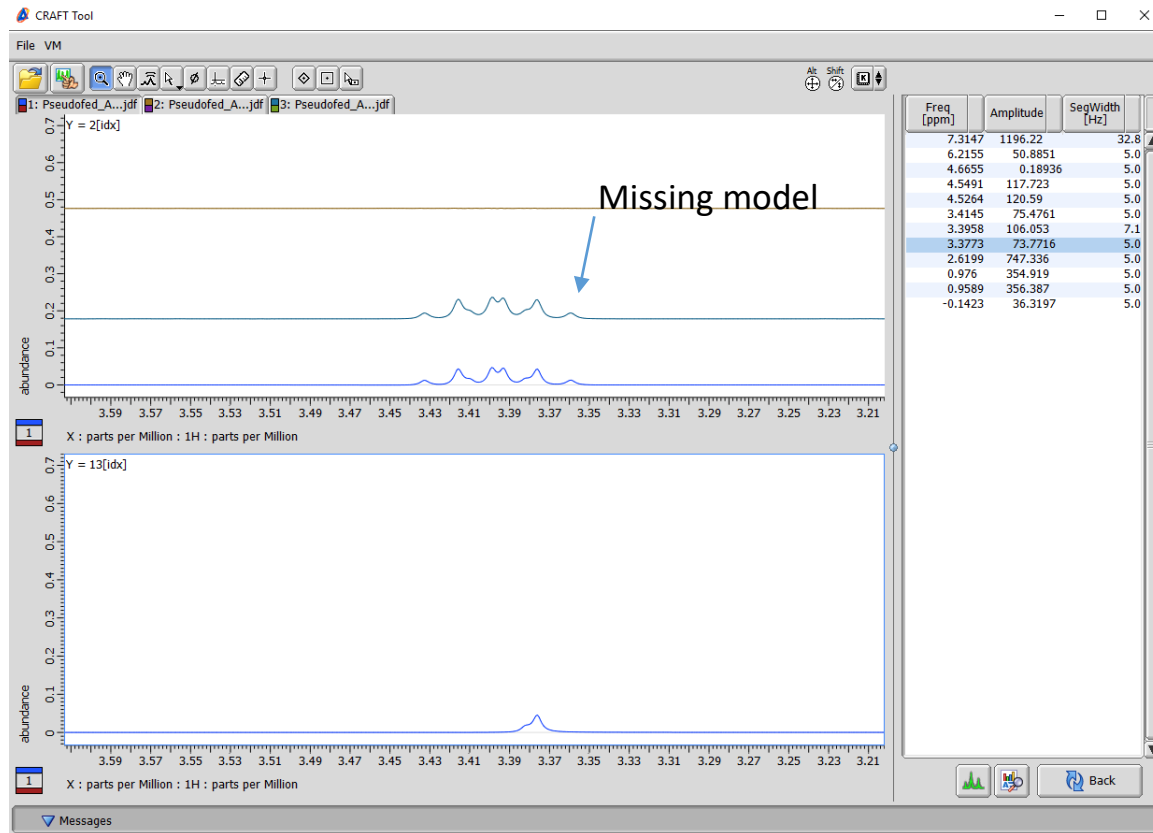
HD CRAFT Result



Note that in the HD CRAFT result in this example the selected solvent signal for D2O is not in the result and totally in the CRAFT residual.

We will now look more deeply at this result and refine the line list to show all desired frequencies by expanding the multiplet near 3.4ppm.

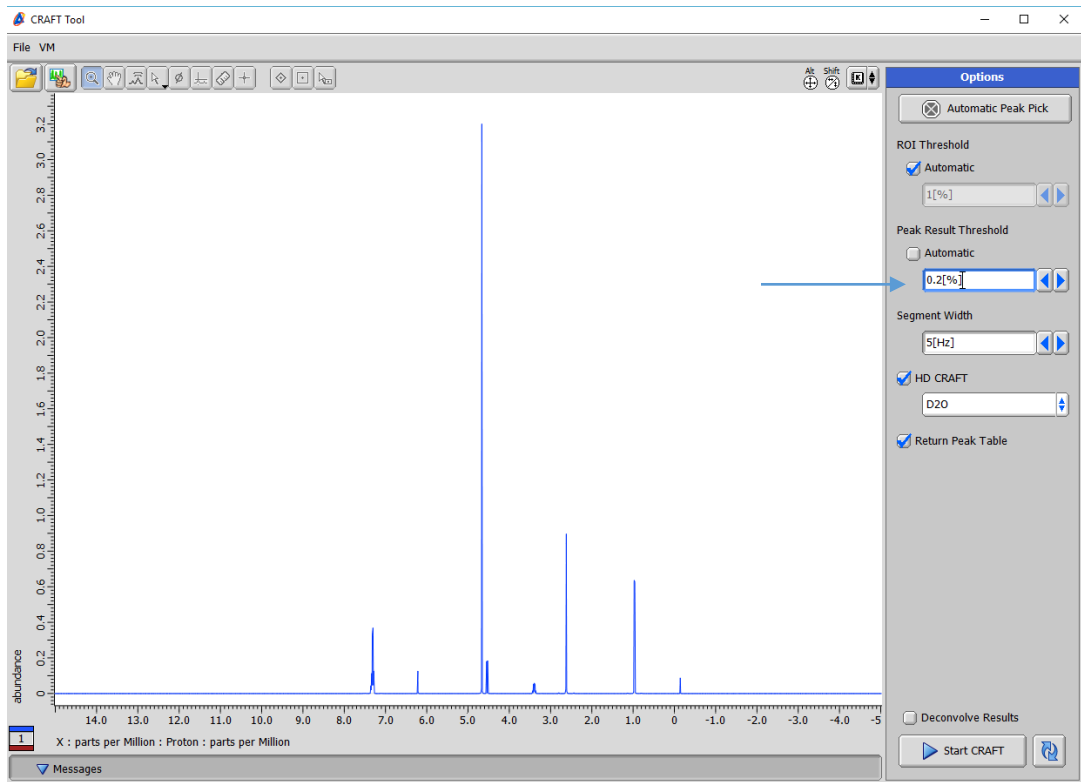
Show Missing Line List result.



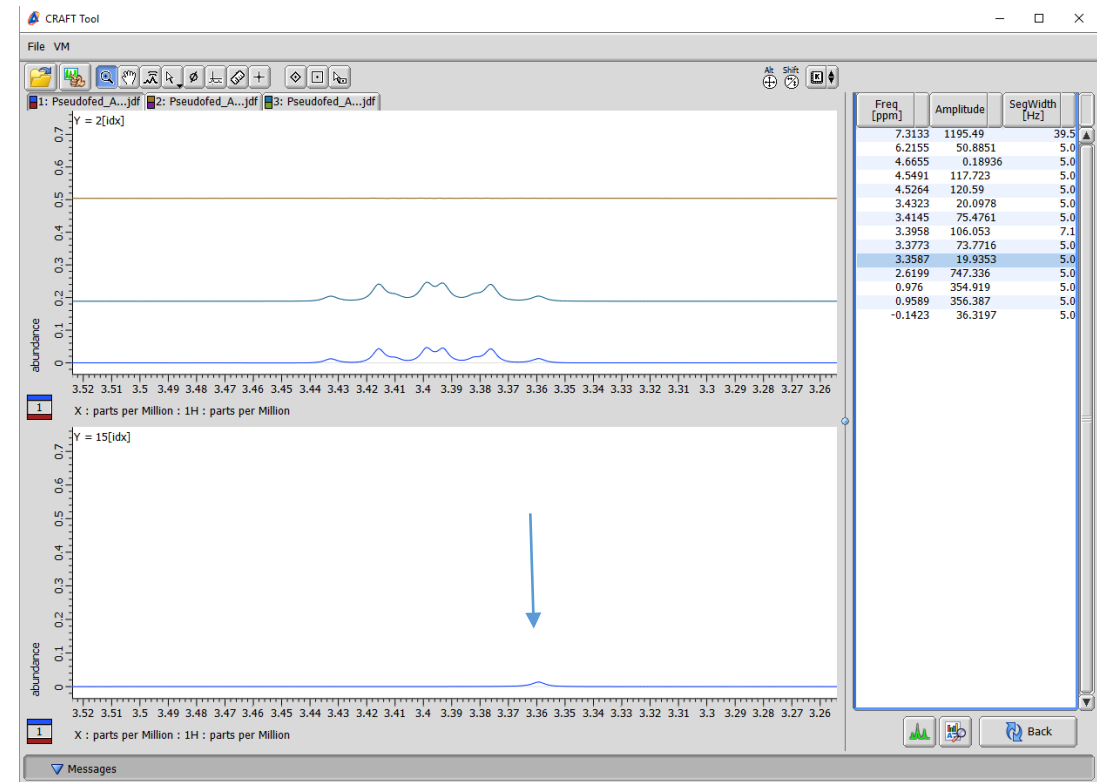
Here it can be seen by model selection that the smallest peak of the multiplet is in the CRAFT result but is absent from the line list report. The model selection is the highest field signal present in the line list and the display shows on the bottom pane that it is not the actual smallest signal in the multiplet.

In this example where a resonance is in the CRAFT result but missing from the line list report, simply lower the threshold for the line list and resubmit. A new CRAFTING is not required. Just a new line list is generated.

Lower Line List Threshold for missing peak.

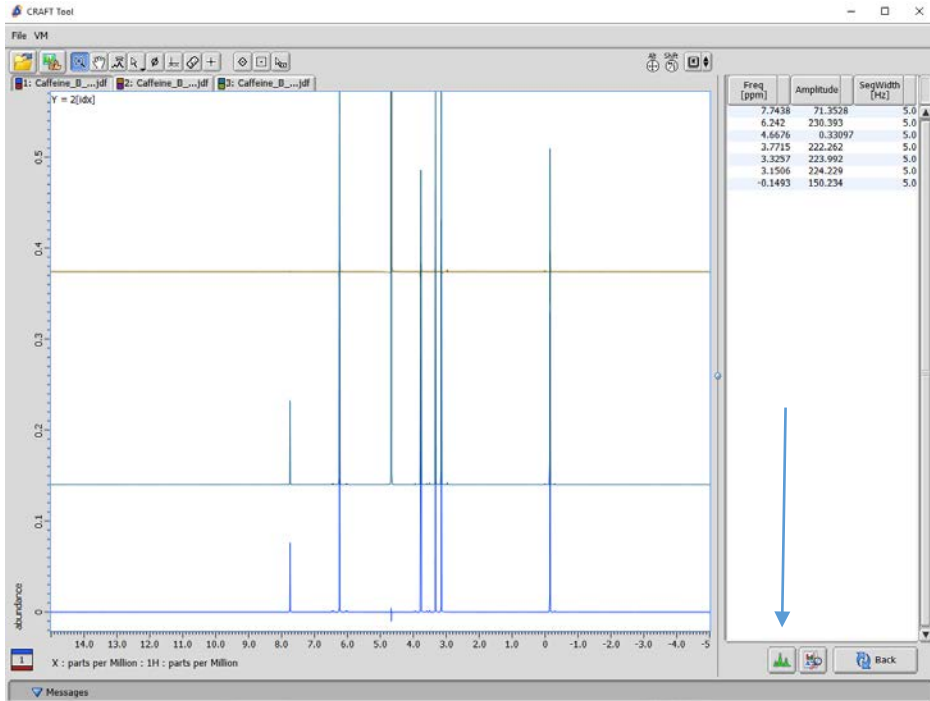


The new lower threshold for line list can be found by horizontal cursor or simply typed into Peak Result Threshold Box. Resubmission is very fast as CRAFTING is not repeated. Details for using a horizontal cursor to define threshold follow on next figures.

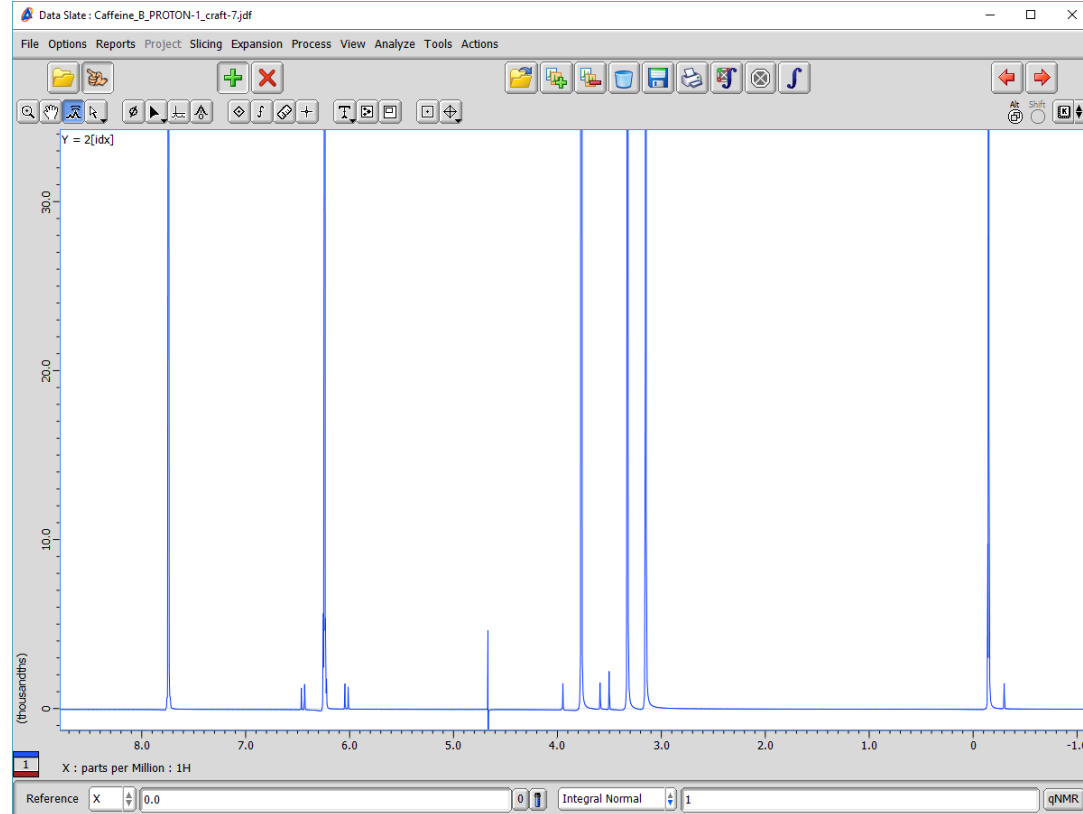


Now selection of the CRAFT model reveals the missing peak has been captured.

Adjusting CRAFT ROI Threshold

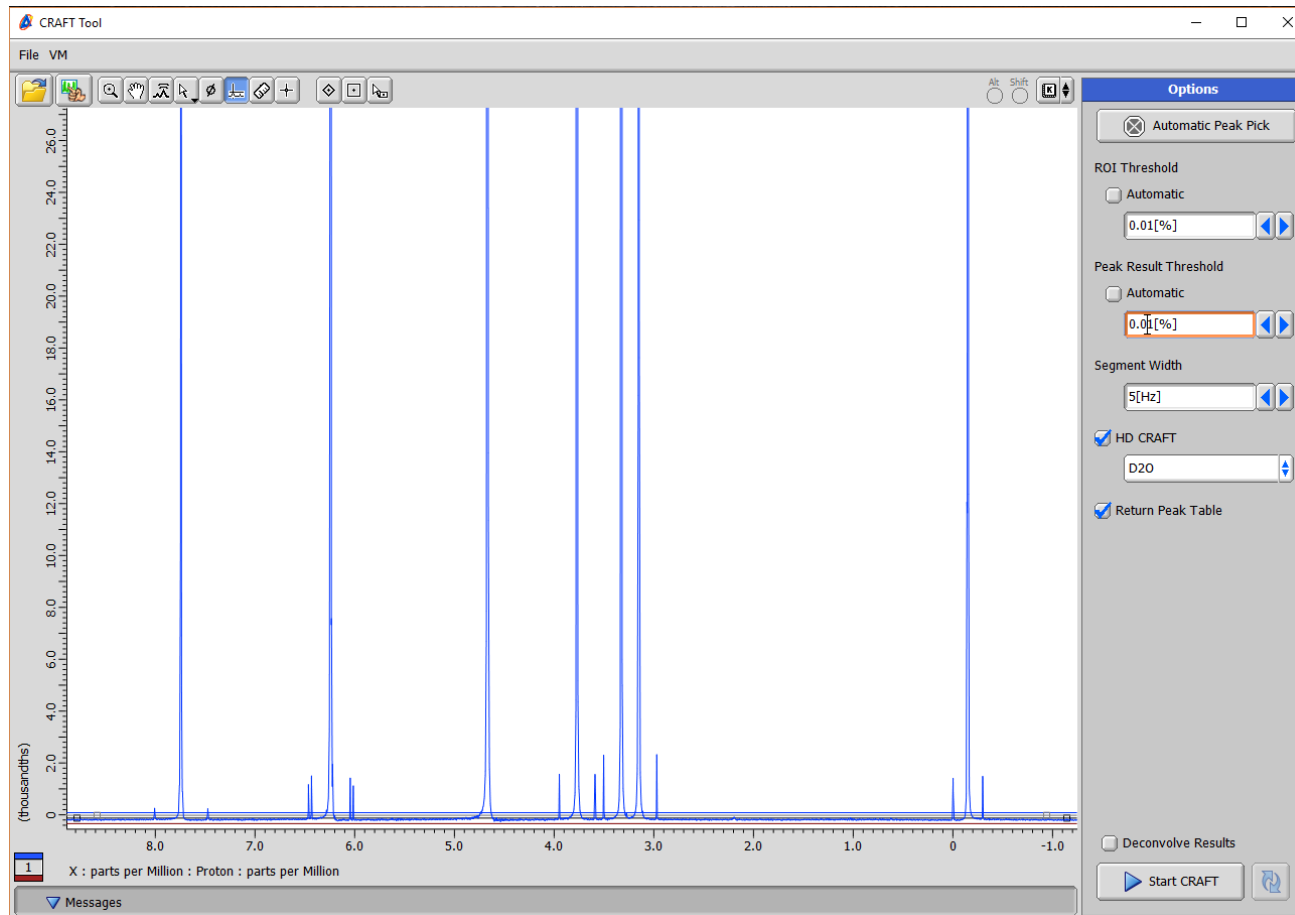


Automatic CRAFT result for Caffeine.
Exported to data slate for closer inspection.



Note how automatic HD CRAFT result missed a few ^{13}C satellites.

Lowering ROI threshold interactively

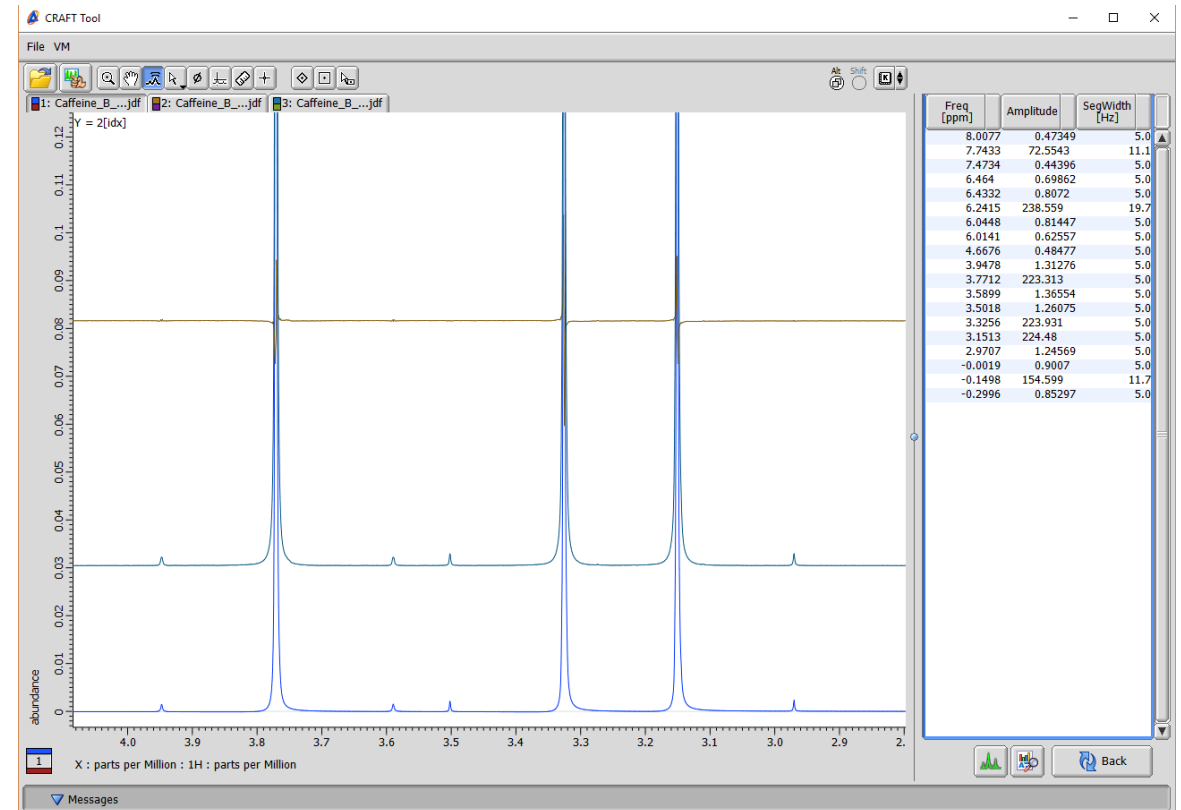
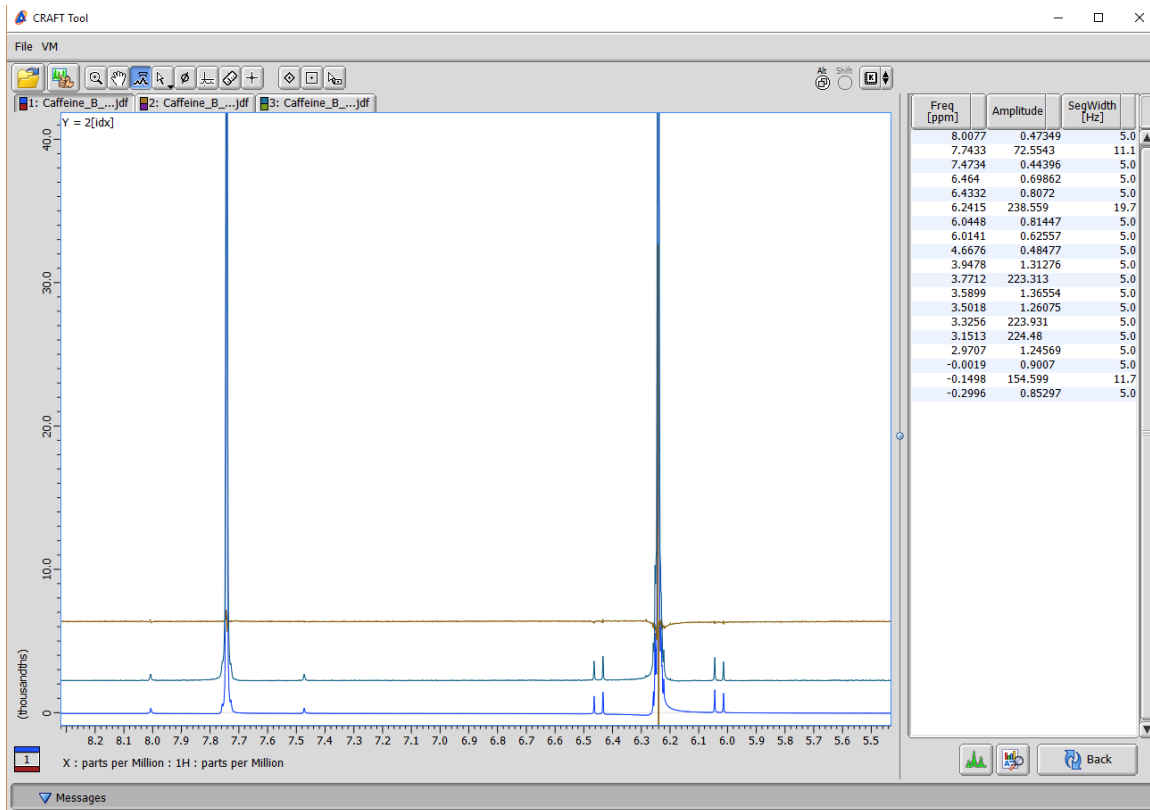


In CRAFT Tool vertical scale increased to see desired small signals. The horizontal threshold is dropped to capture all signals.

Because the ROI threshold has been changed, on submission a complete new CRAFT analysis will be done.

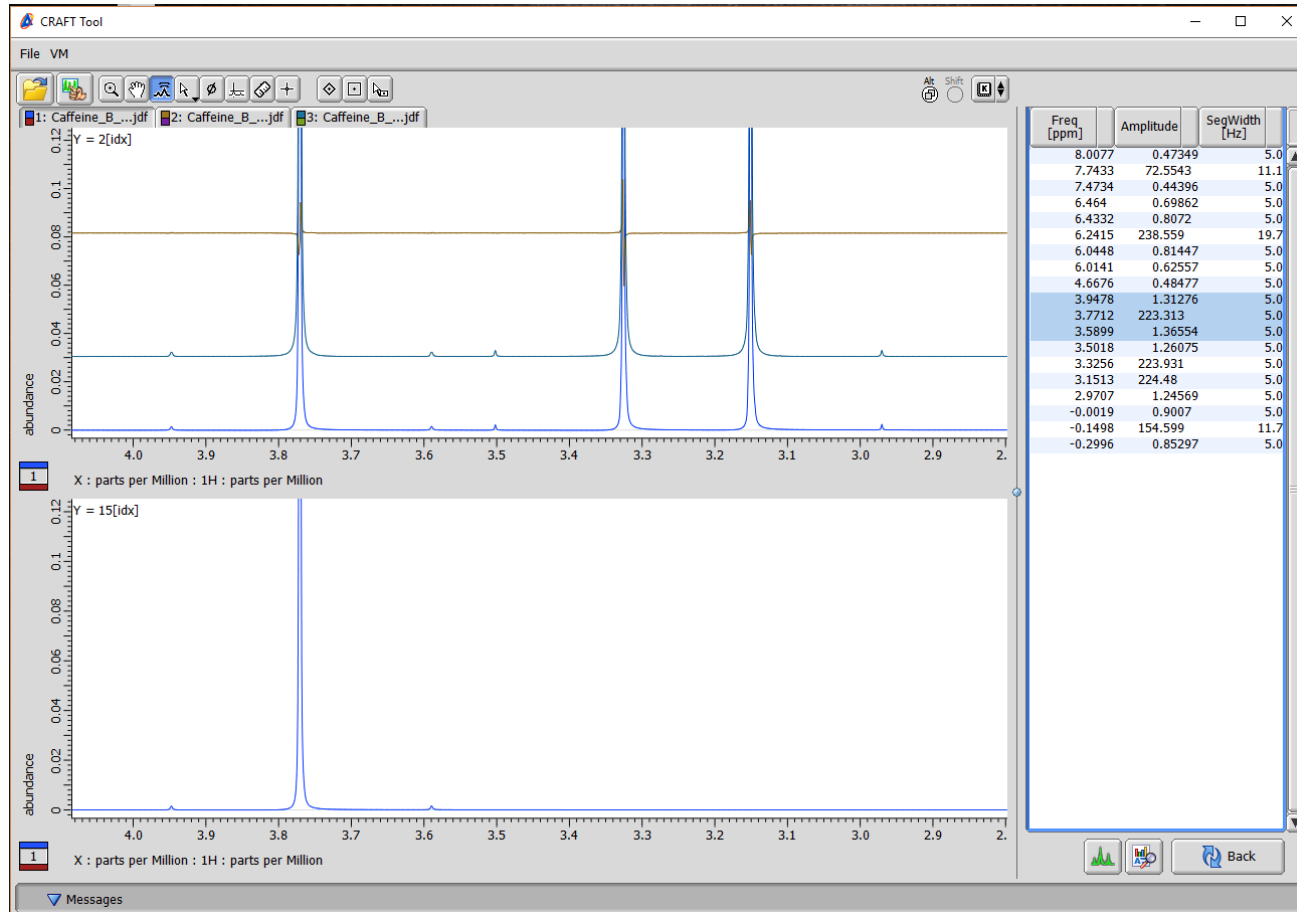
Lower ROI Threshold Results

Note that this time the CRAFT result (blue trace) has indeed captured desired ^{13}C satellite signals.



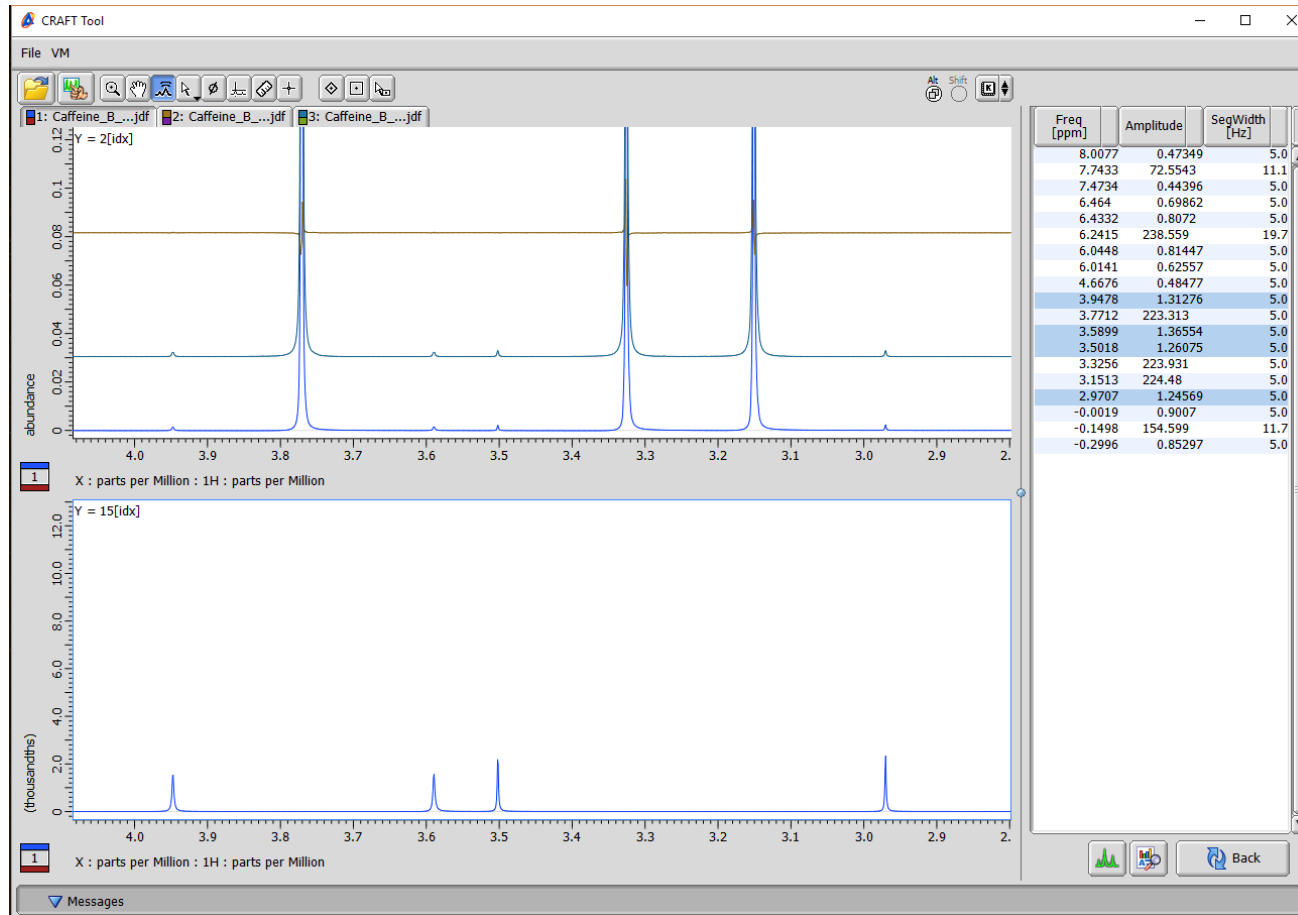
Note also by inspection of the amplitudes in the table how the relative amplitudes of main peaks and ^{13}C satellites agree very closely with the expected 0.55% values.

Selecting models from lower ROI result.



Here we are selecting just the models for the CH₃ at 3.7712ppm along with its associated ¹³C satellites in the line list report to display just those models.

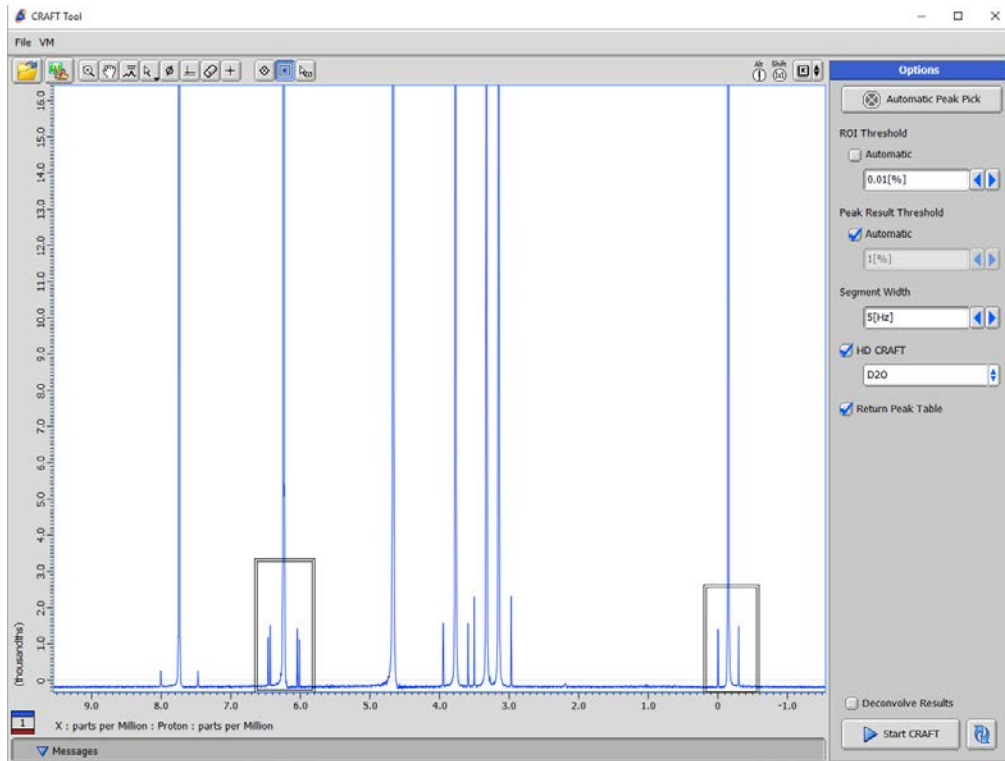
Control-click to choose only desired ^{13}C satellite signals



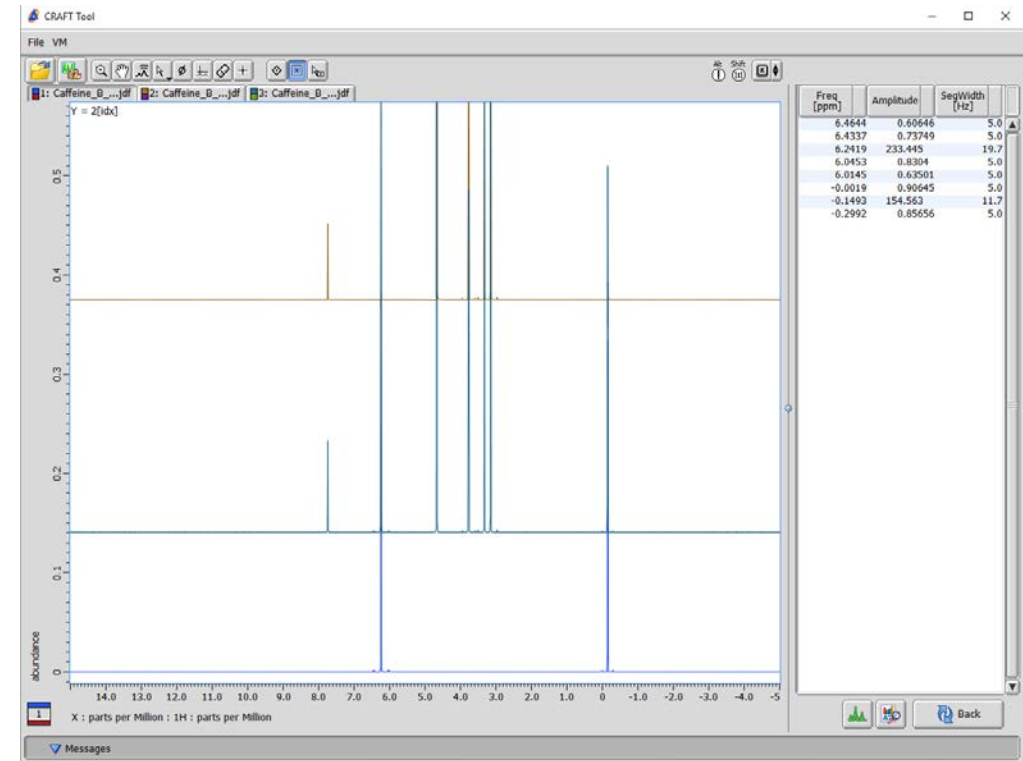
The user can use normal mouse click conventions to selectively choose whatever models that are desired.

In this example only the resolved ^{13}C satellite signals are shown. Note also how the relative amplitudes of the 3 large methyl signals (3.7712, 3.3256, & 3.1513ppm) reveal the presence of hidden 0.55% peaks under the last two.

Combining Selection Tools.

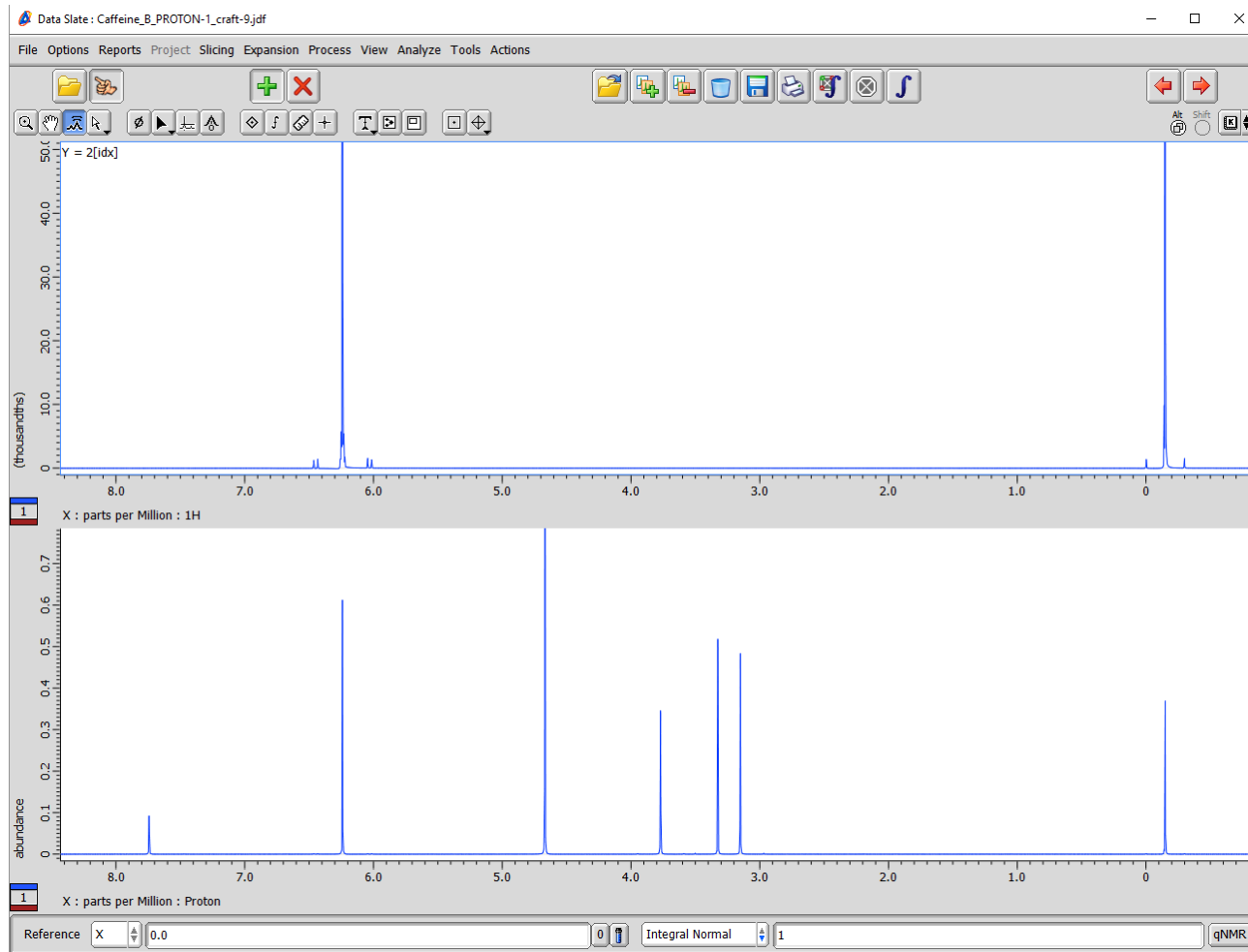


Region boxes and threshold chosen to just analyze the maleic acid and TSP regions.



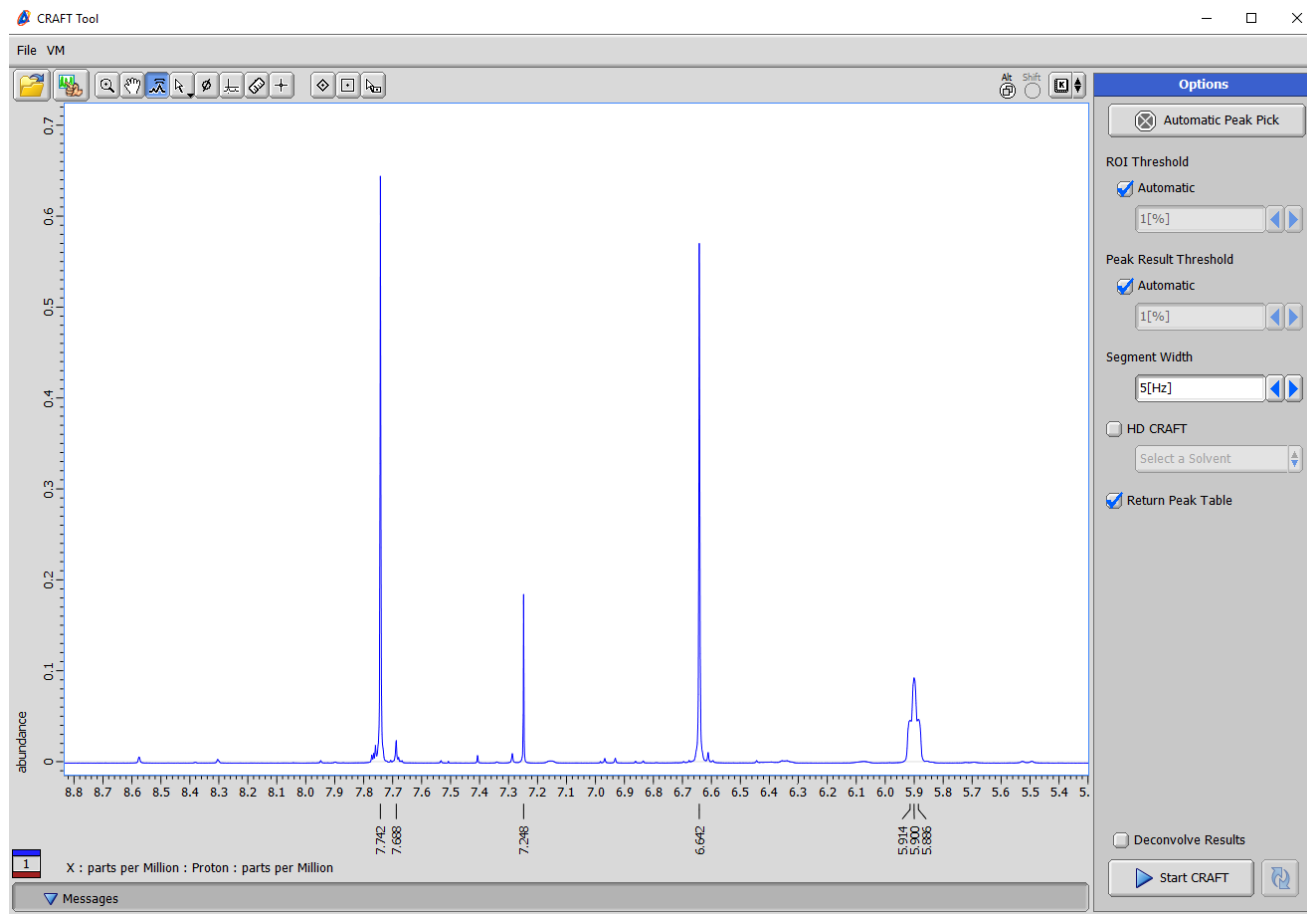
CRAFT result cleanly extracts only the internal calibrants and their ^{13}C satellite signals from the data.

Original Data and CRAFT result in Data slate



The Craft Tool supports all standard Delta display features. In this example the selected CRAFT result is shown with the original data exported to a data slate.

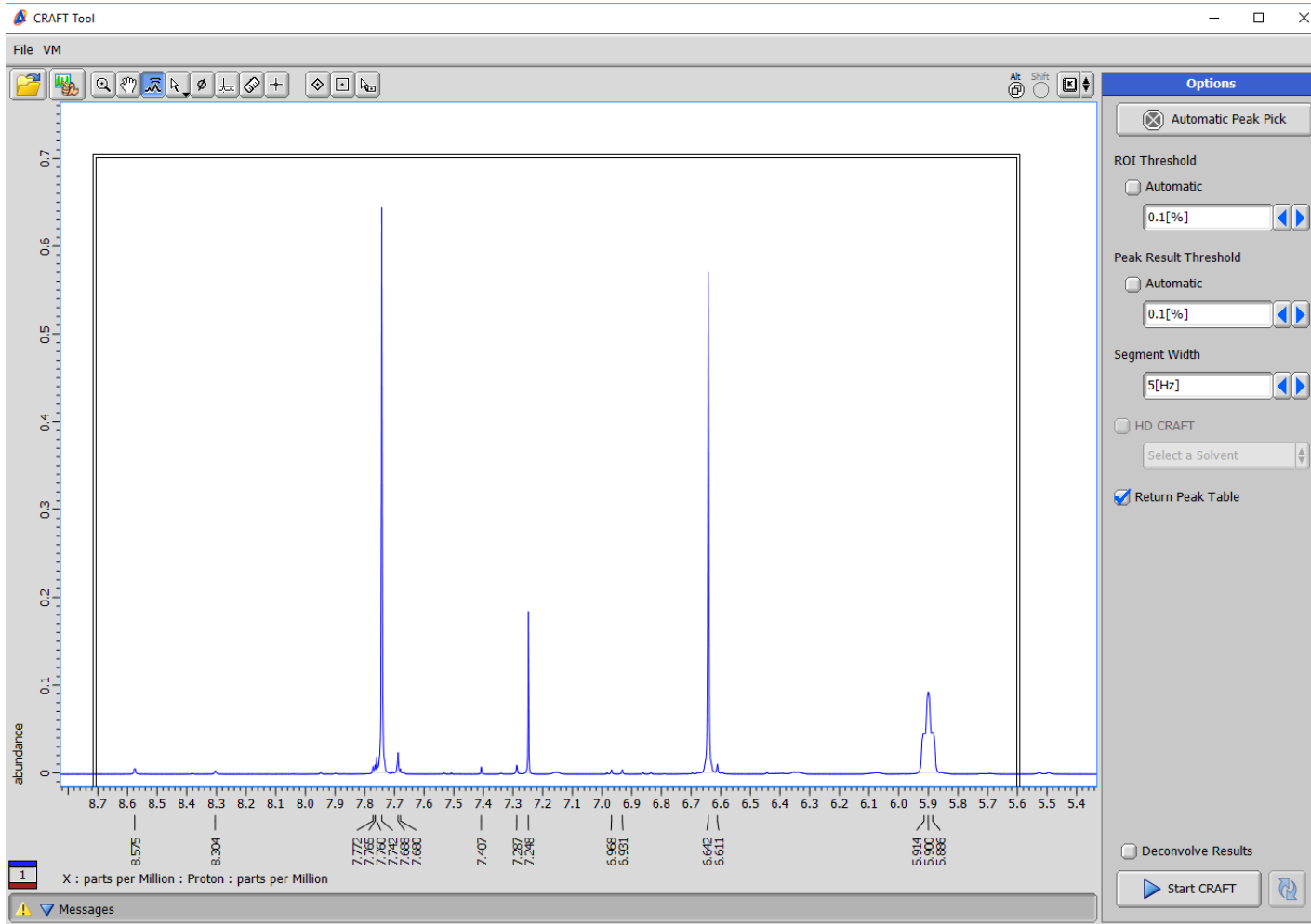
Using Delta Peak Pick to Guide CRAFTing



The Automatic Peak Pick button can be used to guide and preview what CRAFT will search for. The peak listing for selected peaks can be seen under the ppm scale.

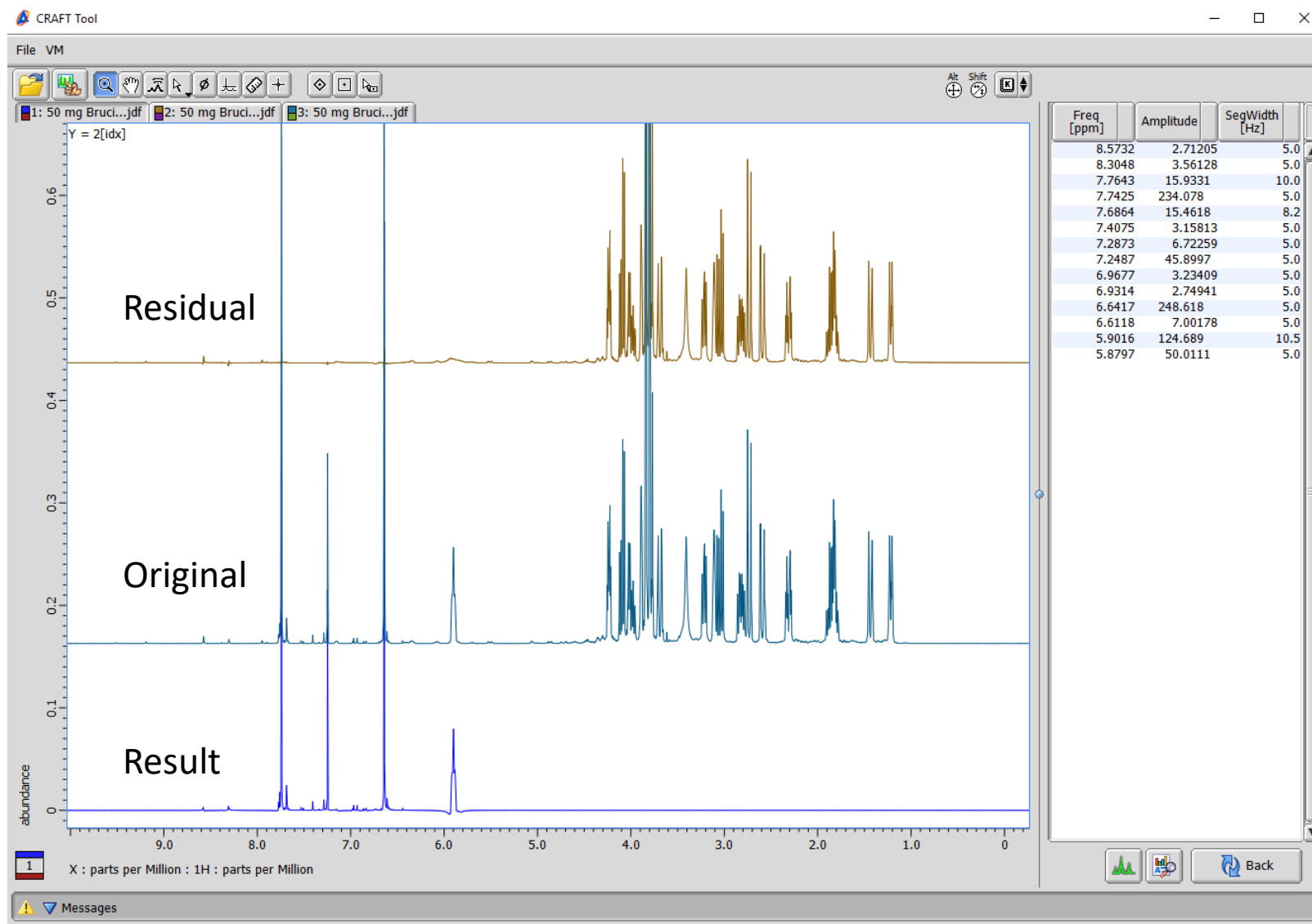
In this example it can be seen that quite a number of small peaks which might be of interest will be missed by the default 1% threshold.

Peak listing after lowering threshold



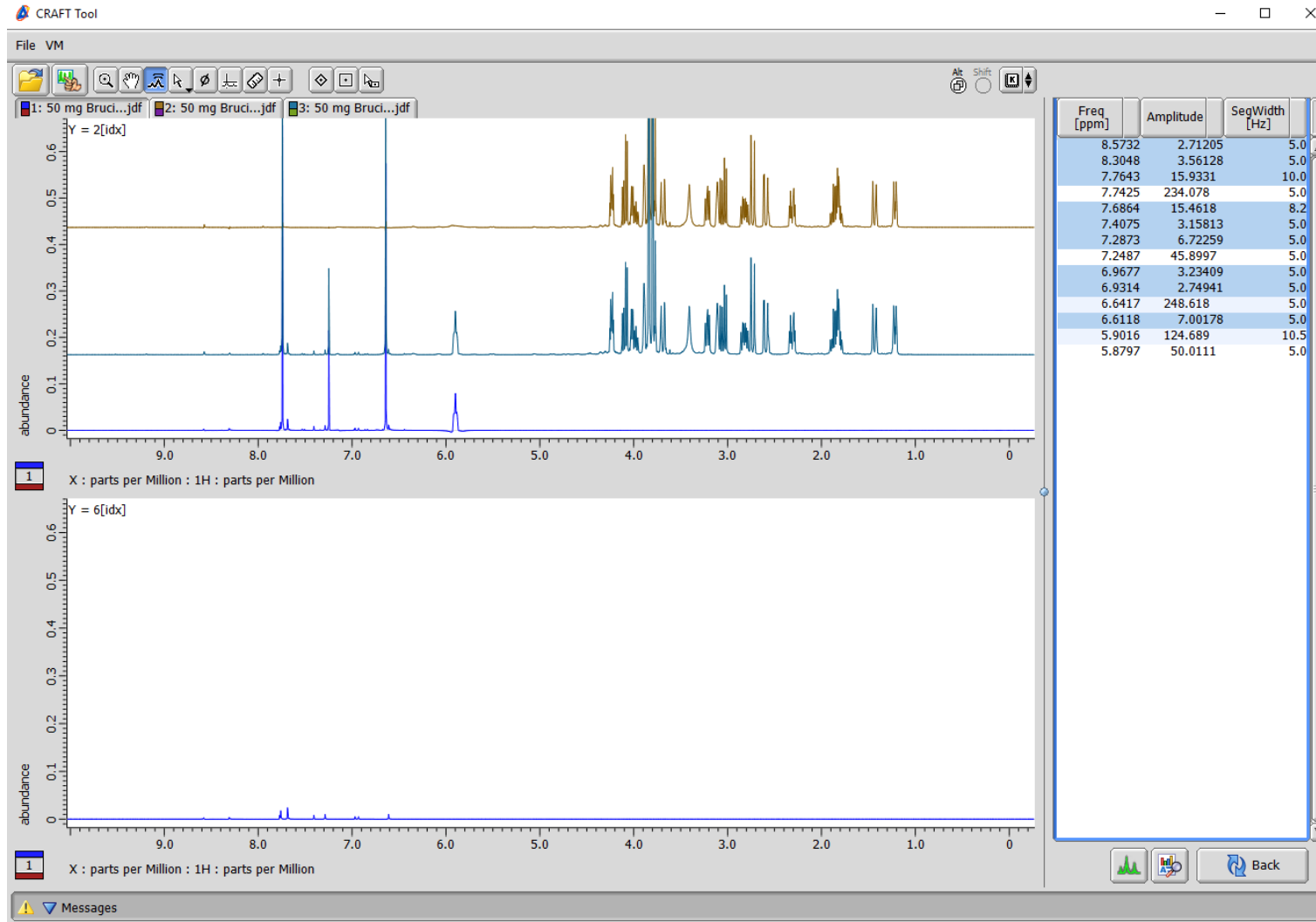
With the same data as previous figure the thresholds for both region of interest and peak result threshold for linelist result have been lowered to 0.1% and the Automatic Peak Pick button clicked once more. Now the peak pick under the ppm scale reveals that the small peaks of interest will be captured. In addition a box has been drawn around the region so that CRAFT will be guided by the peak list but also only for the selected region. It is important to always keep in mind that all of the Craft selection tools will work in combination.

CRAFT result after guidance by peak list and region box



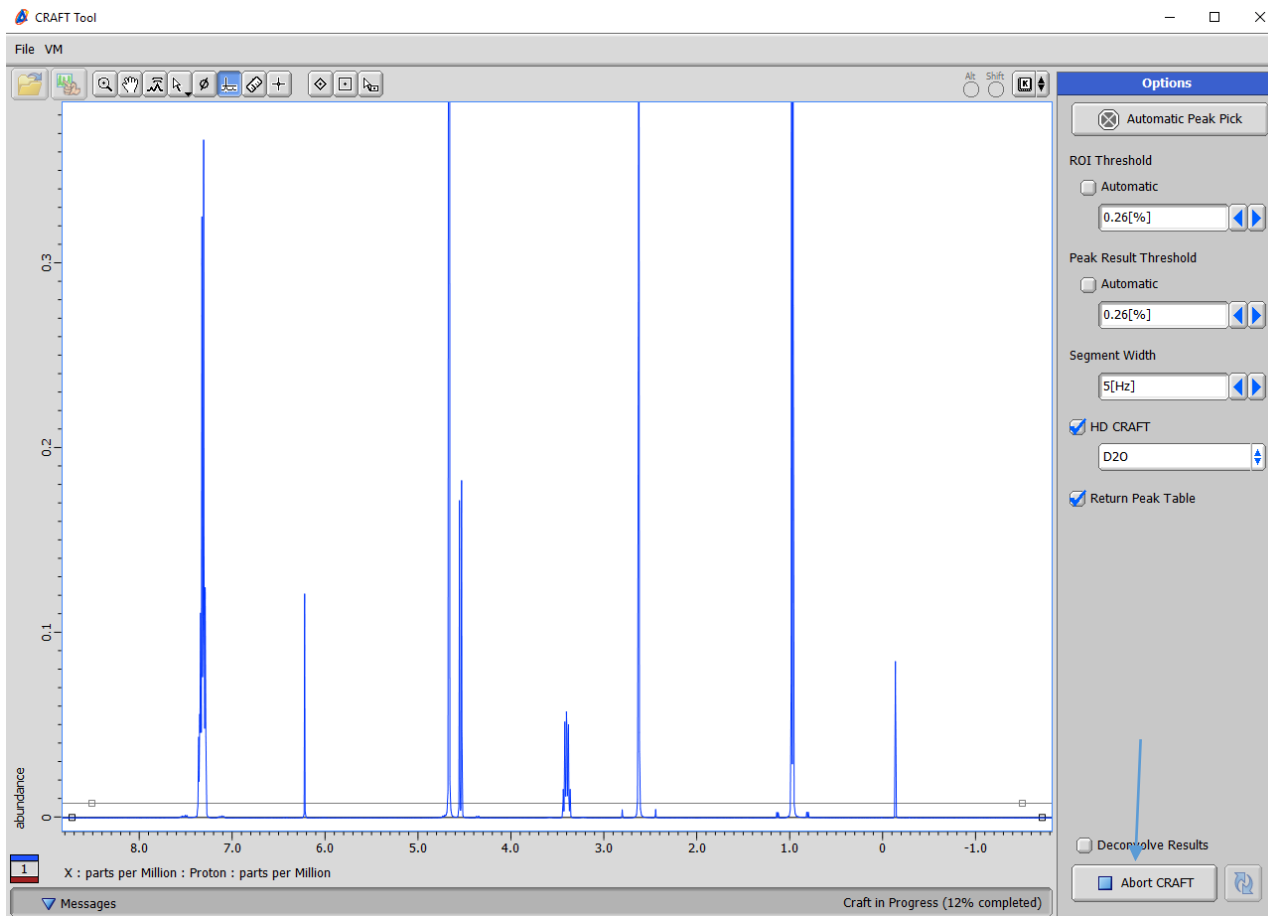
The full CRAFT result showing captured small impurities and complete rejection of the high field region.

Control-click to select just impurity models



Reinforcing once again how selection of linelist results by shift-click or control-click makes it easy to do things like selectively display only the CRAFT models from impurities and/ or ^{13}C satellites.

Monitoring CRAFT Events



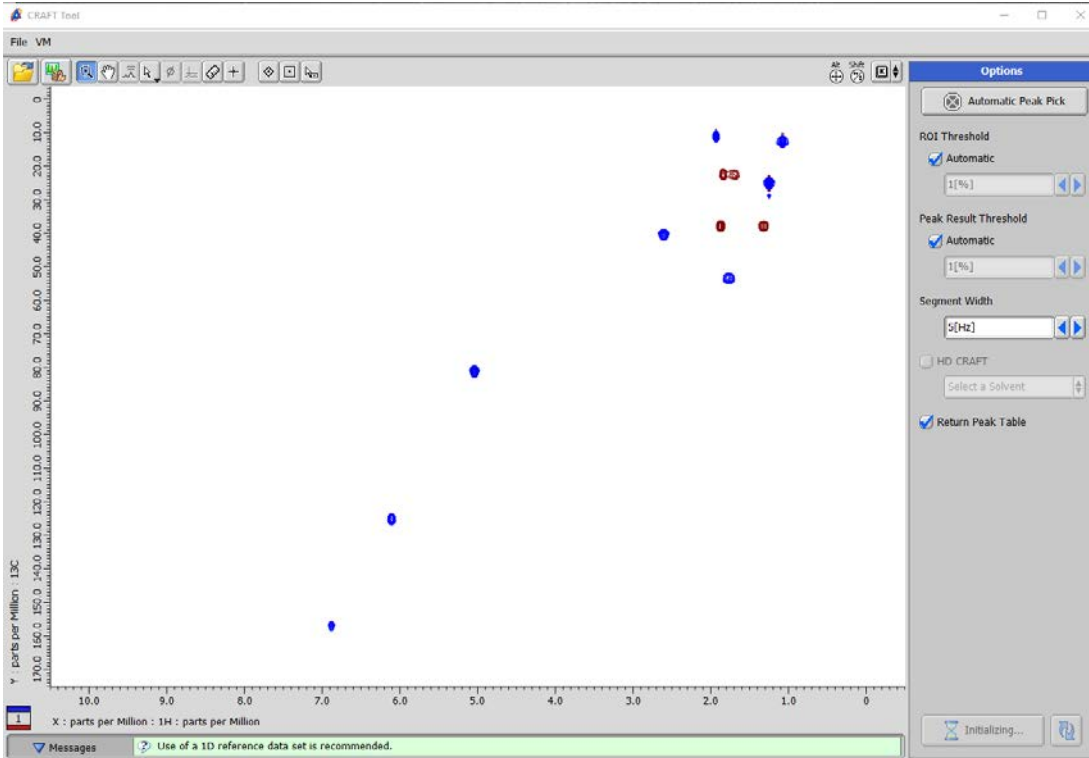
The Message pane at bottom of Craft Tool shows the progress of events. The Craft progress and line list generation stages completion status are shown at all times. If desired the process can be aborted by the Abort CRAFT button which replaces the Start CRAFT button while the processes are running.

In this example the vertical scale has been increased to show all peaks of interest and a horizontal threshold defined so that line list output will capture everything of interest but avoid the ^{13}C satellites.

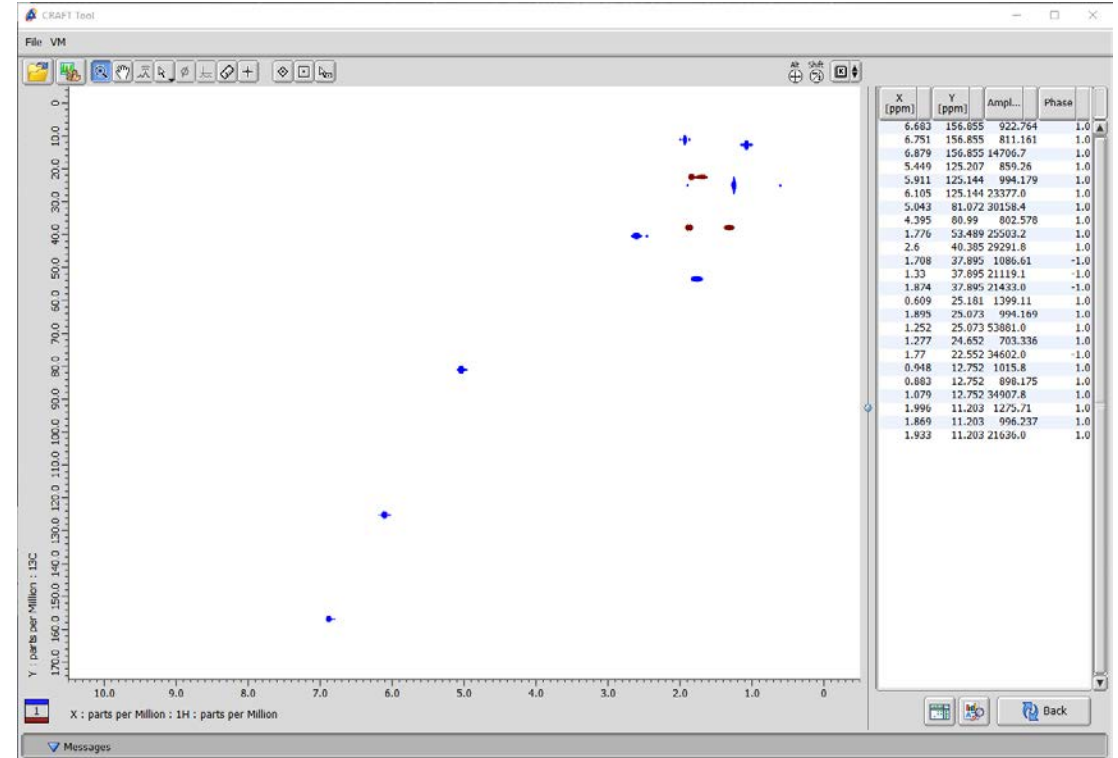
2D Craft Introduction

- To do 2D CRAFT in Delta-CRAFT simply open the desired 2D data set.
- The 2D experiment file should have a couple of parameters defined to allow translation of the X/Y axis phase definition from Delta to VnmrJ. If not included the defaults are $\langle x_display_mode = 'ph', y_display_mode = 'ph1' \rangle$. > For an experiment such as adiabatic H2BC which is absolute value in F2 (X) and phased in F1 (Y); $x_display_mode = 'av'$, and $y_display_mode = 'ph1'$.
- For 2D CRAFT of HSQC-type experiments the full window can be reliably Crafted with no 1D reference spectrum provided for guidance. For experiments with a “1:1” correlation between X and Y axis it is strongly suggested to add a 1D reference spectrum. For HSQC the 1D reference provides a convenient and easy way to tell the CRAFT process to only work on a specific region of the data.
- Following slides show 2D CRAFT in Delta in action.

CRAFT with 2D data set loaded

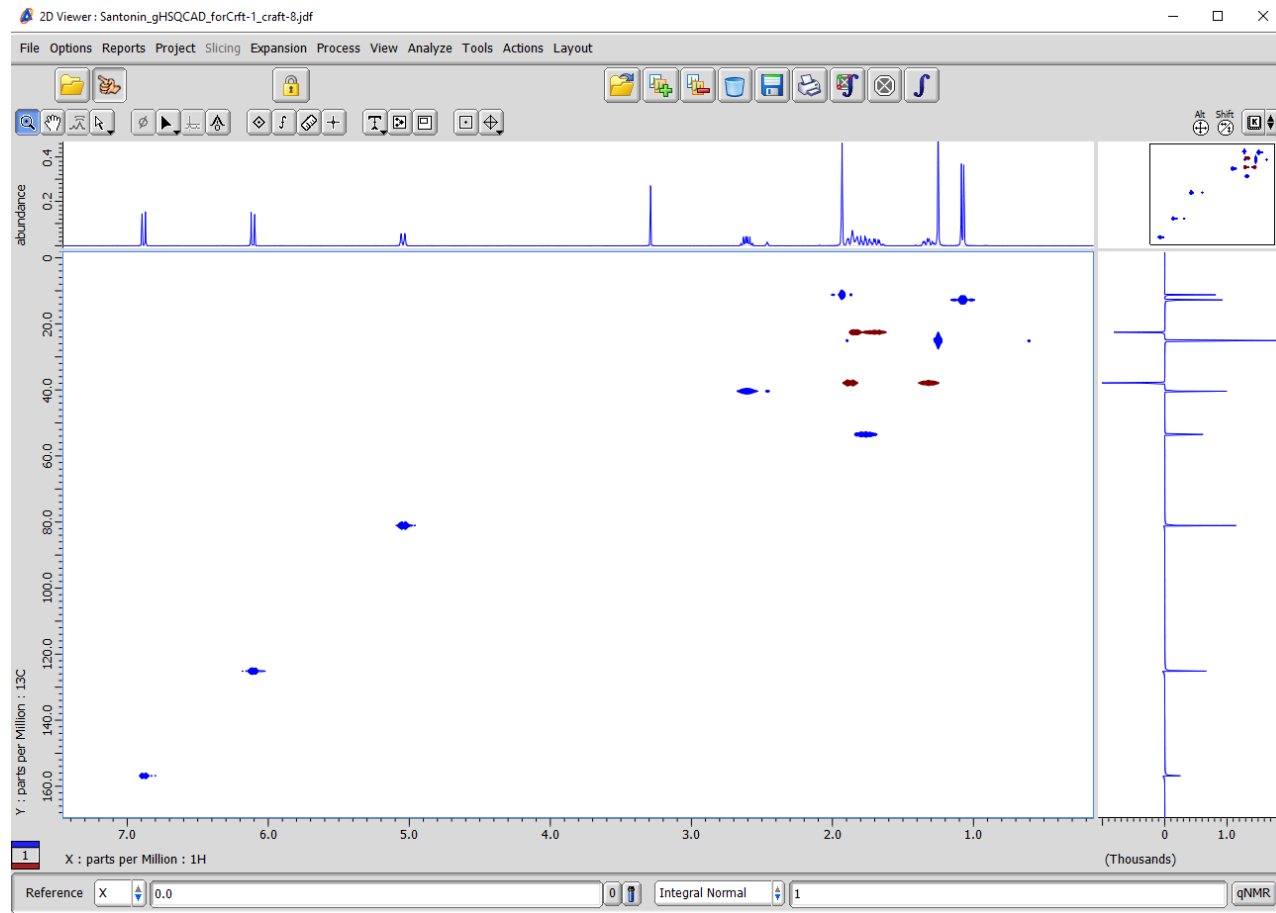


Note how loading 2D asks for 1D reference



2D CRAFT result without 1D reference. Fine for HSQC-type data. Data can be exported to normal 2D Data slate with the extended F1 (Y) projection created by CRAFT by button push

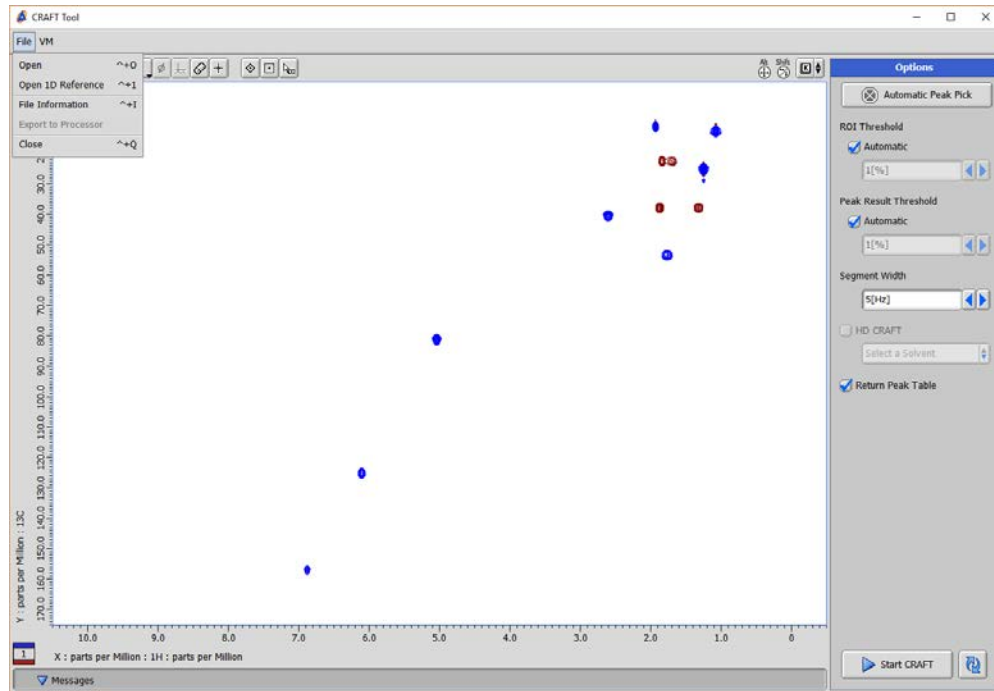
2D CRAFT result gHSQCAD on Santonin



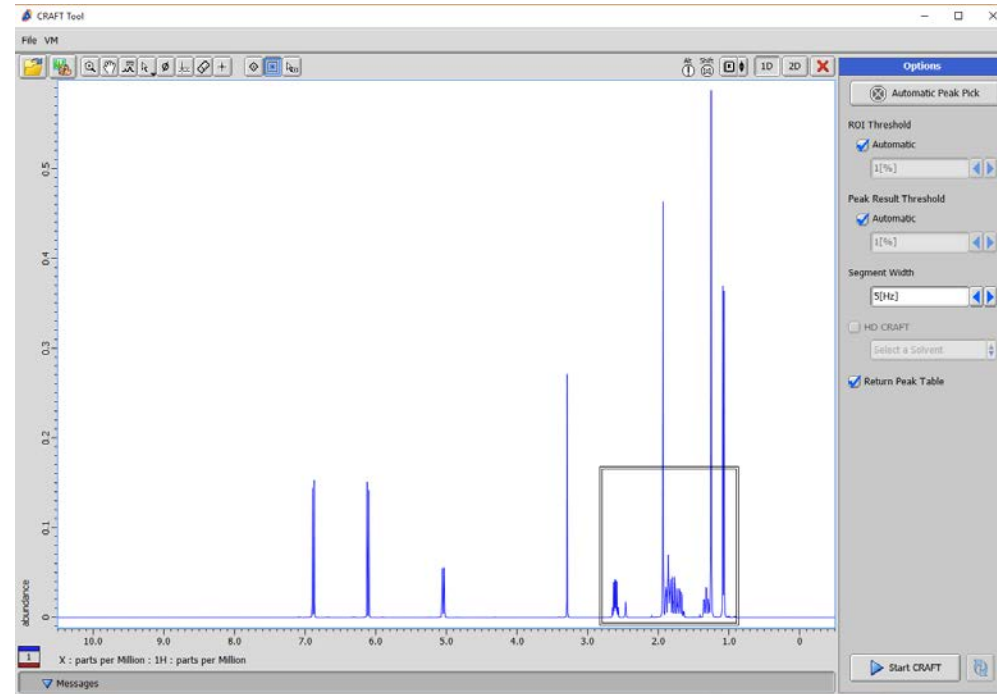
The Y projection here is at a very high 1024 point resolution irrespective of the actual number of 2nd dimension increments used to record the data.

Such an extended y projection is created as a part of all 2D CRAFT processing.

Using a 1D Reference with 2D CRAFT

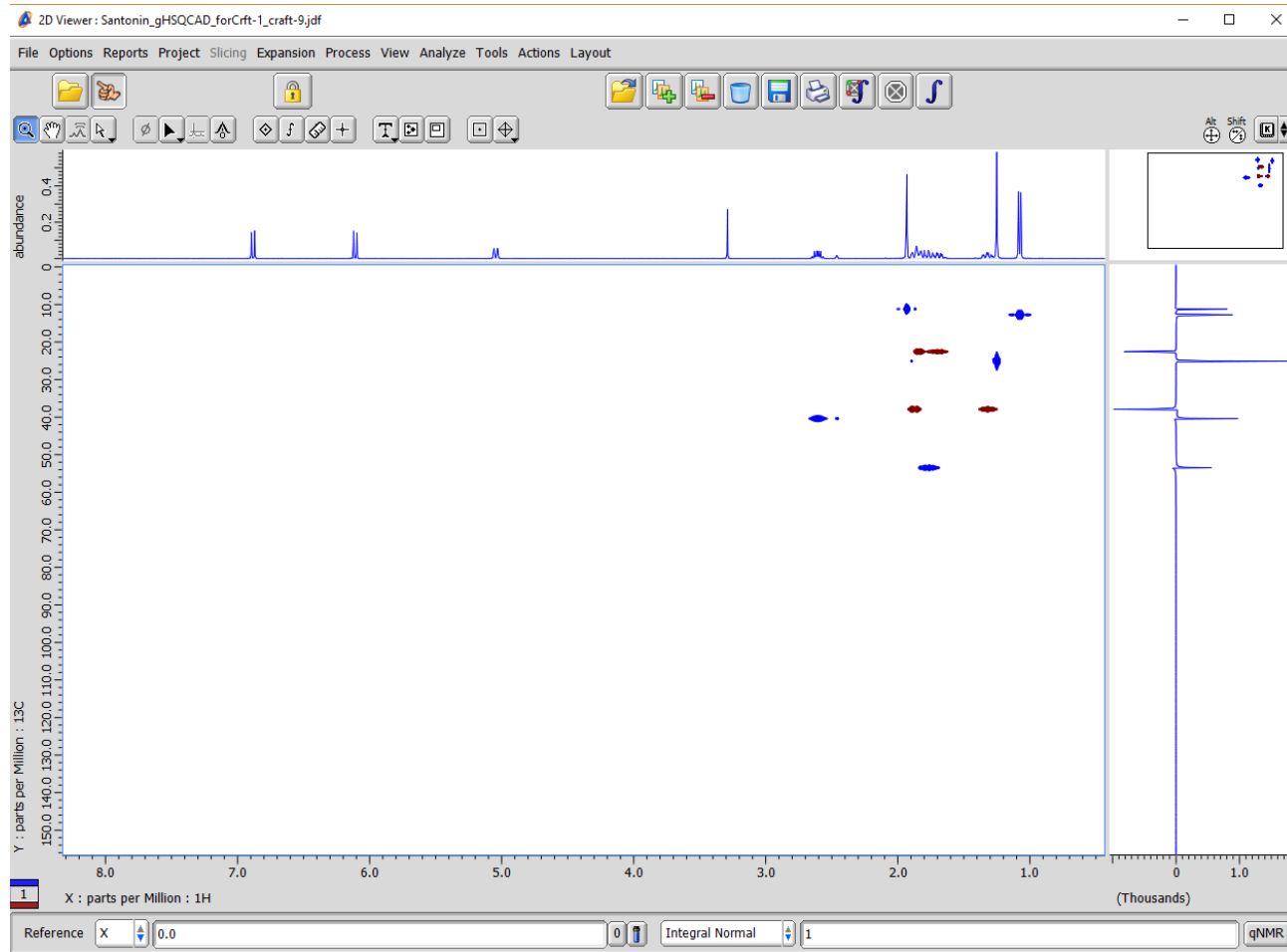


To load a 1D Reference, after opening 2D return to File and choose “Open 1D Reference”.



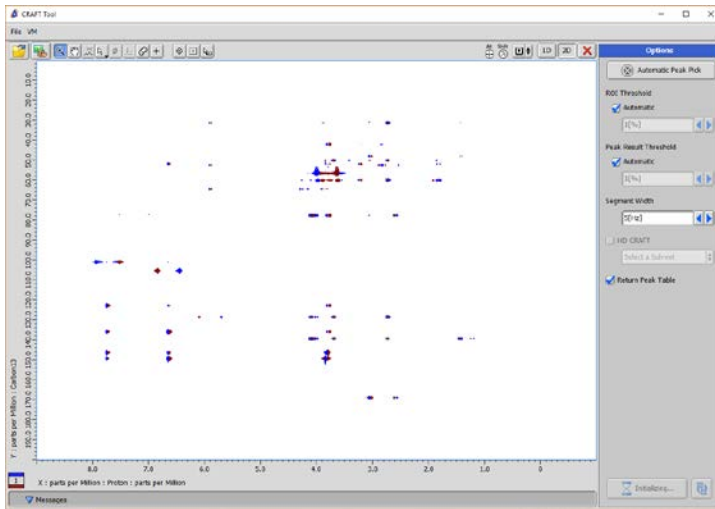
Then with 1D reference open, if desired draw as many selection boxes as desired to limit region of CRAFT analysis. Cursor peak level can also be used in combination.

2D CRAFT Result from region selected multiplicity-edited gHSQCAD experiment

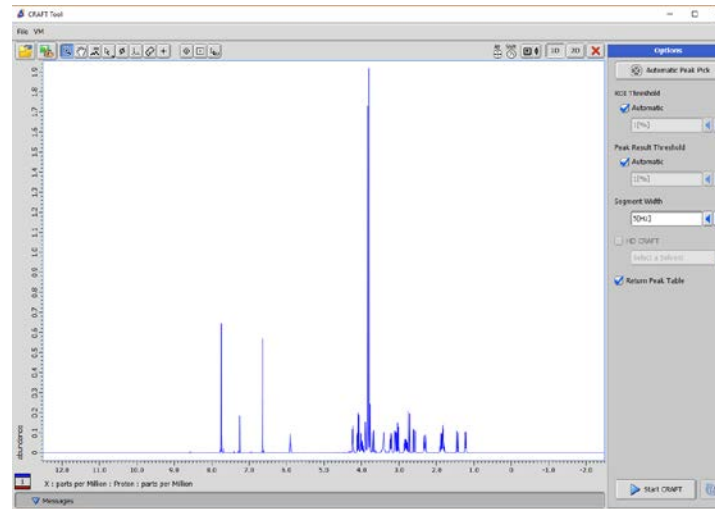


Region-selected 2D CRAFT again with the extended 1024 point f1 (y) projection. The projection is saved separately and can be opened and used for any purpose with all Delta display and output options.

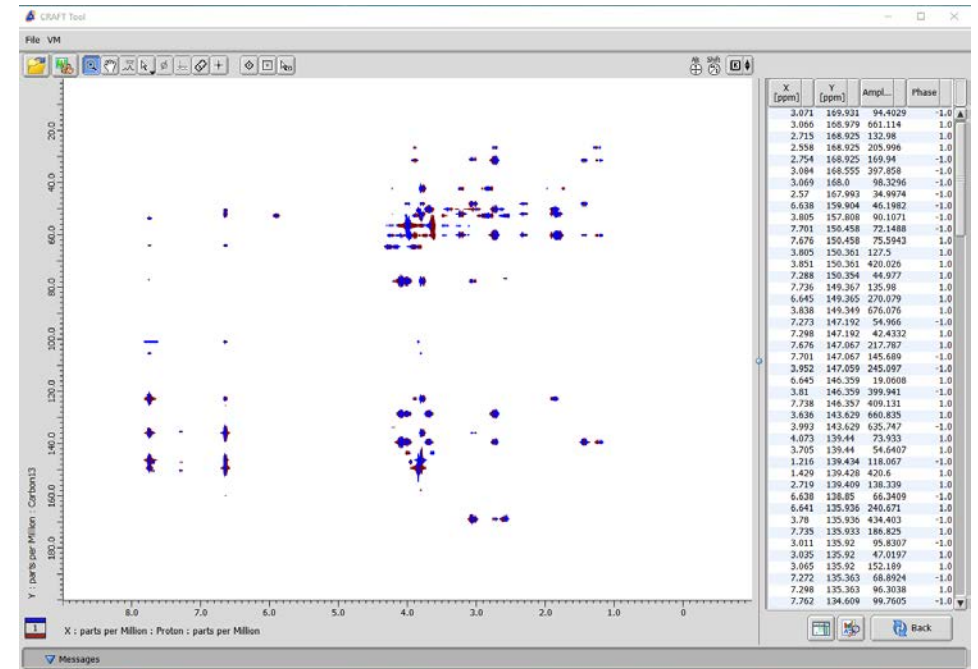
Workflow with 2D gHMBCAD-type Data



1.) Open 2D data



2.) Open 1D reference
(really required for non-
HSQC 2Ds for good result)

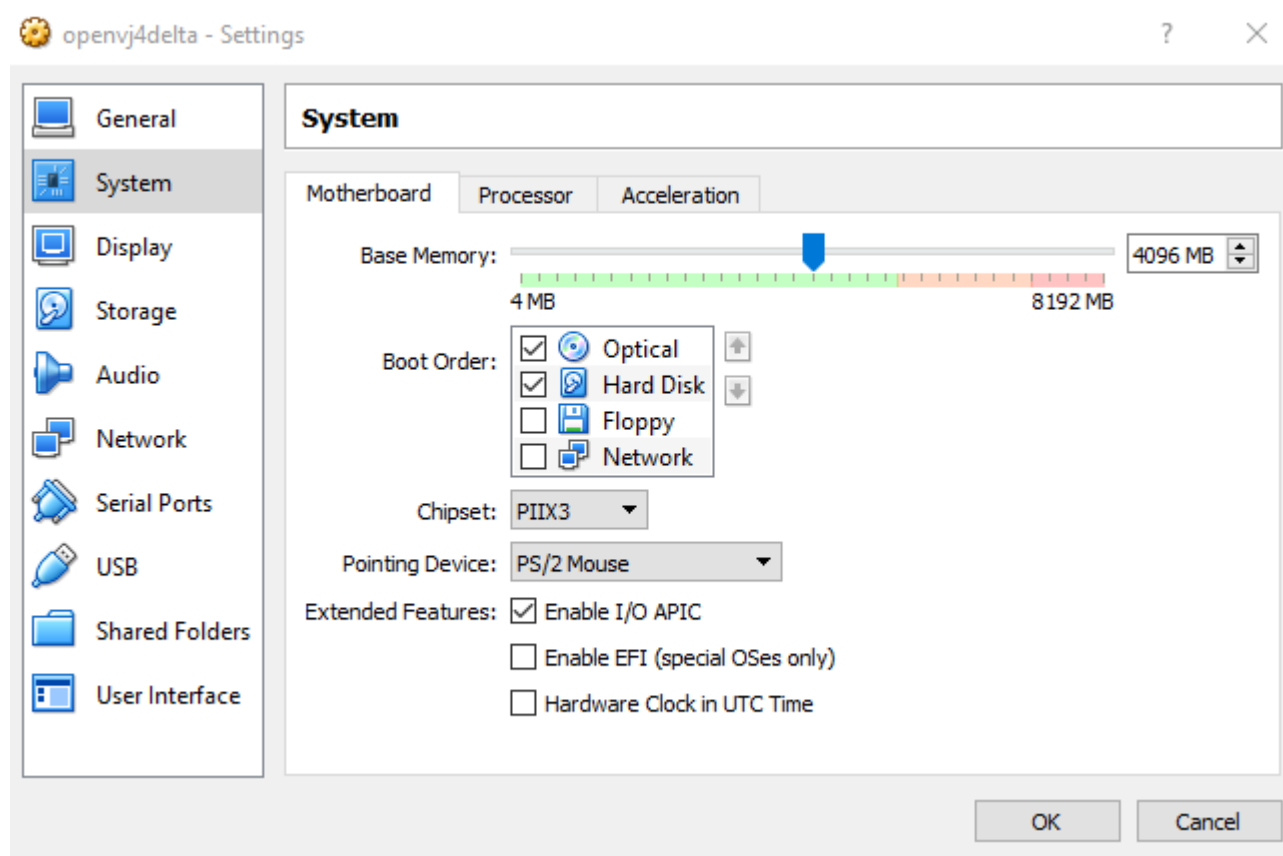


3.) Click Start Craft and get result.

A bit more about the Virtual Machine

- At this time Virtual Box version 5.2.18 is certified for use by Delta-CRAFT. Avoid upgrading to newer versions of Virtual box because doing so may adversely affect communications between Delta and the Virtual machine.
- The Virtual Machine requires a 64bit host for either MS Windows or Apple MacOS.
- The user can control allocation of RAM and processor cores. The default is for the VM to use 2GB RAM and one CPU core. If available more RAM and CPU allocated to the Virtual Machine will improve performance.
- It is highly recommended to use a Solid State disk (SSD) for best performance.

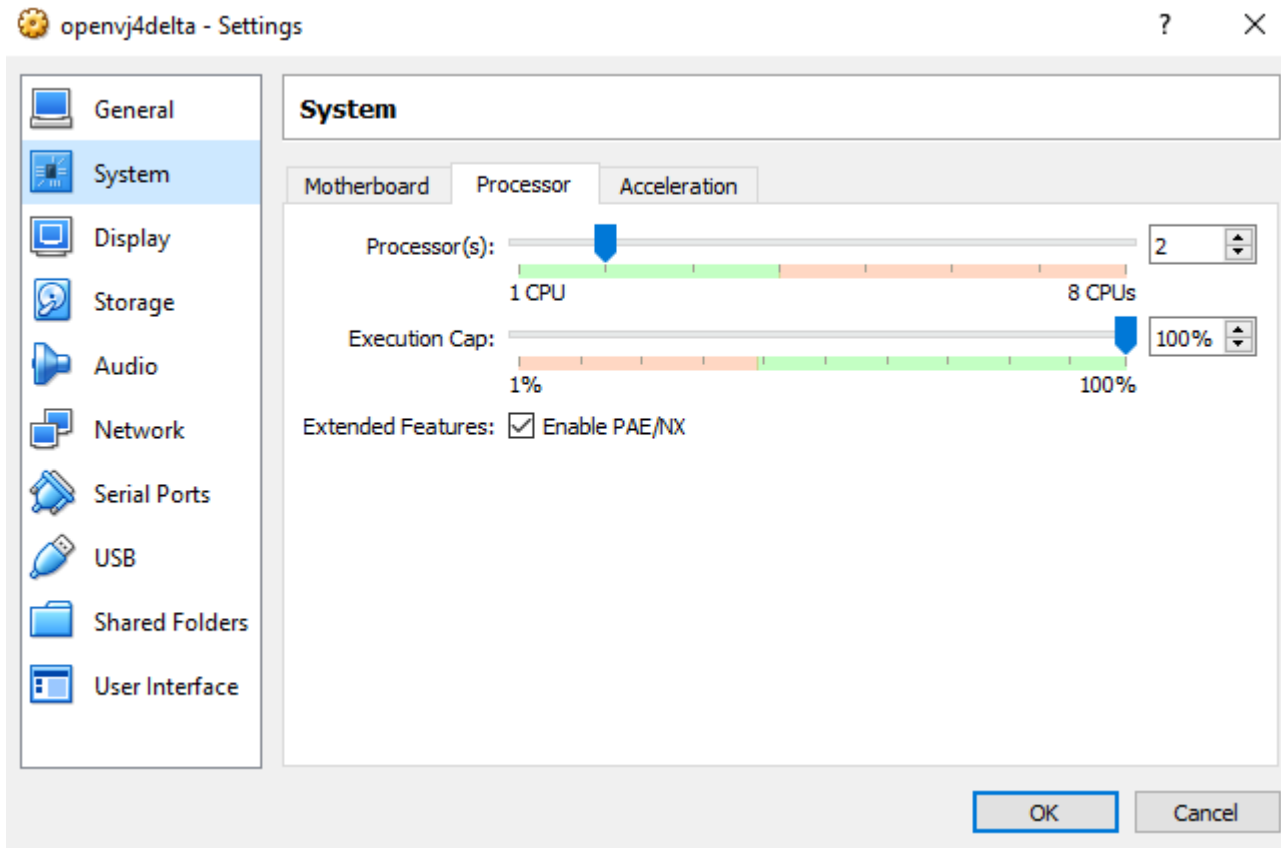
RAM Allocation



With the Virtual machine shutdown
Virtual Box software Settings->System.

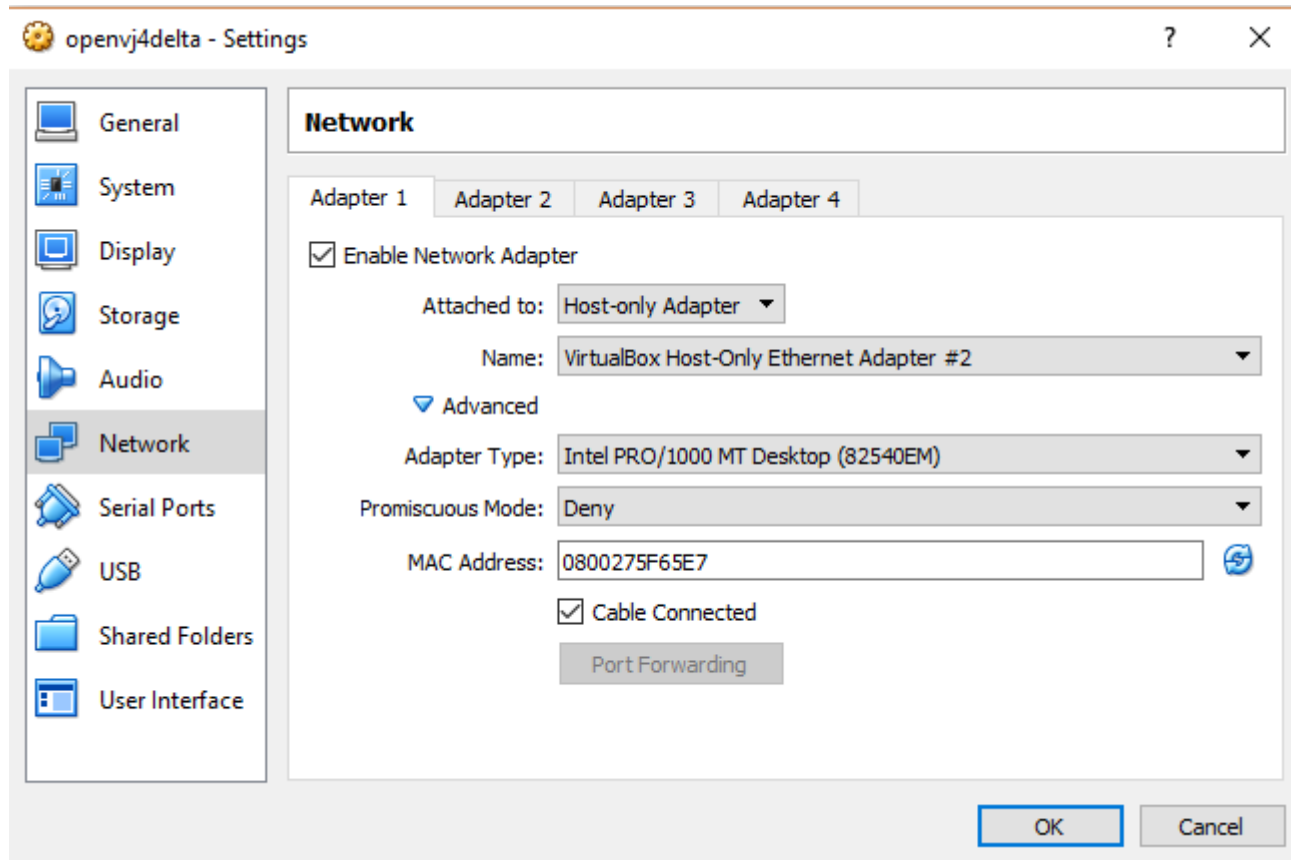
In this example RAM allocation has
been increased from 2GB to 4GB.
Note that this allocation will persist
until the Virtual Box application has
been closed.

Cores Control



The same Virtual Box settings tool provides the place to increase number of CPU cores. This only has effect if the computer has hardware supported Virtualization on it's mother board.

Networking



Anytime a virtual machine is copied from one computer to another it is recommended to choose the Network tab / Advanced settings and click the refresh icon on the right side of the virtual MAC Address line. In addition if a laptop computer is moved from a local area network via wired Ethernet to a wifi connection this might also be required.

Finally, because of the way Windows works moving a laptop to a Public network may even block the internal host-only virtual network from functioning. In that case the wifi may need to be turned off while CRAFTing in public locations.

Configuration of Delta for Craft – more Details

- There are 5 Delta global parameters required for Delta-CRAFT operations. Three of them are handled automatically.
- These three are set automatically in Delta. Can be viewed in Delta->Preferences->Tools. Unlikely these will need adjustment.
 - “SYSTEM.OPENVNMRJ_TO_IPADDRESS” => “192.168.56.11”.
 - “SYSTEM.OPENVNMRJ_FROM_IPADDRESS” => “192.168.56.10”.
 - “OpenVnmrJ: Appliance Name” => <blank> which defaults to “openvnmrj4delta”.
- The final two are in Delta->Preferences->Directory and must be entered by system admin and will be described in some detail.
 - “OpenVnmrJ: CRAFT Result Folder”
 - “OpenVnmrJ: Shared Folder”

Communications between Delta and the Virtual machine.

- In use the technical details are not a concern for the user but having access to the basic information would be useful for solving problems should any arise.
- CRAFT runs on a CentOS 6.8 Linux virtual machine in OpenVnmrJ software. Delta and CRAFT communicate through a private host-only network with ip-addresses of 192.168.56.10 for the Delta host and 192.168.56.11 for the Linux host.
- The Craft_Tool in Delta constructs, sends, and receives the requests and data to and from the Linux host based on the user's requests. The Craft_Tool can even control the startup, shutdown, and sleep of the Linux host.
- In routine use the Linux virtual machine is completely invisible though it can be run and used just like a normal computer whenever it is desired. On the virtual machine the user login called deltauser is provided with no password and is that account is where CRAFTing occurs.
- Installation of the Virtual machine is largely automatic but will be covered in another document.