

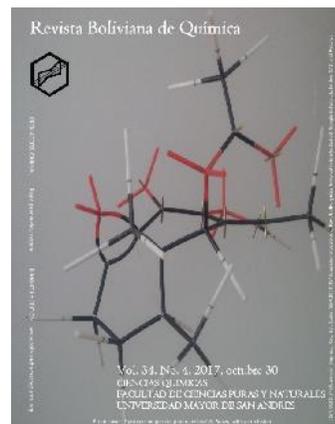


NMR MESTRENOVA, SHORT MANUAL FOR BEGINNERS

NMR MESTRENOVA, PEQUEÑO MANUAL PARA DEBUTANTES

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Short manual, review

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ABSTRACT

This short review presents in a very compact manner the few steps to manipulate 1D and consequently 2D NMR spectra generated in the MestReNova® system. The manual explains how easily we can transform spectral data into different expositive manners. In this sense, we started showing the way to put together ¹³C and DEPT spectra in the same page with peaks aligned. The way to write, erase or edit the name of spectra is also explained. The way to manipulate ¹H NMR spectra is exposed. The treatment and development of most common use 2D spectra is detailed; this includes COSY/NOESY spectra as well as HSQC/HMBC spectra.

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RESUMEN

La presente breve reseña expone de manera muy compacta los pocos pasos para manipular espectros RMN 1D y, en consecuencia, los espectros RMN 2D generados en el sistema MestReNova®. El manual explica con qué facilidad

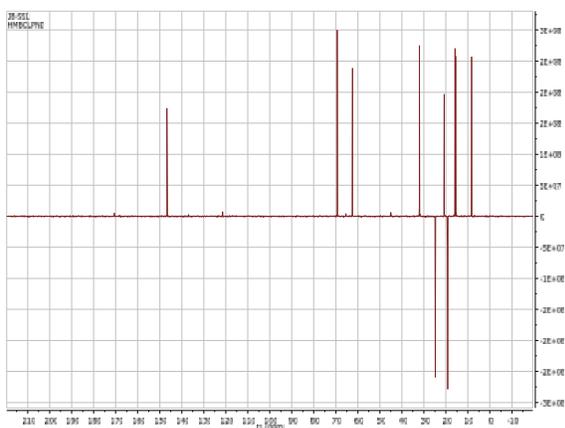


podemos transformar los datos espectrales en diferentes maneras expositivas. En este sentido, comenzamos a exponer la manera de armar los espectros ^{13}C y DEPT en la misma página con los picos alineados. También se explica la forma de escribir, borrar o editar el nombre de los espectros. La forma de manipular los espectros de ^1H RMN también está expuesta. El tratamiento y desarrollo de los espectros 2D de uso más común se detalla; esto incluye los espectros COSY / NOESY así como los espectros HSQC / HMBC.

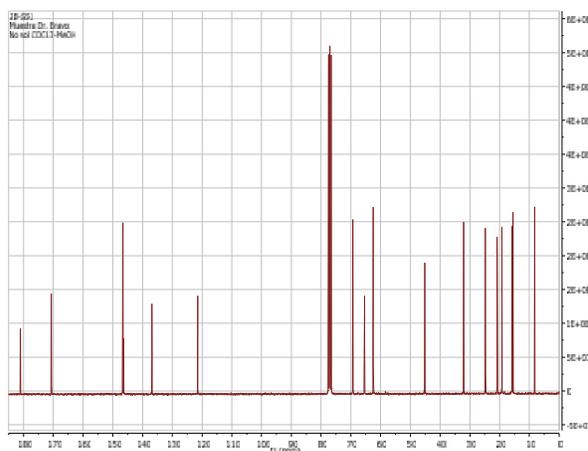
^{13}C NMR

To put ^{13}C and DEPT together in the same page and with peaks aligned [1]

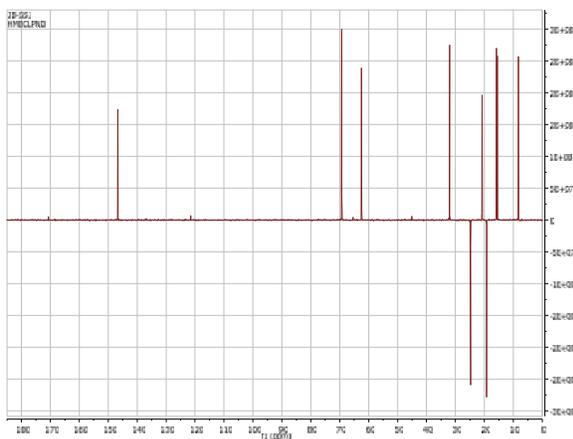
Opening of files. In MestReNova select “open” in the toolbar and open the folder containing the 13C data, then open sequentially: “pdata”, “1” and “1r”; the 13C appears on screen. Select “open” again and open the DEPT data folder and sequentially open: “pdata”, “1” and “1r”; the DEPT appears on screen. Also, both spectra in a shorter size appear in the same page at left under the title “pages”; the DEPT below the 13C. The last screen shows:



Defining of the spectral window. To prepare a doubly 1D spectrum (or two spectra in the same page) we use the same ppm values to define a zoom by means of the “Manual Zoom” option, in the 13C first, and then in the DEPT. Hence, click over the shorter size 13C spectrum at left. It appears on screen. Define a spectral window in ppm with “Manual Zoom” in the toolbar (i.e. 185 ppm left, 0 ppm right). We see:



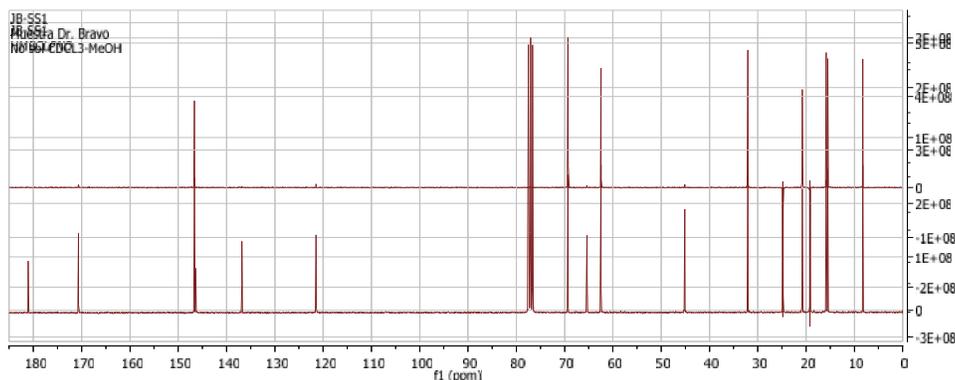
Click over the shorter size DEPT spectrum at left. It appears on screen. Chose the same spectral window as the one defined for the 13C with “Manual Zoom” in the toolbar. We see:



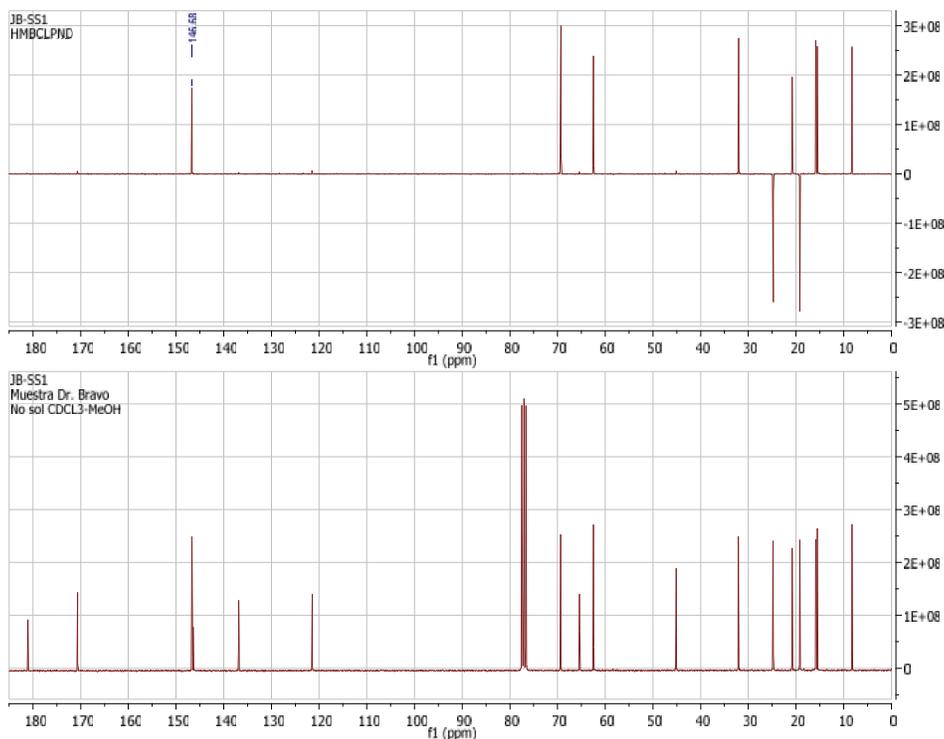
Accommodation of both spectra in the same screen, and peak-picking. Click over the shorter size DEPT spectrum at left. It appears on screen. Click once on the spectrum on the screen, the spectrum becomes reducible. Using the mouse click on the small green square on top and slide the mouse downwards until the spectrum is half the size of the original spectrum. Next, click over the shorter size 13C spectrum at left. It appears on screen. Repeat the previous operation. Click once on the reduced 13C spectrum. Once selected this spectrum, “copy” by clicking the right-hand key of the mouse. Click over the shorter size DEPT spectrum at left. It appears on screen. “Paste” over the spectrum DEPT on screen by using the



right-hand key of the mouse. The view of the screen now is two overwhelmed or merged spectra in the bottom of the screen with all peaks aligned: The DEPT and the ^{13}C spectra. We see:



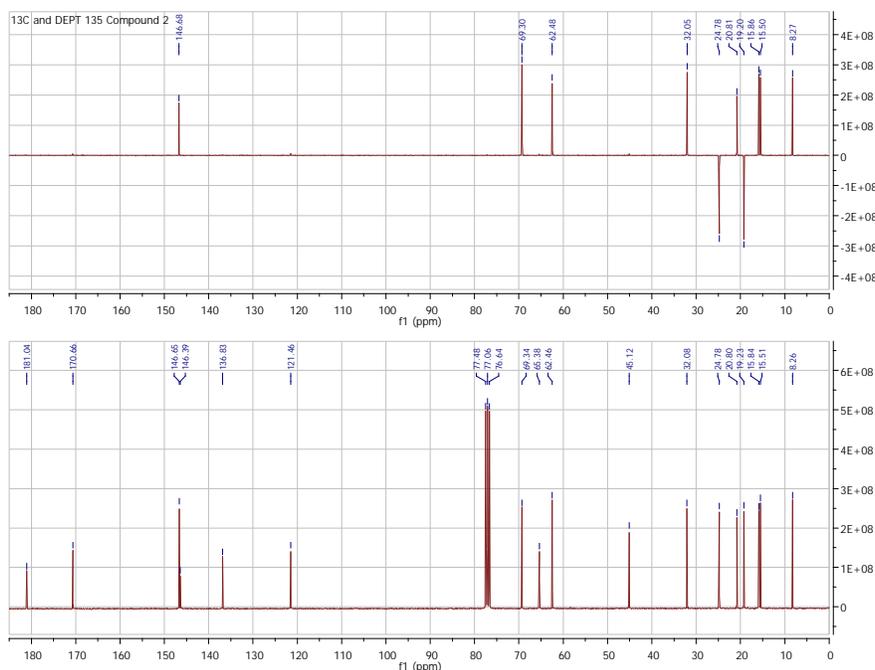
Now you're free to displace any of the spectra downwards or upwards using the shifting arrows on your PC keyboard. We see:



Once separated one spectrum from each other (i.e. put the DEPT in the upper region of the screen and the ^{13}C in the downer region of the screen), choose the option "peak picking" in the toolbar. Once unfolded such option select the option "Manual Threshold". Place the cursor at the left hand but close enough of the most deshielded carbon signal (at lowest field), and below the top of the peak. Press on the left key of the mouse and move it rightwards until the peak has been covered. The cursor is now at the right hand of the peak. Release the right key of the mouse. The signal has been picked and shows its chemical shift value. Repeat this operation with the rest of signals in the spectrum. Do the same with the ^{13}C spectrum currently in the downer region of the screen. Once finished the peak picking operation click on the "peak picking" option in the toolbar. It will unroll and show the option "Manual Threshold". Click on it and the cursor will return to its basic function and it will not be anymore useful only for the peak picking operation. To put a title in the screen double click on the DEPT spectrum (in the upper region of the screen). The window "Properties" appears on screen. Choose option "common" in the menu. Click option "title" if a



title is desired or unclick if not. If the option “title” is chosen then pick up a font and then click in option “...” The “Macro editor” option appears and you can write a title in “Text” and click “OK”. The windows “Properties” appears, and click in “Apply” and “OK”. The title appears on the left upper corner of the spectrum.



Select in the toolbar the option “File”. Select the option “save as”. A new window named “Save as” appears on the screen. Choose either the folder containing the 13C data or the folder containing the DEPT data. The folder “pdata” appears; open it and sequentially open the folder “1”. Name the file and choose the “save” option at the bottom of the current window. A new file appears using the MestReNova logo.

Transfer of the just generated screen containing both spectra with aligned peaks. Select “edit” in the toolbar and then select the “select all” option. The full screen containing both spectra already displaced and accommodated results selected. Unroll the “edit” option in the toolbar. Select the “copy” option. Open a new document in *Word*. “Paste” in the current document of *Word*. The picture is ready to be manipulated as any figure on that page of *Word*. You can rotate it and reduce or enlarge the picture containing both spectra.

TITLE

To put, edit or erase a title of a spectrum [1]

The spectrum appears after clicking “open” and choosing a file or a spectrum (called 1r if 1D or 2r if 2D). In MestReNova do double click over the spectrum. The window “Properties” appears on screen. Choose option “common” in the menu. Click option “title” if a title is desired or unclick if not. If the option “title” is chosen then pick up a font and then click in option “...” The “Macro editor” option appears and you can write a title in “Text” and click “OK”. The windows “Properties” appears, and click in “Apply” and “OK”. The title appears on the left upper corner of the spectrum.

¹H NMR

¹H Peak-picking [1]



In the ^1H spectrum proceed as explained above for the ^{13}C and DEPT spectra when applying the peak-picking operation. Double click on the spectrum on screen. The window “Properties” appears. Select the options “NMR spectrum” and then the “ID spectrum” option. Choose in the vertical toolbar at left the option “peaks”. Mark all four options if not yet done. In the option “peaks” select the font, the “units” (ppm or Hz), “number of decimals”, and “label position”. The other options are already marked (“show position”, “show annotations”, “show tick”). At “label position” chose either “top” or “bottom” or “peak simple”, “peak crowded”, “peak simple” or “peak groups”. Singlets or doublets at lowerfield should prefer “peak simple”. Cluster signals of methylene groups at higher field should prefer “peak crowded”. Once defined these parameters, do “apply” and OK”. Go to the toolbar and select “Peak-picking” and then “Manual threshold”. Proceed selecting each peak to be picked in the spectrum. You’re done. Save as already explained above.

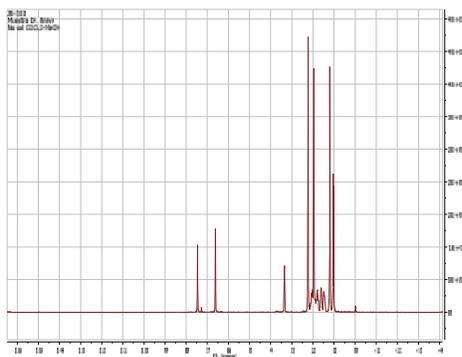
^1H Integration [1]

Double click on the spectrum on screen. The window “Properties” appears. Choose in the vertical toolbar at left the option “integrals”. Mark on “integrals” and select: Font, decimals, position, margin, for the “label”. Mark color, position, maximum height for the “curve”. Do “apply” and “OK”. Go to the tool bar and select “Integrations” and there click on “manual”. In order to set the number of protons for the integral of a signal, right-click the mouse on the integral curve on the spectrum. Choose “edit integral” option. The Integral manager window appears on screen. Choose in “Normalized” the number of protons for the signal and “apply to all”. You’re done. Save as explained.

HOMONUCLEAR 2D SPECTRA

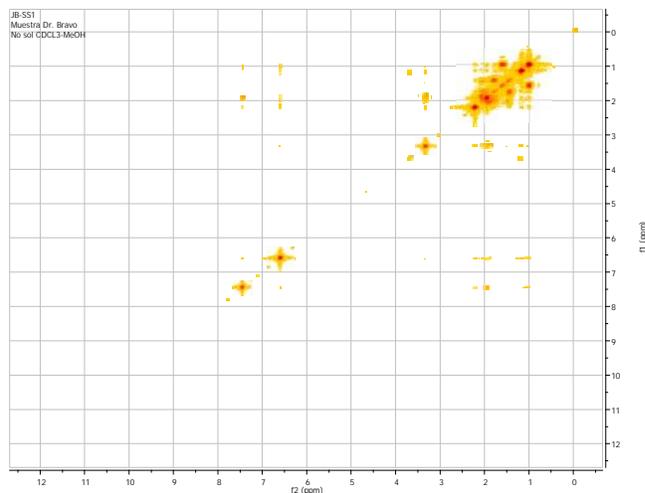
A COSY/NOESY spectrum [1]

Opening of files. In MestReNova select “open” in the toolbar and open the folder containing the COSY/NOESY data, then open sequentially: “pdata”, “1” and “2rr”; the unsymmetrized COSY/NOESY appears on screen. Select “open” again and open the ^1H data folder and sequentially open: “pdata”, “1” and “1r”; the ^1H appears on screen. Also, both spectra in a shorter size appear in the same page at left under the title “pages”; the ^1H below the COSY/NOESY. We see:



Symmetrizing and choosing size of crosspeaks in COSY. Before defining the spectral window it is convenient to symmetrize the COSY spectrum. Click on the small COSY spectrum at the left of the screen. The COSY spectrum appears on screen. Click in the COSY spectrum in the screen. Search in the toolbar the option “processing” then choose “symmetrize” and then the sub-option “COSY-like”. The COSY spectrum symmetrized appears on screen. Choose the desired and most fitting intensity of the COSY contour plot by means of the mouse’s wheel (to increase or decrease the crosspeaks’ size) or by pressing on the “+” (increase) or “-“ (decrease) keys on your PC keyboard.

Choosing size of crosspeaks in NOESY. The ^1H spectrum is on the screen. Click over the shorter size NOESY spectrum at the left of the full screen. The NOESY spectrum appears on screen. Choose the desired and most fitting intensity of the NOESY contour plot by means of the mouse’s wheel (to increase or decrease the crosspeaks’ size) or by pressing on the “+” (increase) or “-“ (decrease) keys on your PC keyboard. We see:

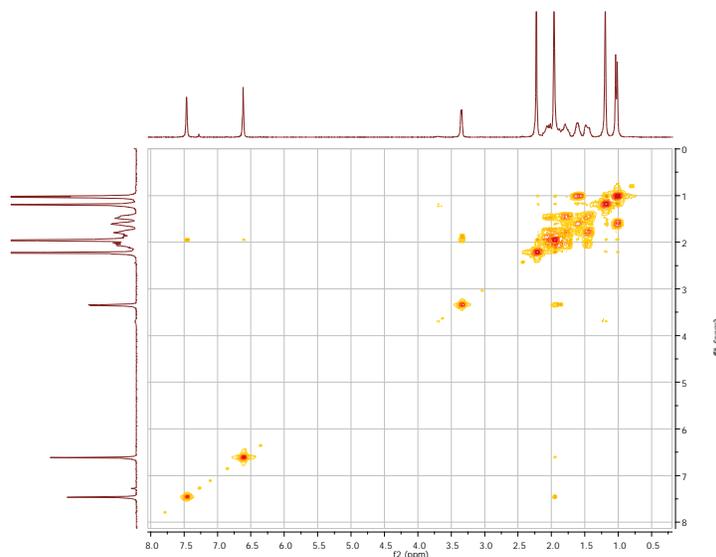
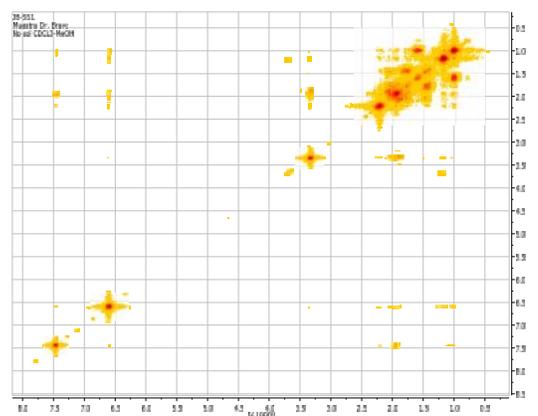


Plotting the proton projection and applying the “Contour Plot” option.

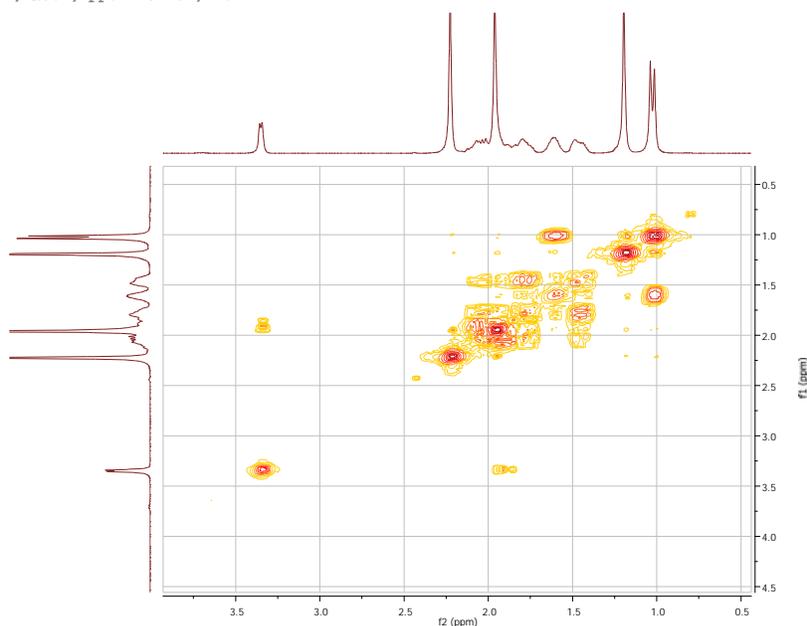
Check for the vertical toolbar at the left side of the screen of MestReNova. Look for the “Bitmap Plot” icon. Click on the two arrows below such icon. An extra icon appears at the right side of the “Bitmap Plot” icon under the name “Show traces”.

Unfold “Show traces” and choose “Setup”. The window “Setup traces” appears on screen. Click on the title of the ^1H spectrum appearing in the square “Available 1D spectra:”. Select the horizontal trace by clicking in the corresponding box. Click again on the title of the ^1H spectrum appearing in the square “Available 1D spectra:”. Select the vertical trace by clicking in the corresponding box. Click OK and the current window disappears. The COSY/NOESY spectrum appears on screen with both projections (traces). Check for the vertical toolbar at the left side of the screen of MestReNova. Look for the “Bitmap Plot” icon and unfold the corresponding menu clicking on the button at right, four options appear, chose “contour plot”. “Save as” the current COSY/NOESY spectrum. The window “Save as” appears on screen. Choose the folder containing the COSY/NOESY data. Open sequentially the folders “data” and “1” and click on “save” previously giving a name to the file.

Defining of the spectral window. Click on the COSY/NOESY spectrum on screen. Select “zoom in” in the toolbar. Select a spectral window on the COSY/NOESY pressing the left key of the mouse and moving throughout the spectrum including all pertinent crosspeak. Release the mouse’s key. The spectral window appears on screen. We see:



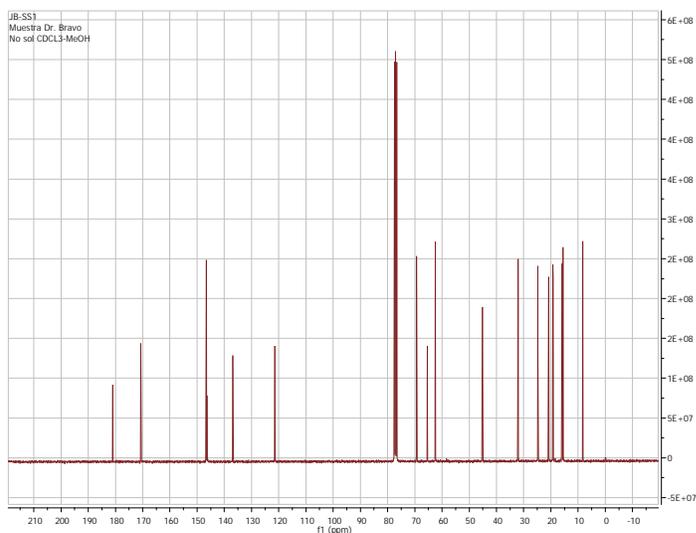
You can zoom on this spectrum on screen with “zoom in” in the toolbar. Save as: _____ this zoom. We see:



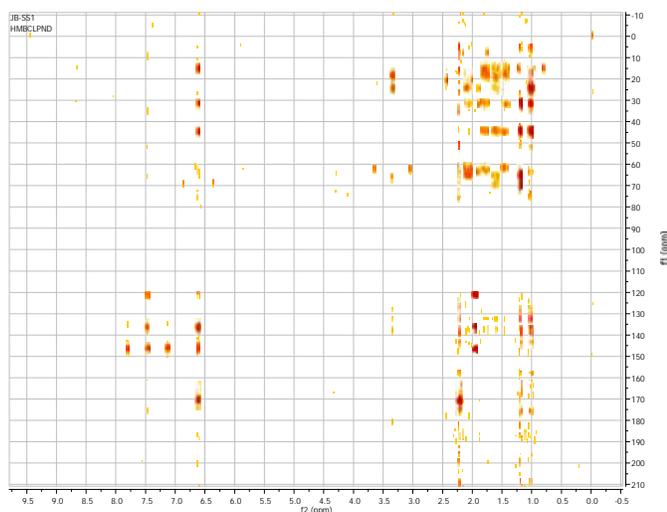
HETERONUCLEAR 2D SPECTRA

A HSQC/HMBC spectrum [1]

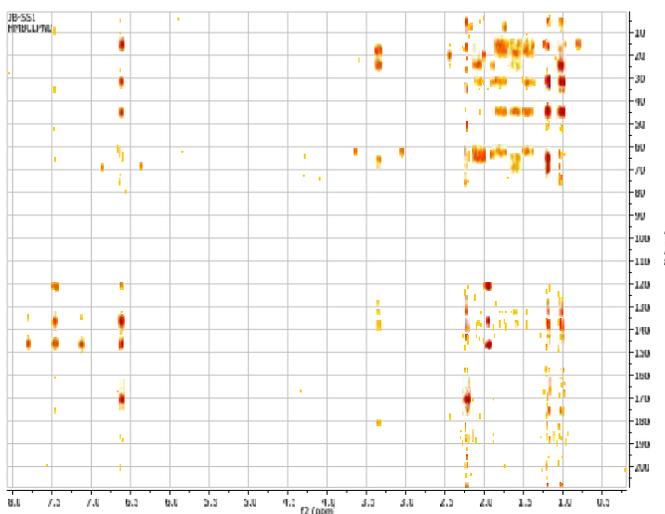
Opening of files. In MestReNova select “open” in the toolbar and open the folder containing the HSQC/HMBC data, then open sequentially: “pdata”, “1” and “2rr”; the HSQC/HMBC appears on screen. Select “open” again and open the 1H data folder and sequentially open: “pdata”, “1” and “1r”; the 1H appears on screen. Select “open” again and open the 13C data folder and sequentially open: “pdata”, “1” and “1r”; the 13C appears on screen. Also, the three spectra in a shorter size appear in the same page at left under the title “pages”; the 1H below the HSQC/HMBC, and the 13C below the 1H. we see:



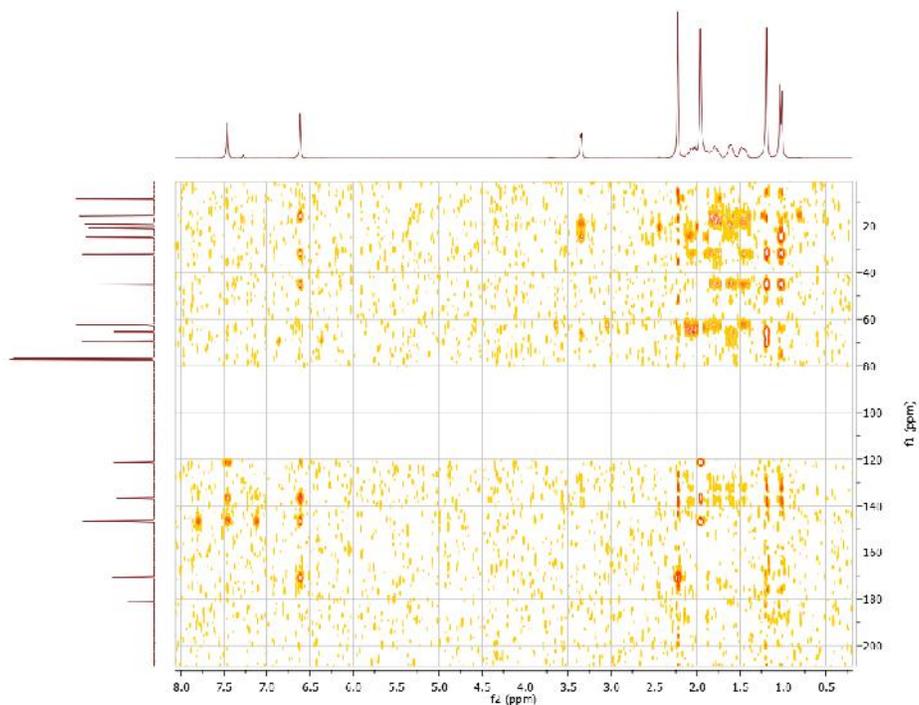
Choosing size of crosspeaks in HSQC/HMBC. The 13C spectrum is on the screen. Click over the shorter size HSQC/HMBC spectrum at the left of the full screen. The HSQC/HMBC spectrum appears on screen. Choose the desired and most fitting intensity of the HSQC/HMBC contour plot by means of the mouse's wheel (to increase or decrease the crosspeaks' size) or by pressing on the “+” (increase) or “-” (decrease) keys on your PC keyboard. We see:



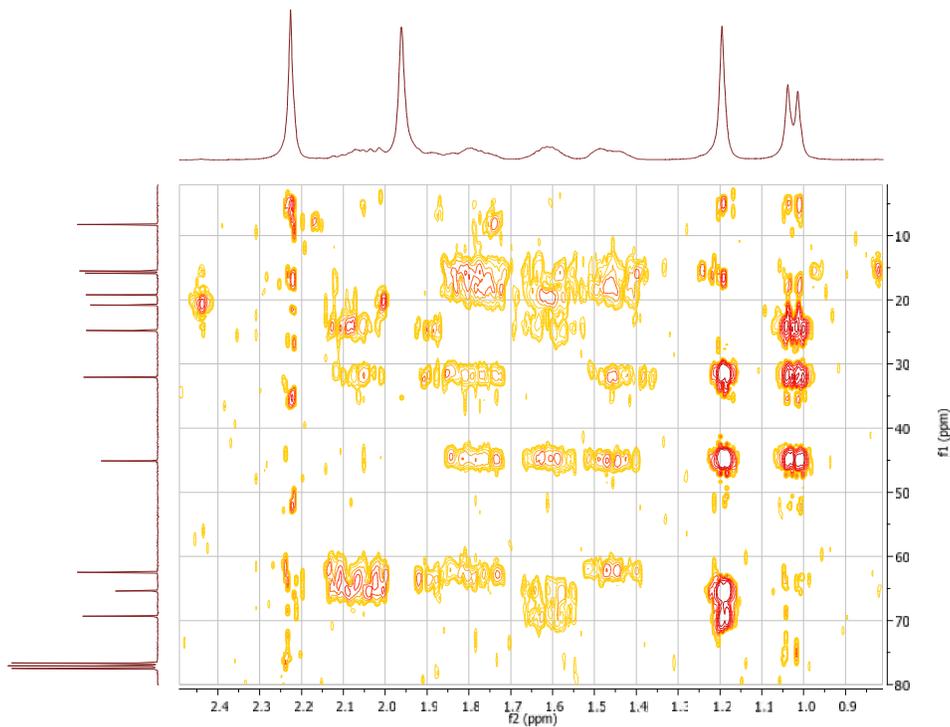
Defining of the spectral window. Click on the HSQC/HMBC spectrum on screen. Select “zoom in” in the toolbar. Select a spectral window on the HSQC/HMBC pressing the left key of the mouse and moving throughout the spectrum including all pertinent crosspeak. Release the mouse’s key. The spectral window appears on screen. We see:



Plotting the proton and carbon projections. Check for the vertical toolbar at the left side of the screen of MestReNova. Look for the “Bitmap Plot” icon. Click on the two arrows below such icon. An extra icon appears at the right side of the “Bitmap Plot” icon under the name “Show traces”. Unfold “Show traces” and choose “Setup”. The window “Setup traces” appears on screen. Click on the title of the 1H spectrum appearing in the square “Available 1D spectra:”. Select the horizontal trace by clicking in the corresponding box. Click on the title of the 13C spectrum appearing in the square “Available 1D spectra:”. Select the vertical trace by clicking in the corresponding box. Click OK and the current window disappears. The HSQC/HMBC spectrum appears on screen with both projections (traces). Check for the vertical toolbar at the left side of the screen of MestReNova. Look for the “Bitmap Plot” icon and unfold the corresponding menu clicking on the button at right, four options appear, chose “contour plot”. “Save as” the current HSQC/HMBC spectrum. The window “Save as” appears on screen. Choose the folder containing the COSY/NOESY data. Open sequentially the folders “data” and “1” and click on “save” previously giving a name to the file. We see:



You can zoom on this spectrum on screen with “zoom in” in the toolbar. Save as: _____ this zoom. We see:



REFERENCES

1. MNOVA, MESTRELAB. 2010, MestReNova Manual, MestreNova 6.2.1.