

Two-dimensional experiments with Vnmrj 2.2

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This manuscript is designed to provide only the minimum information required to perform the most common routine two dimensional experiments with VnmrJ 2.2 and above and Chempack 4.1 in the NMR Facility at the University of Michigan. It doesn't include detailed information about the optimization of parameters or about the NMR techniques themselves. The user must be aware that further optimization, before and after acquisition is possible and recommended. Vnmrj is very complex, with many more options and commands than a casual user would want to know, and making this manuscript more comprehensive would render it tedious for such kind of user. Extensive information can be found in the references. Please read this manuscript in its entirety before attempting to run or process your first 2D spectrum as some procedures may become clearer after you finish reading a whole section.

When doing 2D spectroscopy, it is advantageous (but not required) to have the high resolution 1D spectra of the compound in other workspaces for fast access to them. In this guide, we will assume you have a proton spectrum in workspace 1, a carbon in workspace 2 if needed, and the 2D spectra in workspaces 3 or 4.

Acquisition

Just like in an arrayed experiment, a two-dimensional spectrum is acquired as a series of one-dimensional FIDs (free induction decays). But in contrast to arrayed 1D experiments, the data set is Fourier-transformed twice to provide the two frequency dimensions. The directly detected dimension is called *F2* and is usually plotted along the x-axis, while the dimension detected indirectly is called *F1* and is usually plotted along the y-axis. The number of collected spectra is called the *number of increments* (n_i) and it determines the resolution in *F1* and the total acquisition time.

Experiment selection

There are many 2D experiments available in vnmrj. Here we will only describe the general features of the most common ones. They can be broadly separated into homonuclear or heteronuclear experiments.

- **Homonuclear correlation experiments** are those where the signals to be correlated belong to the same nucleus. Examples are: COSY, gCOSY, TOCSY, zTOCSY, NOESY and ROESY.
- **Heteronuclear correlation experiments** are those where the signals to be correlated come from different nuclei. Due to instrumental limitations, early 2D heteronuclear spectra were measured by detecting the least sensitive nucleus. This is called *direct detection* and an example of such experiment is the classic HETCOR. These have been largely superseded by the **Indirect Heteronuclear correlation experiments**, where the nucleus actually detected is proton or, less commonly, fluorine and thus they are more sensitive. Nowadays, the term indirect is retained only for historic reasons. Examples are ^1H - ^{13}C correlations like HSQC, HMQC and HMBC.

Besides the major classification as heteronuclear or homonuclear, 2D experiments have different designations based on how the pulse sequences are implemented:

- **Gradient selected experiments** are 2D experiments which use magnetic field gradients to select coherences (correlations). They are faster than classical experiments, but there is a slight sensitivity loss compared to the non-gradient version. Examples are Gradient COSY (gCOSY) and Gradient HSQC (gHSQC).
- **Experiments with Adiabatic pulses** use complex, shaped, frequency-swept pulses (so called “adiabatic”) instead of only rectangular pulses in the pulse sequence. With this, some phase distortions along the X dimension, common in heteronuclear experiments, are avoided. Examples are HSQCAD, HMBCAD and CIGARAD.
- **CRISIS2 experiments** use more complex adiabatic pulses and broadband inversion pulses (bip). These experiments yield better sensitivity and line shape than the adiabatic and non-adiabatic counterparts. They are especially recommended when the spectral widths in either dimension are large, for example for ^{19}F - ^{13}C correlations. Examples are: c2hsqc and gc2hmbc.
- **Band selective experiments** allow the acquisition of 2D spectra where only a portion of the F1 spectral width is excited with no peak folding. With these experiments, the resolution in F1 can be greatly improved. They are recommended when it is important to resolve very close signals in F1. Examples: selective NOESY (bashdNOESY) and selective HSQCAD (bsHSQCAD).
- **X-edited TOCSY experiments** combine elements from heteronuclear correlations and TOCSY pulse sequences to provide 2D heteronuclear spectra where the correlation from one carbon is transferred to several other protons in a chain. They are useful to make assignments in crowded regions of a spectrum. Examples are HSQC-TOCSY and gHMQC-TOCSY.
- **Hadamard experiments** use a technique for acquiring multidimensional data sets using a small number of transients and reconstructing the 2D spectrum using a Hadamard transform.
- **DOSY (Diffusion) experiments** are not real 2D experiments, but they can be plotted in a two-dimensional fashion. They can be used to measure self diffusion coefficients.

So what experiments should you use? For the routine analysis of small and middle size organic and inorganic compounds, the following are recommended.

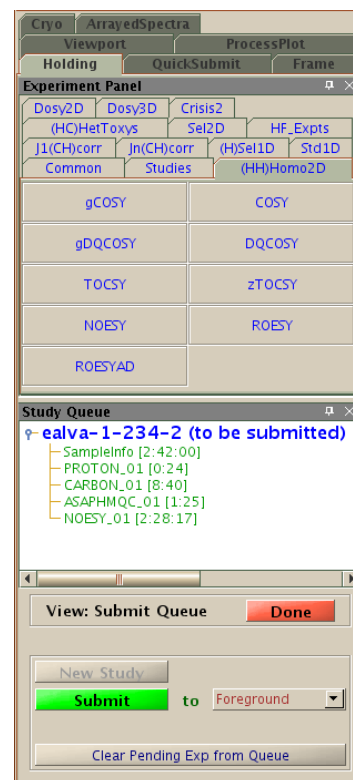
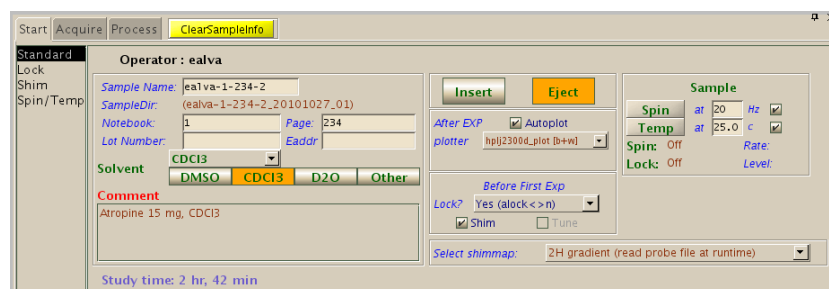
- ^1H - ^1H correlations: 2 or 3 bonds gCOSY; multiple bonds: TOCSY.
- ^1H - ^1H through-space correlations: NOESY for molecular weights up to 1000 and ROESY for larger molecules.
- ^1H - ^{13}C or ^1H -X correlations: one-bond: gHSQCAD, ASAPHMQC or gc2hsqc; 2 or 3 bonds: CIGARAD or gHSQCAD-TOCSY.
- ^{19}F - ^{13}C correlations: one bond: gc2hsqc; 2 or 3 bonds: gc2hmbc.

If you have enough material (more than 5 mg), a gradient-selected experiment will usually give you an acceptable spectrum with the default parameters in a few minutes; but if your sample is dilute, you will get better sensitivity with a non-gradient experiment and a number of transients (n_t) larger than 8 or 16. If you need good resolution in the F1 dimension, increase the number of experiments (n_i).

Automated acquisition

Perhaps the easiest way to acquire a 2D spectrum –but not necessarily the best–, is with automation. After you load the sample in the magnet, vnmrj can automatically set the lock, shim the sample, tune the probe, acquire a series of 1D and 2D spectra, and process, plot and save the spectra. You may only need to manually plot expansions. If you want to do this, proceed as follows:

1. Insert the sample in the magnet as usual.
2. Click **New Study** in the lower part of the **Holding** vertical panel (shown on the right), or select from the menu **Automation > New Automation**.
3. Enter a **Sample Name** in the **Start, Standard** panel, and select the solvent and other options. You can select **Autoplot** to have each spectrum plotted; autolock (**Lock? Yes**) to capture the lock automatically, **Shim** to shim the sample and



Tune to tune the probe if you haven't done it yourself.

4. In the **Experiment Panel**, click the buttons for the desired experiments (some, like NOESY1D will require further customization).
5. Click **Submit**.

The experiments selected in step 4 will have default acquisition parameters that may or may not be suitable for you. Typically, you may need to change at least the number of transients or the spectral width. The experiments may be customized by double clicking the experiment name (in green in the the previous figure at right) in the **Study Queue** vertical panel (not the experiment button in the **Experiment Panel**). The parameter panels for that experiment will open up to allow you to make modifications as usual. Click **Save** on the the row of buttons just above the panel to save any modified parameter and return to the queue entry panel. Also on the **Study Queue** panel, notice the acquisition times for the individual experiments displayed inside brackets to the right of each experiment's name. The total time for the queue is displayed at the bottom of the **Start, Standard** panel.

After each acquisition, the spectrum will be processed and plotted automatically (if requested) and the group of experiments (the queue) will be saved in a folder with a name consisting of the sample name, followed by the date and a sequence number separated by underlines; for example: ealva-1-234-2_20101026_01. Each FID will be saved inside that folder with a name consisting of the sample name followed by the

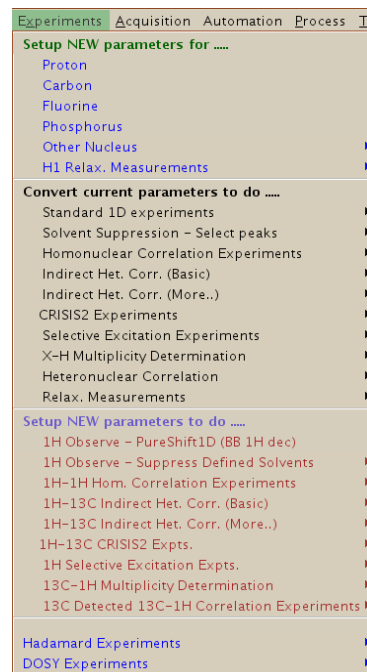
experiment name followed by a sequence number; for example: ealva-1-234-2_HMQC_01.fid. If you need expansions of your spectra, you will have to open each spectrum and plot them manually yourself.

Manual setup

You can easily setup a 2D experiment. If you don't want to optimize parameters, choose the experiment from the menu **Setup NEW parameters to do...** and then click **Acquire** or type go to start the acquisition.

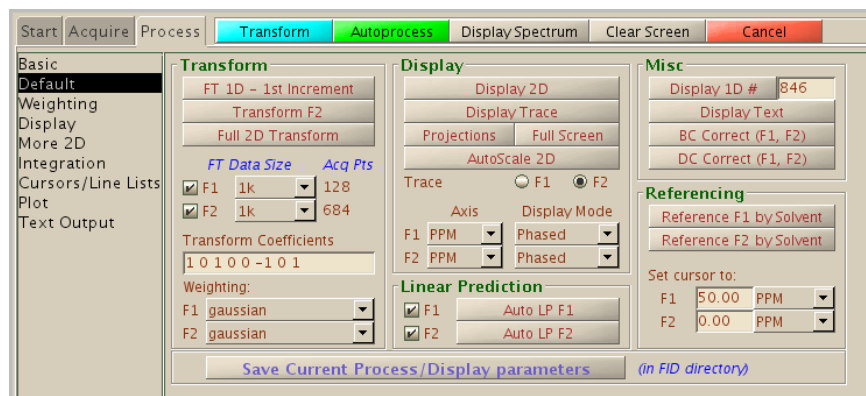
For best results, you should at least reduce the spectral width and inspect/change a few parameters.

- In workspace 1 take a proton spectrum and process it. If you need solvent suppression in your 2D, use an appropriate suppression scheme (WET or PRESAT) and make sure your 1D has enough suppression. The parameters you choose will also be used later in the 2D.
- To eliminate large empty spaces with no peaks on both sides of the spectrum (e.g., above 9 ppm and below 0 ppm) and improve resolution, enclose the actual spectrum inside the two cursors (leave only about 0.5 ppm of empty space on each side), click **MoveSW** and take a new spectrum.
- Copy this spectrum to workspace 3 and go to this workspace: `mf(1,3) jexp3`.
- From the **Experiments** menu, under **Convert current parameters to do...** select the target experiment. The parameters from the customized 1D, like spectral width and solvent suppression will be used in the 2D.
- In the **Acquire, Defaults** panel, inspect and change, if desired, the number of scans, the number of increments, and other relevant parameters.
- Other pulse sequence-specific parameters that may be adjusted appear in the **Acquire, Pulse Sequence** panel. Many of these are already calibrated; if you don't understand what they do, don't change them.
- Some experiments require that you adjust some parameters. For example: for NOESY and ROESY experiments you should adjust the mixing time according to the size of your molecule; for TOCSY experiments you should also adjust the mixing time according to the desired amount of coherence transfer; for ^{19}F - ^{13}C correlations you should check the size of the One-Bond Coupling; for band-selective experiments you should select the spectral width in *F1* and click **Make Shape** to generate the appropriate selective pulses. Check the pulse sequence documentation or the scientific literature before you start.
- Click **Show Time** to display the experimental time. The parameters that most directly determine the experimental time are the number of transients and the number of increments. The former depends on the concentration of your sample; the latter determines the resolution in *F1*. Readjust parameters if needed.
- Click **Acquire** to start the acquisition.



Processing









Most 2D processing tasks can be done from the **Process, Default** panel.








Fourier transformation

Usually 2D spectra are automatically processed after acquisition finishes or after loading the spectrum from the hard disk. To manually process them, click **Full 2D Transform** or select from the menu **Process > Full Process 2D** to do a 2D Fourier transformation with default Weighting and Linear Prediction parameters. Generally, these parameters are appropriate and there is no need to change them, but if you want they can be modified in the **Weighting** and **More 2D** panels. If you get the error “*value for strtext1 is too large*”, click **Auto LP F1** and try again.

Interactive display

- If the 2D spectrum is not already displayed on the screen: click the  button on the vertical toolbar to display it. You will have a cursor that appears as a vertical and a horizontal red line and you can move it around the spectrum by dragging it with the left mouse button just as in a 1D spectrum. If the cursor is not shown, display it with the  button.
- To increase or decrease the intensity of the peaks, use the  and  buttons.
- The number and separation of the contours can be changed in the **Process, Plot** panel as described below (see **Manual Plotting**).
- To expand a region you will need two cursors. Display the second cursor with the  button. The second cursor can be moved by dragging it with the right mouse button. Move the cursors to delineate a box where the left mouse button defines the lower left corner, and the right button, the upper right corner of the region to be zoomed in. The behavior of the cursors may seem a little awkward at the beginning, and some experimentation may be required. In particular, the right cursor cannot move past the left one; and once the two cursors are displayed, the left cursor also moves the position of the right one and cannot modify the size of the box defined by the two cursors. Once the region is selected, click the Zoom In button . Click Show Full Spectrum  to zoom out to the full spectrum or  to zoom out to the previous display.

- Another way to expand a region is by clicking the Zoom Mode button  and dragging the left mouse button over the spectrum. The highlighted region will be immediately expanded when the mouse button is released. To get out of this mode of operation, click the top button in the toolbar.
- You can display a slice of the 2D matrix along the horizontal position of the cursor with the Trace button . If you want a vertical slice, rotate the spectrum along the diagonal first with the Rotate button  and then use the Trace button.
- If a region has been expanded, you can move the spectrum with the Pan and Stretch button .
- The Projections button  will display horizontal or vertical projections on the top and side of the spectrum. These projections are formed from the 2D matrix and are not the same as high resolution projections normally used for plotting. However, when it is not practical to obtain a high resolution 1D spectrum, these projections can be quite useful. For example, you can obtain a ^{15}N “spectrum” much more quickly from a ^1H - ^{15}N HSQC or HMBC than from direct acquisition of a 1D spectrum.

Baseline correction

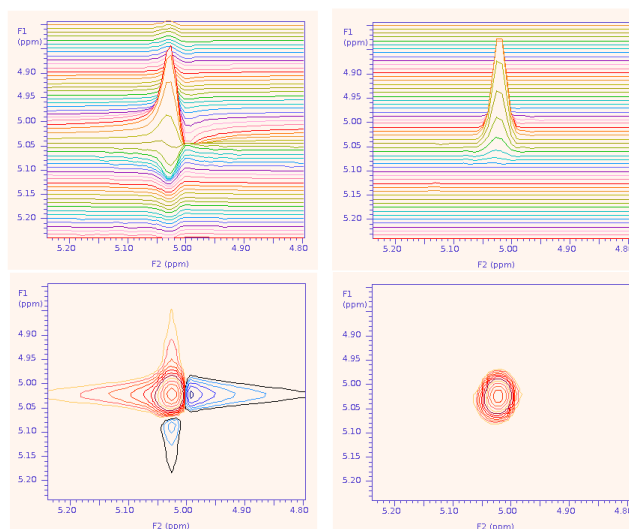
Vnmrj has limited baseline correction tools.

- In the **Default** panel, select **Trace: F2** and click **Transform F2**.
- Type `ds (1)` and proceed to define integral regions as usual.
- In the **Display** panel click **BC F2**.
- In the **Default** panel click **Full 2D Transform**
- If the spectrum is homonuclear, click **BC Correct (F1, F2)**; otherwise, in the **Display** panel click **BC F2**.

Phasing

Some 2D spectra, like COSY and HMBC, are displayed in *absolute mode*. Phase correction is not necessary in these experiments as all peaks have positive intensity.

Most other 2D spectra, however, are collected in *phase-sensitive* mode and they *may* require phase correction. The need for phase correction becomes evident in the spectrum. For example, the figure below shows an out of phase correlation on the left and the properly phased correlation on the right. For a 3D view, the spectra on top are the stacked plots of the contour views below them. Notice how the spectrum on the right is completely positive and looks “clean” and




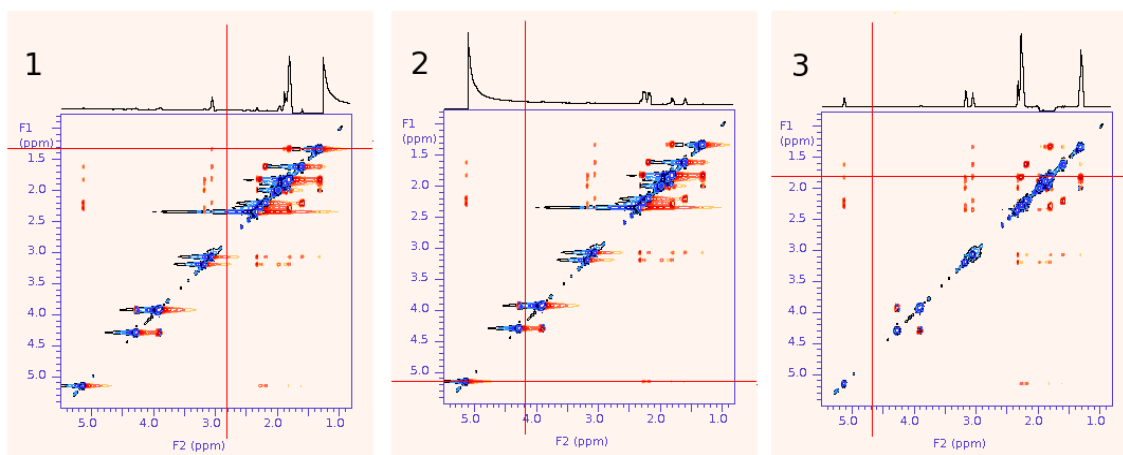
symmetrical, while in the spectrum on the left the peak has both positive and negative components and its contour looks like a star, with horizontal and/or vertical “tails”.




If the acquisition of the 2D spectrum was started from an optimized and properly phased 1D spectrum, it is usually not necessary to phase it or it may only require fine adjustment in F2. However, some experiments, notably NOESY and ROESY, usually require some phase correction in F2 and/or F1.

In Vnmrj, a 2D spectrum is phased by selecting two horizontal traces with peaks (slices of the 2D). One is selected near the top of the spectrum and must contain peaks close to the right side; the other is selected near the bottom and should contain peaks close to the left side. If a correction is also needed in the vertical dimension (usually F1), the 2D matrix must be rotated first so that the vertical dimension is oriented horizontally and the correction can be applied to the horizontal traces.




Follow this procedure to phase a 2D spectrum:

1. Click the **Phase 2D** button .
2. Left-click on the 2D and drag the mouse up until the *horizontal* cursor is located on top of a trace containing a diagonal or a strong signal on the right side of the trace (spectrum 1 in the figure below). The trace is shown on top of the spectrum and its intensity can be changed by dragging the mouse over the trace with the middle button.



3. Click the **Select Spectrum 1** button  (of the two similar-looking buttons on the vertical toolbar, the one on top).
4. Drag the mouse over the spectrum with the left mouse button until the *horizontal* cursor is located over a trace containing a diagonal or a strong signal on its left side (spectrum 2 in the figure).
5. Click the **Select Spectrum 2** button  (the lower button). Note: the icons on the buttons should be interchanged to truly reflect the position of the trace they select; this appears to be a bug in vnmrj.
6. Click the **Phase Selected Spectra** button .
7. The first selected trace is shown. Phase only the right side by clicking over a region close to the right side of the spectrum and dragging the mouse with the left (coarse) or right (fine) buttons. The middle button controls the intensity as usual. Note that in a NOESY spectrum, like the one shown in the figure, the

peaks in the diagonal should be negative, and the cross peaks can be either positive for positive NOEs, or negative for negative NOEs or for correlations due to chemical exchange.

8. Click **Phase Spectrum 2** .
9. The second trace appears on the screen; phase it as follows. First, just click on the right side of the trace but do not drag the mouse (this is done in order to keep the right phase correction previously determined in step 7). Then click on the left side of the trace and drag the mouse to phase the peaks on that side.
10. Click the **Phase Spectrum 1**  button to verify that the first trace is still properly phased and repeat steps 7-9 if necessary.
11. Click the **Return** button  twice. The spectrum should be properly phased (spectrum 3 in the figure: notice the diagonal peaks are all negative or blue and the cross peaks are all positive or red).

Setting the reference

If the 1D spectrum used to start the 2D was properly referenced, the F2 dimension of the 2D should also be properly referenced. If not, read the next paragraphs.

Once F2 is referenced, the F1 dimension can be referenced by typing `reff1`. This is especially useful for heteronuclear spectra when there is no solvent or internal standard peaks available in the F1 dimension, for example in a ^1H - ^{15}N HSQC.

If there is no internal standard or solvent peaks in the F2 dimension (usually ^1H), but the sample was locked, typing `setref reff1` should reference both dimensions using the known deuterium chemical shift of the deuterated solvent used to engage the lock. If the solvent is not deuterated, or if the lock was not engaged, you will need either a solvent peak or an arbitrary peak that can be used as a reference and set its chemical shift manually.

To manually adjust the chemical shift reference:

1. Expand a very small region around the internal standard or chosen reference peak and position the cursor exactly in the center of the contour. It may help to display more contour lines to clearly see the top of the peak; use the **show** buttons in the **Plot** panel.
2. In the **Process, Default** panel, in the **Referencing** box, set the units to PPM in both F1 and F2 and enter the desired chemical shifts in the **Set cursor to:** fields followed by the enter key in both cases.

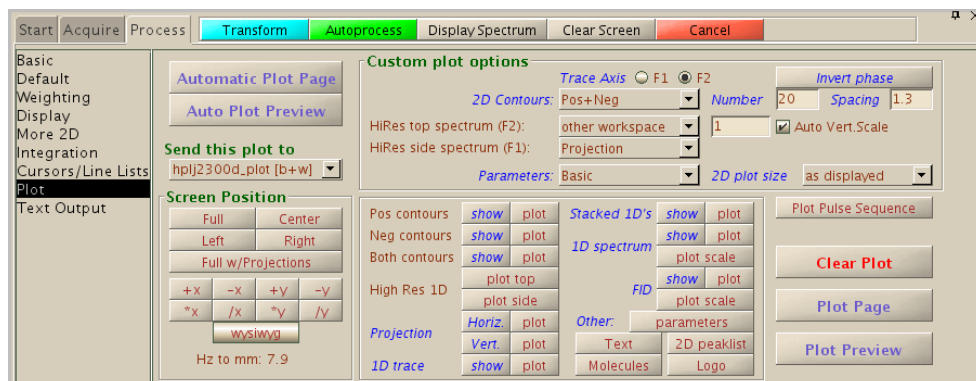
Symmetrization

A homonuclear 2D experiment with a diagonal can be symmetrized to help reduce T1 noise and other artifacts. During symmetrization, the intensity of the most intense point of each pair of points symmetrically located relative to the diagonal is substituted with the intensity of the weakest point. The resulting spectrum is symmetric along the diagonal and artifacts (and weak signals!) appearing only on one side of the diagonal are eliminated.



To apply it, type `foldt`. Be aware that the process may reduce the spectrum's signal to noise ratio.

Plotting

Almost all plotting tasks or operations are done from the **Process, Plot** panel.

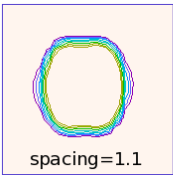


Automatic plotting

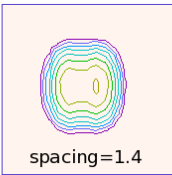
1. Expand the region of interest.
2. Select an intensity just above the noise level with the  and  buttons. Select other **Custom plot options** if desired (see below).
3. Click either **Automatic Plot Page** or **Auto Plot Preview**.

Manual plotting

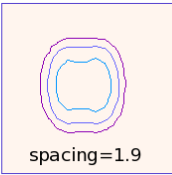
Start by expanding the region of interest and selecting an intensity just above the noise level.

- Select the **Screen Position** for your plot; **Full w/Projections** usually is the best choice. You can also move the position of the plot around the page with the **+x**, **-x**, **+y** and **-y**, buttons and change its size with the ***x**, **/x**, ***y** and **/y** series of buttons.
 - In the **Custom plot options** box, select the **Number** and **Spacing** of the contours. The spacing is a number (must be higher than 1.0) that determines how the contours are separated from each other. For example, with a spacing of 1.1 all the contours may be closely spaced at the base of the peaks, usually appearing as “hollow” peaks in the plot; with a spacing of 1.9 the contours may be more separated but weak peaks may be traced by only a few contours. Choose a number around 1.2-2.0 such that all peaks have contours going from the base to the tip. Expand a small region and click the **show** button on the right side of **Both contours** to display them on the screen.
- 

spacing=1.1



spacing=1.4



spacing=1.9
- **HiRes top spectrum** and **HiRes side spectrum** allow you to choose the spectra to be plotted on top and side of the 2D. Choose from **Projection** to use a low resolution projection of the 2D; **other workspace** to use a high resolution spectrum loaded in another workspace and enter the workspace number and select **Auto Vertical Scale**; and **None** to plot nothing. Since we already have a

proton spectrum in workspace 1, select **other workspace** for the top spectrum (F2) and enter the number 1 in the field that opens up. You can manually adjust the desired intensity in workspace 1 or select **Auto Vert. Scale** to let vnmrj adjust it for you. Proceed similarly for the side spectrum, selecting again workspace 1 if your 2D is homonuclear or 2 (if you have a carbon spectrum available there) if it is heteronuclear.

- The group of buttons on the bottom part of the panel: **show** and **plot**, allow you to display the plot's elements on the screen or to plot them. For example, for a typical 2D plot you would click the **plot** buttons for **Both contours, High Res 1D (plot top and plot side)**, **Projection (Horiz. and Vert.)** and **Other: parameters**.
- After all the elements have been plotted, you can either click **Plot Page** to send the plot to the printer; click **Plot Preview** to preview the plot as a pdf page; or type `UMmailpage` on the command line to have the plot sent to you via email as a high resolution image (png and eps formats) ready to be pasted into word processing and presentation software.

To plot in color, load or create a color Theme for postscript printers in the *Styles and Themes* dialog box. This box is opened when you select the menu **Edit > Display Options**. The procedure is briefly explained in the *Vnmrj Training Guide* and in the *Frequently Asked Questions* writeup.

Experiment selection and optimization guidelines

For all experiments, it is recommended to optimize the spectral width in both F1 and F2. Choose an appropriate number of scans and number of increments. When using a non-gradient experiment, use at least 8 scans to remove artifacts. Parameters that usually need to be adjusted are found in the **Acquire, Defaults** panel. Other less common parameters appear in the **Pulse Sequence** panel.

Homonuclear correlations

Name	Remarks	Parameters commonly adjusted
COSY, gCOSY	Classical correlation experiments. Absolute mode. Very easy to run. Variations are possible: COSY-45, COSY-LR. Default time about 5 min.	
qDQCOSY, DQCOSY	Double Quantum filtered Correlation Spectroscopy. Phase sensitive. Suppresses singlets in the diagonal. Less sensitive than COSY but higher resolution. Measurement and analysis of coupling constants is possible.	
NOESY	Dipolar (through space) correlation experiments. Phase sensitive. Diagonal peaks are negative, positive cross peaks are positive NOEs, negative cross peaks are negative NOEs or chemical exchange correlations. COSY-type artifacts may appear between coupled protons as antiphase cross peaks.	Mixing time can be 0.1 s to 0.3 s for large molecules or 0.3 s to 0.8 s for small molecules.

Name	Remarks	Parameters commonly adjusted
ROESY, ROESYAD	Rotating Frame Overhauser spectroscopy. Phase sensitive. Diagonal peaks are negative, all direct NOEs are positive cross peaks, indirect NOEs are negative and chemical exchange correlations appear as negative cross peaks. TOCSY-type artifacts may appear as antiphase cross peaks. Use ROESY instead of NOESY when the molecular weight is above 1000.	Mixing time can be 0.1 s to 0.3 s for large molecules or 0.3 s to 0.8 s for small molecules.
TOCSY, zTOCSY	Total Correlation Spectroscopy. Phase sensitive. Correlates a proton to all other protons in a spin system. Very useful to see individual residues in peptides, oligosaccharides, and some other organic compounds. The zTOCSY variant uses a z-filter to eliminate phase distortions that sometimes appear in the cross peaks.	Mixing time 30 - 500 ms.

Heteronuclear correlations

Name	Remarks	Parameters commonly adjusted
ASAPHMQC	As Soon As Possible HMQC. If you have at least 5 mg of sample, you will get a good HMQC in about 1 minute.	
HSQC, gHSQC, HSQCAD, gHSQCAD, HMQC, gHMQC	Heteronuclear single bond correlation. Phase sensitive. Cross peaks in HSQC are multiplicity edited by default; CH and CH ₃ are positive and CH ₂ are negative, so you get the equivalent information of a DEPT experiment at the same time. Can also be used to obtain ¹⁵ N chemical shifts at natural abundance. HSQC gives higher resolution and is preferred over HMQC. Default time ~15 min.	The average value of the one bond coupling constant can be optimized if necessary. The default is 140 Hz.
HMBC, gHMBC, gHMBCAD	Heteronuclear Multiple Bond Correlation. Phase sensitive. Shows correlations through 2 and 3 bonds (C-C-H) and (C-C-C-H). Useful for assignment of quaternary carbons or tertiary amines. Default time ~40 min.	The average value of the 2,3 bond coupling constant can be optimized. The default is 8 Hz.
CIGAR, CIGARAD	Heteronuclear multiple bond correlation. Phase sensitive. Attempts to extract more long range correlations than HMBC over a range of coupling constants. Preferred over HMBC. Default time ~ 40 min. Can discriminate between correlations due to 2 bonds and those due to 3 bonds.	The value of the 2,3 bond coupling constant will be changed between two values. The default is between 5 and 8 Hz.

Name	Remarks	Parameters commonly adjusted
gH2BC, gH2BCAD	Heteronuclear 2-bond correlation. H2BC almost exclusively correlates protons and proton-bearing carbons separated by only two bonds. It solves the problem of distinguishing two- and three-bond correlations in HMBC and HSQC-TOCSY and is independent of the heteronuclear two-bond coupling constant. Correlations to quaternary carbons do not appear.	
bsHSQCAD, bsgHSQCAD, bsgHMBC, bsTOCSY, bsNOESY, bsROESY	Band-selective experiments. Useful to get high resolution over a small region in F1 without other peaks folding over the region.	Select a narrow C13 Spectral Width (Acquire, Defaults panel) and click Make Shape before starting the acquisition.
HSQCADTOCSY, gHSQCADTOCSY, HSQCTOCSY, gSHQCTOCSY, HMQCTOCSY, gHMQCTOCSY	Heteronuclear TOCSYs. Heteronuclear multiple bond correlation with tocsy coherence transfer. Provides correlations from a carbon to several other hydrogens along a chain (CH-CH-CH-CH). Useful for interpretation of crowded proton spectra.	Same as HSQC and TOCSY. Long values for Spinlock Duration (mixing time) produce more extended correlations along aliphatic chains.
EXSIDE	Can be used to determine the value of two- or three-bond C-H coupling constants.	
c2hsqc, gc2hsqc, c2hsqcse, gc2hsqcse, gc2hmabc, gc2h2bc, gc2h2bcme, gc2hmabcme	CRISIS 2 experiments. They have slightly better sensitivity and lineshape than other experiments. Names ending in <i>me</i> are <i>multiplicity-edited</i> : cross peaks due to CHs and CH ₃ s have different signs than CH ₂ s. Those ending in <i>se</i> are sensitivity-enhanced.	Same as non-crisis experiments.
HETCOR, COLOC	Heteronuclear correlation. Uses direct ¹³ C detection and it is therefore much less sensitive than newer methods. Obsolete, do not use unless you have a very good reason.	

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