Abstract

VnmrJ 3 Software provides easy-to-use, interactive tools for setting up advanced experiments. This allows even novice users to get critical information about their research samples using the most advanced NMR experiments available. This application note is just one of a series designed to provide step-by-step guidance for setting up sophisticated experiments to collect exactly the data you need for your analyses.
Introduction

The long-range connectivity data provided by the heteronuclear multiple bond correlation (HMBC) experiment are a fundamental building block for most structure elucidation problems. As each carbon atom in a given molecule can generate numerous responses, this experiment creates a spectrum that is very information-dense. In regions where the carbon spectrum is congested this can cause serious spectral overlap, leading to ambiguous structural assignments.

One way to decrease the number of responses, and thereby reduce the chances of spectral overlap, is to use multiplicity as a spectral editing technique. When using the multiplicity-editing technique, the user can choose to suppress those responses arising from carbon atoms bearing either an odd number of protons (i.e., methyl and methine carbons) or an even number of protons (i.e., methylene and quaternary carbons). The editing is accomplished by collecting two arrayed sets of spectra and processing them together to selectively remove the unwanted responses.

Multiplicity-editing in HMBC has two major drawbacks. First, the multiplicity-editing feature relies on the compensation of refocusing inefficiency with synchronized inversion sweep (CRISIS) condition as the fundamental mechanism behind the editing process. This technique is based on a hypothetical relationship between the one-bond $J_{CH}$ coupling constant and carbon chemical shift. Carbons with coupling constants that deviate significantly from this relationship will have imperfect editing. Second, since the experiment is obtained as an array of two spectra, it takes twice as long to acquire as a comparable nonedited data set.

A Multiplicity-Edited HMBC Example

As an example of how multiplicity-editing can improve the effective resolution of an HMBC spectrum, a representative gradient-selected heteronuclear multiple bond correlation, multiplicity-edited, adiabatic (gHMBCmeAD) data set was acquired on paclitaxel (Figure 1).

![Chemical structure of paclitaxel](image-url)
The portion of the spectrum displayed in Figure 2 demonstrates how the data can be processed to yield three different results. The center panel has been processed to retain all the responses, which is equivalent to a standard HMBC data set. When the data are processed to remove the responses from methylene and quaternary carbons, the resulting spectrum is less crowded (bottom panel). The top panel shows the data set processed to remove responses from methyl and methine carbons, rendering the assignment of the three ipso carbon resonances much more facile.

Experimental Method

The multiplicity-edited HMBC experiment is easy to set up in VnmrJ 3 using the following steps:

1. Select the gHMBCmeAD protocol from the Experiment Selector under the Jn(CH)corr tab. The experiment can be run as a new study, a continuation of an existing study, or in foreground (Figure 3).

2. The default parameters include the required parameter array to collect multiplicity-edited data. The other parameters shown on the Default panel of the Acquire tab are similar to the parameters for a standard HMBC.

3. Once the data set is complete, the separate edited spectra are created by differentially processing the data. Software buttons to perform the editing during processing can be found on the Default processing panel under the Process tab (Figure 4).

Figure 2. An expansion of the multiplicity-edited HMBC spectrum of paclitaxel processed to show responses from all carbons (center), CH₂ and CH carbons (bottom), and CH₃ and quaternary carbons (top).
Conclusions

The multiplicity-edited HMBC experiment can provide data that are significantly simpler to interpret than a standard HMBC data set by removing responses from various carbon atoms. This is particularly valuable in spectra where some regions are highly congested.

References

