

# Reducing the Measurement Time of DEPT NMR Experiments

Julien Furrer<sup>a</sup>, Sebastiano Guerra<sup>b</sup>, Robert Deschenaux<sup>b</sup>

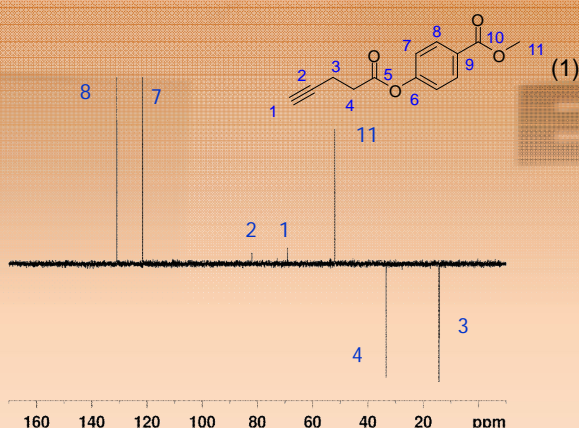
<sup>a</sup>Service Analytique Facultaire, Institut de Chimie, Université de Neuchâtel, Avenue de Bellevaux 51, CH-2009 Neuchâtel, Switzerland

<sup>b</sup>Institut de Chimie, Université de Neuchâtel, Avenue de Bellevaux 51, Case Postale 158, CH-2009 Neuchâtel, Switzerland

## Introduction

NMR spectroscopy is one of the techniques of choice for analyzing molecules at an atomic level. The DEPT experiment, which provides edited <sup>13</sup>C spectra based on the number of attached protons, is probably the most useful NMR experiment for initial analysis. The final <sup>13</sup>C signal intensity of DEPT experiments is strongly influenced by the magnitude of the one-bond coupling <sup>1</sup>J<sub>CH</sub>. If a molecule contains several functional groups with different one-bond coupling constants, maximum <sup>13</sup>C signal intensity cannot be obtained for all functional groups simultaneously, because DEPT experiments are typically recorded using a nominal (average) value for <sup>1</sup>J<sub>CH</sub>.

## Illustration



As an illustrative example, a DEPT135 spectrum of (1), optimized for <sup>1</sup>J<sub>CH</sub> = 145 Hz, is shown above. A first inspection of that DEPT spectrum can be misleading. Indeed, the signals emerge with good signal to noise ratio and it seems that all expected resonances are obvious. After some rudimentary analysis, however, it turns out that the terminal acetylenic carbon C-1, appears as a very weak positive signal around 70 ppm.

## Theoretical background

The effectiveness of polarization transfer I and thus the final <sup>13</sup>C intensity in DEPT spectra is directly influenced by the magnitude of the <sup>1</sup>J<sub>CH</sub> coupling constant and the duration of the precession periods (Δ). The intensity of the final <sup>13</sup>C resonance can be approximated as following:

$$I_{DEPT} \propto \sin^2(\pi \Delta^1 J_{CH}) \quad \text{Equation 1}$$

Equation 1 shows that for molecular systems containing several CH fragments with very different <sup>1</sup>J<sub>CH</sub> coupling constants, the proper choice of the delay Δ becomes tricky. In molecule (1), the <sup>1</sup>J<sub>CH</sub> coupling constants range from 138 Hz (<sup>1</sup>J<sub>C11H11</sub>) to 250 Hz (<sup>1</sup>J<sub>C1H1</sub>). Maximum polarization transfer cannot be achieved for all CH fragments simultaneously because Δ is set to an average <sup>1</sup>J<sub>CH</sub> coupling value. As shown by the spectra 1-3, significant intensity losses results from these compromises.

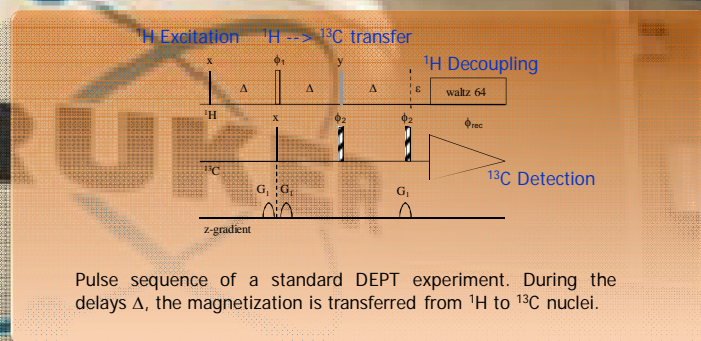
- If Δ is optimized for a <sup>1</sup>J<sub>CH</sub> coupling of 145 Hz, equation 1 and spectrum 1 reveal that the C3, C4 and C11 carbons appear with maximal intensity, while C1 is hardly visible.
- If Δ is optimized for a <sup>1</sup>J<sub>CH</sub> coupling of 200 Hz, equation 1 and spectrum 2 reveal that the C3, C4 and C11 carbons appear with reduced intensity, while C1 is clearly visible
- If Δ is optimized for a <sup>1</sup>J<sub>CH</sub> coupling of 250 Hz, equation 1 and spectrum 3 reveal that the C3, C4 and C11 carbons are hardly visible, while C1 appears with maximal intensity.

## Conclusion

Using accordion optimization, we have shown that the experimental time for obtaining DEPT spectra can be substantially reduced. Thus, our proposed DEPT experiment is a valuable alternative to standard DEPT experiments for molecular systems containing several CH fragments with very different <sup>1</sup>J<sub>CH</sub> coupling constants. In addition, the experiment we propose does not require any supplementary calibration procedures and can be used as it is starting from a standard parameter set by inexperienced users and under automation conditions. These features make the accordion optimized DEPT experiment a very promising tool in NMR spectroscopy of molecules containing a large range of <sup>1</sup>J<sub>CH</sub> coupling constants.

## Reference

(1) Bodenhausen, G.; Ernst, R. R. *J. Am. Chem. Soc.* 1982, 104, 1304.



Pulse sequence of a standard DEPT experiment. During the delays Δ, the magnetization is transferred from <sup>1</sup>H to <sup>13</sup>C nuclei.

## Our solution

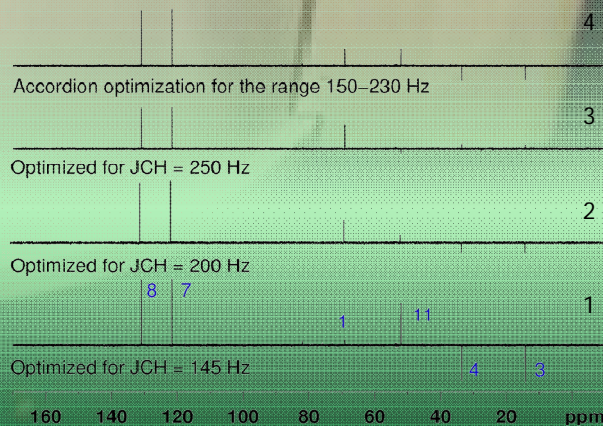
The solution we propose takes advantage of the so-called accordion-optimization<sup>1</sup>. Instead of recording the DEPT experiment using one single <sup>1</sup>J<sub>CH</sub> coupling constant value for all transients, this value is changed after each transient. The sum of all transients results in a DEPT spectrum with equalized intensities over wide range of one-bond coupling constants. The intensity is given by:

$$I_{Acc} \propto \frac{1}{n} \sum_{i=1}^n \sin^2(\pi \Delta_i J_{CH})$$

where n is the number of values used for sampling the chosen coupling constant range and Δ<sub>i</sub> the evolution delay of the i<sup>th</sup> transient.

As a proof of concept, this strategy has been applied to compound (1). The Δ value has been optimized for a [150-230 Hz] <sup>1</sup>J<sub>CH</sub> coupling range. The resulting ACCORD-DEPT spectra (4) display all expected resonances but require much less spectrometer time than conventional DEPT experiments.

## Experimental validation



## Acknowledgment

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