

Modified ^{13}C DEPT Experiments for Facilitating the NMR Analysis of Dendrimers. 2. ACCORD-DEPT

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NMR spectroscopy is obviously one of the techniques of choice for analyzing dendrimers at an atomic level. The DEPT experiment, which provides edited ^{13}C spectra based on the number of attached protons, is probably the most useful NMR experiment for chemists involved in dendrimer synthesis. The effectiveness of polarization transfer from ^1H nuclei to ^{13}C nuclei and therefore the final ^{13}C signal intensity of DEPT experiments is strongly influenced by the magnitude of the one-bond coupling $^1J_{\text{CH}}$. If a molecule contains several functional groups with different one-bond coupling constants, maximum ^{13}C signal intensity cannot be obtained for all functional groups simultaneously, because DEPT experiments are typically recorded using a nominal (average) value for $^1J_{\text{CH}}$. The risk that some resonances are missed increases, particularly when the amount of sample available is limited. High-molecular weight dendrimers may contain several functional groups and typically very low amount of sample can be dissolved for NMR purposes. For such dendrimers, recording NMR experiments like DEPT may become challenging, even with high-field spectrometer and probehead of state-of-the-art technology.

In this contribution, we present a modified DEPT NMR experiment to tackle this problem. The ACCORD-DEPT experiment we propose takes advantage of the so-called accordion-optimization¹. Accordion optimization partly equalizes polarization transfer from ^1H to ^{13}C nuclei over wide range of one-bond coupling constants. As a proof of concept, this strategy has been applied to high-molecular weight dendrimers containing a large range of one-bond coupling constants. The resulting ACCORD-DEPT spectra display all expected resonances but require much less spectrometer time than conventional DEPT experiments.

References

- (1) Bodenhausen, G.; Ernst, R. R. *J. Am. Chem. Soc.* **1982**, *104*, 1304-1309.