Scalar Overhauser Dynamic Nuclear Polarization at 14.1 T

Dubroca, T., van Tol, J., Trociewitz, B., Wi, S. (NHMFL); Frydman, L. (Weizmann Institute, Israel, Chemistry, and NHMFL); Hill, S. (FSU, Physics, and NHMFL)

Introduction

Nuclear Magnetic Resonance (NMR) is an important technique to quantify and study a large range of chemicals with applications in metabolomics, natural products, and pharmaceuticals. Dynamic Nuclear Polarization (DNP) can dramatically increase the intrinsic low sensitivity of NMR. In the liquid state, DNP is achieved via Overhauser mechanisms, scalar and/or dipolar, which have complex dependences on the experimental conditions. In DNP, the sample is mixed with organic radicals containing unpaired electron spins. These spins are driven out of equilibrium by irradiating the sample with microwaves matching the electron Larmor frequency at the associated magnetic field. Through electron-nuclear cross-relaxation processes, the initial large electron polarization is transferred to the nuclei of interest, which can lead to dramatic increases in the observed NMR signal. Improving the sensitivity of NMR, or polarization enhancement, by DNP in liquids is more challenging with increasing magnetic field. Our group has developed a new method for performing DNP at high magnetic field (14.1 T) via the scalar Overhauser effect.

Experimental

^{13}C labeled model compounds CCl_4 and $CHCl_3$ NMR spectra were collected with and without microwaves, see **Fig.1**. The newly completed Overhauser liquid DNP spectrometer part of the NHMFL EMR Facility operating at 14.1 T was used to perform these experiments.

Results

Here, we illustrate our recent instrumentation development progress by showing enhancements of 160 for ^{31}P [1] and 70 for ^{13}C nuclei at room temperature in large sample volumes (100 µL) that were obtained using a 395 GHz gyrotron at 14.1 T (1H 600 MHz) while maintaining the highest resolution (~0.1 ppm), see **Fig.1**.

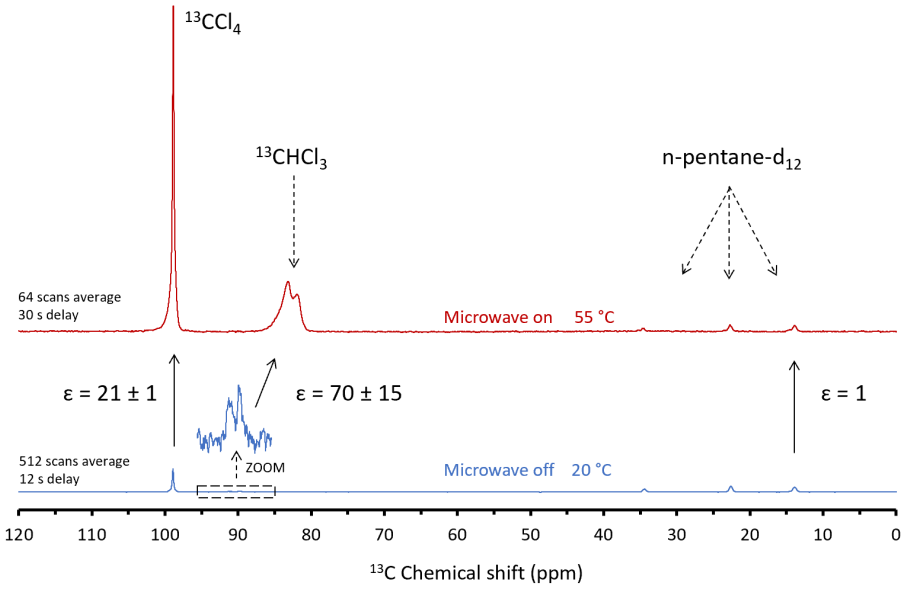

Fig.1 ^{13}C NMR spectra of labeled CCl_4 and $CHCl_3$ measured at 14.1 T without (blue trace) and with microwaves (red trace). The CCl_4 linewidth remained essentially identical at 0.1 ppm. The natural abundance ^{13}C peaks from the solvent, n-pentane-d_{12}, are also visible. Sample information: $^{13}CCl_4$ (9% vol.) $^{13}CHCl_3$ (1% vol.) in n-pentane-d_{12} with 10 mM TEMPO. Microwave power: 13 W.

Acknowledgements

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References

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