Magic Angle Spinning Dynamic Nuclear Polarization Instrument

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Introduction

The Dynamic Nuclear Polarization ad hoc group at the Maglab has developed a new characterization tool, a Magic Angle Spinning Dynamic Nuclear Polarization (MAS DNP) spectrometer at high field (14.1 T / 600 MHz) to study molecules in the solid state. DNP is a novel approach to nuclear magnetic resonance (NMR). Sensitivity is increased up to 2 orders of magnitude compared to traditional NMR, which leads to additional information for molecules being studied. We have designed, assembled and currently operate a MAS DNP spectrometer. This new spectrometer is now part of the user program.

Instrument Development

The MAS DNP spectrometer is a customized version of the commercially available MAS DNP Bruker system. The customization involved a new approach to the microwave transmission as well as use of an existing NMR magnet. This resulted in very large savings compared to the purchase of a complete brand new system. This instrument development also takes advantage of an existing high power microwave source, i.e. gyrotron, which is now shared between the MAS DNP spectrometer and the Overhauser DNP instrumental effort.
In short, microwaves are used to polarize radicals in a solid solution. The resulting polarized electron spins of these radicals are in turn polarizing nuclear spins in molecules of interest via the cross or solid effect. This method increases the number of polarized nuclear spins tremendously. Molecular characterization using this technique results in very high sensitivity.

Results and Conclusions

Upon installation, a standard proline sample mixed with AMUPol (the microwave sensitive polarizing agent) was used to characterize the instrument itself. A 74 fold enhancement was observed (Figure 1). This is in line with the Bruker commercial DNP instrument.

Acknowledgements

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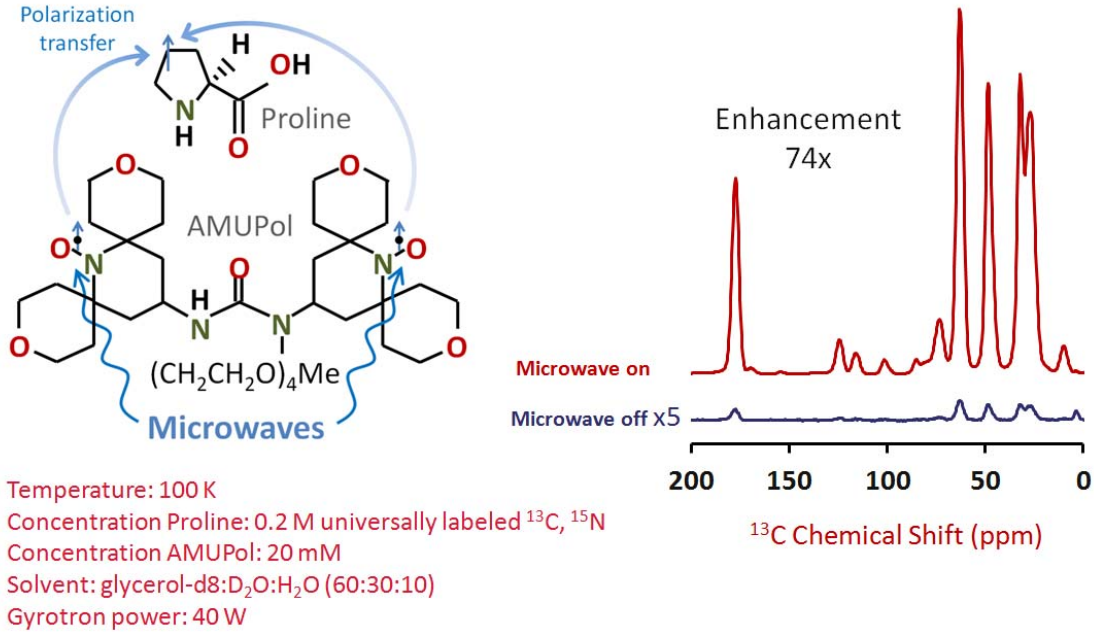

Fig.1 DNP cross effect schematic on proline (left). NMR spectra of standard proline with and without microwaves (right).