\(^{13}\text{C}/^{14}\text{N} \text{ DISTANCE MEASUREMENT IN NATURAL ABUNDANT SOLIDS USING R-RESPDOR NMR}\)

Z. Gan (NHMFL)

**Introduction**

The capability of measuring internuclear distance has made solid-state NMR a powerful tool for structural characterization. This report presents a robust solid state NMR method for simultaneously measuring multiple \(^{13}\text{C}/^{14}\text{N} \) distances in natural abundant solids. \(^{14}\text{N} \) is a highly abundant (99.6\%) spin-1 quadrupolar nucleus and its distances with surrounding carbons provide useful information for molecular structure and spectral assignment. The rotary resonance echo saturation pulse double resonance (R-RESPDOR) method described here introduces two important changes to the rotational echo adiabatic passage double resonance (REAPDOR) [1] previously developed for measuring distances between a spin-1/2 and a quadrupolar spin. First, it uses a simple recoupling scheme called rotary resonance which is susceptible to spinning frequency fluctuation. Second, it uses saturation pulse to induce \(^{13}\text{C}/^{14}\text{N} \) dipolar dephasing which not only achieves efficient dipolar dephasing but also smoothes out the orientation and other parameter dependences allowing simple data interpretation [2].

**Experimental**

The \(^{13}\text{C}/^{14}\text{N}/^{1}\text{H} \) triple-resonance experiment was performed on a Bruker DRX-600 spectrometer at the NHMFL with a 4mm magic-angle spinning probe.

**Results and Discussion**

Figure 1 shows the R-RESPDOR pulse sequence and the \(^{13}\text{C}/^{14}\text{N} \) dipolar coupling measurement using L-tryptophan as a model compound. \(^{13}\text{C}/^{14}\text{N} \) dipolar dephasing curves between the ring nitrogen and eight aromatic carbons are measured simultaneously from a single R-RESPDOR experiment. Figure 1 also shows the simulations using dipolar coupling frequencies obtained from a separate \(^{13}\text{C}/^{15}\text{N} \) REDOR. With the saturation pulse, the universal R-RESPDOR function depends on only the product of dipolar coupling frequency \(D\) and dephasing time \(\tau\).

\[
\Delta S / S_0 = \frac{4}{9}[1 - S_0(\frac{\pi}{4}Dr)] + \frac{2}{9}[1 - S_0(\frac{\pi}{2}Dr)]
\]  

[1]

The listed dipolar coupling frequencies can lead to carbon/nitrogen distances ranging from one to four chemical bonds.

**Conclusions**

The capability of measuring multiple \(^{13}\text{C}/^{14}\text{N} \) distances simultaneously at natural abundance makes R-RESPDOR a potentially useful tool for spectral assignment and structural analysis of small to medium size molecules without the need of isotope labeling.

**Acknowledgements**

This work has been supported by the National High Magnetic Field Laboratory and National Science Foundation through Cooperative Agreement (DMR-0084173)

**References**