NUCLEAR MAGNETIC RESONANCE THERMOMETRY FOR ALIGNED PROTEINS IN LIPID BILAYERS

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RF heating of protein samples is a well known problem in high frequency solid-state NMR [1]. For magic angle spinning experiments with narrow linewidths, NMR chemical shift thermometry (CST) has served as a valuable means to monitor sample temperature [2]. However, CST has not been successfully applied to static uniformly aligned membranes. The recently introduced compound TmDOTP [3] appears to be compatible with these samples and to have a chemical shift that is sufficiently sensitive to temperature to overcome the inhomogeneous broadening inherent in aligned membrane samples. Preliminary experiments have been carried out to evaluate TmDOTP as a practical CST.

To evaluate TmDOTP as a chemical shift thermometer, a small amount of TmDOTP was added to the constituents of a phospholipid bilayer sample. The sample was prepared in the standard way but without a protein. The sample was spread onto 20 glass slides, dried, stacked, hydrated and enclosed in a wax-sealed rectangular glass cell. Both the ^{1}H and ^{31}P resonances of TmDOTP are reported to have a strong dependence of chemical shift on temperature [3]. For these tests, the ^{31}P resonance was observed. The experiments were carried out on a Bruker DRX 400 spectrometer running at 400.487 MHz ^{1}H Larmor frequency. The sample temperature was regulated using the standard spectrometer temperature controller. For each temperature, 512 transients were accumulated. In the bilayer sample, the ^{31}P resonance of TmDOTP was measured to have a full width at half maximum of 7-8 ppm. Fig. 1 shows that the ^{31}P chemical shift in TmDOTP varies linearly with temperature between 23°C and 38°C at 3.0 ppm/°C. A value of 2.18 had been previously reported for a solution of TmDOTP in D_2O [3] at 7.05 T.

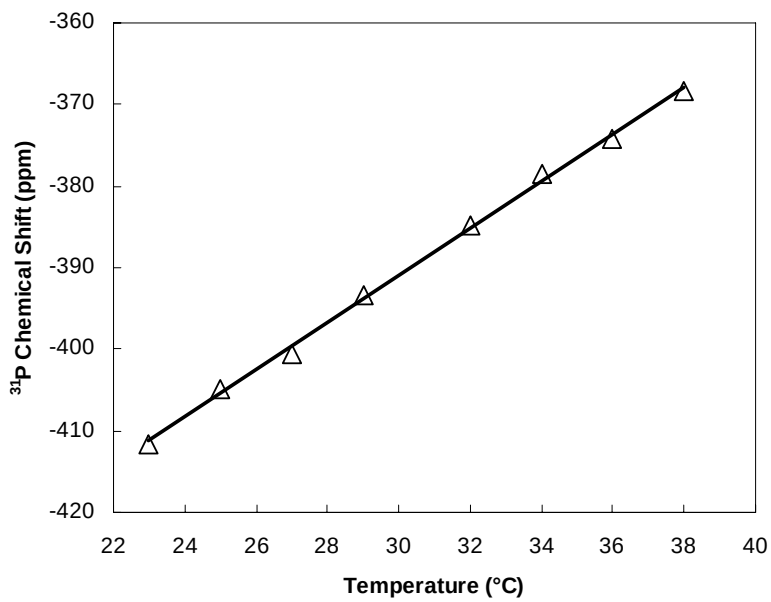

Figure 1. TmDOTP ^{31}P chemical shift as a function of temperature

This project has demonstrated that TmDOTP can be used to measure temperature changes in uniformly aligned membrane samples to a sufficient precision to evaluate the level of RF heating.

References

[1] Li, C. *et al.*, J. Magn. Reson., **180**, (2006) 51-57.
[2] Stringer, J.A., *et al.*, J. Magn. Reson. **173** (2005) 40-48.
[3] C. S. Zuo, *et al.*, J. Magn. Reson. **133** (1998) 53-60.

Acknowledgement

JMJ was an REU student at the NHMFL in Summer 2006.
The research was supported in part by MCB 0235774