Analysis of RF Heating and Sample Stability in Aligned Static Solid-State NMR Spectroscopy

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One prominent problem in the solid State NMR experiment for membrane protein is RF heating, which can induce 40 °C above the cooling air temperature in some sample and experiment conditions. In order to find the origin of the heating effect and optimize sample condition then minimize the RF heating effect in our NMR experiment, RF heating effect under different sample conditions were investigated.

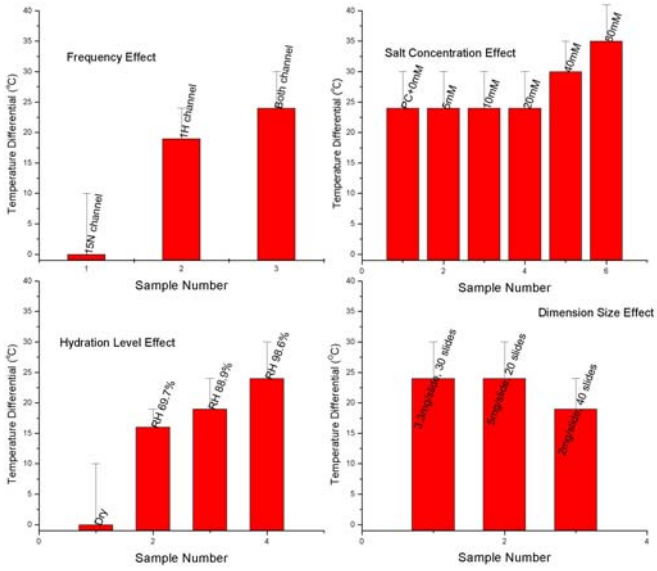

Figure 1: Experimental condition used for this experiment is: CP contact time is 1ms, acquisition time is 7ms, duty cycle delay is 2s. Decoupling power is 50KHz. Air flow rate is ~4L/Min at a temperature of 30 °C. Sample is DOPC/DOPG=9/1, 3.3mg/slide, 33 slides together. (Top) Sample heating frequency effect. (Middle) Influence of the salt concentration in the dialysis buffer on sample heating. (Below) Influence of the hydration level on sample heating. The error bar is the value between the hole that has become black and the neighboring hole that has not become black. All experiments were carried out on a Bruker DRX 400 spectrometer at a ^{15}N Larmor frequency 40.596MHz and ^{1}H Larmor frequency 400.533MHz. A home built double resonance probe was used.

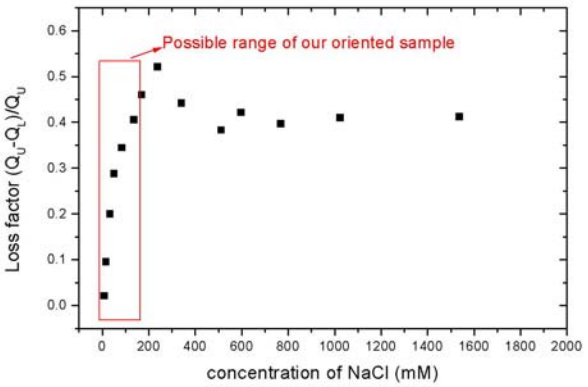

Figure 2: Loss factor as a function of concentration of sodium chloride.6 turn coil is used.

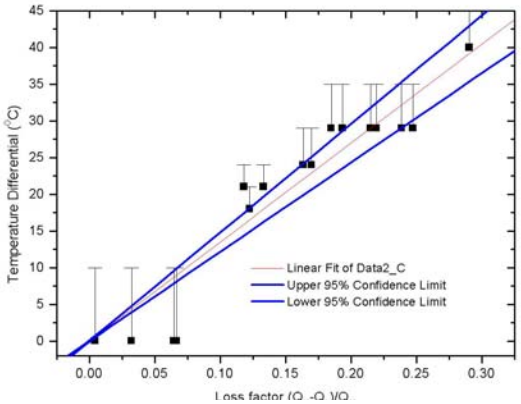

Figure 3: Temperature rise in the sample as a function of loss factor of the samples that were used to measure the equilibrium temperature at various sample conditions

Our experiments shown that the heat induced by the RF, in some sample conditions, can be very intensive and the main contribution of the RF heating comes from dielectric loss in our sample conditions. Water content not the salt concentration played a primary role in the RF heating during PISEMA experiment. A way to estimate the equilibrium temperature from loss factor and the temperature measurement method in situ for oriented static samples was also provided. The main reason leading to high temperature of the sample during PISEMA experiment is the poor heat transfer properties of the air gap, lipid bilayer and square glass tube.