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Supplemental Information

MAS ^{1}H NMR Probes Freezing Point Depression of Water and Liquid-Gel Phase Transitions in Liposomes

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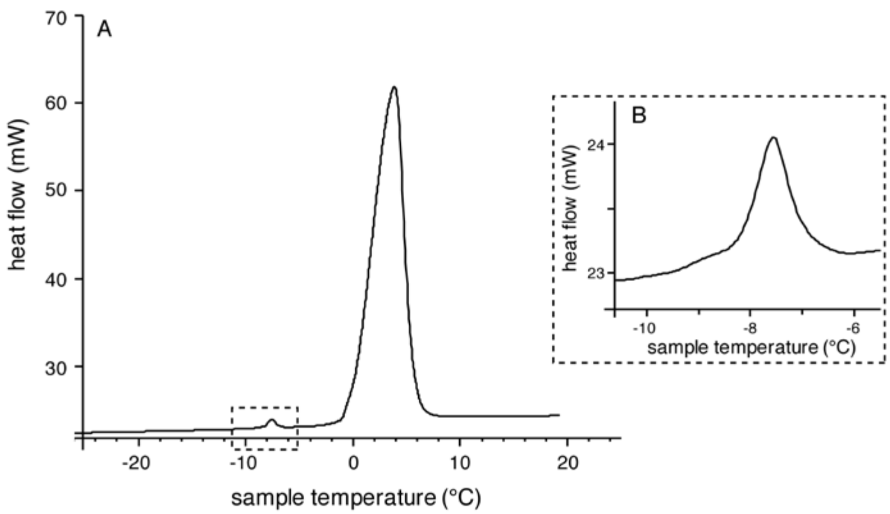

Figure S1 – Differential scanning calorimetry (DSC) of hydrated TOCL. (A) TOCL MLVs, hydrated in excess water, were submitted to DSC measurements using a scanning rate of 2 °C/min. Panel (A) shows the complete heating scan, which includes the TOCL melting transition T_m (boxed) as well as the melting of the water solvent. (B) Enlargement of the lipid transition, showing the TOCL T_m at -8.4 °C. We were unable to locate a prior report of the T_m of TOCL, but note that prior reports on other CL species showed a similar pattern of a CL T_m that is higher than the T_m of the corresponding PC variant (i.e. DOPC in this case) (1, 2).

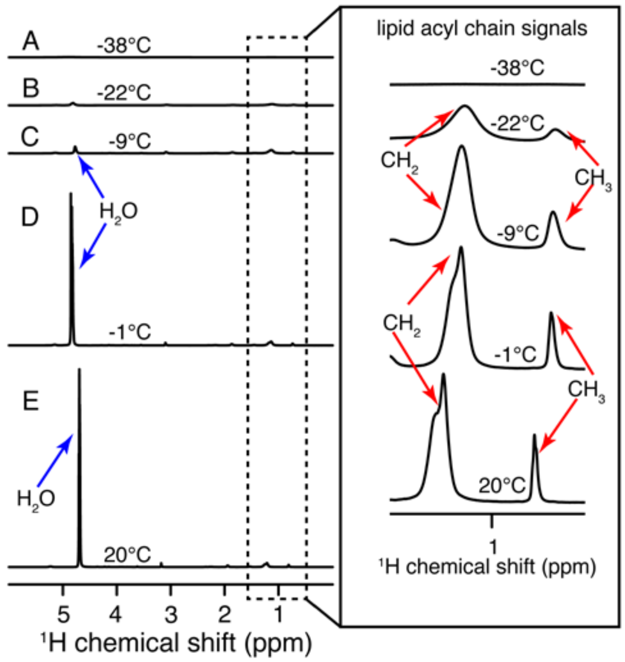

Figure S2 – Heating runs for MAS ^{1}H NMR analysis of DOPC/TOCL MLVs. (A) At -38 °C all ^{1}H peaks are broadened beyond detection. (B) At -22 °C: the water shows up as a low intensity, very broad peak (~4.8 ppm), indicating that most of the water is frozen. Lipid CH_2 and CH_3 peaks become visible but are still strongly attenuated. (C) At -9 °C: The water signal is still attenuated, but lipid acyl chain signals have returned to almost full intensity. (D) At -1 °C: Both water and lipid peaks are now narrow and intense indicating liquid-like motion. (E) At room temperature the peaks remain narrow. The inset to the right shows enlarged versions of the lipid acyl chain peaks. The measurements were performed at 750 MHz.

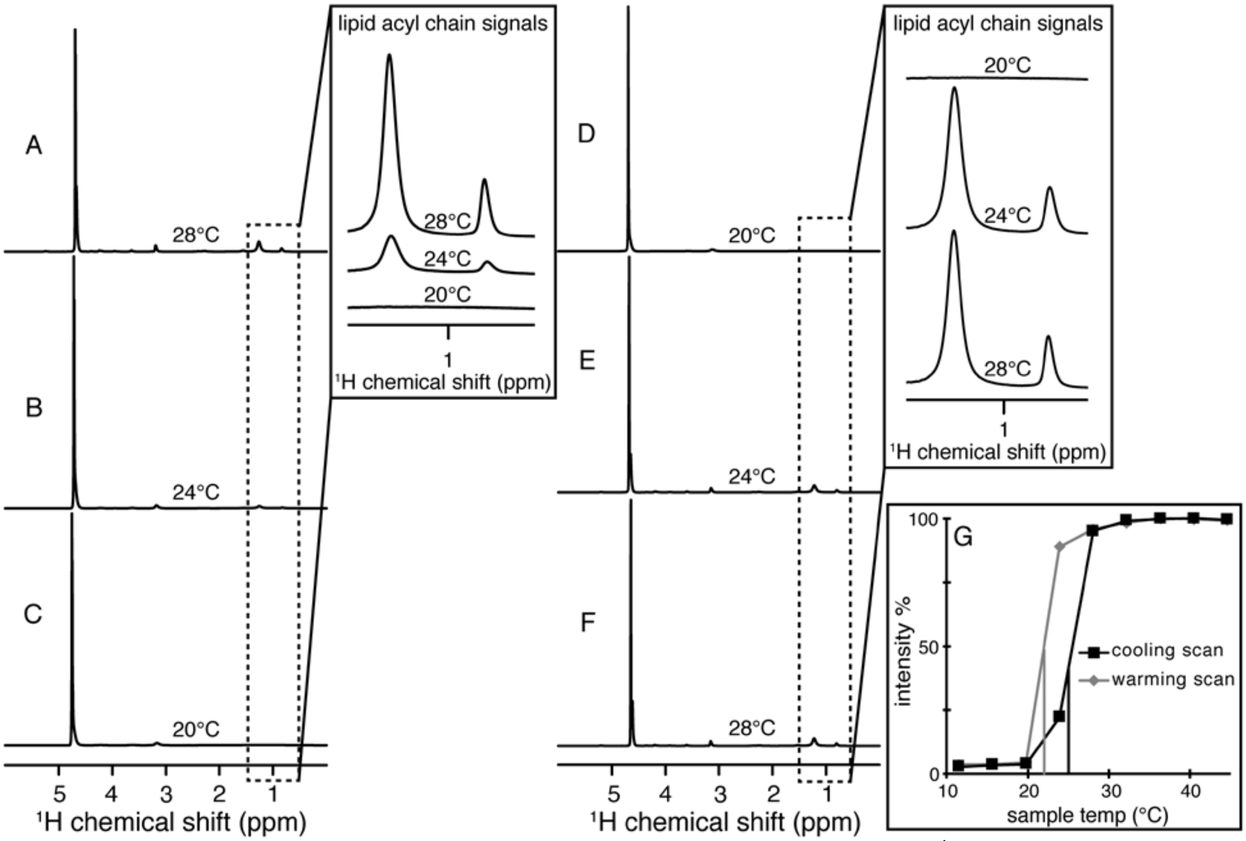

Figure S3 – Heating and cooling runs for hydrated DMPC MLVs. (A-C) ^{1}H MAS NMR spectra from the cooling run show that the transition occurs between 28 and 20 °C. (D-F) Spectra from the warming run show the transition between 20 and 24 °C. (G) Temperature dependent peak heights for lipid acyl chain CH_2 peaks. The transition mid-points in the cooling and heating scans occur at 25 and 22 °C, respectively. All spectra were acquired at 750 MHz.

Supporting References.

1. Lewis, R. N. A. H., and R. N. McElhaney. 2009. The physicochemical properties of cardiolipin bilayers and cardiolipin-containing lipid membranes. Biochim. Biophys. Acta 1788:2069-2079.
2. Boscia, A. L., B. W. Treece, D. Mohammadyani, J. Klein-Seetharaman, A. R. Braun, T. A. Wassenaar, B. Klösgen, and S. Tristram-Nagle. 2014. X-ray structure, thermodynamics, elastic properties and MD simulations of cardiolipin/dimyristoylphosphatidylcholine mixed membranes. Chem Phys Lipids 178:1-10.