Paramagnetic Effects on NMR Spectra of Isotropic Bicelles with Headgroup Modified Chelator Lipids and Metal Ions

Supplementary Information

Materials and Methods

Materials. 1,2-Dimyristoyl-1-sn-glycero-3-phosphocholine (DMPC), 1,2-dihexanoyl-1-sn-glycero-3-phosphocholine (DHPC) and 1,2-dimyristoyl-sn-glycero-3-phosphoethanolamine-N-diethylenetriaminepentaacetic acid ammonium salt (DMPE-DTPA) were obtained from Avanti Polar Lipids (Alabaster, AL).

Sample Preparation. The appropriate amount of DMPC (final concentration 84 mM), DHPC (final concentration 67 mM) and DMPE-DTPA (final concentration 3 or 6 mM corresponding to 2 or 4 mol% of total lipids) were mixed and the solvent chloroform was evaporated under the N_2 gas flow. The dry mixture was dissolved in cyclohexane and lyophilized for 3 hours. The powder mixture was dissolved in water solution with NaN_3 (final concentration 0.02%) and KCl (final concentration 50 mM). The mixture solution was then subjected to freeze and thaw cycles for 5 times to ensure that uniform bicelles are formed. Different metal ions solutions were added to different batches of bicelles to achieve final concentration of 3 or 6 mM. The phosphate buffer (pH 6.5, final concentration 10 mM) and D_2O were added to the bicelles before transfer into NMR tubes.

NMR Spectroscopy. ^1H and ^{13}C spectra were acquired on a Agilent/Varian VNMRS 600 MHz spectrometer with a 5 mm cryoprobe (^1H frequency: 599.936 MHz, ^{13}C frequency: 150.869 MHz). 1D single-pulse experiments were used and standard water suppression sequence was used for ^1H spectra. ^1H T_1's were measured by ^1H 1D inversion recovery experiment. 31P spectra were acquired on a Agilent/Varian 300 MHz spectrometer with a 5 mm double-resonance probe (31P frequency: 121.441 MHz). 1D single-pulse experiments were used. All the spectra were acquired at 25 ºC. Chemical shifts were referenced to TMS with an external standard. Spectra were processed and analyzed with ACD/NMR Processor Academic edition (ACD labs, Toronto, Ontario, Canada). Back linear prediction, zero filling and Lorentzian-to-Gaussian apodization were used before Fourier transformation.