Experimental report

15/04/2020

Proposal: 9-10-1591 **Council:** 10/2018

Title: Distribution of water and alcohol in and around diyne phospholipid nanotubes

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: H2O
D2O
Methanol
Ethanol
ethanol-d6
Methanol-d4
1,2-bis(tricosa-l0,l2-diynoyl)-sn-glycero-3-phosphocholine

Instrument	Requested days	Allocated days	From	To
D11	2	2	02/07/2019	04/07/2019

Abstract:

The formation of nanotubes from chiral molecules such as diyne phospholipids can be achieved in alcohol/water mixtures by either cooling from a vesicular state [1] or by fast addition of water to the alcoholic solution at room temperature [2]. The second method has proven to yield nanotubes of very well-defined radius, showing dozens of oscillations in SAXS. It is well established that solvent quality plays an important role for this system [3]. Hence, we propose to study the solvent distribution in and around the nanotube walls by employing different contrast conditions, thereby complementing experiments at ID02, ESRF. If the solvent composition is enriched in alcohol or water around the nanotubes, we expect to see effects on both the forward scattering intensity and the tube form factor in general.

[1] Thomas B.N. et al., Science 267, 1635-1638 (1995)
[2] Georger J.H. et al., J. Am. Chem. Soc. 109, 6169-6175 (1987)
[3] Spector M.S. et al., J. Am. Chem. Soc. 119, 8533-8539 (1997)

1 Experiment details

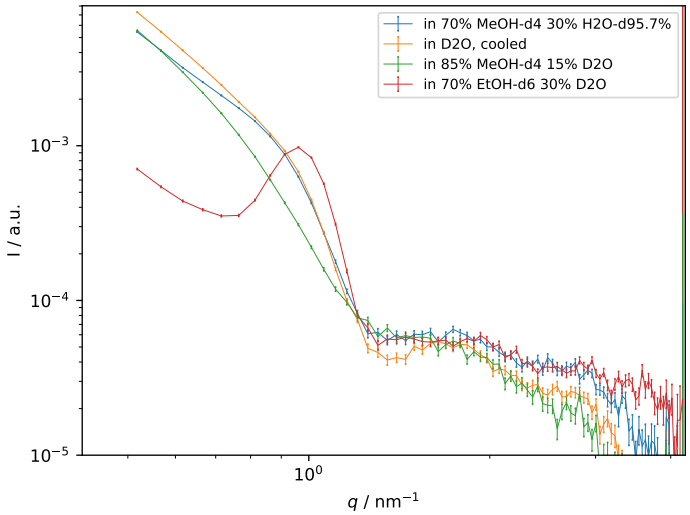

Figure 1: Measurement results for the high-flux configuration at 1.7 m. The high-q details obtained are similar to results from SAXS.

We measured tubular aggregates of the diyne phospholipid $DC_{8,9}PC$ in mixtures of methanol or ethanol in water. Unless otherwise specified, samples are at a lipid concentration of 1 g/L with a volume fraction of 70 % perdeuterated alcohol (methanol-d4/ethanol-d6) in the solvent. The temperature was controlled using a water bath set to 25 °C and samples were prepared by rapid addition of the aqueous solvent phase to an alcoholic solution of our phospholipid.
To cover the q -range from $7 \times 10^{-4}\,\mathrm{nm}^{-1}$ to $4\,\mathrm{nm}^{-1}$, we employed 4 configurations: 1.7 m, 8 m and 39 m at 5.6 Å and 39 m at 11.2 Å. These measurements were complemented using a high-flux configuration at 1.7 m which shows details of the bilayer structure upon subtracting the corresponding measurement of the solvent that would otherwise be hidden in the background.
We varied both the contrast between lipid and solvent as well as the contrast between the alcoholic and aqueous constituents of our solvent, thereby checking for any demixing that might occur near the aggregate surface. Specifically, we varied the H/D-ratio of the aqueous phase from 100% to 0% D2O (5 samples) and used fully deuterated alcohol for maximum signal-to-noise ratio. One sample with fully hydrogenated alcohol + D2O was measured per system.
As reference, a sample with 95.7% D2O in the water phase and 100% MeOH-d4 as the alcohol was used that elim-

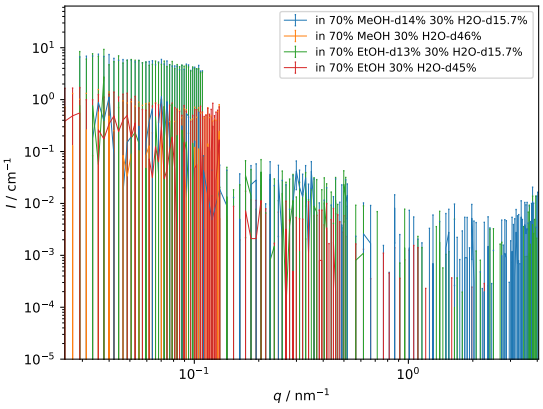

Figure 2: contrast match, all samples

inates contrast within the solvent for the methanol system. This inter-solvent matching point occurs at 100% D2O in the case of EtOH-d6. Further references used were solutions of the lipid in d-alcohol at two concentrations each.

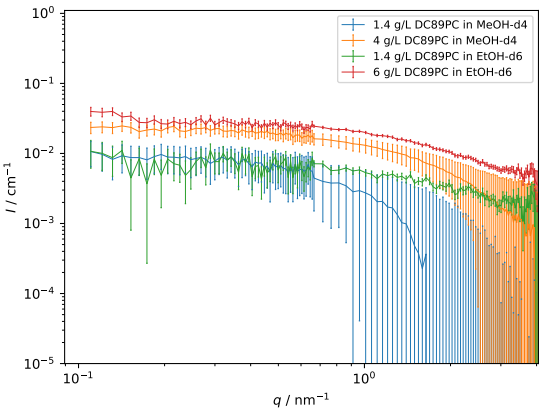

Figure 3: DC89PC in pure alcohol

Samples with solvent mixtures designed to match the lipid SLD were measured both when each solvent (alcoholic and aqueous) matches the lipid (13% EtOH-d6, 14% MeOH-d4, and 15.7% H2O) and when the mixture alcohol + water matches (MeOH + 46% D2O in the aqueos phase and EtOH + 45% water-d2, respectively).

2 Preliminary Results

1. There is a feature (shoulder to peak) transition in the contrast variation experiment at about 1 nm^{-1} (70% methanol). See figure 4

2. Features are too small to be seen in contrast match condition (within feasibility). See figure 2

3. There is a marked difference between 70% and 85% methanol (figure 1) and also small structures present in (roughly) pure alcohol[1] (figure 3)

[1] not dried

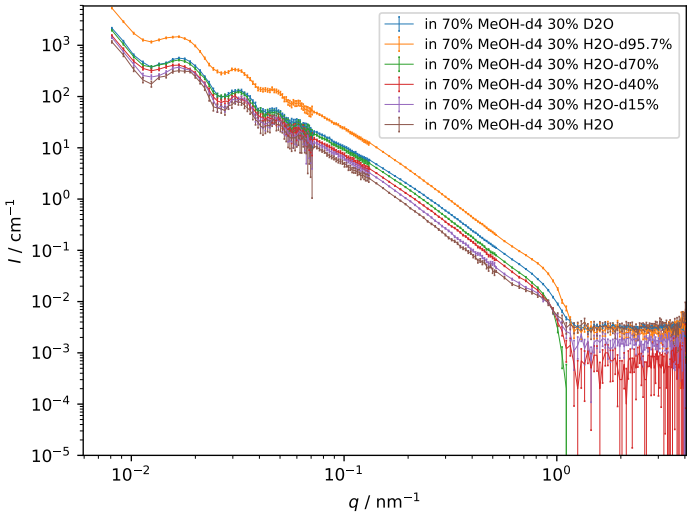

Figure 4: Contrast variation for unilamellar nanotubes at 25 °C showing changes of slope at high q (70% MeOH-D4).

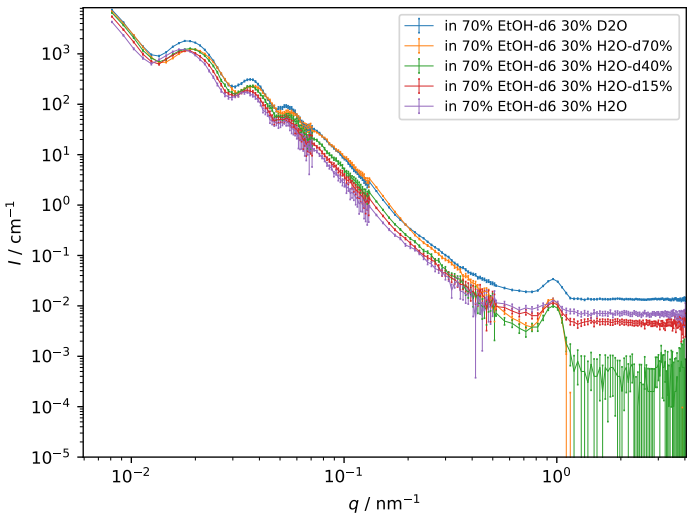

Figure 5: Contrast variation for unilamellar nanotubes at 25 °C showing changes at high q (70% EtOH-D6).