Experimental report

16/09/2022

Proposal: TEST-3179 **Council:** 4/2021

Title: Stability of lipids solution during the freezing

Research area:

This proposal is a new proposal

Main proposer: Olga MATSARSKAIA

Experimental team:

Local contacts: Viviana CRISTIGLIO
Bruno DEME

Samples: Lipids in buffer solution - DSPC/DOPC/Cholestrol

Instrument	Requested days	Allocated days	From	To
D16	2	2	01/03/2021	03/03/2021

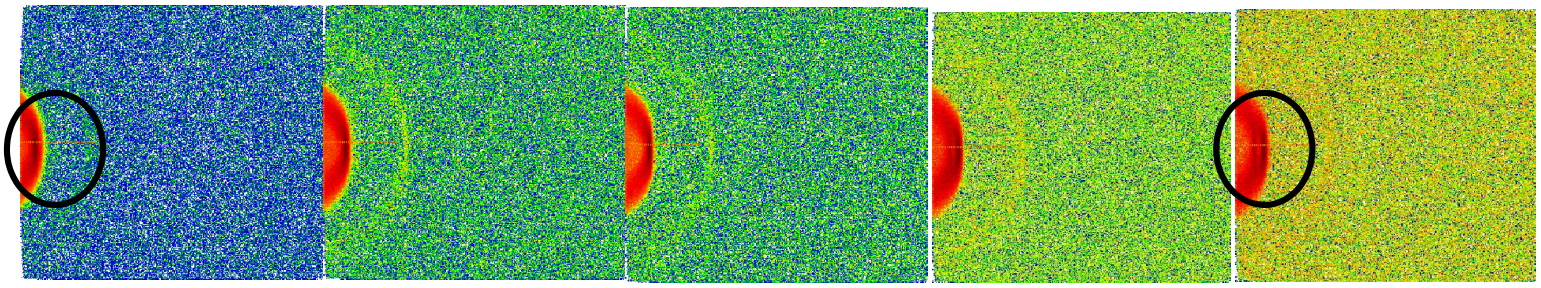
Abstract:

Report of TEST-3179:

We have performed temperature scans (standard orange cryostat) and collect data at defined steps successively at low angles (0.02 – 0.7 $Å^{-1}$) to investigate the lipid phase changes and at wide angles where ice crystal peaks are observed (between 1.5 and 1.9 $Å^{-1}$).

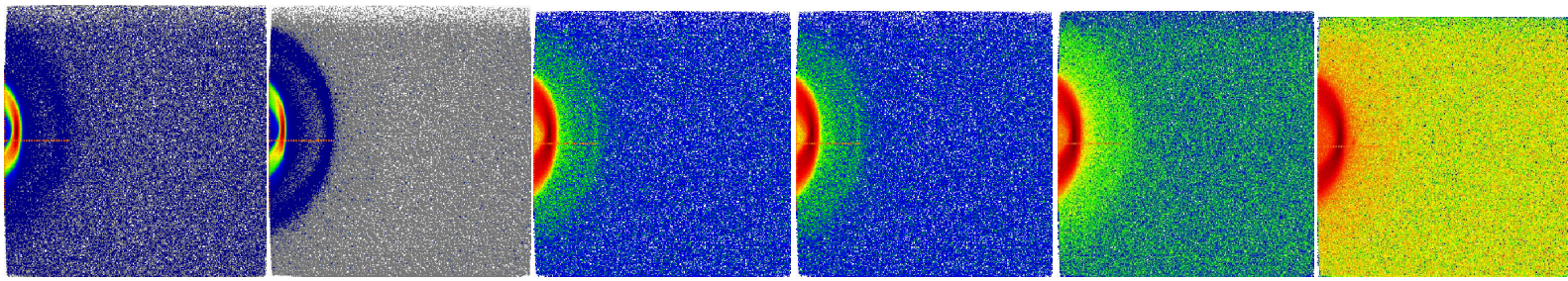
The SANS data have been collected at 1 detector position while the peaks resulting from the crystallization of water have been collected in 2 positions (gamma=12, 75 and 114).
The cooling/heating rate we used is similar to those used in DSC experiments (1 C/min). The test was performed with 2 samples: pure DSPC and a DSPC/Cholesterol binary mixture in phosphate buffer.

Pure DSPC



40 C 25 C -5 C -10 C -80 C

DSPC/Cholesterol

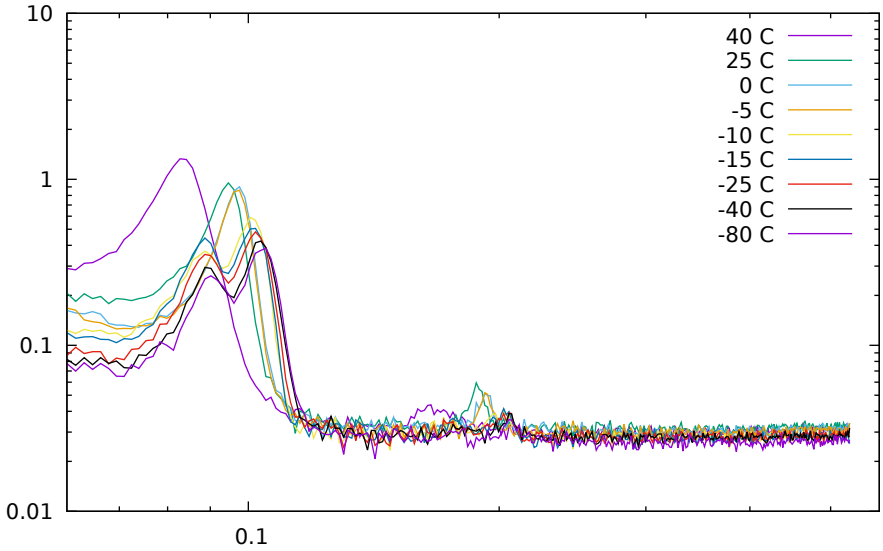


40 C 25 C -5 C -10 C -15 C -80 C

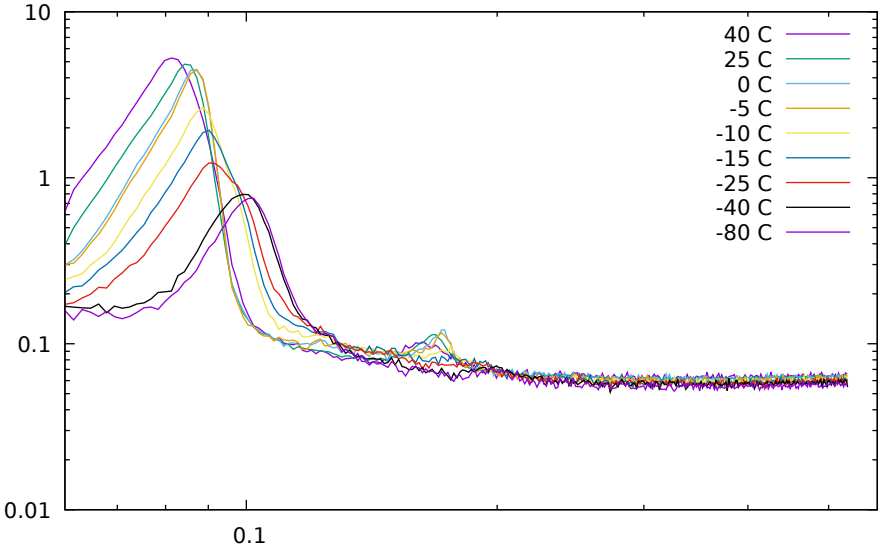
At small angle in pure DSPC we observed that the lamellar phase undergoes a phase transition to two coexisting lamellar phases between -5°C and -10°C, when the excess water in the sample crystallizes. Interestingly, when cholesterol is added, the lamellar phase spacing decreased upon cooling, but no phase transition was observed.

In the integrated 1D curves we can detect the diffraction peaks corresponding to the lamellar phases for each samples at different temperatures.

Pure DSPC

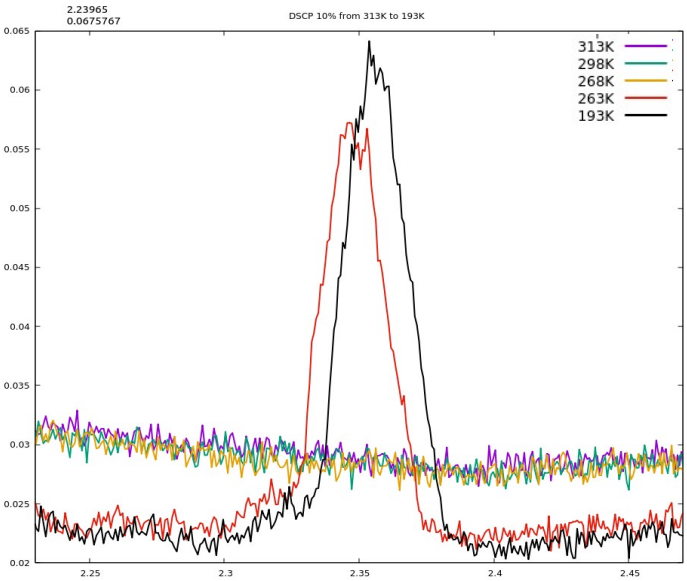


DSPC/Cholesterol

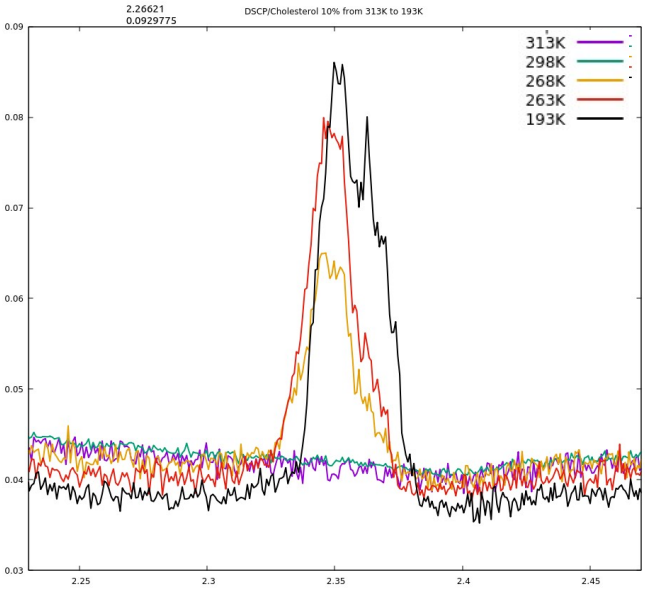


The ice formation can be followed looking at wider angles and the results are shown in fig.3.

Pure DSPC



DSPC/Cholesterol



We observed that for pure DSPC the ice formation temperature is lower than DSPC with cholesterol.

This test has confirmed the feasibility of the experiment in the temperature (T) range of interest: quality of the signal, evolution of the lamellar phase and at the same time the water crystallization upon cooling, and the reversibility of the phase transitions.