# Support information

# Reentrant structural phase transition in amphiphilic self-assembly

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**Material**:

The triterpenoid saponin Quil A and Chol were obtained from Accurate Chemical, Westbury, USA and Sigma Aldrich, Taufkirchen, Germany, respectively. Tris - (hydroxymethyl) - aminomethane (TRIS) was supplied by Merck KGaA, Darmstadt, Germany. The deuterium oxide ($D_2O$) was from Deutero GmbH, Kastellaun, Germany.

**Preparation of Quil A/Chol solution:**

The formulations with Quil A/Chol were prepared according to the lipid film hydration method.[S1] Quil A solutions were dissolved in TRIS buffer 140 mM (pH 7.4) with $D_2O$. Chol was weighted into glass vials and dissolved in chloroform. The solvent was then evaporated under vacuum at room temperature for one hour. The resulting film was hydrated with solutions of Quil A. Quil A/Chol systems were stirred at 293 K for 24 hours and filtrated through a 0.22 μm membrane filter (PVDF filter, Millipore, USA). The concentration of Quil A/Chol (sum of Quil A and Chol) was 6.7 mg/ml for all the systems.

**Small-angle X-ray scattering measurements:**

SAXS experiments were carried out with a Kratky-compact camera that produces a slit-collimated beam (Anton Paar, Graz, Austria), equipped with Cu-K radiation (wave length 0.1542 nm) from the Generator PW1830 (Philips, Kassel, Germany), a Peltier heating/cooling sample holder (Anton Paar, Graz, Austria) and a position-sensitive detector OED 50M (MBraun, Munich, Germany). The $q$ range was 0.12 – 5 $nm^{-1}$. The experimental SAXS data were processed using the indirect transform program Gnom 4.5a.[S2]Desmearing of slit collimated x-ray scattering data was also implemented in Gnom.

**Small-angle neutron scattering measurements:**

SANS experiments were performed by the SANS I at the FRG1 research reactor in Helmholtz-Zentrum Geesthacht, Germany. The wavelength $\lambda$ was 0.85 nm. The $q$ range was 0.05 – 2.5 $nm^{-1}$ obtained by 4 different detector distances (0.7 – 9.7 m). Samples were kept in quartz cuvettes with a path length of 2 mm at 25 ± 1°C. The raw spectra were corrected for sample transmission, room background, and sample cell scattering by conventional procedures.[S3]The two-dimensional isotropic scattering patterns were azimuthally averaged, converted to an absolute scale, and corrected for detector efficiency by dividing with the incoherent scattering spectra of 1 mm thick pure water. The smearing induced by the different instrumental set-ups was included in the data analysis. For each instrumental setting the appropriate resolution function was used to smear the ideal model scattering curves when the model scattering intensity was compared to the measured one using the least-squares method.[S4]

**References**

S1. Copland, M.; Rades T. and Davies, N. Hydration of lipid films with an aqueous solution of Quil A: a simple method for the preparation of immune-stimulating complexes, *Int. J. Pharm.* **2000**, 196, 135.
S2. Svergun, D. I. Determination of the regularization parameter in indirect-transform methods using percettual criteria, *J. Appl. Cryst.* **1992**, 25, 495-503.
S3. Wignall, G. D.; Bates, F. S. Absolute calibration of small-angle neutron scattering data, *J. Appl. Cryst.* **1987**, 20, 28-40.
S4. Pedersen, J.S.; Posselt, D.; Mortensen, K. Analytical Treatment of the Resolution Function for Small-Angle Scattering, *J. Appl. Cryst.* **1990**, 23, 321-333.