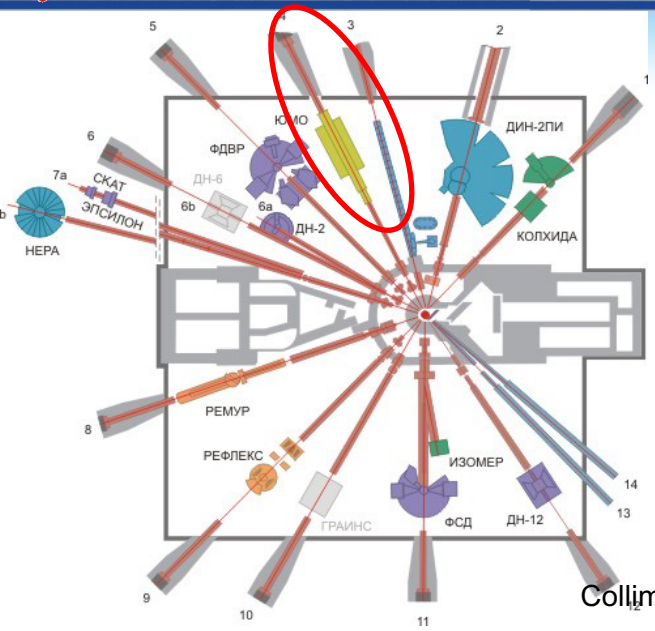
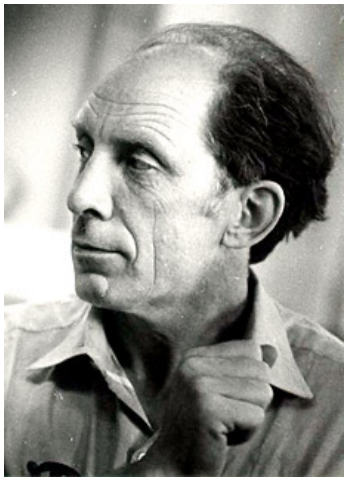
Small angle neutron scattering spectrometer YuMO. Status and prospects

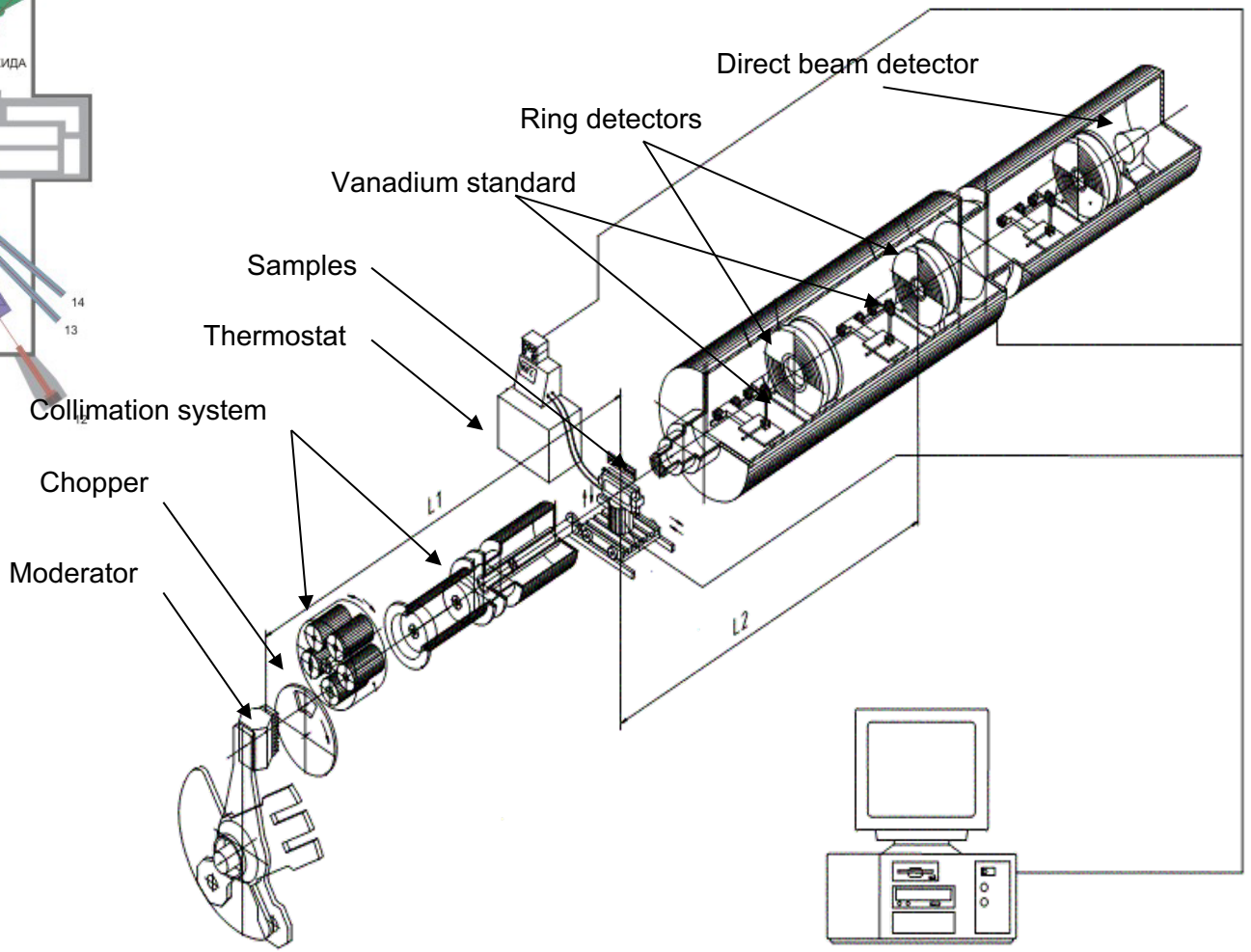
O. Ivankov , D. Soloviov, A.I. Kuklin

54th meeting of the PAC for Condensed Matter Physics

YuMO spectrometer





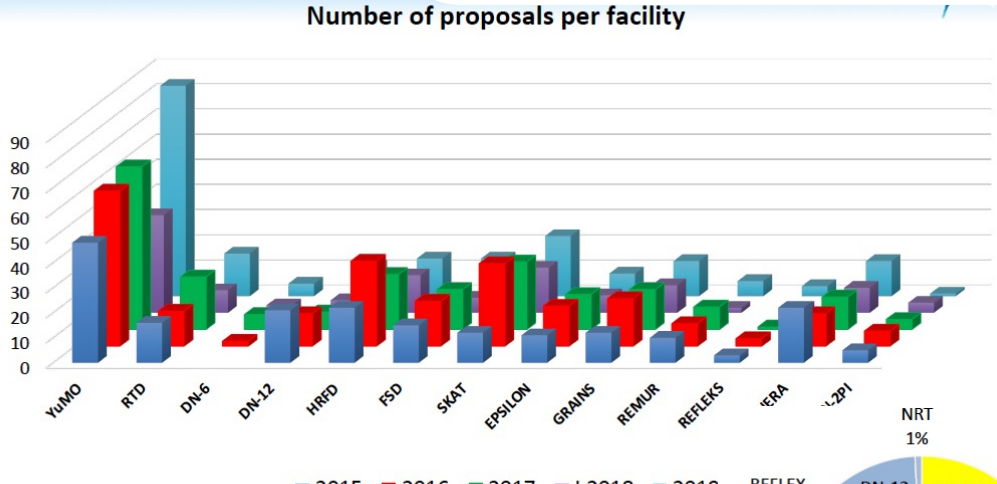


Kuklin, A.I., Ivankov O.I., Rogachev A.V., Soloviov D., Islamov A., Skoi V.V., Kovalev Y., Vlasov A., Rizhikau Y.L., Soloviev A., Kucerka N., Gordeliy V., *Small-Angle Neutron Scattering at the Pulsed Reactor IBR-2: Current Status and Prospects.* Crystallography Reports, 2021. **66**(2): p. 230-241.

Main parameters of the YuMO spectrometer

Parameters	Value
Flux on the sample (thermal neutrons)	$10^7 \div 4x10^7$ n/(s cm^2) [1]
Used wavelength	0.7 Å to 8 Å (10 Å with 30K cold moderator)#
Q-range	$7x10^{-3} \div 0.5$ Å^{-1} (~$4x10^{-3} \div 0.5$ Å^{-1})
Dynamic Q-range	q_{max}/q_{min} up to 100
Specific features	Two detectors system, central hole detectors
Accessible size range of the structural features under investigation *	1000 ÷ 10 Å
Intensity (absolute units -minimal levels)	0.01 cm^{-1}
Calibration standard	Vanadium during the experiment
Beam dimension at the sample position	14 mm diameter
Collimation system	Axial
Detectors	^{3}He filled, , 8 independent wires, in-house design
Detector (direct beam)	^{6}Li-convertor in-house design
Sample changer	Placed in the custom made box, in air
Q-resolution	low , 5-20%
Temperature range	4°C - + 70°C (standard Hellma cells, 1mm, 2mm pathlength) -20°C - + 130°C (custom designed sample holders required)^
Number of the samples in the automated sample changer	25 ***
Background intensity	0.03 – 0.2 cm^{-1}
Average single seta data collection time	1 h
Source pulse frequency	5 Hz
Control computer Operating system	WINDOWS 10
The instrument control software suit	SONIX **[6]**
Controlling parameters	Starts (time of experiments), power, vanadium standard position, samples position, samples changer temperature, vacuum level in the detectors tube .

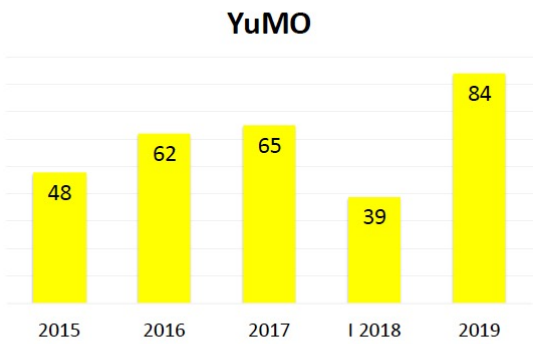
User program for IBR-2 facility

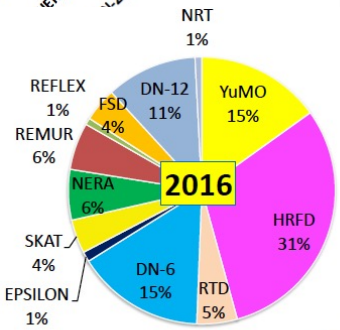


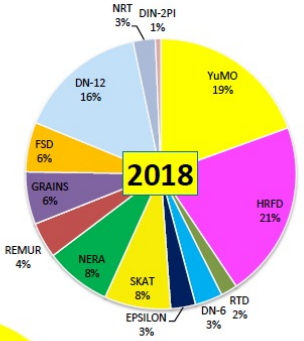
D. Chudoba
Information about the FLNP User Programme
51st meeting of the PAC for Condensed Matter Physics
20 - 21 January 2020

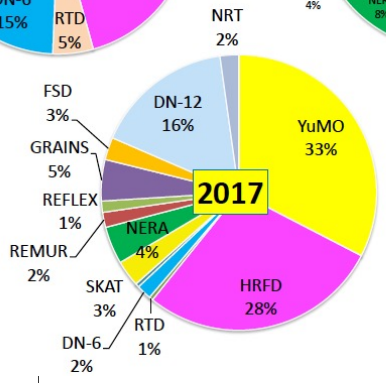
JOINT INSTITUTE FOR NUCLEAR RESEARCH
Frank Laboratory of Neutron Physics
Лаборатория нейтронной физики им.И.М.Франка

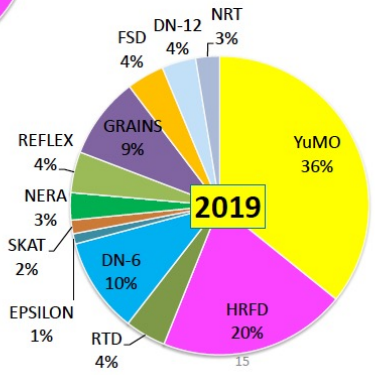
Number of proposals – Small Angle Neutron Scattering





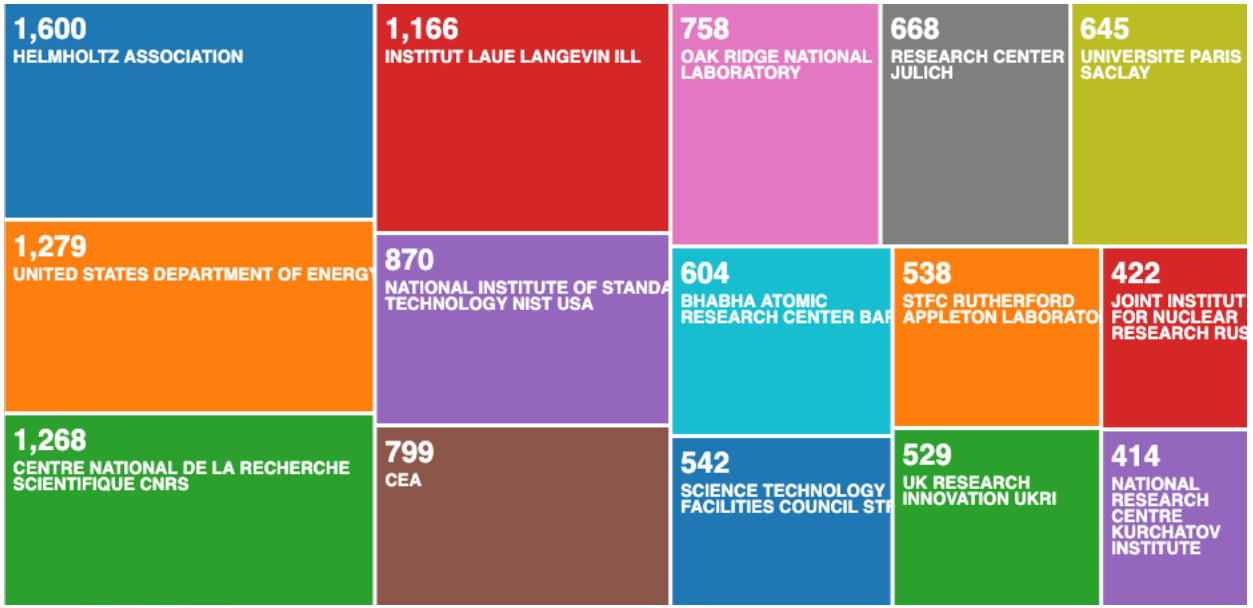


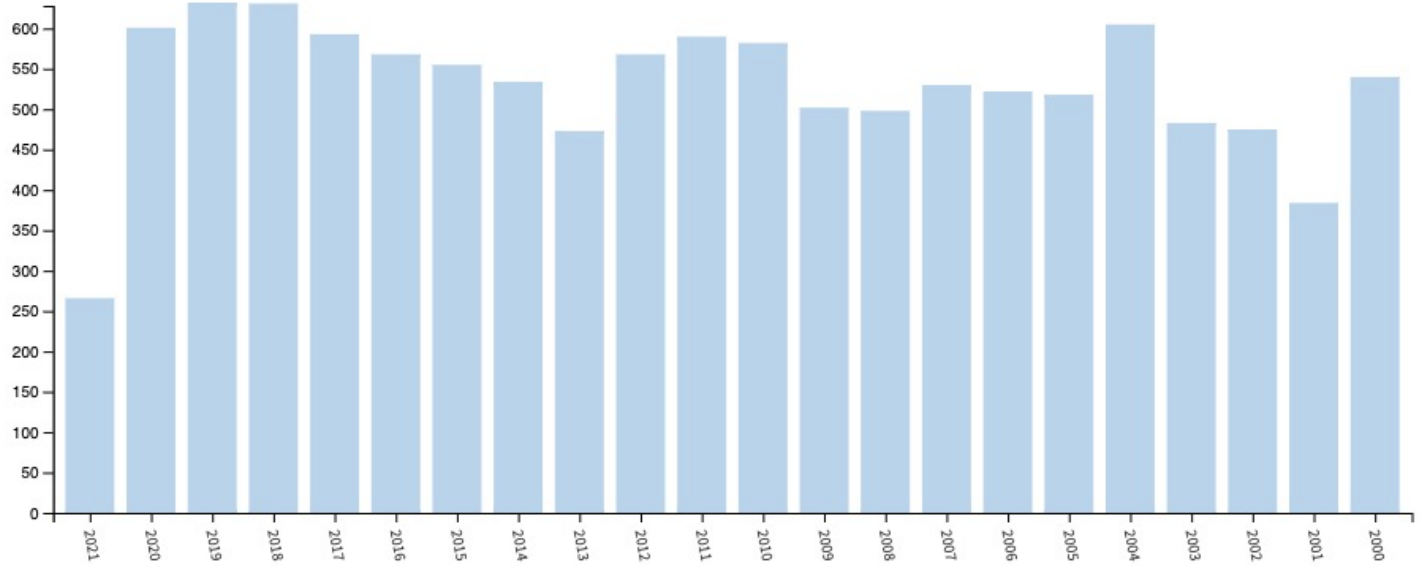




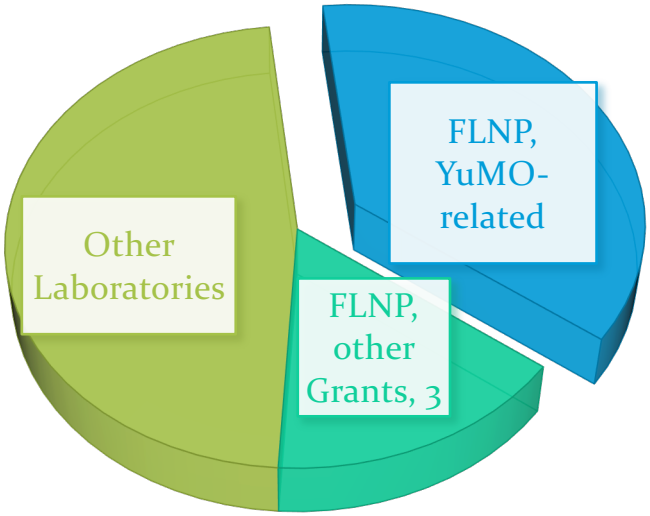
Instrument Scientific Outcome - the statistics after applying a weighting factor that is based on the journal impact factor.

Web of science summary





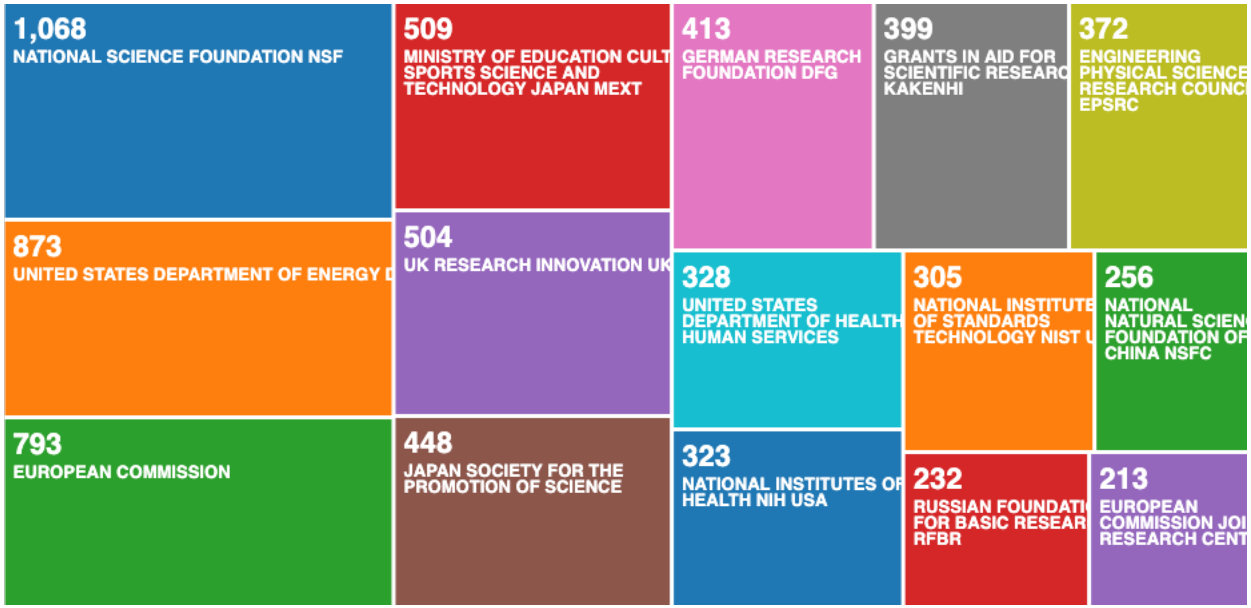
Russian Science Foundation Projects in JINR



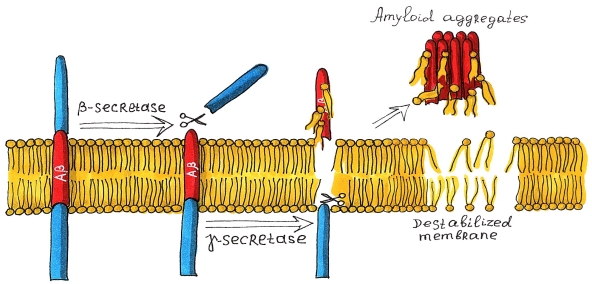
19 - winning projects RSF in JINR
10 - FLNP laboratory
7 – YuMO related projects

RSF Projects implemented on the large research infrastructure facilities

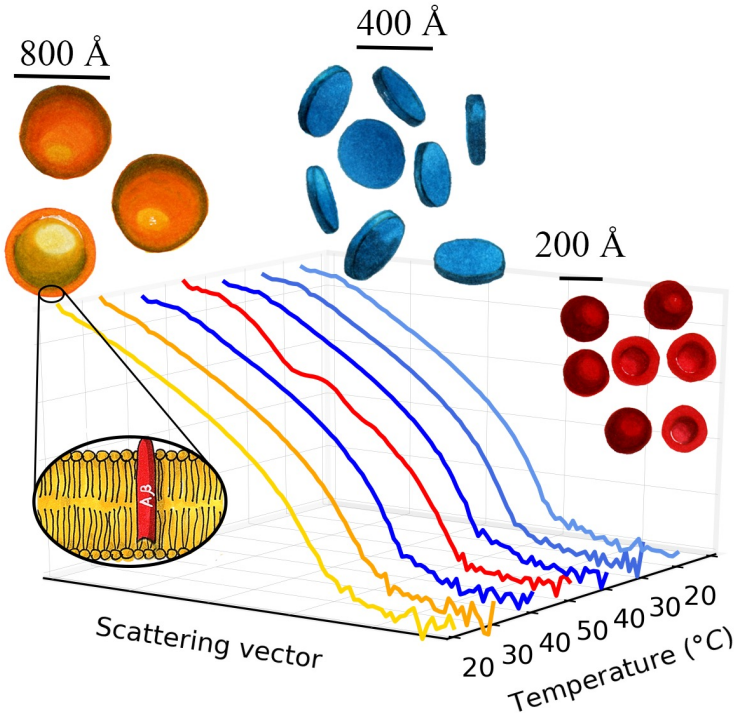
IBR-2 is one from **18** winning from **189** facilities in 2019.
4 projects for IBR-2 facility were approved for funding
3 projects requires **SANS** measurements

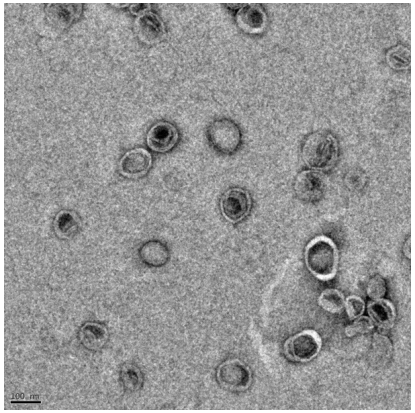


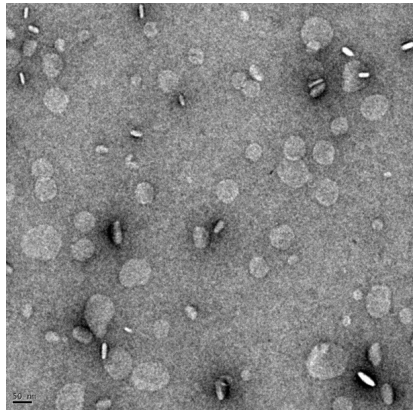
Interactions in Disease Modeling Membranes



Neutron scattering allows to study model membranes that replicate pre-clinical conditions of **Alzheimer's disease**





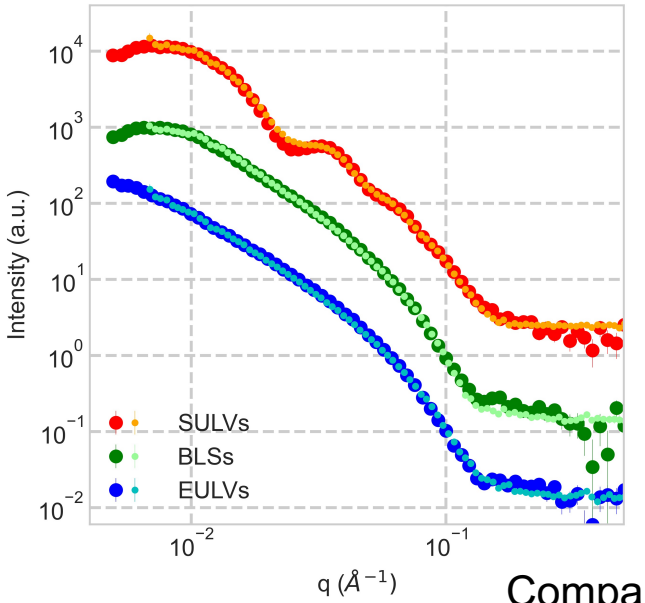


TEM images of the DMPC (left) and DMPC/Aβ25-35 (right) systems collected at 20˚C. The dark bars (100 and 50 nm, respectively) in the lower left corners allow to assess the length scales. Objects in the left-hand panel match the typical vesicular objects with mostly unilamellar walls. The right-hand panel reveals randomly oriented discs also consisting of single layers.

Changes in the membrane self-organization happen during the thermodynamic phase transitions of lipids and are interpreted as the **peptide driven membrane damage**.

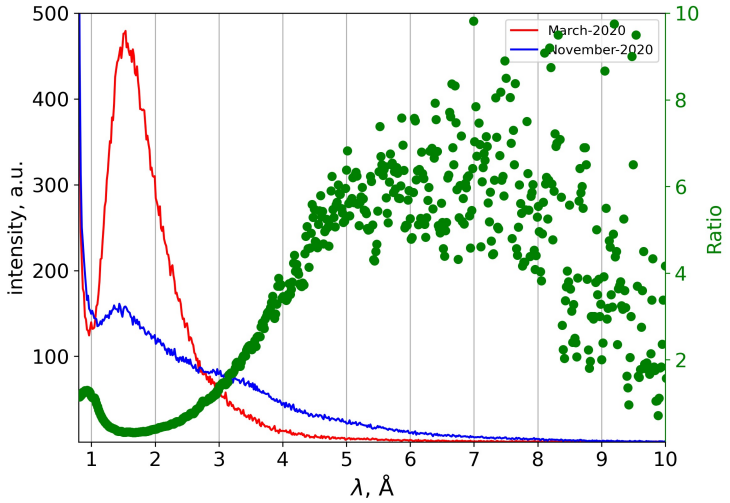
O. Ivankov, T.N. Murugova, E.V. Ermakova, T. Kondela, D.R. Badreeva, P. Hrubovčák, D. Soloviov, A. Tsarenko, A. Rogachev, A.I. Kuklin, N. Kučerka , *The Journal of Physical Chemistry Letters (under review)*

Cold moderator implementation

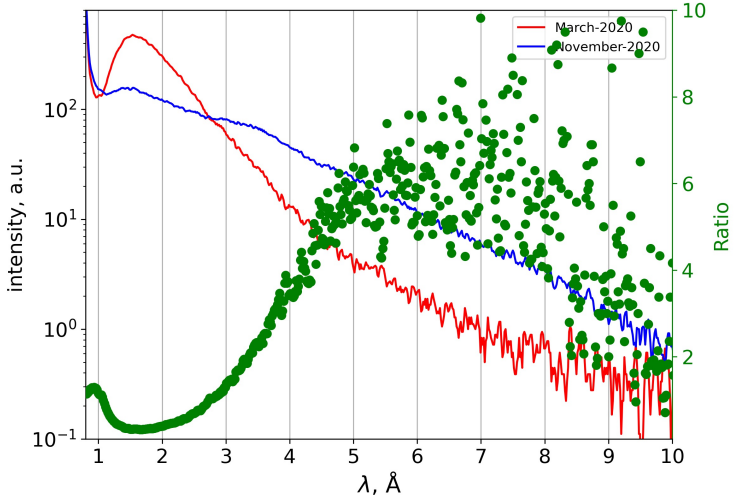


The low-q enhanced data (large circles) compared to the data of standard setup (small circles). The different forms of scattering curves allow to distinguish the overall shape of membrane organization (SULVs vs. BLSs vs. EULVs).

Comparison of the counts on the detectors with the cold moderator at ~ 300K and ~34K



Gain Factor (Ratio):
min ~ 0.3 (1.5 Å)
max ~ 6 (> 5Å)



Kinetics system for YuMO spectrometer

Time of measurement – 2 min

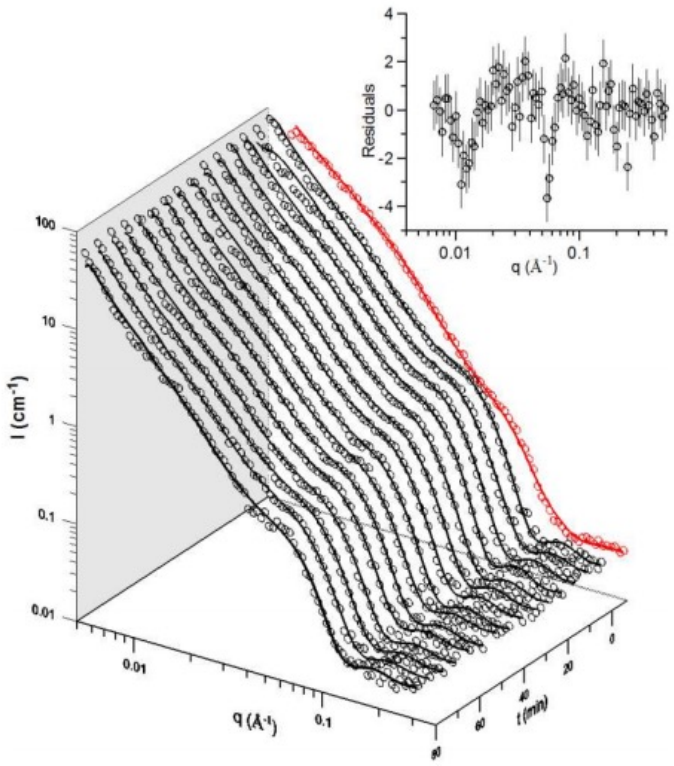


Figure 4. Dependences of SANS intensity $I(q)$ on scattering vector q for DNA – C_{12}NO/DOPE dispersion as a function of time; prior (red points) and after DCl injection. Full lines show fits using a paracrystal lamellar model.
Inset: An example of the distribution of residuals.

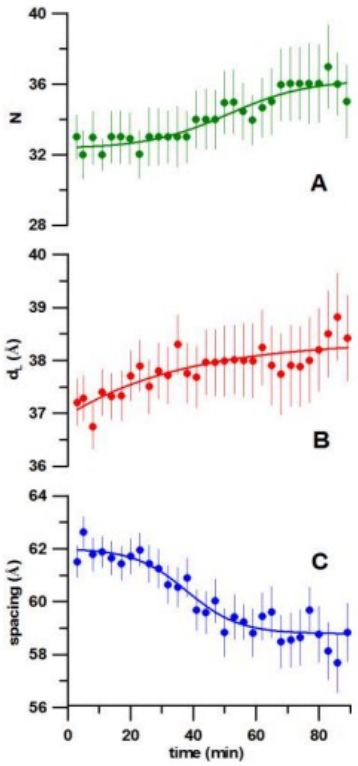
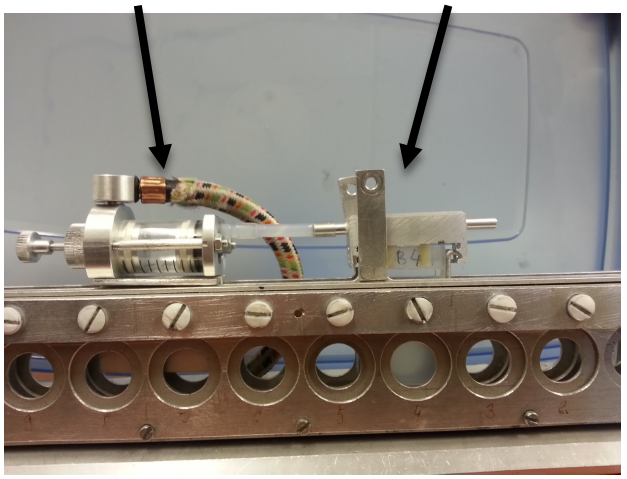


Figure 5. Time dependence of the structural parameters: the number of layers (A), the lipid bilayer thickness (B) and the spacing (C).

Container for the sample to be injected

Standard cuvette



Samples holder

D. Uhríková, J. Teixeira, L. Hubčík, A. Búcsi, T. Kondela, T. Murugova, and O. I. Ivankov, Journal of Physics: Conference Series **848,** 012007 (2017).

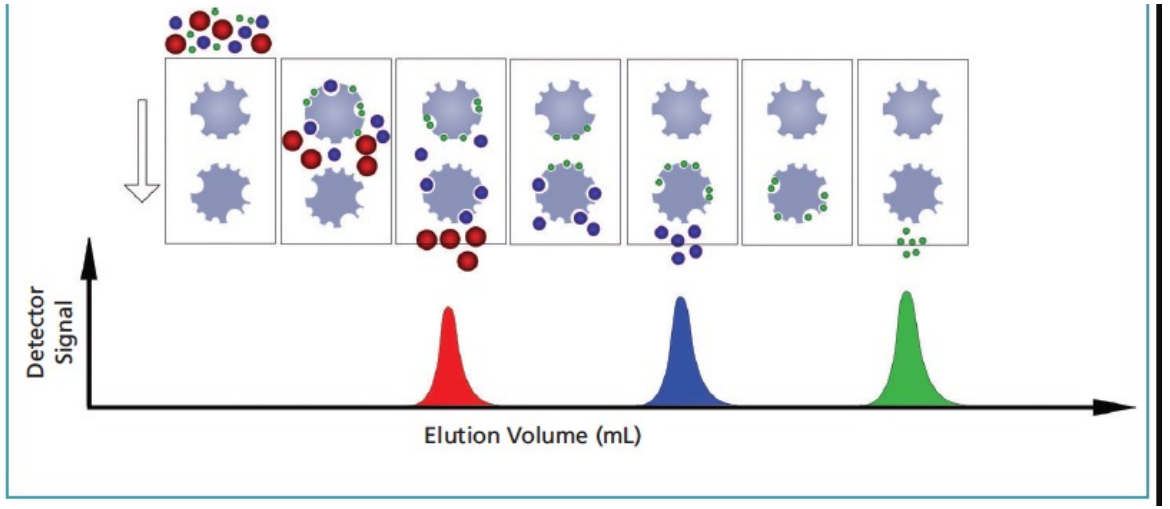
Kinetics system was developed and manufactured in Commenius University of Bratislava, Slovakia

Size-exclusion chromatography (SEC)

Applications:

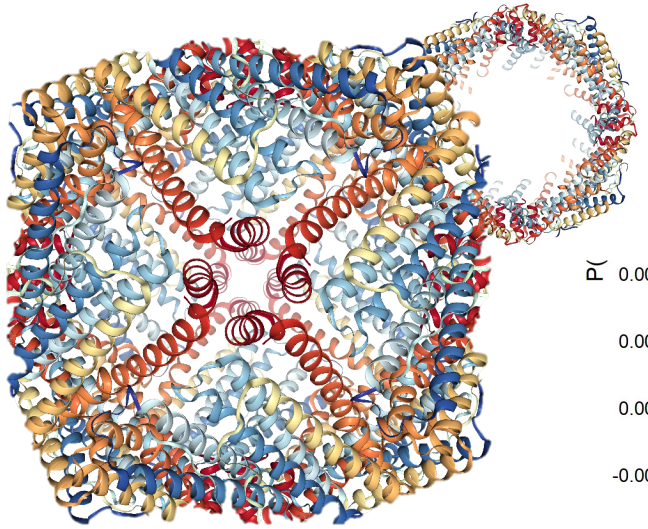
- Separation of macromolecules from complex mixtures according to their size, charge, selective non-covalent interaction and other properties.
- Protein and polimers purification.
- Affinity-tagged protein purification.
- Desalting and buffer exchange.
- Identification and quantitation of macromolecules (evaluation of hydrodinamic size of a macromolecule).
- Detects the unknown compounds and purity of mixture.

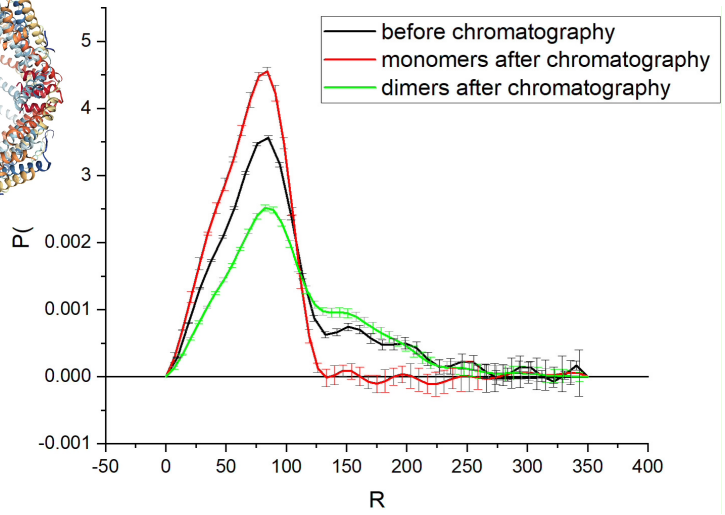
Funding:
- RSF Grant (Kucerka N.)
- Department of Spectrometers Complex IBR-2 (Kulikov S., Bodnarchuk V.)
- JINR-Poland Grant (Kuklin A.)

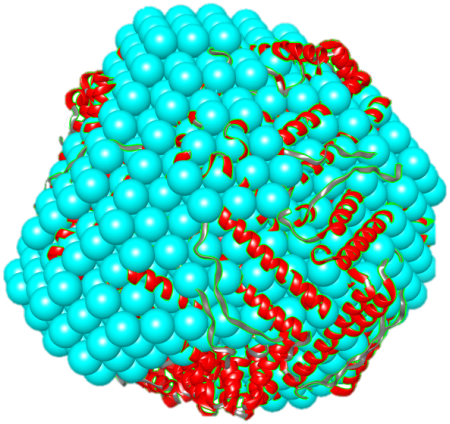


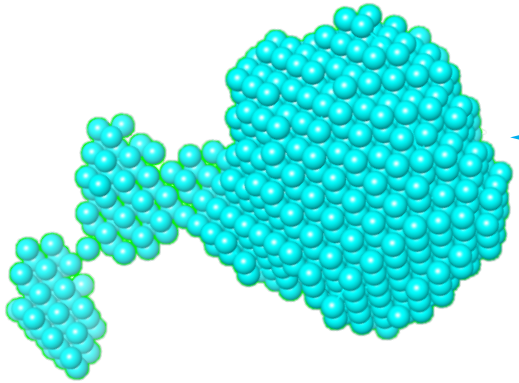
Implementation of SEC on YuMO spectrometer

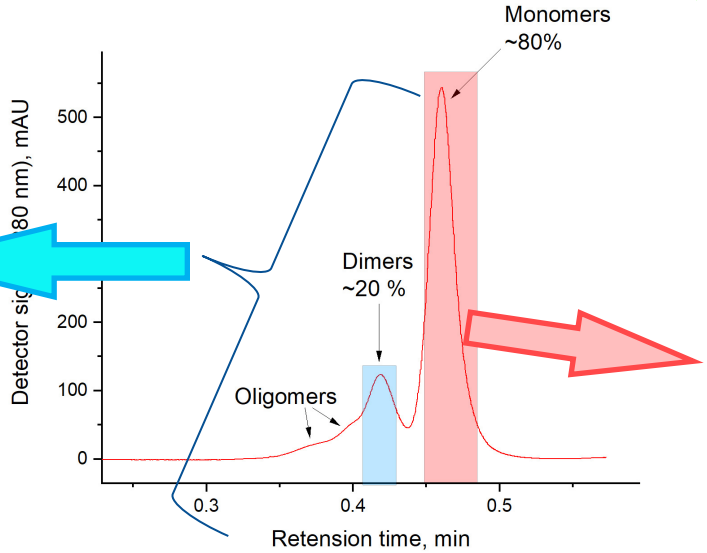
Apoferritin protein

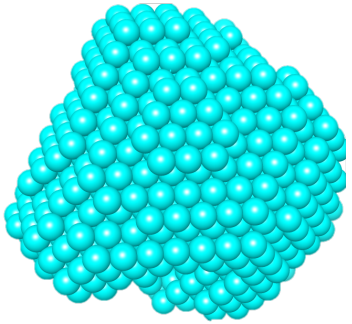






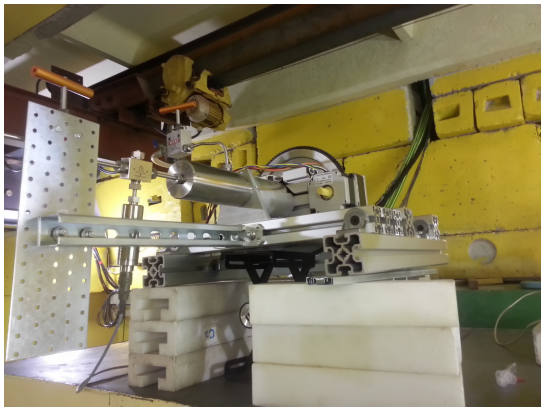


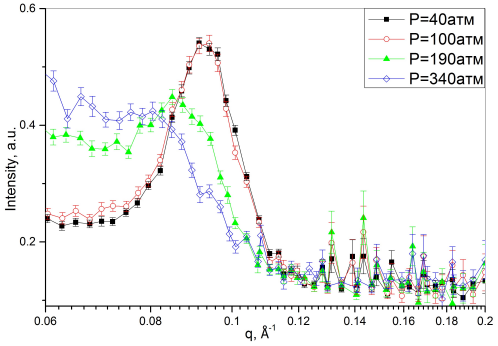


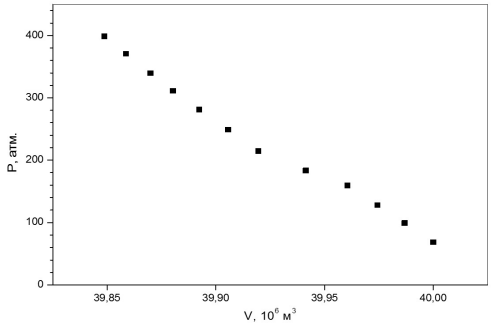


D. I. Svergun (1999) Restoring low resolution structure of biological macromolecules from solution scattering using simulated annealing. *Biophys J.* 2879-2886

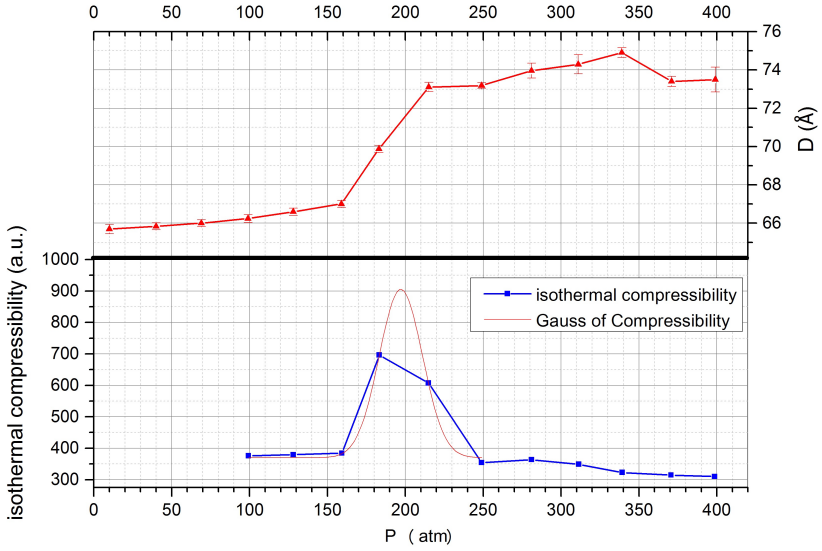
Volumetric setup for YuMO spectrometer







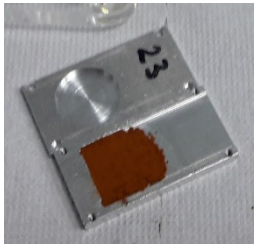
- Volume of the sample required: 3-4 ml
- Pressure up to 2 kBar

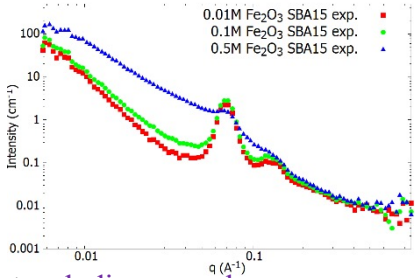


Soloviov, D., et al., *Changes in the Area per Lipid Molecule by P–V–T and SANS Investigations.* Macromolecular Symposia, 2014. **335**(1): p. 58-61.

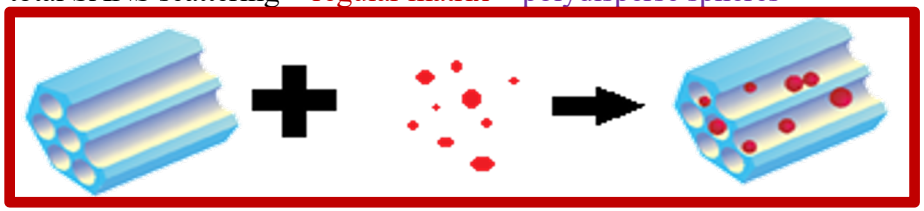
Nanopores for Magnetic and Biomedical Applications

SANS experiments at YuMO





total SANS scattering = regular matrix + polydisperse spheres



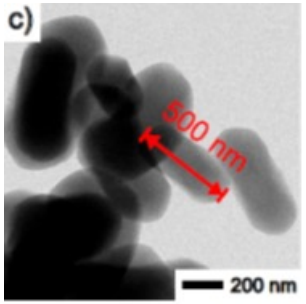
$$I(q) = K_C S(q)|F_C(q)|^2 + K_S|F_S(q)|^2 + I_d(q) + I_i$$

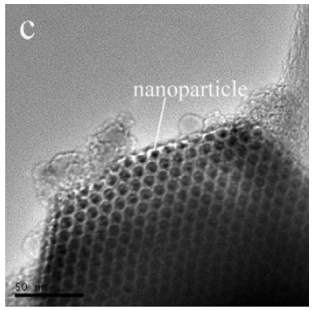
- pores - size & mutual distance
- NPs - size distribution & concentration

Material

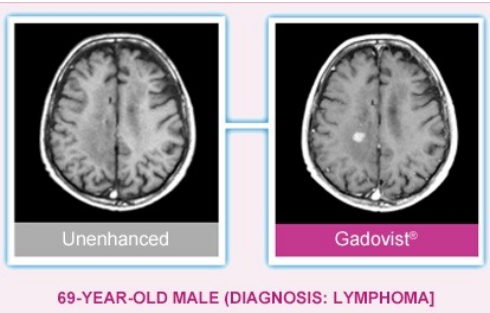
Periodic nanoporous silica

- perfect regular structure
- biocompatibility
- thermal stability and durability
- high specific surface

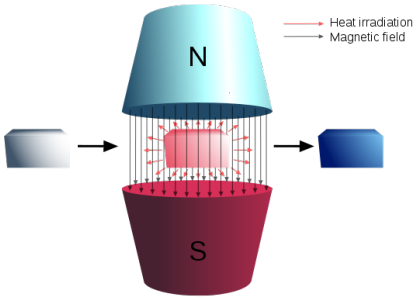




Applications



- therapeutic agents in tumor treatment and drug delivery
- MRI contrast media (Gadovist)
- magnetic refrigeration due to the large magnetocaloric effect

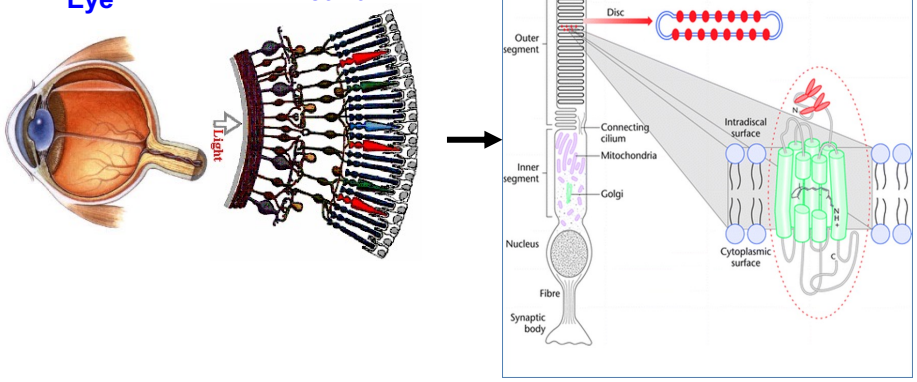


Zeleňáková, A., Hrubovčák P., Kapusta O., Kučerka N., Kuklin A., Ivankov O., Zeleňák V., *Size and distribution of the iron oxide nanoparticles in SBA-15 nanoporous silica via SANS study.* Scientific Reports, 2019. **9**(1): p. 15852.

Analysis of the supramolecular organization of rhodopsin in photoreceptor membrane

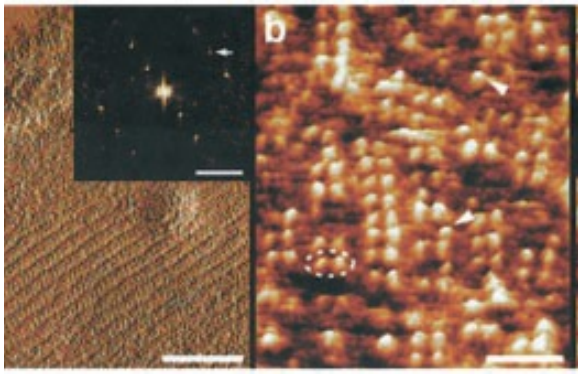
Eye

Retina

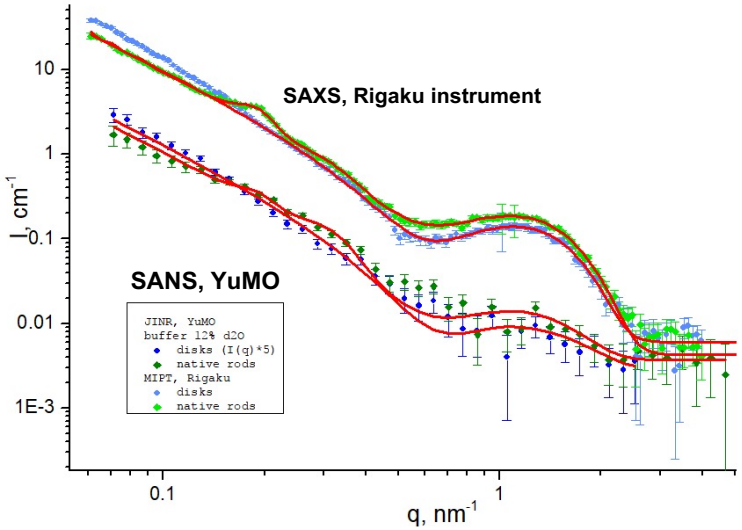


Photoreceptor cell (rod cell)

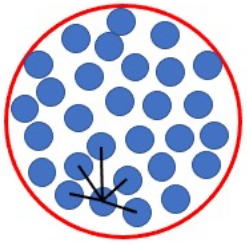
AFM of outer shape of disc with the rhodopsin protein from rod cell



Distance between rhodopsin centers 38 Å.
Rhodopsin molecule size is 35 Å.



Photoreceptor membrane model (fragment)



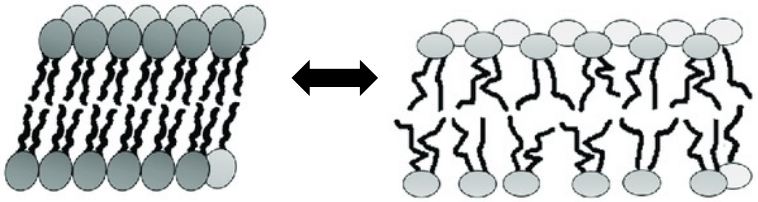
The average distance between the rhodopsin molecule centers is 58 Å

Feldman, T.B., Ivankov O.I., Kuklin A.I., Murugova T.N., Yakovleva M.A., Smitienko O.A., Kolchugina I.B., Round A., Gordeliy V.I., Belushkin A.V., Ostrovsky M.A., *Small-angle neutron and X-ray scattering analysis of the supramolecular organization of rhodopsin in photoreceptor membranes.* Biochimica et Biophysica Acta **(BBA) - Biomembranes, 2019**.

Anomalous swelling effect

Gel phase ($L_{\beta'}$)

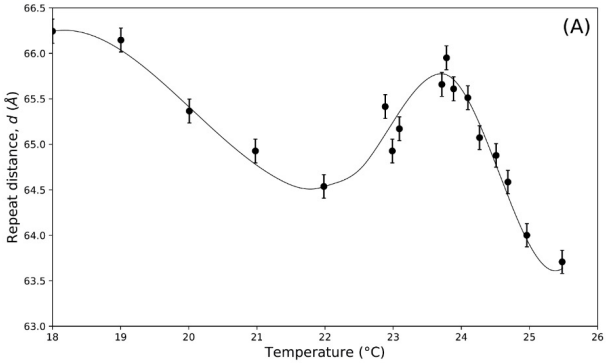
Fluid phase (L_{α})



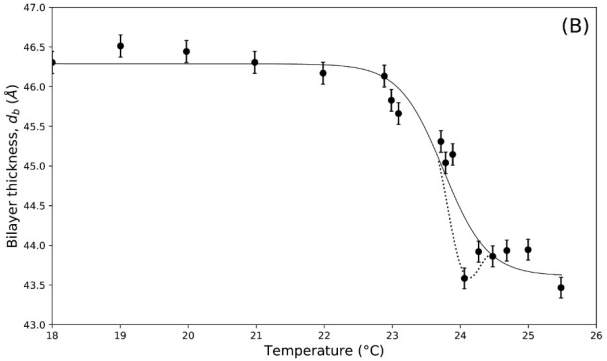
T_m – *Main phase transition temperature*
T^* – Spinoidal point

$(T_m - T^*)/T_m \simeq 10^{-2}$

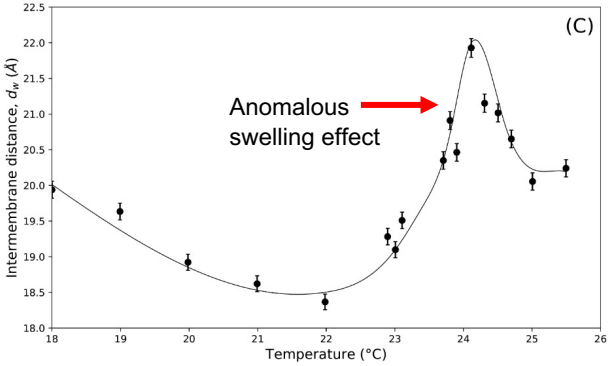
Anomalous swelling effect is not coupled to the formation of a ripple phase and occur at the spinoidal point T^*, which is close, but do not coincide with the main phase transition temperature T_m



DMPC MLVs in D_2O



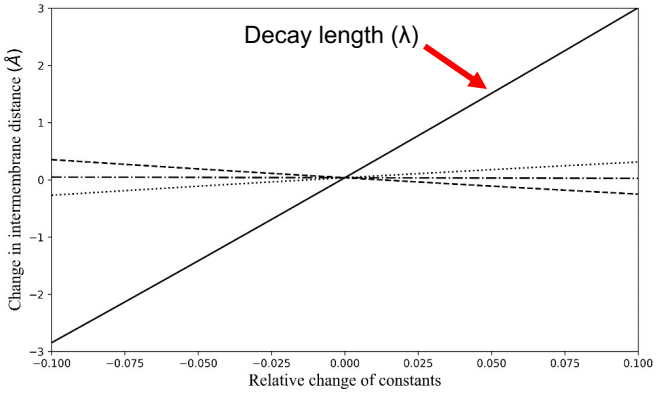
DMPC ULVs in D_2O



Intermembrane distance

A Kuklin, D Zabelskii, I Gordeliy, J Teixeira, A Brûlet, V Chupin, Vadim Cherezov, Valentin Gordeliy. On the Origin of the Anomalous Behavior of Lipid Membrane Properties in the Vicinity of the Chain-Melting Phase Transition. **Sci Rep 10, 5749 (2020).**

Anomalous swelling effect
Conclusions



Predicted changes in the intermembrane distance induced by variations in the decay length (λ), a Hamaker constant (H), short range repulsion pre-exponential factor (P_0) and bending rigidity (K_c) plotted as solid, dashed, dotted and dash-dotted lines respectively

Balance of forces:

$$P_{srr} + P_{und} = P_{VdW}$$

$$P_{srr} = P_0 e^{-d_w/\lambda} \qquad P_{und} = \frac{3\pi^2 (kT)^2}{128 K_c d_w^3}$$

$$P_{VdW} = \frac{H}{6\pi}\left(\frac{1}{d_w^3} - \frac{2}{(d_w + d_b)^3} + \frac{1}{(d_w + 2d_b)^3}\right)$$

Our calculations show that, despite the fact that the bending rigidity depends strongly on the membrane thickness, the reduction of the membrane thickness **cannot completely account for the change of the membrane bending elasticity.**

Thus, critical density fluctuations lead to a more disordered lipid bilayer and therefore to the reduction of the bending rigidity to its softening by a factor of 1.5 as it is observed in the experiment.

A mere 6% increase in the value of λ can ensure that the theoretical estimate matches the measured increase in the intermembrane distance.

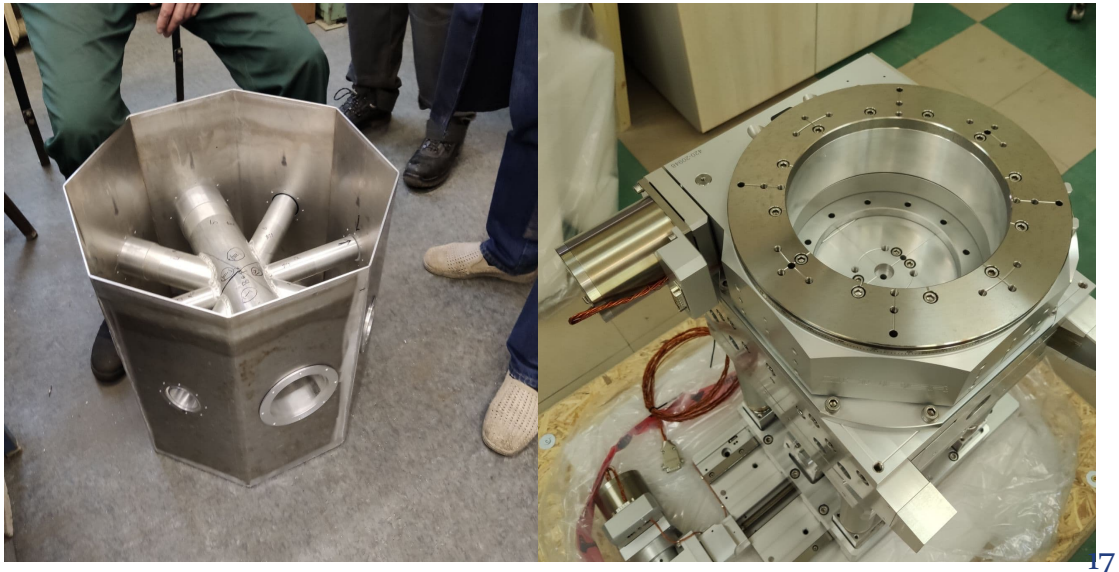
A Kuklin, D Zabelskii, I Gordeliy, J Teixeira, A Brûlet, V Chupin, Vadim Cherezov, Valentin Gordeliy. On the Origin of the Anomalous Behavior of Lipid Membrane Properties in the Vicinity of the Chain-Melting Phase Transition. **Sci Rep 10, 5749 (2020).**

New adjustable collimator for the YuMO spectrometer

Spectrometer upgrade:

- adjustable collimator
- direct beam detector
- scattering detectors
- reconstruction of the collimating base

Photographs of the new adjustable collimator and the goniometrical part.



Sample preparation room

Responsible: T. Murugova

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Thank you for your attention!

25 samples holder with connected Lauda liquid thermostat