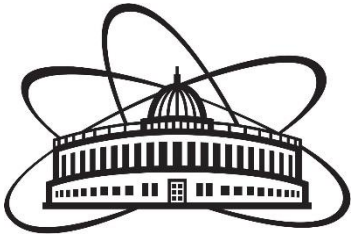
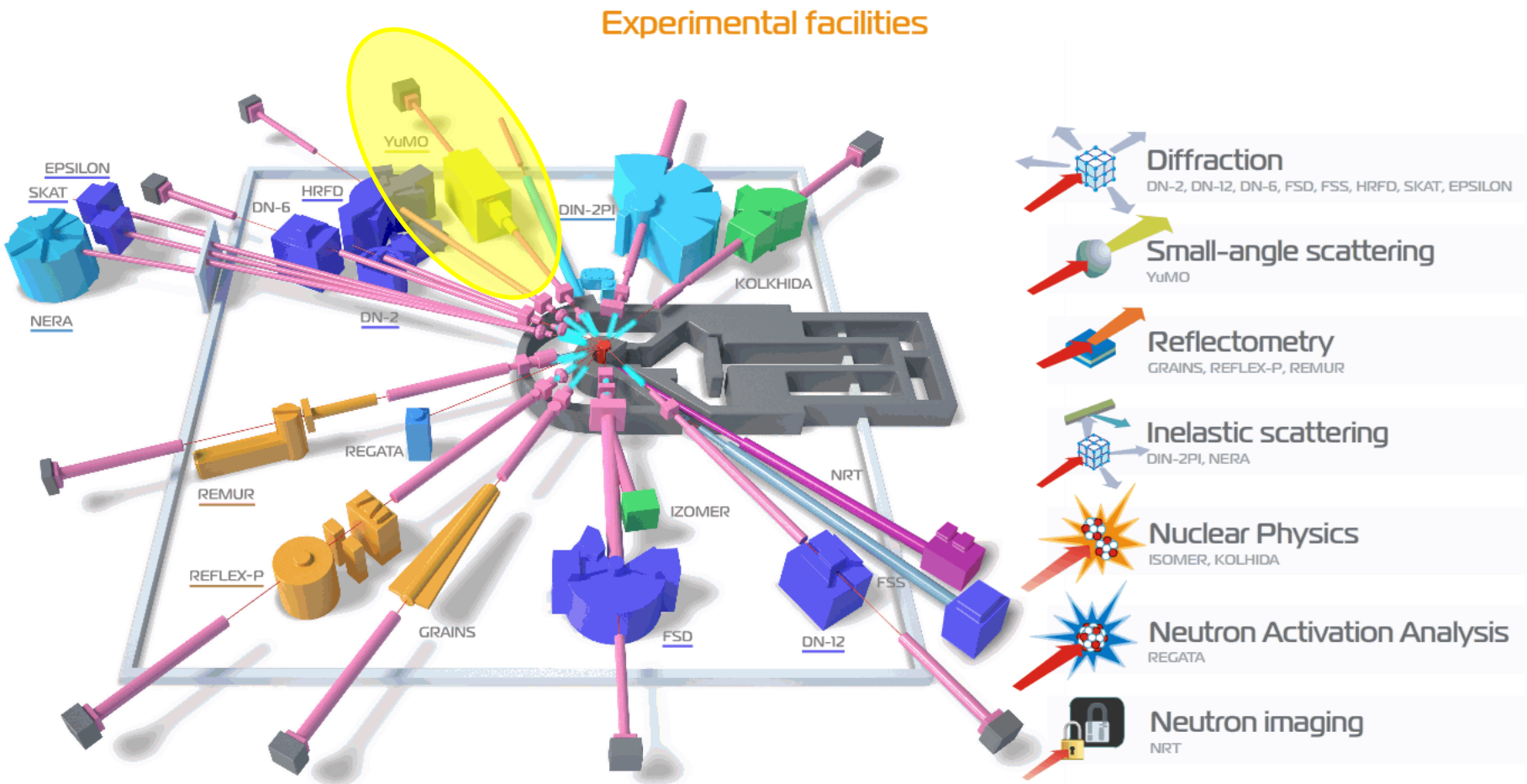
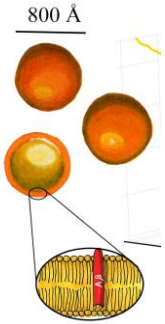
Small Angle Neutron Scattering at the IBR-2 and Science

O.I. Ivankov*, T.N. Murugova, A.Kh. Islamov, A. Nabiev, S.A. Kurakin, A.V. Rogachev, V.V. Skoi, A.H.A. Elmekawi, M. Balasoiu, Yu.S. Kovalev, Yu.L. Ryzykau, A.V. Vlasov, A.G. Soloviev, V. Gordeliy, N. Kucerka, and A. I. Kuklin



Suite of Spectrometers





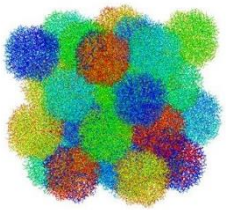
Small Angle Scattering

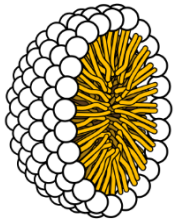
- Biology
- Colloidal chemistry
- Material science
- Solid state physics
- Polymers physics

Structural information:
10-1000 Å

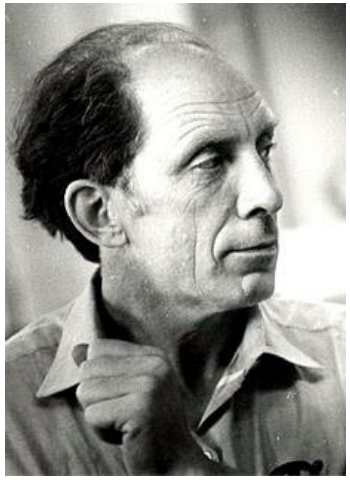
- Size, volume, molecular weight
- Shape
- Conformation
- Oligomeric state
- 3D structures with resolution ~10 Å

- Macromolecules and it aggregates in solvents
- Alloys
- Films
- Powders

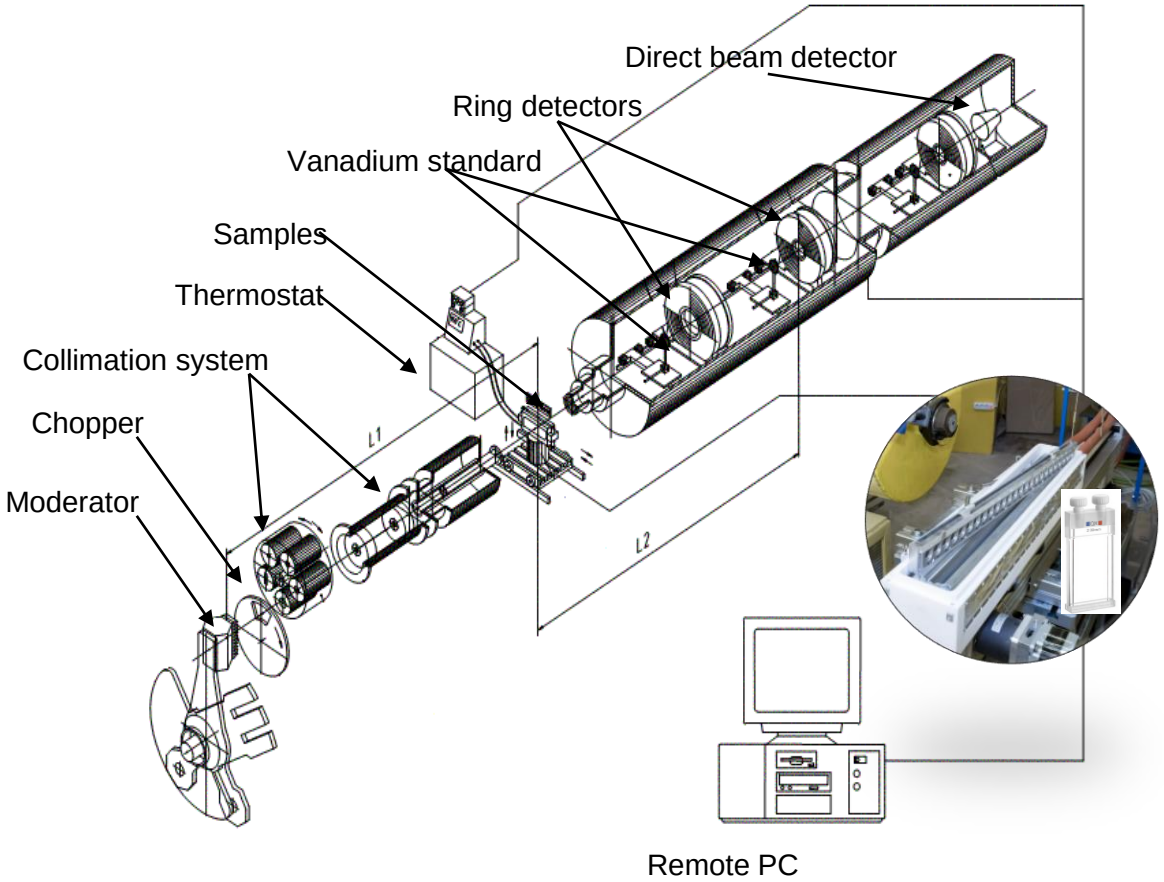




YuMO Spectrometer



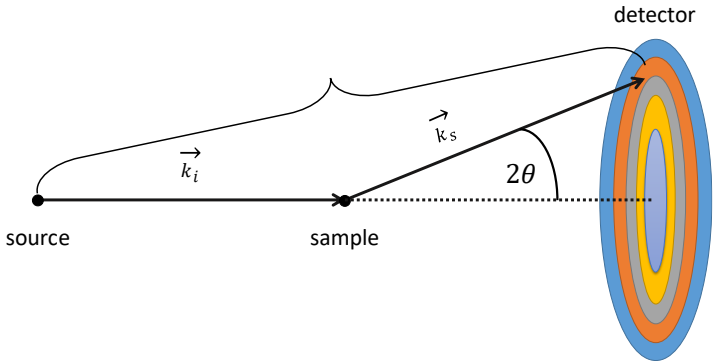
Yuri
Mechislavovich
Ostanevich

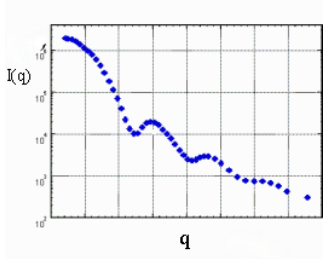


В.А.Вагов, А.Б.Кунченко, Ю.М.Останевич, И.М.Саламатин, Установка малоуглового рассеяния нейтронов по методу времени пролета на импульсном реакторе ИБР-2// Сообщения ОИЯИ, Р14-83-898, Дубна, 1983.

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.

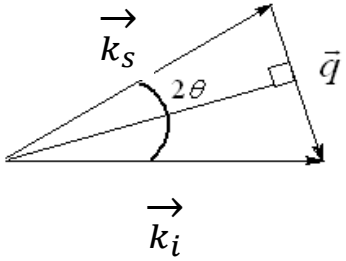
SANS experiment scheme





Scattering curve

$$I(q) = \frac{d\sigma}{d\Omega} = \iint_V \rho(\vec{r}_i)\rho(\vec{r}_k)\frac{\sin q|\vec{r}_i - \vec{r}_k|}{q|\vec{r}_i - \vec{r}_k|} d\vec{r}_i d\vec{r}_k .$$



Scattering vector q

$$\vec{q} = \vec{k}_i - \vec{k}_s$$

$$\lambda = const$$

or

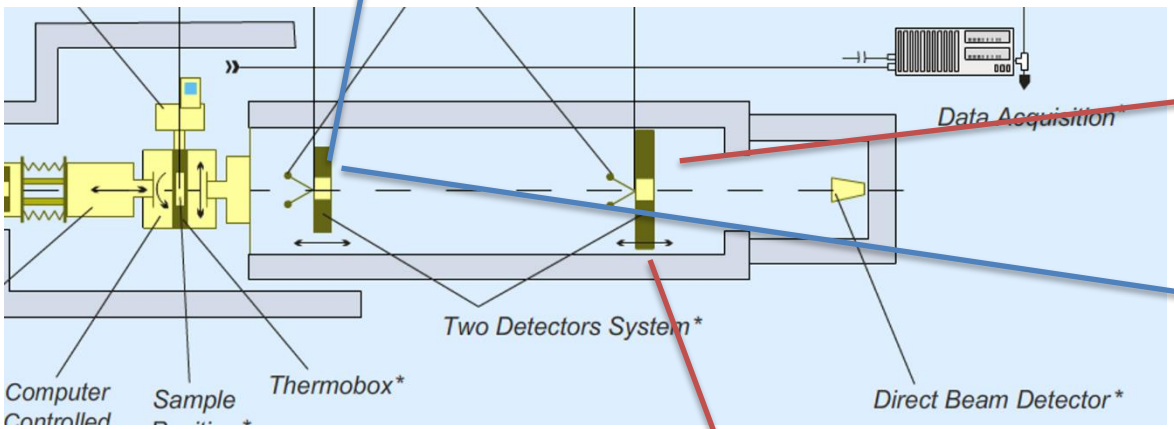
$$q = 2k\sin\theta = \frac{4\pi}{\lambda}\sin\theta$$

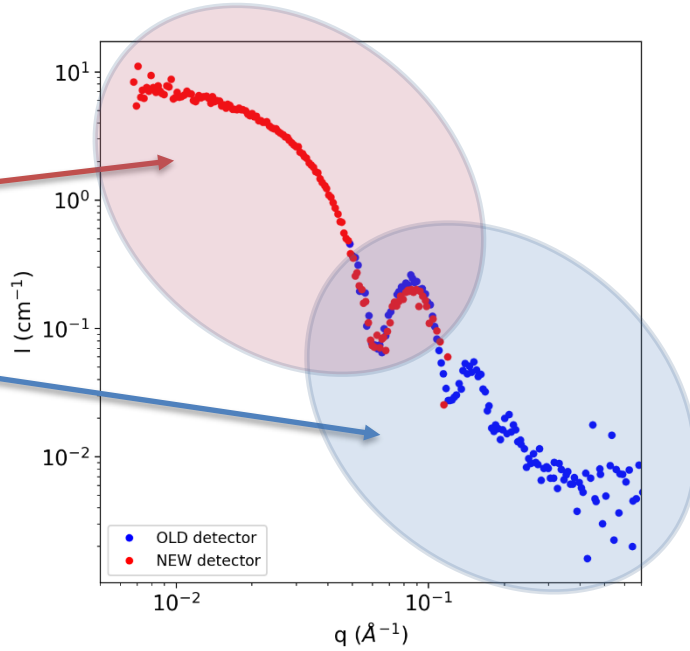
$$\lambda = \frac{h}{mv} = \frac{t \cdot h}{m \cdot L}$$

Two-detector system of YuMO

1978 y.

Б.Н.Ананьев, А.Б.Кунченко, В.И.Лазин, Ю.М.Останевич, Е.Я.Пикельнер, Кольцевой многонитевой детектор медленных нейтронов с гелием-3 // Сообщения ОИЯИ, 3-11502, Дубна, 1978.



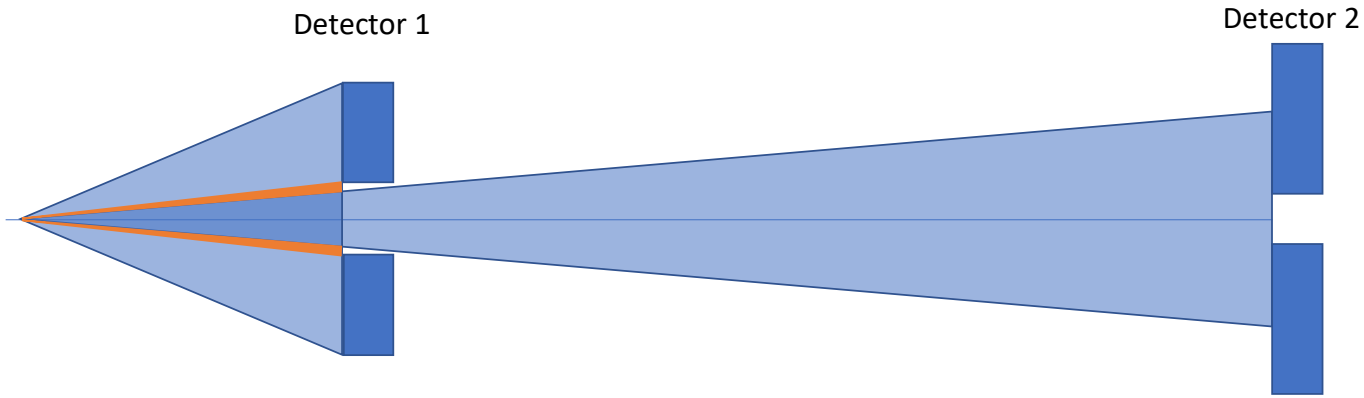


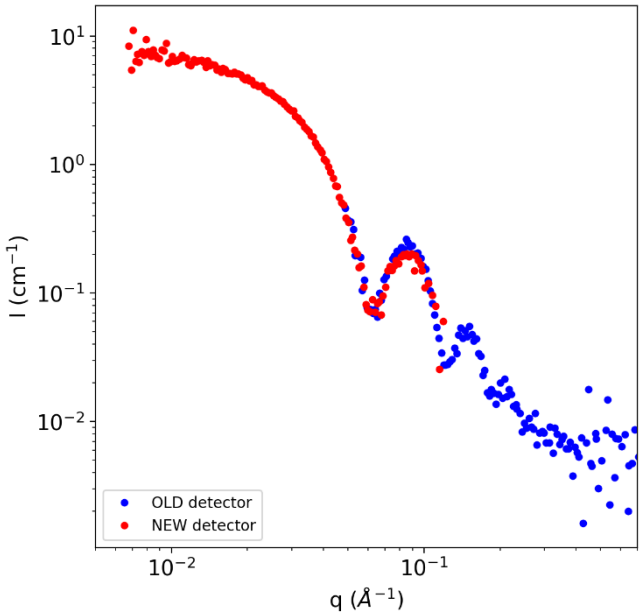
1983 y.

$$\frac{q_{max}}{q_{min}} = 100$$

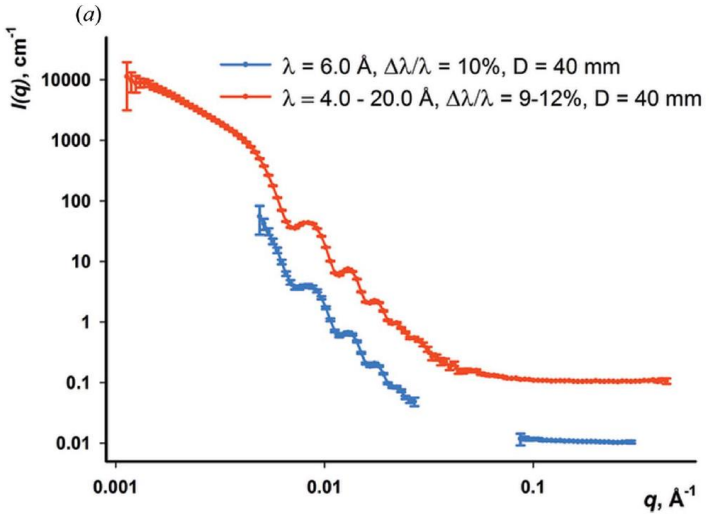
* For the thermal neutrons spectra

Two-detector system of YuMO





YuMo spectrometer



Spectrometer BILBY (ANSTO, Australia)

Anna Sokolova et al., Performance and characteristics of the **BILBY** time-of-flight small-angle neutron scattering instrument // *J. Appl. Cryst.* (2019). **52**, 1-12

Raw spectra data treatment

$$I(\theta,\lambda) = I_0(\lambda)\cdot\frac{d\Sigma}{d\Omega}\cdot\epsilon(\lambda)\cdot e^{-\Sigma_t(\lambda)d_s}\cdot\Omega_s + B(\theta,\lambda)$$

Incident beam

Differential cross section

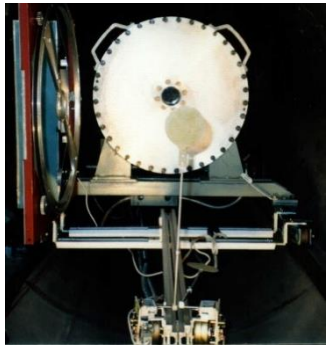
Detector effectivity

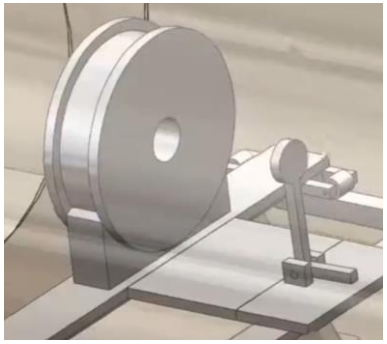
Sample transmission

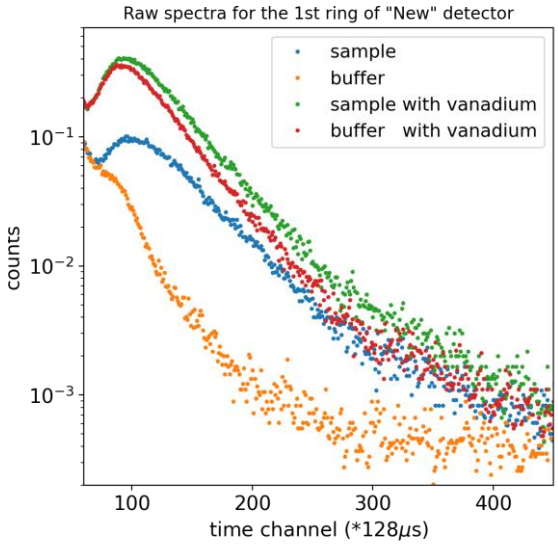
Solid angle

Background

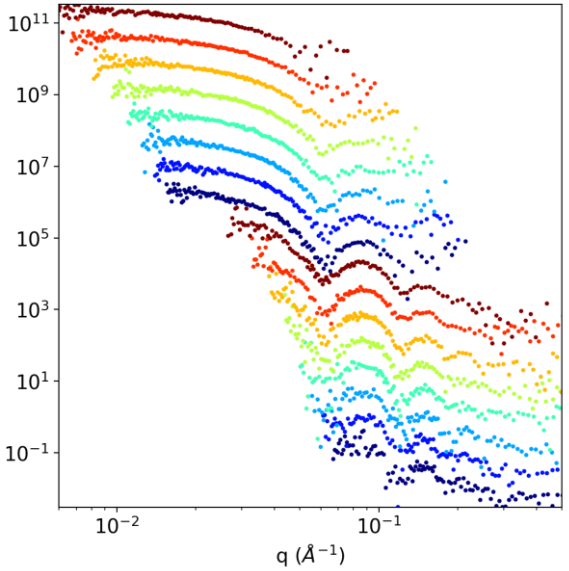
$$\frac{d\Sigma}{d\Omega} = \frac{I_{sample} - I_{buffer}}{I_{sv} - I_{sample}}\cdot\frac{\Omega_v}{\Omega_s}\cdot T_v\cdot d_v\cdot\left(\frac{d\Sigma}{d\Omega}\right)_v\cdot\frac{1}{d_s}$$

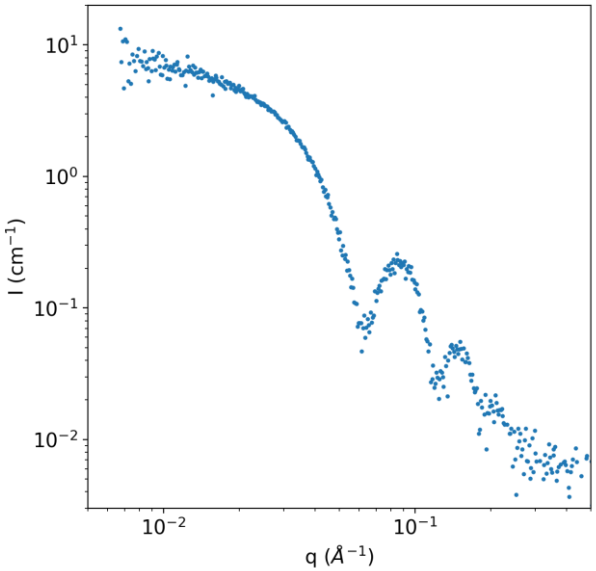




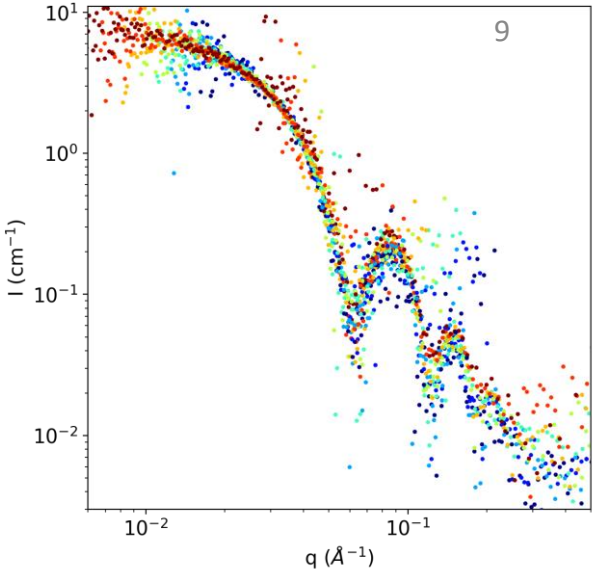


$$\frac{d\Sigma}{d\Omega} = \frac{I_{sample} - I_{buffer}}{I_{sv} - I_{sample}} \cdot \frac{\Omega_v}{\Omega_s} \cdot T_v \cdot d_v \cdot \left(\frac{d\Sigma}{d\Omega}\right)_v \cdot \frac{1}{d_s}$$

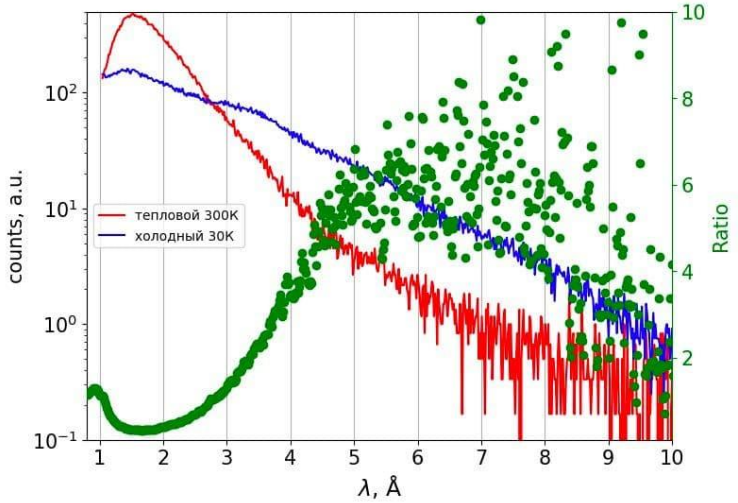




Averaging



Cold moderator

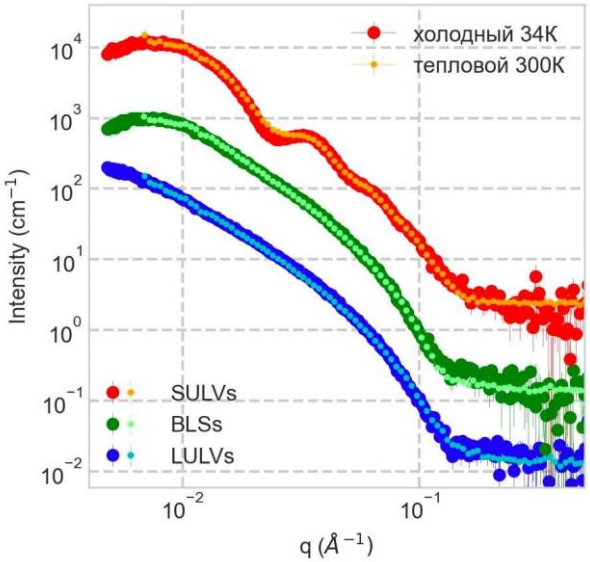


Comparison of the counts on the detectors with the cold moderator at ~ 300K and ~34K

gain factor wavelength

(1Å-3Å):	0.47
(1Å-6Å):	0.65
(1Å-8Å):	0.67
(1Å-10Å):	0.67
(3Å-10Å):	3.02

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



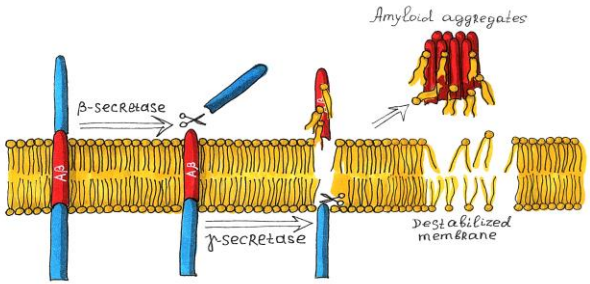
Dynamic q-range

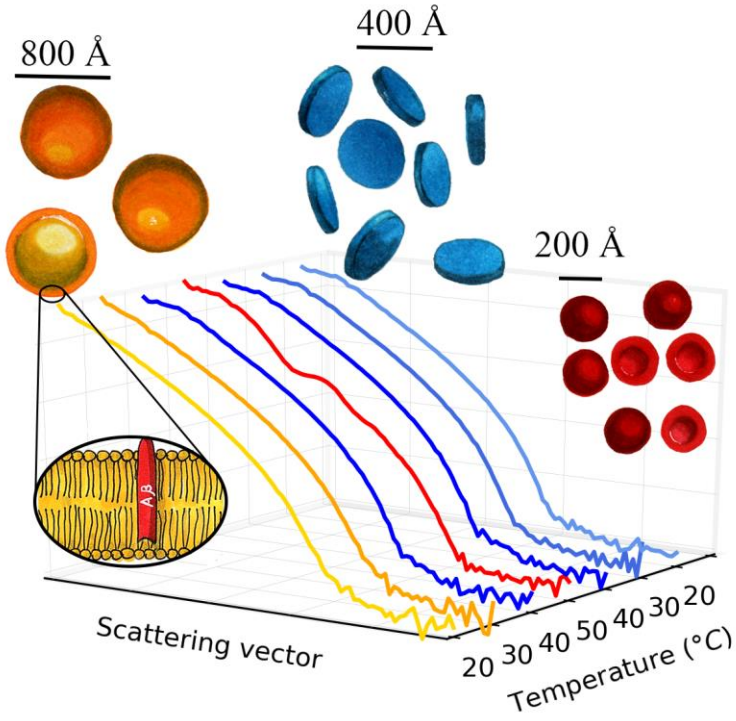
$$\frac{q_{max}}{q_{min}} = 140$$

Parameters of YuMO spectrometer

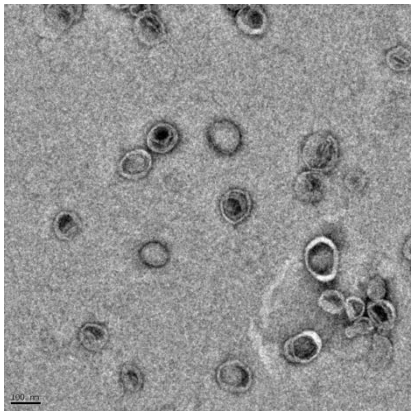
Flux on the sample (thermal neutrons)	10^7– 4x10^7 n/(s cm^2)
Used wavelength	0.5 Å to 8 Å (10Å with cold moderator 30K)
Q-range	0.007 – 0.7 $Å^{-1}$ (0.005-0.7 $Å^{-1}$ cold moderator)
Dynamic Q-range	q_{max}/q_{min} up to 100 (140 cold moderator)
Specific features	Two detectors system, central hole detectors
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	He^3 -filed, in-house design, 8 independent wires
Detector (direct beam)	^{6}Li-convertor (in-house design)
Sample changer	In-house designed box, in air
Q-resolution	5-15%
Temperature range	4^{o}C - + 70^{o}C (standard Hellma cells, 1mm, 2 mm) -20^{o}C - + 130^{o}C (in-house design sample holders)
Number of the samples in the automated sample changer	25
Average single data collection time	1 h
Reactor pulse frequency	5 Hz

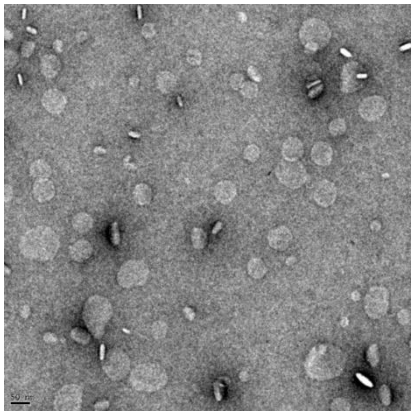
Interactions in Disease Modeling Membranes





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**.

Ivankov O., Murugova T.N., ..., Kučerka N., *Scientific Reports* (2021).

Kinetics system for YuMO spectrometer

Time of measurement – 2 min

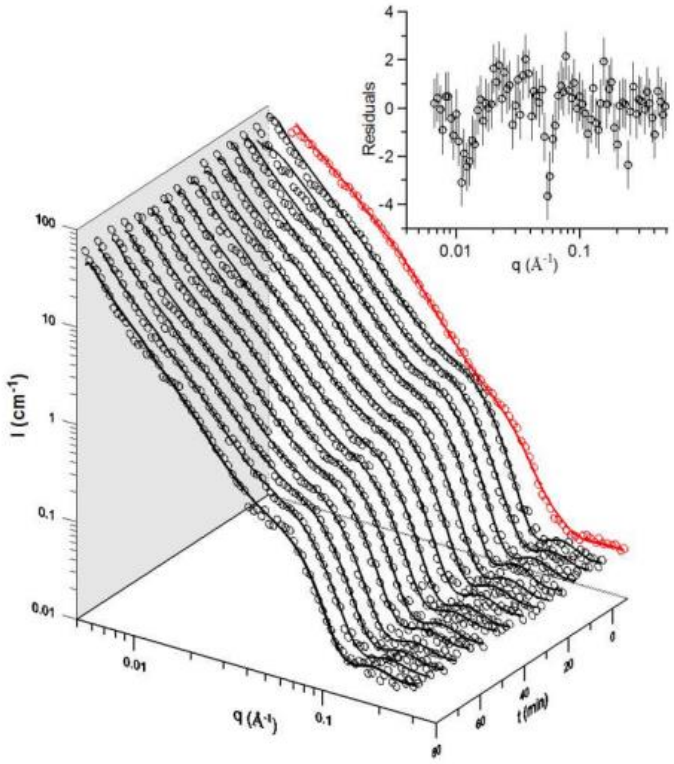


Figure 4. Dependences of SANS intensity $I(q)$ on scattering vector q for DNA – $C_{12}NO$/DOPE dispersion as a function of time; prior (red points) and after DCl injection. Full lines show fits using a paracrystal lamellar model.
Inset: An example of the distribution of residuals.

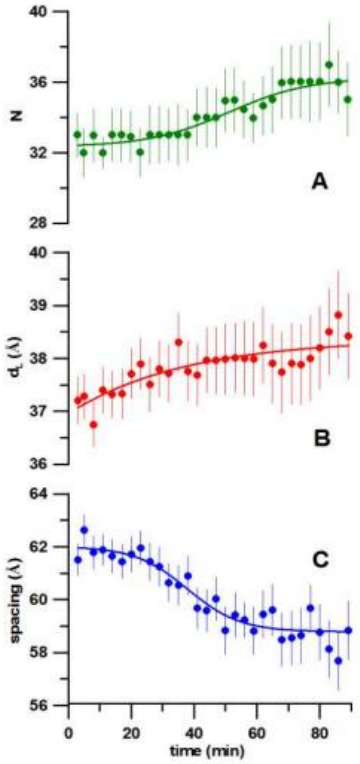
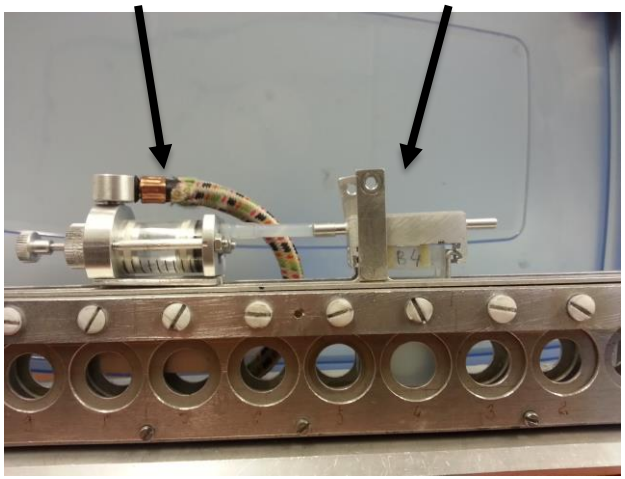


Figure 5. Time dependence of the structural parameters: the number of layers (A), the lipid bilayer thickness (B) and the spacing (C).

Container for the sample to be injected

Standard cuvette

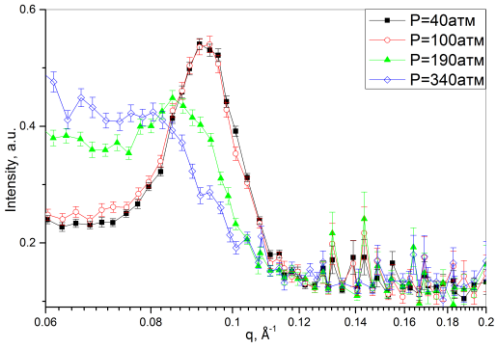


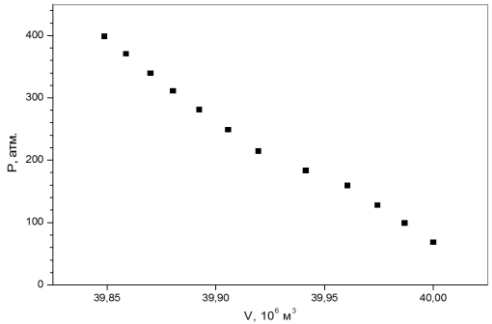
Samples holder

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

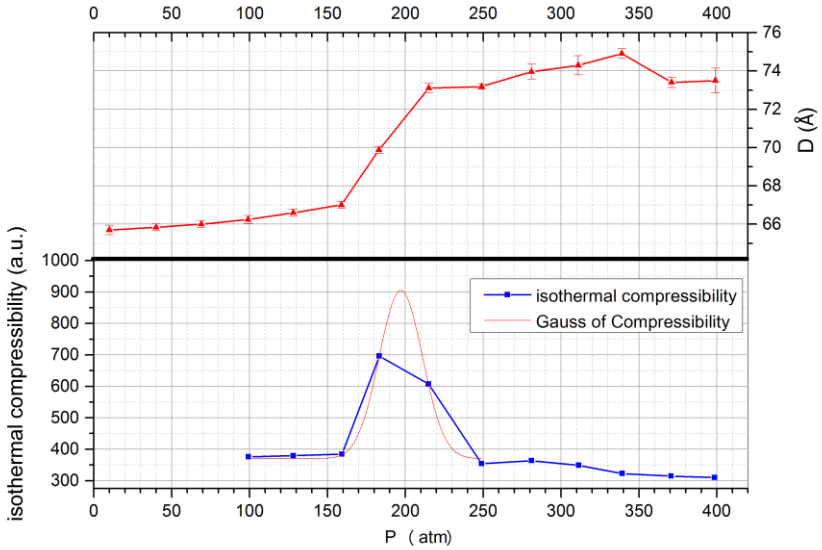
Kinetics system was developed and manufactured in Commenius University of Bratislava, Slovakia

Volumetric setup for YuMO spectrometer





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



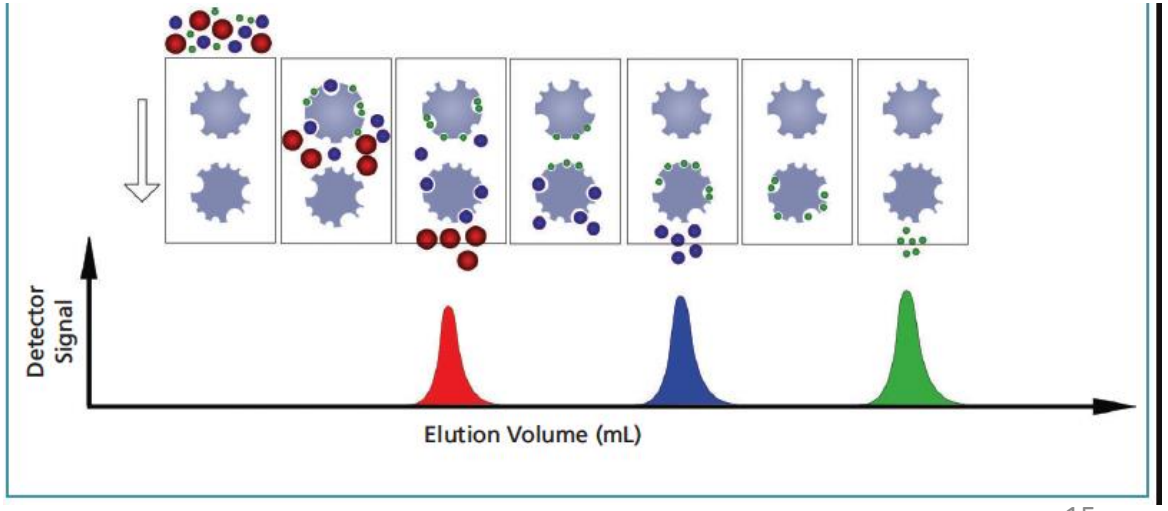
Soloviov, D., et al., *Changes in the Area per Lipid Molecule by P–V–T and SANS Investigations.* Macromolecular Symposia, 2014. **335**(1): p. 58-61.

Size-exclusion chromatography (SEC)

Applications:

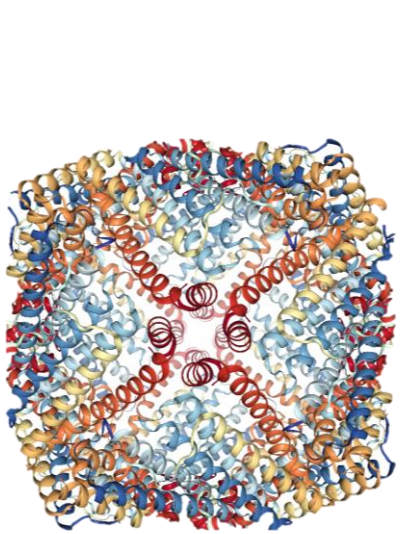
- Separation of macromolecules from complex mixtures according to their size, charge, selective non-covalent interaction and other properties.
- Protein and polimers purification.
- Affinity-tagged protein purification.
- Desalting and buffer exchange.
- Identification and quantitation of macromolecules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

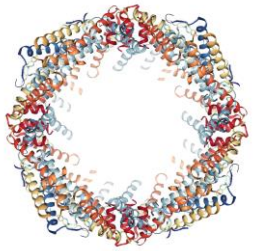
Funding:
- RSF Grant (Kucerka N.)
- Department of Spectrometers Complex
  IBR-2 (Kulikov S., Bodnarchuk V.)
- JINR-Poland Grant (Kuklin A.)

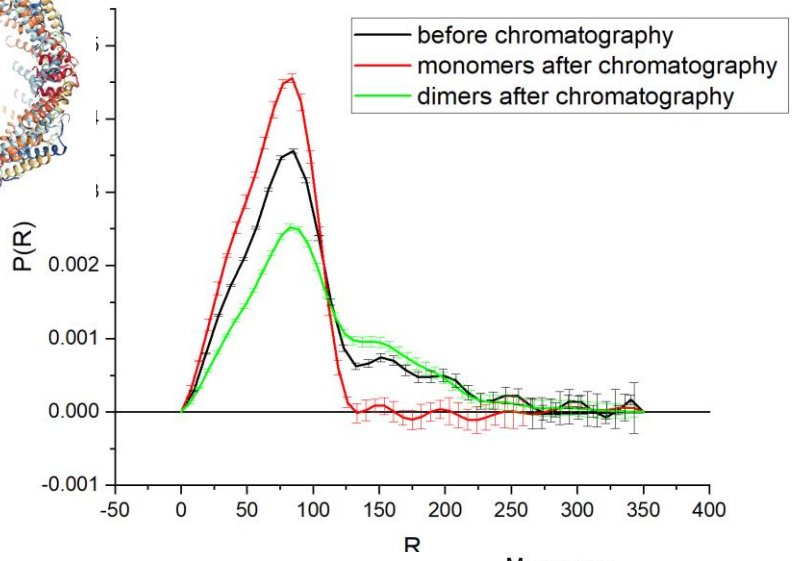


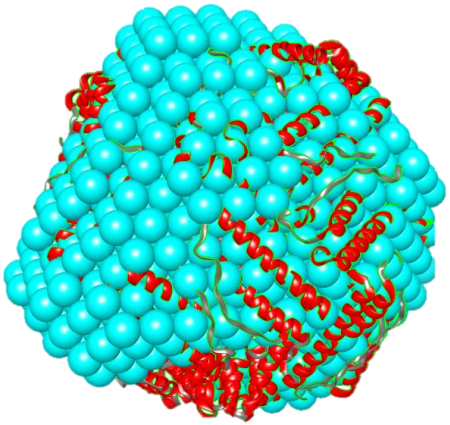
Implementation of SEC on YuMO spectrometer

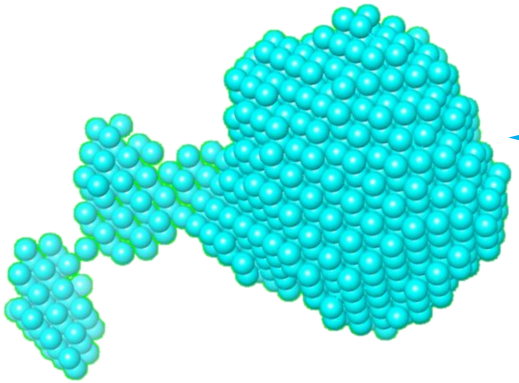
Apoferritin protein

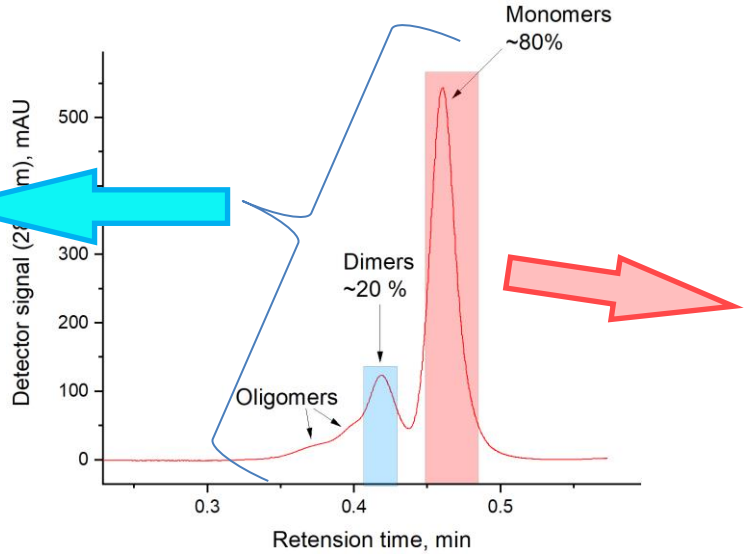


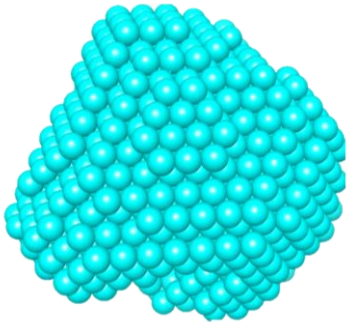




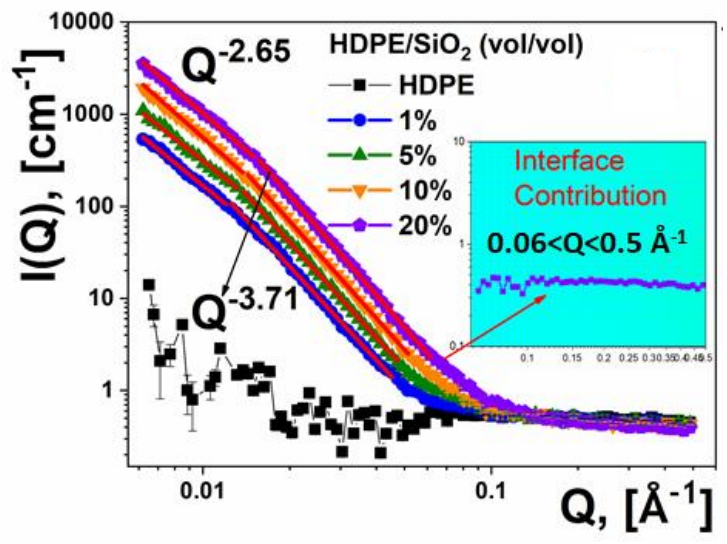


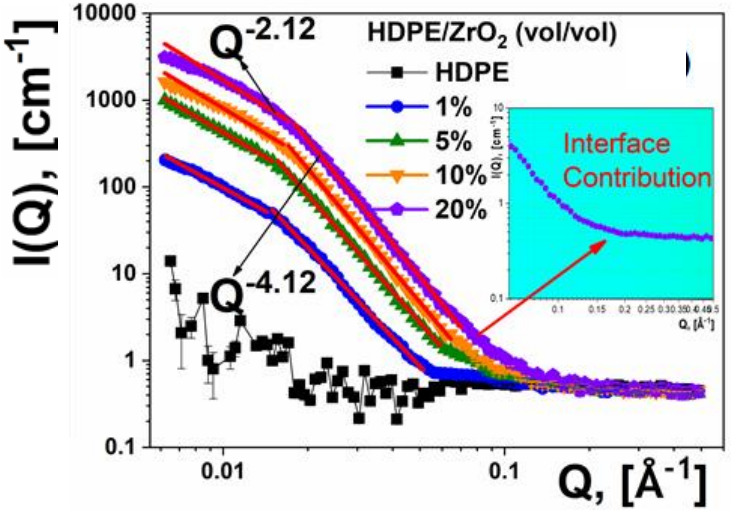


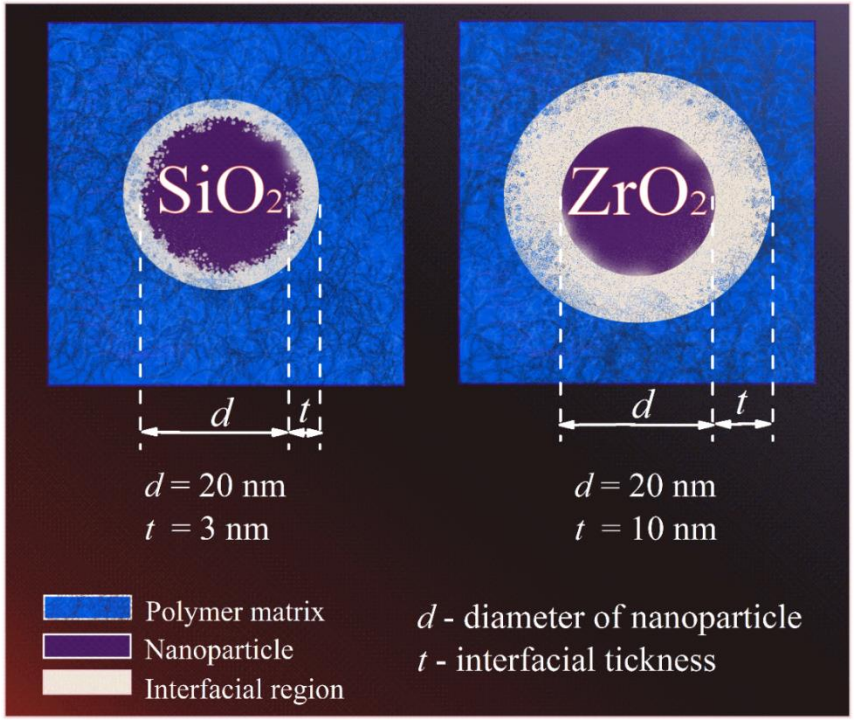




Interfacial effects in polymer-nanoparticle composite films by small-angle neutron scattering method



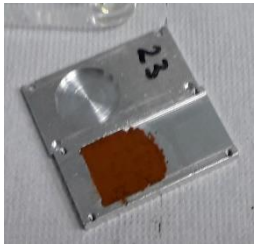


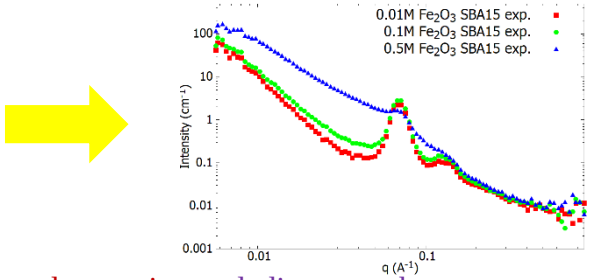


Nabiyev, A.A.; Olejniczak, A.; Islamov, A.K.; Pawlukojc, A.; Ivankov, O.I.; Balasoiu, M.; et al. Composite Films of HDPE with SiO2 and ZrO2 Nanoparticles: The Structure and Interfacial Effects. Nanomaterials 2021, 11, 2673. https://doi.org/10.3390/nano11102673

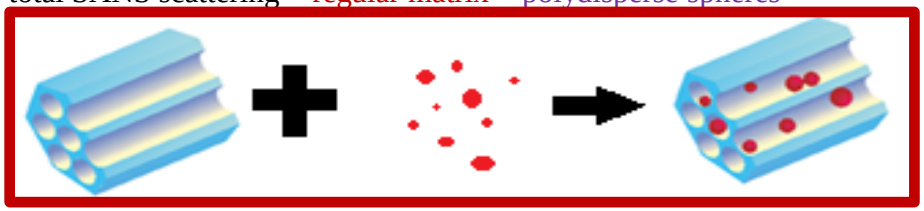
Nanopores for Magnetic and Biomedical Applications

SANS experiments at YuMO





total SANS scattering = regular matrix + polydisperse spheres



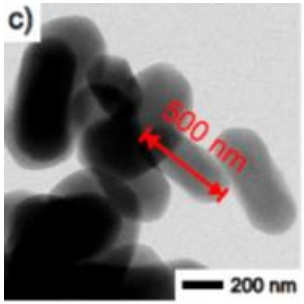
$$I(q) = K_C S(q)|F_C(q)|^2 + K_S|F_S(q)|^2 + I_d(q) + I_i$$

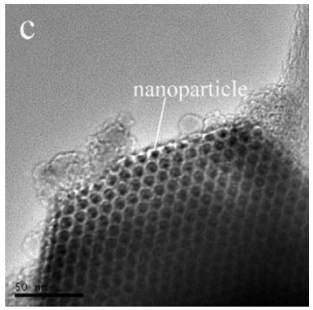
- pores - size & mutual distance
- NPs - size distribution & concentration

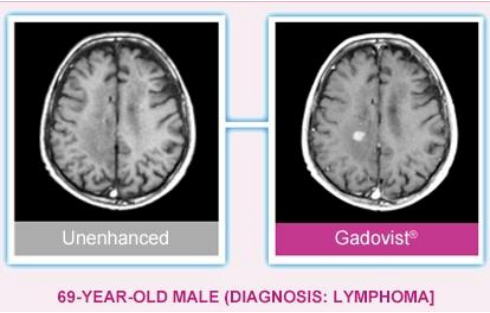
Material

Periodic nanoporous silica

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

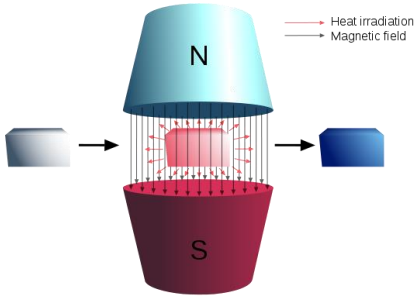






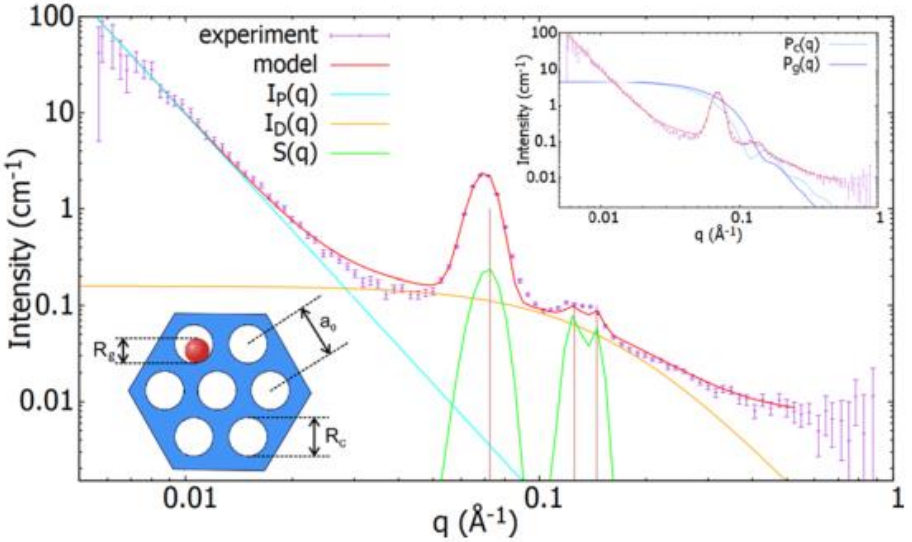
Applications

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



Size and distribution of the iron oxide nanoparticles in SBA-15 nanoporous silica via SANS study

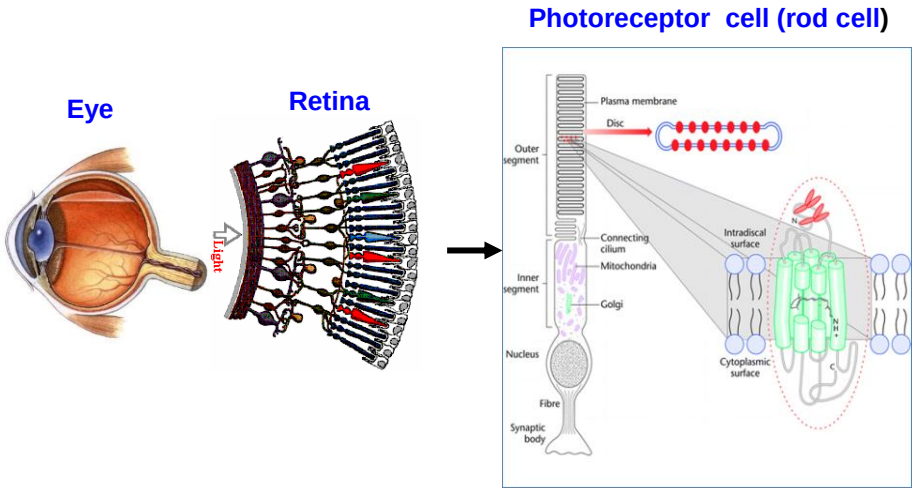
Illustration of the Fe2O3@SBA-15 nanocomposite preparation and structure organization. Polydisperse nanoparticles of Fe_2O_3 are randomly distributed in the longitudinal cylindrical pores of perfect hexagonal order within silica matrix.

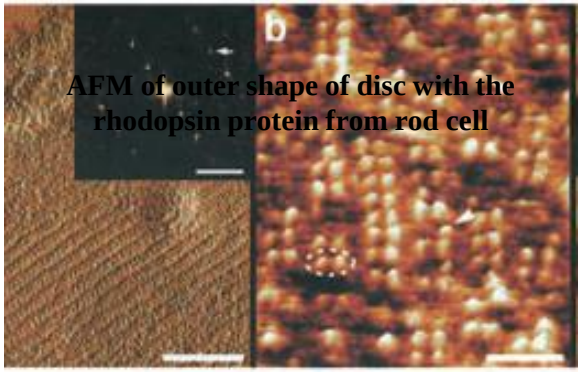


Fit of the model to the experimental SANS data corresponding to nanocomposite Fe2O3@ SBA-15 (1) with very sparse occupation of the pores by nanoparticles

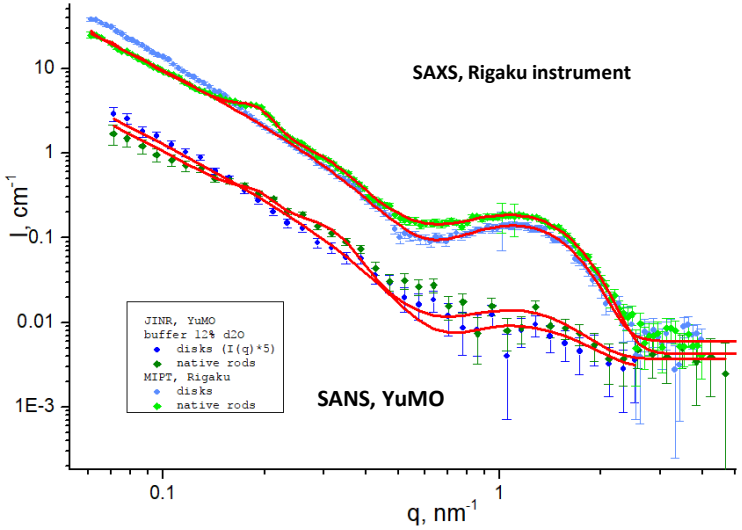
Zeleňáková, A., Hrubovčák, P., Kapusta, O., Kučerka, N., Kuklin, A., Ivankov, O., & Zeleňák, V. (2019). Size and distribution of the iron oxide nanoparticles in SBA-15 nanoporous silica via SANS study. **Scientific Reports, 9(1), 1-9.**

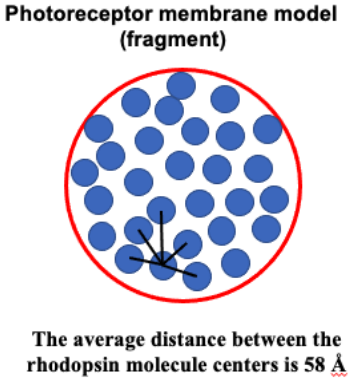
Supramolecular organization of rhodopsin





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



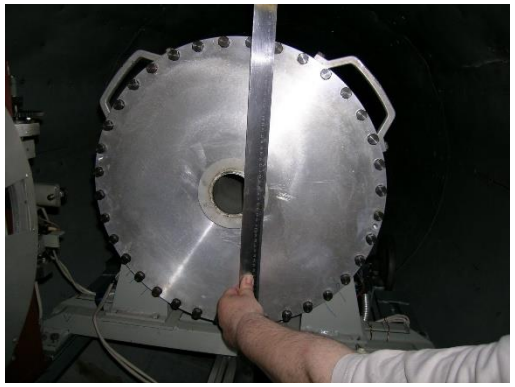


Feldman, T.B., Ivankov O.I., Kuklin A.I., Murugova T.N., Yakovleva M.A., Smitienko O.A., Kolchugina I.B., Round A., Gordeliy V.I., Belushkin A.V., Ostrovsky M.A., *Small-angle neutron and X-ray scattering analysis of the supramolecular organization of rhodopsin in photoreceptor membranes.* Biochimica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

Magnetic system

Main parameters:

- Magnetic field: up to 2 T.
-
- 2 rotary planes
- 2 replaceable poles with the ability to change the distance between poles
- The gap is up to 130 mm.
- Weight about 2 tons.



THANK YOU FOR YOUR ATTENTION!