Neutronographic investigations of supramolecular structures on upgraded small-angle spectrometer YuMO

A I Kuklin\textsuperscript{1,2*}, A V Rogachev\textsuperscript{1,2}, D V Soloviov\textsuperscript{1,2,3}, O I Ivankov\textsuperscript{1,2,3}, Yu S Kovalev\textsuperscript{1,2}, P K Utrobin\textsuperscript{2}, S A Kutuzov\textsuperscript{1}, A G Soloviev\textsuperscript{1}, M I Rulev\textsuperscript{1,4} and V I Gordeliy\textsuperscript{1,2,5}

\textsuperscript{1} Joint Institute for Nuclear Research, Dubna, Russia
\textsuperscript{2} MIPT, Dolgoprudny, Russia
\textsuperscript{3} Institute for Safety Problems NPP, Chernobyl, Ukraine
\textsuperscript{4} Lomonosov Moscow State University, Moscow, Russia
\textsuperscript{5} Institute Biological Structure, Grenoble, France
E-mail: kuklin@nf.jinr.ru

Abstract. The work is a review of neutronographic investigations of supramolecular structures on upgraded small-angle spectrometer YuMO. Here, key parameters of small-angle spectrometers are considered. It is shown that two-detector system is the basis of YuMO upgrade. It allows to widen the dynamic q-range twice. In result, the available q-range is widened and dynamic q-range and data collection rate are doubled. The detailed description of YuMO spectrometer is given. The short review of experimental researches made on the spectrometer in the polymers field, biology, material science and physical chemistry is given. The current investigations also have a methodological aspect. It is shown that upgraded spectrometer provides advanced world level of research of supramolecular structures.

1. Introduction
In 2012 the first work about scientific orientation of small-angle spectrometer (SANS – MURN, JINR, Dubna) celebrated its fortieth anniversary [1]. Authors considered many methodical aspects of small-angle scattering: contrast variation, homogeneous and heterogeneous particles and deuterium exchange. Average scattering densities are shown for different proteins and RNA. Biological orientation of YuMO was obvious for the authors. Despite the success in proteins crystallization and synchrotron science in recent decades defines the trend in structural biology, there are still a number of problems connected with the native state of proteins and the importance of SANS for them is difficult to over-estimate. At the same time, other areas of research arose and some of which are presented in this short review.

In recent years the spectrometer was significantly upgraded and got a name “YuMO” in honor of one who created the spectrometer ideology (Yuri Mechislavovich Ostanevich). In this work, some methodical and scientific aspects of research with the help of small-angle neutron scattering on example of investigations on the small-angle neutron spectrometer on pulsed reactor IBR-2 (after 2011 IBR-2M) are shown.
Investigation of supramolecular objects acquires more relevance with regard to the intensive development of nanotechnologies and corresponding scientific tasks [2, 3]. Also the requirements to the methods of investigations of substances on nanoscales are increasing.

Small-angle scattering of thermal neutrons method is one of the major means of structural characteristics determination for condensed matter in range 2 – 100 nm. Small-angle spectrometer YuMO on the 4th channel of reactor IBR-2, JINR, Dubna is almost the only spectrometer of this type that is systematically and efficiently used for condensed matter research in different organizations all over the Russia and membership countries of JINR.

As a rule in leading world centers there are two or more spectrometers of this type. Such number of spectrometers is connected with big amount of users of small-angle neutron scattering. Subsequently it is defined by the importance of structural information obtained by the method and the wide spectrum of tasks it solves – from material sciences to structural biology [4].

In the review mentioned above [1] the authors have seen biological objects as the main field of research on the small-angle spectrometer. Mentioned in the article the detailed consideration of small-angle scattering is still relevant. In fact, the questions of H₂O/D₂O contrast variation with isotopic exchange taking into account are considered and formula of average scattering lengths calculation for ribosome proteins, RNA and DNA are shown. Authors used research experience in the world and in Laboratory for Neutron Physics of the Joint Institute for Nuclear Research. When considering questions about high-resolution method for protein crystals investigation, SANS is pointed out as non-destructive method. However, as practice showed, the main disadvantage of neutron sources for high-resolution neutronography is their weak flux. High fluxes are also the key features for small-angle spectrometers.

Small-angle spectrometers belong to the class of installations with low-resolution. Because of the small-angles of scattering the main contribution into resolution is usually made not by the time parameter (that is limited for IBR-2 by the neutron pulse width), but by the angular resolution that is defined by collimation and the size of the minimal sensible element of the detector part of spectrometer. This statement is also true for other pulse sources. There is another situation for stationary reactors working practically with the only one wavelength (more precisely – with very narrow neutron wavelengths range) and using mechanical velocities selector for monochromatization of neutrons.

In order to have satisfactory wavelength resolution one has to reduce the neutron flux on the sample. Usually the resolution is lower than 5% (under adequate flux decrease) in case one does not use perfect crystal (usual resolution for wavelength selector is approximately 10% [5]). In result under the same resolution neutron fluxes on the sample on stationary reactors and pulse sources become equal and on IBR-2 with comparably low average power (2 MW) neutron fluxes are comparably high [6]. Subsequently the data collection rate can also be high.

In 2013 small-angle spectrometer on the 4th channel of IBR-2 celebrated its thirtieth anniversary [7]. It was the first time the installation got through the drastic upgrading in 1990, which was connected with personal computers rapid development. This allowed to make significant spectrometer management automatization even in comparison with contemporary situation. The basic progress was the concept of measuring scattering curve in absolute scale at the feasibly shortest time. This allowed obtaining interesting results in different fields of condensed matter physics [8-17]. New paradigm of installation involved flexibility when making an upgrade and preserving the existing instrument advantages. In 2000th was done modernization in basis of it is two-detector system [17-20].

Small-angle spectrometer with direct geometry (the sample “is oriented” to the zone) is more suitable to fast pulsed reactor IBR-2 with current length of starting “pulse” (nearly 300 us). The instrument upgrade (first of all usage of two-detector system) was based on maximization of advantages of method of neutron detection versus their time of flight on pulse neutron source. It was the use of the full spectrum of
thermal neutron wavelengths, which ensured the overlap of the spectra of small-angle neutron scattering obtained from two detectors.

2. Upgraded small-angle neutron scattering spectrometer

With a significant variety of small-angle spectrometers all over the world the key parameters are: intensity and density of the flux on a sample, [21], q-range and dynamic q-range (the range of q-vectors that is available simultaneously [22, 23], that is defined by the ratio $q_{\text{max}}/q_{\text{min}}$) and resolution. Upgrading of these key parameters is that was done on the spectrometer YuMO. The opportunities of sample investigation were broadened as to allow studying samples under pressure, in external magnetic field and under light illumination [24]. In addition, we did optimization of spectrometer configuration and created user-friendly interface for experiments management and data treatment for spectrometer users.

In [19, 20, 25] there is a description of new software and in [26] – details of nodes of automated spectrometer YuMO. Contemporary methods of smoothing while treating experimental data for obtaining SANS curve with spectrometer resolution taken into account are given in [27]. In [28, 29] the specificities of spectra calibrations for one-detector spectrometer case with vanadium standard, the ratio between time of measurement with typical standard and sample, spectra correction with regard to detector gap time and opportunities of hydrogen detection in mixtures of water and heavy water are given.

Key feature of the upgrading was the usage of two-detector system that is the basic element of YuMO spectrometer at the moment [18, 23]. This system allows not only widen the dynamic q-range and lessen twice the time of experimental data collection, but also it allows conducting measurements for unique samples in wide q-range simultaneously. Consequently, the widened q-range implies the increase of accuracy of obtained results that for model object is usually the only way to recover the form-factor and space distribution unequivocally for investigated objects [30]. The increase of dynamic q-range is also important for short-living samples measurements or when investigating kinetics.

The other feature of YuMO spectrometer in new installation scheme is obtaining SANS curve in absolute scale even during the experiment [31]. This gives an opportunity of obtaining additional information about the structure of the object and its interaction for example with solvent.

It is worth mentioning about further perspectives of YuMO development. New unique position-sensitive detector was created, already passed successfully preliminary tests and was mounted in working position on the spectrometer. It has high spatial resolution and will make it possible to investigate structure and properties of anisotropic objects and provide unique resolution of small-angle spectrometer YuMO [32].

2.1. Upgraded spectrometer description

The scheme of upgraded spectrometer is shown on Figure 1. Behind the shelter after reactor zone with thermal (or cold) moderator there is chopper, partially decreasing background and satellite pulses of neutron power, which are formed by additional reflector.

The flux of thermal neutrons was formed by collimator system (4,6 Figure 1) so that neutrons coming onto the sample were forming a neutron beam with maximum cross-section 22 mm and intensity of $3 \times 10^7$ neutrons/sec [6, 21].

The first collimator (4) together with adjustable collimator (7) that have a retractable “nose” and two discs (5,8) with discrete set of cadmium rings that have inner holes with different sizes, constitute new system of forming a neutron beam. The system is automatically controlled with the help of computer.
Figure 1. The scheme of main nodes of upgraded small-angle spectrometer YuMO on IBR-2 reactor. 1 is two reflectors, 3 is chopper, 4 and 6 is collimation system, 9 is samples table, 10 is thermostat, 11 – holder for 25 samples, 13, 15 is OLD and New detector correspondently, 12, 14 is vanadium standards, 16 is changeable vacuum tube, 17 is direct beam detector.

Vanadium windows on the retractable part of adjustable collimator and on vacuum tube of detectors replaced with quartz glass “Heraeus”.

Sample table can move in horizontal and vertical directions. The samples are put in the box made of aluminum alloy inside of which there are channels for the thermal liquid. The box is connected with computer controlled thermostat (10). Working range of temperatures is −20°C до +180°C. The box windows are made of aluminum foil (0.1 mm). At the same time, 25 cuvettes “Helma” (24x47x7 mm) could be placed inside the box. The sample table is not under vacuum that makes easy to mount different devices on it, for example, light source, pressure chamber, thermostatic volume (temperature box), magnetic installation etc.

Neutron flux scattered on the sample comes into stage vacuum volume where ring neutron detectors (13, 15) and direct beam detector (17) are placed. Vanadium standards that are placed directly in front of each detector (12, 14) during the experiment are periodically put into and out of the beam that allows avoid unpleasant effects of long-periodical power oscillations and obtain SANS curve in absolute units of scattering cross-section. Besides, such a procedure allows data treatment simultaneously with the experiment conduction.
Vanadium standards are preliminary adjusted relative to the axis of spectrometer with the help of the laser. In front of the second (NEW) detector, the computer-controlled mask (15) is placed for checking the anisotropy of the scattering picture. The main electronic modules made in WINDOWS 10 standard. Both detectors can move with the help of computer controlled stepping motors inside of vacuum tube with length of approximately 10 meters. The software for controlling the installation is a system that controls temperature, motors and data collection [19]. During the experiment conduction, the system controls the main spectrometer parameters. Also there is an option of remote experiment control.

The monitor counter placed in front of the sample can be used for spectra normalization, checking the work of vanadium standards and at the same time tracking the neutron beam and its intensity.

Direct beam detector is used for normalization in case of samples that have strong scattering or strong adsorption when standard procedure works not accurately enough [29].

2.2. Two-detector system
YuMO spectrometer upgrading based on development, creation and usage of two-detector system [22, 23] based on ring scattering detectors [33]. In the vast majority of experiments using slow neutrons requires the widest possible q-range. The standard solution of the problem is detector movement along the base sample-detector and/or creation of a detector with a big surface. The disadvantage of detector movement is the increase of data collection time at least twice and losing the sample state in case it has irreversible phase transition of limited lifetime. Creation of big-surface detector is limited by technical reasons and big costs. Multi-detector system has no such disadvantages. Its working principle is the following. A part of neutrons scattered on the sample is registered by the nearest (first) to the sample detector and the other part (scattered under lower angles) comes through the central hole of the detector and is registered by the next (second) detector (Figure 1) [34]. Having come through the central hole of the second detector the part on neutrons is registered on the third detector etc. The overlapping of the spectra is provided by time-of-flight technique. At the time of this writing the spectrometer had two detectors for tracking scattered neutrons and one direct beam detector.

In case of two-detector system it is necessary to take into account additional scattering on vanadium standard of the first detector in active state (inside the neutron beam) that is registered on the second detector (for the first detector all the formula are as for one-detector installation) [34]:

\[
\frac{d\Sigma}{d\Omega} = \frac{I_s - I_{sv}}{I_{sv} - I_s} \frac{d_s \Omega_s T_s}{d_v \Omega_v T_v} \left(1 + \frac{d_v \Omega_v}{d_s \Omega_s} \right) \]

(1.1)

where \( I_s \) и \( I_{sv} \) – scattering intensity on the sample that is registered on the second detector and scattering intensity on the sample and vanadium standard simultaneously registered on the second detector; \( \Omega_s \) – the solid angle formed from the sample position to the second detector; \( \Omega_v \) – solid angle formed from vanadium standard position to the second detector; \( \Omega_{sv} \) – solid angle formed from the position of vanadium standard of the first detector to the second detector; \( \frac{d\Sigma}{d\Omega} \) – differential cross-section of vanadium standards; \( d_s \), \( d_v \), \( T_s \) and \( T_v \) – thicknesses and transmissions for vanadium standards of the first and the second detectors respectively. In the right part of (1.1) there are constants and measured values. Consequently, it is possible to save the intensity scale (cross-section scattering) in absolute units.
Formula mentioned above are used only in case of low-scattering samples (cross-section is lower than 10 cm$^{-1}$). For strongly scattering samples (cross-section is higher than 10 cm$^{-1}$) the expression $I_{\phi + v}^o - I_{\phi}^o$ from (1.1) becomes approximately zero and fluctuate significantly that implies fluctuation of the scattering intensity. In this case it is necessary to use another normalization and instead of expression $I_{\phi + v}^o - I_{\phi}^o$ use $I_{\phi + v}^o - I_{\phi + v}^o$, where $I_{\phi}^o$ and $I_{\phi + v}^o$ – scattering intensity on background sample (solvent of matrix) and background sample and vanadium standard registered on the second detector simultaneously. In this case formula is the following (for the first detector all the formula are as for one-detector installation):

$$
\frac{(d\Sigma/d\Omega)_v}{(d\Sigma/d\Omega)_v} = \frac{I_{\phi + v}^o}{I_{\phi}^o} \frac{d_{v}^o \Omega_{v}^o}{d_s \Omega_s^o T_s} \left(1 + \frac{d_{v}^o \Omega_{v}^o}{d_s \Omega_s^o T_s}\right)
$$

(1.2)

where $I_{\phi + v}^o$ – scattering intensity on the sample that is registered on the second detector and scattering intensity on the sample and vanadium standard simultaneously registered on the second detector; $\Omega_{v}^o$ – solid angle formed from the sample position to the second detector; $\Omega_{v}^o$ – solid angle formed from vanadium standard position to the second detector; $\Omega_{v}^o$ – solid angle formed from the position of vanadium standard of the first detector to the second detector; $(d\Sigma/d\Omega)_v$ – differential cross-section of vanadium standards; $d_s$ – sample thickness; $d_{v}^o$, $d_{v}^s$ and $T_{v}^o$, $T_{v}^s$ – thicknesses and transmissions for vanadium standards of the first and the second detectors respectively. In the right part of (1.2) there are constants and measured values. Consequently, it is possible to save the intensity scale (cross-section scattering) in absolute units.

Experiments in most neutron research centers are conducted with several positions of the detector and then all the data are merged and shown on one plot with preliminary normalization of each spectrum. Such approach requires rationale with taking into consideration boundary conditions and, for example, its application to solutions where the concentration of investigated particles is time-dependent or to solutions with low kinetics is problematic. Such problematic samples could be lipids or big organelles.

For time-of-flight spectrometers, q-range is defined by experimenter with regard to sample cross-section and ratio signal/background. Two-detector system allows solving this problem. When overlapping two spectra from different detectors q-range limitations become experimentally defined.

For ring detectors with central pinhole there is no necessity to make spectrum correction in the near zero angles as in case with beam-stop. These additional advantages of YuMO spectrometer allow controlling scattering curve boundary conditions.

2.3. Spectrometer resolution

It is worth to remind that small-angle spectrometers belong to the class of low-resolution installations. Nowadays, within the development of mathematical methods and for tasks with lattice parameter smaller than 50Å the spectrometer resolution often plays the key role. In terms of obtaining the best resolution, a multi-detector system has important advantages. Indeed, under the same spatial resolution the nearest position of the detector to the sample allows obtaining higher resolution. Consequently using of two detectors gives better resolution if compare them with using one (big) detector with the same q-range, but placed in the between.
Pulse neutron sources have significant advantage not only because the time resolution (and consequently neutron wavelength) is much better than on stationary reactors, but also they have a possibility of widening q-range. In our work [27] it is shown that using of dynamic window in smoothing procedure allows widen q-range in case of bad statistics. Widening of q-range implies increase in accuracy of obtained results that for model object is usually the only way to recover the form-factor and space distribution unequivocally for investigated objects.

Commonly used for calibration of X-ray spectrometers silver behenate can be used and for neutrons. However, nuclear contrast is not so high. We evaluated the possibility of using silver behenate not only for calibration purposes but also for resolution [35].

3. Physical parameters of spectrometer and investigation of supramolecular structures

The scheme of experiment mentioned above allows widening q-range of slow neutrons that are scattered on the sample and registered on detectors. Consequently, it widens the range of transferred momentum at least twice in comparison with moving detector on base sample-detector at the same time of experiment.

On the Figure 2 the comparison of two spectra (before and after upgrade) is shown. For some samples the minimum q-value is $4 \times 10^{-3} \text{Å}^{-1}$.

![Figure 2. SANS curves for single lipidic membranes measured in 1999 with one detector (left) and two-detector system in 2000 (right: red – NEW detector and blue – OLD detector). Measurements time was 20 min.](image)

Figure 3 demonstrates the possibilities of spectrometer for low-scattering sample with short time of measurement and the value of dynamic q-range that is eventually limited by the sample size. On the Figure 4 there is a curve where intensity of scattered neutrons has a five orders change. The ratio $q_{\text{min}}/q_{\text{max}}$ (dynamic q-range) in this case is more than 90.

In addition, the optimization of spectrometer parameters was made. The questions of data consistency in the area of overlapping experimental points from two detectors were investigated [22]. Devices allowing studies of the samples under pressure and with external magnetic field are created. Available
temperature range on the sample is widened and new device for experiments with high pressure opens new direction for investigation of condensed matter [36-38] on YuMO spectrometer.

![SANS curves for polyelectrolyte membranes Nafion 105 measured in 1999 with one detector (green circles) and two-detector system in 2000 (red squares).](image)

**Figure 3.** SANS curves for polyelectrolyte membranes Nafion 105 measured in 1999 with one detector (green circles) and two-detector system in 2000 (red squares).

During the experiment, the parameters and all spectrometer motors states are tracked and the protocol is logged. The results obtained have systematic errors excluded and there is always the possibility to watch the logs when incorrect work of the spectrometer takes place. Thereby the accuracy of obtained results increased significantly.

Qualitatively another level of experiment control is provided by the automated nodes of the spectrometer.

Finally, there are papers that present the results of the upgrade, which became possible because of the changes in spectrometer. The part of them is [30, 37-65]. For example, in [39, 40] the investigation of influence of substances interesting in terms of science and pharmacy on lipid membranes is described. In [41] and [42-46] – fractal arrangement in biological objects and in soils respectively. In all cases, it was the wide dynamic q-range that allowed obtaining the further mentioned results. Theoretical studies [66-70] show that for determined fractals investigation it is required not only wide dynamic q-range but also good resolution.

Authors of the work [41] studied intact chicken erythrocyte nuclei on several installations (Dubna, Juelich). It is shown that on the scale of 15 nm – 1.5 um the scattering curve could be interpreted in terms of mass fractal. Fractal dimension of the protein parts of cell nucleus is constant and approximately
equals 2.5. In comparison, the DNA arrangement is two-phase with the value of 2 for the scale less than 300 nm and tends to 3 on the larger scale.

![SANS curves for water solution of apoferritin measured in 2017 with two-detector system and smoothing procedure [25]. It is shown low level of background and good resolution.](image)

Figure 4. SANS curves for water solution of apoferritin measured in 2017 with two-detector system and smoothing procedure [25]. It is shown low level of background and good resolution.

Methodic value of this work is to check the coincidence of scattering curves obtained on upgraded spectrometer YuMO and installations KWS-2 and KWS-3 (Forschungzentrum, Juelich, Germany). Obtained lines in log-log scale coincide in area of overlapping ranges of transferred momentum.

Fractal arrangement of colloidal compartments of soil is considered in [42-46]. Scattering curves obtained on these samples have a dynamic q-range 90 (q-range is from 0.007 to 0.6Å⁻¹). More than 20 types of soils and horizons are considered. It is shown that fractal dimension depends on the type of soil and soil horizon, degree of water saturation and temperature. Conclusions about the fractal structure were made because of wide dynamic q-range.

It is worth to point out that future nanotechnology breakthrough will be probably connected with creating monodisperse objects. The question of small-angle scattering curves in case of monodisperse deterministic fractals is theoretically considered in [66-69, 71]

It is shown that at a given iteration m, both surface fractal models can be represented as a sum of mass fractals at iterations from zero to m. This confirms that in general, any surface fractal can be represented as a sum of mass fractals [70].

The work [47] is about studying the kinetics of bacteriorhodopsin crystallization in lipidic cubic phase. The beginning of crystallization processes when adding salt implies significant decrease of lattice
parameters. The symmetry type and the lattice parameters were determined and the kinetics of parameters change was studied. In terms of method a good resolution of spectrometer is demonstrated in that work. The time of one measurement for studying kinetics was 3 min.

The questions of phase decay in bismuth superconductor are considered in [48].

The object of studies in polyelectrolyte hydrogels was a phenomenon of charge-induced microphase separation [49, 50]. The aggregation number was determined. It was shown that the hydrophobic part decreases when the charge increases. When adding salt the microphase separation vanishes. The model was suggested for such behavior of polyelectrolyte hydrogels. In addition, the wide dynamic q-range is used in that work because there is a structural arrangement of polyelectrolyte hydrogels at different scales.

One of the direction in polymers physical chemistry is searching and creation of controlled gels. In the frameworks of this direction the work about studying of rigid polyelectrolyte rods in polyacrylamide gel was done [51]. It was shown that as inside gel as in water solution polyelectrolyte rods are self-organizing in cylindrical aggregates containing 8-9 single polymer chains. The chains are oriented along the axis of such aggregate. When the aggregates occurred, the gel becomes more homogenous at large scales. Conclusions are based on systematic rods studies immersed into gel or solvent with a screening of gel (solvent) percentage relatively to the rods and studying the influence of salt addition on structural arrangement of the system in general.

The effect of insertion of antibiotic AmB in membranes of egg phosphatylcholine in concentration range from 0.01 to 5 molar percent per a structure and dynamic properties of lipid bilayers were studied by three methods – SANS (YuMO spectrometer), X-ray diffraction (DRON-4) and infrared spectroscopy (FTIR) [52]. The results showed that antibiotic is predominantly placed near head groups of membrane under the concentration lower than 1 molar percent and under higher concentrations AmB inserts the hydrophobic membrane part.

Started at the beginning of 90-th works on magnetic liquids [12] where it was shown how to distinguish nuclear and magnetic counterparts in scattering intensity on the installation without magnetic field and polarized neutrons have found their continuation on upgraded YuMO spectrometer [53-55]. Authors [12] showed that by contrast variation method in experiment of small-angle scattering of non-polarized neutrons one can distinguish nuclear and magnetic counterparts. This, as it was mentioned, was absolutely new result at that time.

In review [55] the questions of relation between magnetic and nuclear counterparts and influence of the solvent type on the behavior of SANS curve are considered. The size of magnetic particle and surfactant external shell are given for several types of deuterated solutions and surfactants. In particular it is pointed out that in water solutions (that is important for practice) the particles with surfactant shell associate into clusters. The conclusion was based on the power law of small-angle curve for water solutions. In the work the comparative table for four SAS instruments is shown: YuMO, IBR-2, Dubna; Yellow Submirine, KFKI, Budapest, Hungary; SANS, PSI, Willigen, Switzerland; SANS-1, FRG-1, GKSS, Geschacht, Germany. Under approximately the same q-range spectrometer YuMO makes a single measurement when the other spectrometers require three-four independent measurements (and consequently three-four times more the measurement time). At the same time it is noted that all non-Russian spectrometers equipped with magnetic devices (filed from 1.2 to 2.5 T). The project of installation for creating magnetic field (up to 2.6 T) was successfully implemented in Dubna on small-angle YuMO spectrometer and the first test experiments were already done.

In structural investigation of biological objects it is about to jump from single protein, membranes and even complexes investigation to investigation of these objects as a part of the whole organelles. In works [56-58] made on upgraded YuMO spectrometer the structures of membranes of intact mitochondria were
investigated. Small-angle experiments showed that when the system of volume regulation is switched on the ultrastructural rearrangements in mitochondria from liver and heart of mouse take place. Peaks positions showed that if liver mitochondria are put under hypotonic conditions the cristae rearrange from disordered package to ordered two-membrane package with distance between membranes centers 190 Å. Under the same conditions in heart mitochondria the cristae rearrange from lamellar package with distance 220 Å to presumably hexagonal with lattice parameter 250 Å.

The spectrum of the beam on YuMO spectrometer includes also fast neutrons and gamma quants [6]. For decreasing the destructive radiation of the object the time of measurement should be decreased. This was achieved in the frameworks of two-detector system of tracking scattered neutrons. After the measurements a respiratory control of the mitochondria was carried out, which showed their resistance to radiation within 0.76 Sv for the gamma-ray flux and 49.9 Sv for fast and thermal neutrons. Thus, the possibility of using the method of small-angle neutron scattering to study the structure of functioning mitochondria was demonstrated.

Development and installation on the spectrometer the device with high hydrostatic pressure on the sample allowed investigating the phase transitions in micellar solutions of tetradecyl trimethyl ammonium bromide / heavy water (TTAB / D2O) in wide temperature range, pressure and surfactant concentration [34,35]. The kinetics of the phase transition micelles – solid phase was studied. The phase diagram of solvents was calculated and the temperature, pressure and surfactant concentration dependency of the transition velocity was shown. Two-phase coexistence of micelles and precipitates (solid phase) under certain conditions on concentration, pressure, and temperature was observed.

Recently the unique PVT installation was created. With its help, one can simultaneously conduct neutron structural investigations and measure the volume change[72]. Experiments made on upgraded reactor IBR-2M on the PVT installation for lipid membrane mixture for the first time showed that in the mixture of DPPC/POPC in excess of water the simultaneous jump of isothermal compressibility and minimum in the derivative of the periodicity [73]. Dynamic q-range is extremely important when studying lipid membranes. Indeed, in case of wide dynamic q-range it becomes possible to track the changes of periodicity of lipid membranes and the thickness of lipid bilayer at the same time. Taking into account the kinetics of solvent this consequence allows avoiding systematical errors [72].

In the series of works [30, 31, 59-61], aimed at investigation of the structure of polyallylcarbosilane dendrimers, the specificities of interactions such nanodisperse objects with solvent and their arrangement inside the solution, some quantitative parameters characteristics were obtained, such as sizes of several generations and average scattering density. Appliance of modern data treatment methods and the usage of good resolution of upgraded spectrometer YuMO allowed characterizing the form of dendrimers of several generations for 3 and 4-functional core. The fact of solvent penetrates inside dendrimer allows expecting the possibility of practical application of these new synthetic big molecules. The wide dynamic q-range (from 0.007 to 0.4Å⁻¹) and absolute intensity scale allowed solving the structure of dendrimers and understanding their interaction with solvent. Using modern mathematical data analysis algorithms to treat SANS data the spatial distribution of scattering density of investigated type of dendrimers was obtained [30]. SANS contrast variation showed that dendrimers molecules in solvents do not contain closed inner cavities where the solvent cannot penetrate into. Knowing the SANS intensity in absolute scale the partial dendrimer volume in solvent was obtained and the volume ratio (30-40%) of opened inner cavities inside effective dendrimer volume where the solvent can penetrate into was calculated. The anisometry of dendrimer macromolecules form that was inconsistent with AFM data [62] found the confirmation in results obtained by molecular dynamics [63].

Investigation of structure and solubilized behavior of three-layer nanoparticles obtained by using radiation polymerization from methyl methacrylate was done by two methods – NMR and SANS. SANS
measurements were done on two installations – YuMO, IBR-2, Dubna and Yellow Submirine, KFKI, Budapest, Hungary. The absorption kinetics and two low molecular weight compounds (benzene and chloroform) equilibration were studied. SANS curves obtained on YuMO were in full consistency with the data obtained in neutron center in Budapest.

Lipid membranes is an adequate object for SANS structural studies. Using this method it is feasible to obtain as periodicity as thicknesses of single vesicles. Q-range plays a key role here because only in case it is widely enough these parameters could be obtained simultaneously. With the help of YuMO spectrometer the influence of DMSO \((\text{CH}_3)_2\text{SO}\) on the phospholipid (DMPC) membrane structure was investigated in wide DMSO concentration range \(0.0 \leq \chi_{\text{DMSO}} \leq 1.0\) in excess of solvent at \(T=12.5^\circ\text{C}\) and \(T=55^\circ\text{C}\) [64]. The dependency of periodicity \(d\) of multi lamellar vesicles and thickness of single vesicles \(d_b\) in gel and liquid-crystalline phases versus \(X_{\text{DMSO}}\) in excess of solvent DMSO/water was determined. The complementary use of the values \(d\) and \(d_b\) made it possible to determine the intermembrane distance \(d_s\). It is shown that the intermembrane distance decreases significantly with increasing DMSO concentration, and at a concentration of \(X_{\text{DMSO}}=0.4\), the membranes in the neighborhood are practically in a steric contact with each other, which results in the fusion of single membranes into multilayer lamellar structures. The use of deuterated DMSO and the contrast variation method on water allowed determining for the first time the number of DMSO molecules strongly bounded to the membrane. The number is 6.9, and the total volume of such molecules is 820 Å³, which is comparable with the volume of the polar head of the lipid molecule itself and explains the previously observed phase transition of the lipid membrane to the phase with mutual penetration of chains.

The investigation of influence \(\text{Ca}^{2+}\) on structure DMPC multilamellar vesicles in [74] was done.

The study of binary mixtures of branched polyethyleneimine and cationic surfactants aggregation in aqueous solutions was also carried out by several methods [65]. In that work, the dependency of shape and size of polymer-colloid aggregates, as well as their catalytic activity on the polymer concentration, the structure of surfactants and substrates was obtained. It has been found out that in some aqueous compositions it is possible to achieve significant acceleration (up to \(10^4\) times) of decomposition of esters of tetrakodonated phosphorus acids in comparison with their hydrolysis reaction in water at the same pH in both cases. The methodological feature of this experiment is a combination of conductometry, dynamic light scattering and SANS methods, which in this case are complementary and give self-consistent data about the sample structure.

During the SANS experiment the sample is placed in the air gap between the neutron guide and the detector tube. Thus, it is possible to illuminate the sample with a laser beam or a specific beam of light. In a sample environment system, a Schott KL 1500 electronic (SCHOTT Glas) source of visible light with a high and constant light intensity was used on the YuMO spectrometer. Light is directed to the sample with a flexible fiber. During the experiment, the sample is not exposed to excessive stress heat. With the help of this light source, the influence of light on the structure of bacteriorhodopsin (bR) protein on the YuMO spectrometer was studied.

Mentioned above several examples of investigations from material science to biology show new qualitative state of spectrometer after the upgrade.

Further instrument development is connected first of all with the method of thermal neutrons detection. Since March 2002 the project connected with creating the contemporary 2D position-sensitive detector (PSD) was launched. The detector is created, preliminary tests were done in LLB (France) and the detector was tested on YuMO[32]. PSD will make it possible to start working at usage of important advantage of pulsed reactor in comparison with the stationary one – good resolution on the momentum transfer. When PSD start working it will become possible to obtain additional information about the
structure and consequently about the properties and processes occurring in samples under the action of temperature, pressure, light illumination and humidity.

4. Conclusion
Let us sum up the main advantages of upgraded YuMO spectrometer in comparison with the old instrument and the advantages of new nodes and devices of the spectrometer. Firstly, it is a high rate of experimental data collection and a wide dynamic q-range because of a two-detector system, secondly, it is a high level of service and remote control; thirdly, there is the possibility of quick commissioning of practically any new device on the spectrometer and integrating them into the existing control program.

The scheme of the experiment makes it possible to obtain the intensity curve in absolute units immediately after the experiment is completed and during the experiment. This gives an undeniable advantage and additional information when studying the molecular and supramolecular structures before the commonly used scheme (after the experiment additional measurements of water, carbon or other normalizers are made). New programs for the initial processing of experimental data and the fitting of obtained curves were updated, revised, and created.

The expanded opportunities led to the increase in the number and quality of experiments, and, correspondingly, to the increase of scientific publications.

Thus, the main result of the upgrade was the appearance of a small-angle world-class neutron spectrometer.

We dedicate this work to the memory of Yu.M. Ostanevich, our colleague and creator of the unique small-angle neutron spectrometer at the IBR-2 rector, the 80th anniversary of his birth was recently celebrated[75].

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