High-throughput SANS experiment on two-detector system of YuMO spectrometer

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Abstract. Using a multidetector system on the YuMO spectrometer allows shortening the time of measurements. The quantitative comparison of the measurement time using one and two-detector mode is done. The time range for experiments was from several minutes up to 12 hours. It was shown that two-detector system shortens more than twice the time of the measurement. While making a structural investigation using advanced software the two-detector system allows to treat the data at a qualitatively new level. An example illustrating the features of the channels choice and measurement time on the spectrometer was shown. The results of this paper could be used when planning the experiments on the YuMO spectrometer, for modernization of the installation and for equipment using time-of-flight method.

1. Introduction
For the most commonly used scientific instruments the time of measurements is defined by experimenter. Occurrence of the spectrometers on reactors has changed this paradigm. The operation time of the spectrometer became strongly dependent on the reactor operation time. The experiment went into industrial scale. The increasing interest in nanotechnologies and consistent structural problems [1, 2] has raised the busyness of reactor instruments. The difficulty of shortening the time of measurements without the loss of data quality has occurred especially for time-of-flight facility. The problem could be solved via full automatization of the spectrometer [3, 4] and eliminating the experimenter-made errors.

The system that uses two detectors simultaneously [5-7] was designed and mounted on the small angle spectrometer YuMO on the reactor IBR-2. It allowed significantly shortening the time of experiment. The system was also extremely important especially because modern data treatment software requires wide Q-range [8-12] in contrary to the earlier periods of small angle neutron scattering.
development [13]. The problem of experimenter at the moment is to obtain data in wide Q-range with satisfying statistical error, which is determined by the time of measurement. The measurement time depends on the scattering cross-section at the other similar conditions, first of all, the Q-range.

In the work [6] the scheme of the experiment, calibration methods for samples that have low and high scattering, optimization of sizes and distances for vanadium standards are described. The idea of shortening the time of experiment could arise after two decades of two-detector system exploitation and several times increase of scientific publications made with the help of the instrument [7].

A new word in modernization of the YuMO spectrometer is a design of the second unique position sensitive detector (PSD) in the framework of two-detector project. As soon as the question about duration of the exposure on the small angle neutron spectrometer on the reactor IBR-2 was still unclear, it will be described in details in this work.

2. Time of measurement factors
The time of measurement significantly depends on the cross-section scattering of the sample. The available intensity range is determined by the statistical error that directly depends on the measurement time. Another factor is the wideness of the Q-range that is necessary for experiment. If the value is bigger than the dynamic range of the spectrometer \( \frac{q_{\text{max}}}{q_{\text{min}}} \), the experimenter should make additional measurements. For stationary reactors producing monochromatic irradiation the dynamic range is determined by the size of the detector. For pulsed neutron sources (accelerator, IBR-2) the terms such as Q-range and dynamic Q-range are quite relative. The limitation of the YuMO spectrometer for samples that have a low scattering is nearly 0.05 - 0.1 cm\(^{-1}\). In this case the Q-range in Guinier approximation area is about 0.02 A\(^{-1}\) and in the area of Porod approximation is up to 0.2 A\(^{-1}\). The dynamic range in this case is only 10. For the samples that have high scattering the Q-range is 0.006 – 0.6 A\(^{-1}\), and the dynamic range is more than 100. Consequently, the Q-range, dynamic Q-range and time of measurement significantly depend on the sample. It is worth to highlight that the samples with low scattering when it seems that using one detector satisfies the dynamic Q-range, the time of measurements could also be shortened twice if using the second detector in optimal position. This happens due to the using of the time-of-flight method and the possibility of moving the detectors so that the Q-range will be approximately similar for both detectors. In addition, experimenter often makes test measurements with samples that could have specific behavior in different points of Q-range. Such experiments commonly last several minutes and experimenter does not move the detector. In case of one-detector system the effect could be lost. Consequently the using of two-detector system the data quality is much higher.

The smoothing technique [14] for small angle data could also shorten the time of measurement except "tails" effect. This could be achieved only by using of two-detector system and advanced data treatment methods.

3. The comparison of the time of measurements: one detector at two positions vs two detectors simultaneously
The question about the comparison of the time of measurements (one detector at two positions and two detectors simultaneously) could be reduced to the question of the time of measurements in the nearest position (big values of Q) and the further position (small values of Q) at the optimal collimation for each position in case of one detector. These times of measurements are almost similar. So the low scattering cross-section at big values of Q (the nearest position) and the consequent necessity of long exposure is compensated the small number of neutrons and consequently almost the same exposure time at low values of Q (the further position). It is worth to point out that the measurement at nearest position (for shortening the time of the experiment) implies information loss for monodisperse samples (dendrimers, proteins, tracking membranes) and samples that have a structural factor (lipid membranes, polymers, etc.). Two-detector system has no such disadvantage. The need of high resolution at the nearest position for the wide spectrum of samples (alconates [15], proteins [16-19], dendrimers [20-25], nanotubes [26], lipids [27-29]) leads to conducting the experiments at the nearest position of the well-collimated
detector. Consequently the time of measurements range on the spectrometer YuMO is from 3 min [30, 31] up to 12h [32, 33].

On the spectrometer YuMO there is a specific procedure of normalization [34, 35]. For two-detector system this procedure has some corrections [6]. The same reasoning is true for the time of measurements with and without vanadium standards [36]. There is also the ratio

\[ \frac{t_{sv}}{t_s} = \left( \frac{I_s}{I_{sv}} \right)^{1/2} \]  

(1)

where \( t_{sv} \) is the time of measurement of the sample simultaneously with vanadium standard into the beam, \( t_s \) is the time of the measurement of the sample, \( I_s \) – scattering intensity of the sample, \( I_{sv} \) – scattering intensity of the sample and vanadium standard in the beam simultaneously. The ratio could be defined by the minimum scattering of the vanadium standard. It is important that the longer the measurement of the background the wider the dynamic range.

Here is the qualitative evaluation, because the quantitative calculation requires exact values of Q-range and depends on the sample. The efficiency of detector grows versus wavelength and the transmission decreases almost at the same order, so these coefficients cancel each other. The ratio of fluxes for different wavelengths [37] is nearly \( 10^4 \) and the ratio of differential cross-sections is some units of \( 10^3 \) and the ratio of solid angles is approximately 1/6. So, taking in account the square root from the formula (1) the times of measurements is the same order values. This is only a qualitative evaluation. For every sample the ratio of the times of measurements should be calculated individually. In practice the experimenter has an adequate statistical error taking into account the real operation time and consequently correcting big or low values of Q so that the times of measurements at small angle and wide angle area of Q are equal in the case of two-detector system.

The SAS package [35, 38-39] is used to process the spectra measured on YuMO spectrometer. And with modernizing the spectrometer SAS has been developing in parallel. It has preserved the fundamentals of TOF data treatment, but its architecture was refactored significantly. In general, processing with SAS includes these main stages:

- normalization of the time spectra over the scattering from the vanadium standard;
- conversion time spectra into the space of neutron momentum transfer.

First stage is performed independently for each detector ring. And on the second one the resulting spectra are averaged over the set of rings.

Modern SAS does not operate with "OLD" and "NEW" detectors separately, as before, when spectra for them have to be sewed. Instead, it operates with the whole set of rings for both detectors and produces the final spectrum in a natural way [35]. Moreover, this approach improves the statistics for the middle region of the averaged spectra. This region corresponds to the boundary between "OLD" and "NEW" detectors, and when averaging them separately boundary effects made it difficult to sew the spectra.

In the Fig. 1 there are SANS curves for carbon powders smoothed (for clarity) as described in [14]. Guinier approximation area of the curve has Q-shift due to decreasing of time channels range for obtaining the scattering picture at «true» intensity scale. Otherwise the scattering intensity will be in arbitrary units so the slopes of the scattering curves (Fig. 1) and the discrepant parts of the curves in the Guinier approximation region are not represent the true scattering picture. The same is also true for the right border. However, there the effect is not so prominent because of the small scattering cross-section.

The fact that SANS intensities for 60, 30 (overlap fully visually) and 10 min of measurements are almost similar means that for samples with high scattering the time of measurement 10 min is enough. However, there are some discrepancies near the right border.
Figure 1. Powder carbon SANS curves for different measurement time. For some curves the selection of TOF channels was managed. From left curves to right: time of measurement 60 min (55-450 time-of-flight channels), 30 min (55-450 time-of-flight channels), 10 min (55-450 time-of-flight channels), 5 min (65-350 time-of-flight channels), 2.5 min (75-300 time-of-flight channels), 70 second (85-220 time-of-flight channels), 30 second (95-200 time-of-flight channels). First two curves (60 min and 30 min) overlap fully visually.

The optimal Q-range has especially to be taken into account when measuring samples with «fractal» properties. The fractal structures with determined dimension have a linear behavior of scattering curve in log-log scale [40-45]. Boundary conditions determine the size-range where the fractal structure could be observed. That is why the optimal Q-range is crucial in this case.
Figure 2. Apoferritin solution with 7 mg/ml concentration in capillary (2 mm light path) SANS curves for different measurement times: 80 min (red curve), 60 min (blue curve), 40 min (yellow curve) and 20 min (black curve). All curves are smoothed for obvious vision by procedure, described in [14].

In Fig.2 shown example of curves demonstrates good possibility of measurement in capillary 2 mm light path.

4. Additional time loss during experiments with one-detector system

Another difference between one- and two-detector system modes of data collection on the spectrometer YuMO is the following.

For example if there is an experiment on the spectrometer YuMO that includes measurements of 25 samples with 4 different temperatures each (in 30 degrees centigrade range) using one-detector system in two different positions or two-detector system. Additional time loss connected with detector moving 10 meters is about 0.25h. Nearly the same time loss is for changing the samples. To change the temperature – minimum 30 min and for its stabilization – 1.5h. The resulting time loss is nearly 3h. If the time of measurement is 20 min for each sample (high scattering samples) then there is 65% of time loss. If there are low scattering samples (the time of measurement is 60 min for each) the loss is about 20%. If the time of measurement of buffer simultaneously with temperature changing is taken into account (despite it is not always correct) the ratio of the time loss will be lower, but nevertheless significant part of the operation time.

Besides, the nearest to the sample detector decreases the background for the second detector, that increases the signal/noise ratio and results in shortening the time of measurement. The quantitative evaluation of this effect is not simple enough and is out of the scope of this paper.

Concerning kinetic experiments and unique samples with short lifetime, clustering effects (especially biological or colloids) the only way of obtaining structural information in wide dynamic range is using
two-detector system. This is also works for investigating samples at temperature and/or pressure changes and phase transitions.

5. Conclusion
The dynamic range and Q-range are significantly dependent on sample. Consequently, as the time of measurement for most of the samples in near position is practically the same as in far position, the whole time of measurement shortens twice. While measuring samples with low scattering intensity and consequently narrow Q-range the second detector could twice the statistical data collection that is equal to shortening twice the time of measurement if optimal positions for the both detectors have been setup.

When using only one detector there is an additional loss of the time of measurement (from 20 to 60%) connected with detector movement, sample and temperature changing. It means that the time shortening for two-detector system in comparison with one-detector system is even more than twice for most of the experiments. Expected ratio of the times of measurement for other small angle spectrometers is nearly the same if the sizes of detectors are equal.

The results of the paper are proved by almost two decade experience of two-detector system exploitation on the YuMO spectrometer.

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