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Wavelength calibration in conventional SANS setup with a mechanical velocity selector

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Abstract. In the conventional setup of a small-angle neutron scattering instrument a nearly monochromatic neutron beam is formed by means of a mechanical velocity selector. We studied the influence of the wavelength distribution in the incident beam on the wavelength of the neutrons reaching the sample. The assumption that the wavelength spectrum behind the selector equals to the product of the incident spectrum of the thermalized neutrons and the transmission window of the selector predicts satisfactorily the wavelength measured at sample position. An approximate relation is given between the effective wavelength and the selector rotation speed. The described method of wavelength calibration can improve the precision of size determination in SANS experiment.

Introduction

In the conventional setup of a small-angle neutron scattering instrument the neutron beam is monochromatized by means of a mechanical velocity selector. The main factors that determine the wavelength of the neutrons reaching the sample are the wavelength distribution in the incident beam, the transmission characteristics of the selector and those of the mirror-coated neutron guides. The knowledge of the right wavelength is essential for obtaining size parameters with precision as high as one percent. A possible source of error in wavelength calibration can be the assumption that the wavelength is inversely proportional to the selector rotation speed.

In the present work, we derived an approximate analytical formula that gives the wavelength of the neutrons reaching the sample in function of the selector speed, and compared it with the experimentally determined effective wavelength. The latter was measured using a silver behenate powder sample.

The neutron spectrum transmitted by the selector

The transmission function of a velocity selector is determined by its geometrical parameters, it has usually a triangular shape, and the position of its center, $\lambda^{selector}$, is related to the speed of rotation:

$$\lambda^{selector} = C / \omega , \qquad (1)$$

where C is a constant, and ω is the selector rotation speed. Due to its simplicity, equation (1) is used widely for the calculation of the neutron wavelength from the selector rotation speed. The constant C is determined by the geometry of the selector and the tilt angle between the selector axis and the neutron beam; it can be determined experimentally by measuring the wavelength at certain selector speed, or calculated from the specifications of the selector.
In a real experiment the neutrons that reach the sample have some unknown wavelength distribution, which can often be well approximated by a Gaussian distribution, centered at λ^{eff} . The effective wavelength however does not necessarily coincide with $\lambda^{selector}$. For the SANS instrument at NIST, the observed difference between the calculated wavelength and the measured one was attributed to the slope of the incident spectrum [1]. Calibrating the selector with wavelength resolution 10% FWHM at ILL, a relation $\lambda^{eff} = A + B / \omega$ has been obtained [2]. The small offset parameter A led to a deviation from equation (1) less than 1% over the investigated λ-range. Authors of a recent paper [3] noted that the non-flat incident spectrum deforms the transmitted spectrum, and therefore the actual wavelength should be measured rather than calculated.

It is well-known that the wavelength distribution of the neutrons in the beam behind the selector can be written as the product of the selector transmission function and the wavelength distribution in the incident beam [4, 5]. In λ-space, the distribution $\Phi^{transmitted}(\lambda, \omega)$ of the neutrons transmitted by the selector is:

$$\Phi^{transmitted}(\lambda, \omega) = \phi_{selector}(\lambda, \omega)\ \Phi^{incident}(\lambda) \quad , \qquad (2)$$

where $\phi_{selector}(\lambda, \omega)$ is the selector transmission function at speed ω (ω is omitted in the further formulas) , and $\Phi^{incident}(\lambda)$ is the neutron wavelength distribution in the incident beam. For simplicity, we do not consider the angular distribution of neutrons in the different parts of the beam.
The scattered neutrons are registered by detectors with wavelength-dependent efficiency. For a given detector, an 'effective' neutron spectrum can be introduced: $\Phi^{incident\_effective}(\lambda) = \Phi^{incident}(\lambda) \cdot \eta(\lambda)$, where $\eta(\lambda)$ is the detector efficiency. All measurements described below were performed with the same detector, so for simplicity we keep the notation $\Phi^{incident}(\lambda)$ for the efficiency-weighted neutron spectrum.

If the incident spectrum is not flat (for example Maxwell-like distribution), then, according to equation (2), the peak of the wavelength distribution $\Phi^{transmitted}(\lambda)$ does not coincide with the position of the center of the selector transmission function $\phi_{selector}(\lambda)$, and so the effective wavelength of neutrons reaching the sample is shifted with respect to $\lambda^{selector}$.
The wavelength shift can be calculated in linear approximation, using Gaussian selector transmission function $\phi_{selector}(\lambda) = \exp\left(-(\lambda-\lambda^{selector})^2/2\sigma^2\right)$ and approximating the shape of the incident spectrum within the selector transmission window by a constant slope:

$$\Phi_{lin}^{incident}(\lambda) = m \lambda + b , \tag{3}$$

where m is the slope of the spectrum: $m = d\Phi^{incident} / d\lambda\big|_{\lambda^{selector}}$. From equations (2) and (3) the shift in the position of the peak of $\Phi^{transmitted}(\lambda)$ can be calculated:

$$\lambda^{eff} - \lambda^{selector} = D\left(\sqrt{1+\left(\frac{\sigma}{D}\right)^2} - 1\right), \tag{4}$$

where $D = \Phi^{incident}(\lambda^{selector}) / 2m$. Here $\lambda^{selector}$ is the nominal wavelength given by equation (1), in which the constant *C* must be determined at the position of the maximum of the spectrum. The such calculated effective wavelength λ^{eff} corresponds to the position of the maximum of $\Phi^{transmitted}(\lambda)$; this is the quantity useful in the analysis of the experimental scattering curves, if the non-symmetric shape of $\Phi^{transmitted}(\lambda)$ is not taken into account.

Experimental

The effective wavelength at different selector speeds was determined using a silver behenate powder sample. Silver behenate gives a strong reflection at $q = 0.1076$ Å^{-1} [6], which can be conveniently measured by a position sensitive detector within a wide range of wavelengths and sample-detector distances, making it suitable for wavelength and resolution calibrations of SANS instruments [6-8].
The experiments have been performed on the SANS instrument operating at a cold neutron beamline at the Budapest Research Reactor [9], equipped with a multidisk type velocity selector [10, 11]. The rotation speed can be varied between 700 and 7000 rot/min, transmitting neutrons with wavelengths between 3.8 and 38 Å at zero tilt angle between the selector axis and the direction of the neutron beam. The tilt angle can be adjusted within ±4°. In the present experiment the angle was set at -3.6°, in order to provide a large wavelength spread of 0.45 relative FWHM. The selector speed was varied from 1750 to 5500 rot/min, cor-

responding to a nominal wavelength window from 2.2 to 7.0 Å. The scattered neutrons were registered by a 64 × 64 cm BF_3-filled position sensitive detector with 1 cm pixel size.
The radii of the Debye-Scherrer rings from the silver behenate powder were obtained by fitting the two-dimensional scattering patterns by a corresponding two-dimensional function, in which the radial cross-section of the peak had a Gaussian form. The effective wavelength λ^{eff} was calculated at each rotation speed from the position of the first Bragg peak.
The form of the intensity spectrum was determined by measuring the scattering from a 1 mm thick light water sample at the same rotation speeds; the count rate on the detector is shown in figure 1. The change of the apparent forward scattering of water with the wavelength has been taken into account by applying the Jacrot factor [12]. No corrections were made for the wavelength dependence of the detector efficiency. For the elastically scattered neutrons it is taken into account by using the notion of the efficiency-weighted neutron spectrum. However, the energy gain of a certain part neutrons scattered by water could not be taken into account in this way. Use of another scatterer, or the attenuated beam could be more advantageous for the measurement of the beam intensity.

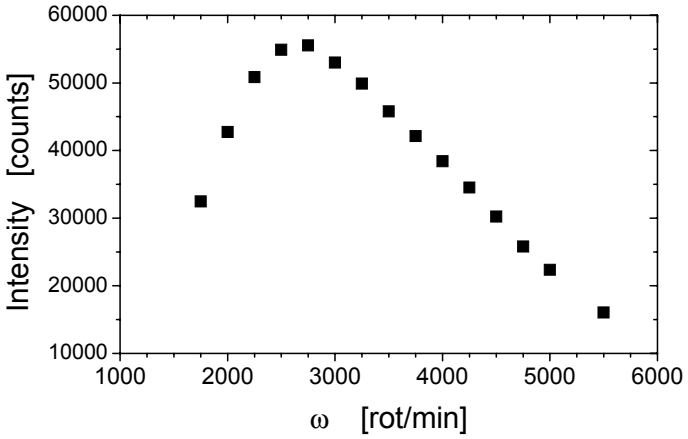

Figure 1. The scattering intensity from a water sample at different selector speeds.

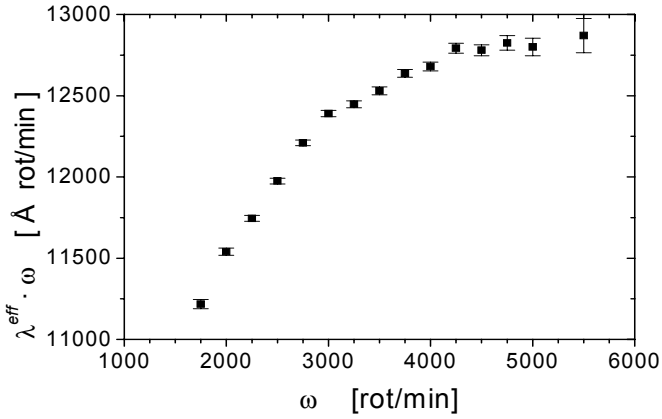

Figure 2. Product of the measured wavelength and the selector rotation speed.

Results and discussion

In figure 2 the product of the measured effective wavelength λ^{eff} and the rotation speed is shown in function of the selector rotation speed. The deviation of $\lambda^{eff} \cdot \omega$ from a constant value indicates that the measured wavelength is not inversely proportional to the selector speed. Comparing this curve with the form of the spectrum measured using a water sample (figure 1), the correlation between the spectrum and the wavelength shift is well seen. The relative wavelength shift, $(\lambda^{eff} - \lambda^{selector}) / \lambda^{selector}$, can be read out from figure 2. In the present experiment, with a broad selector transmission window, it reached values up to 10%. Such uncertainty in the wavelength is much larger than the desired precision of the size determination in SANS experiments.

In figure 3 the experimental wavelength shift is compared to the shift calculated using equation (4). In spite of the various approximations used, there is a rather good agreement between the measured and the calculated wavelength shift. The difference between the two curves is larger at small wavelengths; this can probably be attributed to the higher curvature of the spectrum in this region.

The wavelength resolution in this experiment was rather moderate. A narrower wavelength spread improves the approximation used in equation (3), as the linearity of the shape of the incident spectrum is better within a narrow transmission window. Instead of using the unknown $\Phi^{incident}(\lambda)$, we used the transmitted spectrum smeared by the selector. With a narrow wavelength spread, the difference between $\Phi^{incident}(\lambda)$ and the intensity distribution measured behind the selector would be smaller. Therefore, the removing of the selector

for the determination of the incident spectrum can be avoided, and measurements in the time of flight mode are not necessary.
The described procedure can serve as a convenient and efficient method of wavelength calibration, and can improve the accuracy in SANS experiments.

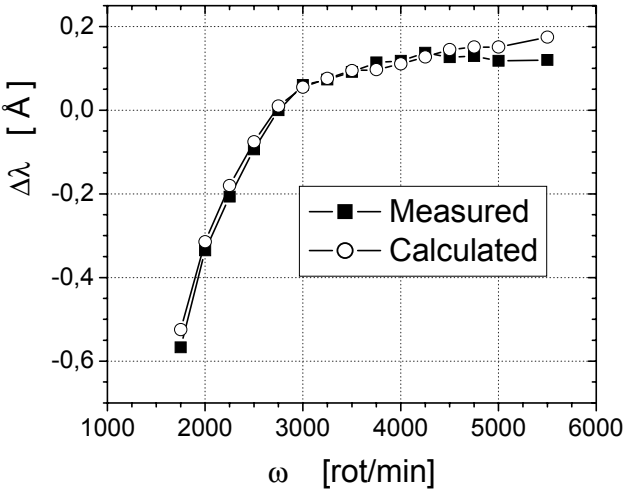

Figure 3. The measured wavelength shift $\Delta\lambda = \lambda^{eff} - \lambda^{selector}$ *and the shift calculated using equation (4).*

References

1. Glinka, C.J., Rowe, J.M., LaRock, J.G., 1986, *J. Appl. Cryst.*, **19**, 427.
2. Lindner, P., 1998, ILL Technical Report LI 11 T.
3. Strunz, P., Mortensen, K., Janssen, S., 2004, *Physica B,* **350**, e783.
4. Von Höhne, P., 1961, *Annalen der Phys.,* **7**, 50.
5. Friedrich, H., Wagner, V. & Wille, P., 1989, *Physica B*, **156-157**, 547.
6. Gilles, R., Keiderling, U. & Wiedenmann, A., 1998, *J. Appl. Cryst.*, **31**, 957.
7. Keiderling, U., Gilles, R. & Wiedenmann, A., 1999, *J. Appl. Cryst.*, **32**, 456.
8. Gilles, R., Keiderling, U., Strunz, P., Wiedenmann, A., Fuess, H., 2000, *Materials Science Forum*, **321-324,** 264.
9. Rosta, L., 2002, *Appl. Phys. A*, **74**[Suppl.], S52.
10. Rosta, L., 1989, *Physica B,* **156-157**, 615.
11. Rosta, L., 1991, *Physica B,* **171**, 562.
12. Jacrot, B., 1976, *Rep. Progr. Phys.*, **39**, 911.