National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3600

Absolute Intensity Calibration Standard for Small-Angle X-ray Scattering

This Standard Reference Material (SRM) is intended for use in the scattering intensity calibration of small-angle X-ray scattering (SAXS) instruments. SAXS measurements characterize the microstructure and nanostructure of heterogeneous material systems: specifically, the size distributions of microscale and nanoscale features and, with scattering intensity calibration, their volume fractions or number concentrations, and their surface areas [1]. A unit of SRM 3600 consists of a single glassy carbon coupon specimen of approximate dimensions 10 mm x 10 mm x 1 mm.

Certified Values: The certified values are for absolute X-ray scattering intensity, given in terms of the X-ray differential scattering cross-section, $d\Sigma/d\Omega$, per unit sample volume of glassy carbon, *versus* the magnitude, Q, of the scattering vector. A schematic of the ultra-small-angle X-ray scattering (USAXS) instrument, the primary instrument used to measure the certified X-ray scattering intensity values, is shown in Figure 1. Certified $d\Sigma/d\Omega$ values are tabulated in Table 1 for the certified Q range of 0.008 $Å^{-1}$ to 0.25 $Å^{-1}$, and are plotted in Figure 2. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account [2]. The certified values are based on measurements performed under ambient conditions.

Reference Values: Small-angle neutron scattering (SANS)-based values for $d\Sigma/d\Omega$ *versus* Q, rescaled for SAXS, are provided in Table 2 and plotted in Figure 3. The measured SANS scattering intensities have been rescaled for SAXS using the ratio of X-ray and neutron scattering contrast factors, based on traceable tables for the X-ray and neutron scattering properties of the carbon atom [3,4]. Reference values are noncertified values that represent the best estimate of the true values based on available data; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2].

Information Values: The mean thickness of the SRM 3600 coupons, based on two separate micrometer thickness measurements on each of 56 coupons sampled is 1.055 mm ± 0.025 mm. The uncertainty listed with the value is the expanded uncertainty with coverage factor $k = 2$, corresponding to a 95 % confidence interval, expected for a single coupon, and is derived from the standard deviation of replicate measurements. An information value is considered to be a value that may be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 3600** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate this notification.

Overall direction and coordination of the technical measurements leading to the certification of SRM 3600 was under the leadership of A.J. Allen of the NIST Materials Measurement Science Division.

Development of SRM 3600 utilized the Advanced Photon Source (APS) at the U.S. Department of Energy (DOE), Office of Science, Argonne National Laboratory (ANL), Argonne, IL and the SANS instrument at the NIST Center for Neutron Research (NCNR) funded by the National Science Foundation (NSF).

John A. Small, Chief
Materials Measurement Science Division

Gaithersburg, MD 20899
Certificate Issue Date: 17 June 2016

Steven J. Choquette, Acting Director
Office of Reference Materials

Design and USAXS measurements at the APS were performed by A.J. Allen and F. Zhang of the NIST Materials Measurement Science Division and J. Ilavsky of the ANL X-ray Science Division.

Supplemental SAXS measurements at NIST were performed by A.J. Allen, F. Zhang; and R.J. Kline of NIST Materials Science and Engineering Division. SANS validation measurements at NCNR were performed by A.J. Allen.

Data analysis, based on the USAXS/SAXS instrument software developed by J. Ilavsky, was performed by A.J. Allen and F. Zhang.

Statistical consultation was provided by W.F. Guthrie of NIST Statistical Engineering Division.

Sample cutting and initial packaging for this SRM were performed by J.N. Brandenburg, M. McGlauflin and J. Fuller of NIST Fabrication Technology Office.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

NOTICE TO USERS

Values provided in Tables 1 and 2 are available in a digital format at http://www.nist.gov/srm/. At the SRM Program homepage, enter the SRM number to access the Material Details web page for SRM 3600. The file can be accessed under the Data Files icon for Data and Information Files. This file provides the user of this SRM with the ability to modify the data in order to interpolate the intensity values for the Q points they actually measure with their own instrumentation. Once the user modifies the data, NIST will not be responsible for the file's content.

When SRM 3600 is used to calibrate SAXS intensities, we should emphasize that all certified intensity values are based on measurements made under ambient conditions. While significant deviations in calibration are not expected for measurements at different temperatures (0 °C to 200 °C or 300 °C) or pressures (vacuum to a few hundred kPa), this is not guaranteed. Furthermore, while SANS measurements made using a specific instrument configuration have been used to provide an independent validation of the certified SAXS calibration intensity values, SRM 3600 is not currently certified for SANS intensity calibration for all SANS measurement configurations.

INSTRUCTIONS FOR STORAGE AND USE

Storage: SRM 3600 should be stored within the plastic membrane box supplied under dry ambient conditions, away from excessive moisture or heat (maximum of 60 °C). SRM 3600 glassy carbon material has been shown to remain stable for calibration purposes over extended periods, with an indefinite shelf-life [5] under these storage conditions.

Use: To use SRM 3600 to provide SAXS intensity calibration for an "unknown" sample using a conventional SAXS instrument with a two-dimensional (2D) position-sensitive detector (PSD), several steps must be followed. Using attenuators as needed to protect the 2D PSD from the incident X-ray beam (same attenuation for blank, sample and standard), measure the transmissions of the sample, T_S, and of the standard, T_{STD}, with respect to the incident beam (blank) measured intensity at $Q = 0$, where $Q = (4\pi/\lambda)\sin\theta$, λ is the X-ray wavelength, and θ is half of the scattering angle [1]. Also determine the position of the incident beam on the 2D PSD from these transmission measurements. Then, with a beam stop in place to protect the 2D PSD from the incident X-ray beam, make SAXS measurements under identical conditions for the "unknown" sample, the blank (no sample present), for an electronic background (X-ray beam off) and for the SRM 3600 standard coupon. Normalize all measured intensities for every 2D PSD pixel to the incident beam intensity, and then subtract out the electronic background for each pixel. For both sample and standard, subtract out the blank scattering, attenuated by the factor, T_S or T_{STD}, respectively, for each PSD pixel. Then circularly-average the 2D PSD data with respect to the incident beam position on the detector (position where $Q = 0$), and divide by both the respective transmission, T_S or T_{STD}, and by the sample or standard thickness, τ_S or τ_{STD}. Note that for SRM 3600, $\tau_{STD} = 1.055$ mm should be used.

Caution: Care must be taken in measurement of the transmissions, T_S and T_{STD}, to minimize both their statistical and systematic fractional uncertainties, as these must be combined with the uncertainties in the certified calibration values presented here in order to determine the total uncertainty in the user's calibration of an "unknown" sample.

Assuming all measurements have been made under the same conditions (including the same incident beam size and collimation), the circularly averaged data, $I'_S(Q)$ and $I'_{STD}(Q)$, are normalized to their respective sampling volumes and are corrected for attenuation in the sample or standard, respectively. Now compare the measured $I'_{STD}(Q)$ *versus* Q with the certified calibration curve, $I_{STD}(Q) = d\Sigma/d\Omega_{STD}$ *versus* Q, on a log-log scale. If the two curves are parallel over the certified Q range, then no further flat background subtraction is required. However, if this is not the case for the data at high Q, then subtract a flat background from the experimental data such that the two curves become parallel

over the certified Q range on the log-log scale. (Some flat background subtraction may also be necessary for the "unknown" sample, but this must be left to the judgment of the user.) Then, by dividing the certified calibration standard curve, $I_{STD}(Q) = d\Sigma/d\Omega_{STD}$ *versus* Q by the *measured* curve for the standard, $I'_{STD}(Q)$ *versus* Q, in the certified Q range specified for valid calibration (Q = 0.008 Å^{-1} to 0.25 Å^{-1}), obtain the intensity calibration factor, CF. Multiply $I'_S(Q)$ by the same factor, CF, to convert the "unknown" sample scattering curve to an absolute calibrated scale in differential scattering cross-section per unit sample volume, and give a curve for absolute-intensity calibrated $I_S(Q) = d\Sigma/d\Omega_S$ *versus* Q. Note that, provided the calibration standard is measured within the valid certified calibration range of Q to determine CF, the calibrated intensity for the "unknown" sample should be valid for all of its measured Q range.

SOURCE, PREPARATION AND ANALYSIS[(1)]

Source and Preparation: Feedstock in the form of Type 2 glassy carbon plates was procured from Alfa Aesar (Ward Hill, MA), a Johnson-Matthey company (London, UK). Each plate was from product no. 38021 and batch no. L27X007, and had dimensions of 100 mm x 100 mm x 1 mm thick (nominal) [6]. The composition of the glassy carbon plates was 100 % carbon by mass. The bulk density for the glassy carbon plate was 1.42 g/cm^3. Given that the theoretical X-ray density of carbon is 2.25 g/cm^3, a total internal porosity of 36.9 % is inferred within the glassy carbon plate material, with the X-ray scattering contrast between the pores and solid glassy carbon ribbons providing the SAXS intensity observed over the extended range in Q of interest for certification.

Each of the glassy carbon plates was sectioned into one hundred 10 mm x 10 mm (nominal) square coupons. Wax was applied to one side of each plate and then a gentle heating was used to effectively "glue" the plate to a flat metal surface on cooling. Ten straight cuts were made across each plate in each of two orthogonal directions using a water-cooled diamond saw to create one hundred ≈ 10 mm square pieces. The assembly was warmed to remove specimens from the metal surface and the wax was then cleaned off in acetone. The grid locations of the individual coupons on each plate were retained for packaging. For each plate, the 100 coupons were oven-heated for 12 h at 120 °C to remove any residue accumulated on the specimens during cutting. Finally, each coupon was packaged into an individual plastic membrane box, which was labeled to indicate the plate and indexed location on the plate.

Certified Values: The certified values are based on USAXS measurements performed under ambient conditions at the APS [7] shown schematically in Figure 1, supplemented by SAXS measurements in a vacuum using the CDSAXS instrument at NIST [8]. The values have been validated by independent SANS measurements at NCNR [9]. Certification of this SRM is for absolute X-ray scattering intensity, given in terms of the X-ray differential scattering cross-section, $d\Sigma/d\Omega$, per unit sample volume of glassy carbon, *versus* the magnitude of the scattering vector, Q, where $Q = (4\pi/\lambda)\sin\theta$, λ is the X-ray wavelength, and θ is half of the scattering angle [1,7]. The scattering angle is determined directly by the scattering geometry of the measurement, while the wavelength, λ, is related to the X-ray energy, E, by $\lambda = hc/E$, where h is Planck's constant and c is the speed of light in vacuum. Calibration of the X-ray energy, E, is traceable using X-ray absorption foils to tables of the fundamental X-ray absorption energies of the elements [3]. First-principle measurements of the $d\Sigma/d\Omega$ values are directly traceable to the following definition: the macroscopic SAXS differential scattering cross-section is the probability per unit incident X-ray flux and per unit sample volume of X-ray scattering into unit solid angle about a direction associated with a given scattering vector, **Q**, where **Q** bisects the incident and scattered beam directions and has magnitude, Q [7]. For an isotropic microstructure such as that of glassy carbon, there is no dependence on azimuthal angle, and the calibrated SAXS intensity consists simply of $d\Sigma/d\Omega$ *versus* Q. Certified $d\Sigma/d\Omega$ values are tabulated in Table 1 for the certified Q range of 0.008 Å^{-1} to 0.25 Å^{-1}, and are plotted in Figure 2.

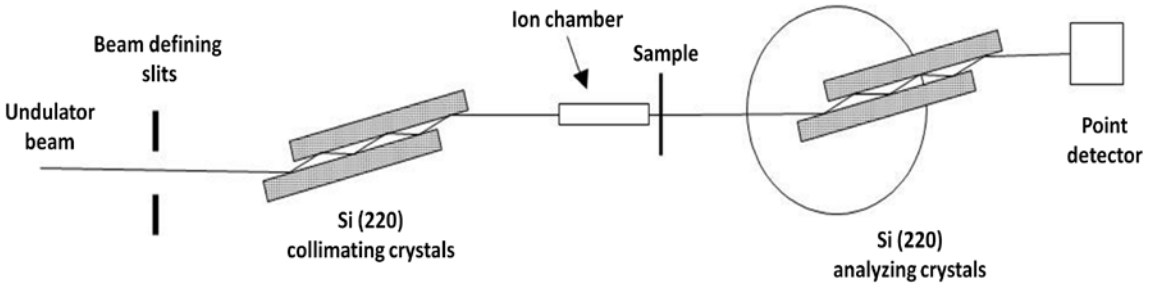

Figure 1. Schematic of USAXS measurement.

(1) Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institutes of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

In USAXS measurements X-ray crystal optics are used both to define the collimated, monochromatic, incident beam (collimating crystals), and to determine the small-angle scattering intensity as a function of Q (analyzing crystals). This is done by rotating the analyzer crystal monolith through and away from the Bragg condition at $Q = 0$. For a given rotation angle, φ, measured from that at $Q = 0$ where the Bragg condition for diffraction through the analyzing crystals is satisfied for the incident beam, only X-rays that have been scattered through an angle $\varphi = 2\theta$ satisfy the Bragg condition of the analyzer crystals. The X-ray point detector incorporates a photodiode that collects the entire beam intensity diffracted by the analyzing crystals as a function of the scattering angle, φ (or 2θ). It has a 10-decade intensity linear dynamic range that is sufficient to capture both the weak SAXS intensity and the full intensity of the primary X-ray beam (at $Q = 0$) within a single scan, without distortions arising from detector saturation or from X-ray and electronic background noise. Note that USAXS scans are made in one azimuthal direction in Q and are not circularly-averaged. Also, the intensity data are "slit-smeared" in the direction orthogonal to the scan over a transverse scattering-angle range given by the photodiode detector aperture subtended at the sample position. The data are later desmeared as described below. The ion chamber, placed before the sample, records any temporal variations in the incident beam flux and is used to normalize out any corresponding temporal fluctuations in the photodiode signal not associated with the sample. The APS USAXS instrument has evolved from previous instrumentation developed by NIST at the National Synchrotron Light Source (NSLS), Brookhaven National Laboratory (Upton, NY) [10]. Its principles of operation, sample requirements, and evolving capabilities and performance specifications over more than 28 years have been documented in a series of publications by NIST authors and our collaborators [7,11]. The instrument has operated in its present form for 16 years providing primary (standard-less) absolute intensity calibration of the USAXS data collected from every sample studied, in terms of the SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega$.

The experimentally measured $d\Sigma/d\Omega$ for USAXS measurements of SRM 3600 are given initially by:

$$\frac{d\tilde{\Sigma}}{d\Omega}_{STD} = \frac{I(Q)}{I_0(0)} \frac{1}{T_{STD}\tau_{STD}\Delta\Omega} \tag{1}$$

where the tilde indicates the data are slit-smeared; $I_O(0)$ is the measured intensity without a sample present at $Q = 0$; T_{STD} is the coupon X-ray transmission, i.e., the ratio of the intensity at $Q = 0$ with the coupon present, $I(0)$, to that with no coupon present, $I_O(0)$; τ_{STD} is the coupon thickness and $\Delta\Omega$ is the solid angle associated with the scattered intensity measurement at a given Q value.

For USAXS measurements, $\Delta\Omega$ is given by:

$$\Delta\Omega = \Delta\varphi_C 2\Theta_H \tag{2}$$

where $\Delta\varphi_C$ is the angular full width at half maximum (FWHM) of the analyzing crystals rocking curve in the diffraction plane and $2\Theta_H$ is the angle subtended at the sample position by the photodiode detector aperture in the orthogonal plane to the diffraction plane. The photodiode detector response to X-ray flux has sufficient linear dynamic range in measured intensity to permit measurements over the incident X-ray beam profile at $Q = 0$ to give $d\Sigma/d\Omega$ directly using equation (1). USAXS provides an accessible Q range from 0.0001 Å^{-1} to 1.0 Å^{-1}, which encompasses the range of virtually all SAXS instruments. The range certified for SRM 3600 SAXS intensity calibration is more restricted: 0.008 Å^{-1} to 0.25 Å^{-1}, due to the form of the glassy carbon scattering curve as a function of Q. However, this remains sufficient to cover the range of most practical SAXS instruments, worldwide.

Sources of uncertainty with reference to the terms in equations (1) and (2) are as follows:

The uncertainty in Q for every USAXS scan is that associated with fitting the angular position of $\varphi = 0$ (and $Q = 0$) for the main encoded rotational stage motion of the analyzing crystal pair. This is the stage rotation that gives the scattering angle, φ, from which the corresponding Q value is derived during a USAXS scan. This zero position is determined from modified Gaussian fits to the intensity peak profiles of the incident beam, $I(Q)$ and $I_O(Q)$, etc., in the vicinity of $\varphi = 0$ (hence, $Q = 0$). The fits determine the peak position, which then defines $Q = 0$ for a given USAXS scan to an uncertainty at least one order of magnitude smaller than the rocking curve width of 10^{-4} Å^{-1}, which is the Q resolution for the USAXS scan. The uncertainty with which $Q = 0$ is determined is matched by the precision of the encoded analyzing crystal stage rotation in tracking φ and Q throughout the USAXS scan. Thus, we conclude that the uncertainty in Q, based on geometric uncertainty is $< 10^{-5}$ Å^{-1} while the Q resolution is 10^{-4} Å^{-1}. There is also a contribution to the uncertainty in Q arising from that in the X-ray energy or wavelength, λ. However, the APS undulators and monochromators are all ultimately energy-calibrated to within an uncertainty of ± (2 to 3) eV, using standard absorption foils with known X-ray absorption edges for each element derived from traceable X-ray absorption tables [3,12]. Given the APS X-ray energies used for the certification (10.5 keV and up), we conclude that the overall uncertainty in Q is always < 0.05 %, which can be neglected for this certification.

The user is instructed to set τ_{STD} = 1.055 mm for use of SRM 3600. The mean thickness of the SRM 3600 coupons, based on two separate micrometer thickness measurements on each of 56 coupons sampled is 1.055 mm ± 0.025 mm. The uncertainty listed with the value is the expanded uncertainty with coverage factor k = 2, corresponding to a 95 % confidence interval, expected for a single coupon, and is derived from the standard deviation of replicate measurements. However, users should use a value of 1.055 mm, rather than a measurement of their own coupon thickness, because the thickness variability is already incorporated into the uncertainties due to coupon variability tabulated later in this document. The intensity measurements with and without sample present, $I(Q)$ and $I_O(Q)$, are based on measures of photodiode current, which cannot be treated with conventional Poisson statistics. Ion chamber counts for the incident beam (to which $I(Q)$, etc. are normalized) do follow Poisson statistics but the ion chamber counts are sufficiently high as to render their statistical uncertainty negligible. In any case, individual point-to-point scatter in intensity for neighboring $I(Q)$ values is not relevant to the calibration certification; it is the uncertainty in the overall intensity of $I(Q)$ *versus* Q over the certified Q range that is relevant here. Uncertainties in the primary (incident) beam intensity at $Q = 0$ with and without the coupon present, $I(0)$ and $I_O(0)$, are more significant for calibration. They are determined by modified Gaussian fits to the intensity peak profiles in the vicinity of $Q = 0$. Uncertainties in the fit values, and hence in their ratio, T_S, are significant contributors to the overall calibration uncertainty for repeated USAXS measurements with a given USAXS set-up configuration. Uncertainties in $\Delta\Omega$ comprise those in $\Delta\varphi_C$ and those in $2\Theta_H$. The value of $\Delta\varphi_C$ is determined from the modified Gaussian fit of the peak profile in $I(Q)$ in the vicinity of $Q = 0$. Again, uncertainties in the fit values for successive USAXS scans are significant contributors to the overall calibration uncertainty for repeated USAXS measurements with a given USAXS set-up configuration. However, we note that the uncertainty in the fitted $\Delta\varphi_C$ value may not be independent of that in the corresponding fitted $I(0)$ value. The uncertainty in $2\Theta_H$ arises differently and is fixed for a given USAXS set-up. The value of $2\Theta_H$ is the photodiode detector aperture orthogonal to the USAXS diffraction plane, 5.5 mm with negligible uncertainty for calibration purposes, divided by the sample-to-detector distance, SDD. Typically, SDD ≈ 900 mm, but it is physically measured for each USAXS set-up with a typical uncertainty of a few millimeters. This contributes a USAXS set-up uncertainty in the intensity, i.e. a bias for all USAXS measurements made with the given set-up, of ≈ 0.5 %. A more significant USAXS set-up uncertainty of a few percent arises from the need to adjust and minimize the crystal tilts within the collimating and analyzer crystal pairs [7]. The tilt angle of any one crystal orientation is the transverse deviation of its own diffraction plane out of the USAXS diffraction plane shown in the schematic of Figure 1 (vertical at APS). During USAXS set-up for measurements at any given X-ray energy, the relative tilts of the two crystals within each of the collimating and analyzing crystal pairs must be minimized, as must the tilt of the overall collimating and analyzing crystal pair diffraction planes with respect to each other. Depending on where it occurs, failure to minimize the crystal tilts can lead to intensity calibrations that are too high or too low. This was found to be the largest uncertainty in the certification of SRM 3600.

To determine the actual measurement uncertainties, replicate measurements were made, both for given USAXS energy set-ups, and across multiple USAXS energy set-ups. Thus, all uncertainties were treated as Type A in accordance with the ISO/JCGM [13], and no significant Type B uncertainties are known.

As a first step in the certification of SRM 3600, 56 coupons were carefully selected that had been cut from the centers, edges, corners and intermediate regions of the different glassy carbon plates. USAXS measurements were made, on all 56 coupons at a X-ray energy of 12.0 keV, some repeatedly. Following data reduction using the USAXS instrument data reduction and analysis algorithms [14] as outlined above, the slit-smeared differential scattering cross-section per unit sample volume *versus* Q data were compared and a full regression analysis carried out using global spline, spatial, and local models. Both within the measurement uncertainties found at the time, and within the overall uncertainties now established for the certification of SRM 3600, it was established that no significant dependence exists in the SRM 3600 calibrated intensity with respect to the plate from which a given coupon was cut, or with respect to the position on that plate. Thus, further certification for given USAXS energies and set-ups, and with respect to different USAXS energies and set-ups could be conducted with a smaller sub-set of SRM 3600 coupons.

All further certification has been carried out with regard to desmeared USAXS data. The slit-smeared, calibrated USAXS data, determined using equation (1), have been desmeared using the well-known algorithm by Lake [15]. This algorithm iteratively removes the slit-smearing effect to provide desmeared data that reproduce the starting slit-smeared data when smeared using the known instrument configuration, to within the experimental uncertainties provided with the input data. The Lake algorithm has been well-tested for desmearing USAXS data over its entire twenty-eight-year history [7,10,11]. Within statistical uncertainties, desmeared USAXS data are found to be in agreement with other unsmeared SAXS and SANS data where available, and desmeared data agree with predicted models for the unsmeared data. Similarly, direct agreement, within statistical uncertainties, is obtained between desmeared USAXS data for strongly scattering samples, derived from slit-smeared data using the standard configuration of Figure 1, and unsmeared USAXS data, measured with additional transverse crystal optics present that remove the slit-smearing effect, altogether. (Unfortunately, glassy carbon does not scatter strongly enough to permit a direct test.) The main correction to the data on desmearing is to increase the scattering at Q values less than the

transverse slit length (measured in Q units), increasingly so for smaller Q values. The point-to-point data scatter in Q is also increased at low Q, but this does not affect the calibration uncertainty.

For certification purposes, slit-smeared, calibrated $d\Sigma/d\Omega_{STD}$ *versus* Q data were obtained for multiple SRM 3600 coupons at each of the following X-ray energies: 10.5 keV (four coupons), 11.5 keV (16 coupons), 12.0 keV (16 coupons) and 16.8 keV (four coupons). The four coupons measured at 10.5 keV and 16.8 keV were also included among the 16 measured at 11.5 keV and 12.0 keV. In each case, the data were averaged and the deviations of the N individual datasets from the mean at each Q were used to obtain the experimental mean standard deviation at each Q, consistent with the $\{N - 1\}$ degrees of freedom [13]. These were used as the uncertainties for each slit-smeared dataset on input for desmearing. The process was repeated again for the desmeared, calibrated $d\Sigma/d\Omega_{STD}$ *versus* Q data. However, for the calibration certification, the individual desmeared datasets were compared with the average dataset in each case using the USAXS data comparison algorithms. The absolute intensity calibration of each individual desmeared USAXS dataset was compared with the mean dataset over the Q range for certification. The fractional deviations of the individual calibrated datasets with respect to the mean were used to obtain the experimental mean fractional standard deviation, again consistent with the $\{N - 1\}$ degrees of freedom [13]. In this way, the standard uncertainties for calibration associated with repeated measurements and coupon variability were determined for each USAXS energy set-up. These were found comparable for each set-up, and did not vary with X-ray energy in any systematic way. Thus, after the supplemental 2D PSD SAXS measurements (discussed below) were used to separate out the uncertainty due to coupon variability, the uncertainty for repeated USAXS measurement with a given set-up was based on 40 measurements but 36 degrees of freedom. Meanwhile, it was found that the results for the two X-ray energies with 16 coupons (15 degrees of freedom) were not significantly changed if the analysis was confined to just the four coupons measured at 10.5 keV and 16.8 keV (three degrees of freedom); so just these four coupons were used to determine the uncertainty associated with USAXS set-up at a given X-ray energy. Then, by repeating the statistical analysis for the four mean desmeared datasets for each of the USAXS energy set-ups, with respect to the mean of all four, the experimental mean fractional standard uncertainty for calibration associated with USAXS energy set-up was determined (three degrees of freedom) [13]. No monotonic or other systematic variation in the intensity calibration is found with X-ray energy, and we assume this uncertainty to be statistically associated more with USAXS set-up, rather than selection of a specific X-ray energy as such.

Supplemental conventional 2D PSD SAXS measurements were carried out at NIST on 16 SRM 3600 coupons using the CDSAXS instrument [8]. This instrument used Copper $K\alpha$ radiation with X-ray energy, 8.063 keV and wavelength, 1.5418 Å. Only part of the certified Q range was covered in these measurements ($Q > 0.04$ Å^{-1}). Data were normalized to the incident beam intensity, and the *Blank* (empty beam) data were subtracted out using standard SAXS instrument algorithms [16]. The normalized 2D data were circularly averaged, but the intensity was not calibrated and the sample transmission and thickness were not divided out. For these measurements, the instrument 2D detector counts for a given time interval obeyed Poisson statistics, and the counts were sufficiently high that statistical uncertainties were negligible. Furthermore, these measurements confirmed that the maximum Q of 0.25 Å^{-1} for certification did not require any flat background subtraction for certification. With appropriate normalization to the calibrated desmeared USAXS data, these uncalibrated SAXS datasets could be readily merged with the desmeared USAXS data and used to smooth the data at high Q. However, the 16 reduced datasets of uncalibrated intensity *versus* Q were actually averaged among themselves, and the intensity of each individual dataset compared to the mean to give the experimental mean fractional standard uncertainty for the intensity (with 15 degrees of freedom) [13]. This fractional standard uncertainty was found to be less than that for the coupon thickness. This is expected because no allowance had been made to normalize out the product, $\tau_S T_S$, and at this X-ray energy, variations in T_S partially compensate for those in τ_S in affecting the overall relative SAXS intensity. We conclude, therefore, that variations in the relative SAXS intensity found for the 2D PSD SAXS measurements must be dominated by that due to coupon variation, and that the coupon variability in SAXS intensity must be largely due to variation in coupon thickness. With this information, the uncertainty in USAXS calibrated intensity (above) associated with repeated measurements for a given USAXS set-up was separated from those associated with coupon variation. We note that the higher USAXS energies have T_S much closer to one than at the Cu $K\alpha$ energy, and the reduction in intensity variations due to τ_S variability being partially compensated by a complementary variability in T_S is much reduced. Given this point, we conservatively estimate the intensity calibration uncertainty due to sample variability to match that of the coupon thickness variation in the general case, and we have used this larger uncertainty due to coupon variation in computing the total measurement uncertainty.

Uncertainties: The expanded uncertainties listed in Table 1 were calculated according to the ISO/JCGM Guide [13]. These uncertainties correspond to the expanded uncertainties in the SAXS differential scattering cross-section per unit sample volume, and define a confidence interval of approximately 95 %. The combined and expanded uncertainties were derived using inputs from the coupon variability, sampling reproducibility, and USAXS set-up uncertainties. All were treated as Type A (random) statistical uncertainties, arising from the sources described above.

Table 1. Certified values for $d\Sigma/d\Omega_{STD}$ with Measurement Uncertainties for SRM 3600

Q[a] (Å^{-1})	$d\Sigma/d\Omega_{STD}$[b] (cm^{-1} sr^{-1})	Coupon[c,d] Variability (N = 56) (u = 1.14 %) (cm^{-1} sr^{-1})	Given Set-up[c,e] Measurement Replicability (N = 40) (0.88 %) (cm^{-1} sr^{-1})	Different USAXS[c,f] Set-ups Replicability (N = 4) (2.14 %) (cm^{-1} sr^{-1})	u_C (2.58 %) (cm^{-1} sr^{-1})	U (6.25 %) (cm^{-1} sr^{-1})
0.00827568	34.933380	0.398241	0.307414	0.747574	0.901092	2.183336
0.00888450	34.427156	0.392470	0.302959	0.736741	0.888034	2.151697
0.00954735	34.042170	0.388081	0.299571	0.728502	0.878103	2.127636
0.01026900	33.698553	0.384164	0.296547	0.721149	0.869240	2.106160
0.01105780	33.352529	0.380219	0.293502	0.713744	0.860314	2.084533
0.01191830	33.027533	0.376514	0.290642	0.706789	0.851931	2.064221
0.01286110	32.665045	0.372382	0.287452	0.699032	0.842581	2.041565
0.01389340	32.306665	0.368296	0.284299	0.691363	0.833337	2.019167
0.01502510	31.970485	0.364464	0.281340	0.684168	0.824665	1.998155
0.01626850	31.559099	0.359774	0.277720	0.675365	0.814053	1.972444
0.01763650	31.183763	0.355495	0.274417	0.667333	0.804372	1.948985
0.01914320	30.861805	0.351825	0.271584	0.660443	0.796067	1.928863
0.02080510	30.514300	0.347863	0.268526	0.653006	0.787103	1.907144
0.02264220	30.084982	0.342969	0.264748	0.643819	0.776029	1.880311
0.02467500	29.690414	0.338471	0.261276	0.635375	0.765852	1.855651
0.02692890	29.249965	0.333450	0.257400	0.625949	0.754490	1.828123
0.02943170	28.889970	0.329346	0.254232	0.618245	0.745204	1.805623
0.03221560	28.449341	0.324322	0.250354	0.608816	0.733839	1.778084
0.03531810	28.065980	0.319952	0.246981	0.600612	0.723950	1.754124
0.03878270	27.704965	0.315837	0.243804	0.592886	0.714638	1.731560
0.04265880	27.331304	0.311577	0.240515	0.584890	0.704999	1.708207
0.04700390	26.974065	0.307504	0.237372	0.577245	0.695784	1.685879
0.05188580	26.676952	0.304117	0.234757	0.570887	0.688121	1.667309
0.05738140	26.401158	0.300973	0.232330	0.564985	0.681007	1.650072
0.06358290	26.177427	0.298423	0.230361	0.560197	0.675236	1.636089
0.07059620	25.904683	0.295313	0.227961	0.554360	0.668200	1.619043
0.07854840	25.528734	0.291028	0.224653	0.546315	0.658503	1.595546
0.08758630	24.917743	0.284062	0.219276	0.533240	0.642743	1.557359
0.09788540	23.946472	0.272990	0.210729	0.512455	0.617689	1.496655
0.10965500	22.472101	0.256182	0.197754	0.480903	0.579658	1.404506
0.11431200	21.777228	0.248260	0.191640	0.466033	0.561734	1.361077
0.11839500	21.112938	0.240687	0.185794	0.451817	0.544599	1.319559
0.12262400	20.401110	0.232573	0.179530	0.436584	0.526238	1.275069
0.12314200	20.287060	0.231272	0.178526	0.434143	0.523296	1.267941
0.12700400	19.685107	0.224410	0.173229	0.421261	0.507769	1.230319
0.13154000	18.909809	0.215572	0.166406	0.404670	0.487770	1.181863
0.13623900	18.089242	0.206217	0.159185	0.387110	0.466604	1.130578
0.13864300	17.679572	0.201547	0.155580	0.378343	0.456037	1.104973
0.14110500	17.264117	0.196811	0.151924	0.369452	0.445321	1.079007
0.14614500	16.372848	0.186650	0.144081	0.350379	0.422331	1.023303
0.15136500	15.458350	0.176225	0.136033	0.330809	0.398742	0.966147
0.15651300	14.587700	0.166300	0.128372	0.312177	0.376284	0.911731
0.15677100	14.563071	0.166019	0.128155	0.311650	0.375648	0.910192
0.16237100	13.616671	0.155230	0.119827	0.291397	0.351236	0.851042

[a] The scattering vector magnitude, Q.

[b] The SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega_{STD}$, the standard uncertainty components (u), the combined standard uncertainties (u_C) and the expanded uncertainties (U) in the SAXS differential scattering cross-section per unit sample volume (95 % confidence interval). All u, u_C and U uncertainties in calibration are also indicated as percentage fractions of the certified $d\Sigma/d\Omega_{STD}$ value. The k value of 2.4231 was determined by statistical methods. All calculations made according to the JCGM Guide [13].

[c] Type A uncertainties evaluated by statistical methods.

[d] Uncertainties for coupon variability are for a single coupon.

[e] Uncertainties based on 40 measurements distributed across 4 USAXS set-ups, hence 36 degrees of freedom.

[f] Uncertainties based on four USAXS set-ups at different X-ray energies, hence three degrees of freedom.

(continued on next page)

Table 1. Certified values for $d\Sigma/d\Omega_{STD}$ with Measurement Uncertainties for SRM 3600 (Continued)

Q[a] (Å^{-1})	$d\Sigma/d\Omega_{STD}$[b] (cm^{-1} sr^{-1})	Coupon[c,d] Variability (N = 56) (u = 1.14 %) (cm^{-1} sr^{-1})	Given Set-up[c,e] Measurement Replicability (N = 40) (0.88 %) (cm^{-1} sr^{-1})	Different USAXS[c,f] Set-ups Replicability (N = 4) (2.14 %) (cm^{-1} sr^{-1})	u_C (2.58 %) (cm^{-1} sr^{-1})	U (6.25 %) (cm^{-1} sr^{-1})
0.16817000	12.668549	0.144421	0.111483	0.271107	0.326780	0.791784
0.17417700	11.752287	0.133976	0.103420	0.251499	0.303145	0.734518
0.17718100	11.311460	0.128951	0.099541	0.242065	0.291774	0.706966
0.18039800	10.862157	0.123829	0.095587	0.232450	0.280185	0.678885
0.18684100	9.961979	0.113567	0.087665	0.213186	0.256965	0.622624
0.19351500	9.116906	0.103933	0.080229	0.195102	0.235167	0.569807
0.20042700	8.325578	0.094912	0.073265	0.178167	0.214755	0.520349
0.20116500	8.224897	0.093764	0.072379	0.176013	0.212158	0.514056
0.20758600	7.541931	0.085978	0.066369	0.161397	0.194541	0.471371
0.21500000	6.854391	0.078140	0.060319	0.146684	0.176806	0.428399
0.22267900	6.216070	0.070863	0.054701	0.133024	0.160341	0.388504
0.22909500	5.715911	0.065161	0.050300	0.122321	0.147439	0.357244
0.23063300	5.582366	0.063639	0.049125	0.119463	0.143995	0.348898
0.23887100	4.999113	0.056990	0.043992	0.106981	0.128950	0.312445
0.24740200	4.463604	0.050885	0.039280	0.095521	0.115137	0.278975

[a] The scattering vector magnitude, Q.

[b] The SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega_{STD}$, the standard uncertainty components (u), the combined standard uncertainties (u_C) and the expanded uncertainties (U) in the SAXS differential scattering cross-section per unit sample volume (95 % confidence interval). All u, u_C and U uncertainties in calibration are also indicated as percentage fractions of the certified $d\Sigma/d\Omega_{STD}$ value. The k value of 2.4231 was determined by statistical methods. All calculations made according to the JCGM Guide [13].

[c] Type A uncertainties evaluated by statistical methods.

[d] Uncertainties for coupon variability are for a single coupon.

[e] Uncertainties based on 40 measurements distributed across 4 USAXS set-ups, hence 36 degrees of freedom.

[f] Uncertainties based on four USAXS set-ups at different X-ray energies, hence three degrees of freedom.

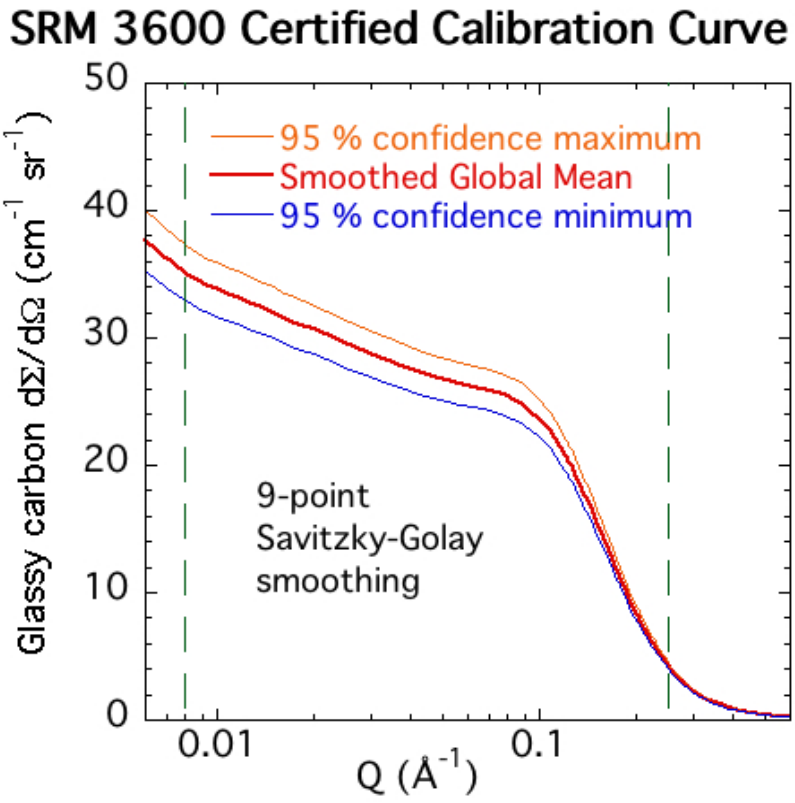

Figure 2. Smoothed calibrated SAXS intensity *versus* Q. Vertical dashed lines indicate the certification range in Q.

The SRM 3600 certified calibration curve for $d\Sigma/d\Omega_{STD}$ *versus Q*, is presented in Figure 2, together with the computed 95 % confidence minimum and maximum envelope, based on the certified values presented in Table 1. In computing the mean SAXS intensity calibration curve and its uncertainties, we note that the latter arise from fractional uncertainties in the calibrated intensity, regardless of *Q*. Also, 9-point Savitzky-Golay smoothing has been applied to reduce the residual point-to-point scatter in the USAXS data [17].

REFERENCE VALUES FOR SANS MEASUREMENTS

The primary means of validating the USAXS/SAXS certified values using a second independent method has been to conduct independent measurements at NCNR, using the NIST/NSF 30 m SANS instrument [9] to obtain the neutron $d\Sigma/d\Omega_{SANS\ STD}$ *versus Q* data, and rescale these for SAXS. The principles of small-angle scattering apply both to SAXS and to SANS [1]. SANS measurements were made using a 2D PSD sensitive to neutrons. Using the standard NCNR SANS data reduction and analysis algorithms [18], scattering intensity in each detector pixel was corrected for detector sensitivity and for any effects due to the sample and detector geometries. Parasitic (empty beam) scattering and electronic backgrounds were subtracted out, and correction made for sample attenuation (mainly sample absorption). The corrected scattering intensity was then calibrated with respect to the incident beam intensity, the latter being measured by a fission monitor detector. Because the scattering intensity and incident beam intensity were measured on the same relative scale, the absolute scattering intensity, i.e., the differential scattering cross-section, $d\Sigma/d\Omega_{SANS\ STD}$, could be measured directly from the sample configuration and scattering geometry. In practice, the small-angle scattering was measured with a beam-stop in front of the 2D PSD to protect it from being damaged by the incident beam. The incident beam intensity was measured with the beam-stop removed, with and without the sample present. A calibrated neutron beam attenuator was used to protect the detector from the full incident beam intensity. Prior calibration of a complete set of SANS instrument attenuators was carried out with the beam stop in place by comparing the measured 2D PSD scattering intensities from a strongly scattering sample using different attenuator thicknesses. Since the small-angle scattering from SRM 3600 coupons is circularly symmetric, the SANS data were circularly averaged around the incident beam position on the 2D PSD to provide 1D SANS data of $d\Sigma/d\Omega_{SANS\ STD}$ *versus Q*. This "calibrated attenuator" mode of SANS intensity calibration is now in widespread use around the world. Details of the 30 m SANS instruments at NCNR, as well as the data reduction and analysis software packages are given elsewhere [9].

SANS measurements were carried out on eight SRM 3600 glassy carbon coupons. A sample thickness of 1.055 mm was assumed for all eight coupons. The shortest possible neutron wavelength, λ, of 5.05 Å was used with $\Delta\lambda/\lambda$ = 13.1 %, defined by a rotating helical-slot velocity selector, which gives a triangular, not Gaussian transmission function in λ. This implies that $\Delta Q/Q \approx 26$ % due to wavelength spread alone, with geometrical collimation effects making a small additional contribution. While the *Q* resolution is broad, it is found sufficient to capture the shape of the scattering profile in *Q* in all practical cases. Three sample-to-detector measurement configurations were used, each with approximately matching incident beam collimation conditions defined by inserting a different number of neutron waveguides in the beam. Data were reduced, calibrated and circularly averaged for each measurement configuration, separately. Then, the three 1D datasets associated with each SRM 3600 coupon were internormalized and merged using the SANS instrument data reduction package [18] to obtain a single 1D SANS dataset covering a *Q* range from 0.0046 $Å^{-1}$ to 0.39 $Å^{-1}$, which covers the range for certification. While data for the three SANS instrument configurations were calibrated independently, and their internormalization factors were close to one, the 1D dataset corresponding to the largest sample-to-detector configuration (data at smallest *Q* values) was used as the primary file for calibration. Thus, the overall SANS intensity calibration was effectively with respect to the most tightly collimated incident neutron beam (longest incident and scattered collimation distances). This situation most closely resembles the USAXS absolute intensity calibration with X-rays. Meanwhile data from all three configurations were merged together to form one calibrated 1D dataset for each SRM 3600 coupon.

The counting statistics for both the fission beam monitor and main SANS instrument 2D PSD obey Poisson statistics. However, the eight complete datasets for the SRM coupons were treated statistically in the same way as for the USAXS/SAXS datasets in accordance with the ISO/JCGM guide [13]. The eight datasets were averaged and the deviations of the eight individual datasets from the mean were used to obtain the experimental mean standard deviation at each *Q*, consistent with seven degrees of freedom. As in the X-ray case, these data included point-to-point scatter that does not contribute to the uncertainty in overall intensity calibration. Therefore, each dataset was compared with the mean to determine the overall fractional deviations from the mean calibrated dataset. These fractional deviations of the individual calibrated datasets with respect to the mean were used to obtain the experimental mean fractional standard deviation, again consistent with the seven degrees of freedom [13]. The statistical uncertainty obtained arises from a combination of coupon variability and repeated SANS measurements. Again, the supplemental 2D SAXS results were used to separate these two sources of uncertainty, and determine that due to repeated SANS measurements, alone. However, as with the X-ray case, the full coupon variability, associated with thickness variation was used in computing the overall combined uncertainties.

In the case of SANS, a much more significant uncertainty in the intensity calibration is associated with instrument set-up. This arises from multiple sources including neutron guide and velocity selector alignment, and alignment of the various beam apertures, as well as the beam stop for the selected instrument configurations. Based on more than 20 years of operational experience at NCNR, the standard fractional uncertainty in calibration is estimated to be approximately 5 % [9]. However, since only one set of three instrument configurations (with data merged and normalized to the lowest Q configuration) used in the validation process, this is treated here as a Type B uncertainty [13].

For validation of the USAXS/SAXS intensity calibration, the neutron $d\Sigma/d\Omega_{\text{SANS STD}}$ data have been re-scaled as follows. In both SAXS and SANS, the SRM 3600 small-angle scattering arises from the contrast between the glassy carbon ribbons (carbon) and voids (air or vacuum). For SAXS the scattering contrast factor (to which the intensity is proportional) is given by the square of the atomic form-factor density near $Q = 0$ for the solid carbon in each SRM 3600 coupon. For SANS, the scattering contrast factor is the square of the nuclear coherent scattering length density for the solid carbon in the coupon. Traceable look-up tables are available both for the atomic X-ray form-factors of the elements [3], and for the neutron coherent scattering lengths of each element (and isotope) [4]. While the absolute solid glassy carbon density can only be estimated (likely to be slightly denser than graphite, but much closer to graphite than to diamond [8]), the ratio of the scattering contrast factors is simply the ratio of the squares of the carbon X-ray form-factor and neutron scattering length, whatever the exact glassy carbon density. The ratio = 6.409 with variations of < 0.5 % over the range of X-ray energies used, and this factor was used in the rescaling of the $d\Sigma/d\Omega_{\text{SANS STD}}$ values.

In computing the mean SANS intensity calibration curve and its uncertainties, rescaled for SAXS, we note that the uncertainties arise from fractional uncertainties in the calibrated intensity, regardless of Q. Once again, nine-point Savitzky-Golay smoothing has been applied to reduce the residual point-to-point scatter in the SANS data [17].

Table 2. Reference values for $d\Sigma/d\Omega_{\text{STD}}$ with Measurement Uncertainties for SRM 3600

Q[a]	$d\Sigma/d\Omega_{\text{STD}}$[b] rescaled for SAXS by factor of 6.409	Coupon[c,d] Variability (N = 56) (u = 1.14 %)	Given Set-up[c,e] Measurement Replicability (N = 8) (0.22 %)	Different SANS[f,g] Set-ups Replicability (N = large) (5.00 %)	u_C (5.13 %)	U (10.06 %)
(Å^{-1})	(cm^{-1} sr^{-1})	(cm^{-1} sr^{-1})	(cm^{-1} sr^{-1})	(cm^{-1} sr^{-1})	(cm^{-1} sr^{-1})	(cm^{-1} sr^{-1})
0.008398	36.254550	0.413302	0.079760	1.812728	1.860957	3.648034
0.008878	35.713234	0.407131	0.078569	1.785662	1.833171	3.593565
0.009358	35.280862	0.402202	0.077618	1.764043	1.810977	3.550059
0.009838	34.894925	0.397802	0.076769	1.744746	1.791167	3.511225
0.010320	34.517623	0.393501	0.075939	1.725881	1.771800	3.473260
0.010740	34.158574	0.389408	0.075149	1.707929	1.753370	3.437131
0.010800	34.157291	0.389393	0.075146	1.707865	1.753304	3.437002
0.011280	33.993941	0.387531	0.074787	1.699697	1.744919	3.420565
0.011760	33.746618	0.384711	0.074243	1.687331	1.732224	3.395679
0.012010	33.603744	0.383083	0.073928	1.680187	1.724890	3.381303
0.012240	33.570798	0.382707	0.073856	1.678540	1.723199	3.377987
0.012720	33.389290	0.380638	0.073456	1.669465	1.713882	3.359724
0.013200	33.120235	0.377571	0.072865	1.656012	1.700072	3.332651
0.013270	33.091010	0.377238	0.072800	1.654550	1.698572	3.329710
0.013680	33.019949	0.376427	0.072644	1.650997	1.694924	3.322560
0.014160	32.768811	0.373564	0.072091	1.638441	1.682033	3.297289
0.014540	32.574223	0.371346	0.071663	1.628711	1.672045	3.277709
0.014640	32.499631	0.370496	0.071499	1.624982	1.668216	3.270204
0.015120	32.387256	0.369215	0.071252	1.619363	1.662448	3.258896
0.015600	32.229122	0.367412	0.070904	1.611456	1.654331	3.242984
0.015800	32.131216	0.366296	0.070689	1.606561	1.649305	3.233133
0.016080	32.098653	0.365925	0.070617	1.604933	1.647634	3.229856

[a] The scattering vector magnitude, Q.

[b] The SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega_{\text{SANS STD}}$ rescaled for SAXS, the standard uncertainty components (u), the combined standard uncertainties (u_c) and the expanded uncertainties (U) in the SAXS differential scattering cross-section per unit sample volume (95 % confidence interval). All u, u_C and U uncertainties in calibration are also indicated as percentage fractions of the rescaled $d\Sigma/d\Omega_{\text{SANS STD}}$ value. The k value of 1.9603 was determined by statistical methods. All calculations made according to the JCGM Guide [13].

[c] Type A uncertainties evaluated by statistical methods.

[d] Uncertainties for coupon variability are for a single coupon.

[e] Uncertainties based on eight measurements with one SANS set-up, hence seven degrees of freedom.

[f] Type B uncertainties evaluated by other means.

[g] Uncertainties based on literature estimate and long instrument history.

(continued on next page)

Table 2. Reference values for $d\Sigma/d\Omega_{STD}$ with Measurement Uncertainties for SRM 3600 (Continued)

Q[a] (Å^{-1})	$d\Sigma/d\Omega_{STD}$[b] rescaled for SAXS by factor of 6.409 (cm^{-1} sr^{-1})	Coupon[c,d] Variability (N = 56) (u = 1.14 %) (cm^{-1} sr^{-1})	Given Set-up[c,e] Measurement Replicability (N = 8) (0.22 %) (cm^{-1} sr^{-1})	Different SANS[f,g] Set-ups Replicability (N = large) (5.00 %) (cm^{-1} sr^{-1})	u_C (5.13 %) (cm^{-1} sr^{-1})	U (10.06 %) (cm^{-1} sr^{-1})
0.016560	31.922813	0.363920	0.070230	1.596141	1.638608	3.212163
0.017040	31.754424	0.362000	0.069860	1.587721	1.629964	3.195219
0.017060	31.737441	0.361807	0.069822	1.586872	1.629092	3.193510
0.017520	31.564552	0.359836	0.069442	1.578228	1.620218	3.176113
0.018000	31.375155	0.357677	0.069025	1.568758	1.610496	3.157056
0.018330	31.240158	0.356138	0.068728	1.562008	1.603567	3.143472
0.018480	31.190148	0.355568	0.068618	1.559507	1.601000	3.138440
0.018960	31.093882	0.354470	0.068407	1.554694	1.596058	3.128753
0.019430	31.072508	0.354227	0.068360	1.553625	1.594961	3.126603
0.019590	31.077029	0.354278	0.068369	1.553851	1.595193	3.127057
0.019910	30.984730	0.353226	0.068166	1.549236	1.590456	3.117770
0.020390	30.923391	0.352527	0.068031	1.546170	1.587307	3.111598
0.020860	30.786942	0.350971	0.067731	1.539347	1.580303	3.097868
0.020870	30.779594	0.350887	0.067715	1.538980	1.579926	3.097129
0.021350	30.557618	0.348357	0.067227	1.527881	1.568532	3.074793
0.021830	30.405512	0.346623	0.066892	1.520276	1.560724	3.059488
0.022120	30.387206	0.346414	0.066852	1.519360	1.559784	3.057646
0.022310	30.330074	0.345763	0.066726	1.516504	1.556852	3.051897
0.022790	30.235904	0.344689	0.066519	1.511795	1.552018	3.042421
0.023270	30.167856	0.343914	0.066369	1.508393	1.548525	3.035574
0.023380	30.133794	0.343525	0.066294	1.506690	1.546777	3.032147
0.023750	29.969732	0.341655	0.065933	1.498487	1.538355	3.015638
0.024230	29.924238	0.341136	0.065833	1.496212	1.536020	3.011060
0.024650	29.928915	0.341190	0.065844	1.496446	1.536260	3.011531
0.024710	29.888051	0.340724	0.065754	1.494403	1.534163	3.007419
0.025190	29.845786	0.340242	0.065661	1.492289	1.531993	3.003166
0.025670	29.858232	0.340384	0.065688	1.492912	1.532632	3.004419
0.025910	29.828586	0.340046	0.065623	1.491429	1.531110	3.001436
0.026150	29.740201	0.339038	0.065428	1.487010	1.526574	2.992542
0.026630	29.561376	0.337000	0.065035	1.478069	1.517394	2.974548
0.027110	29.550138	0.336872	0.065010	1.477507	1.516818	2.973417
0.027170	29.507322	0.336383	0.064916	1.475366	1.514620	2.969109
0.027590	29.337116	0.334443	0.064542	1.466856	1.505883	2.951983
0.028070	29.328828	0.334349	0.064523	1.466441	1.505458	2.951149
0.028440	29.227804	0.333197	0.064301	1.461390	1.500272	2.940983
0.028550	29.214541	0.333046	0.064272	1.460727	1.499591	2.939649
0.029030	29.122231	0.331993	0.064069	1.456112	1.494853	2.930360
0.029700	28.946823	0.329994	0.063683	1.447341	1.485849	2.912710
0.030960	28.619719	0.326265	0.062963	1.430986	1.469059	2.879796
0.032230	28.363527	0.323344	0.062400	1.418176	1.455908	2.854017
0.033170	28.230638	0.321829	0.062107	1.411532	1.449087	2.840646
0.033490	28.214313	0.321643	0.062071	1.410716	1.448249	2.839003
0.034750	28.040823	0.319665	0.061690	1.402041	1.439344	2.821546
0.036020	27.975786	0.318924	0.061547	1.398789	1.436006	2.815002
0.036330	27.939175	0.318507	0.061466	1.396959	1.434126	2.811318
0.037280	27.822820	0.317180	0.061210	1.391141	1.428154	2.799610
0.038540	27.689378	0.315659	0.060917	1.384469	1.421304	2.786183
0.039490	27.612941	0.314788	0.060748	1.380647	1.417381	2.778491
0.039800	27.576466	0.314372	0.060668	1.378823	1.415508	2.774821
0.041070	27.441883	0.312837	0.060372	1.372094	1.408600	2.761279
0.042330	27.344473	0.311727	0.060158	1.367224	1.403600	2.751477

[a] The scattering vector magnitude, Q.

[b] The SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega_{SANS\ STD}$ rescaled for SAXS, the standard uncertainty components (u), the combined standard uncertainties (u_c) and the expanded uncertainties (U) in the SAXS differential scattering cross-section per unit sample volume (95 % confidence interval). All u, u_C and U uncertainties in calibration are also indicated as percentage fractions of the rescaled $d\Sigma/d\Omega_{SANS\ STD}$ value. The k value of 1.9603 was determined by statistical methods. All calculations made according to the JCGM Guide [13].

[c] Type A uncertainties evaluated by statistical methods.

[d] Uncertainties for coupon variability are for a single coupon.

[e] Uncertainties based on eight measurements with one SANS set-up, hence seven degrees of freedom.

[f] Type B uncertainties evaluated by other means.

[g] Uncertainties based on literature estimate and long instrument history.

(continued on next page)

Table 2. Reference values for $d\Sigma/d\Omega_{STD}$ with Measurement Uncertainties for SRM 3600 (Continued)

Q[a] ($Å^{-1}$)	$d\Sigma/d\Omega_{STD}$[b] rescaled for SAXS by factor of 6.409 (cm^{-1} sr^{-1})	Coupon[c,d] Variability (N = 56) (u = 1.14 %) (cm^{-1} sr^{-1})	Given Set-up[c,e] Measurement Replicability (N = 8) (0.22 %) (cm^{-1} sr^{-1})	Different SANS[f,g] Set-ups Replicability (N = large) (5.00 %) (cm^{-1} sr^{-1})	u_C (5.13 %) (cm^{-1} sr^{-1})	U (10.06 %) (cm^{-1} sr^{-1})
0.042640	27.324635	0.311501	0.060114	1.366232	1.402582	2.749481
0.043590	27.197318	0.310049	0.059834	1.359866	1.396047	2.736670
0.044850	27.080566	0.308718	0.059577	1.354028	1.390054	2.724922
0.045800	26.998297	0.307781	0.059396	1.349915	1.385831	2.716644
0.046120	26.986052	0.307641	0.059369	1.349303	1.385202	2.715412
0.047380	26.862557	0.306233	0.059098	1.343128	1.378863	2.702985
0.048640	26.808082	0.305612	0.058978	1.340404	1.376067	2.697504
0.048960	26.796780	0.305483	0.058953	1.339839	1.375487	2.696367
0.049900	26.737215	0.304804	0.058822	1.336861	1.372429	2.690373
0.051160	26.690831	0.304275	0.058720	1.334542	1.370048	2.685706
0.052110	26.652975	0.303844	0.058637	1.332649	1.368105	2.681897
0.052430	26.630031	0.303582	0.058586	1.331502	1.366928	2.679588
0.053690	26.585299	0.303072	0.058488	1.329265	1.364631	2.675087
0.054950	26.493033	0.302021	0.058285	1.324652	1.359895	2.665803
0.055260	26.492674	0.302016	0.058284	1.324634	1.359877	2.665767
0.056210	26.451887	0.301552	0.058194	1.322594	1.357783	2.661663
0.057470	26.417365	0.301158	0.058118	1.320868	1.356011	2.658189
0.058420	26.398150	0.300939	0.058076	1.319908	1.355025	2.656256
0.058730	26.392520	0.300875	0.058064	1.319626	1.354736	2.655689
0.059990	26.316155	0.300004	0.057896	1.315808	1.350816	2.648005
0.061250	26.247067	0.299217	0.057744	1.312353	1.347270	2.641053
0.061570	26.235012	0.299079	0.057717	1.311751	1.346651	2.639840
0.062510	26.249483	0.299244	0.057749	1.312474	1.347394	2.641296
0.063770	26.249639	0.299246	0.057749	1.312482	1.347402	2.641312
0.064720	26.242958	0.299170	0.057735	1.312148	1.347059	2.640640
0.065030	26.235626	0.299086	0.057718	1.311781	1.346683	2.639902
0.066290	26.239541	0.299131	0.057727	1.311977	1.346884	2.640296
0.067550	26.199259	0.298672	0.057638	1.309963	1.344816	2.636243
0.067870	26.151000	0.298121	0.057532	1.307550	1.342339	2.631387
0.068810	26.108488	0.297637	0.057439	1.305424	1.340157	2.627109
0.070070	26.029905	0.296741	0.057266	1.301495	1.336123	2.619202
0.071020	25.973353	0.296096	0.057141	1.298668	1.333220	2.613511
0.071330	25.945622	0.295780	0.057080	1.297281	1.331797	2.610721
0.072590	25.924336	0.295537	0.057034	1.296217	1.330704	2.608579
0.073850	25.896775	0.295223	0.056973	1.294839	1.329289	2.605806
0.074170	25.917482	0.295459	0.057018	1.295874	1.330352	2.607889
0.075110	25.897155	0.295228	0.056974	1.294858	1.329309	2.605844
0.076370	25.871780	0.294938	0.056918	1.293589	1.328006	2.603291
0.077310	25.848582	0.294674	0.056867	1.292429	1.326816	2.600956
0.077630	25.820259	0.294351	0.056805	1.291013	1.325362	2.598107
0.078890	25.744981	0.293493	0.056639	1.287249	1.321498	2.590532
0.080150	25.692106	0.292890	0.056523	1.284605	1.318784	2.585211
0.080460	25.654044	0.292456	0.056439	1.282702	1.316830	2.581382
0.081400	25.629631	0.292178	0.056385	1.281482	1.315577	2.578925
0.082660	25.580526	0.291618	0.056277	1.279026	1.313056	2.573984
0.083600	25.532093	0.291066	0.056171	1.276605	1.310570	2.569110
0.083920	25.524943	0.290984	0.056155	1.276247	1.310203	2.568391
0.085180	25.438540	0.289999	0.055965	1.271927	1.305768	2.559697
0.086430	25.319783	0.288646	0.055704	1.265989	1.299672	2.547747
0.086750	25.301280	0.288435	0.055663	1.265064	1.298722	2.545885
0.087690	25.218590	0.287492	0.055481	1.260929	1.294478	2.537565

[a] The scattering vector magnitude, Q.

[b] The SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega_{SANS\ STD}$ rescaled for SAXS, the standard uncertainty components (u), the combined standard uncertainties (u_c) and the expanded uncertainties (U) in the SAXS differential scattering cross-section per unit sample volume (95 % confidence interval). All u, u_C and U uncertainties in calibration are also indicated as percentage fractions of the rescaled $d\Sigma/d\Omega_{SANS\ STD}$ value. The k value of 1.9603 was determined by statistical methods. All calculations made according to the JCGM Guide [13].

[c] Type A uncertainties evaluated by statistical methods.

[d] Uncertainties for coupon variability are for a single coupon.

[e] Uncertainties based on eight measurements with one SANS set-up, hence seven degrees of freedom.

[f] Type B uncertainties evaluated by other means.

[g] Uncertainties based on literature estimate and long instrument history.

(continued on next page)

Table 2. Reference values for $d\Sigma/d\Omega_{STD}$ with Measurement Uncertainties for SRM 3600 (Continued)

Q[a] (Å^{-1})	$d\Sigma/d\Omega_{STD}$[b] rescaled for SAXS by factor of 6.409 (cm^{-1} sr^{-1})	Coupon[c,d] Variability (N = 56) (u = 1.14 %) (cm^{-1} sr^{-1})	Given Set-up[c,e] Measurement Replicability (N = 8) (0.22 %) (cm^{-1} sr^{-1})	Different SANS[f,g] Set-ups Replicability (N = large) (5.00 %) (cm^{-1} sr^{-1})	u_C (5.13 %) (cm^{-1} sr^{-1})	U (10.06 %) (cm^{-1} sr^{-1})
0.088950	25.106052	0.286209	0.055233	1.255303	1.288701	2.526241
0.089890	24.987916	0.284862	0.054973	1.249396	1.282637	2.514354
0.090210	24.973175	0.284694	0.054941	1.248659	1.281881	2.512871
0.091460	24.894596	0.283798	0.054768	1.244730	1.277847	2.504964
0.092720	24.821476	0.282965	0.054607	1.241074	1.274094	2.497606
0.093030	24.763399	0.282303	0.054479	1.238170	1.271113	2.491762
0.093970	24.690993	0.281477	0.054320	1.234550	1.267396	2.484477
0.095230	24.610680	0.280562	0.054143	1.230534	1.263274	2.476395
0.096170	24.521740	0.279548	0.053948	1.226087	1.258708	2.467446
0.099310	24.214046	0.276040	0.053271	1.210702	1.242914	2.436485
0.102400	23.944226	0.272964	0.052677	1.197211	1.229064	2.409335
0.105600	23.633494	0.269422	0.051994	1.181675	1.213114	2.378068
0.108700	23.274262	0.265327	0.051203	1.163713	1.194675	2.341921
0.111800	22.857259	0.260573	0.050286	1.142863	1.173270	2.299961
0.115000	22.412435	0.255502	0.049307	1.120622	1.150437	2.255202
0.118100	21.946720	0.250193	0.048283	1.097336	1.126532	2.208340
0.121200	21.440636	0.244423	0.047169	1.072032	1.100554	2.157417
0.124400	20.898846	0.238247	0.045977	1.044942	1.072744	2.102900
0.127500	20.376032	0.232287	0.044827	1.018802	1.045908	2.050293
0.130600	19.849971	0.226290	0.043670	0.992499	1.018905	1.997360
0.133700	19.299975	0.220020	0.042460	0.964999	0.990674	1.942017
0.136800	18.734065	0.213568	0.041215	0.936703	0.961625	1.885074
0.140000	18.154859	0.206965	0.039941	0.907743	0.931894	1.826793
0.143100	17.606044	0.200709	0.038733	0.880302	0.903724	1.771569
0.146200	17.073375	0.194636	0.037561	0.853669	0.876382	1.717971
0.149300	16.538783	0.188542	0.036385	0.826939	0.848941	1.664179
0.152400	16.014714	0.182568	0.035232	0.800736	0.822040	1.611445
0.155500	15.514954	0.176870	0.034133	0.775748	0.796387	1.561158
0.158600	15.003181	0.171036	0.033007	0.750159	0.770118	1.509662
0.161700	14.490267	0.165189	0.031879	0.724513	0.743790	1.458051
0.164800	13.980814	0.159381	0.030758	0.699041	0.717639	1.406789
0.167900	13.482934	0.153705	0.029662	0.674147	0.692083	1.356690
0.171000	12.984537	0.148024	0.028566	0.649227	0.666500	1.306540
0.174100	12.493814	0.142429	0.027486	0.624691	0.641311	1.257162
0.177200	12.035300	0.137202	0.026478	0.601765	0.617776	1.211026
0.180300	11.579527	0.132007	0.025475	0.578976	0.594381	1.165164
0.183400	11.127615	0.126855	0.024481	0.556381	0.571184	1.119692
0.186400	10.717036	0.122174	0.023577	0.535852	0.550109	1.078378
0.189500	10.309091	0.117524	0.022680	0.515455	0.529169	1.037330
0.192600	9.922081	0.113112	0.021829	0.496104	0.509303	0.998388
0.195700	9.544609	0.108809	0.020998	0.477230	0.489928	0.960405
0.198800	9.170826	0.104547	0.020176	0.458541	0.470741	0.922794
0.201800	8.825891	0.100615	0.019417	0.441295	0.453036	0.888086
0.204900	8.462488	0.096472	0.018617	0.423124	0.434382	0.851519
0.207900	8.118883	0.092555	0.017862	0.405944	0.416745	0.816945
0.211000	7.784807	0.088747	0.017127	0.389240	0.399596	0.783329
0.214100	7.472734	0.085189	0.016440	0.373637	0.383578	0.751927
0.217100	7.184433	0.081903	0.015806	0.359222	0.368779	0.722918

[a] The scattering vector magnitude, Q.

[b] The SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega_{SANS\ STD}$ rescaled for SAXS, the standard uncertainty components (u), the combined standard uncertainties (u_c) and the expanded uncertainties (U) in the SAXS differential scattering cross-section per unit sample volume (95 % confidence interval). All u, u_C and U uncertainties in calibration are also indicated as percentage fractions of the rescaled $d\Sigma/d\Omega_{SANS\ STD}$ value. The k value of 1.9603 was determined by statistical methods. All calculations made according to the JCGM Guide [13].

[c] Type A uncertainties evaluated by statistical methods.

[d] Uncertainties for coupon variability are for a single coupon.

[e] Uncertainties based on eight measurements with one SANS set-up, hence seven degrees of freedom.

[f] Type B uncertainties evaluated by other means.

[g] Uncertainties based on literature estimate and long instrument history.

Table 2. Reference values for $d\Sigma/d\Omega_{STD}$ with Measurement Uncertainties for SRM 3600 (Continued)

Q[a] (Å^{-1})	$d\Sigma/d\Omega_{STD}$[b] rescaled for SAXS by factor of 6.409 (cm^{-1} sr^{-1})	Coupon[c,d] Variability (N = 56) (u = 1.14 %) (cm^{-1} sr^{-1})	Given Set-up[c,e] Measurement Replicability (N = 8) (0.22 %) (cm^{-1} sr^{-1})	Different SANS[f,g] Set-ups Replicability (N = large) (5.00 %) (cm^{-1} sr^{-1})	u_C (5.13 %) (cm^{-1} sr^{-1})	U (10.06 %) (cm^{-1} sr^{-1})
0.220200	6.894059	0.078592	0.015167	0.344703	0.353874	0.693700
0.223200	6.617605	0.075441	0.014559	0.330880	0.339684	0.665882
0.226300	6.345461	0.072338	0.013960	0.317273	0.325714	0.638498
0.229300	6.092117	0.069450	0.013403	0.304606	0.312710	0.613006
0.232300	5.848382	0.066672	0.012866	0.292419	0.300199	0.588481
0.235400	5.612116	0.063978	0.012347	0.280606	0.288072	0.564707
0.238400	5.400463	0.061565	0.011881	0.270023	0.277207	0.543410
0.241400	5.193931	0.059211	0.011427	0.259697	0.266606	0.522628
0.244500	4.992766	0.056918	0.010984	0.249638	0.256280	0.502386
0.247500	4.800052	0.054721	0.010560	0.240003	0.246388	0.482995

[a] The scattering vector magnitude, Q.
[b] The SAXS differential scattering cross-section per unit sample volume, $d\Sigma/d\Omega_{SANS\ STD}$ rescaled for SAXS, the standard uncertainty components (u), the combined standard uncertainties (u_C) and the expanded uncertainties (U) in the SAXS differential scattering cross-section per unit sample volume (95 % confidence interval). All u, u_C and U uncertainties in calibration are also indicated as percentage fractions of the rescaled $d\Sigma/d\Omega_{SANS\ STD}$ value. The k value of 1.9603 was determined by statistical methods. All calculations made according to the JCGM Guide [13].
[c] Type A uncertainties evaluated by statistical methods.
[d] Uncertainties for coupon variability are for a single coupon.
[e] Uncertainties based on eight measurements with one SANS set-up, hence seven degrees of freedom.
[f] Type B uncertainties evaluated by other means.
[g] Uncertainties based on literature estimate and long instrument history.

The SRM 3600 validation calibration curve for rescaled $d\Sigma/d\Omega_{SANS\ STD}$ *versus* Q is presented in Figure 3, together with the computed 95 % confidence minimum and maximum envelope, based on the values presented in Table 2. The certified calibration curve with computed 95 % confidence minimum and maximum envelope is also recalled from Table 1 and Figure 2 for comparison.

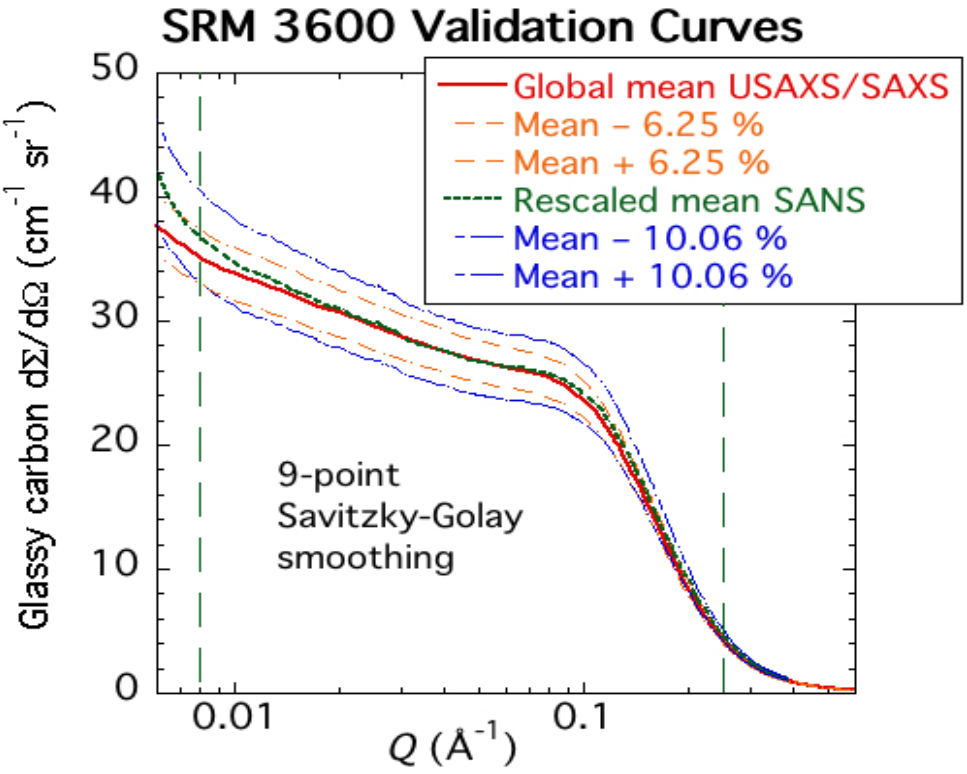

Figure 3. Comparison of smoothed rescaled SANS and smoothed calibrated USAXS/SAXS intensity data *versus* Q. The uncertainty bands represent the minimum and maximum limits for overall 95 % confidence expanded uncertainties on the mean results. The vertical dashed lines indicate the certification range in Q.

REFERENCES

[1] Guinier, A.; Fournet, G.; *Small-angle Scattering of X-rays*; John Wiley & Sons, New York, NY (1955); see also Glatter, O.; Kratky, O.; *Small-angle X-ray Scattering*; Academic Press, London (1982).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at: http://www.nist.gov/srm/publications.cfm (accessed June 2016).

[3] *X-ray data booklet*; Thompson, A.; Ed.; Lawrence Berkeley National Laboratory: Berkeley, CA (2009); see also Chantler, C.T.; Olsen, K.; Dragoset, R.A.; Chang, J.; Kishore, R.A.; Kotochigova, S.A.; Zucker, D.S.; *X-ray Form Factor, Attenuation, and Scattering Tables (version 2.1)*; NIST, Gaithersburg, MD (2005); available at http://physics.nist.gov/ffast (accessed June 2016).

[4] Sears, V.F.; *Neutron Scattering Lengths and Cross Sections*; Neutron News, Vol. 3(3), pp. 26–37 (1992); NIST, Gaithersburg, MD (2013); available at http://www.ncnr.nist.gov/resources/n-lengths/ (accessed June 2016).

[5] Zhang, F.; Ilavsky, J.; Long, G.G.; Quintana, J.P.G.; Allen, A.J.; Jemian, P.R.; *Glassy Carbon as an Absolute Intensity Calibration Standard for Small-angle Scattering*; Metall., Mater. Trans. A, Vol. 41, pp. 2010–2015 (2010); see also Dreiss, C.A.; Jack, K.S.; Parker, A.P.; *On the Absolute Calibration of Bench-top Small-angle X-ray Scattering Instruments: A Comparison of Different Standard Methods*; J. Appl. Cryst., Vol. 39, pp. 32–38 (2006).

[6] *Glassy Carbon Plate*; MSDS, Alfar Aesar, Johnson Matthey Catalog Co., Inc.: Ward Hill, MA (2009); see also Jenkins, G.M.; Kawamura, K.; *Structure of Glassy Carbon*; Nature, Vol. 231, pp. 175–176 (1971).

[7] Ilavsky, J.; Jemian, P.R.; Allen, A.J.; Zhang, F.; Levine, L.E.; Long, G.G.; *Ultra-small-angle X-ray Scattering at the Advanced Photon Source*; J. Appl. Cryst., Vol. 42, pp. 469–479 (2009); see also Ilavsky, J.; *USAXS Documentation*; Argonne National Laboratory: Argonne, IL (2014); available at http://usaxs.xray.aps.anl.gov/docs/index.html (accessed June 2016).

[8] Ho, D.L.; Wang, C.; Lin, E.K.; Jones, R.L.; Wu, W.L.; *A Laboratory Scale Critical-dimension Small-angle X-ray Scattering Instrument*; AIP Conf. Proc., Vol. 931, pp. 382–386 (2007).

[9] Glinka, C.J.; Barker, J.G.; Hammouda, B.; Krueger, S.; Moyer, J.J.; Orts, W.J.; *The 30 m Small-angle Neutron Scattering Instruments at the National Institute of Standards and Technology*; J. Appl. Cryst., Vol. 31, pp. 430–445 (1998); see also Hammouda, B.; *SANS Manuals & Data Reduction*; NIST, Gaithersburg, MD (2013); available at http://www.ncnr.nist.gov/programs/sans/ (accessed June 2016).

[10] Long, G.G.; Jemian, P.R.; Weertman, J.R.; Black, D.R.; Burdette, H.E.; Spal, R.; *High-resolution Small-angle X-ray Scattering Camera for Anomalous Scattering*; J. Appl. Cryst., Vol. 24, pp. 30–37 (1991).

[11] Ilavsky, J.; Zhang, F.; Allen, A.J.; Levine, L.E.; Jemian, P.R.; Long, G.G.; *Ultra-small-angle X-ray Scattering Instrument at the Advanced Photon Source: History, Recent Development, and Current Status*; Metall. Mater. Trans. A, Vol. 44, pp. 68–76 (2013).

[12] Dejus, R.J., Lai, B., Moog, E.R.; Gluskin, E.; *Undulator A Characteristics and Specifications: Enhanced Capabilities*; Report ANL/APS/TB-17; Argonne National Laboratory: Lemont, IL (1994).

[13] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed June 2016); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed June 2016).

[14] Ilavsky, J.; Jemian, P.R.; *Irena: Tool Suite for Modeling and Analysis of Small-angle Scattering*; J. Appl. Cryst., Vol. 42, pp. 347–353 (2009).

[15] Lake, J.A.; *An Iterative Method of Slit-correcting Small Angle X-ray Data*; Acta Cryst., Vol. 23, pp. 191–194 (1967).

[16] Ilavsky, J.; *Nika: Software for Two-dimensional Data Reduction*; J. Appl. Cryst., Vol. 45, pp. 324–328 (2012).

[17] Savitzky, A.; Golay, M.J.E.; *Smoothing and Differentiation of Data by Simplified Least Squares Procedure*; Anal. Chem., Vol. 36, pp. 1627–1639 (1964).

[18] Kline, S.R.; *Reduction and Analysis of SANS and USANS Data Using IGOR Pro*; J. Appl. Cryst., Vol. 39, pp. 895–900 (2006).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.