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The Absolute Calibration of a Small-Angle Scattering Instrument with a Laboratory X-ray Source

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Abstract. Absolute calibration of small-angle scattering data (in units of differential cross-section per unit sample volume per unit solid angle) is necessary for the determination of molecular weights, the number density of particles, the scattering-length densities of phases in multiphased systems, volume fraction, the specific surface area of the scatterers and to restrict the parameters of a given model to the set which reproduces the observed intensity. It is also a useful means for the detection of artefacts in SAS experiments. Absolute intensities from the same sample also allows intercalibration among different instruments. This work details the absolute calibration procedure of a small-angle X-ray scattering instrument, the Rigaku S-Max3000. Absolute calibration is achieved by using two standards: homogeneous and stable glassy carbon and water. The scattering intensity of glassy carbon is calibrated by comparison with the absolute-calibration measurements taken on the USAXS instrument located at the 32ID beamline of the Advanced Photon Source in Argonne National Laboratory. This instrument has primary calibration capability. Scattering from water is angle-independent and only depends on the physical property of isothermal compressibility. Absolute calibrations using two standards were compared. The agreement of scale factors obtained using two standards suggests that precalibrated glassy carbon can serve as a convenient standard for all types of materials under study.

1. Introduction

Small-angle X-ray scattering (SAXS) is a well established technique that allows us to obtain structural information about inhomogeneities in materials with a characteristic length from 1nm to 100nm. SAXS is thus an appropriate technique to characterize a very large variety of samples, ranging from colloids of all types, metals, cements, nanoparticles, oils, polymers, plastics, proteins, surfactants, foods and pharmaceuticals. Absolute calibration relates the measured (arbitrary) intensity I to the differential scattering cross section per unit volume, $\partial\Sigma/\partial\Omega$, in units of cm^{-1} . While small-angle neutron scattering data is commonly converted to an absolute scale taking advantage of strong incoherent scattering of water [1-3], a significant fraction of X-ray data is still published in arbitrary units. Particularly, absolute calibration is less of a routine procedure for SAXS instruments with laboratory X-ray sources. Several reasons may exist for this lack of calibration. The use of suitable calibration techniques is by no mean universal and straightforward. The first method, the so-called direct method or premier method, attenuates the beam mechanically by using a rotating disc [4] or a moving slit [5]. This method is not well suited for use with position-sensitive detectors [6]. Detailed analysis of the attenuator transmission measurements is often nontrivial as well [7]. The second method compares the scattering intensity from the sample with that from a calibration standard sample

of theoretically known scattering cross-section, such as water, or pre-calibrated standards. Given the lower flux, when compared to synchrotrons, laboratory SAXS measurements of water can be time consuming. Obtaining stable pre-calibrated secondary standards is also difficult. Some bench-top SAXS cameras may also face the practical complexity and difficulty in routine processes such as removing and realigning the beamstop [8] and data smearing issues. The Rigaku S-Max3000 SAXS camera uses pinhole collimation and a beamstop with an embedded photodiode to record direct and transmitted beam intensities. The problems of data smearing and removing and realigning the beamstop are thus avoided. With improved flux using a MicroMax 002+ X-ray source and focusing confocal optics, the Rigaku S-Max3000 enhances the routine use of water as a premier standard.

The absolute scale is not necessary for the measurement of the interference functions [9]. For example, determination of radius of gyration, phase transitions and the derivation of the correlation function can be done using measured arbitrary intensity. However, placing the data on an absolute scale is necessary for the determination of molecular weights [10], the number density of particles [11], the scattering-length densities of phases in multiphase systems [12], volume fraction, the specific surface area of the scatterers and to restrict the parameters of a given model to the set which reproduces the observed intensity [13,14,1,2]. An absolute scale is also useful for the detection of artefacts in small angle scattering (SAS) experiments [9]. The absolute intensity is independent of sample thickness, SAXS camera geometry and measurement time. Therefore, absolute intensities from the same sample also allow inter-calibration among different instruments [9].

Due to the aforementioned difficulties of the direct method, the use of calibration standards is more common for absolute calibration in SAXS. Water and pure liquids are typically used as primary standards [15-17] since their differential scattering cross sections depend only on the temperature and isothermal compressibility and therefore can be calculated. However these standards are not suitable for a laboratory SAXS camera with low-flux sources because the scattering is very weak and measurement time is long. In addition, these standards are more suitable for biosciences; however, their X-ray scattering cross sections are too low to serve as a standard in the areas of materials science, polymer science, ceramics, chemistry, etc. Alternatively, monodisperse colloidal suspensions have been used as primary intensity calibration standards [18], for which the differential cross section can be obtained from Porod's law as the interfacial region is well defined. However, the metastable nature of these colloidal systems leaves their long-term usage in question. As secondary standards, vitreous or glassy carbon [19], aluminium-silver alloys [20], silica suspensions [21, 18], Lupolen [22-24] and calibrated foils have been used [25-27].

Among the many intensity calibration standard materials, glassy carbon is a strong scatterer and has a good temperature tolerance [19]. It has been informally used as a calibration standard. As with other secondary standards, its differential cross section needs to be measured. Traditionally, this measurement has been done either at a neutron facility or through calibration, using X-ray data of primary standards. Now there is the possibility to measure it directly at a Synchrotron. The versatile ultra-small-angle X-ray scattering (USAXS) instrument at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL) offers quantitative primary calibration of X-ray scattering cross sections without the need for beam attenuation or primary standards of any kind [28]. Designed after the Bonse-Hart double-crystal configuration [29-31], the APS USAXS instrument takes advantage of a silicon photodiode detector that is linear over 9 decades of scattering intensity. This broad linearity enables the instrument to scan through the full incident beam without loss of linearity in measurement of the beam intensity. This instrument is not limited to ultra small angles. A wide scattering vector range from 0.0001 to 1 \AA^{-1} can be covered.

This work details the absolute calibration procedure of a small-angle X-ray scattering instrument, the Rigaku S-Max3000. One should be note that this procedure is applicable to any type of SAXS camera

with a laboratory source. We first give a short review of the principle and theory for absolute calibration, followed by an investigation of two standards: water and glassy carbon. The intensity of glassy carbon in absolute scale is obtained by measuring the sample on the USAXS instrument located at the 32ID beamline of APS at Argonne National Laboratory. We compare scale factors obtained using these two standards. We discuss the suitability of two standards and suggest glassy carbon as a general standard material for absolute intensity calibration.

2. Theory

Detailed theory and relevant expressions for absolute calibrations can be found elsewhere [32, 33, 34]. Here we restate the formulas used for extracting the calibration scale factor. The intensity scattered from a sample can be described as

$$I_s(q) = I_0(\lambda)A\Delta\Omega\eta(\lambda)T_s(\lambda)d_s\left(\frac{\partial\Sigma}{\partial\Omega}\right)_s(q) + BG_s, \quad (1)$$

where $q = 4\pi\sin(\theta)/\lambda$ is the momentum transfer; θ is half of the scattering angle; λ is the wavelength; $I_s(q)$ is measured intensity of the sample; $I_0(\lambda)$ is the incident flux (in units of photons/s⁻¹/cm⁻²); $\Delta\Omega$ is the solid angle element defined by the size of a detector pixel; A is the area illuminated by the beam; η is the detector efficiency; $T_s(\lambda)$ is the transmission of the sample; d_s is the thickness of the sample; BG_s is the scattering background; $\left(\frac{\partial\Sigma}{\partial\Omega}\right)_s$ is the differential scattering cross section(cm⁻¹) of the sample and is the quantity obtained from the absolute calibration of the measured intensity.

The absolute intensity of the sample can be obtained by using standards with known differential scattering cross section $\left(\frac{\partial\Sigma}{\partial\Omega}\right)_{st}(q)$ and thickness d_{st} :

$$\left(\frac{\partial\Sigma}{\partial\Omega}\right)_s(q) = \left(\frac{\partial\Sigma}{\partial\Omega}\right)_{st}(q) \frac{(I_s(q) - BG_s)}{d_s T_{s+cell}} \bigg/ \frac{(I_{st}(q) - BG_{st})}{d_{st} T_{st+cell}}, \quad (2)$$

where $T_{s+cell}(\lambda)$ and $T_{st+cell}(\lambda)$ are the transmissions of the sample and standard in the containers (cells), respectively. $I_{st}(q)$ is the measured intensity of the standard. Therefore, the intensity of the sample in absolute scale can be obtained by multiplying the measured intensity by the scale factor. Please note that the measured intensity of the sample should first be normalized by transmission and thickness of the sample.

$$ScaleFactor = \left(\frac{\partial\Sigma}{\partial\Omega}\right)_{st} \bigg/ \frac{(I_{st}(q) - BG_{st})}{d_{st} T_{st+cell}} \quad (3)$$

3. Experimental Methods

3.1. Materials

Distilled water and glassy carbon were studied for the calibration of SAXS experiments. We used commercially available glassy carbon plate from Alpha Aesar. We chose type 2 (stock number: #38021), which has stronger scattering than type 1 (stock number: #38024). The glassy carbon plates have dimensions of 50 mm × 50 mm × 1mm and ≈ 3.55 g/ 50 x 50 mm. The glassy carbon was cut to a smaller piece for X-ray measurements. Please note that scattering from glassy carbon may vary from plate to plate even if the plates have the same stock number. Therefore, the absolute intensity data for a particular piece of glassy carbon may not be suitable for calibration of another glassy carbon plate.

For accuracy, the exact same sample should be used for the calibration measurements at the synchrotron and home labs.

3.2. Laboratory-based small angle X-ray scattering measurements

The measurements were performed on a Rigaku three-pinhole SAXS camera S-Max3000 (Figure 1). This camera is a dedicated SAXS instrument suitable for measuring both isotropic and anisotropic systems. The instrument uses Cu K α radiation ($\lambda = 1.54 \text{ \AA}$). The combination of sealed tube microfocus source MicroMax 002+ and focusing confocal optics provides a small beam spot and high flux on the sample. The high brilliance microfocus source MicroMax 002+ requires low maintenance and can operate at low power consumption, typically at a current of 0.88 mA and a potential difference of 45 kV. The scattering data were collected by a fully integrated 2D multiwire proportional counting gas-filled detector that has low background. The diameter of the detector was 120 mm. A photodiode embedded in the beamstop was used allowing continuous intensity monitoring and direct measurement of the transmission. The two sample chambers provide flexibility in covering a large q range without the need to move the detector and subsequent alignment. The water sample was placed in a sealed capillary with a diameter of 0.15 cm. The glassy carbon was directly mounted into the beam path without a container. The measurements were performed in vacuum and at room temperature. Samples were placed in two sample chambers and measured at two sample-detector distances (1.5 m and 0.44 m) to cover total q range from 0.0067 \AA^{-1} to 0.55 \AA^{-1} . The empty capillary, empty beam, dark current and transmissions of the samples were also measured for data correction. The data were reduced and analyzed using Rigaku's SAXSGUI and Nanosolver software packages.

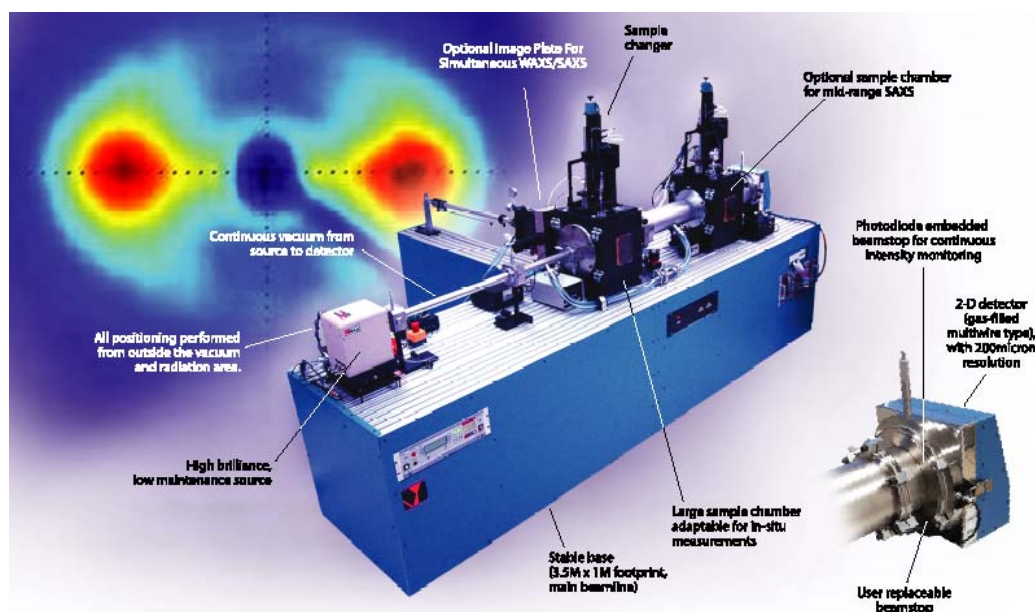


Figure1. Rigaku three-pinhole SAXS camera S-Max3000

3.3. Ultra Small angle X-ray scattering measurements at synchrotron

The absolute intensity of glassy carbon was measured on a USAXS instrument at beamline 32-ID of the APS, ANL. This USAXS instrument offers quantitative primary calibration of X-ray scattering cross sections, a scattering vector range from 0.0001 to 1 \AA^{-1} , and intensity range of up to 9 decades [28]. The data were collected using monochromatic 12 keV radiation, beam size 2 mm x 1 mm (width x height), with exposure of 1 second at each point, resulting in total data collection time about 10 minutes for the measurement. USAXS data were reduced, calibrated, and corrected for all instrumental effects (such as slit smearing) using the Indra and Irena software [35].

4. Results and discussions

Figure 2 shows the scattering curves (with error bars) of water in the capillary, empty capillary and pure water after subtraction of these two. Data were collected using the Rigaku SAXS camera S-Max3000. Each curve is a combination of two data sets collected in two sample chambers e.g. at two different sample-detector distances. Water is a very weak scatterer hence the dark current is significant and can't be neglected. The raw data were corrected for dark current and transmission following standard procedures during the data reduction using SAXSGUI software, but not yet normalized with the thickness of the sample.

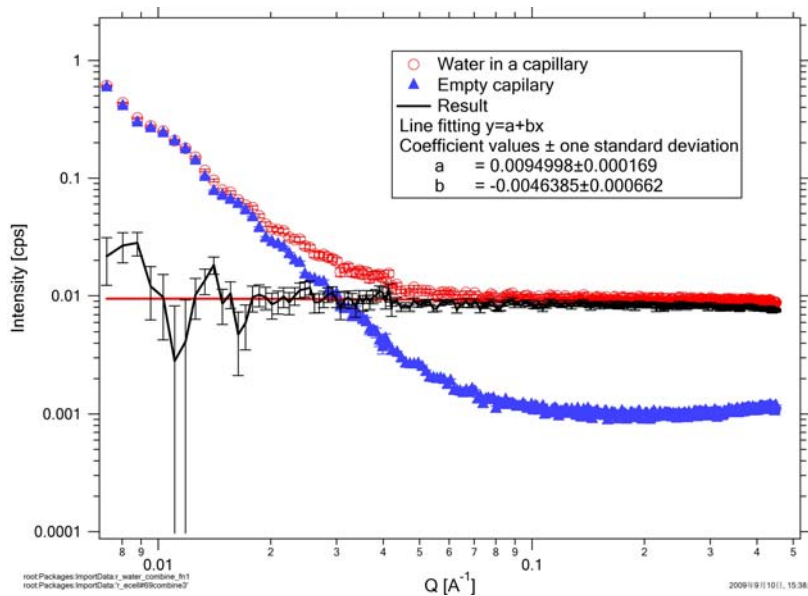


Figure 2. Scattering profiles: scattering from water in capillary are red circles; empty capillary- blue triangles; pure water-dark; horizontal linear fitting in solid red line.

The data exhibits a straight line after $q = 0.02 \text{ \AA}^{-1}$. The linear fitting extrapolates to intensity at zero angle of $I_{\text{H}_2\text{O}}(0) = 9.5 \times 10^{-3} \text{ s}^{-1} \pm 0.2 \times 10^{-3} \text{ s}^{-1}$. The scattering of water only depends on temperature and the physical property of the isothermal compressibility according to

$$\left(\frac{\partial \Sigma}{\partial \Omega}\right)_{\text{H}_2\text{O}}(0) = \rho^2 K T \chi_T, \quad (4)$$

where ρ is the scattering-length density and χ_T is the isothermal compressibility. The differential scattering cross section is $1.65 \times 10^{-2} (\text{cm}^{-1})$ at 293 K and 10^5 Pa with the isothermal compressibility of $4.591 \times 10^{-10} \text{ Pa}^{-1}$ [36].

As we mentioned above, the data in Figure 2 was corrected with the transmission factor, but not the thickness of the sample. The diameter of the capillary is 0.15 cm. Therefore, the scale factor should be:

$$\text{SF} = \frac{\left(\frac{\partial \Sigma}{\partial \Omega}\right)_{\text{H}_2\text{O}}(0)}{I_{\text{H}_2\text{O}}(0)} \times d_{\text{H}_2\text{O}} = 1.65 \times 10^{-2} \frac{0.15}{9.5 \times 10^{-3}} = 0.261$$

To scale to absolute intensity, the intensity (after correction of background, transmission and thickness) measured on the lab-based SAXS should be multiplied by this scale factor. Taking into account the

error of $0.2 \times 10^{-3} \text{ s}^{-1}$ for $I_{\text{H}_2\text{O}}(0)$, the error for the scale factor from water is estimated to be 0.003(0.3%).

Please note in the calculation above we used the thickness of capillary, provided by the manufacturer, as 0.15 cm and the error in the thickness was not taken into account. Since the X-ray beam spot is not a point, the actual beam path length through the capillary is not the diameter of the capillary and it is not trivial to define. It is better to use a flat quartz cell for the measurements. More accurate estimation of the thickness (averaged beam path length) of water in the capillary can be done by measuring the transmissions of water in the capillary and empty capillary and using the known mass attenuation coefficient of water. The transmission of a sample can be described as

$$T = I_t / I_0 = e^{-(\mu/\rho)X} = e^{-\sum_i w_i (\mu/\rho)_i X},$$

where I_0 and I_t are the intensities of incident beam and transmitted beam, respectively; x is the mass thickness obtained by multiplying the thickness t by the density ρ i.e., $x = \rho t$. For mixtures and compounds, the values of the mass attenuation coefficient, μ/ρ , can be obtained by simple additivity: $\sum_i w_i (\mu/\rho)_i$, where w_i is the fraction by weight of the i^{th} atomic constituent. Therefore, the transmission of water in a capillary can be described as

$$T_{\text{water+cell}} = T_{\text{cell}} \times e^{-(\mu/\rho)_{\text{water}} \rho_{\text{water}} t}$$

The mass attenuation coefficient for water, $(\mu/\rho)_{\text{water}}$, at a photon energy of 8 Kev is 10.37 (cm^2/g) according to <http://physics.nist.gov/PhysRefData/XrayMassCoef/ComTab/water.html>. For the particular quartz capillary used for our measurements, $T_{\text{water+cell}} = 0.1458$ and $T_{\text{cell}} = 0.6098$. The density of water is 1 g/cm^3 . Then the thickness of water can be calculated as 0.1384 cm. Comparing to the nominal diameter of the capillary of 0.15 cm, the error is $\sim 7.7\%$. Therefore the error in the water thickness can dominate the error in the scale factors. It needs to be carefully defined. Taking into account the errors from sample thickness and intensity at zero angle, the total error for the scale factor from water was estimated to be 0.02. The scale factor with new sample thickness of 0.1384 cm can be recalculated as $\text{SF} = 0.24 \pm 0.02$.

For the above described Rigaku instrument setup, 2 hours exposure time can produce scattering data from water with reasonably good statistics. To improve the statistics, longer measuring time is needed. Our measurements were repeated 4 times with an exposure time of 2 hours each. Four data sets were summed for data reduction and analysis. The data showed in Figure 2 have a total of 8 hours exposure time for each sample. For other low flux SAXS cameras, even longer measuring time would be required. The Rigaku S-Max3000 provides a second optional sample chamber closer to detector that allows exploration of a middle q range without moving the detector or realigning the beam. This setup also makes water calibration much less time consuming. The integrated counts for water in the second chamber are 1.92×10^6 per 4 hours, where as 7.56×10^5 per 8 hours for water in first chamber. The count rate for water in second chamber is about 4 times for water in first chamber that means for same statistics only about 1/4 measuring time is needed. Particularly, the flat region of scattering profile of water used for the calibration is in larger q area. The second chamber covers this q area. Therefore, practically, data from water in second chamber alone are enough for intensity calibration. Measuring water in first chamber is optional.

For intensity calibration, accurate transmission is required. Another advantage of the Rigaku S-Max3000 system is that it uses a photodiode embedded beamstop. It allows continuous monitoring of the beam intensity yielding a direct measurement of the sample transmission. A series of transmission measurements can be easily measured and then averaged. The obtained transmission is pretty accurate. The standard deviation is normally within 0.005.

Traditionally, the intensity in absolute scale for glassy carbon has been measured at a neutron facility. Using neutron data for X-ray intensity calibration has the following disadvantages: first, the scattering contrasts for neutrons and X-rays are different as determined by the nuclear scattering length and by the electron density difference, respectively. Therefore, the differential cross section obtained with neutrons needs to be converted to that for X-rays as follows:

$$\left(\frac{\partial \Sigma}{\partial \Omega}\right)_{x\text{-ray}}(q) = \left(\frac{\partial \Sigma}{\partial \Omega}\right)_{\text{neutron}}(q) \frac{(\rho_{\text{matrix}} - \rho_{\text{particle}})_{x\text{-ray}}^2}{(\rho_{\text{matrix}} - \rho_{\text{particle}})_{\text{neutron}}^2} \quad (5)$$

Second, establishing the linear region of the scattering profile from glassy carbon with any certainty is very difficult. Therefore, a non-negligible error is obtained when the differential cross section at $q=0$ is extrapolated. Using absolute intensity data measured at the synchrotron X-ray instrument allows one to scale the data collected at lab-based SAXS directly into absolute units.

The same glassy carbon with a thickness of 1mm was measured on the USAXS instrument at APS of ANL and on a Rigaku S-Max3000 SAXS. The exposure time was 10 and 5 minutes, respectively. The scattering profiles are shown in Figure 3. The data measured on the Rigaku S-Max3000 is scaled to USAXS data. We can see that two data sets coincide well within a common q range.

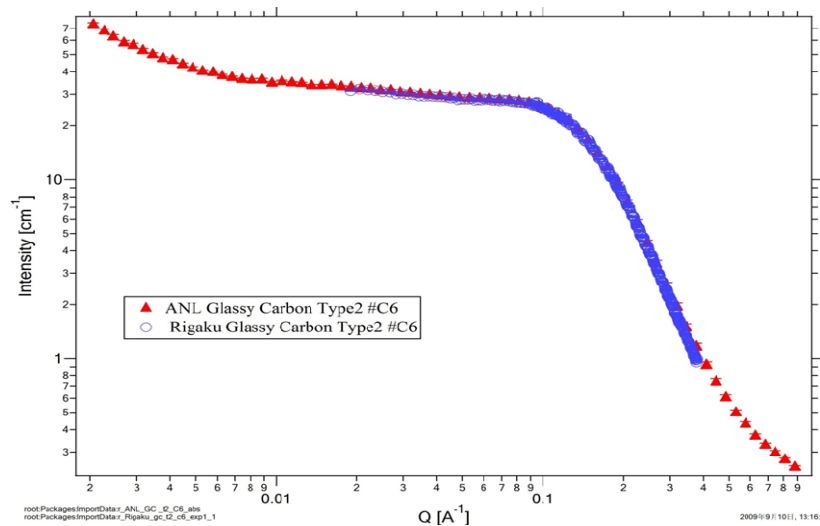


Figure 3. SAXS from glassy carbon in absolute units. Data collected on USAXS at APS, Argonne National Lab are in red; Rigaku S-Max3000 data are in blue.

We selected several q positions to evaluate the scale factors. For data obtained on Rigaku S-MAX3000, the transmission factor of the glassy carbon was corrected during data reduction, but the thickness is not yet normalized. Therefore the scale factor is calculated as $SF = (I_{ANL}/I_{Rigaku}) \times d_{GC}$, where I_{ANL} and I_{Rigaku} are measured intensities on USAXS at Advanced Photon Source of Argonne National Laboratory and on Rigaku S-Max3000 with laboratory X-ray source, respectively. The glassy carbon is in a form of a flat plate. Its thickness can be easily measured. d_{GC} is $0.1 \pm 0.8\%$ cm. The q positions and obtained I_{ANL}/I_{Rigaku} are listed in Table 1.

Table 1. I_{ANL}/I_{Rigaku} at different q positions

q (\AA^{-1})	I_{ANL}/I_{Rigaku}
0.0525	2.64
0.0753	2.64
0.1606	2.62
Average	2.63
Standard deviation	0.01

Using $I_{ANL}/I_{Rigaku} = 2.63 \pm 0.01$ and $d_{GC} = 0.1 \pm 0.8\%$ cm, we estimated the scale factor from glassy carbon as $SF = 0.26 \pm 0.02$. Once determined, the scaling factor can be used to convert other SAXS data, collected on the same SAXS instrument configuration, to absolute units by a simple multiplication.

The scale factor obtained with the glassy carbon standard is 0.26 ± 0.02 which is in an agreement within the error bars with the value obtained using water, 0.24 ± 0.02 . The measuring time for glassy carbon is just 5 minutes on the Rigaku S-Max3000, compared to 8 hrs for water plus 8 hrs for capillary making glassy carbon a much more convenient standard for a lab-based SAXS instrument. Over the years our lab at Rigaku Innovative Technologies, Inc., has regularly used glassy carbon from Alpha Aesar to check the performance of the SAXS camera. We found that the scattering profiles are repeatable within experimental errors and they are considered stable with no radiation damage. The temporal stability of glassy carbon was systematically studied [37], and it was proven that the glassy carbon microstructure is stable over time and is not affected by atmosphere or radiation exposure. During the inter-laboratory calibration, although the same block of glassy carbon is measured, different sample area may be probed due to the difference in beam sizes and sample location. Therefore, the spatial homogeneity of glassy carbon is concern for the calibration. Recently, the spatial homogeneity of one type of commercially available glassy carbon was studied [37]. It was demonstrated that glassy carbon is sufficiently homogeneous. These factors suggest that pre-calibrated glassy carbon is capable of serving as a general intensity calibration standard.

The high scattering power, temporal stability and sufficient homogeneity make glassy carbon a convenient standard. One should note that such a strong scatterer might cause a saturation problem on some detectors both at synchrotrons and in home laboratories [38]. For a CCD detector, the photon counts can be reduced by measuring for shorter times or using a filter. For lab-based gas proportional counters, the count rate is the key factor for evaluating the saturation. For many gas proportional counters, the count rate from the sample should be kept below 10^5 cps. There are many types of glassy carbons. One should be carefully chosen to avoid saturation effects. In our studies, the count rates were 2.17×10^4 and 3.57×10^4 cps for glassy carbon located in first and second chamber, respectively.

One should note that water still remains a useful calibration standard for dilute solutions of weakly scattering particles (proteins, micelles etc). The exposure time for the samples is similar to that of water and therefore the measurement of water does not represent a large 'overhead'. Particularly, when a capillary is used as the solution container, it is not trivial to estimate the effective sample thickness due to the beam spot size. Using water as a standard and using the same capillary for the measurements of the solution samples, the thicknesses for water and samples are cancelled out. Therefore, solution sample thickness is no longer needs to be determined.

5. Conclusions

This work has detailed the absolute intensity calibration procedures for lab-based SAXS instruments using two standards: water and homogeneous/stable glassy carbon. Water has a known differential cross section and no pre-calibration is required; however, the measurement is time consuming. Moreover, the beam path length through the capillary is not trivial to define due to beam spot size. We described a method to evaluate this value by measuring transmissions of the water in the capillary and empty capillary. The absolute calibration method using glassy carbon was based on a primary calibration X-ray instrument (the USAXS instrument at the APS at ANL). In this method the data collected on the lab-based camera can be scaled directly to data in absolute units measured on USAXS. This procedure avoids the non-negligible error produced by converting the differential cross section obtained with neutrons to X-rays. We compared scale factors obtained using water and glassy carbon. The good agreement of the scale factors suggest that pre-calibrated glassy carbon can serve as a general intensity calibration standard for all types of materials. Particularly, in the fields of materials science, polymer science, ceramics, and chemistry, when samples are more likely to have strong scattering, glassy carbon should be the standard. Water still remains a suitable standard for diluted solutions and serves bioscience well.

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