USAXS Absolute Intensity*

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Conversion of the scattering data into differential cross-section in units of cm^{-1} involves measuring the ratio of the number of photons scattered per second into unit solid angle to the number of photons in the incident beam.

The as-measured slit-smeared intensity profile (in some arbitrary units),

$$\tilde{I}(Q) = I_0 \ \Omega \ t \ T_s \ \frac{d\tilde{\Sigma}}{d\Omega}(Q) \tag{1}$$

is measured by a detector subtending a solid angle, Ω, with the sample. I_0 is the apparent source intensity incident on the sample, with identical units as $I(Q)$ and identical efficiency of detection. $d\tilde{\Sigma}(Q)/d\Omega$ is the slit-smeared differential scattering cross-section per unit volume per unit solid angle. The measured transmission is given by $T_s = e^{-\mu t}$ where μ is the linear absorption coefficient of the sample and t is the sample thickness.

With the USAXS instrument, the same detector that measures $I(Q)$ is used to measure I_0 with the sample removed from the instrument, the detector efficiency cancels and primary conversion of the measured intensity into units of $\mathrm{cm}^2/\mathrm{cm}^3/\mathrm{sr}$ involves only the measurement of t, T, Ω, and I_0. That is, the slit-smeared SAXS in absolute units measured by the USAXS instrument is

$$\frac{d\tilde{\Sigma}}{d\Omega}(Q) = \frac{R_s(Q)/T_s - R_b(Q)}{R_b(0) \ \Delta\theta_w \ \Delta\theta_l \ t} \tag{2}$$

where $\Delta\theta_w$ and $\Delta\theta_l$, which define the detector solid angle Ω, are the angular width and length of the beam intercepted by the detector.

The ratio, $R(Q)$, of detector counts, D, in time, t, to monitor counts, M, corrected for detector range dark current, d_r, and adjusted for detector range amplification, A_r, measured at each different Q, is

$$R(Q) = \frac{A_r \left[D(Q) - \tau d_r\right]}{M(Q)}. \tag{3}$$

The intensity ratio, $R(Q)$, is a superposition of the small-angle scattering from the sample, small-angle scattering from any windows between the collimating and analyzing crystals, convolution of the X-ray source profile with the rocking

*from the 1990 PhD thesis: http://www.uni.aps.anl.gov/~ jemian/jemian-phd.pdf curves of the monochromator and USAXS optics, and parasitic background scattering of the X-ray beam in the air path between the sample position and the detector. The source convolution will be shown later to be negligible. At $Q = 0$, the USAXS analyzer crystal conducts the full intensity of the unscattered beam (I_0, the intensity apparent at the detector) so the sample transmission,

$$T_s = \frac{R_s(0)}{R_b(0)}, \tag{4}$$

where $R_s(Q)$ and $R_b(Q)$ are the curves measured for the sample and blank beam, respectively, normalized to a constant monitor count rate. The blank beam is a scan which is the same as a SAXS scan in every regard except that the sample is removed.

It may be assumed that all effects except the scattering from the sample and a small additional background will be found in both R_s and R_b. The instrumentally-smeared small-angle scattering plus small additional background in arbitrary units is then separated by scaling the sample data by its transmission factor and subtracting the empty beam, as

$$\tilde{I}(Q) = R_s(Q)/T_s - R_b(Q). \tag{5}$$

In a SAXS camera that utilizes geometric collimation with a concomitantly small illuminated area, e.g. (Wignall & Bates, 1987), the solid angle is defined by the area of the beam on the detector element, Δa, and the distance between the sample and detector, L_{SDD}. However, in the USAXS instrument, the angle in the scanning direction is more highly-collimated by the rocking curve of the crystal optics. Therefore, $\Delta\theta_w$ is measured from the experiment as the full width at half maximum of the empty beam, $R_b(Q)$. With no crystal optics in the slit-length direction, $\Delta\theta_l$ is defined in the geometric sense. For a large illuminated sample area defined by the width and length of the source slits, where $A = W_S \times L_S$, and a detector diameter of L_D, where $L_S < L_D$,

$$\Delta\theta_l = \frac{L_D + L_S}{2L_{SDD}} + \frac{L_D - L_S}{2L_{SDD}} = \frac{L_D}{L_{SDD}}. \tag{6}$$

The detector diameter (diameter of the active area of the unbiased PIN photodiode) used in the UNICAT USAXS instrument is 5.5 mm and the sample-to-detector distance is typically 300 mm. Since L_{SDD} need be known to only a precision of 5 mm or so, the additional X-ray path distance due to the mutiple reflections in the analyzer is negligible with respect to this precision. Thus it is evident that although primary intensity calibration usually requires special equipment and a standard sample, and can be difficult and time-consuming to perform, the USAXS instrument requires no additional beam time for calibration since all of the parameters are available in the normal course of performing scattering measurements.