

Absolute Scattering Standards



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Absolute Scattering Standards



Why should we care?

- Compare scattering data from different instruments – laboratory SAXS, Synchrotron SAXS, SANS.
- For Standardless absolute scattering, a universal standard provides a self-consistent check.
- Desirable Qualities for a Standard
 - Strong Scatterer so that Background is orders of magnitude lower intensity
 - Relatively uniform from batch to batch
 - Stable over time
 - Free Standing – No windows to subtract
 - Definitive Peak or Plateau
- Glassy Carbon (GC) is a good candidate
 - **Pro:** Plateau in SAXS is a good reference intensity
 - **Con:** relies on a standardless scattering instrument for calibration and we don't have clear consensus to its value.

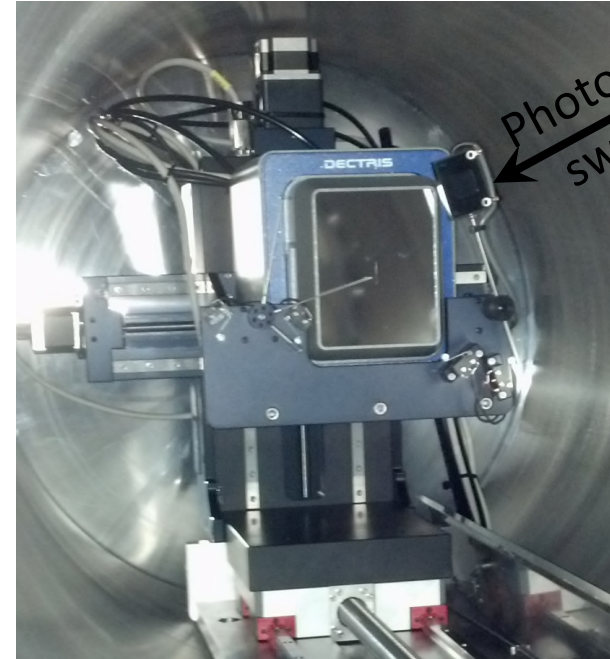
Water Standard Comments



- Water or other low Z liquids are poor choice for reference
- Long laboratory integration times (>30 min) for both sample and container
- Relies on subtraction of 2 weak signals – cell with and without sample
- Noise is usually larger than desired precision.
- Compressibility limit requires linear or second order fit – $I(q) \rightarrow 0$ is not a flat line.
- Flat window cell non-trivial in vacuum because path length can change from differential pressure against windows.
- Capillary cells have unknown Inner Diameter and curvature presents non-constant path length.

Standardless Measurement

Ganesha from SAXSLAB



Characteristics

- Sealed Tube Microsource with 2D Multilayer Optic (**Cu** or **Mo**).
- 2 or 3 Aperture (“scatterless” slits).
- Moving Pilatus 300 Detector (172 x 172 μm^2 pixel size) in vacuum. Continuous SAXS to WAXS like a SANS line.
- Non-Paralyzable Deadtime correction applied internally and calibrated by Dectris.
- Can measure 1MCps/pixel/sec.
- Use the flat field correction provided by Dectris.
- Photodiode used to measure direct beam and transmission, calibrated to the Pilatus.

Absolute Scattering for Strong Scatterer



η_1 and η_2 are efficiencies for direct beam and scattered intensities, respectively. These are equal if the photodiode is scaled to the Pilatus.

Φ is the incident beam flux and A is the area. ΦA is the total incident intensity in ph/s. t is the measurement time.

T is the transmission measured by the photodiode with and without sample. δ is the sample thickness.

$\Delta\Omega$ is the solid angle of detector for a given pixel. Its value is $\frac{p^2}{L^2} / \{\cos(2\vartheta)\}^{-3}$, where the $\cos(2\vartheta)^{-2}$ accounts for the pathlength at higher angles and the additional $1/\cos(2\vartheta)$ factor is the projection cosine of the pixel normal to the scattered beam.

Background Corrections: Cosmic Ray Corrections only.
Measurement in Vacuum

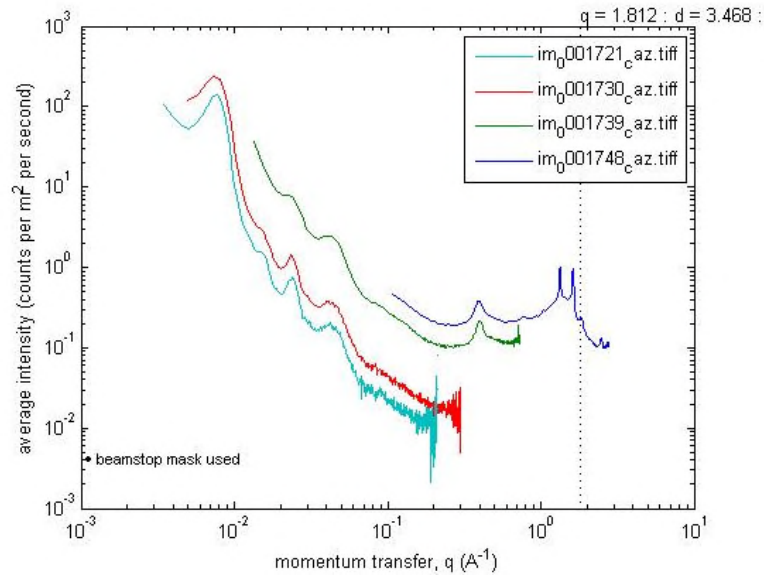
$$I(q) = \frac{1}{V} \left(\frac{d\sigma}{d\Omega} \right), \quad q = \frac{4\pi \sin(\theta)}{\lambda}$$

$$= \frac{\eta_2 I_{ij}}{\eta_1 (\Phi A) T t} \frac{1}{\Delta\Omega} \frac{1}{\delta}$$

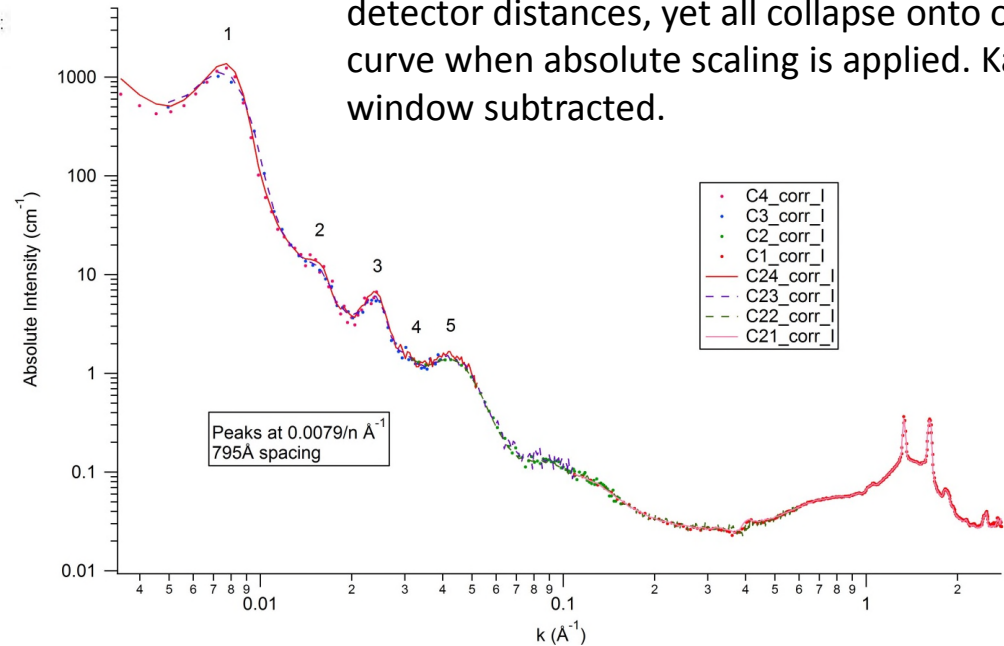
– *Background*

Motivation

No Scaling Applied. Angularly integrated scans from Raw 2D data



These data were collected in both 2 and 3 aperture geometries, different sample-to-detector distances, yet all collapse onto one curve when absolute scaling is applied. Kapton window subtracted.



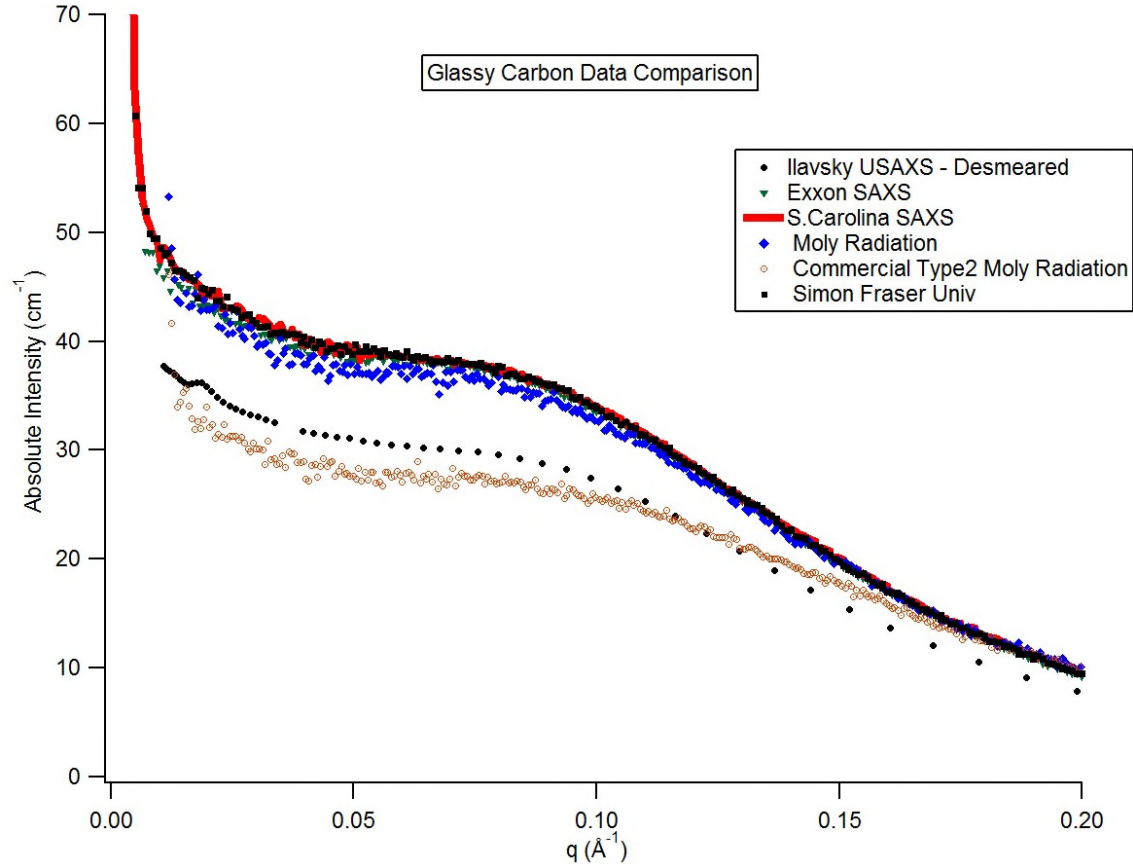
Additional Pilatus Corrections



- Scans are composed of addition of shorter frame scans
 - Frame time adjustable 0.1s to 15sec typical
- Cosmic ray corrections applied by looking for above-average events within a given frame compared to the final additive image.
- Vacuum measurement in minutes. No significant background signal
- No readout noise with Pilatus

Comparison of Glassy Carbon Results

- These are data measured on a GC standard provided by Jan Ilavsky from APS.
- The dotted black curve at the the plot of standard data provided by Jan Ilavsky.
- The upper cluster of curves are data reduced data from 3 different laboratory instruments with Cu Ka and 1 with Mo Ka.
- The lower curve with open circles is a commercial type2 GC measured with Moly Ka



- Where is discrepancy with Ilavsky data coming from?
 - How reliable is the GC standard as received?
 - We received a batched sample
 - A different commercial sample is in line with expected intensity
 - We're getting a new sample that was measured directly at SR facility
 - Is there a background issue?
 - No. All data scale by a multiplicative factor.
 - Is there a detector offset from flat field correction?
 - Not likely, since Cu and Moly data are in agreement.
 - Offset from deadtime correction?
 - Probably not because we see excellent overlap in corrected data from different I_0 .