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

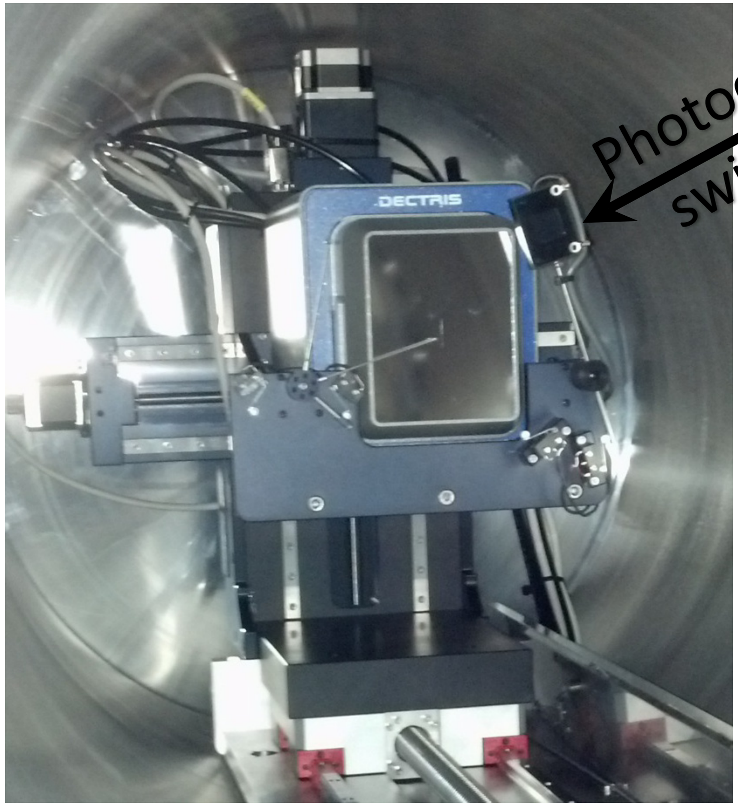
- Glassy Carbon (GC) is a good candidate
  - **Pro:** Plateau in SAXS is a good reference intensity
  - **Con:** relies on a standardless scattering instrument for calibration and we don't have clear consensus to its value.

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



Photodiode detector swings into beam

Characteristics

- Sealed Tube Microsource with 2D Multilayer Optic (**Cu** or Mo).
- 2 or 3 Aperture ("scatterless" slits).
- Moving Pilatus 300 Detector (172 x 172 mm^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 $\frac{p^2}{L^2}/\{\cos(2\theta)\}^{-3}$, 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

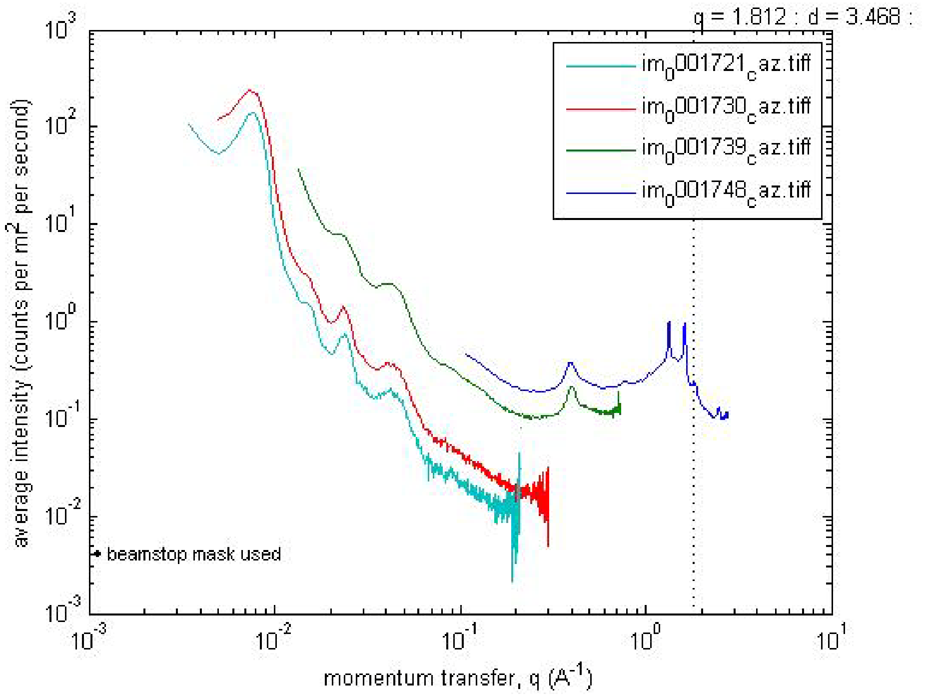
$$I(q) = \frac{1}{V}\left(\frac{d\sigma}{d\Omega}\right), \quad q = \frac{4\pi \sin(\theta)}{\lambda}$$

$$= \frac{\eta_2 I_{ij}}{\eta_1(\phi A)Tt}\frac{1}{\Delta\Omega}\frac{1}{\delta}$$

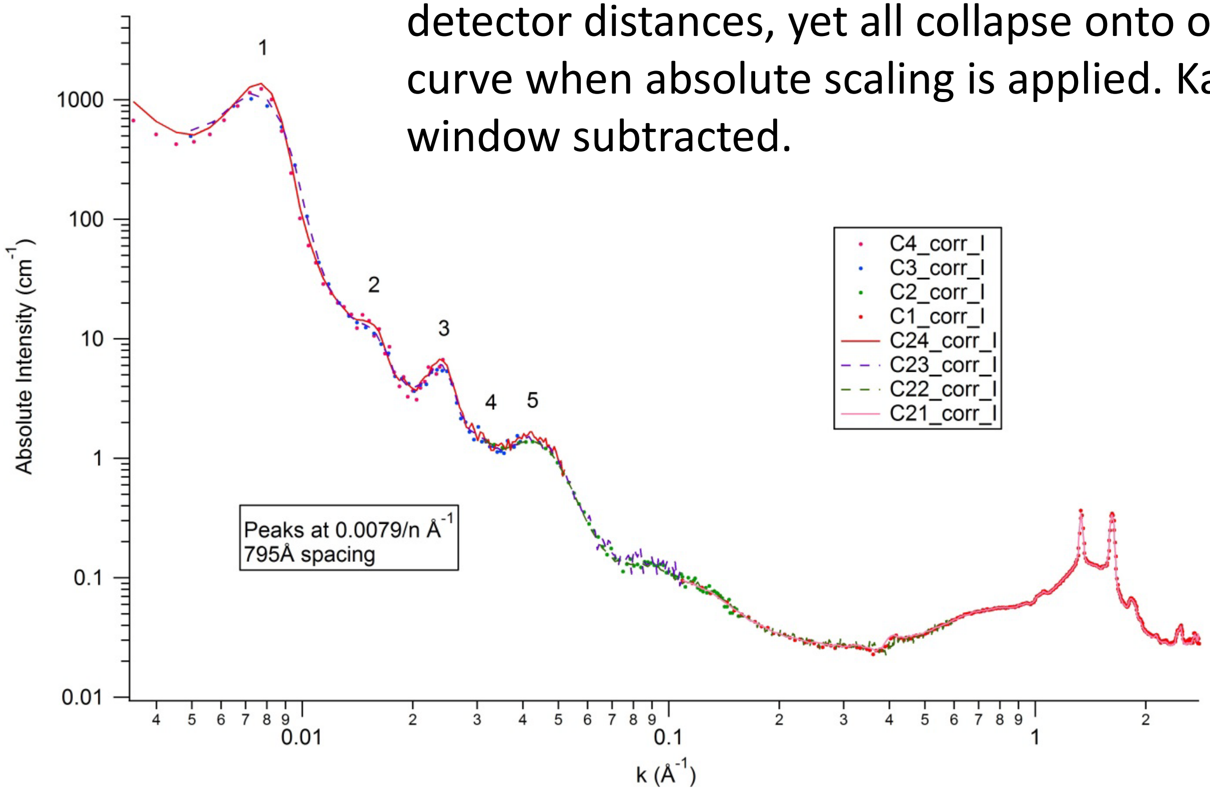
$$- Background$$

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.

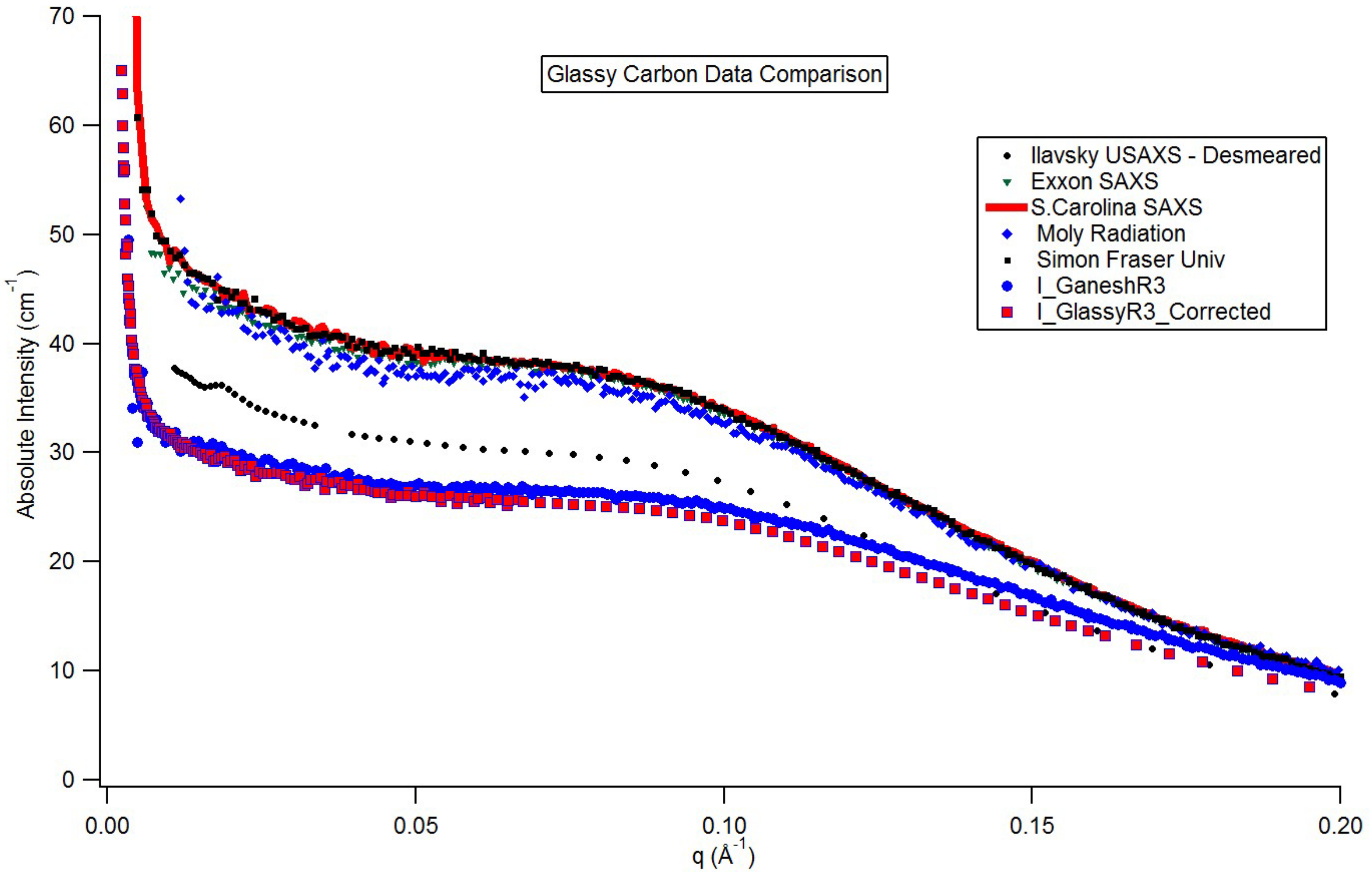


Additional Pilatus Corrections

- 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

- Ilavsky USAXS are data provided for sample M12
- The upper cluster of points are scattering on the M12 sample measured at different Ganesha SAXS instruments and another equipped with Moly Ka using the SAXSLAB reduction routine.
- The lower 2 curves are for the R3 sample measured directly at the APS USAXS and on the UMASS Ganesha



Observations and Conclusions

- The M12 measurements are consistent on the different Ganesha instruments (with Cu Ka) and also on another instrument with Moly Ka.
  - The M12 Ilavsky data are scalable to our measurements.
- The R3 Measurements agree, once the thickness for the APS sample is corrected from 1mm to .885mm.
- We have to conclude that
  - **The M12 sample provided has the incorrect calibration curve. To our understanding, this sample was not measured directly, but rather was a sample selected from a statistically measured batch. It is an outlier.**
  - **The R3 sample we received was measured by Jan Ilavsky directly at APS. The sample thickness was assumed to be 1mm, but it is actually .885mm +/- .005 thick. The APS data were adjusted (divided by 0.885) and are within 5% of our measured data.**