An Ultra-SAXS Instrument on a Shoestring Budget

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The Source

While the proof-of-concept (POC) version utilized a repurposed rotating anode source (with molybdenum target), its high power requirements, high maintenance cost and fragility led to its eventual disposal (after developing yet another expensive fault). By then, only a few measurements had been recorded, which demonstrated the instrument feasibility and functionality.

The acquisition of a new source sparked efforts to upgrade instrument components as well, leading to the current prototype version. The new source was chosen to be a Xenocs microfocus source with a molybdenum target (emitting 17.5 keV photons), with integrated low divergence optics.

The higher energies appear well suited to Ultra-SAXS experiments, resulting in lower air interaction and higher transmission through samples (e.g. metals, abrasives, powders and aerogels).

Collimation

The collimation in this instrument offers coarse beam shaping ability, and largely removes the truly useless radiation (the majority of the angular filtering of the incident beam will be done by the upstream channel-cut crystal). An additional shutter and attenuation wheel provides extra beam control, but will probably be removed in future instrument iterations.

The collimation is largely constructed from parts printed in ABS plastic. These parts hold recycled JEOL slits in place (two each, mounted perpendicularly). The beam path is protected from accidental entry by foreign objects using DIN KF-flanged flexible vacuum tubing. Due to the slit slots, however, the system is not vacuum-tight in its current design.

The construction using XT95 rails allows for high flexibility in collimation component placement and provides a stable platform.

Rotations

The upstream crystal has a very narrow angular filter window, but only needs a rotation accuracy matching the divergence of the impinging beam. The upstream rotation, therefore, consists of a commercial rotation stage for convenience.

The high precision rotation which *is* required for the downstream crystal, is achieved using a sine-bar rotation constructed from inexpensive components. The core components are a cross-roller ring that forms a stable rotation center, and a linear actuator acting upon an arm several hundred mm from the rotation center. As this sine bar only effects a rotation of about 2°, a coarse manual rotation sits on top.

To simplify alignment, either rotation stage assembly can be slid out of place on linear guides at the base. Vertical alignment is achieved using a large jack.

Future versions will further reduce the complexity of the rotation stage design, and augment the design with an encoder on the arm.

Detector System

The detector system on the POC version consisted of a repurposed 25mm diameter NaI scintillation detector. This was connected to a shaping amplifier, a single-channel analyser and a counter/ratemeter unit. As the latter was not available in the collection of repurposed equipment, and with new NIM variants costing too much, such a unit was built by hand instead. An "Arduino" programmable microcontroller was used for this purpose, mounted in an old NIM housing. With a straightforward program, this unit could serve as counter/ratemeter for rates up to 50000 CPS. However, the "darkcurrent" countrate of the scintillation tube remained at 0.1 CPS, problematic at high Q.

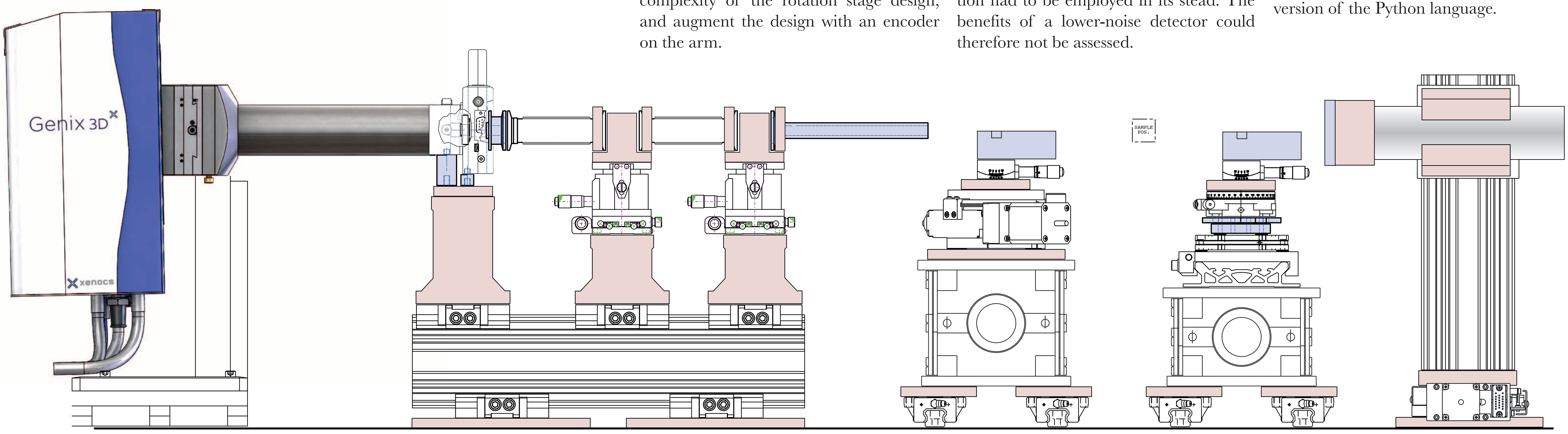
The prototype version was to resolve this issue by using a silicon drift detector instead. However, hardware failure of the new detector system coupled with time pressure meant that the old detector solution had to be employed in its stead. The benefits of a lower-noise detector could therefore not be assessed.

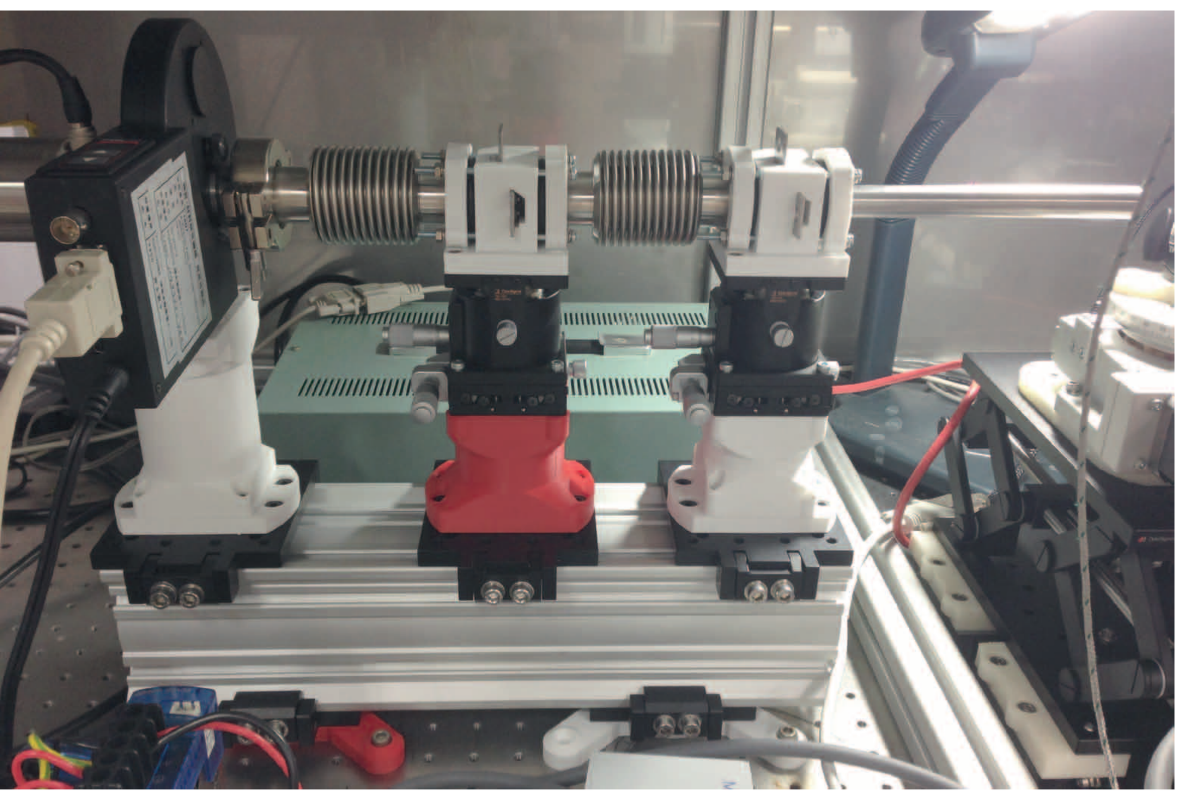
The Odds and Ends

This instrument was built on top of a large, repurposed optical table. While nice and flexible, buying such a table (when not available) would be too cost-prohibitive. For the next version, therefore, a less expensive solution is sought, and will probably be found in the shape of 8020 profile.

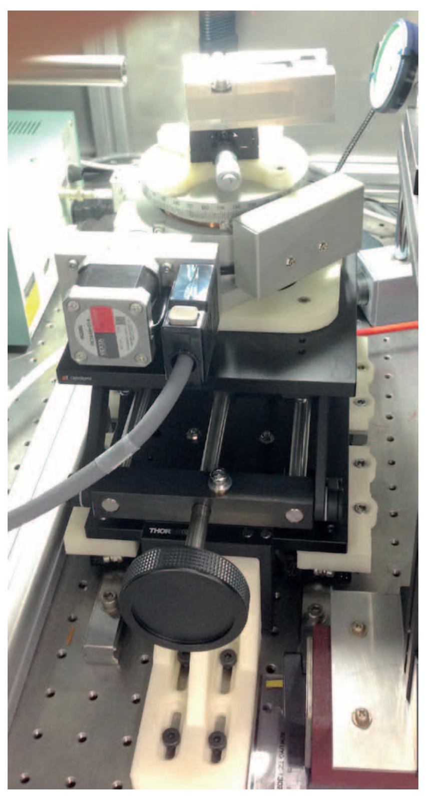
Shielding around the instrument consisted of a box made from aluminium profile, in which 2mm steel sheet was suspended. Such shielding would guarantee that no photon would escape within the lifetime of the sun (to 99% certainty). Lighter, and more transparent shielding can, perhaps, be considered for future versions.

The instrument electronics are connected to the computer via ethernet, where necessary with an intermediate ethernet-to-serial translation device. The controlling computer can be any computer with a network connection capable of running a version of the Python language.

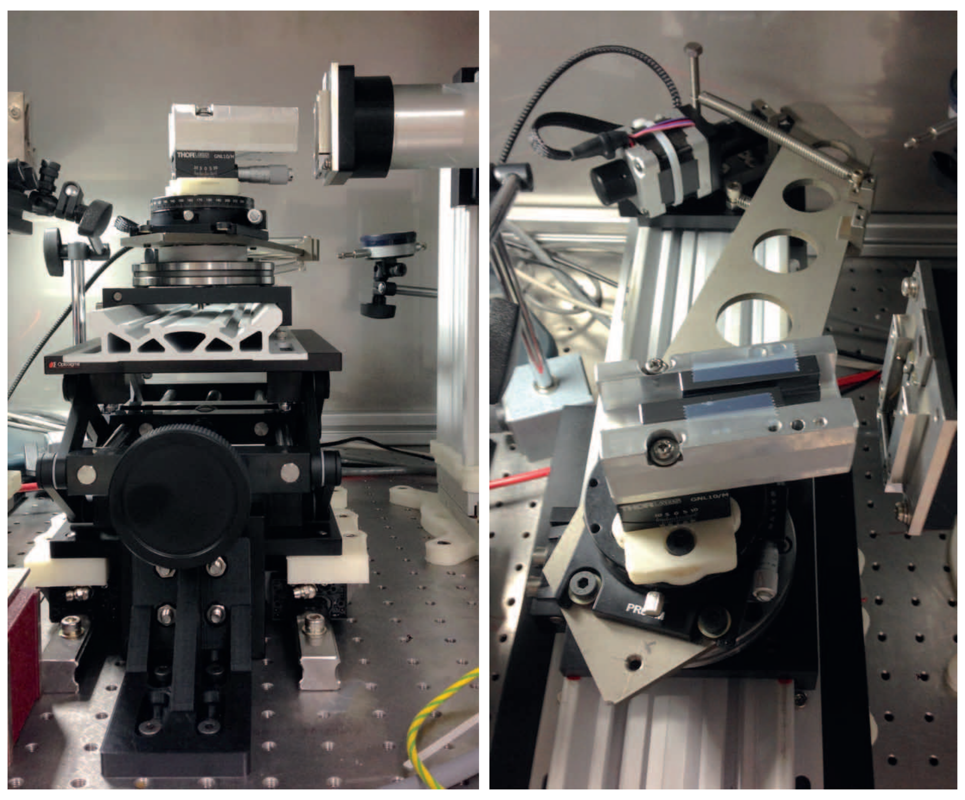




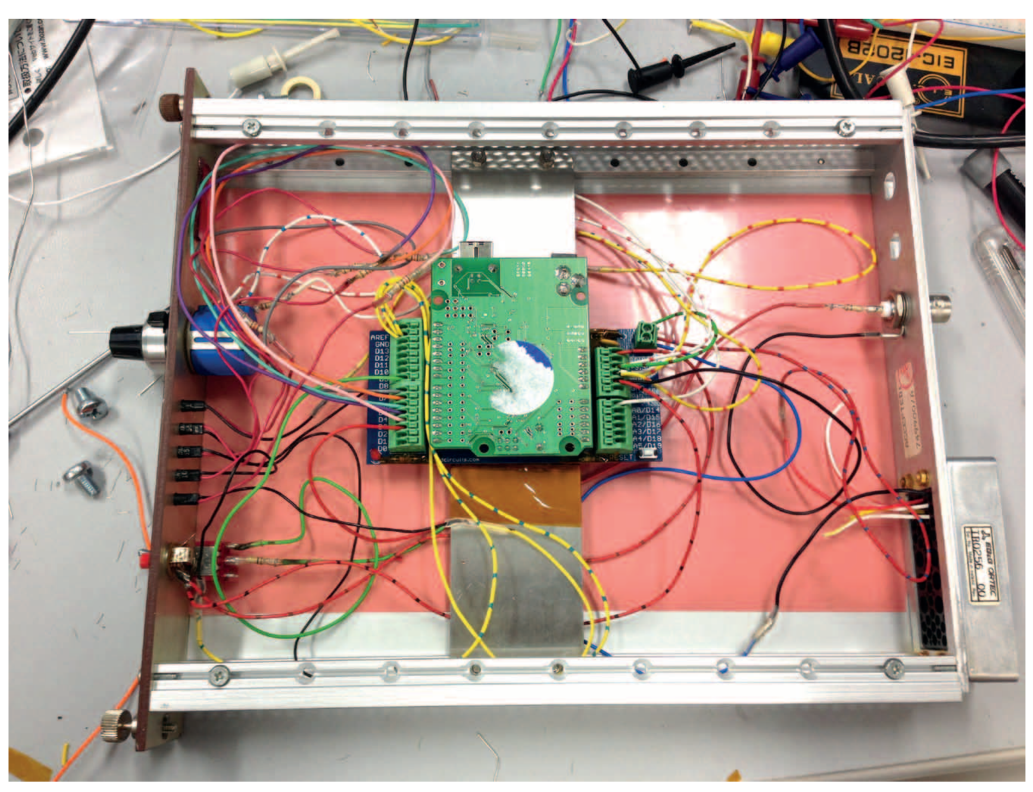
The collimation section



The upstream rotation



The downstream rotation with sine-bar mechanism



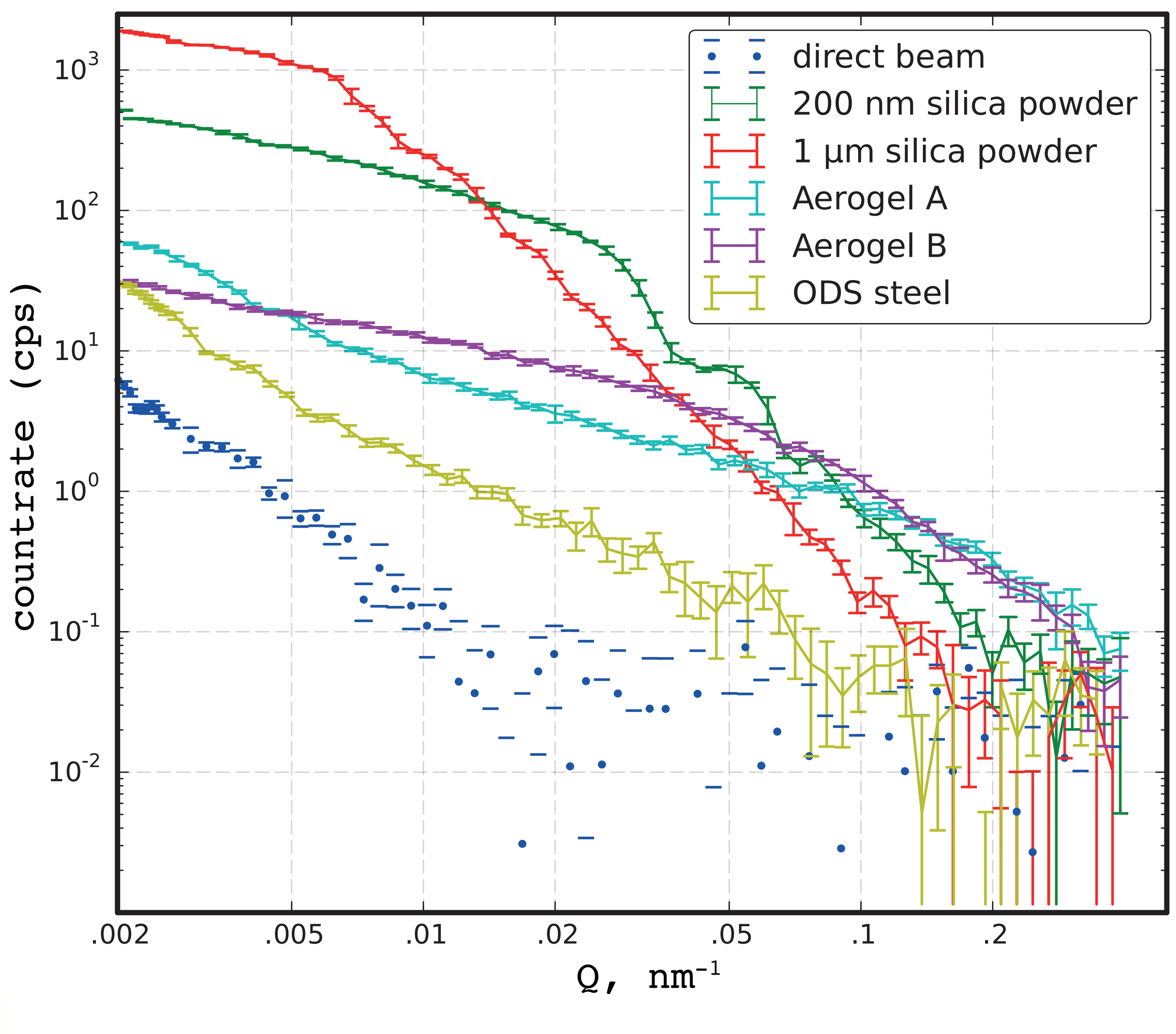
The NIM-duino: an Arduino-based counter/ratemeter

The Bill

	kJP¥	kEUR
Source:	10000	73.420
Collimation:	363	2.665
Upstream Rot.	435	3.194
Downstream Rot.	437	3.208
Detector (sdd)	1823	13.384
TOTAL:	13058	95.871

The bill does not include:
- Tools (easily 20kEuro)
- 3D printer (3 kEuro)
- Crystals (5 kEuro / crystal)
- Shielding (2 kEuro)
- Base (unknown cost)
- Time (priceless)
- Lab space (unknown cost)

The Results



The Conclusion:

Provided a source (and optionally, a detector) is available, an in-house Ultra-SAXS instrument can be constructed for about 10 kEuro. The design is suitable for 17.4 keV X-ray photons, allowing for thicker samples and iron-containing materials to be measured.

While the signal-to-noise ratio in the low-Q region of the measurement is decent, the performance deteriorates towards high-Q due to a relatively high darkcurrent level (0.13 cps) of the scintillation tube detector. This issue is expected to be largely resolved with the replacement of the detector by a modern (fast) silicon drift detector. At the moment, air scattering is not a measurable contributor to the scattering patterns. This is likely due to the high energy, the short distance between the two crystals, and the high darkcurrent level.

The measurements show a high suitability of the instrument for strongly scattering samples, in particular for powders and aerogels. Steel samples may be appropriate as well.

De-smearing, however (not shown here) rapidly amplifies the uncertainties to untenable levels. Therefore, any analysis of the Ultra-SAXS patterns should be performed by smearing of the fitting models rather than attempting to de-smear the scattering patterns.

When the instrument is to be married to an already available SAXS instrument (to extend its measurement range), the quality of the SAXS instrument starts to matter. The practically achievable minimum Q of such a SAXS instrument is recommended to be better than 0.1 nm^{-1}.