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White paper

Advancing Scattering Experiments with Hybrid Photon Counting Detectors

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Small and Wide Angle X-ray Scattering are powerful techniques for investigating structural properties of materials in different sizes and shapes, from nanometers to atomic resolution. This white paper highlights how Hybrid Photon Counting detectors enable the high-resolution data collection, free of detector background, in both laboratory and synchrotron environments. We present two case studies: one on dynamic biological studies at a synchrotron beamline and another on detailed polymer analysis using a laboratory source, both demonstrating how advanced structural characterization benefits from Hybrid Photon Counting technology.

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1. Introduction

Small-Angle (SAXS) and Wide-Angle X-ray Scattering (WAXS) are powerful techniques for characterizing structures across multiple length scales. Both rely on X-ray scattering, which encodes information about a sample's internal organization. SAXS probes large-scale features (1–100 nm) such as macromolecular complexes, colloids, porous systems, and biological samples in their native state [1]. WAXS reveals atomic- and molecular-level details, including crystalline arrangements, polymer conformations, and phase transitions [2]. Specialized methods such as Ultra-Small-Angle X-ray Scattering and Grazing-Incidence Small-Angle X-ray Scattering further expand the accessible length scales and geometries [3, 4].

2. Collecting high quality scattering data with HPC detectors

Direct detection with Hybrid Photon Counting (HPC) detectors, such as EIGER2 and PILATUS4, eliminates the signal blurring common in scintillator-based area detectors and provides the high spatial resolution needed to resolve fine scattering features. Their high dynamic range allows simultaneous measurement of both intense and faint signals, reducing the need for multiple exposures, improving data consistency, and minimizing radiation damage. In addition, frame rates of up to 4000 images per second without dead time enable true time-resolved studies at synchrotron facilities [5-7].

Because many scattering signals are inherently weak, experiments are typically conducted in vacuum to suppress air scattering, which would otherwise interfere with the signal — a setup fully compatible with EIGER2 and PILATUS4. Furthermore, their adjustable energy threshold rejects electronic noise, ensuring even the weakest signals are measured with precision. This also allows for long exposure times without noise pileup, enabling accurate data collection with laboratory sources [8].

By combining resolution, speed, sensitivity, and noise suppression, HPC detectors are the optimal choice for SAXS and WAXS experiments.

3. High precision scattering measurements in Synchrotron Radiation Facilities

Synchrotrons provide highly intense and focused X-ray beams, enabling high-resolution measurements over a wide range of scattering angles. These facilities are ideal for time-resolved experiments, high-throughput studies, and advanced SAXS/WAXS techniques. The combination of synchrotron radiation with state-of-the-art detectors like the DECTRIS EIGER2 or PILATUS4 enables the study of fast processes, such as protein folding, phase transitions, and structural changes in materials.

The advancements in the 4th generation synchrotrons, based on multi-bend achromat lattices, offer dramatically improved brilliance and spatial coherence. These beam properties enable techniques such as scanning SAXS, X-ray Photon Correlation Spectroscopy (XPCS), and coherent diffraction imaging, expanding the frontiers of structural and dynamic studies [9]. The use of fast, noise-free detectors has also made possible ultrafast experiments, including picosecond pump-probe studies [10], offering new insights into rapid physical and biochemical processes.

3.1 Enhancing Time-Resolved SAXS with PILATUS4

A prototype PILATUS4 detector was employed at the P12 SAXS beamline at EMBL, PETRAIII, DESY, Hamburg, to study dynamic structural changes in biological macromolecules. These experiments demonstrated its capability to capture high-quality SAXS data with exceptional temporal resolution when combined with state-of-the-art sample environment tools, such as fast mixing devices. Specifically, time-resolved SAXS (TR-SAXS) measurements utilizing the Micro Stopped-Flow Mixer (μSFM) enabled precise control over reaction initiation, allowing researchers to monitor rapid structural transitions [11]. For example, the dissociation 24-mer spherical of apoferritin multicomplex into singular subunits under acidic conditions was captured

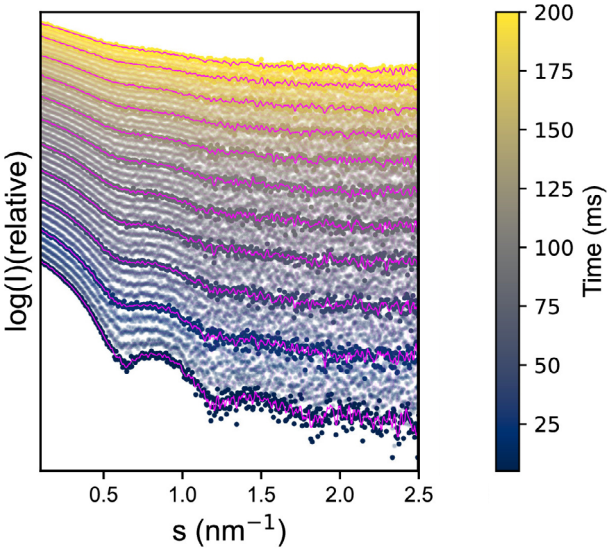


Figure 1: Time-resolved evolution of the dissociation of Apoferritin. Data were collected using a PILATUS4 260K prototype in combination with a stopped-flow setup.Image provided courtesy of Aleksi Sutinen, P12, EMBL, DESY.

over hundreds of milliseconds, providing valuable insights into the macromolecular dynamics associated with intraparticle distances with sub-domains or interparticle interactions between two macromolecular species.

The PILATUS4 detector supports frame rates of up to 4 kHz, making it ideal for time-resolved experiments. In this study, data were collected at 2 kHz. Temporal fine slicing enables retrospective optimization of the trade-off between time resolution and signal intensity — a strategy often referred to as 'collect before destruction'. Combined with the µSFM stopped-flow system at P12, this setup ensures efficient sample usage while achieving high temporal resolution. The PILATUS4's ability to handle high photon flux and support gated acquisition modes further makes it well-suited for TR-SAXS studies involving rapid conformational changes triggered by pH shifts, ligand binding, or other stimuli. The full story can be read in [11].

These advancements address key challenges in TR-SAXS, such as minimizing radiation damage and optimizing sample consumption. Providing the high temporal resolution necessary for capturing dynamic processes before the sample is compromised requires fast data to be read out with high precision. Combining the P12 beamline infrastructure with advanced detector technology such as PILATUS4 establishes a robust platform for time-resolved SAXS. Future upgrades at P12, including integration with the PETRA IV synchrotron source, will further enhance opportunities for high-resolution TR-SAXS studies, enabling deeper insights into macromolecular dynamics.

4. Versatile measurements in laboratories/using laboratory systems

Laboratory SAXS/WAXS systems offer convenience and accessibility, allowing researchers to perform routine measurements without the need to wait for beamtime and to travel to a synchrotron facility. Although the X-ray flux is lower in laboratory setups, advanced HPC detectors effectively counteract this limitation by having zero detector background noise and high quantum efficiency. Their superior sensitivity enables high-quality data collection even with longer exposure times. DECTRIS detectors, such as PILATUS4 and EIGER2, are widely used in both laboratory and synchrotron setups, providing reliable and precise measurements by maximizing signal-to-noise ratio and ensuring optimal data quality under varying experimental conditions.

4.1 Benefits of the DECTRIS EIGER2 for Laboratory SAXS with Specific Applications

The DECTRIS EIGER2 R 4M photon-counting detector significantly enhances laboratory SAXS capabilities, especially for high-throughput and detailed nanostructure characterization. For example, at the BioPACIFIC SAXS-WAXS beamline, the EIGER2 R 4M has been utilized in combination with a MetalJet X-ray source from EXCILLUM to study bio-derived polymers and nanoparticles. The detector's small pixel size (75 µm) and large active area (155.1 × 162.2 mm) enabled high-resolution measurements across a broad q-range, providing insights into structures from 0.1 nm to 100 nm. This capability allowed researchers to investigate particle size distributions and hierarchical structures in bio-derived polymers crucial for material innovation.

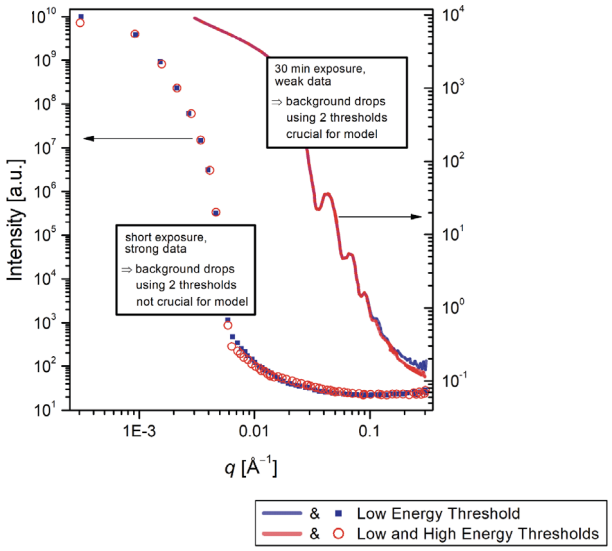


Figure 2: Background reduction using dual-threshold data acquisition. Data provided by Phillip Kohl and Youli Li from the BioPACIFIC NSF Materials Innovation Platform at UC Santa Barbara.

A specific advantage of the EIGER2 R 4M is its dual energy discrimination capability. For weakly scattering samples such as dilute polymer solutions, the two thresholds helped minimize environmental background, improving the clarity of the scattering patterns. This was particularly evident during long exposure times, where precise buffer subtraction was required. In one study, the detector's sensitivity enabled clear differentiation between scattering signals from the sample and noise, which is essential for accurate modeling of nanostructures.

The high frame rate of the EIGER2 R 4M of 20 Hz also facilitated rapid data collection, critical for experiments involving dynamic environments such as temperature changes or in situ mixing. For instance, during mix-SAXS experiments on polymer blends, the detector captured structural changes in real-time, enabling the

study of reaction kinetics and phase transitions with high temporal resolution. These specific applications highlight the EIGER2's ability to provide accurate, high-quality data for complex systems, making it a powerful tool for advancing SAXS research in laboratory settings.

5. Conclusion

HPC detectors, such as EIGER2 and PILATUS4, provide significant benefits for SAXS and WAXS applications. Their low noise, high dynamic range, fast frame rates, and radiation hardness make them ideal for both routine and advanced scattering experiments. EIGER2's small pixel size and PILATUS4's high-speed capabilities offer specific advantages for high-resolution and time-resolved studies, respectively. By using HPC detectors, researchers can obtain high-quality data that enable deeper insights into the structure and dynamics of materials.

6. Acknowledgements

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