The new PHOTON III HE detector – unbeatable sensitivity for short wavelength crystallography

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Introduction

Figure 1: PHOTON III Family.

Single crystal X-ray diffraction (SC-XRD) is undisputedly the most powerful tool to determine the (absolute) 3D structure of crystalline matter. A core component of every SC-XRD instrument is the X-ray detector for accurate, reliable and fast data collection. Over the past decade, X-ray detectors with single photon sensitivity became state-of-the-art also for the home lab.

The latest generation of mixed-mode detectors* have demonstrated significantly improved data quality. With the PHOTON III Bruker has made the latest achievements available for the home lab. While silicon-based photon-counting detectors suffer from notoriously low detection efficiency for high energy radiation, the PHOTON III detector family prevailed with good detection efficiency for these applications.

The new PHOTON III HE even goes beyond and presents a dedicated X-ray detector for hard Ag-radiation.

*'Mixed-mode' refers to integrating detectors with photon counting capability.

X-ray Detection in the PHOTON III

The PHOTON III combines latest development mixed-mode technology (e. g. JUNGFRAU[1], AGIPD[2]), with well proven scintillator-based X-ray detection and low noise CMOS technology. The PHOTON III integrates the charge produced by the X-rays incident on the detector during a short (14 msec) exposure time. Photon counting is then achieved by locating and counting single X-ray events in the individual frames. This fine-sliced charge integration approach in combination with subsequent photon counting has several advantages compared to previous generation photon counters:[3]

- No charge sharing noise
- Elimination of pulse pile-up effects
- High X-ray absorption for all wavelength
- Large active area
- No gaps/dead areas
- Very low parallax
- Scintillator can be optimized for specific applications

Active Area	Up to 240 x 200 mm²
Pixel Size	135 µm
Gaps, dead areas	None
Conversion	Indirect
Conversion Gain	25e⁻/keV
Electrical noise	< 10 e⁻
Energy range	6 – 30 keV
DQE	> 90%
Frame rate	70 – 140 Hz
Point spread function	< 90µm
Parallax	< 1 pixel
Stability	< 0.1% variation
Linearity	< 1% over full range
Cosmic ray rejection	> 99.99% eliminated
Charge sharing noise	None

Table 1: Specifications of the PHOTON III X-ray detector.[4]

Why hard radiation? Is Ag the new Mo?

Mo radiation is still the most popular wavelength for chemical crystallography. However, the great progress in Ag IµS microfocus sources has made shorter wavelength experiments much more feasible. Furthermore, the introduction of the METALJET complemented the high energy spectrum further with In radiation. These high-intensity, hard-radiation sources provide several benefits in range of diffraction experiments including:

- Higher maximum resolution
  (PDF, Charge density)

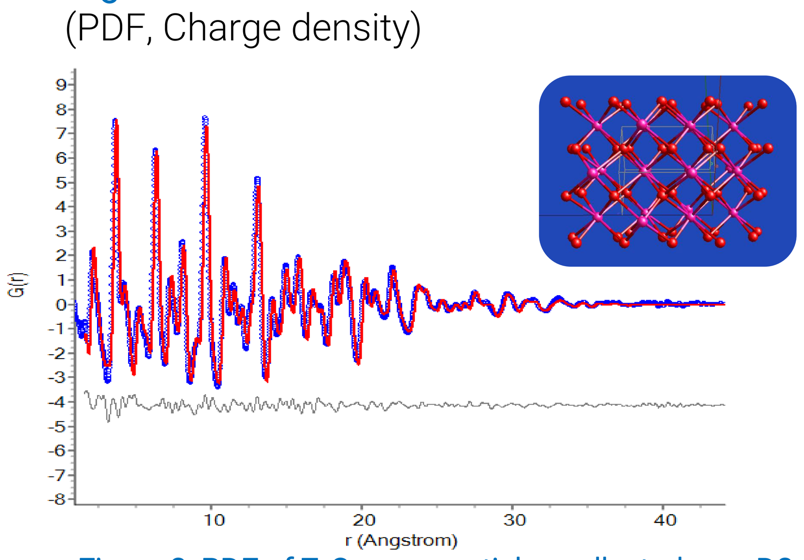


Figure 2: PDF of ZrO_2 nanoparticles collected on a D8 VENTURE

- Less absorption and scaling issues
  (Heavy elements, anisotropic crystals)

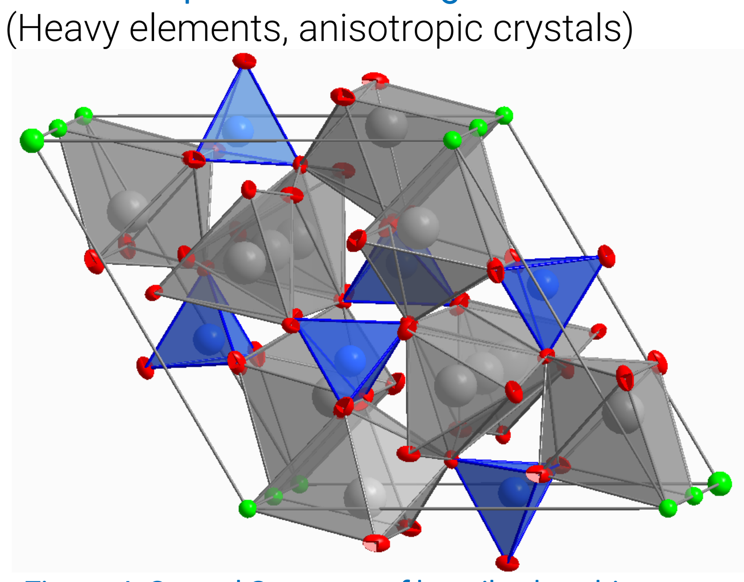


Figure 4: Crystal Structure of heavily absorbing $Pb_5(VO_4)_3Cl$.

- More data in geometrically restricted setups
  (DAC, Gas cell, flow cell, crystallization plate, …)

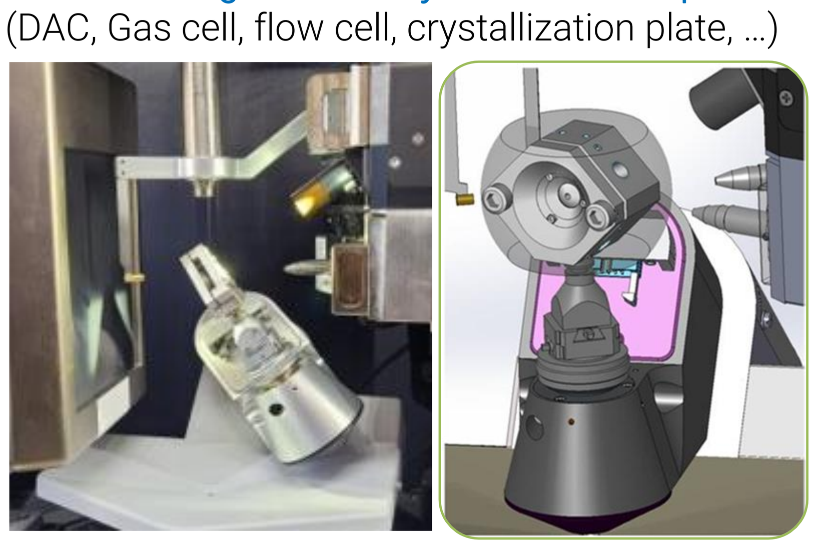


Figure 3: High-pressure setup using a D8 VENTURE.

However, hard radiation poses practical challenges for the detector. The quantum efficiency of the detector is proportional to the atomic number, Z, of X-ray sensor. Therefore, detectors with conventional silicon sensors exhibit very low quantum efficiency for Ag radiation, typically on the order of 30% only.

Here the PHOTON III HE makes the difference!

Optimized Scintillator – Best data

To a large extent the data accuracy obtained by modern detectors is based on the capability to count single photons. However, you can only count what you "see". In conventional silicon sensors two-thirds of Ag radiation passes right through the sensor and is thus never detected. Absorption efficiency is therefore a key property of every detector as it directly influences the I/σ and especially data quality of very weak reflections. The Bruker PHOTON III combines the latest mixed-mode technology with optimized X-ray scintillators. This approach optimizes X-ray absorption and signal gain, eliminates parallax effects and achieves more accurate data. The new PHOTON III HE extends this concept with a new, optimized scintillator for high energy and achieves near-ideal quantum efficiency for Mo, Ag and In.

Check out our video on X-ray absorption.

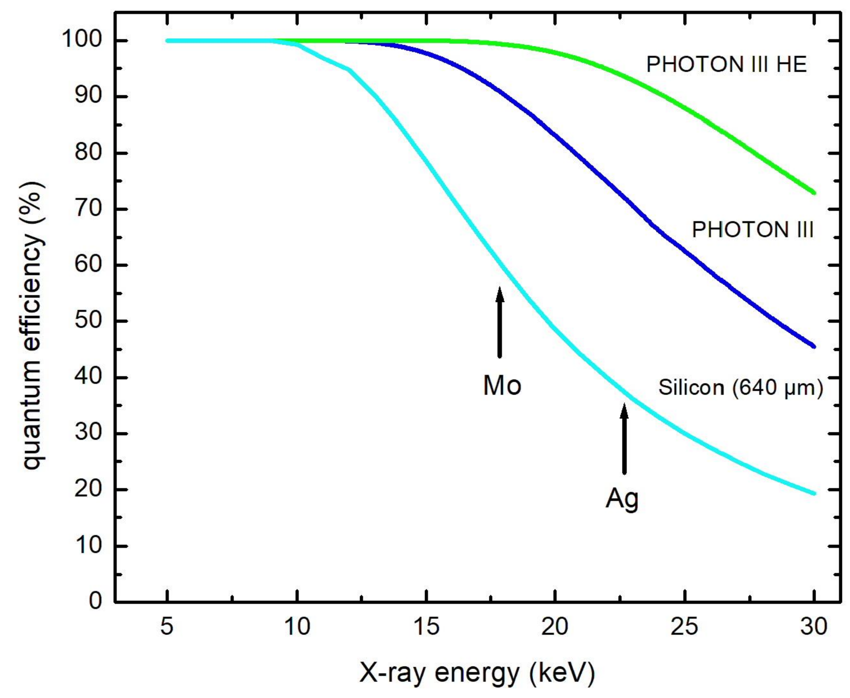


Figure 5: Energy dependent X-ray absorption for the PHOTON III detectors.

Absorber Material	Mo	Ag
Typical HPC Sensor (640mu, Si)	58 %	33%
PHOTON III Scintillator	91%	69%
PHOTON III HE Scintillator	99%	95%

Table 2: Experimentally absorption values determined by transmission experiments using Mo or Ag radiation.

PHOTON III vs. PHOTON III HE

A D8 VENTURE equipped with an Ag IµS 3.0 microfocus X-ray source and a KAPPA goniometer was used to compare measurement times and data quality from the new PHOTON III HE with the PHOTON III. A small crystal of $CaSiF_6 \cdot 2H_2O$ was measured at Ag Kα (22.5keV). The crystal unit cell parameters were a = 5.7293(3)Å, b = 9.1802(4)Å, c = 10.4773(4)Å, β = 98.947(2)°, and V = 544.36(4)Å³ with sapce group $P2_1/n$. Identical strategies were used to collect complete data on both detectors to 0.60Å.

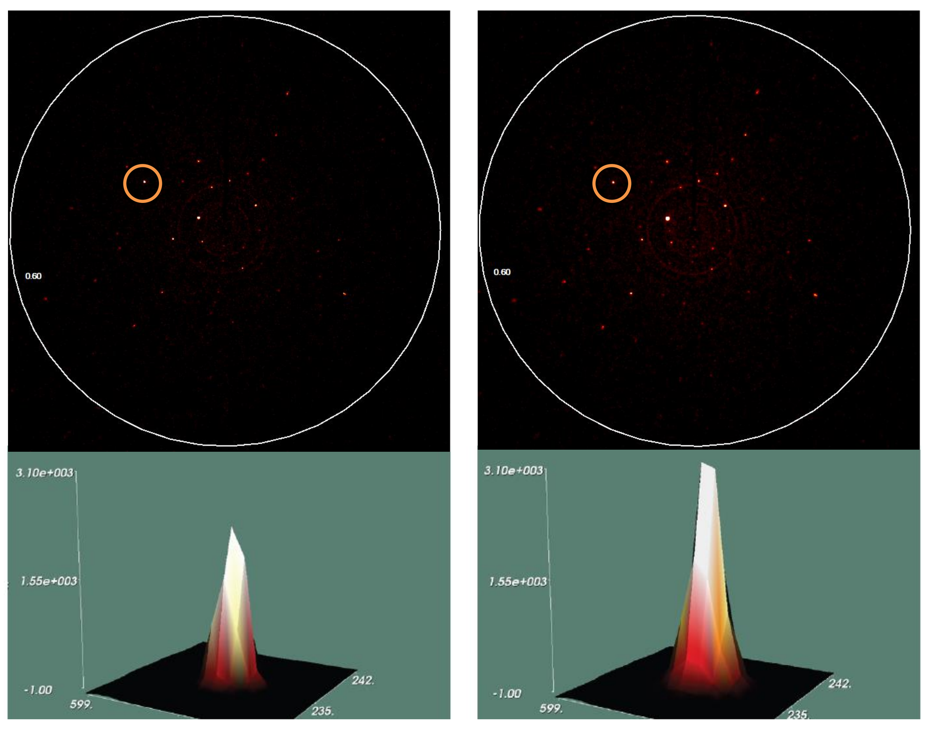


Figure 5 shows two equivalent frames recorded with the PHOTON III and the PHOTON III HE. The highlighted reflection exhibits a peak intensity 40% higher recorded with the PHOTON III HE. This is directly proportional to the higher DQE of the PHOTON III HE and nicely demonstrates the highly linear count rate and the zero noise of the PHOTON III detectors.

Figure 5: 0.6s diffraction images using Ag radiation recorded with the PHOTON III (left) and PHOTON III HE (right). The highlighted reflection (hkl -5,1,13) recorded with the PHOTON III HE has an intensity maximum 40% greater than recorded under identical conditions with the PHOTON III (insets).

	PHOTON III	PHOTON III HE
Max. Resolution [Å]	0.60	0.60
R_{int} (all/high Res)	9.54/21.69	9.18/19.33
I/σ (all/highRes)	9.25/5.01	11.36/5.81
%Obs(>2σ(F²))	80.02	82.42
*R*1 (obs/all) [%]	4.39/6.08	3.84/5.32
*wR*2 [%]	15.00	13.59
Res. el. Density [e/Å³]	1.13/-1-17	1.21/-1.16

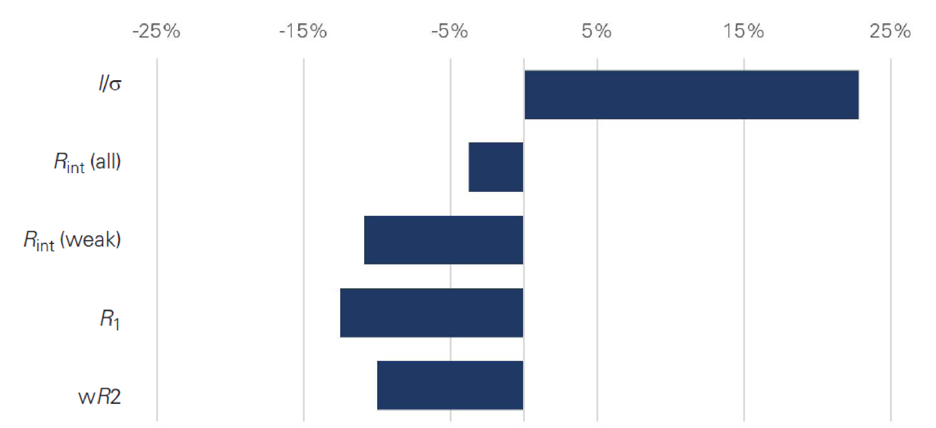
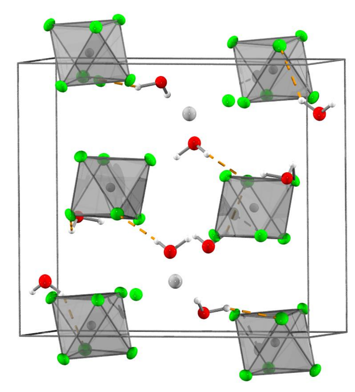


Figure 6: Data statistics and refinement results (left). Improvements in statistics when using PHOTON III HE versus. PHOTON III.(right).

Figure 6 shows the refinement parameters for the PHOTON III and PHOTON III HE data collections. The new PHOTON III HE yields appreciably better-quality data. A direct result of the higher DQE is an overall ~20% higher I/σ(I). This increase is fully consistent with the expected increase for a noise-free detector. The improvement in data quality is particular significant for the weak reflections in the highest resolution shell. The higher quality data from the PHOTON III HE consequently, results in a more accurate structure model. Structure residuals R1 and wR2 are reduced by 13% and 10% respectively.



Conclusions

- The PHOTON III combines latest mixed-mode X-ray detection with modern scintillator technology to achieve the best DQE for all conventional wavelengths.
- The PHOTON III HE is optimized for high energy applications with a DQE >90% for Mo, Ag and In radiation.
- Comparison of the PHOTON IIII and PHOTON III HE demonstrated significant data quality improvements for Ag radiation with small improvements on the Mo side.
- The PHOTON III HE is the perfect in-house X-ray detector for your high energy applications!

SC-XRD

[1] A.Mozzanic, *Synchrotron Radiati. News*, **2022**, *31*(6), 16-20.
[2] B. Henrich, *Nucl. Instrum. Methods Phys. Res. A*, **2011**, *33*, 11-14.
[3] Leonarski et al., *Nat. Methods*, **2018**, *15*, 799–804.
[4] Durst et al., *Proc. of SPIE*, **2021**, *11838*, 118380N-1.