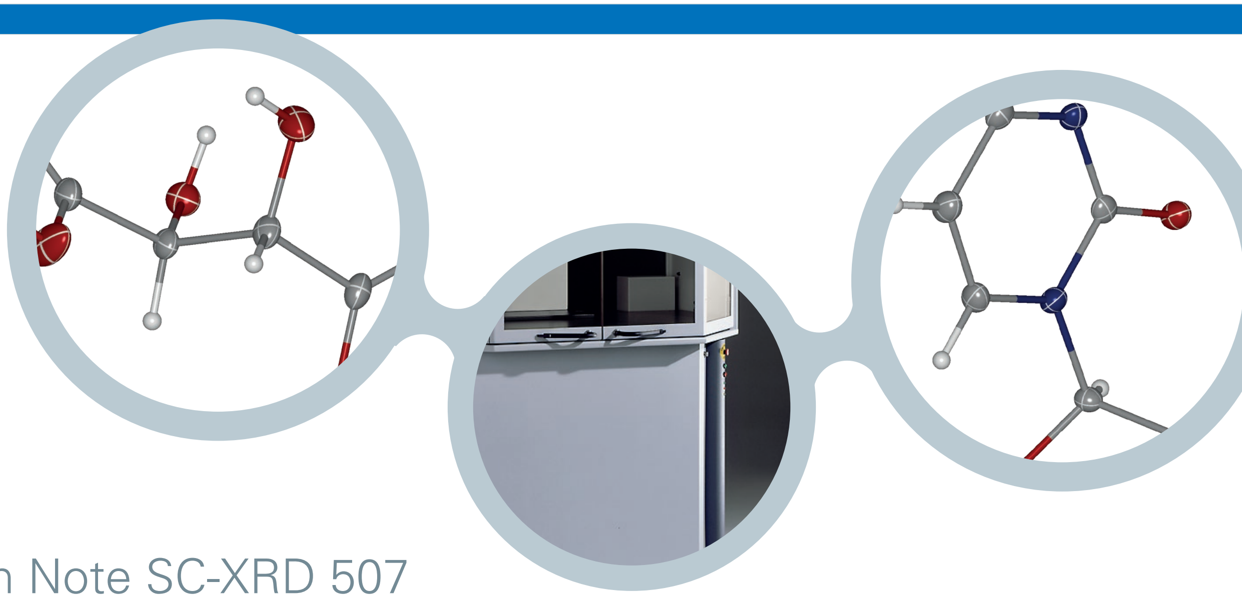

Application Note SC-XRD 507

PHOTON 100 detector upgrade for CCD systems – a structure comparison

Introduction

The PHOTON 100 CMOS APS detector has been very well received in the crystallographic community and provides superb data for both small- and macro-molecules. Its large active area, high sensitivity and unsurpassed speed using shutterless data acquisition make it the ideal detector for crystallographic applications. The PHOTON 100 was introduced with the D8 QUEST and D8 VENTURE systems, and there are more than 200 detectors installed today. The PHOTON 100 detector is available as an upgrade to replace APEX and APEX II detectors on D8 series systems.

This application note presents and compares structure data that were collected before and after upgrading a D8 series SMART APEX system, replacing an APEX detector with a PHOTON 100 detector.

Figure 1: PHOTON 100, the premier CMOS active pixel sensor detector for X-ray crystallography.

Data collection with an APEX detector

Data collection with an APEX CCD detector requires the experiment shutter to be closed during readout of a diffraction frame, as CCD detectors cannot be exposed to X-rays during readout. For each exposure, each image, the goniometer must be ramped to speed for a synchronized opening of the shutter at the beginning of the acquisition. The shutter is then closed at the end of the acquisition. While the detector is read out, the goniometer is repositioned for consecutive data collection or to collect correlated frames, which are typical for CCD detectors, where each frame is collected twice. Inherent to CCD detectors, this stopping and starting introduces mechanical jitter that is detrimental to data quality. In addition, overhead is introduced as the frame is read out, and while the goniometer is repositioned. This overhead can be as long as several seconds, and no data can be acquired during this time. As a result, for short exposure times CCD detectors are idle through most of the experiment.

Data collection with a PHOTON 100 detector

Data collection with a PHOTON 100 CMOS APS detector allows readout while the detector is exposed to X-rays. Powerful onboard electronics and software apply necessary image processing on the fly so that the PHOTON 100 writes out completely corrected frames, and allow the detector to be operated in shutterless mode, using continuous scans without the need to open and close the shutter. Data collection speed is optimized and mechanical jitter is eliminated for improved data quality and overhead-free experiments. Time savings are substantial, especially for short exposure times.

Extended dynamic range

Even during a well-planned experiment, some of the strongest reflections might overload the sensor, requiring special treatment to properly record the intensities of those reflections. Traditionally with Bruker CCD detectors, topped pixels are flagged during processing and the same frame is immediately recollected at a faster scan speed, or with an attenuator inserted, or the combination of both. Topped pixels and a few neighboring pixels then would be replaced with the scaled pixels (scaled according to the faster scan speed or attenuation factor) of the rescanned frame.

With the shutterless data acquisition of the PHOTON 100, it is impractical and inefficient to interrupt the continuous scan during data collection to recollect topped pixels. Instead, the best method to extend the dynamic range and properly record the intensities of the strongest reflections is to perform a *Fast Scan* at the beginning of the experiment. This on-axis scan ($2\vartheta = 0°$) covers a hemisphere of data and is typically collected in 1s/1° frames in only three minutes. This fast scan provides data for crystal screening, initial indexing, and allows for determination of experimental parameters, such as scan width and exposure time. This quick data collection is very often already of sufficient quality for structure solution.

During data processing, this Fast Scan is used to provide intensity data for the strongest reflections that are topped during data collection, after careful scaling of all data runs and Fast Scan(s). The remaining weaker reflections from the Fast Scan are typically discarded for the final structure determination.

Data collection

All data were collected using a D8 series goniometer and a normal fine-focus molybdenum X-ray tube at 1500 Watts coupled to a flat graphite monochromator. APEX data were acquired with a Bruker standard hemisphere strategy covering 360° of data in three omega scans with offsets in phi between the scans. The PHOTON 100 data for comparison were collected in a similar fashion using several runs of omega scans in addition to a Fast Scan. For a Cytidine sample comparison the overall experiment time was kept constant. PHOTON 100 data were either collected with the same exposure time, or with longer exposure time using similar experiment time. In case of a strongly diffracting tartaric acid sample, PHOTON 100 data were acquired with the same exposure time in about half the experiment time or with the same exposure time in the same experiment time.

Conclusion

The PHOTON 100 CMOS APS detector is an exciting replacement for the APEX family of detectors, extending the life of durable, reliable instruments into the next generation of X-ray detectors. The upgrade offers unrivaled data collection speed and a larger active area, which translates to more data, faster. The nearly threefold increase in active area dramatically reduces the number of images required for a given resolution, and simultaneously increases the multiplicity of the data collected. With shutterless operation and consequently no overhead, data collection times can drop by a factor of two or more. Decreased data collection time allows for collection of more redundant data using similar or longer exposure times to improve overall data quality in the same total experiment time. The ability to collect routine data faster and more efficiently gives time to spend examining the more challenging samples that come into the laboratory.

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Tartaric Acid

Detector	APEX	PHOTON 100	
Total time	2 hr 58 min	1 hr 29 min	2 hr 56 min
Multiplicity	2.49	3.73	7.40
*R*int [%]	3.60	3.22	3.31
*R*sig [%]	3.13	3.31	2.49
*R*1 [%]	2.99	2.69	2.53
w*R*2 [%]	7.43	6.61	6.36

Table 1. Selected experimental parameters for Tartaric acid.

The Tartaric acid sample used for the comparison was a well diffracting crystal and its data were collected with short exposure times. Exploiting the larger detection area and shutterless operation of the PHOTON 100, it is possible to achieve 50% higher multiplicity in less experiment time. Taking the same time to complete the experiment allows for a tremendous 3-fold increase in multiplicity, compared to the APEX detector data. The additional observations help to improve data quality which is impressively reflected in the values for the agreement factors *R*1 and w*R*2. From Table 1, it is clear that PHOTON 100 is an exciting replacement for the APEX detector.

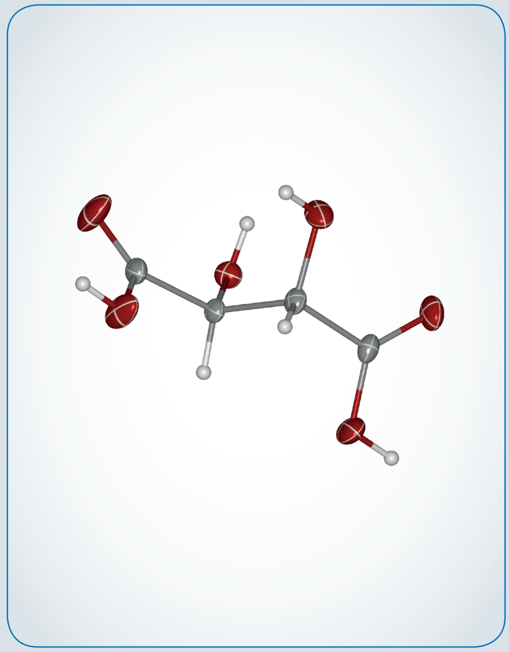

Figure 2: Molecular structure of Tartaric acid.

Cytidine

Detector	APEX	PHOTON 100
Exposure time	15 s/°	30 s/°
Total time	3 hr 25 min	3 hr 13 min
Overhead time	1 hr 39 min	1 min
Multiplicity	3.40	5.79
*R*int [%]	2.41	2.70
*R*sig [%]	2.00	2.09
*R*1 [%]	3.14	2.99
w*R*2 [%]	8.36	7.54

Table 2: Selected experimental parameters for Cytidine.

For a second comparison we have chosen a cytidine crystal, which is a more challenging sample as it diffracts very weakly. Here the PHOTON 100 is capable of collecting longer exposures in the same overall experiment time, as compared to the APEX detector. The data collection performed using the APEX detector required a total elapsed time of almost 3½ hours for a total exposure time of 1¾ hours. The PHOTON 100 on the other hand collects data free of overhead and the remaining 1¾ hours of experiment time can be utilized to collect better data by increasing the exposure time, and almost doubling the multiplicity of observations. PHOTON 100 data provide better overall structural results, compared to the APEX detector, Table 2.

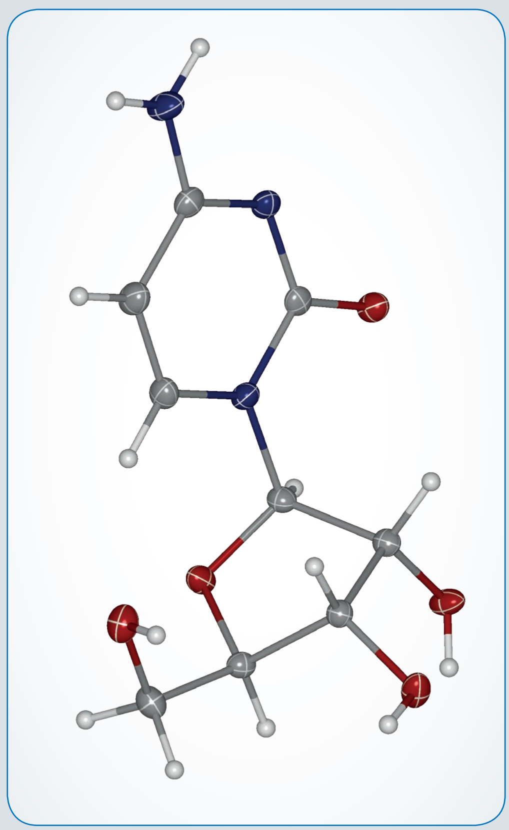

Figure 3: Molecular structure of Cytidine.

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