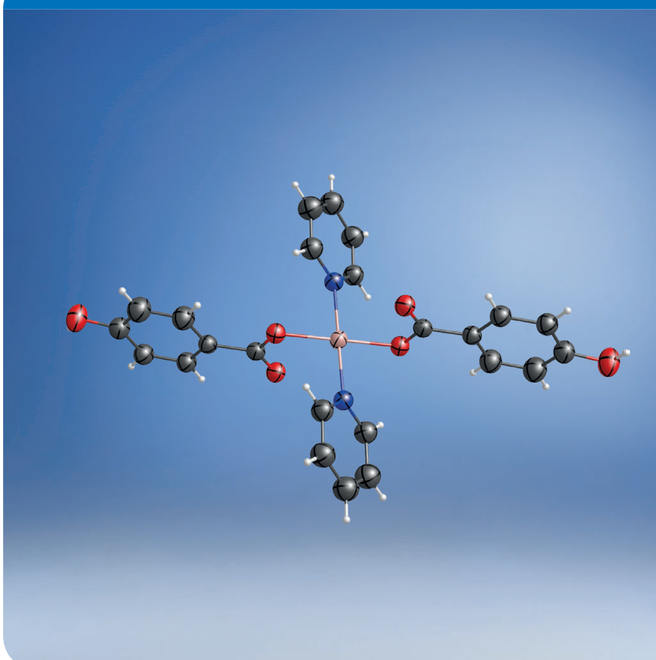


## Application Note SC-XRD 521

# PHOTON II Detector Upgrade

- Keeping Your D8 QUEST or D8 VENTURE at the Forefront of Technology

### Introduction

Starting in 1997, CCD detectors, such as the APEX II, were considered for many years to be state-of-the-art technology in Single Crystal X-ray Diffraction (SC-XRD). Research sites all over the world are still successfully using SMART APEX, KAPPA APEX and X8 PROTEUM systems.

Today, however, charge integrating pixel array detectors (CPAD) represent the state of the art, are in use at a small number of 4th generation XFEL synchrotron beam lines, or are under development for these. Since the end of 2015 Bruker is providing the PHOTON II, an X-ray detector following the very same design concept for in-house crystallography. Fortunately, this detector—featuring a large 10×14 $cm^2$ active area—is also available as an upgrade for existing D8 QUEST and D8 VENTURE systems.

## One-to-one comparison data sets

This application note presents a series of comparisons obtained on D8 QUEST and D8 VENTURE systems, each equipped with a PHOTON II detector and an IµS 3.0 microfocus source, and relates these data sets to data sets from the same systems equipped with a PHOTON 100 detector. Test crystals representing typical samples in service crystallography were chosen to demonstrate the superior performance of the PHOTON II in terms of spatial resolution, data collection speed and data quality. All data sets were processed with the Bruker APEX3 software suite which is included with all D8 QUEST and D8 VENTURE systems.

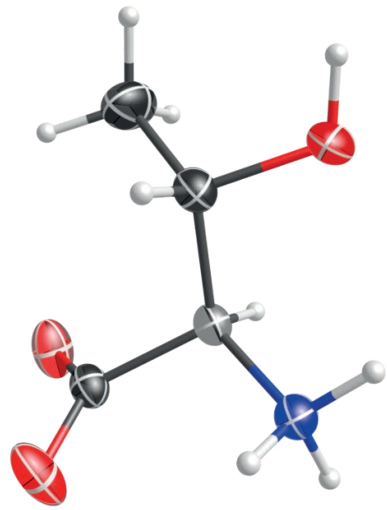

Figure 1: Crystal structure of D-Threonine, $C_4H_9NO_3$.

## D-Threonine, $C_4H_9NO_3$

Data from a relatively small threonine crystal ($0.14 \times 0.12 \times 0.11$ mm$^3$) was acquired using copper (Cu-)K$\alpha$ radiation at a temperature of 100 K. In order to compare detector active areas, the shortest strategy possible was calculated for both setups giving 100% complete data up to 0.84 Å. Exposure times of 4 s for low 2theta angles and 20 s for high 2theta angles were chosen together with a frame width of 2° to give an equal number of frames for both detectors. In total, 963 independent reflections were collected on 786 frames.

Due to the PHOTON II's 40% larger active detector area, in general fewer 2theta runs are necessary to obtain complete data, reducing the total experiment time in this case by almost 10%. Another direct consequence of the bigger PHOTON II is significantly higher data multiplicity (redundancy).

Furthermore, the distinctly improved *R1* and *wR2* values already reflect the superior data quality of the PHOTON II compared to the PHOTON 100. This all goes along with an improved Flack parameter, allowing the assignment of the absolute structure with highest confidence. Note that D-Threonine is an all-light-atom compound with period 2 elements only. The new CPAD detector generation routinely allows absolute structure determination of these compound types, a challenge for older systems for many years.

| | PHOTON 100 | PHOTON II |
|---|---|---|
| Total time [min] | **208** | **190** |
| Exposure/frame | 4/20 s | 4/20 s |
| Frame width [°] | 2 | 2 |
| No of frames | 786 | 786 |
| Redundancy | 4.9 | 5.7 |
| $I/\sigma$ | 37.9 | 54.7 |
| $R_{int}$ [%] | **3.3** | **2.6** |
| *R1* [%] | **2.6** | **2.3** |
| *wR2* [%] | 6.6 | 6.3 |
| Flack | **-0.13(1)** | **-0.05(8)** |
| Residual density [eÅ$^{-3}$] | 0.1/-0.1 | 0.1/-0.1 |

Table 1: Data collection and refinement parameters of D-Threonine.

## Cu Complex

Data of the metal organic Cu complex $[Cu(C_5H_5N)_2(C_7H_6O_3)_2]$ ($0.19 \times 0.11 \times 0.08$ mm$^3$) were acquired using Cu-K$\alpha$ radiation at 300 K. From both detector setups—a system equipped with a PHOTON 100 and another system equipped with a PHOTON II—fast scan measurements were used to determine an optimized data collection strategy leading to 100% data completeness up to a resolution of 0.84 Å. The superior sensitivity of the PHOTON II allowed for the collection of high quality data at high 2theta even with half of the exposure, reducing the total experiment time by more than 40%. As before, the larger active area of the PHOTON II inherently leads to higher data multiplicity.

## Double your productivity: Same quality data in half of the time

As shown in Table 2, both data sets, collected with the PHOTON 100 and the PHOTON II, respectively, result in almost identical quality factors (*R*-values) for the crystallographic model. The significant difference between the two data sets is the exposure time, reduced by as much as 50% for the PHOTON II data set, highlighting the high detection speed, accuracy and sensitivity of this detector. The example nicely demonstrates the gain in time for a given sample or, as the flip side of the coin, the possibility to measure weaker samples on a D8 QUEST or D8 VENTURE system equipped with the new PHOTON II detector.

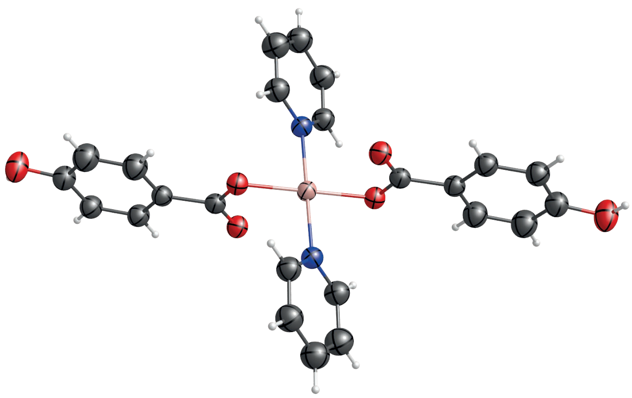

Figure 2: Crystal structure of $Cu(C_5H_5N)_2(C_7H_6O_3)_2$.

| | PHOTON 100 | PHOTON II |
|---|---|---|
| Total time [min] | **135** | **80** |
| Exposure/frame | 8/2 s | 4/2 s |
| Frame width [°] | 2° | 2° |
| No of frames | 1212 | 1236 |
| Redundancy | 7.2 | 8.8 |
| $I/\sigma$ | 40.0 | 45.0 |
| $R_{int}$ [%] | 3.1 | 2.9 |
| *R1* [%] | 2.9 | 2.8 |
| *wR2* [%] | 7.7 | 7.9 |
| Residual density [$eÅ^{-3}$] | 0.3/-0.3 | 0.3/-0.3 |

Table 2: Data collection and refinement parameters of $Cu(C_5H_5N)_2(C_7H_6O_3)_2$.

## Paracetamol, $C_8H_{10}NO_2$

To test the PHOTON II's performance with hard radiation, data of a small, weakly diffracting paracetamol crystal [$C_8H_{10}NO_2$](0.03 × 0.05 × 0.05 $mm^3$) were acquired using molybdenum (Mo) K$\alpha$ radiation at 100 K. Based on fast scan measurements, similar data acquisition strategies were calculated using equal detector distances, with the objective for 100% data completeness up to 0.83 Å, same data collection time, frame width, exposure time per frame and equal multiplicity for both data sets (Table 3). Applying the constraint of equal multiplicity, the advantages of a bigger detector are deliberately eliminated, allowing a direct comparison of the detection accuracies. This direct comparison of data quality (Table 3) impressively demonstrates the superior performance of the PHOTON II: 50% increase of $I/\sigma(I)$ as well as significant improvement of the residual values $R_{int}$, *R1* and *wR2*.

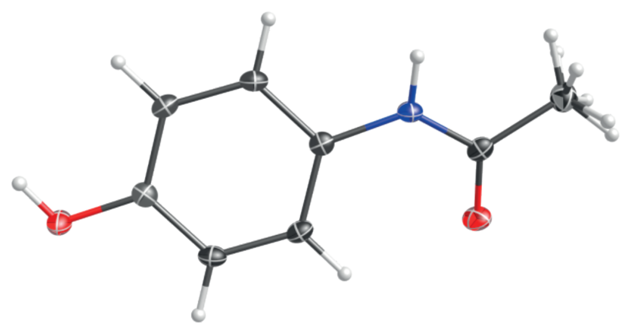

Figure 3: Crystal structure of paracetamol, $C_8H_{10}NO_2$.

| | PHOTON 100 | PHOTON II |
|---|---|---|
| Total time [h] | 5 | 5 |
| Exposure/frame | 35 s | 35 s |
| Frame width [°] | 0.5° | 0.5° |
| No of frames | 517 | 517 |
| Redundancy | 4.7 | 4.8 |
| $I/\sigma(I)$ | 22.0 | 34.1 |
| $R_{int}$ [%] | **4.2** | **2.5** |
| *R1* [%] | **4.0** | **3.2** |
| *wR2* [%] | 9.9% | 7.8% |
| Residual density [$eÅ^{-3}$] | 0.2/-0.3 | 0.2/-0.2 |

Table 3: Data collection and refinement parameters of paracetamol.

### *Cis*-Pt-derivative, $Pt(C_6H_7O_4)(NH_3)_2$

Data of a tiny, heavily absorbing *cis*-Pt complex crystal $[Pt(C_6H_7O_4)(NH_3)_2]$ (0.07 × 0.07 × 0.09 mm³) were acquired using Mo-Kα radiation at 100 K. The linear absorption coefficient of this compound is 15.9 $mm^{-1}$ for Mo with an estimated minimum and maximum transmission of 0.328 and 0.420. Both datasets were collected in a very short overall time, up to a resolution of 0.84 Å, again employing identical strategies with a frame width of 2°/frame and 2 s exposure/frame. Besides the increased multiplicity, the superior data quality of the PHOTON II is impressively reflected by the increased $I/\sigma(I)$, distinctly reduced residual values and residual electron density around the heavy metal atom Pt (Table 4).

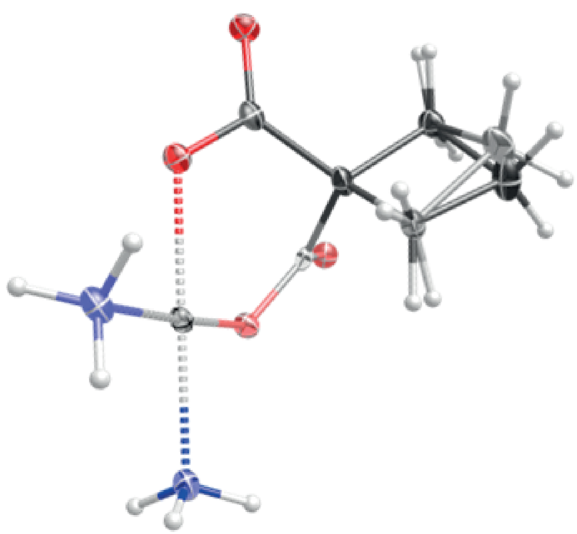

Figure 4: Crystal structure of *cis*-Pt-derivative, $Pt(C_6H_7O_4)(NH_3)_2$.

| | PHOTON 100 | PHOTON II |
|---|---|---|
| Total time [min] | 4.2 | 4.2 |
| Exposure/frame | 2 s | 2 s |
| Frame width [°] | 2 | 2 |
| Redundancy | 6.4 | 8.5 |
| $I/\sigma(I)$ | 21.8 | 34.7 |
| $R_{int}$ [%] | 6.0% | 3.5% |
| *R1* [%] | 1.9% | 1.1% |
| *wR2* [%] | 3.5% | 2.7% |
| Residual density [eÅ$^{-3}$] | 0.9/-1.1 | 0.5/-0.4 |

Table 4: Data collection and refinement parameters of *cis*-Pt-derivative, $Pt(C_6H_7O_4)(NH_3)_2$.

## Conclusion

Compared to the PHOTON 100 detector, the PHOTON II systematically provides same quality data sets in significantly shorter time or better data quality in the same time. This has been demonstrated on a variety of samples for both typical in-house wavelengths, Mo-Kα and Cu-Kα radiation. D8 QUEST and D8 VENTURE systems can be easily upgraded to the new PHOTON II CPAD detector technology, keeping the system with reasonable efforts at the forefront of technology.

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