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# SMX036 - Comparison of two XtaLAB Synergy instruments

## Introduction

Many research and service crystallography laboratories use single crystal diffractometers equipped with two different wavelengths. Typically, CuK $\alpha$  and MoK $\alpha$  are used but also combinations with AgK $\alpha$  are becoming popular. The choice of wavelength for a diffraction experiment is always a tradeoff between intensity, resolution and absorption for sample at hand. Rigaku Oxford Diffraction offers a unique rotating anode system, featuring two wavelengths to overcome these tradeoffs. The intensity gain between a microfocus sealed tube instrument and a rotating anode is in the range of a factor 10-12, depending on the wavelength. By curving the detector around the sample, higher coverage of reciprocal space can be achieved. Additionally, reflections hit the sensor of the detector with a lower obliqueness, thus creating sharper and better-defined spots. Fewer corrections in the data processing are required, therefore the overall data quality is improved.

This application note compares two differently configured XtaLAB Synergy instruments, (Figure 1) covering the middle to high range of single crystal diffraction equipment.



**Figure 1:** (a) XtaLAB Synergy-S, equipped with a HyPix-6000HE and (b) XtaLAB Synergy-DW, equipped with an Arc 150° detector. Low temperature devices are omitted for clarity.

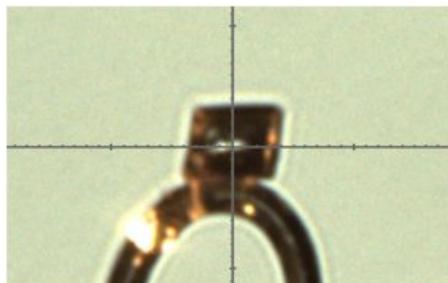
## Experimental

Each instrument is equipped with CuK $\alpha$  and MoK $\alpha$  wavelengths, which will be tested using carefully selected crystals of cytidine and PdCl<sub>2</sub>(dppf) as examples for a small, organic, chiral molecule and a common metal-organic complex (dppf: 1,1'-bis(diphenylphosphino)ferrocene). For comparison, both datasets aimed for an equal redundancy and intensity of data. The measurements were conducted at 100 K, using Oxford Cryostream 800 series cryo-coolers. The crystals were kept frozen under liquid nitrogen between the measurements to prevent sample degradation.

## Results

### Cytidine

A colorless block-shaped crystal was cut from a larger block to a size of 0.063 x 0.064 x 0.087 mm, see Figure 2.



**Figure 2:** Photo of the cytidine crystal used for comparison.

The number of measured frames reflects the influence of detector size: the HyPix-Arc 150° dataset needed around 20% fewer frames than the HyPix-6000HE (7570 vs 6106). Due to the size of the detector, the HyPix-6000HE dataset was collected in two principal theta positions, whilst the HyPix-Arc 150° dataset could be collected in a single principal theta position, see Table 1.

**Table 1:** Comparison results for the cytidine crystal, Cu-K $\alpha$ .

Instrument Setup	(a)	(b)
<b>Experimental</b>		
Runs / Frames	60/7570	44/6106
Scan width [°]	0.5	0.5
Exposure time [s]	0.29 / 1.16	0.05
Total experiment time [hh:mm:ss]	02:06:07	00:17:07
<b>Data quality (inf - 0.84 Å)</b>		
Completeness [%]	100	100
Redundancy	15.7	15.6
I/ $\sigma$ (I)	104.6	122.0
R <sub>int</sub> [%]	3.21	2.45
R <sub>1</sub> [%]	2.20	2.36
wR <sub>2</sub> [%]	5.84	6.23
Flack [x]	0.01(3)	0.03(4)

Due to the high photon flux of the rotating anode, even the high angle reflections could be measured at 0.05 s/0.5°, resulting in a performance increase of factor 23.2 (exposure time 1.16 s vs 0.05 s per frame). Comparing the I/ $\sigma$ (I) of the highest resolution shells, (Tables 2 & 3) equally intense data was collected (47.78 vs 47.24). The overall redundancy

distribution over all resolution shells differs, which is due to the size difference between the detectors: While measuring complete data on high resolution shells, redundancy on inner shells is collected as a by-product in the case of HyPix-Arc 150°.

**Table 2:** Statistics vs. Resolution (taking redundancy into account) – point group symmetry P222: XtaLAB Synergy-S with HyPix-6000HE.

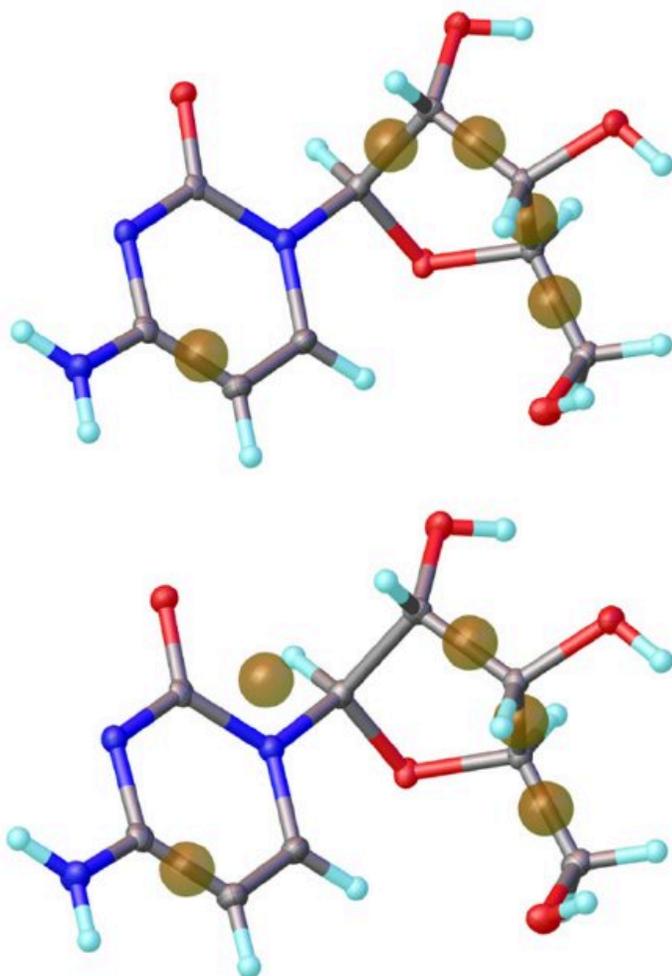
Resolution (Å)	# measured	Completeness	Redundancy	Mean F <sup>2</sup>	Mean F <sup>2</sup> /σ(F <sup>2</sup> )	R <sub>int</sub>	R <sub>pim</sub>	CC 1/2	$ F_+^2 - F_-^2 /\sigma$
inf- 1.84	2558	100	13.8	70097.62	109.70	0.027	0.008	1.000	1.74
1.84- 1.45	3952	100	21.2	30454.53	108.31	0.030	0.007	1.000	1.89
1.45- 1.25	4396	100	23.6	18519.33	95.05	0.036	0.007	1.000	1.79
1.25- 1.14	4256	100	22.9	19496.39	88.69	0.037	0.008	1.000	2.11
1.14- 1.06	3817	100	20.5	19149.88	84.94	0.036	0.008	1.000	1.89
1.06- 0.99	3018	100	16.2	13157.62	72.33	0.040	0.010	0.999	2.27
0.99- 0.95	2264	100	12.2	8662.95	64.92	0.038	0.011	0.999	1.96
0.95- 0.91	2090	100	11.2	6679.30	55.43	0.041	0.013	0.999	1.93
0.91- 0.87	1710	100	9.2	7250.33	55.36	0.038	0.013	0.999	1.91
0.87- 0.84	1438	99.5	7.5	4853.98	47.78	0.044	0.017	0.999	1.81
inf- 0.84	29499	99.9	15.8	21350.19	83.82	0.033	0.008	1.000	1.94

**Table 3:** Statistics vs. Resolution (taking redundancy into account) – point group symmetry P222: XtaLAB Synergy-DW with HyPix-Arc 150°.

Resolution (Å)	# measured	Completeness	Redundancy	Mean F <sup>2</sup>	Mean F <sup>2</sup> /σ(F <sup>2</sup> )	R <sub>int</sub>	R <sub>pim</sub>	CC 1/2	$ F_+^2 - F_-^2 /\sigma$
inf- 1.84	4815	100	25.9	864626.16	210.59	0.019	0.004	1.000	2.75
1.84- 1.45	3785	100	20.3	350178.21	126.28	0.023	0.005	1.000	1.81
1.45- 1.25	3777	100	20.3	211852.79	99.84	0.028	0.006	1.000	1.80
1.25- 1.14	3401	100	18.3	224944.24	89.24	0.028	0.007	1.000	1.86
1.14- 1.06	2844	100	15.3	220427.33	80.88	0.030	0.008	1.000	1.75
1.06- 0.99	2010	100	10.8	144638.65	61.02	0.036	0.011	0.999	1.76
0.99- 0.95	2038	100	11.0	98121.76	50.99	0.040	0.012	0.999	1.74
0.95- 0.91	2451	100	13.2	78070.12	51.71	0.043	0.012	0.999	1.57
0.91- 0.87	2205	100	11.9	78893.06	52.69	0.043	0.013	0.999	1.83
0.87- 0.84	1855	99.5	9.6	56277.09	47.24	0.047	0.015	0.999	1.89

inf- 0.84	29181	99.9	15.6	296120.66	101.43	0.025	0.006	1.000	1.85
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The refined crystal structures are very similar in quality ( $R_1 = 2.20\%$  vs  $2.36\%$ ). Within one standard uncertainty, the Flack-parameter is equal in both measurements (Flack =  $0.01(3)$  vs  $0.03(4)$ ). More internally consistent data was obtained from the HyPix-Arc  $150^\circ$  detector ( $R_{int} = 3.21\%$  vs.  $2.45\%$ ). A significantly lower  $R_{int}$  could be obtained because more symmetry equivalent reflections could be measured in the same frame, thus the scaling of data was improved. The residual electron density is found between the atoms in the covalent bonds, indicating a very high quality structure, see Figure 3.



**Figure 3:** Refined crystal structure models of cytidine, measured on a XtaLAB Synergy-S/HyPix-6000HE (top) and a XtaLAB Synergy-DW/HyPix-Arc  $150^\circ$  (bottom).

#### **PdCl<sub>2</sub>(dppf)**

A plate-like bright orange crystal of size  $0.018 \times 0.039 \times 0.110$  mm was chosen for the comparison measurements with MoK $\alpha$  radiation, see Figure 4.

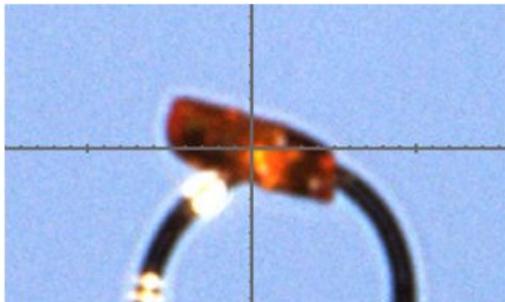
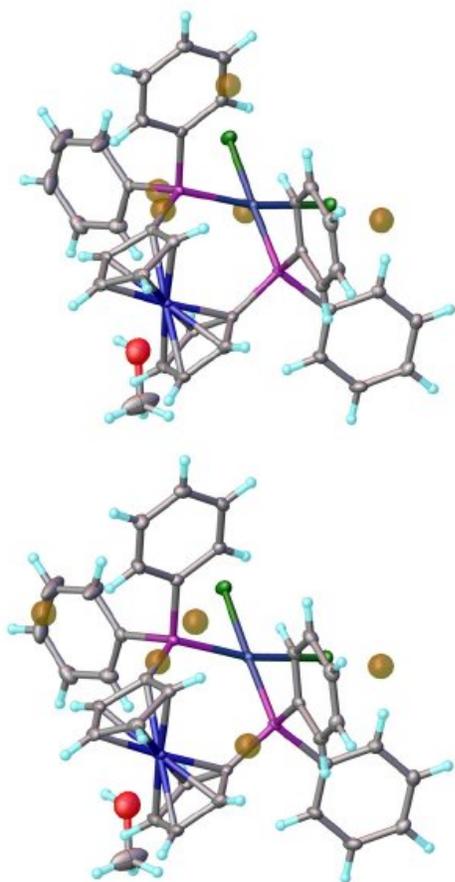


Figure 4. Photo of the PdCl<sub>2</sub>(dppf) crystal used for comparison.

Data collection times lasted over 15 hours on the sealed tube instrument, whereas the measurement on the rotating anode finished in little over 1 hour. With a nearly equal number of frames (1256 vs 1294), the difference in exposure time, 45 s vs. 3 s, shows the performance increase of a factor 15 between the system (a) and (b), see Table 4.

Both data sets allowed for excellent refined crystal structures. The  $I/\sigma(I)$  as well as the  $R_{int}$  are slightly stronger in the sealed tube dataset, which is due to the exposure time. Ultimately the rotating anode dataset yielded an equally well refined structure model within margin:  $R_1 = 3.09\%$  vs.  $3.15\%$ . Both structure models show residual electron density on the chemical bonds. A half molecule of MeOH could be refined without constraints on a special position, see Figure 5.



**Figure 5:** Refined crystal structures of PdCl<sub>2</sub>(dppf), measured on a XtaLAB Synergy-S/HyPix-6000HE (top) and a XtaLAB Synergy-DW/HyPix-Arc 150° (bottom).

Tables 5 and 6 show statistics for both datasets. The dataset measured with the HyPix-Arc 150° shows a different redundancy distribution than the dataset measured with the HyPix-6000HE like in the Cu dataset of cytidine.

**Table 5:** Statistics vs. Resolution (taking redundancy into account) – point group symmetry P1: XtaLAB Synergy-S with HyPix-6000HE.

Resolution (Å)	# measured	Completeness	Redundancy	Mean F <sup>2</sup>	Mean F <sup>2</sup> /σ(F <sup>2</sup> )	R <sub>int</sub>	R <sub>pim</sub>	CC 1/2
inf- 1.72	2622	98.7	4.3	253.07	70.07	0.013	0.007	1.000
1.72- 1.37	3112	100	5.0	159.58	57.21	0.019	0.009	1.000
1.37- 1.20	3330	100	5.4	98.30	44.76	0.028	0.013	0.999
1.20- 1.09	3398	100	5.5	87.13	40.70	0.031	0.014	0.999
1.09- 1.01	3416	100	5.5	83.50	38.59	0.035	0.016	0.999
1.01- 0.95	3403	100	5.5	70.05	33.28	0.040	0.019	0.999
0.95- 0.90	3474	100	5.6	54.34	28.26	0.050	0.023	0.999
0.90- 0.86	3260	100	5.2	49.60	25.78	0.057	0.027	0.998
0.86- 0.83	2793	99.8	4.5	40.68	20.51	0.066	0.034	0.996
0.83- 0.80	2362	99.8	3.8	38.08	18.14	0.070	0.040	0.995
inf- 0.80	31170	99.8	5.0	91.79	37.75	0.031	0.015	1.000

**Table 6:** Statistics vs. Resolution (taking redundancy into account) – point group symmetry P1: XtaLAB Synergy-DW with HyPix-Arc-150°.

Resolution (Å)	# measured	Completeness	Redundancy	Mean F <sup>2</sup>	Mean F <sup>2</sup> /σ(F <sup>2</sup> )	R <sub>int</sub>	R <sub>pim</sub>	CC 1/2
inf- 1.72	3685	97.4	6.1	2986.57	73.30	0.015	0.006	1.000
1.72- 1.37	3118	100	5.0	1876.14	53.70	0.020	0.010	1.000
1.37- 1.20	3300	100	5.3	1164.19	40.13	0.030	0.014	0.999
1.20- 1.09	3369	100	5.4	994.99	35.27	0.038	0.018	0.999
1.09- 1.01	3377	100	5.5	940.86	32.94	0.043	0.020	0.999
1.01- 0.95	3242	100	5.2	773.08	26.93	0.050	0.024	0.998
0.95- 0.90	3090	100	5.0	601.57	21.28	0.059	0.029	0.998
0.90- 0.86	2813	100	4.5	507.62	17.37	0.068	0.036	0.997
0.86- 0.83	2679	100	4.3	439.30	15.14	0.078	0.042	0.996
0.83- 0.80	2403	100	3.9	415.15	13.15	0.079	0.046	0.994

inf- 0.80	31076	99.7	5.0	1132.52	34.57	0.033	0.016	1.000
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## Summary

Both instruments are great tools to analyze small crystals of organic and metal-organic molecules. The gain in performance between the medium range instrument XtaLAB Synergy-S with HyPix-6000HE and the higher range instrument XtaLAB Synergy-DW with HyPix-Arc 150° is in an overall range 10-15. Laboratories with small crystals or needing high throughput will benefit from an intense rotating anode source and a large, curved detector.

## Related products



### HyPix-6000HE

Extremely low noise detector based on direct X-ray detection technology.



### HyPix-Arc 150°

A curved detector based on direct X-ray detection technology with the highest  $2\theta$  range at a single position available for the home lab.



### XtaLAB Synergy-DW

Spectacular performance combined with dual wavelength versatility, provides the perfect answer for high-capacity Chemical Crystallography labs or for X-ray facilities that support Chemical Crystallography and Protein Crystallography.



### XtaLAB Synergy-S

Our most popular diffractometer for Chemical Crystallography and Mineralogy, configured with either single or dual microfocus sealed tube X-ray sources and an extremely low noise direct X-ray detection detector.