Rigaku oxford diffraction

Superior data quality achieved with the XtaLAB Synergy-i, micro-focus Mo source

Introduction

Mo Kα radiation is ideal for evaluating large crystal samples, crystals that contain several heavy atoms and crystal samples that are densely packed with heavy atoms. Some examples of such crystals include: minerals, metal clusters or other inorganic materials like perovskites. By studying these sample types using Mo Kα radiation it is possible to reduce absorption effects and achieve better data quality overall. In addition to studying highly absorbing samples it is also possible to obtain high resolution datasets using Mo Kα radiation and perform charge density measurements. Through sophisticated charge density refinements it is possible to determine the bond connectivity and electron placement for a particular crystal structure in more detail.

The XtaLAB Synergy-i (Fig. 1) is a cutting-edge diffractometer equipped with the latest technology detector, optimized for its small pixel size and sensitivity, and can be configured with up to two bright micro-focus sealed tube sources. The Mo micro-focus source has its own benefits and a typical data collection on an organometallic crystal is discussed herein.

Figure 1. The XtaLAB Synergy-i

Experimental

An organometallic complex containing a first row transition element was mounted on a nylon loop (Fig. 2) and a standard dataset for publication purposes was collected. The data collection details are shown in Table 1.

Table 1: Data collection details.

Crystal-to-detector distance	40 mm
Exposure Time	10 seconds
Total Collection Time	1 hour 48 minutes
Scan width / No. of frames	0.5 ° / 640
Completeness	99.9 % (0.83 Å)
Average I/σ(I)	40.0

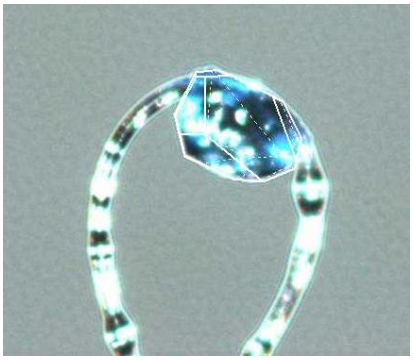

Figure 2. Face-indexed organometallic crystal (0.26 × 0.18 × 0.15 mm) .

Data collection results

Figure 3, illustrates a typical diffraction pattern measured on the XtaLAB Synergy-i with the HyPix-Bantam detector. High intensity reflections can be detected beyond a resolution of 0.83 Å.

The data was processed using the CrysAlis^{Pro} software package, which produced an excellent R_{int} value. CrysAlis^{Pro} also contains powerful, easy-to-use tools for face-indexing which makes it easy to apply face-indexed absorption correction models to every set of data collected using this instrument.

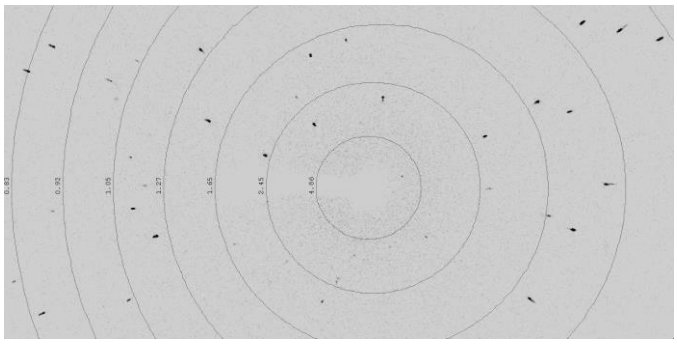

Figure 3. Diffraction image.

Table 2: Crystal data and refinement statistics.

Chemical Formula	$M_2Cl_3C_{12}H_{20}O_{22}$
Space group	Monoclinic, $P2_1/c$
a (Å), α (°) *b* (Å), β (°) *c* (Å), γ (°)	10.103(1), 90 10.8119(3), 142.77(3) 18.237(2), 90
V (Å^3) / Z	1205.3(8) / 2
R_{int} [merged data]	2.17 %
R_1 [merged data]	3.51 %
Largest residual peak/hole	0.44 / -0.75

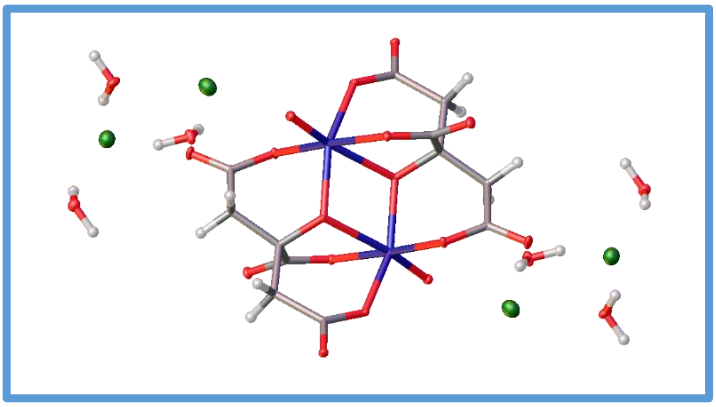

Figure 4. Organometallic structure.

Conclusion

Publishable results were obtained from this sample in a reasonable time frame and this can be attributed to the cutting-edge technology of the of the XtaLAB Synergy-i. The high flux of the Mo micro-focus source, the true digital photon counting technology of the detector, the efficient 4-circle Kappa goniometer and the all-important CrysAlis^{Pro} software are all responsible for the superior data quality.

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