APPLICATION NOTE

Rigaku oxford diffraction

The Universal Goniometer: Complete Data Sets on All Crystals, No Compromise

Introduction

Based on the Universal Goniometer, the Rigaku Oxford Diffraction (ROD) XtaLAB Synergy X-ray diffractometers represent new and innovative tools for X-ray diffraction analysis in the home laboratory. The series include a single/dual wavelength microfocus sealed tube (the XtaLAB Synergy-S), and a microfocus rotating anode (the single wavelength XtaLAB Synergy-R and dual wavelength XtaLAB Synergy-DW). The central component for all ROD systems is the Universal Goniometer, a flexible and efficient full 4-circle kappa goniometer featuring the following:

- ω rotation speed of up to 10°/sec
- ω rotation both in clockwise and counterclockwise directions to minimize goniometer repositioning time in between scans
- Minimum detector distance of 32 mm and maximum 2θ position of 107° to capture the highest resolution diffraction reflections
- Telescopic 2θ arm allowing for a maximum detector distance of 250 mm for large unit cell dimensions
- 2θ and κ motion in both positive and negative directions and a κ range of +/-179° for the shortest data collection strategies

To demonstrate the flexibility and high performance of the Universal Goniometer, we present here the results from data collected on a small crystal of chlorothiazide crystallized in space group P1.

Experimental Overview

A small crystal of chlorothiazide, with dimensions 0.019 x 0.035 x 0.046 mm, was captured within a thin layer of paratone oil inside a fiber loop mounted on a magnetic base (Figure 1). The crystal was mounted on the Universal Goniometer of a microfocus sealed tube XtaLAB Synergy-S X-ray diffractometer and data were collected at 100 K using ROD software, CrysAlis^{Pro} [1].

Table 1: XtaLAB Synergy-S specifications.

X-ray source	PhotonJet-S Cu source with continuously variable divergence slit Beam FWHM = 110 µm
Operating power	50 kV x 1 mA = 50 W
Goniometer **Detector distance range**	4-circle Kappa with telescoping 2Theta arm 32 –250 mm
Detector **Active area** **Frame rate** **Readout speed** **Pixel size** **Cooling**	Hybrid photon counting HyPix-6000HE 77.5 x 80.3 mm^2 Up to 100 Hz 0 ms in ZeroDeadTime mode 100 µm air-cooled

Figure 1: View of the chlorothiazide crystal mounted on the goniometer.

Data collection

A target completeness of 100% and redundancy of 3 at 0.837 Å on the Friedel mates merged was used to calculate the strategy in CrysAlis^{Pro}. 26 scans were calculated, 9 at 2θ = -47.20°, 4 at 2θ = -86.25° and 17 at 2θ = -106.49°. The exposure time was estimated by CrysAlis^{Pro} for a target <I/σ(I)> of 30 at 0.837 Å for the data unmerged, based on the <I/σ(I)> calculated upon screening. The full data collection time was 2 hours and 51 minutes. Table 1 shows the data collection parameters.

Table 1: Data collection parameters.

Generator settings	50 W (50 kV, 1 mA)
Wavelength	1.54184 Å
Temperature	100 K
Detector distance	34 mm
Low resolution exposure time	1 seconds
High resolution exposure time	4 seconds
Scan width	0.5 °
Total frames	3176
Total time	2 hours 51 minutes

With exposure times of 1 second and 4 seconds per 0.5 ° for the low resolution and high resolution scans, respectively, diffraction from the crystal yielded small, very sharp reflections across the resolution range (Figure 2).

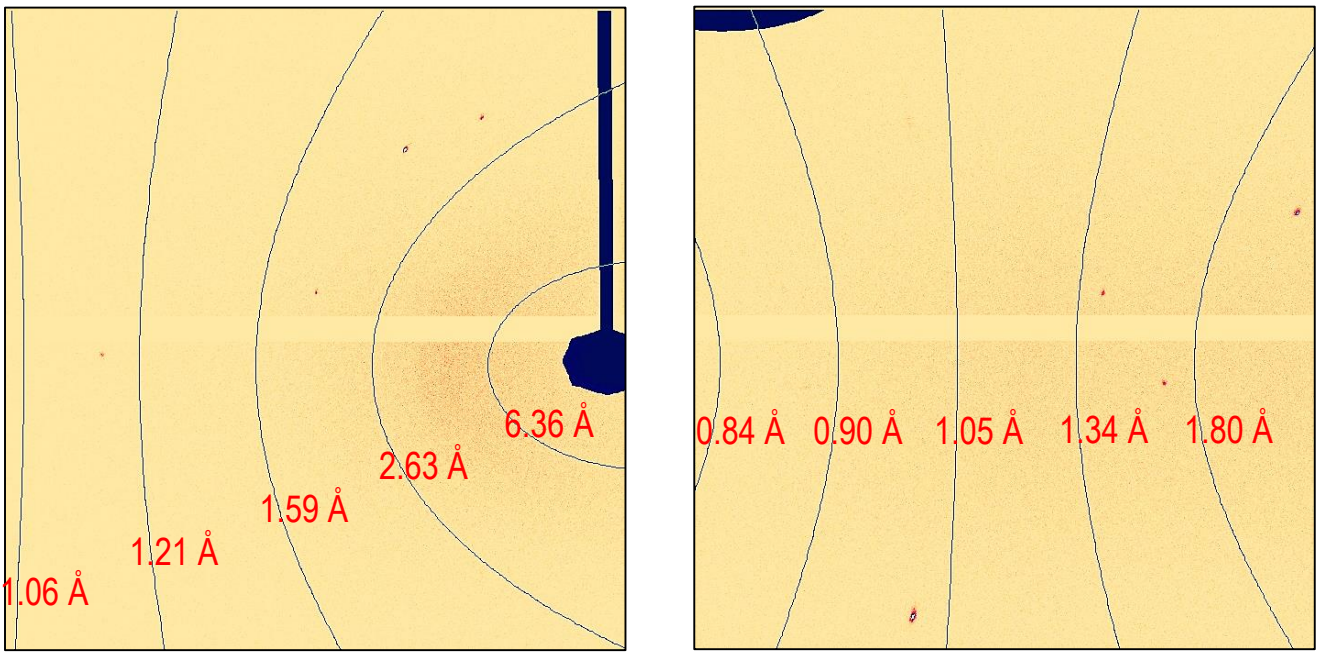

Figure 2: Example of diffraction images obtained at low resolution with 1 sec / 0.50° (left) and at high resolution with 4 sec / 0.50° (right).

Results

Upon processing with CrysAlis^{Pro}, the data set was 100% complete, including in the last resolution shell to 0.837 Å (Table 2). Data processing yielded an overall R_{int} of 3.6% and <I/σ(I)> of 31.9 to 0.837 Å. The crystal structure (Figure 3) was automatically solved by ShelxT[2] in space group P1 and refined with ShelxL[3] within the AutoChem[4] interface. Hydrogen atoms were placed as riding atoms. Excellent structural statistics were obtained, with R_1 at 2.51% and GooF at 1.070.

Table 2: Experiment and refinement details for the Friedel pairs unmerged.

Space group	P1
Unit cell	4.8469(3), 6.3804(3), 8.9211(4) Å 74.317 (4), 83.926(5), 80.475(5) °
Resolution	0.837 Å
Completeness (last shell)	100% (100%)
Redundancy (last shell)	3.1 (1.8)
<I/σ(I)> (last shell)	31.9 (20.1)
R_{int} (last shell)	3.6% (5.8%)
Final R factors [I>2σ (I)]	R_1 = 2.51% wR_2 = 6.29%
Goodness of fit	1.070
Largest residual peak/hole (eÅ^{-3})	0.329 / -0.268

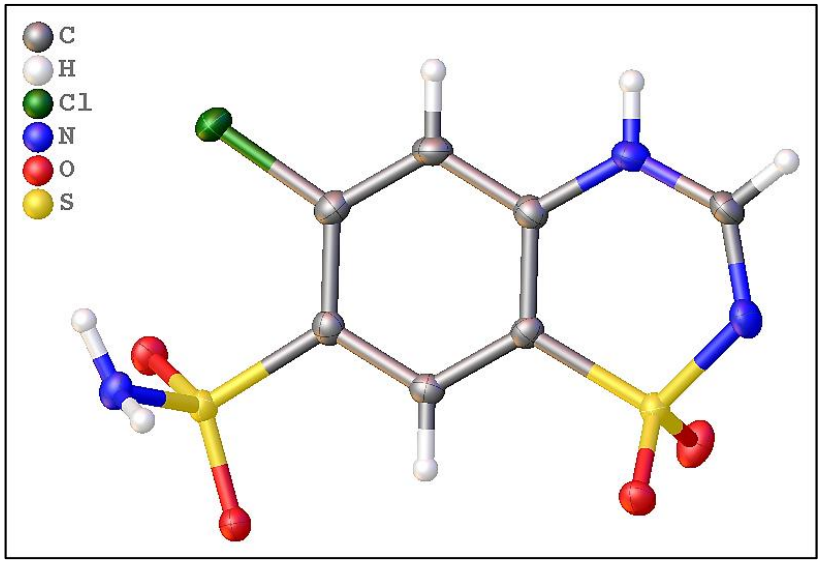

Figure 3: Representation of the model obtained for the chlorothiazide molecule after refinements to 0.837 Å.

Conclusion

A 100% complete data set, both overall and in the last resolution shell to 0.837 Å, was collected on a small crystal crystal of chlorothiazide (0.019 x 0.035 x 0.046 mm^3) in under 3 hours, using the microfocus sealed tube X-ray generator XtaLAB Synergy-S configured with a 4-circles kappa Universal Goniometer. The crystal structure of chlorothiazide was solved readily and yielded excellent statistics. Such results demonstrate that:

- The Universal Goniometer allows for the collection of complete data sets, even in the lowest symmetry possible.
- Combined with the powerful strategy algorithm in CrysAlis^{Pro}, the Universal Goniometer enables the collection of the best data sets in a minimum amount of time.

References

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3. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H., *J. Appl. Cryst.* (2009), **42**, 339-341.
4. Sheldrick, G. M. (2015), *Acta Cryst.* **A71**, 3-8.

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