Moving Crystals
A Software Tool to Correct for Severe Crystal Movement or Mis-Alignment

Application Note

X-ray Crystallography

Authors

Dr. Oliver Presly
Agilent Technologies UK Ltd

Prof. Patrick McArdle
Dr. Andrea Erxleben
School of Chemistry
National University of Ireland
Galway

Introduction

Where crystals are mounted on glass fibers using an epoxy resin, crystal movement can be a common occurrence, especially during room temperature data collections. This is further compounded when needle-like crystals are mounted "end-on", whereby capillary action slowly re-orientates the sample so that the pin is in contact with a larger crystal face.

Using the Agilent Crysalis^{Pro} software, crystal movement can be observed by looking for the incremental movement of reflections in reference frames taken during data collection. These frames are measured by automatically reading images at the same reference position at a user-defined frequency throughout the data collection, for example, every 50 frames (Figure 1).

Figure 1. Xcalibur S installed at the National University of Ireland, Galway.

Agilent Technologies

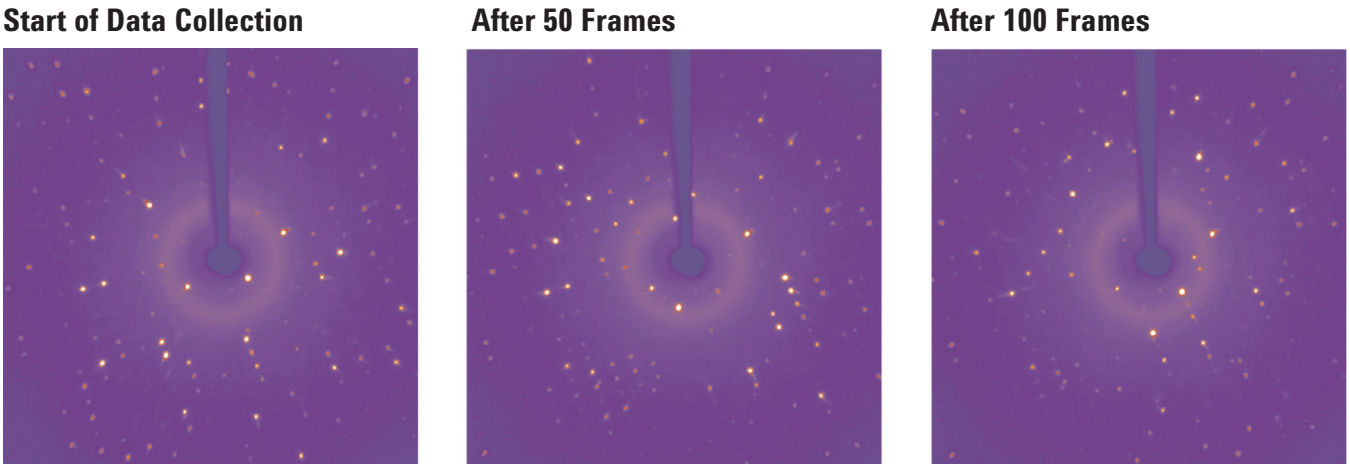

Figure 2. Reference frames illustrating crystal movement (these should be identical).

When crystal movement is significant, automatically integrated data can be severely effected, with structure solution difficult or even impossible. However, movement of the UB matrix can be tracked and the data thus corrected for, using the "Follow sudden changes of sample orientation" tool in CrysAlis^{Pro}'s data reduction software.

Within CrysAlis^{Pro}, frame-based model refinement is performed by carrying out a 2-cycle, 3D peak analysis. The integration masks are refined by modelling the changes in expected reflection positions over a specified angular range. A pre-determined number of steps (or attempts) per degree are used in order to ascertain the best model for following the UB matrix change. For particularly severe cases, a range of up to 10 degrees with 10 steps per degree can be applied, with the only limiting factor being the time taken for the completion of the data reduction process.

One such example where crystal movement was found to be significant is with data collected by Prof. Patrick McArdle and Dr. Andrea Erxleben at the National University of Ireland, Galway using their Xcalibur S system (Figure 2). The crystal was mounted using a "rapid-setting", two-part Araldite epoxy resin. However, over the course of a 9 hour, room temperature data collection the crystal moved significantly, rendering the resultant automatically integrated data of little use (R_{int} = 30.4%). This is particularly demonstrated by the displaced integration masks calculated for frames towards the end of the data collection (Figure 3).

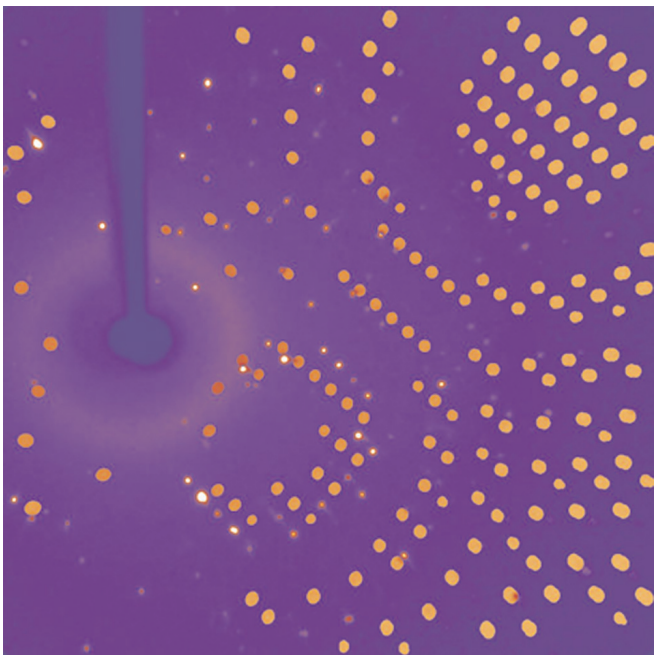

Figure 3. Predicted integration mask from automatic data reduction for frame number 349 (467 total), clearly illustrating the mis-match in reflection mask positions.

Moving-Crystal Data Reduction

resolution(A)	# measured	# unique	# theory	%complete	redundancy	average F2	mean F2/sig(F2)	mean Rint	RsigmaB
inf-1.77	3728	669	685	97.7	5.6	343696.61	155.61	0.018	0.006
1.77-1.40	3201	669	670	99.9	4.8	163004.44	96.43	0.020	0.009
1.40-1.22	2907	669	675	99.1	4.3	107087.47	59.86	0.028	0.016
1.22-1.10	2651	669	676	99.0	4.0	94547.85	47.92	0.031	0.021
1.10-1.02	2416	669	697	96.0	3.6	70808.31	33.83	0.037	0.029
1.02-0.96	2215	669	693	96.5	3.3	48810.27	22.67	0.050	0.044
0.96-0.91	2073	669	703	95.2	3.1	37176.66	16.16	0.059	0.061
0.91-0.87	1912	669	704	95.0	2.9	33052.61	13.69	0.069	0.074
0.87-0.84	1770	669	708	94.5	2.6	27083.25	10.54	0.081	0.097
0.84-0.80	1680	675	728	92.7	2.5	26438.58	9.71	0.089	0.107
inf-0.80	24553	6696	6939	96.5	3.7	117168.30	46.64	0.027	0.022

resolution(A)	# measured	# unique	# theory	%complete	average redundancy	mean F2	mean F2/sig(F2)	Rint	RsigmaB
inf-1.85	3360	1221	1241	98.4	2.8	63866.17	40.24	0.254	0.022
1.85-1.47	2785	1221	1240	98.5	2.3	32889.99	25.65	0.276	0.045
1.47-1.28	2507	1221	1270	96.1	2.1	21265.38	14.46	0.283	0.082
1.28-1.16	2219	1221	1301	93.9	1.8	17254.73	10.15	0.317	0.127
1.16-1.07	2057	1221	1377	88.7	1.7	14776.24	7.49	0.408	0.149
1.07-1.00	1866	1221	1432	85.3	1.5	12835.22	5.64	0.479	0.193
1.00-0.95	1755	1221	1455	83.9	1.4	10786.13	4.12	0.590	0.231
0.95-0.90	1607	1221	1516	80.5	1.3	8781.17	3.42	0.596	0.271
0.90-0.86	1555	1221	1584	77.1	1.3	7628.80	2.63	0.539	0.371
0.86-0.80	1372	1226	2950	41.6	1.1	6672.01	2.14	0.615	0.375
inf-0.80	21083	12215	15366	79.5	1.7	24009.51	11.59	0.304	0.079

Refinement Statistics

Empirical formula	$C_{31}H_{31}Cl_3N_5O_1Sn_{1,5}$
Formula weight	773.99
Temperature	293(2)K
Wavelength	0.71073
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 12.756(1) Å b = 17.290(2) Å c = 15.533(2) Å α = 90° β = 96.831(13)° γ = 90°
Volume	3401.5(6) Å^3
Z	4
Density (calculated)	1.511 Mg/m^3
Absorption coefficient	1.376 mm^{-1}
F(000)	1544 electrons
Crystal size	0.26 × 0.29 × 0.31 mm
Theta range for data collection	2.49° to 26.32°
Index ranges	–15 ≤ h ≤ 15, –21 ≤ k ≤ 21, –18 ≤ l ≤ 18
Reflections collected	23684
Independent reflections	6527[$R_{(int)}$ = 0.0287]
Completeness to theta = 25.00°	97.8%
Refinement method	Full-matriz least-squares on F^2
Data / restraints / parameters	6527 /0 / 379
Goodness of fit	1.128
Final R indices [> 2sigma9(I)]	R_1 = 0.0317, wR2 = 0.0854
Final R indicies (all data)	R_1 = 0.0477, wR2 = 0.0899
Largest diff. peak and hole	0.656 and –0.532 e/Å^3

In the case provided by the authors at the National University of Galway, the data were vastly improved (R_{int} = 2.7%) by using a range of 3 degrees with 4 steps per degree. The average I/σ value increased by a factor of almost 5 when compared to that obtained from standard data reduction, yielding an excellent structure solution and refinement with an $R_1[> 2\sigma(I)]$ of 3.17% (Figure 4).

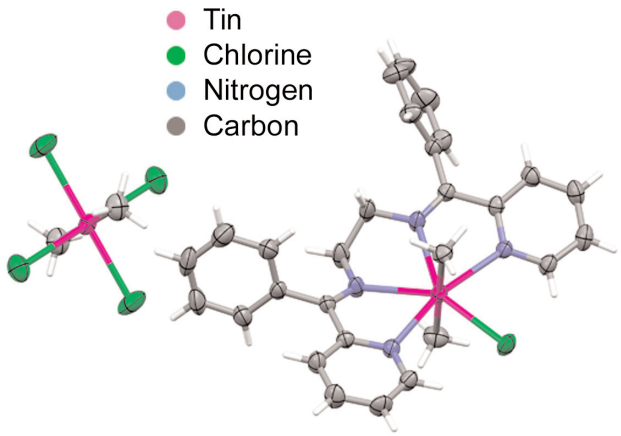

Figure 4. Refined molecular structure (ellipsoids at 50% probability level).

For More Information

For more information on our products and services, visit our Web site at www.agilent.com/chem

www.agilent.com/chem

Printed in the USA
November 1, 2010
SI-A-1379

Agilent Technologies