Copper vs. Molybdenum on a Dual Source SuperNova

Application Note

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Introduction

Since the introduction of CCD based area detectors for X-ray crystallography molybdenum has often been put forward as the best choice for a general purpose diffractometer system. The characteristics of molybdenum radiation, with its short wavelength and low absorption, made it a natural fit for the slower early CCD devices and early software. However, the ever-growing popularity of dual source systems has enabled many chemical crystallographers to gain experience with copper as well as molybdenum sources. With recent advances in micro-focus source technology, CCD detectivity and advanced data processing algorithms in Agilent's CrysAlis^{Pro} software, copper is frequently a better alternative to molybdenum, and is therefore being increasingly used as the default X-ray source in many laboratories.

Figure 1. Agilent dual micro-focus sources

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Background

The wavelength of radiation used in a diffraction experiment dictates the size of the Ewald sphere according to an inverse dependence on the wavelength itself. In terms of the diffraction experiment in direct space, this translates to a requirement to measure a larger portion of direct space as wavelength increases in order to achieve complete data to a given resolution. For a typical data collection it might be expected to collect approximately 6-10 times more frames when using copper as opposed to molybdenum depending on crystal orientation and detector size.

Another factor to consider however is the relative intensity of the two sources. Due to both the higher efficiency of copper focusing optics and higher diffraction power of copper radiation, we generally see more diffracted intensity from our crystal during a copper data collection than when using molybdenum radiation. In terms of experimental set up, this generally means we can use lower frame exposure times in order to achieve measurable data to our target resolution.

These two competing effects, the first requiring more frames for longer wavelengths and the second allowing shorter frame collection times, will together dictate the total experiment length.

Data Collection Challenge – Mo and Cu data sets collected with a 1 hour time limit

In order to compare the SuperNova's Mova (Mo) and Nova (Cu) micro-focus sources, a small organic crystal (fig. 2) was measured on both wavelengths with a total data collection time limit of 1 hour. The challenge was to observe whether or not both wavelengths could provide data of sufficient quality for publication.

Figure 2 – Small sample crystal (encapsulated in glue) with approx. dimensions 0.044 x 0.059 x 0.102 mm

Experimental

The crystal was mounted on a dual source SuperNova equipped with the large area Atlas CCD detector. Two datasets were then collected, one with Mo radiation and another with Cu radiation (details are given in Table 1). The experiments were carried out consecutively on the same diffractometer to ensure the experimental conditions and crystal orientation did not change. Using the CrysAlis^{Pro} strategy module, which has been carefully optimised to produce efficient collection strategies quickly for either Cu or Mo radiation, a 1 hour data collection strategy was devised for both wavelengths. The goal was to first attempt to collect complete data (98.5% or better) up to a minimum resolution of 0.84 Å (2θ = 50° for Mo, 133° for Cu) then to consider diffracted intensity. The frame exposure times were tuned to achieve this, meaning that under the time constraint applied, I/σ statistics for each dataset could then be used as an indication of the relative performance of the two sources.

	Molybdenum	Copper
Wavelength	0.7107 Å	1.5418 Å
Temperature	296 K	296 K
Min Exposure Time (inc. retakes)	5.5 seconds	1 seconds
Max Exposure Time	22 seconds	6 seconds
Detector Distance	53 mm	53 mm
Scan Width	2°	1°
Total Frames	77	650
Total Time	1 hour	1 hour

Table 1 – Data collection parameters for Mo and Cu datasets

Results

Using an average I/σ of 2 as an indication of the diffraction limit, the copper dataset diffracts beyond the intended target of 0.84 Å whilst the molybdenum dataset only reaches approximately 1.06 Å. The effect of this carried across to other data quality indicators such as R_{int} and the conventional R-factor (R1) (see table 2).

	Molybdenum	Copper
Diff. Limit [I>2σ(I)]	1.06 Å	0.84 Å
<I>	880.61	19499
<I/σ>	4.51	6.12
R_{int} [all data]	8.60 %	5.20 %
R1 [all data]	7.54 %	5.85 %

Table 2 – Data quality indicators from 1 hour copper and molybdenum data sets

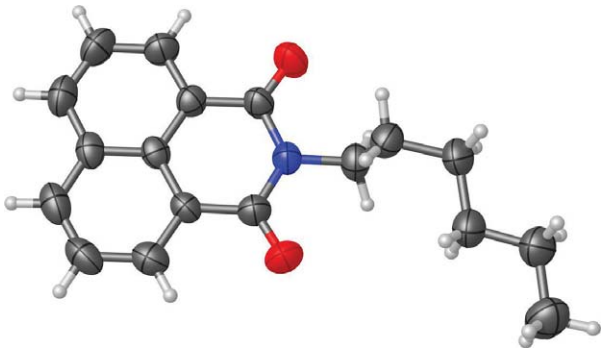

Figure 3 – Structure of N-hexyl-1,8,naphthalimide

Conclusions

In this case the structure solves and refines easily for either data set. However, the poor effective resolution limit of the molybdenum dataset (1.06 Å) would require justification in order to publish the data. The copper data is much stronger and therefore has no such problems.

This fits the general trend that whilst copper datasets require significantly more frames to achieve the same level of completeness, exposure times are shorter and the data obtained in the same overall time is typically better than corresponding molybdenum data. It is therefore no surprise that high intensity copper sources are increasingly being used in routine chemical crystallography as well as in more traditional applications such as absolute configuration studies and protein crystallography.

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