Radiation Damage in Chemical Crystallography

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Introduction

Although crystals suffering radiation damage is a well-known and studied phenomena for macromolecular crystallography[1], as far as we are aware there still appears to be no such published work relating to chemical crystallography. However, there are numerous anecdotal accounts of disintegrating crystals and resolution progressively dropping off that have been ascribed to radiation damage. Since the start of operations on the small molecule synchrotron beamline I19[2] at Diamond Light Source (DLS), there have continued to be anecdotal observations from several users of sample damage in the beam.
The UK National Crystallography Service[3] handles a wide variety of samples and a number (around 2%) of these have experienced what is believed to be radiation damage. In order to understand the causes and symptoms of this effect in greater detail some controlled experiments have been performed.
So far two different samples have been investigated in some detail: a gold complex and a nickel complex. The various experiments involved changing temperature, overall dose, dose rate and X-ray source. For each compound, efforts were made to select as similar sized crystals each time to limit volume based effects. Presented here are a few of the results involving temperature, dose and X-ray source for the gold and nickel complexes.

Gold Complex

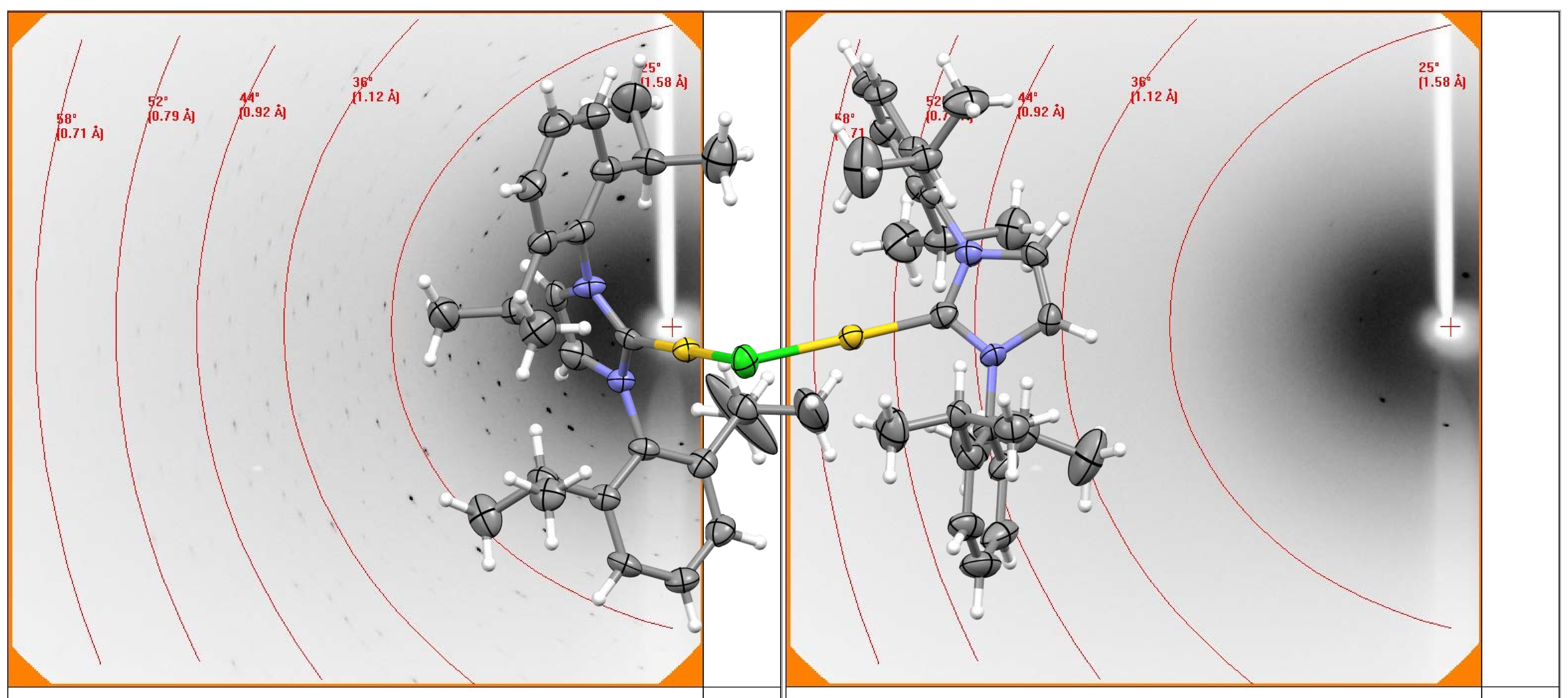
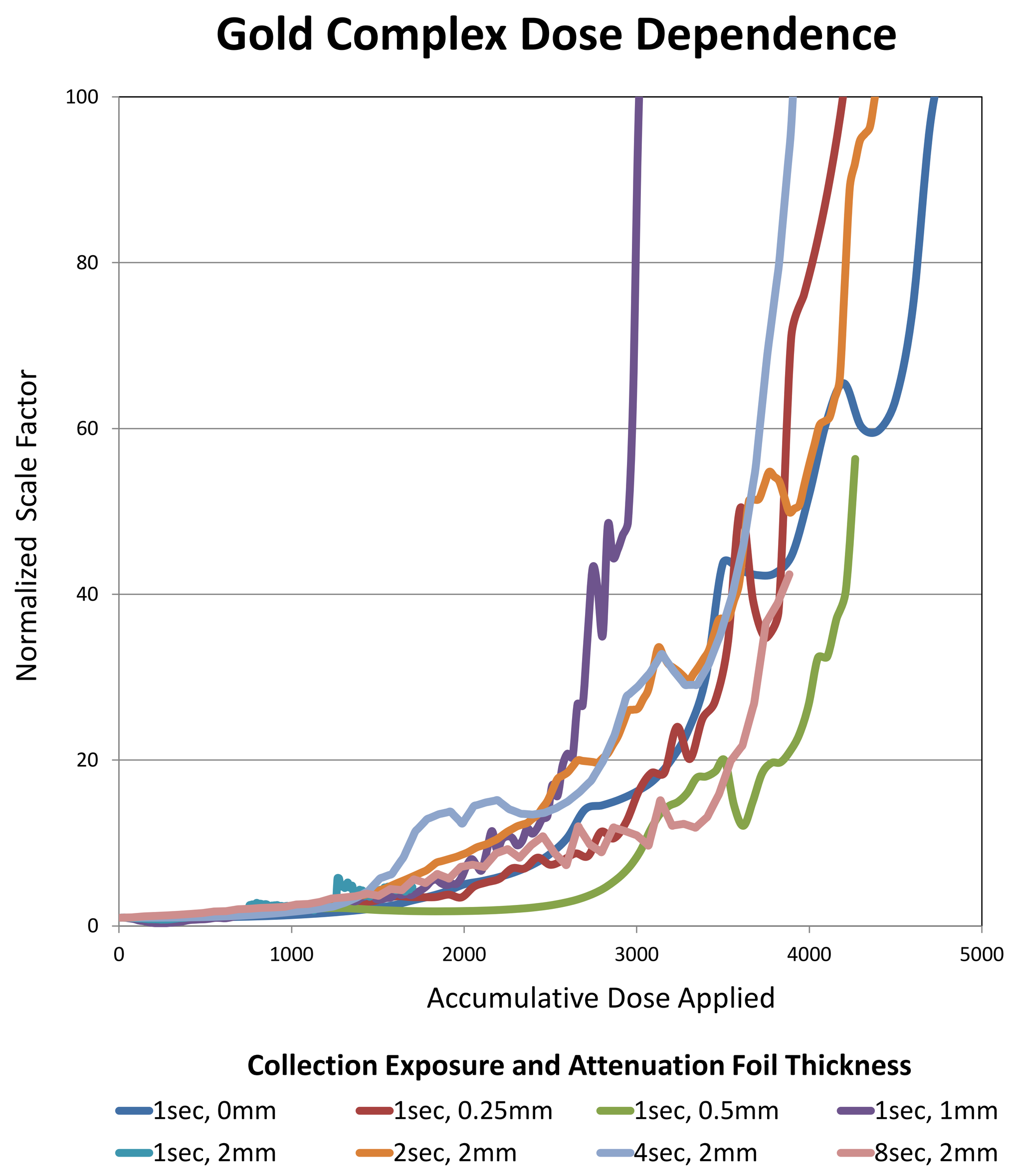


Image 2 | Image 45

The gold complex shows a fairly rapid rate of damage using the standard data collection strategy on I19 (λ = 0.6889Å, 100K, 1 sec/°, 0 attenuation), with the loss of all but the most intense low angle reflections after just over one minutes worth of exposure. This allows for the effects of temperature and dose on radiation damage to be studied.



Dose can be controlled by varying both the thickness of attenuators (Al foil) placed in the beam and the time of each image. To normalise results a one second exposure with no attenuation was given a standardised value of 100 dose. For each 0.25mm of foil with λ = 0.6889Å, a 26.5% dose reduction was applied, enabling a direct comparison between experiments. The actual rate of damage for dose applied is similar in all cases. Thus, in order to obtain the highest possible data completeness (most images) requires the appropriate use of attenuation.

Nickel Complex

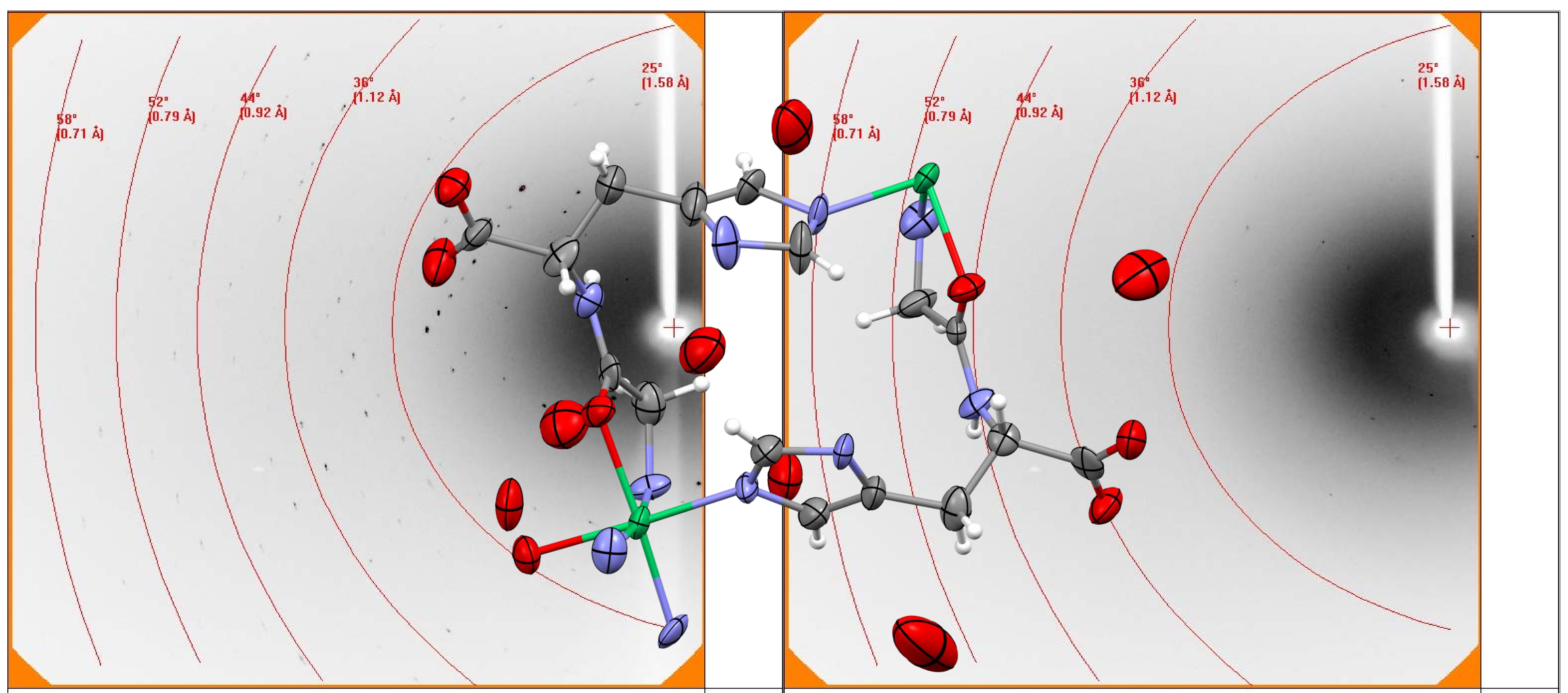
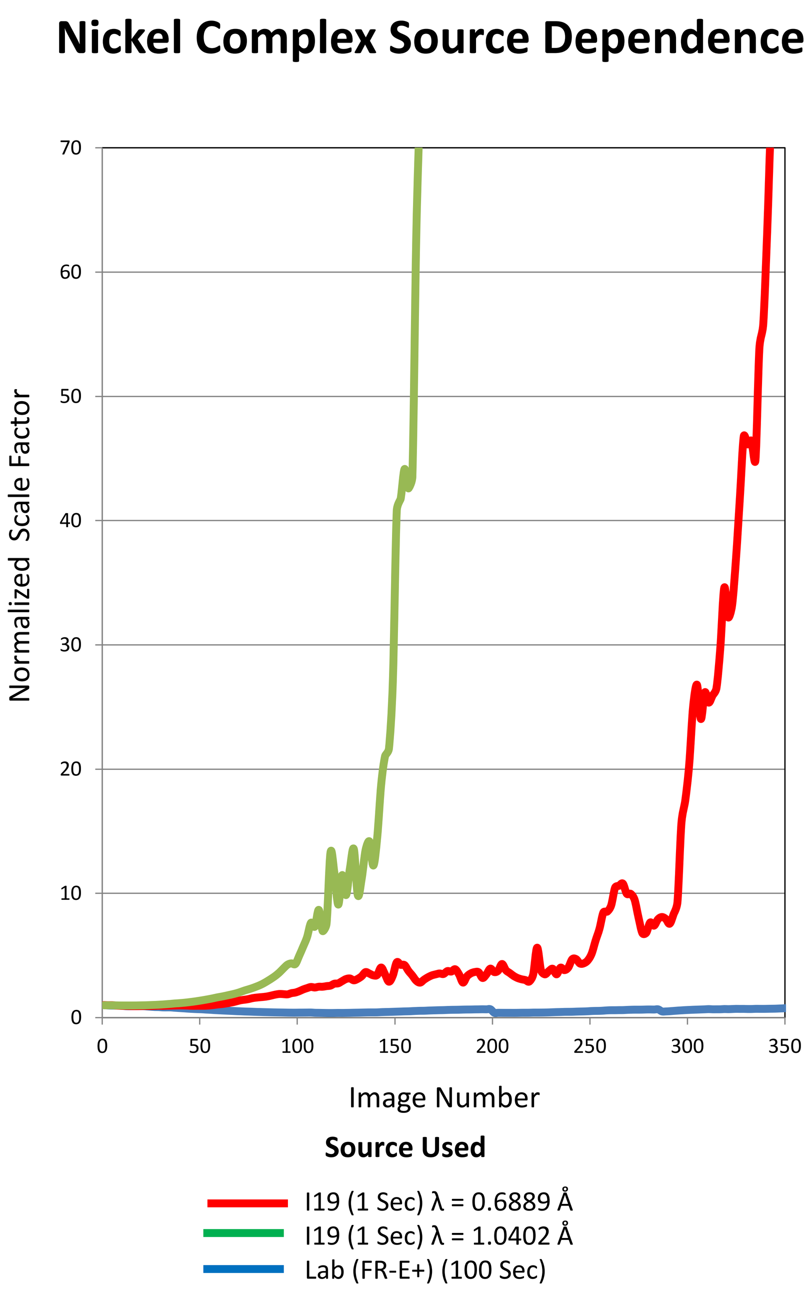
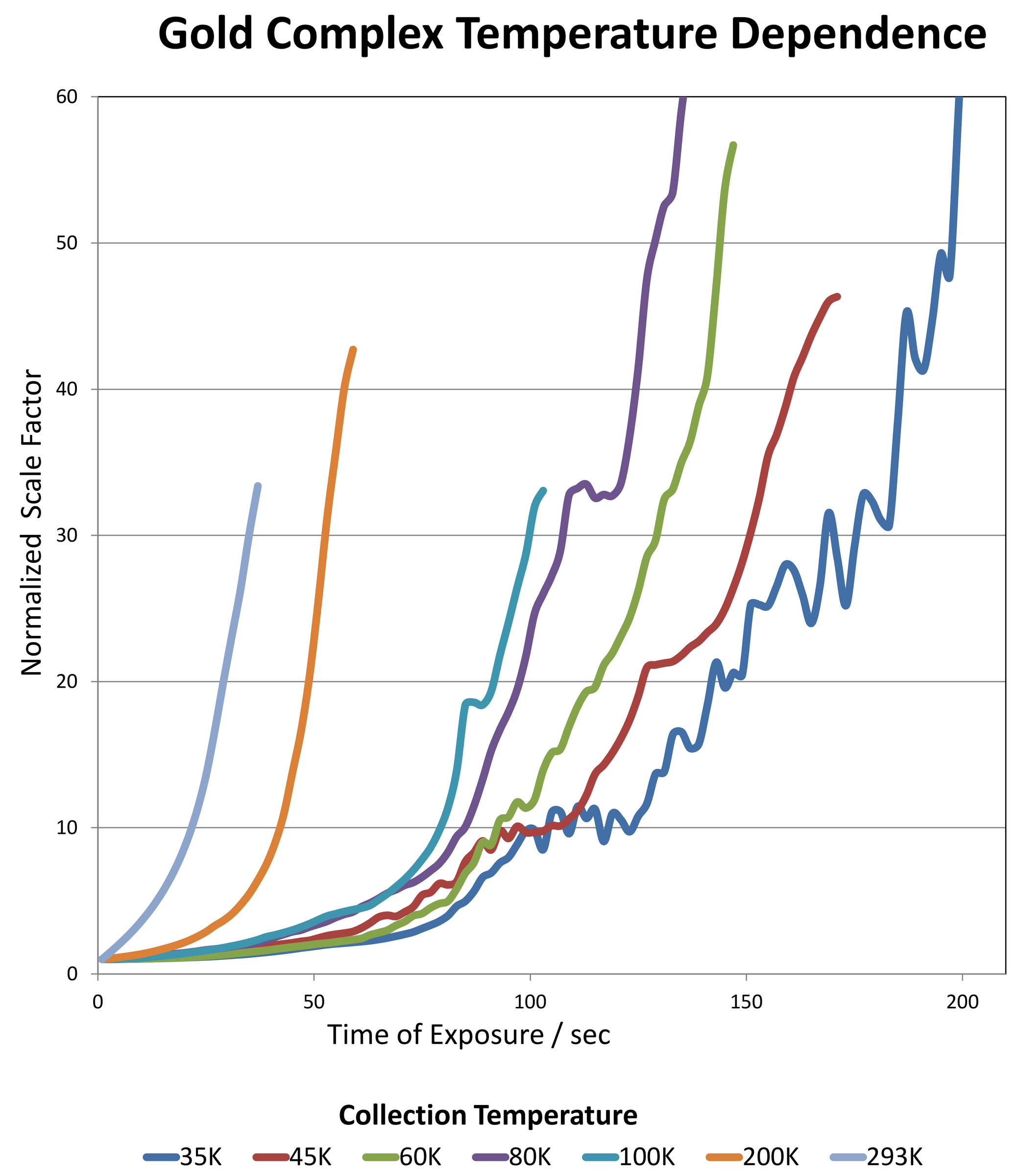


Image 2 | Image 320

The nickel complex[4] shows a relatively slow rate of damage using the standard data collection strategy on I19 (λ = 0.6889Å, 100K, 1 sec/°, 0 attenuation), with some diffraction still observed on the final images of the collection, whereas on a rotating anode laboratory-based diffractometer (FR-E+, λ = 0.71075Å) effectively no damage is observed. Moving to a longer wavelength (λ = 1.0402Å) on I19 produces a markedly increased rate of damage and this effect is also observed for the gold complex. Varying exposure time, attenuation and temperature on I19 produced similar damage trends to the gold complex, albeit at a significantly slower rate and with greater error margins as it is a weaker diffractor. The room temperature lab collection only showed minimal sign of damage.



The effect of altering temperature is as expected, with the lowest resulting in least damage. Using the standard data collection strategy for I19 (λ = 0.6889Å, 0 attenuation, 1 sec/°), a crystal measured at 35K lasts around twice as long as one measured at 100K, while a room temperature (293K) crystal barely survives 30 seconds of exposure. Experiments using a rotating anode laboratory diffractometer (FR-E+) exhibit a slow rate of damage at room temperature and no appreciable damage at 100K



Acknowledgements

We would like to thank the following for allowing us to use information pertaining to the example crystals:-
Professor Manfred Bochmann, University of East Anglia (Gold complex)
Dr Sarah Barnett, Diamond Light Source (Nickel complex and also for useful discussions regarding possible crystal decay on I19)

References

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