

In-situ monitoring of a metal recovery process using dual imaging and diffraction

Susanna S. M. Vance,^{1 a} Rebecca Rae,¹ Luke M. M. Kinsman,¹ Caroline Kirk,¹ Liam Perera,² Carole A. Morrison^{1*} and Jason B. Love^{1*}

¹EaStCHEM School of Chemistry, University of Edinburgh, Edinburgh EH9 3FJ.

²Diamond Light Source, Harwell Science and Innovation Campus, Didcot, Oxfordshire, OX11 0DE, UK.

[*Carole.Morrison@ed.ac.uk](mailto:Carole.Morrison@ed.ac.uk) [*jason.love@ed.ac.uk](mailto:jason.love@ed.ac.uk)

Supplementary Information

Experimental Methods

Sample Preparation

Dry samples of the complexes were prepared prior to beamtime by contacting **L** (0.1 mmol) with a solution of either gold or iron (0.05 M H₂AuCl₄ or FeCl₃ in 6 M HCl) and stirring for 1 h. The samples were then filtered and dried before collection and storage.

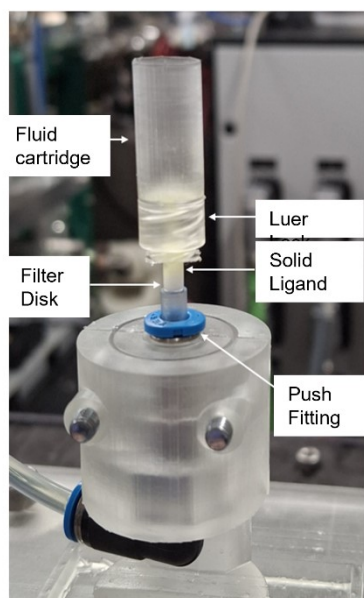
PXRD and Tomography details

The flow cell was designed for and tested on K11-DIAD of Diamond Light Source.¹ K11 is a dual beam instrument with the diffraction and imaging beams configured independently but registered spatially to allow for image guided diffraction. The imaging beam was configured to 18 keV with Tomograms collected using 1100 projections over 180° with a 0.5 second exposure (9.5 minutes total). Projections were collected using a PCO edge 5.5 CLHS detector binned 4 times with a 180 µm GGG: Tb scintillator. Reconstructions were carried out using filtered back projection with ASTRA toolbox via Savu.² Diffraction scans were collected using a 25 µm × 25 µm beam at 21 keV. For each flow step, a vertical line scan of 10 points was collected, with the final diffraction patterns analysed the summed results of these 10 points.

Experimental Set-up

The flow cell consists of a 3D stereolithography printed sample cell (Figure S1a) and a reusable sample cell holder (Figure S1b) printed in Formlabs Clear Resin (V4.1), chosen for its transparent properties (to permit remote monitoring) and stability at low pH. The sample cell consists of two parts, namely a fluid cartridge and the sample holder (Figure S1a). The sample holder consists of a hollow tube with a 4mm base that attaches to a push fitting and a Luer-lock (female) at the top that attaches to the fluid cartridge (capacity 1.3 mL). The sample cell can hold approximately 25 mm³ of sample, which is held in place by a filter disk (2.5 mm) supported by a central integrated pillar.

a)



b)

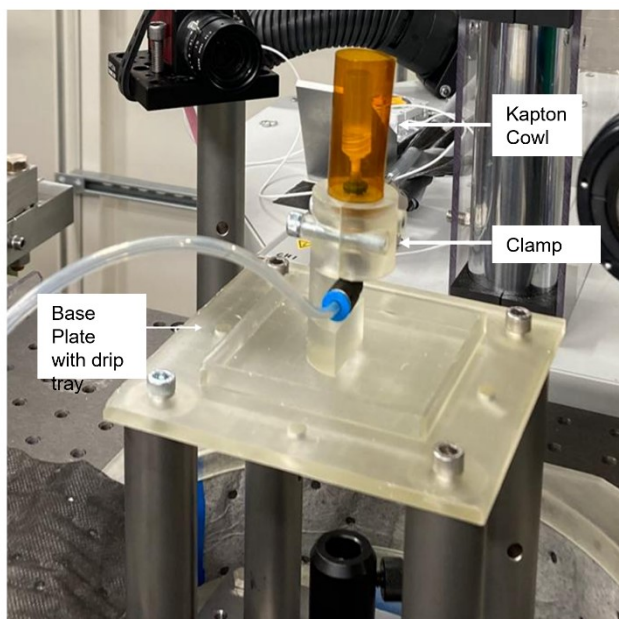


Figure S1: a) Image of the bespoke flow cell, showing both the sample holder and fluid cartridge. b) Expanded view of the flow cell and base plate with drip tray, and Kapton sheath as a leak precaution.

Reaction of Au (0.005 M / 6 M HCl) with L

Solid L (10-15 mg) was added to a sample cell with a drop of the metal solution (0.005 M HAuCl_4 / 6 M HCl). The initial tomography and diffraction pattern suggest that the reaction has gone to completion, indicating a rapid timescale for precipitation of gold by L (Figure S2). At $T=0$, the reconstructed tomography image is significantly obscured due to movement during the scan as a result of the rapid formation of the ligand-metal complex (light regions). The dark regions characteristic of the receptor cannot be seen indicating a rapid reaction timescale. Similarly, the diffraction patterns at $T=0$, 20 mins match that of the expected end-product, confirming the reaction has occurred.

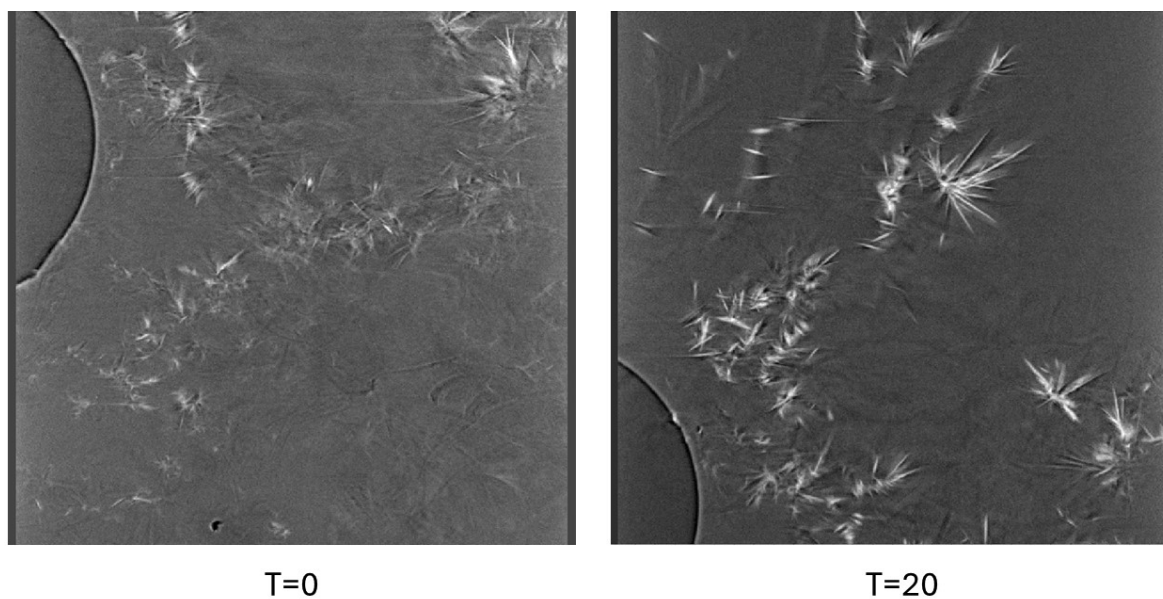


Figure S2: X/Z slices at T=0, 20 mins for the precipitation of gold by L (0.005 M Au / 6 M HCl). At T=0 min. The images are slightly obscured due to the rapid formation of the metal precipitated complex (light regions) resulting in movement during the tomography scan.

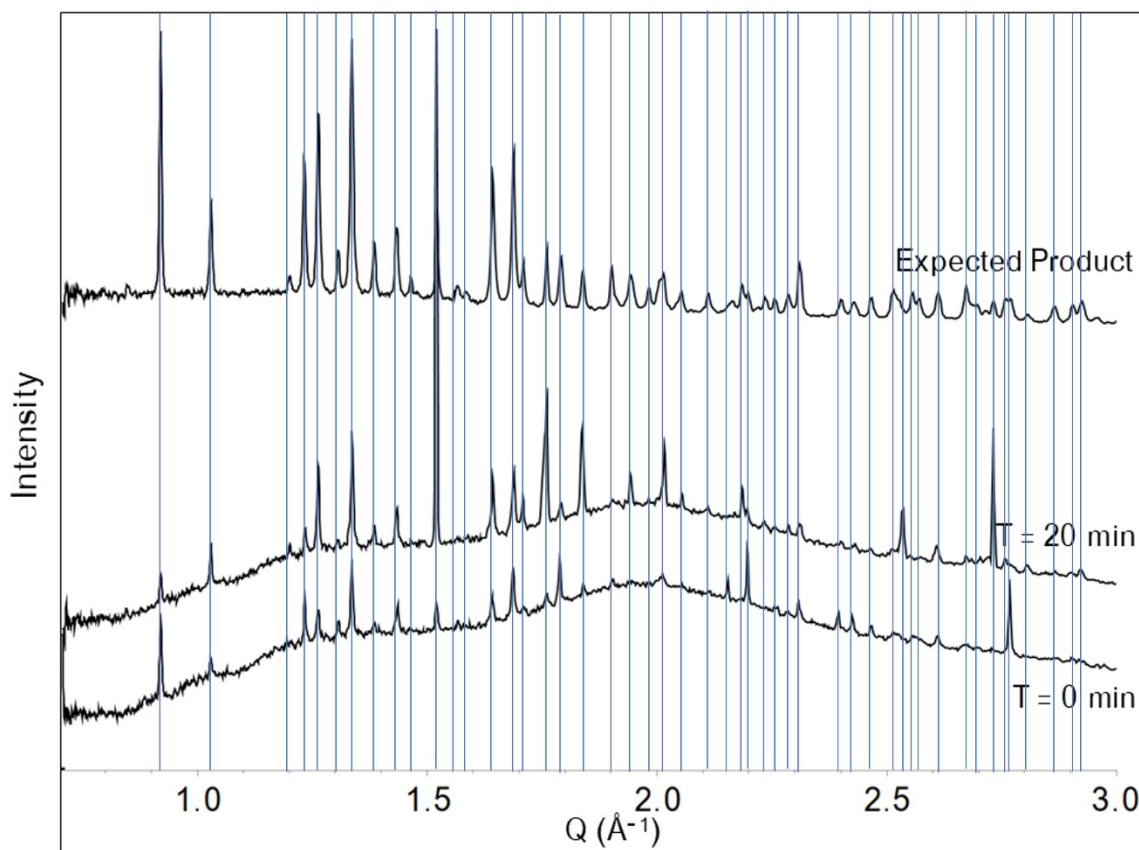


Figure S3: Stacked summed diffraction patterns for the precipitation of gold by L (0.005 M Au / 6 M HCl). The diffraction patterns match those of the expected end-product indicating immediate reaction under these conditions. PXRD patterns have been normalized for clarity.

References

1. L. Perera, P. Garland, C. Kirk, A. Leonardi, J. B. Love, T. Manchester, C. A. Morrison, R. Rae, S. S. M. Vance and S. I. Ahmed, *J. Synchrotron Radiation*, **33**, 2026. DOI:10.1107/S1600577526000123
2. N. Wadson and M. Basham, *Savu: A Python-based, MPI Framework for Simultaneous Processing of Multiple, N-dimensional, Large Tomography Datasets*, 2016.