Electronic Supplementary Material (ESI) for Dalton Transactions

An EXAFS and HR-XANES Study of the Uranyl Peroxides $[UO_2(\eta^2\text{-}O_2)(H_2O)_2].nH_2O$ (n = 0, 2) and Uranyl (Oxy)hydroxide $[(UO_2)_4O(OH)_6].6H_2O$

Aurora Walshe, Tim Prüßmann, Tonya Vitova and Robert J. Baker

Supplementary Information

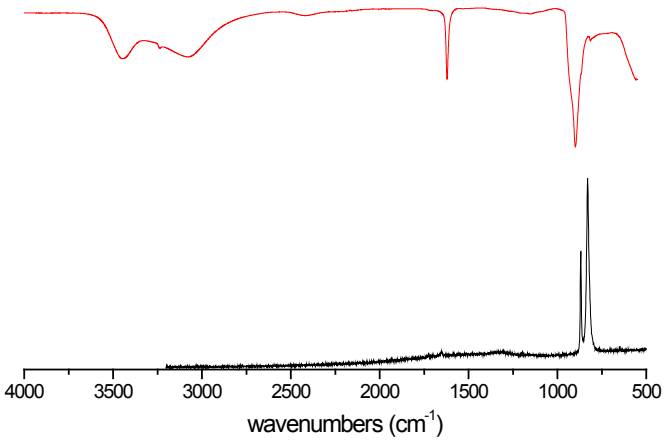

Figure S1: The infrared (top) and Raman (bottom) spectra of synthetic studtite.

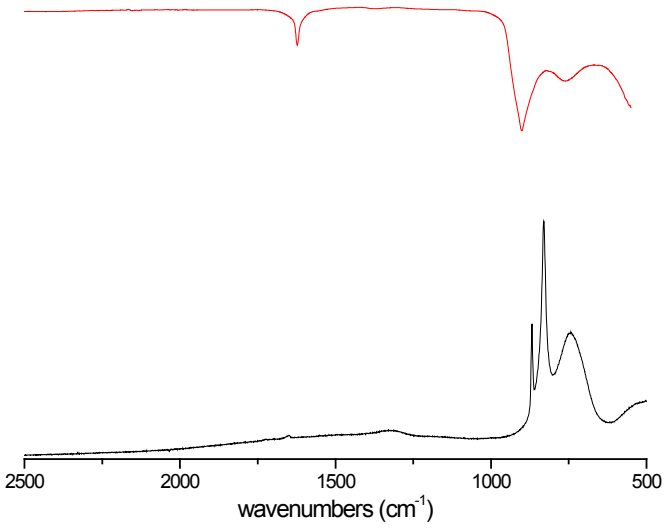

Figure S2: The infrared (top) and Raman (bottom) spectra of synthetic meta-studtite.

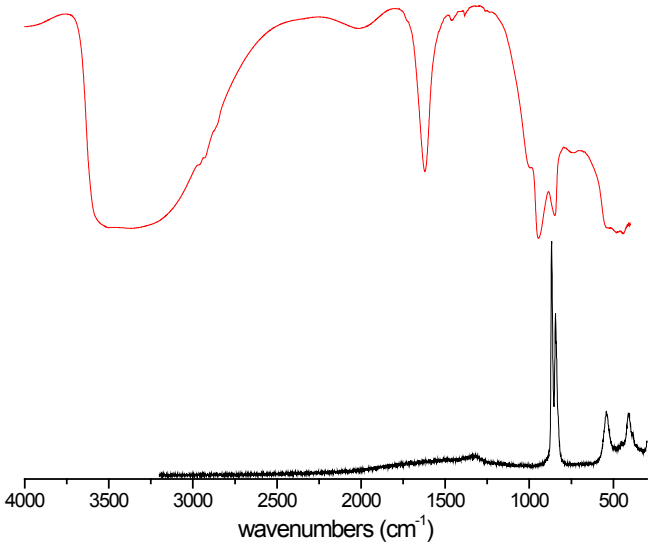

Figure S3: The infrared (top) and Raman (bottom) spectra of synthetic schoepite.

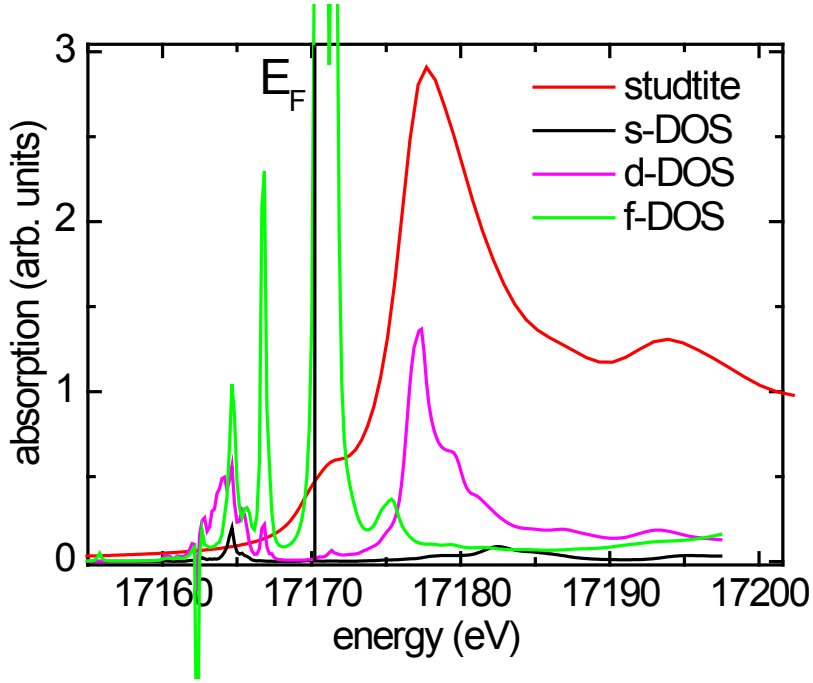

Figure S4: Calculated U-L_3 Edge HR-XANES spectrum and U s-. d- and f-DOS of studtite. The E_F line separates the occupied from the unoccupied DOS.

HR-XANES Data Deconvolution:

The line shape analysis was performed using the open source curve-fitting software Fityk (Version 0.9.8)

[1] with the step function defined as the error function f(x) = step*(erf((x-centre)/width) + 1). The step function was varied for metastudtite and then fixed for the remaining

samples during the fit. The α parameter indicates the Lorentzian component of the pseudo-Voigt (P.V.) function; the Gaussian component of the P.V. function is defined as 1 - α.

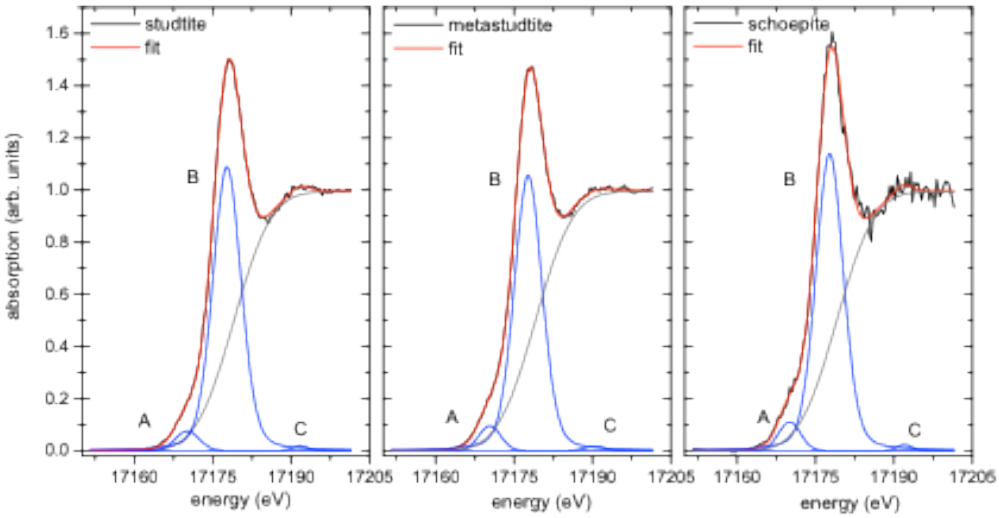

Figure S5: The U L_3 edge HR-XANES spectra of studtite, metastudtite and schoepite. The P.V. functions describe the pre-edge (A), white line (B) and post-edge (C) features. The P.V. parameters are listed below in **Table S1**. Goodness of fit for metastudtite 0.7%, studtite 2.0% and schoepite 10.2%.

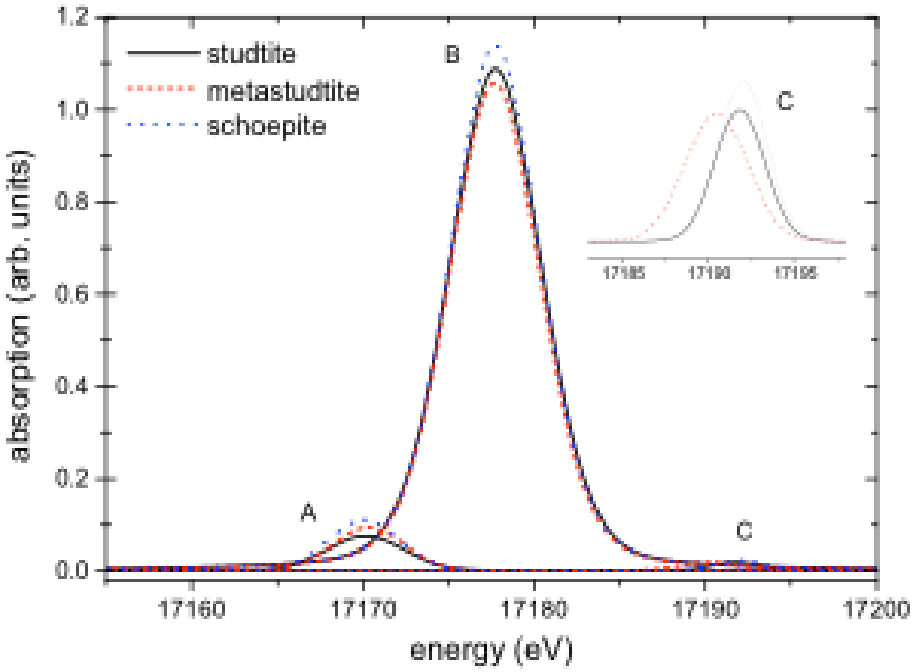

Figure S6: The overlaid P.V. functions describing the pre-edge (A), white line (B) and post-edge (C) features of studtite, metastudtite and schoepite. The expanded inset of C shows the shift in the post-edge feature (**Table S1**)

Table S1: Height, position, and FWHM parameters of the P.V. functions used to model the HR-XANES resonance features A, B and C for studtite, metastudtite and schoepite and the step function to model the edge jump (see **Fig. S5**). The α and the area of the P.V. peaks are also given.

Sample	Feature	Height ±0.001	Position ±0.1	FWHM ±0.3	α ±0.01	Area ±0.06
Studtite	A	0.076	17170.2	2.7	0	0.43
Metastudtite	A	0.112	17170.3	2.5	0	0.50
Schoepite	A	0.111	17170.1	2.5	0	0.58
Studtite	B	1.090	17177.7	3.2	0.3	8.18
Metastudtite	B	1.050	17177.7	3.1	0.3	7.80
Schoepite	B	1.141	17177.7	3.1	0.3	8.33
Studtite	C	0.019	17191.8	1.7	0	0.07
Metastudtite	C	0.021	17190.5	2.1	0	0.08
Schoepite	C	0.023	17192.0	1.7	0	0.08
Studtite	step	0.497	17179.0	8.0	-	-
Metastudtite	step	0.497	17179.0	8.0	-	-
Schoepite	step	0.497	17179.0	8.0	-	-

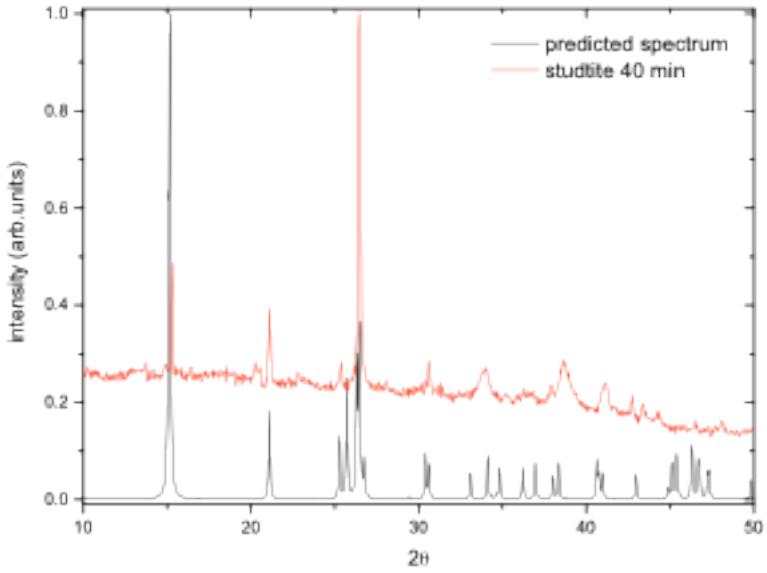

Figure S7: The calculated and found powder data for studtite.

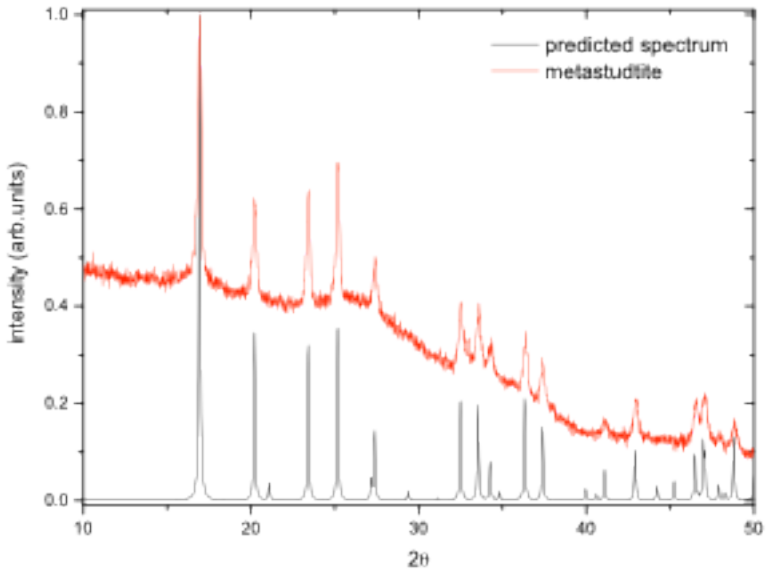

Figure S8: The calculated and found powder data for metastudtite.

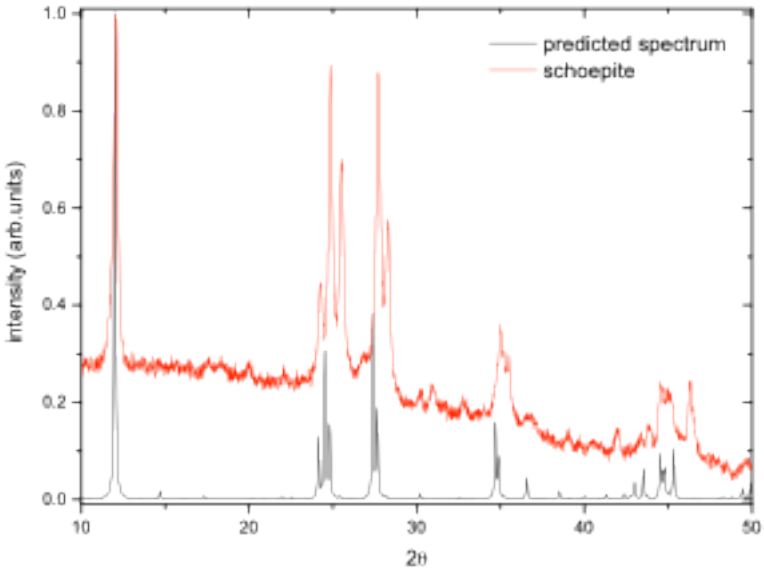

Figure S9: The calculated and found powder data for schoepite.

[1] Wojdyr, M. *Journal of Applied Crystallography* **2010**, *43*, 1126.