Supporting Information

A simple and practical wax-encapsulation method for air-sensitive XAS samples

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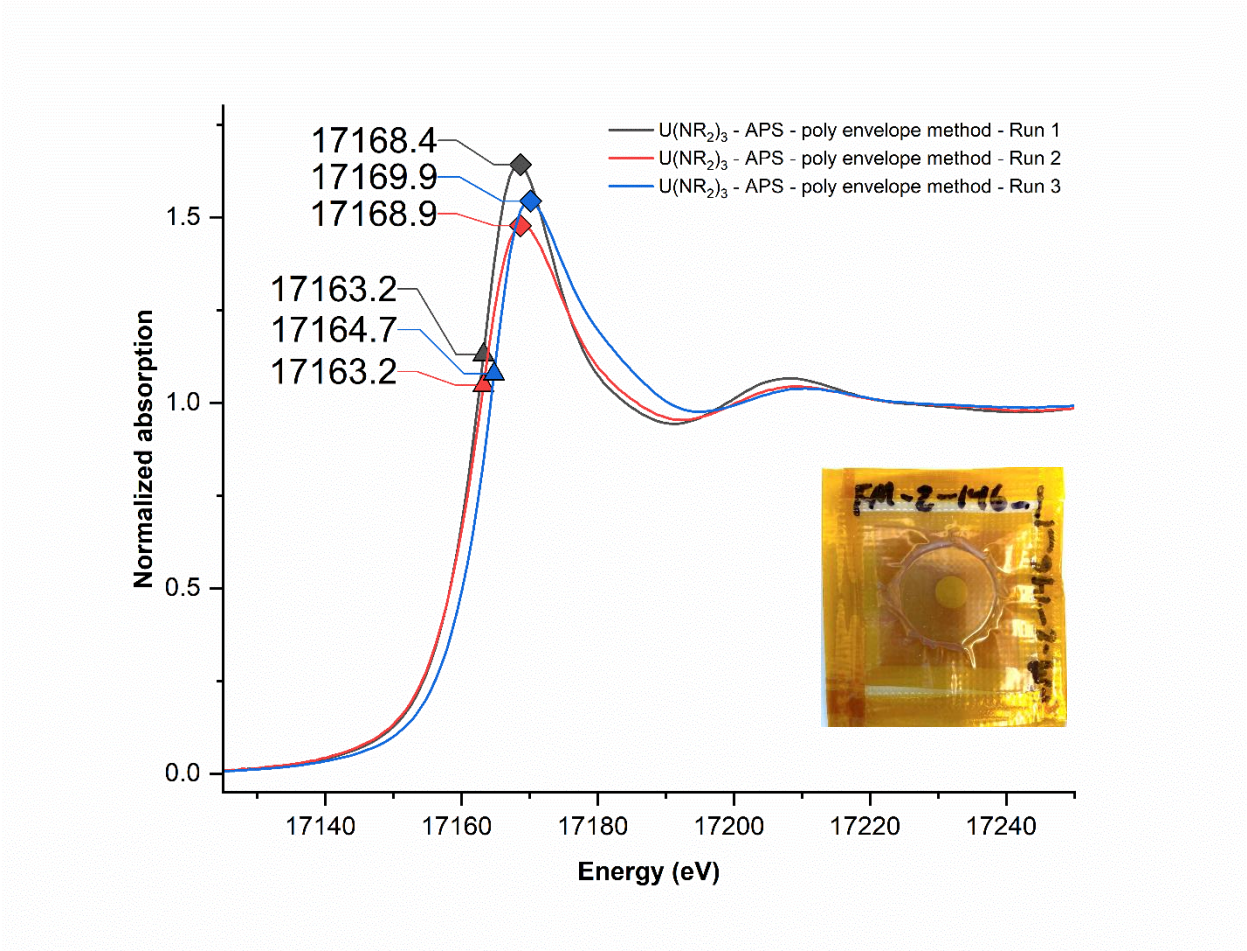

Fig. S1 Uranium L_3-edge XANES spectra of three independently prepared compressed boron nitride pellets of $U(NR_2)_3$ (R = $SiMe_3$) (**1**), each encapsulated using a previously reported method involving vacuum-sealed polyethylene envelopes.[1] An inset photograph shows a representative polyethylene envelope used in this encapsulation technique. The spectra were collected across separate attempts and exhibit misaligned edge energies and variability in spectral features, consistent with adventitious oxidation during handling or prior to measurement. Data were previously collected at the Advanced Photon Source (APS) at sector 10-BM and are referenced to yttrium foil (17038.4 eV).

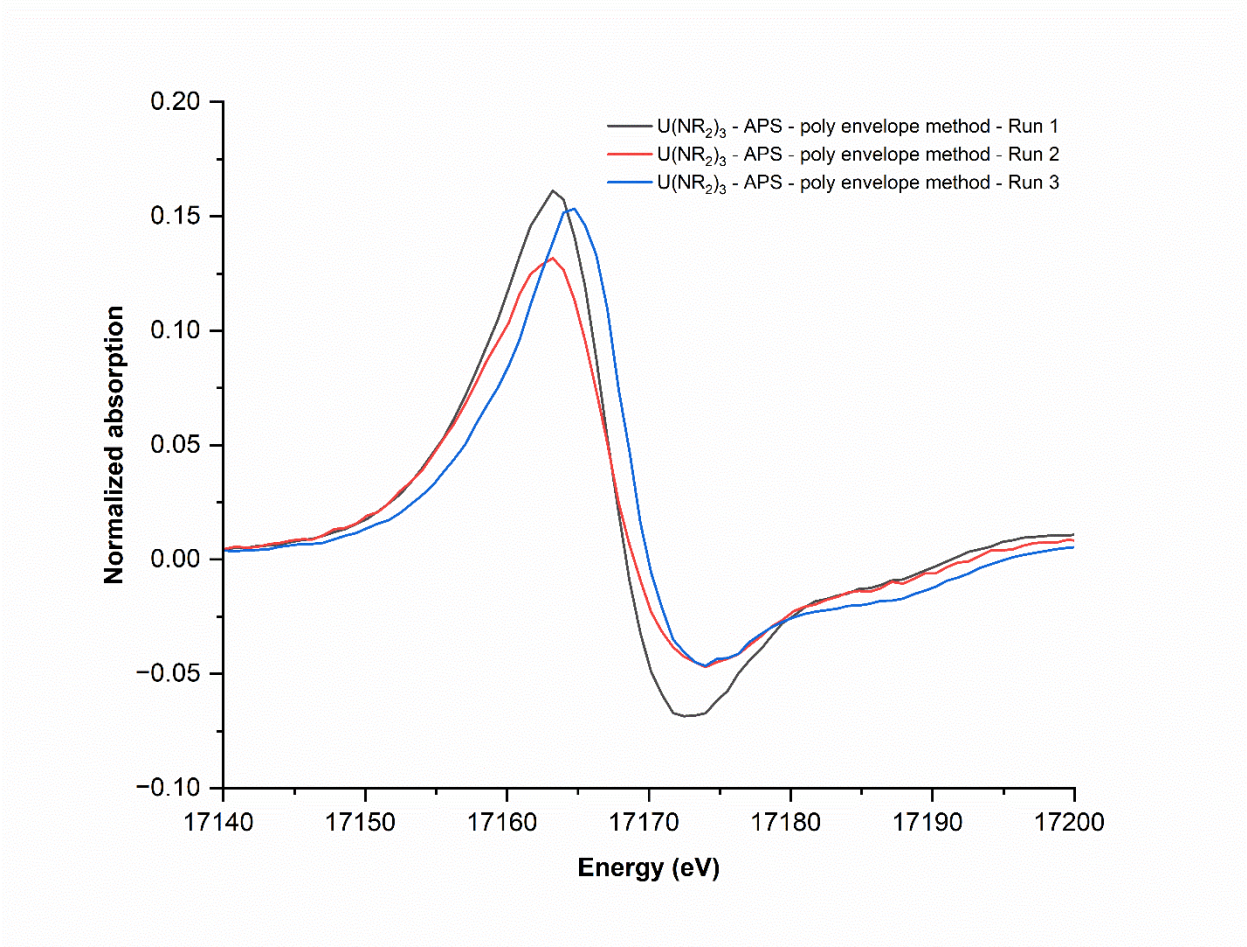

Fig. S2 Uranium L_3-edge XANES spectra, expressed as the first derivative, of three independently prepared compressed boron nitride pellets of **1**, each encapsulated using a previously reported method involving vacuum-sealed polyethylene envelopes (see **Figure S1**).[1] The spectra were collected across separate attempts and exhibit misaligned edge energies and variability in spectral features, consistent with adventitious oxidation during handling or prior to measurement. Data were previously collected at the APS at sector 10-BM and are referenced to yttrium foil (17038.4 eV).

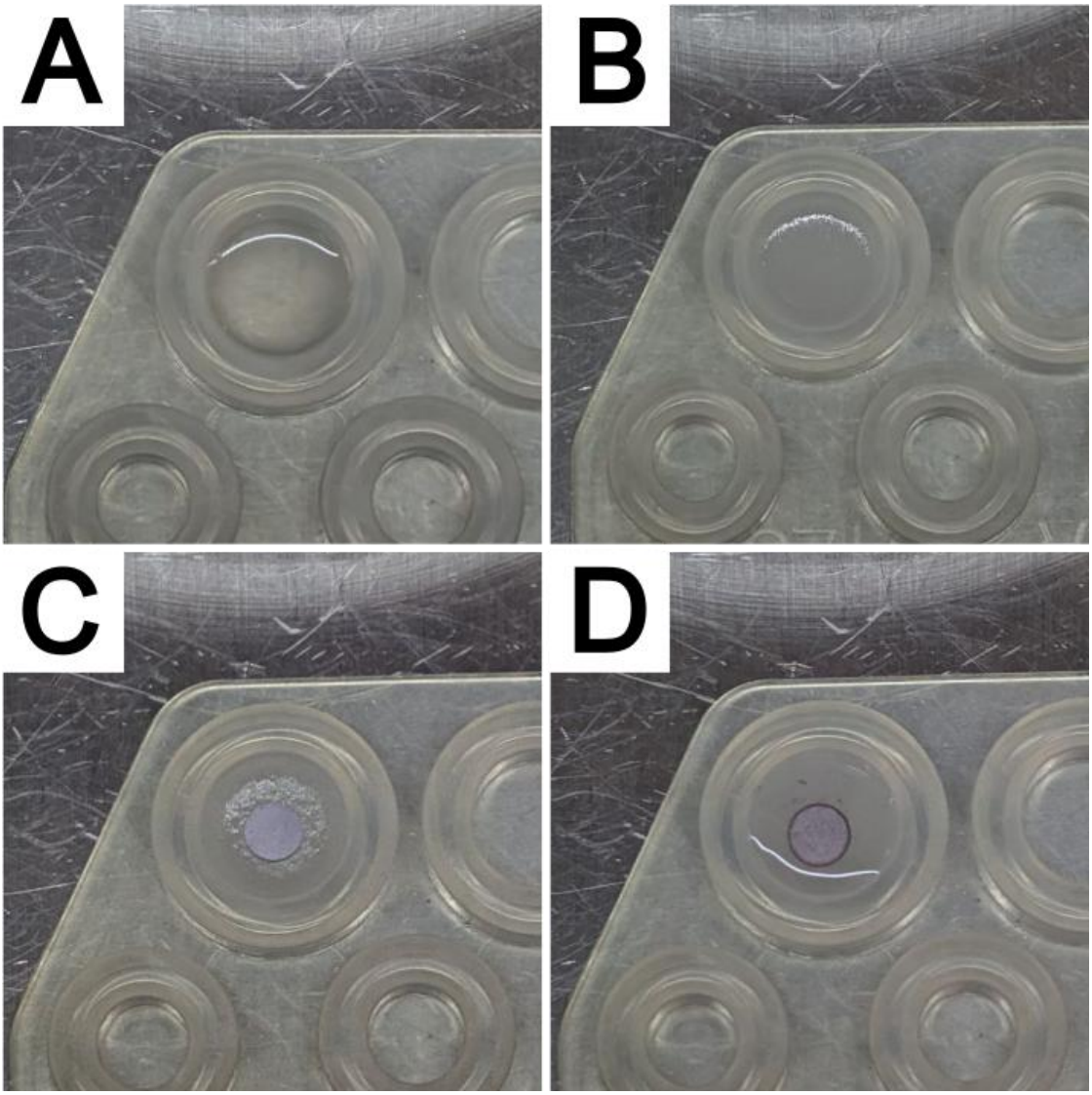

Fig S3 Stepwise preparation of a wax coin of **1**. A bottom layer of molten paraffin wax (**A**) is first partially cooled in a silicone ring mold (**B**), followed by placement of a pressed boron nitride pellet of **1** (**C**) and subsequent encapsulation with additional molten wax (**D**).

Fig S4 Top: Photographs showing stage alignment of the mounted sample rack at the beamline. **Bottom:** Centering of individual sample pellets (visible as dark circles). The partial translucency of the wax coins facilitates visual alignment and centering during measurement.

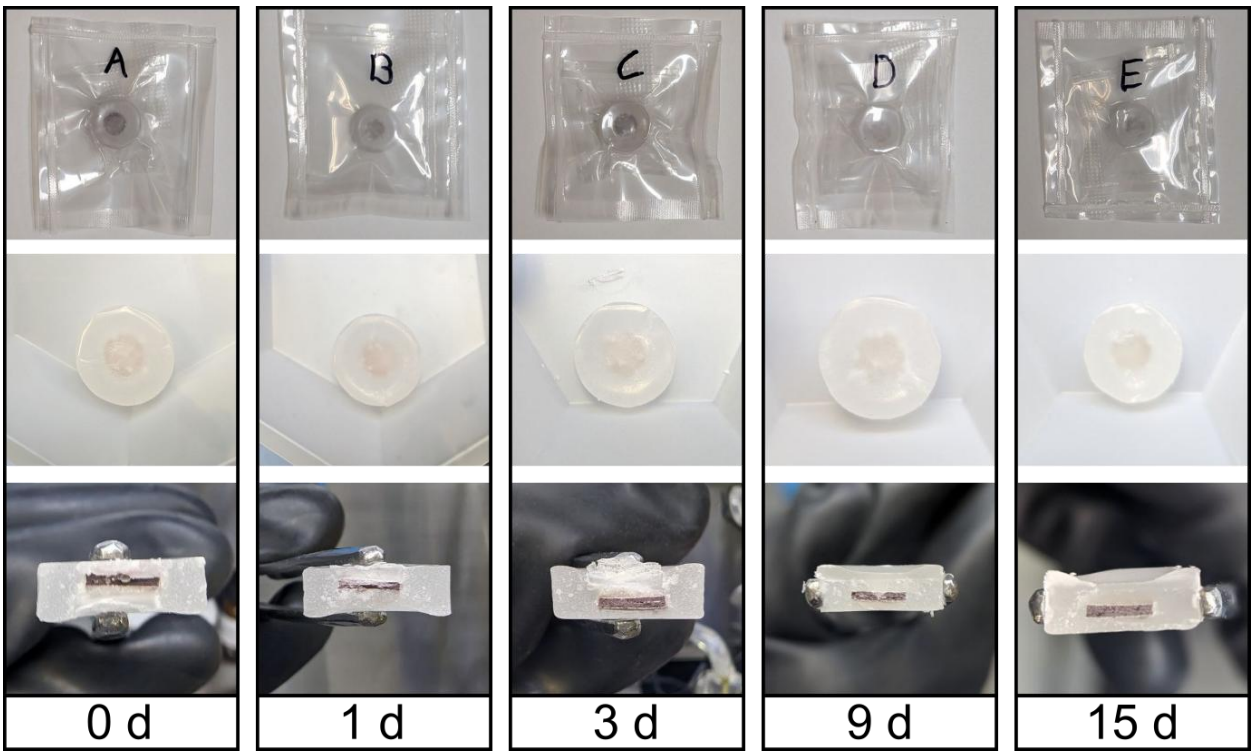

Fig S5 Top: Photograph showing a series of boron nitride pellets of **1**, prepared as wax coins, vacuum sealed within layers of food grade ethylene vinyl alcohol (EVOH, 3 mil, 0.0762 mm) and co-extruded polyethylene nylon (poly-nylon, 5 mil, 0.127 mm) polymers. **Bottom:** Extracted sample pellets and their corresponding cross-sections after storage of the sample pouches under ambient conditions for t = 0, 1, 3, 9, and 15 days.

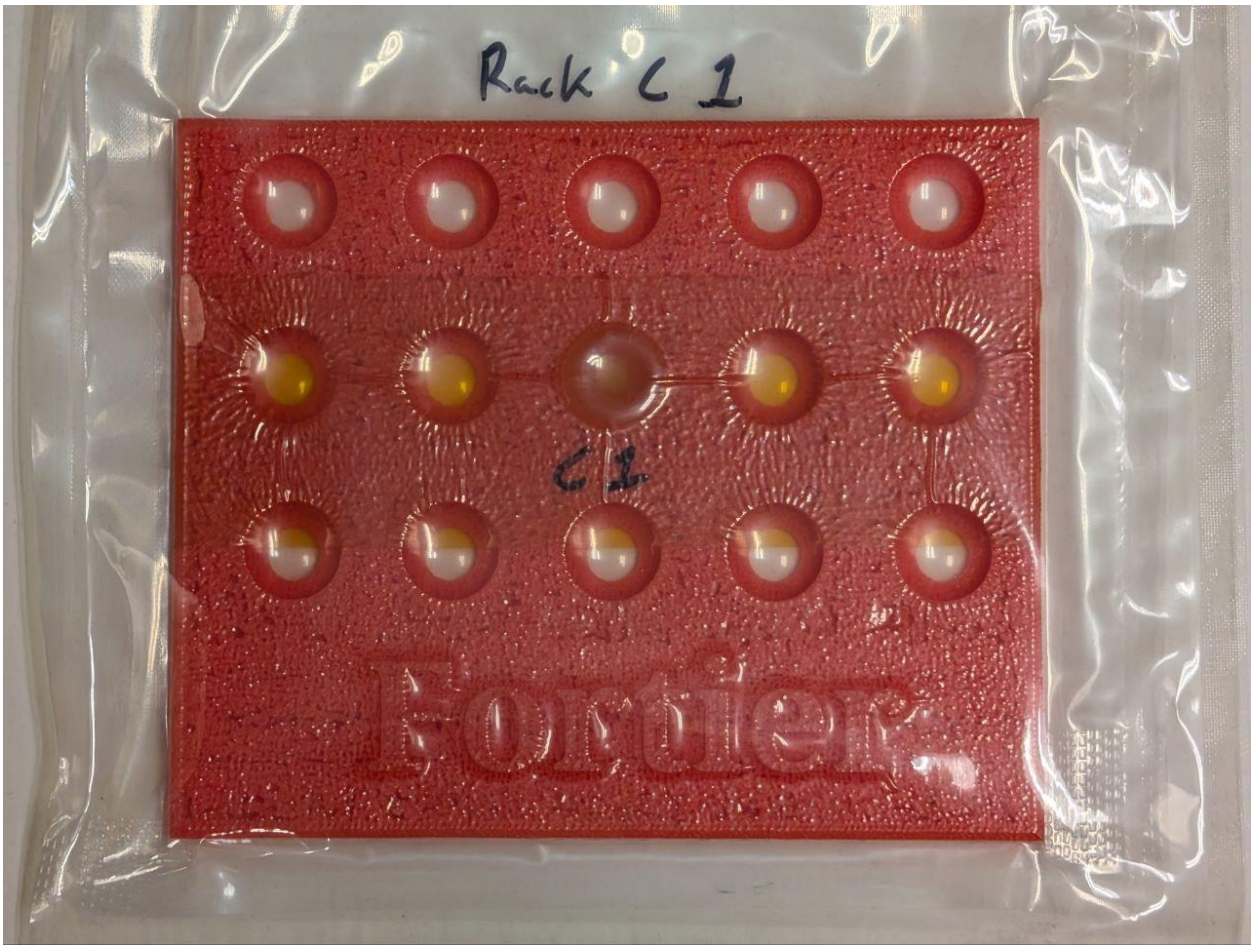

Fig. S6 Photograph of a wax coin fitted within a 3D-printed sample rack (160 mm × 145 mm × 7 mm), vacuum sealed within food-grade EVOH and poly-nylon films.

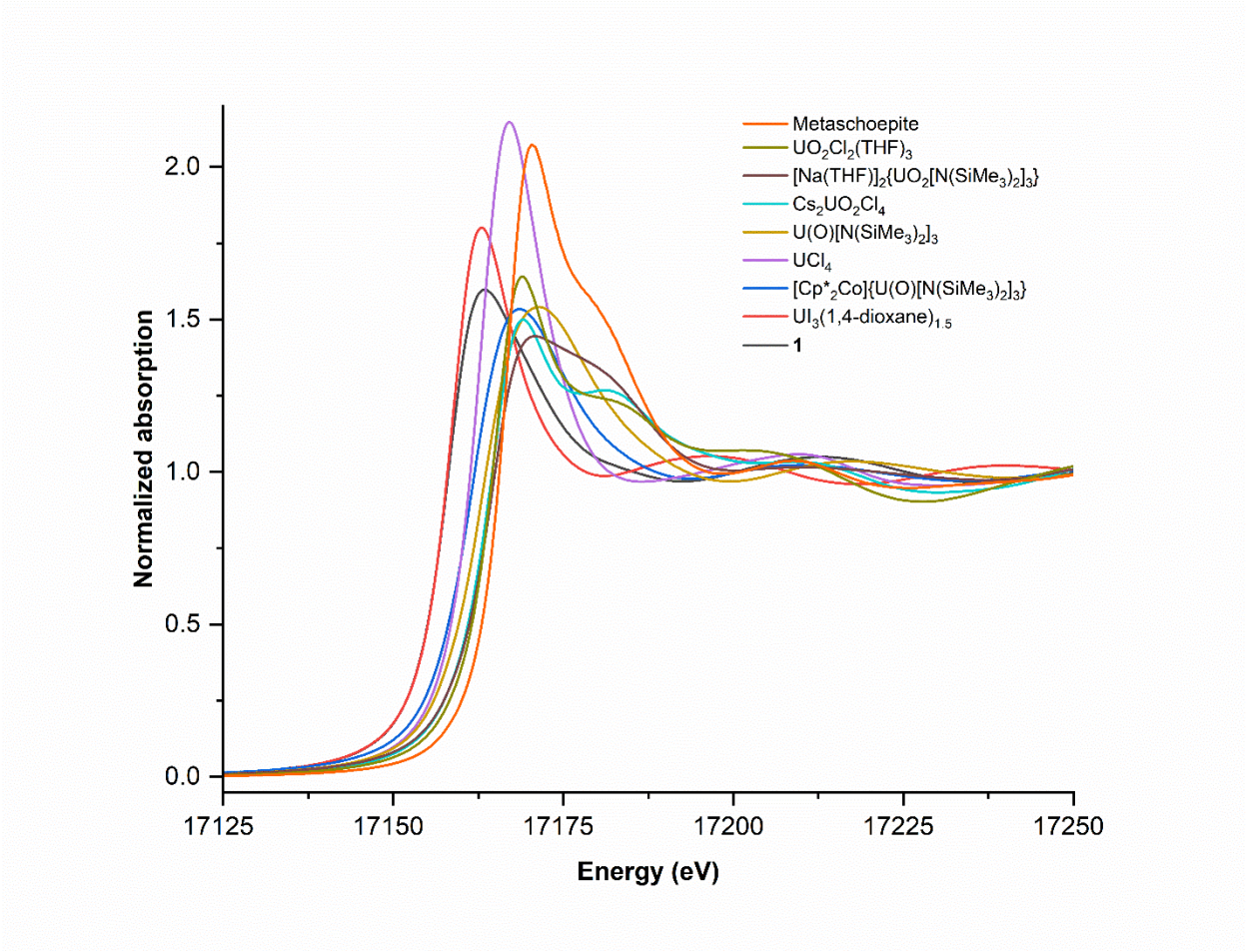

Fig. S7 Normalized uranium L_3-edge XANES spectral plots for **1** and selected reference compounds, each prepared as wax coins.

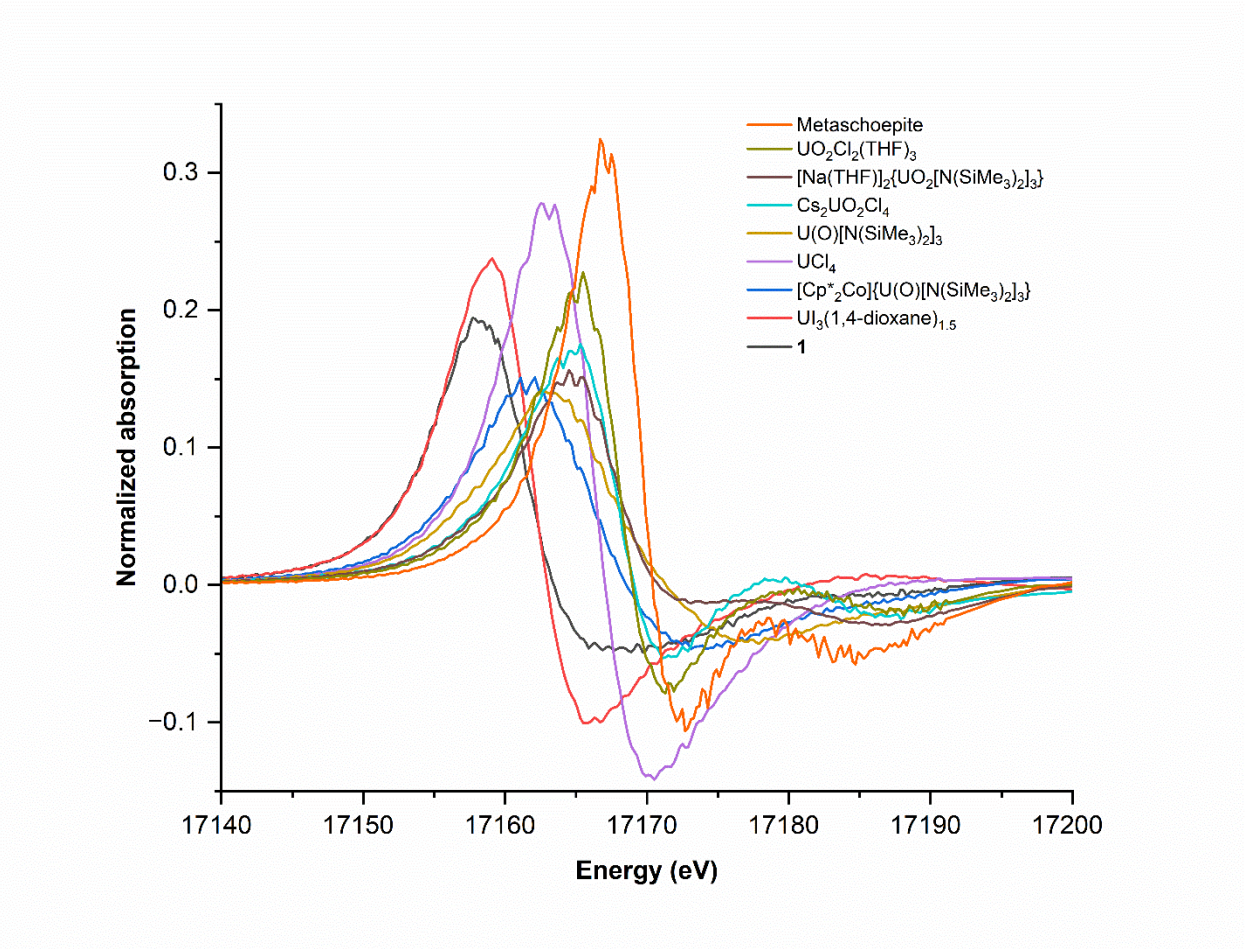

Fig. S8 First derivative uranium L_3-edge XANES spectral plots for **1** and selected reference compounds, each prepared as wax coins.

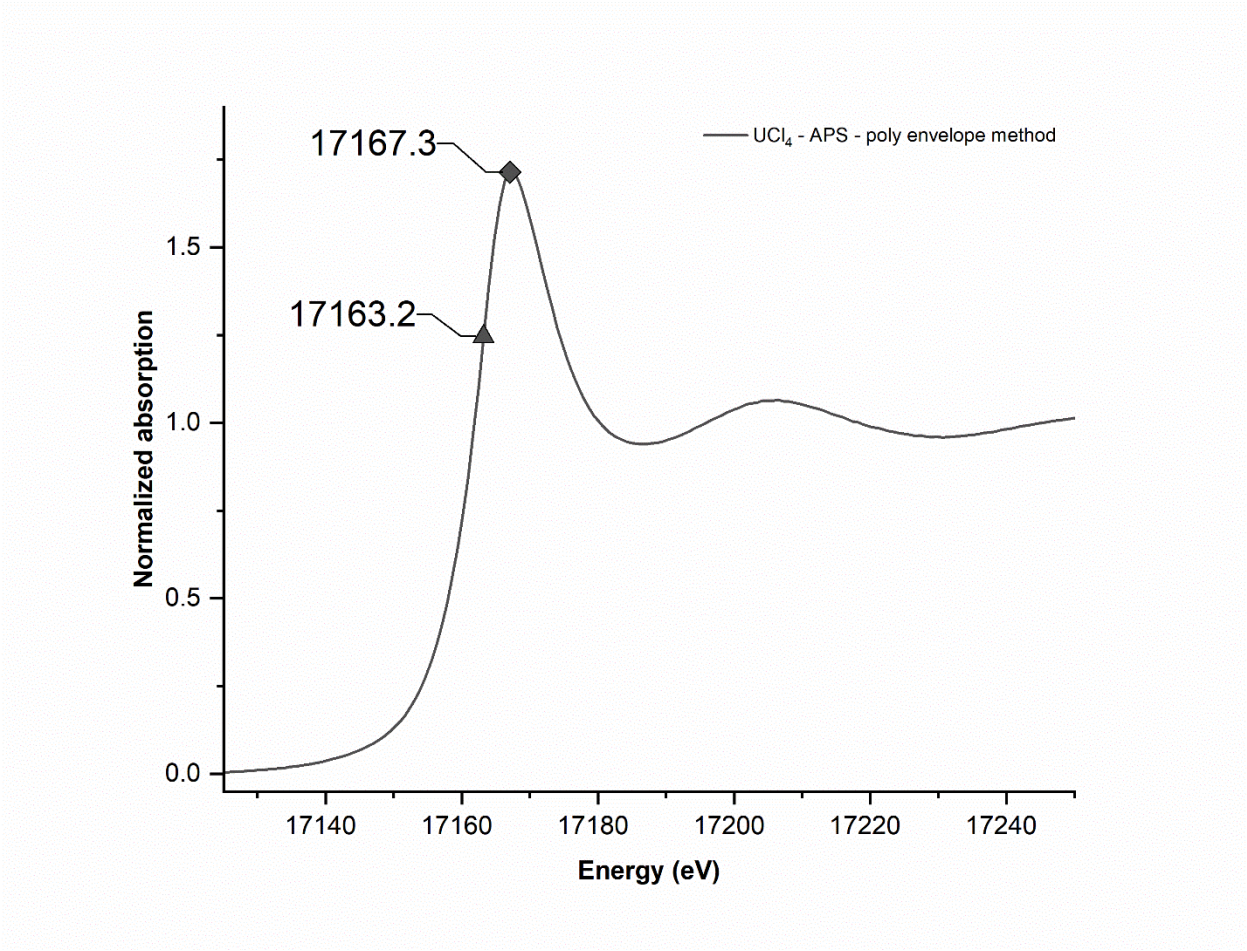

Fig. S9 Averaged uranium L_3-edge XANES spectra of three independently prepared UCl_4 samples, each dispersed in compressed boron nitride and encapsulated using our previously reported vacuum-sealed polyethylene envelope method.[1] Data were collected during a separate run at sector 10-BM of the APS and initially referenced to an yttrium foil (17,038.4 eV), followed by secondary alignment to the edge energy of the shared $UO_2Cl_2(THF)_3$ standard (17,165.1 eV). The resulting edge energy (17,163.2 eV) closely matches the value obtained using the wax coin method, supporting the reproducibility of the measurement and confirming the anomalous nature of our earlier reported UCl_4 value.[1]

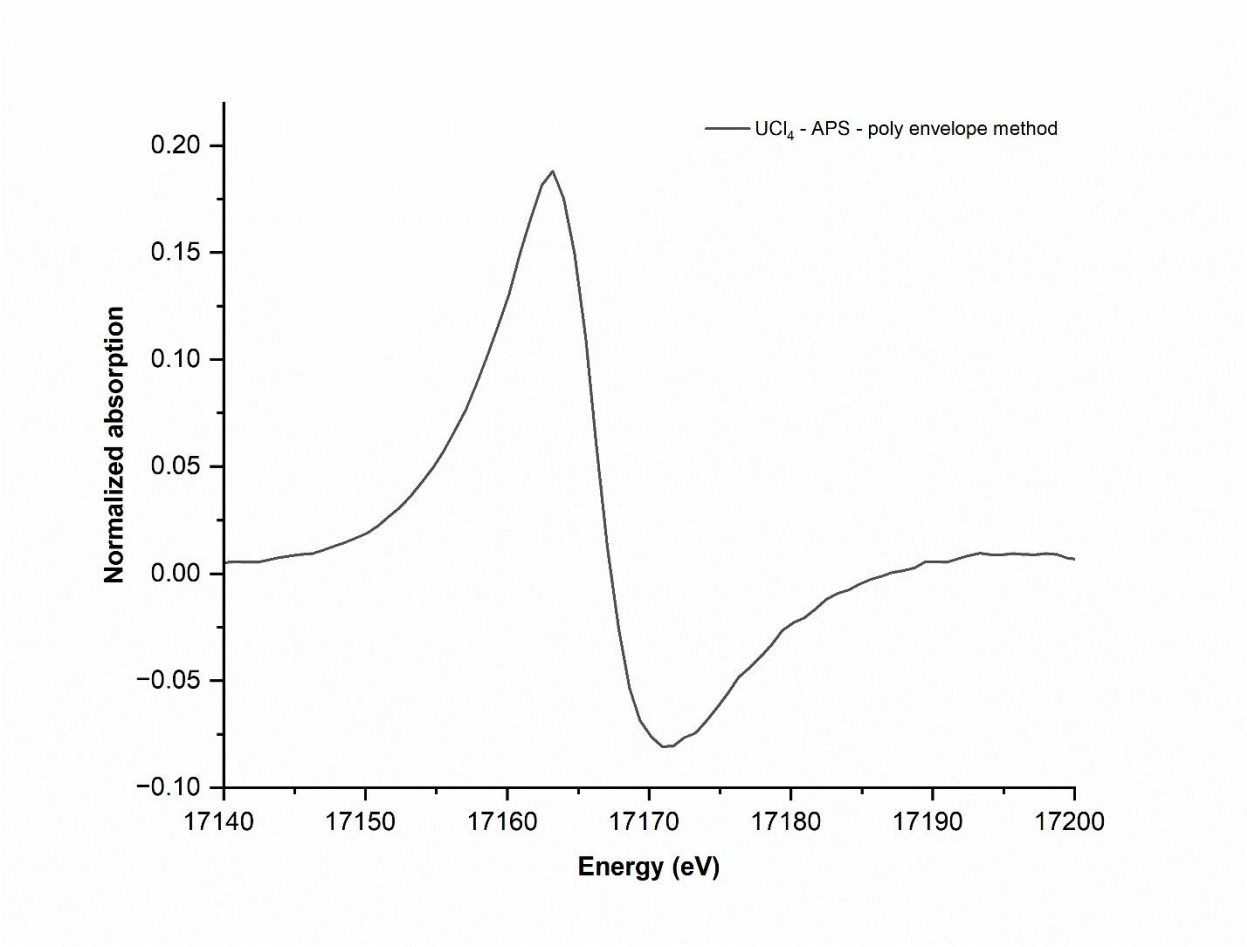

Fig. S10 First derivative uranium L_3-edge XANES spectra of three independently prepared UCl_4 samples, each dispersed in compressed boron nitride and encapsulated using our previously reported vacuum-sealed polyethylene envelope method.[1] Data were collected during a separate run at sector 10-BM of the APS and initially referenced to an yttrium foil (17,038.4 eV), followed by secondary alignment to the edge energy of the shared $UO_2Cl_2(THF)_3$ standard (17,165.1 eV). The resulting edge energy (17,163.2 eV) closely matches the value obtained using the wax coin method, supporting the reproducibility of the measurement and confirming the anomalous nature of our earlier reported UCl_4 value.[1]

Reference

1 J. Murillo, R. Bhowmick, K. L. M. Harriman, A. Gomez-Torres, J. Wright, R. W. Meulenberg, P. Miró, A. Metta-Magaña, M. Murugesu, B. Vlaisavljevich and S. Fortier, Actinide arene-metalates: ion pairing effects on the electronic structure of unsupported uranium–arenide sandwich complexes, *Chem. Sci.*, 2021, **12**, 13360-13372.