Supporting Information

Flux Growth of $[NaK_6F][(UO_2)_3(Si_2O_7)_2]$ and $[KK_6Cl][(UO_2)_3(Si_2O_7)_2]$: The Effect of Surface Area to Volume Ratios on Reaction Products

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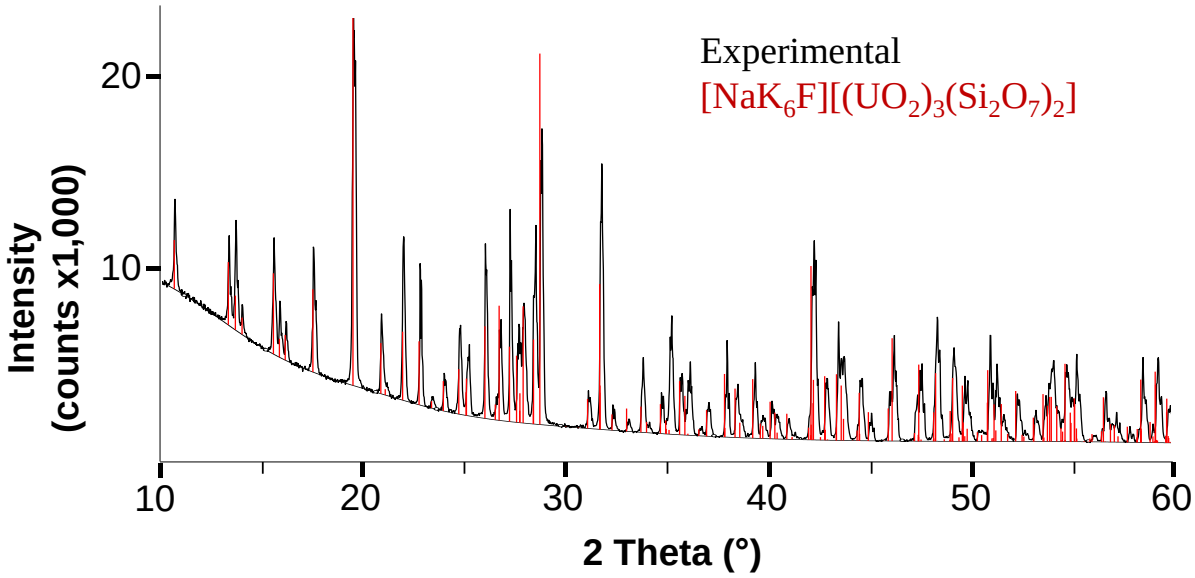


Figure S1. Powder diffraction pattern for $[NaK_6F][(UO_2)_3(Si_2O_7)_2]$.

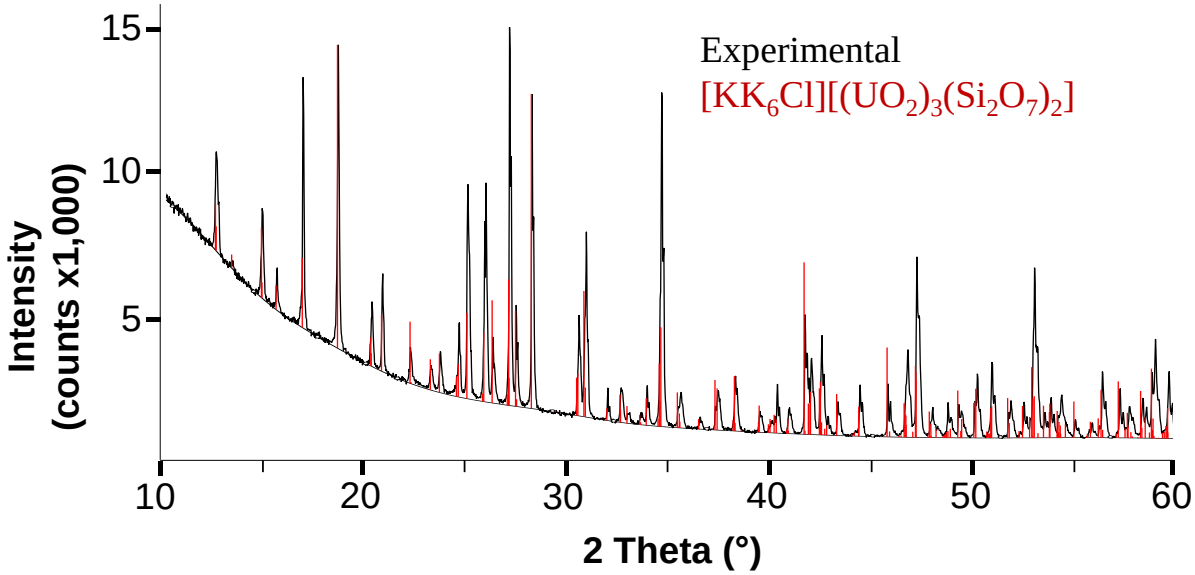


Figure S2. Powder diffraction pattern for $[KK_6Cl][(UO_2)_3(Si_2O_7)_2]$.

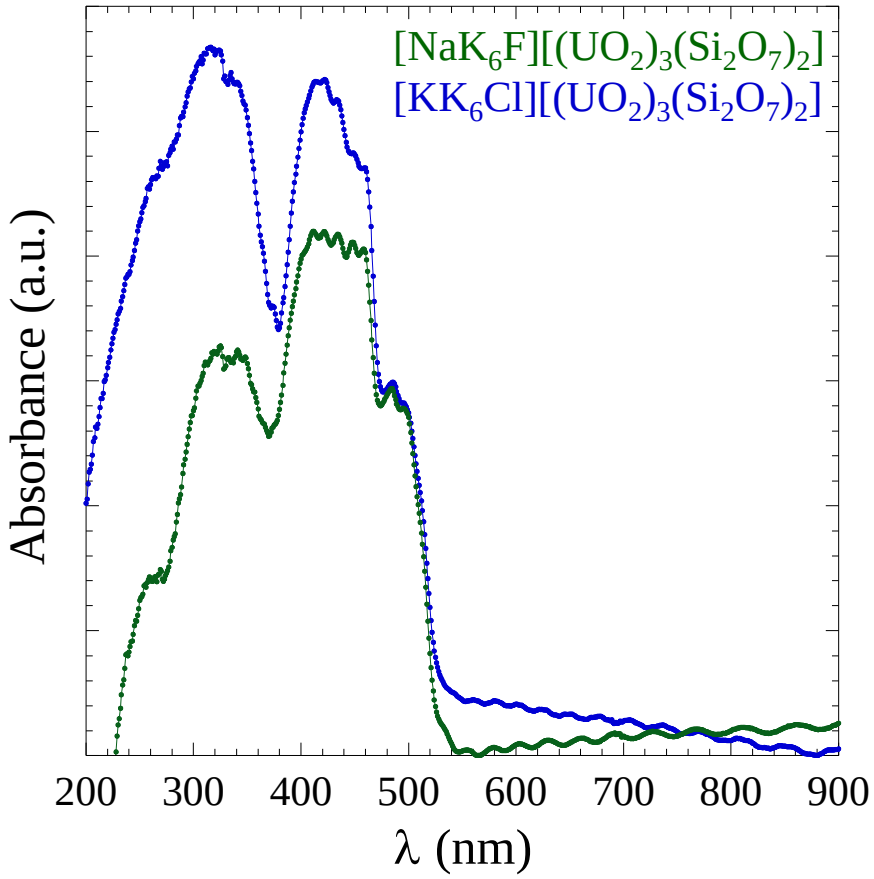
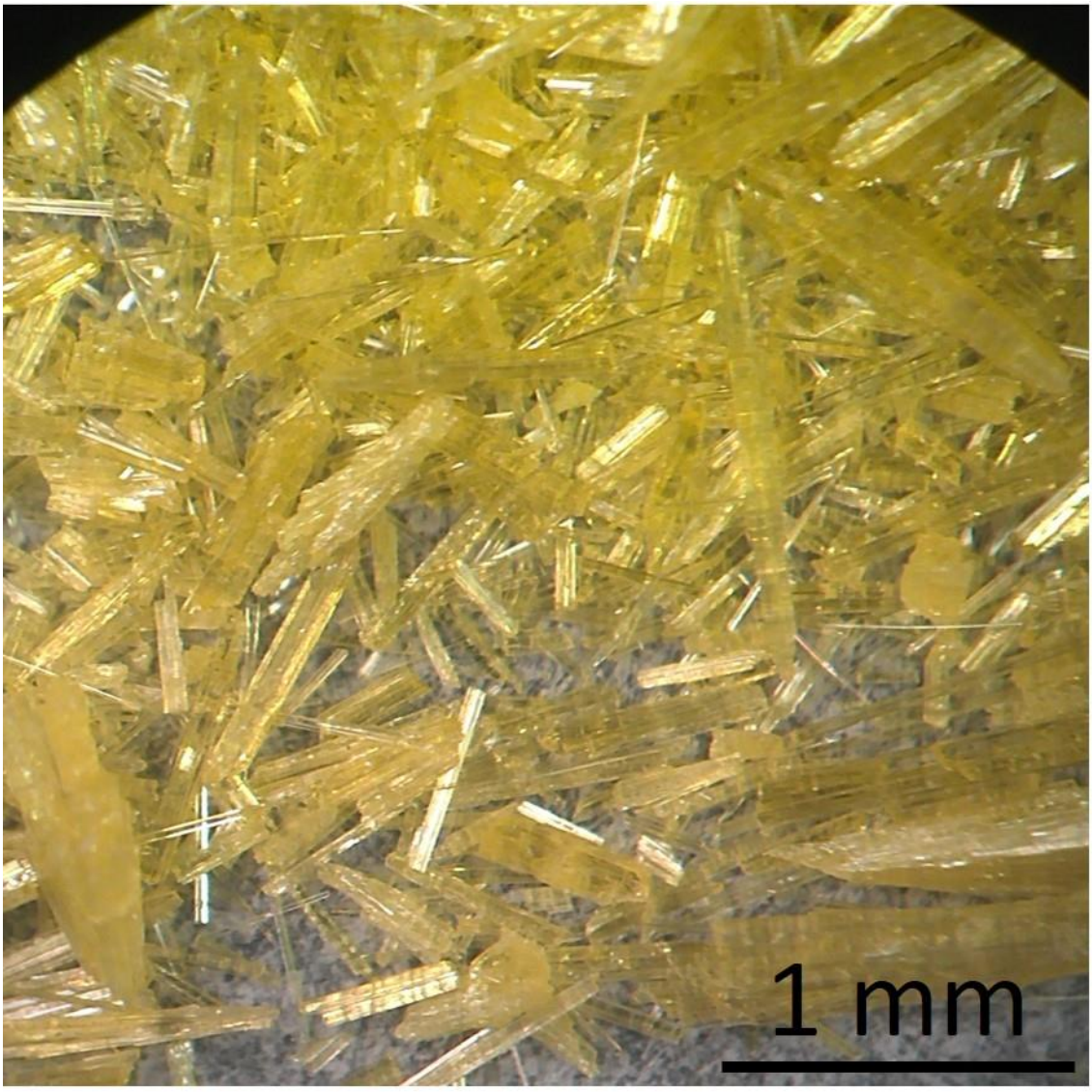


Figure S3. UV-vis absorbance spectra for $[NaK_6F][(UO_2)_3(Si_2O_7)_2]$ and $[KK_6Cl][(UO_2)_3(Si_2O_7)_2]$.

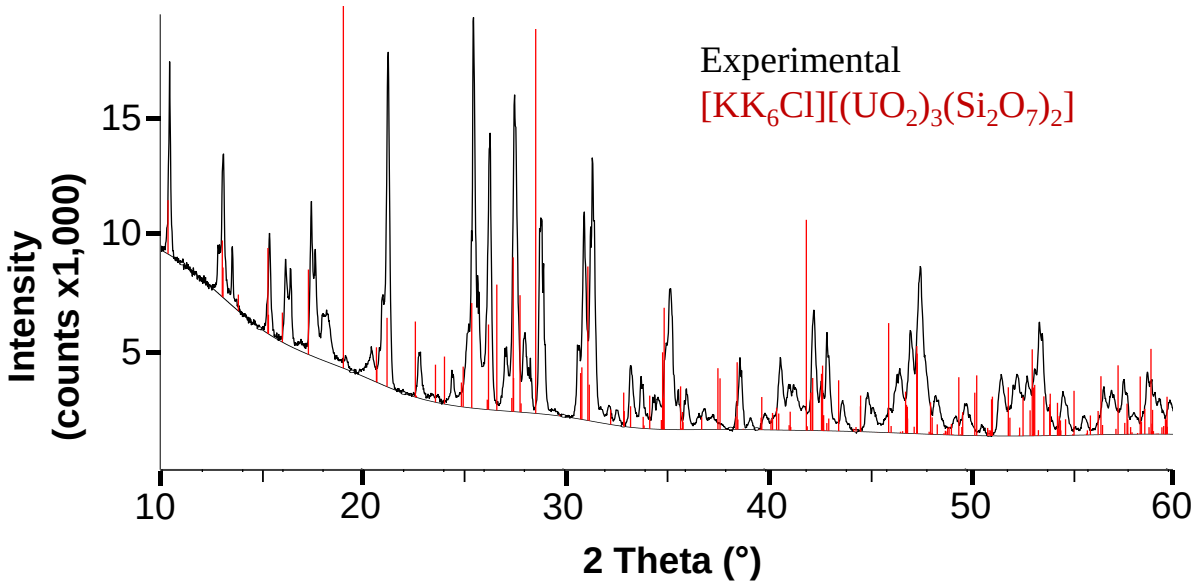
Further information on the unsolved salt-inclusion phase:

Reaction Conditions: For the synthesis of the unsolved salt-inclusion phase, a mixture of 1/3 mmol U_3O_8 and 4/3 mmol SiO_2 was loaded into a cylindrical silver crucible with dimensions 1.2 cm diameter x 5.7 cm height. The U/Si mixture was covered with a mixture of KF/KCl (35 mmol:5 mmol) and the crucible was loosely covered with a silver lid. The reaction was heated to 900 °C in 1.5 h, dwelled at this temperature for 24 h., and slow cooled to 600 °C at 6 °C/h, at which temperature the furnace was shut off.



The above figure shows crystals of the unsolved salt-inclusion phase. These crystals form as pale yellow needles which are striated along the needle axis. In some cases, these needles appear to be made up of lots of individual very thin crystals. Despite good optical quality of some of the thin crystals, single crystal X-ray diffraction collected on multiple thin crystals using a Bruker D8 QUEST diffractometer

equipped with an Incoatec microfocus Mo Kα source and a PHOTON 100 CMOS area detector, displayed sharp peaks within broad streaked peaks indicating poor crystal quality.



Powder diffraction data (shown above), shows some similarities to $[KK_6Cl][(UO_2)_3(Si_2O_7)_2]$. However, there are clearly some missing and additional peaks, along with many shifted peaks. The shifting of the peaks may suggest a smaller unit cell than $[KK_6Cl][(UO_2)_3(Si_2O_7)_2]$. Attempt to index the sharp peaks within the single crystal diffraction pattern suggested two potential unit cells, a *c*-centered orthorhombic cell with a = 7.84 Å, b = 21.97 Å, and c = 13.63 Å or a primitive monoclinic cell with a = 7.83 Å, b = 13.26 Å, c = 11.66 Å, and β = 109.6°.