Supporting Information

High-Temperature, High-Pressure Hydrothermal Synthesis, Characterization, and Structural Relationships of Layered Uranyl Arsenates

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Figure S1. The observed and simulated X-ray powder patterns of Na₁₄[(UO₂)₅(AsO₄)₈]·2H₂O.

Figure S2. The observed and simulated X-ray powder patterns of $K_6[(UO_2)_5O_5(AsO_4)_2]$. Asterisks denote the minor phase of $K_4[(UO_2)_3O_2(AsO_4)_2]$.

Figure S3. The observed and simulated X-ray powder patterns of Rb₄[(UO₂)₃O₂(AsO₄)₂].

Figure S4. The observed and simulated X-ray powder patterns of $Cs_6[(UO_2)_5O_2(AsO_4)_4]$.

Figure S5. The EDS spectra of $Na_{14}[(UO_2)_5(AsO_4)_8] \cdot 2H_2O$, $K_6[(UO_2)_5O_5(AsO_4)_2]$,

 $K_4[(UO_2)_3O_2(AsO_4)_2], \ Rb_4[(UO_2)_3O_2(AsO_4)_2], \ and \ Cs_6[(UO_2)_5O_2(AsO_4)_4].$

Figure S6. Thermogravimetric analysis of $Na_{14}[(UO_2)_5(AsO_4)_8] \cdot 2H_2O$ in flowing nitrogen gas at 2 °C/min.

Figure S7. Room-temperature fluorescence spectrum of 1 excited at 360 nm.



Figure S1



____ observed



Figure S2



Figure S3



Figure S4



 $Na_{14}[(UO_2)_5(AsO_4)_8] \cdot 2H_2O$ (compound 1)



 $K_6[(UO_2)_5O_5(AsO_4)_2]$ (compound 2a)



 $K_4[(UO_2)_3O_2(AsO_4)_2]$ (compound **2b**)



 $Rb_4[(UO_2)_3O_2(AsO_4)_2]$ (compound 3)



 $Cs_6[(UO_2)_5O_2(AsO_4)_4]$ (compound 4)

Figure S5



Figure S6



Figure S7