Supplementary Information

Effect of Synthetic Parameters toward Nanocrystals of Potassium Transition Metal Fluorides Using Colloidal Chemistry

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Synthesis of $KFeF_3$ nanocrystals from a double fluorine source

The colloidal synthesis route had different reactants and consisted of two major steps: making iron trifluoroacetate and then mixing with potassium trifluoroacetate to produce $KFeF_3$. Fe (Sigma Aldrich, 99 %, powder), trifluoroacetic acid (99 %, Sigma Aldrich), potassium trifluoroacetate (98 %, Sigma Aldrich), 1-ocatadecene (tech. grade, 90 %, Sigma Aldrich), Oleic Acid (tech. grade, 90 %, Sigma Aldrich) and Oleylamine (ACROS ORGANICS) were used as received without further purifications. 1 g Fe powder was added to 15 mL trifluoroacetic acid, and the mixture was heated at 10 °C for at least 24 h. The solution darkened after 24 h, and was then left at 100 °C to evaporate in order to obtain the iron trifluoroacetate powder. Then, 1 mmol each of iron trifluoroacetate powder and potassium trifluoroacetate were added to 20 mmol of 1-octadecene and a mixture of 13.3 mmol oleic acid and 6.67 mmol oleylamine in a three-neck flask at room temperature. The mixture was heated to 120 °C for 0.5 h, followed by another heating step at 280 °C for 0.5 h under N_2 flow to obtain a dark solution. After the resulting solution was allowed to cool below 75 °C, excess amount of ethanol was added to precipitate out the reaction product, which were then washed at least three times with ethanol and hexane alternatively. The product was then dried for characterizations.

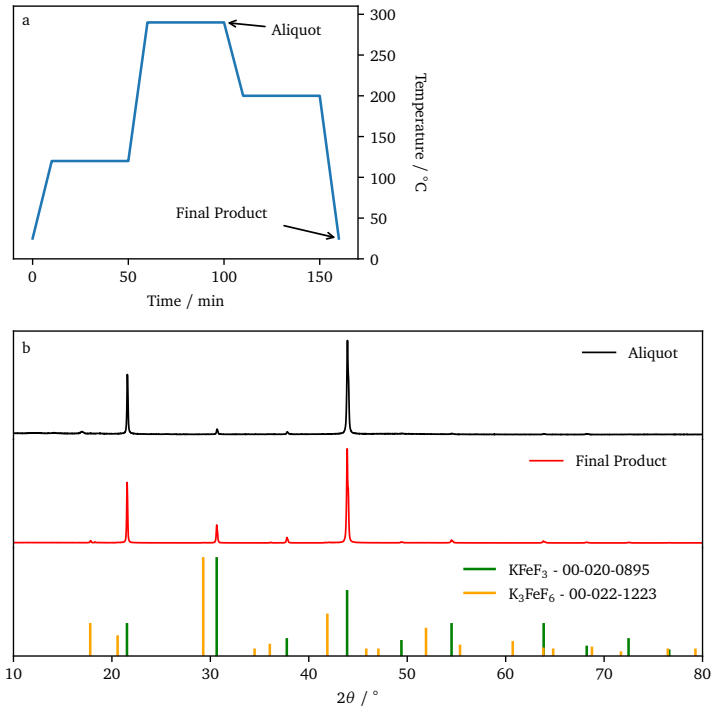

Figure S1 Temperature profile of the experiment denoting where the aliquot and final product were collected (a), and powder x-ray diffraction (XRD) patters showing the composition of these products (b).

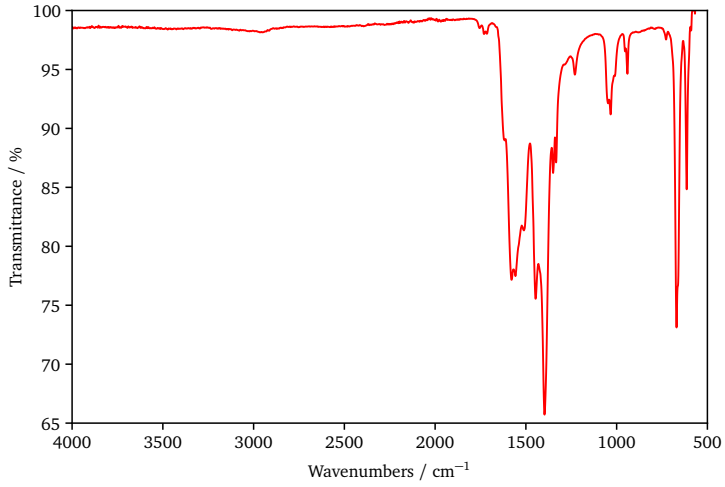

Figure S2 Infrared (IR) spectroscopy of $Fe(CH_3COO)_2$ precursor.

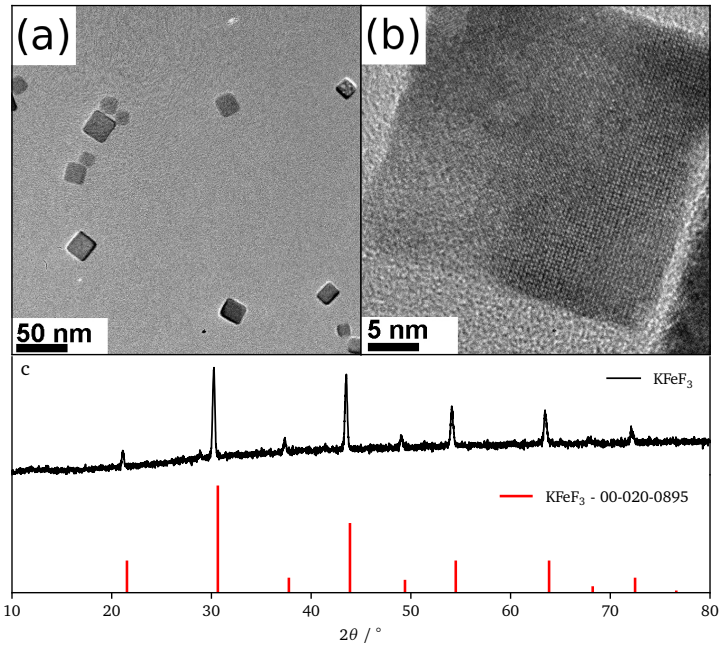

Figure S3 Transmission electron microscopy (TEM) images (a and b) and XRD spectra (c) of $KFeF_3$ nanocrystals synthesized from a double fluorine source method.

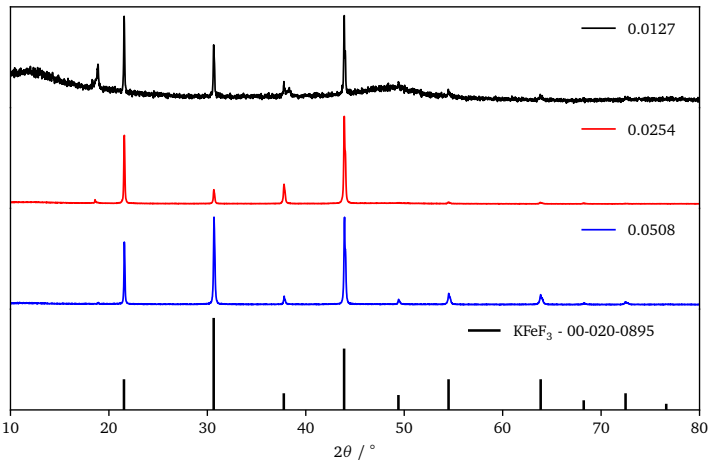

Figure S4 XRD patterns showing the co-thermolysis product, $KFeF_3$, at different molar concentrations of iron precursor in the reaction mixture ($\frac{Fe}{OA+OM}$). The precursor stoichiometry ($\frac{K}{Fe}$) was kept constant during these experiments.

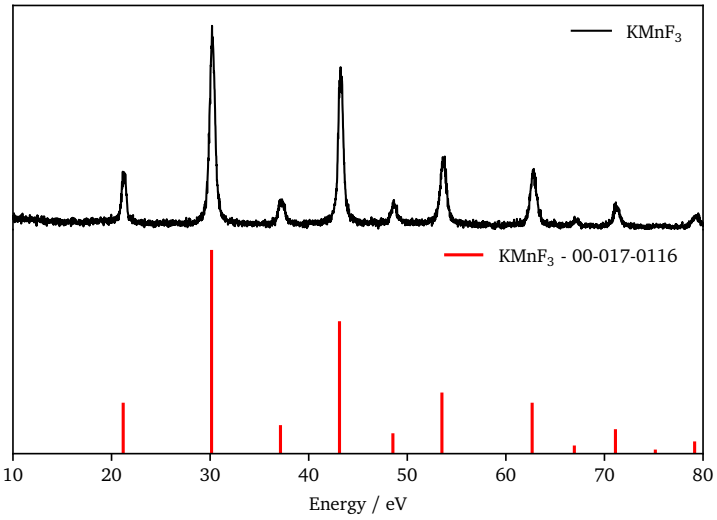

Figure S5 XRD pattern of hot-injection product, $KMnF_3$

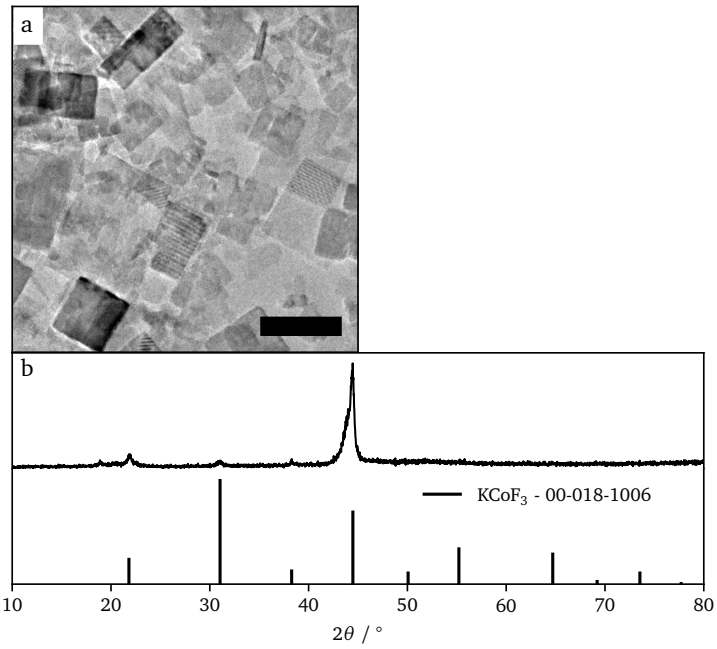

Figure S6 TEM image and XRD pattern of hot-injection product, $KCoF_3$, with stoichiometric ratio $\frac{K}{Co}$=3.00. Scale bar represents 50 nm

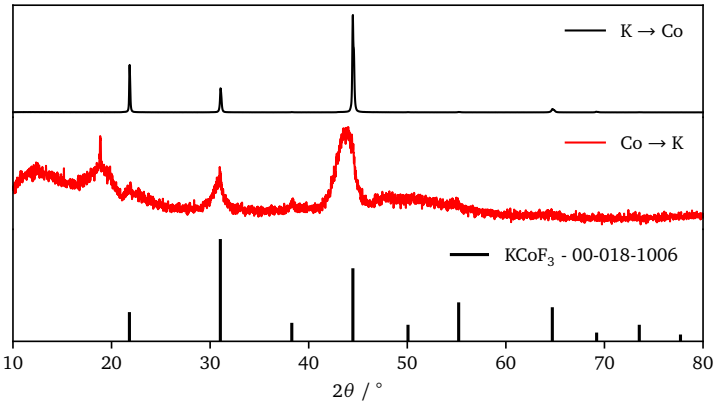

Figure S7 XRD patterns of hot-injection product, $KCoF_3$, with varying the mobile and static hot-injection solutions.

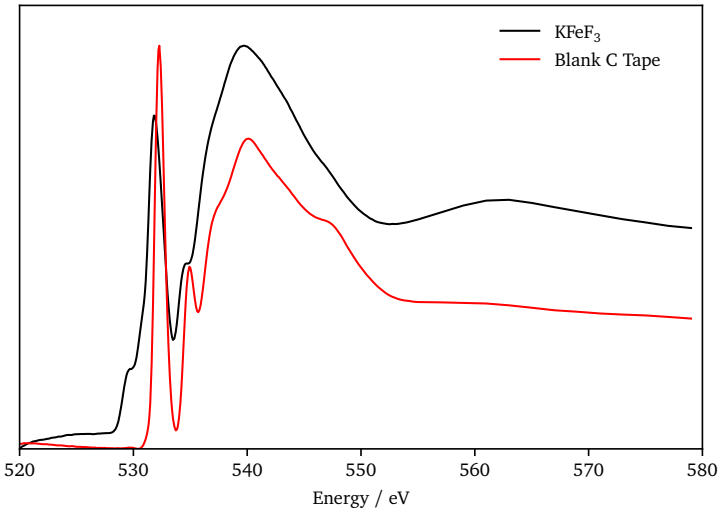

Figure S8 Soft x-ray absorption spectroscopy (XAS) spectra (total electron yield (TEY) signal) showing similar patterns between $KFeF_3$ (black) and carbon tape substrate (red) oxygen *K*-edge data.

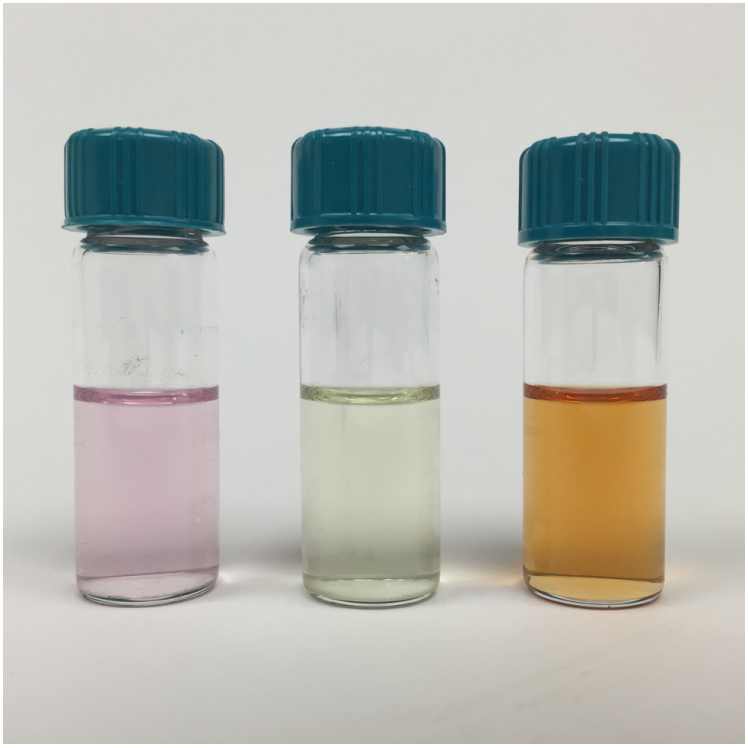

Figure S9 $KCoF_3$ (left), $KNiF_3$ (center) and $KFeF_3$ (right) nanoparticles dispersed in hexane. Colours of the solution are representative of the individual dry powders.

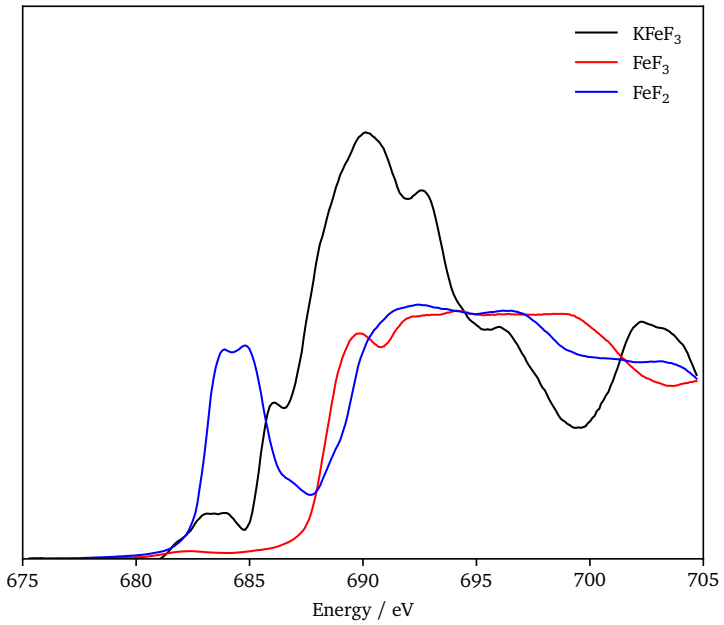

Figure S10 F *K*-edge data (total fluorescence yield (TFY)) of $KFeF_3$ (black), FeF_3 (red), and FeF_2 (blue) taken at the same beamline (4-ID-C, Advanced Photon Source, Argonne National Laboratory.)