

Synthesis, phase characterization and crystal structure comparison of a self-made SmF_2 – SmFCl – SmFO mixture by XRD and EDX

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In an experiment to obtain single crystals of SmF_2 [1–3], a mixture of Sm, SmF_3 and NaCl (as flux) was heated up inside a sealed niobium capsule to 850 °C for four days and slowly cooled down with 5 K/h. In addition to dark red single crystals of the target compound SmF_2 , also orange single crystals of SmFCl [4] were obtained. A PXRD experiment showed an additional phase, which was characterized as SmFO [5]. The ratio of them was determined via the FULLPROF SUITE by PXRD (Cu- K_α radiation) as about 70:5:25. By EDX analysis, the stoichiometry of all three named compounds was confirmed. SCXRD experiments (Mo- K_α radiation) were performed with single crystals (red SmF_2 : CSD-2087284, orange SmFCl : CSD-2087285). While SmF_2 and SmFO crystallize with the CaF_2 -type structure (cubic; $Fm\bar{3}m$; PXRD-data: $a = 579.62(3)$ pm and $a = 556.31(3)$ pm CSD-2087286 for SmFO), SmFCl adopts the PbFCl -type structure (tetragonal, $P4/nmm$; $a = 413.7(1)$ pm and $c = 699.1(3)$ pm). The unit-cell parameters from the SCXRD measurements of SmF_2 ($a = 580.31(4)$ pm) and SmFCl ($a = 413.59(5)$ pm and $c = 699.34(8)$ pm) show a good agreement to them of the PXRD experiment. The charge of the samarium cations in the named compounds was calculated by bond-valence calculations [6] and unambiguously led to Sm^{2+} in SmF_2 and SmFCl , but to Sm^{3+} in SmFO . The measured powder pattern of the three-component mixture can be seen in Figure 1 together with single crystals of SmF_2 and SmFCl and all three unit cells of the title compounds.

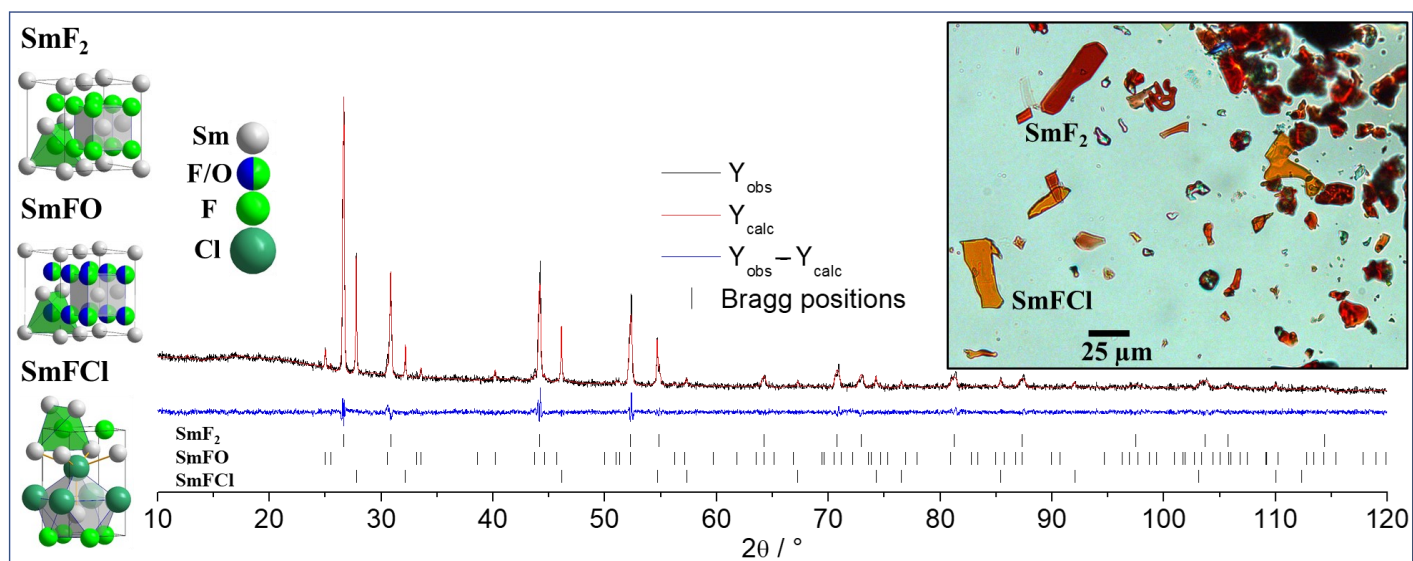


Figure 1. Rietveld refinement of a SmF_2 – SmFCl – SmFO mixture (70:5:25) by using Cu- K_α radiation (*bottom*), unit cells of SmF_2 , SmFO and SmFCl (*left*) and single crystals of SmF_2 and SmFCl (*top right*).

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