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SUPPLEMENTARY MATERIALS

Antimony Oxofluorides
–
a synthesis concept that yield phase pure samples and single crystals

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Table S1: Weight in amount of the reactants for synthesis of the compounds Sb_3O_4F, $Y_{0.5}Sb_{2.5}O_4F$, M-SbOF and α-$Sb_3O_2F_5$.

Compounds	Reactants				
	Sb_2O_3 (g)	SbF_3 (g)	YF_3 (g)	H_2O (ml)	HF (drop)
Sb_3O_4F	0.292	0.045	--	1	--
$Y_{0.5}Sb_{2.5}O_4F$	0.876	--	0.146	1	Added
M-SbOF	0.148	0.090	--	1	--
α-$Sb_3O_2F_5$	0.148	0.090	--	1	Added

Table S2: Bond Valence sum calculations for Sb_3O_4F. R_0-values are from Brown[12]; R_0(Sb–O) = 1.97 Å, R_0(Sb–F) = 1.88 Å, b = 0.37 Å.

Atoms	V
Sb1	2.9
Sb2	3.0
Sb3	3.1
F1	0.8
O2	2.3
O3	2.2
O4	2.1
O5	2.2

Table S3: Bond Valence sum calculations for M-SbOF and $Sb_3O_2F_5$. R_0-values are from Brown[1]; R_0(Sb–O) = 1.97 Å, R_0(Sb–F) = 1.88 Å, b = 0.37 Å.

Compounds	Atoms	V
M-SbOF	Sb1	2.9
	Sb2	3.0
	F1	0.8
	F2	1.3
	O1	2.2
	O2	2.1
$Sb_3O_2F_5$	Sb1	3.0
	Sb2	3.0
	Sb3	3.0
	Sb4	2.8
	O1	2.2
	O2	2.3
	F1	1.0
	F2	0.9
	F3	1.0
	F4	0.9
	F5	0.9

Table S4: Selected atomic distances for Sb_3O_4F and $Y_{0.49}Sb_{2.51}O_4F$.

Atom pair	d (Å) in $Y_{0.49}Sb_{2.51}O_4F$	d (Å) in Sb_3O_4F
Sb1 – O2	1.992 (3)	1.985 (2)
Sb1 – O3	2.011 (3)	2.001 (2)
Sb1 – O5	1.991 (3)	1.976 (2)
Sb1 ··· F1	2.645 (3)	2.630 (2)
Sb1 ··· O3	2.603 (3)	2.586 (2)
Sb2 – F1	2.060 (3)	2.050 (2)
Sb2 – O2	2.029 (3)	2.022 (2)
Sb2 – O2	2.288 (3)	2.266 (2)
Sb2 – O3	1.956 (3)	1.944 (2)
Sb2 ··· O5	2.680 (3)	2.679 (2)
Sb3 – O4	1.977 (3)	1.970 (2)
Sb3 – O4	1.978 (3)	1.964 (2)
Sb3 – O5	1.972 (3)	1.953 (2)
Sb3 ··· O4	2.918 (3)	2.904 (2)
Sb3 ··· F1	2.890 (3)	2.877 (2)

Figure S1: Comparison of PXRD data with the calculated diffractogram from the single crystal XRD data for $Y_{0.49}Sb_{2.51}O_4F$.

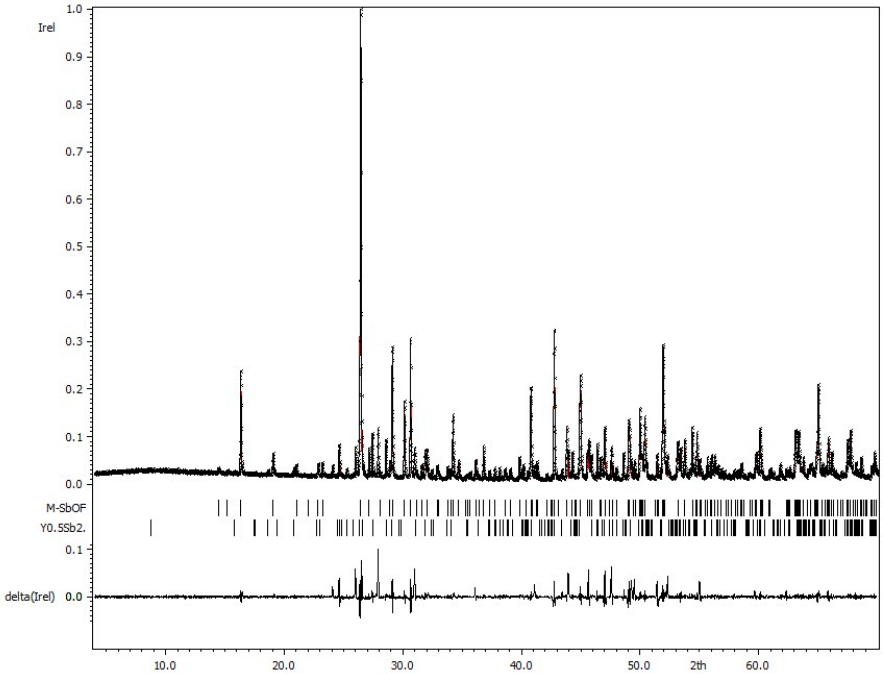

Figure S2: Comparison of PXRD data with the calculated diffractogram from the single crystal XRD data for $Sb_3O_2F_5$.

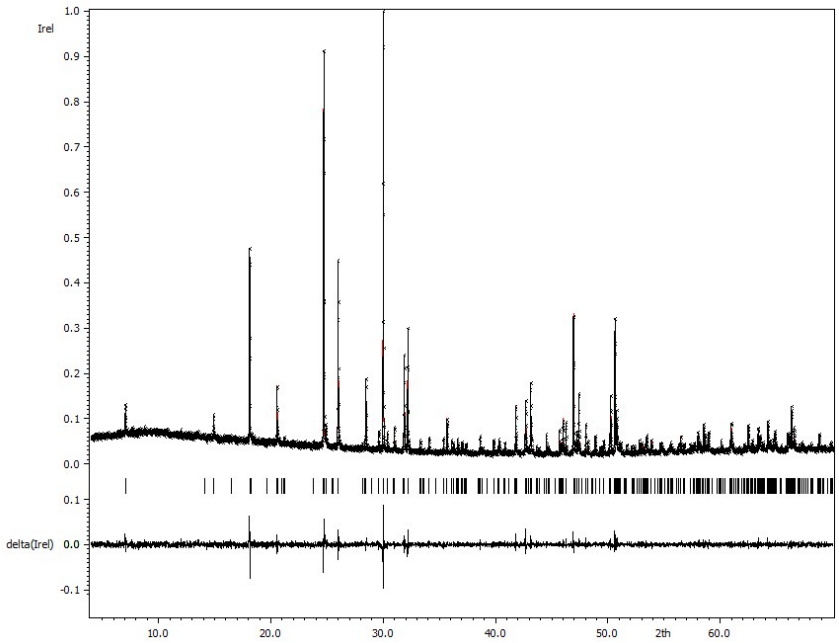

Figure S3: Comparison of PXRD data with the calculated diffractogram from the single crystal XRD data for M-SbOF.

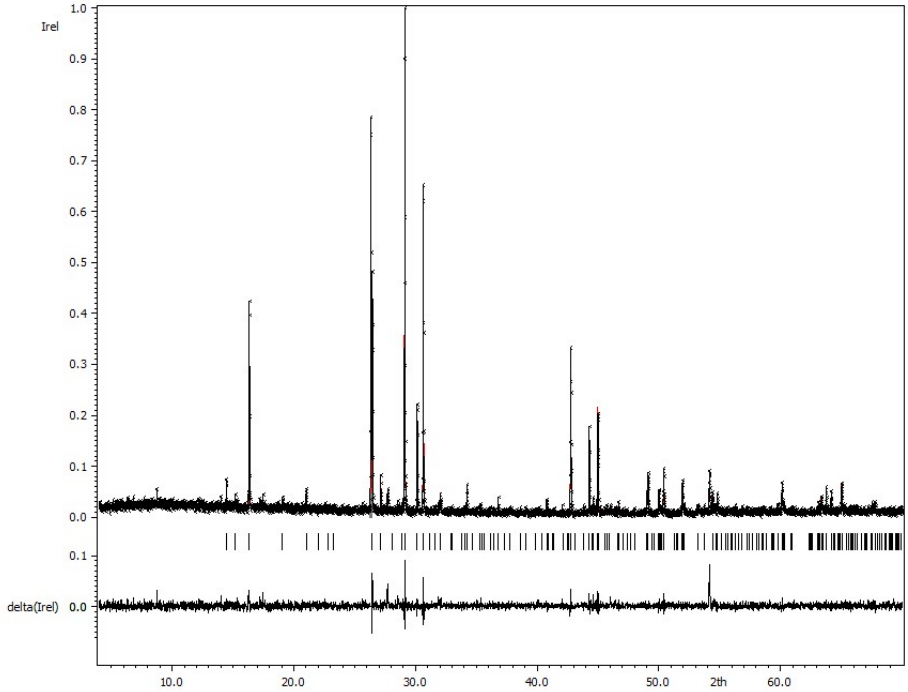

Table S5: EDS analyses on single crystals were performed in by use of scanning electron microscopes (JEOL-7000F, TM3000) showing that all the intended elements are present in $Y_xSb_{(3-x)}O_4F$. The percentage of the heavy elements is calculated considering seven points in each crystal.

Sb_3O_4F	EDS data (at %)		Average values (at %)		Expected values for $Y_{0.5}Sb_{2.5}O_4F$ (at %)	
	Y	Sb	Y	Sb	Y	Sb
Crystal 1	25.3	74.7	23.6	76.4	16.7	83.3
	18.3	81.7				
	23.3	76.7				
	16.5	83.5				
	26.1	73.9				
	26.5	73.5				
	29.2	70.8				
Crystal 2	17.7	82.3	16.8	83.2	16.7	83.3
	14.6	85.4				
	14.5	85.5				
	18.2	81.8				
	18.5	81.5				
	19.3	80.7				
	14.8	85.2				

References

1. I. D. Brown, D. Altermatt, *Acta Crystallogr. Sect. B*, 1985, 41, 244-247.