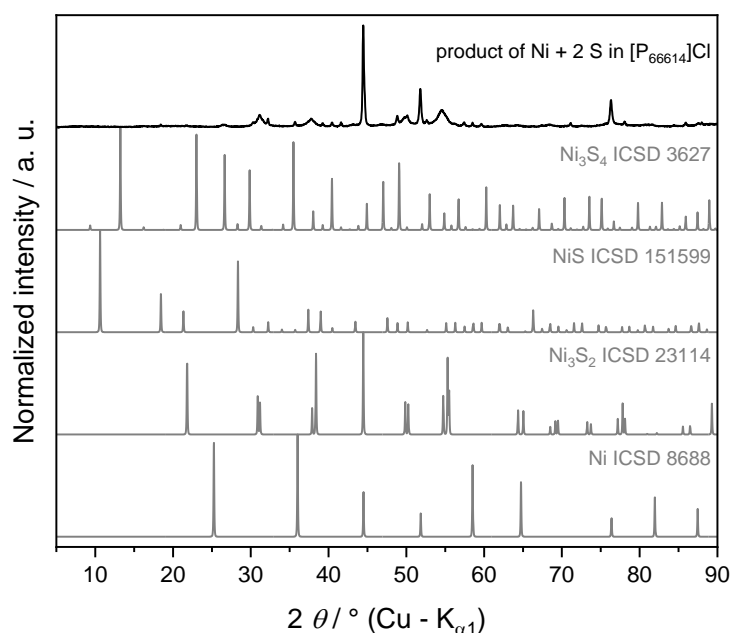


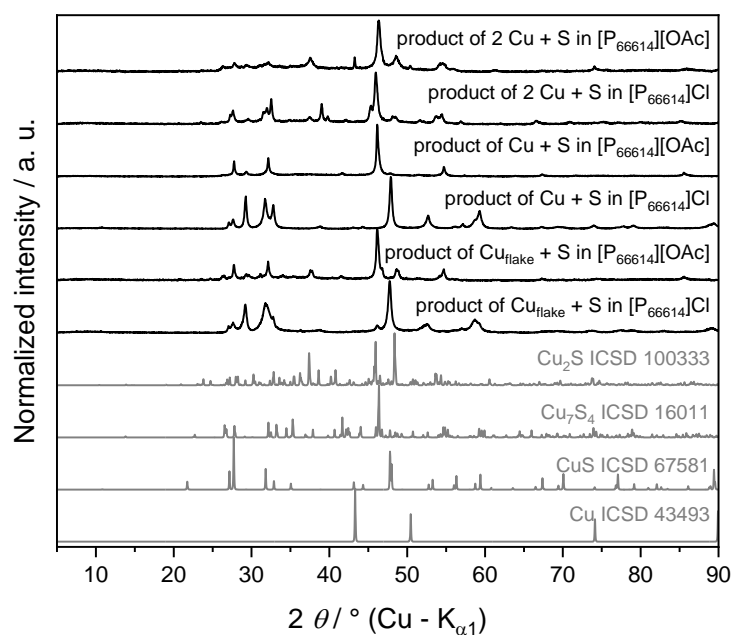
## Exploration of Metal Sulfide Syntheses and the Dissolution Process of Antimony Sulfide in Phosphonium-based Ionic Liquids – Supporting Information

Matthias A. Grasser, Tobias Pietsch, Eike Brunner and Michael Ruck

### PXRD of the reaction products of Ni, Cu and antimony with sulfur in [P<sub>66614</sub>]Cl

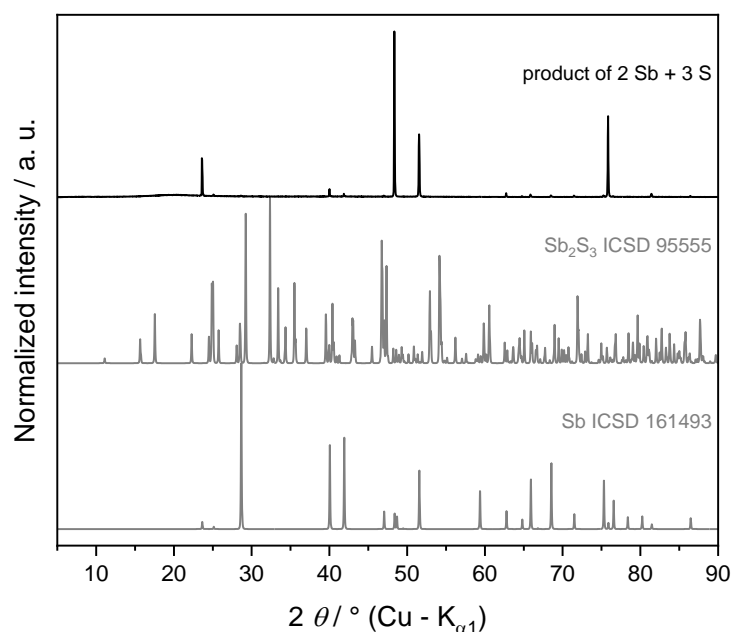


**Figure S1:** Comparison of Powder x-ray diffraction (PXRD) pattern of the precipitate after reaction of Ni + 2 S in [P<sub>66614</sub>]Cl, for 16 h @ 100 °C after washing (3 x 5 mL) with DCM in air, with calculated patterns of nickel (ICSD 8688), Ni<sub>3</sub>S<sub>2</sub> (ICSD 23114), NiS (ICSD 151599) and Ni<sub>3</sub>S<sub>4</sub> (ICSD 23114). Broad reflections indicating formation of Ni<sub>3</sub>S<sub>2</sub> besides small reflections of traces of unreacted nickel and other sulfides.



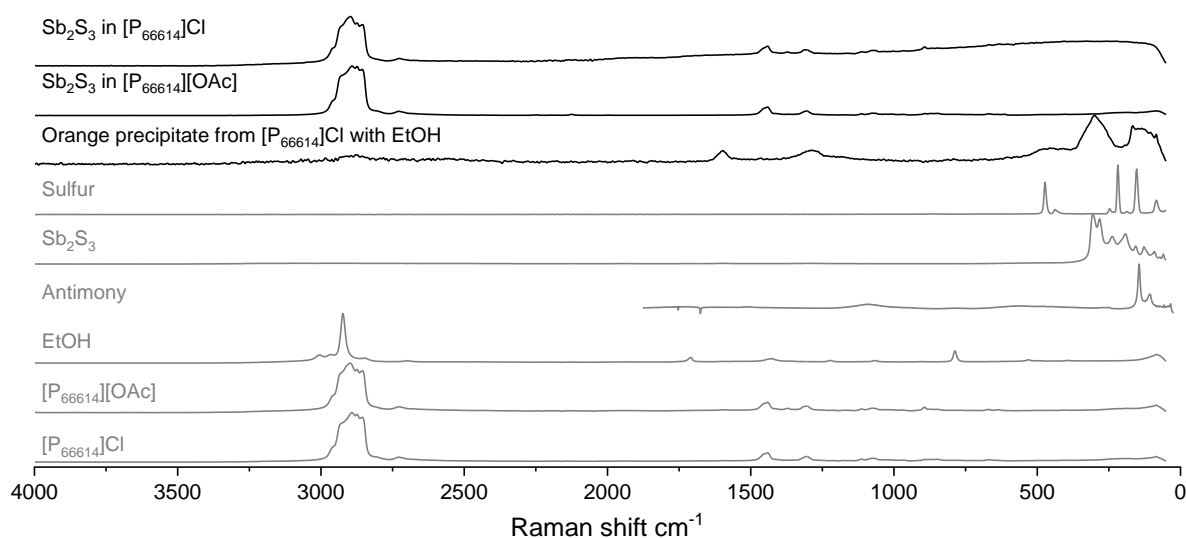
**Figure S2:** Comparison of Powder x-ray diffraction (PXRD) pattern of the precipitate after reaction of Cu + S in [P<sub>66614</sub>]Cl or [P<sub>66614</sub>][OAc], for 16 h @ 100 °C after washing (3 x 5 mL) with DCM in air, with calculated patterns of copper (ICSD 43493) and CuS (ICSD 67581), Cu<sub>7</sub>S<sub>4</sub> (ICSD 16011) and Cu<sub>2</sub>S (100333). PXRD from the reaction starting from Cu powder show formation of CuS with traces of unreacted copper. In contrast reaction from flakes show additionally reflections for Cu<sub>2</sub>S. The reaction of two equivalents of copper shows mainly formation of Cu<sub>2</sub>S. The size of the copper starting material plays a role, as can be seen in reactions

of copper flakes with sulfur. Sulfur seems to form the mobile species, as suggested by the high solubility in ILs. All reactions of copper flakes show unreacted copper macroscopically.

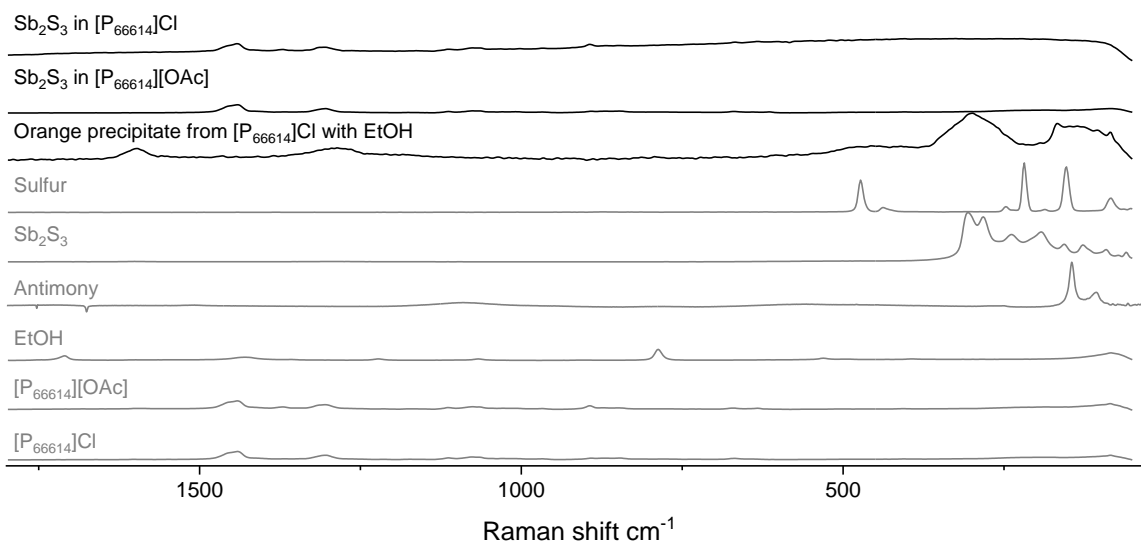


**Figure S3:** Comparison of Powder x-ray diffraction (PXRD) pattern of the precipitate after reaction of 2 Sb + 3 S in  $[P_{66614}]Cl$ , for 16 h @ 100 °C after washing (3 x 5 mL) with DCM in air, with calculated patterns of antimony (ICSD 161493). The powder patterns shows besides a small amorphous background only unreacted antimony, with a strong preferred orientation.

### **Raman spectra of solutions of $Sb_2S_3$ in $[P_{66614}][OAc]$ and $[P_{66614}]Cl$**

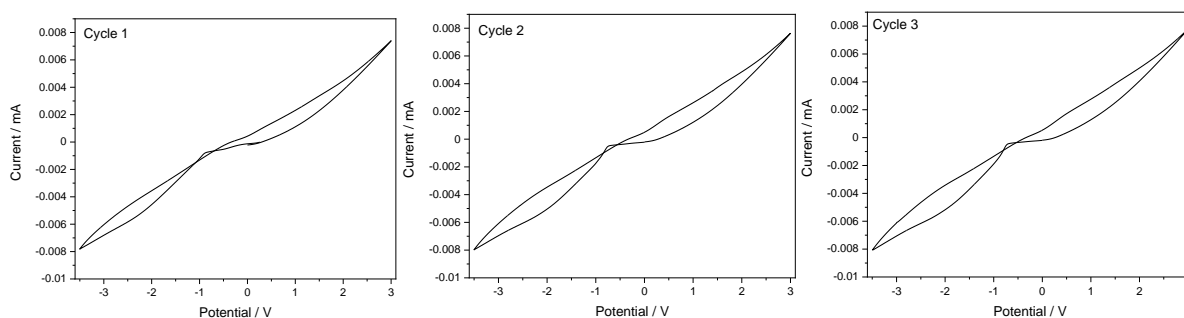


**Figure S4:** Comparison Raman spectra of  $Sb_2S_3$  dissolved in  $[P_{66614}]Cl$  and  $[P_{66614}][OAc]$  with spectra of the starting materials for the synthesis of antimony sulfide (Sb, S) and with crystalline  $Sb_2S_3$ , which was dissolved in the ILs. Self-measured references are grey, samples black. Solutions of  $Sb_2S_3$  show no Raman resonances besides that of the IL. The precipitate show in comparison with references signals for metastibnite (typically broad Raman shift at 300  $cm^{-1}$ ), crystalline sulfur and antimony.



**Figure S5:** Detailed view of the comparison of Raman spectra of  $\text{Sb}_2\text{S}_3$  dissolved in  $[\text{P}_{66614}]\text{Cl}$  and  $[\text{P}_{66614}][\text{OAc}]$  with spectra of the starting materials for the synthesis of antimony sulfide (Sb, S) and with crystalline  $\text{Sb}_2\text{S}_3$ , which was dissolved in the ILs. Self-measured references are grey, samples black.

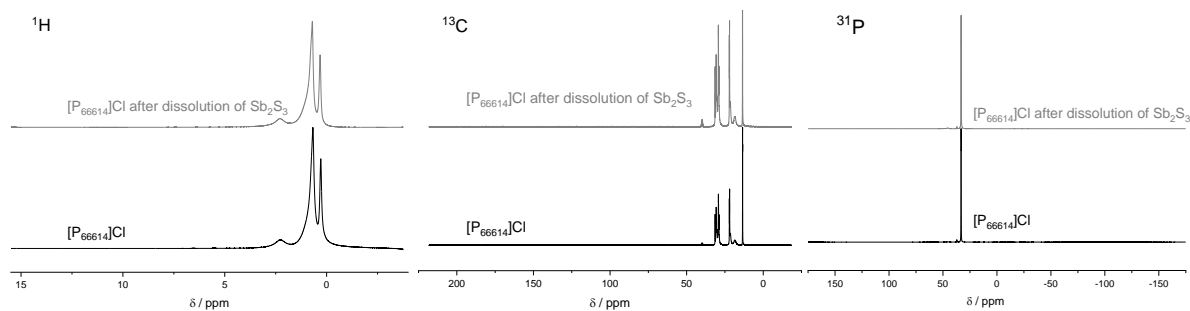
## Electrochemical Characterization



**Figure S6:** Cyclic voltammogram (CV) of  $\text{Sb}_2\text{S}_3$  dissolved in  $[\text{P}_{66614}]\text{Cl}$ . Scan speed 10 mV/s in a potential range from  $-3,5$  V to 3 V at 100 °C. Only very small hysteresis occurs, without any significant signal, indicating the dissolved species to be neutral.

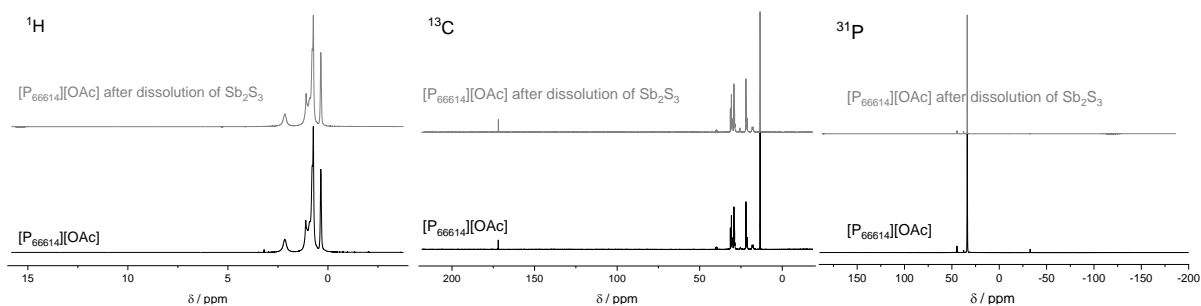
## NMR

### Comparison of $[\text{P}_{66614}]\text{Cl}$ befor and after dissolution of $\text{Sb}_2\text{S}_3$



**Figure S7:** Comparison of NMR spectra ( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ ) of  $[\text{P}_{66614}]\text{Cl}$  befor and after the dissolution of  $\text{Sb}_2\text{S}_3$ . No changes of the resonances shows that the IL is not decomposed by the dissolution process. Chances of the chemical shift would indicate strong interactions of the IL with the dissolved species, as it is not seen, we conclude weak interactions.

## Comparison of [P<sub>66614</sub>][OAc] bevor and after dissolution of Sb<sub>2</sub>S<sub>3</sub>

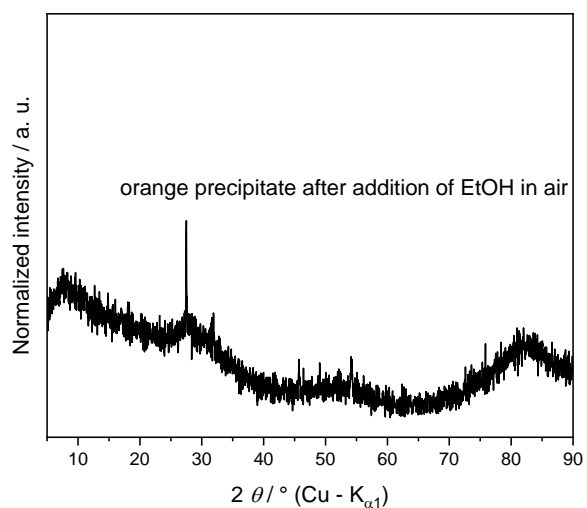


**Figure S8:** Comparison of NMR spectra (<sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P) of [P<sub>66614</sub>][OAc] bevor and after the dissolution of Sb<sub>2</sub>S<sub>3</sub>. No changes of the resonances shows that the IL is not decomposed by the dissolution process. Only weak interactions of the IL with the dissolved species are present, as there is no significant difference in the chemical shift of the signals.

## PXRD: Orange precipitate



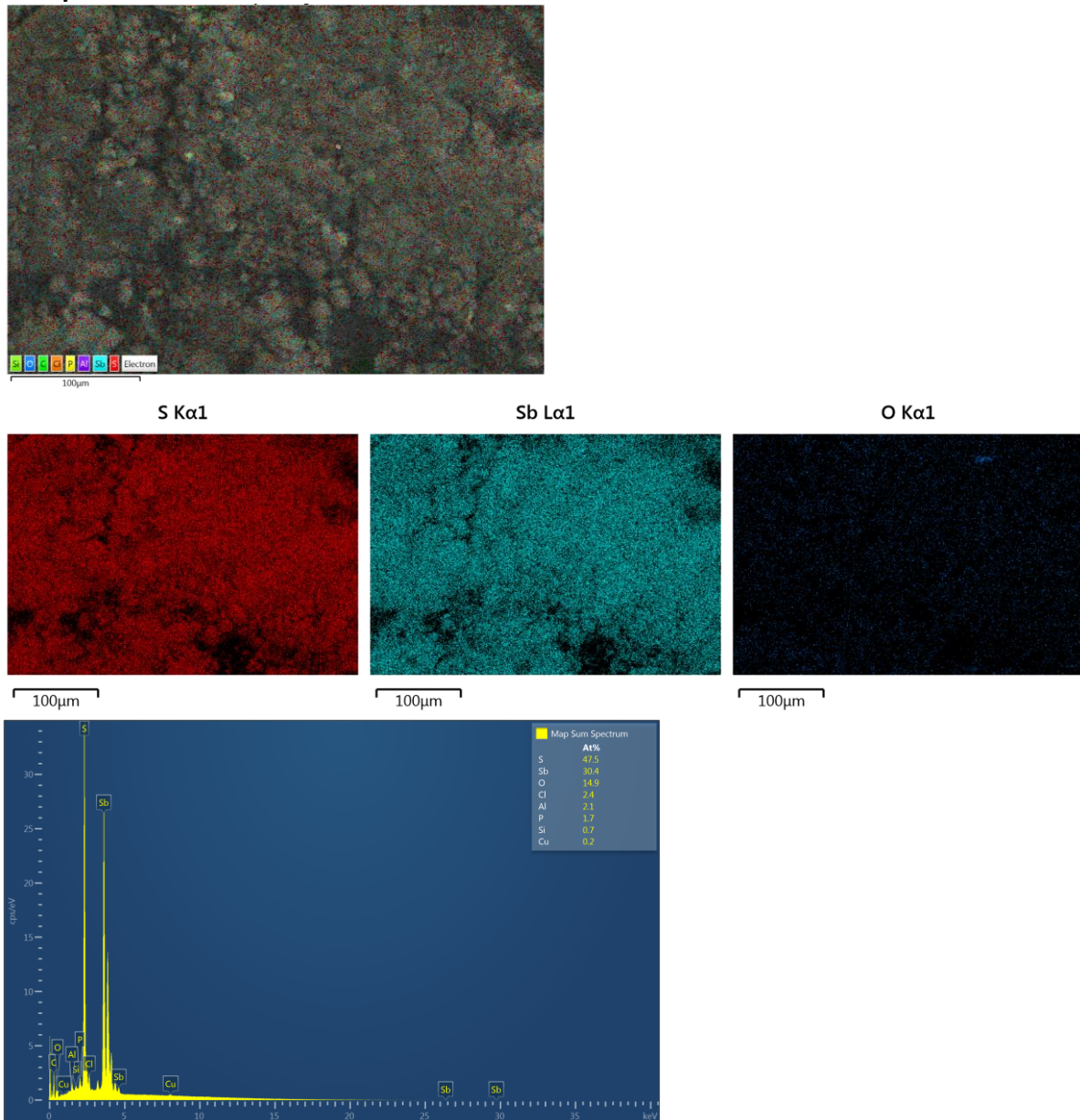
**Figure S9:** Picture of the reflection sample holder after preparation a PXRD sample of precipitate from the synthesis of Sb with S from IL supernatant by dilution with EtOH. In the middle a ring of grey materials, supposed to be Sb, can be seen through the orange precipitate. The large particles are glass from the preparation.



**Figure S10:** Powder x-ray diffraction (PXRD) pattern of the orange precipitate after addition and washing (3 x 5 mL) with EtOH in air. Only one strong reflection at an angle 2θ of 27.3° occurs, which is not enough to identify the material properly.

## SEM/EDX

### Precipitated in air

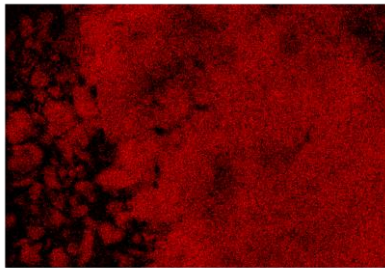


**Figure S11.** SEM/EDX maps of the orange material precipitated in air from  $\text{Sb}_2\text{S}_3$  dissolved in  $[\text{P}_{66614}]\text{Cl}$ . Sb and S show homogeneous distribution in the agglomerates in the ratio of Sb : S; 30.4 : 47.5 (At %). Oxygen is equally distributed over the hole measurement range.

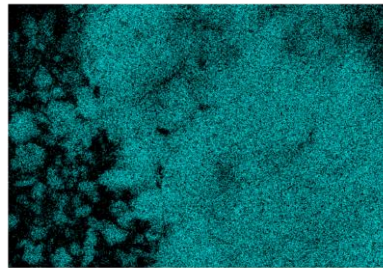
## Precipitated in Ar



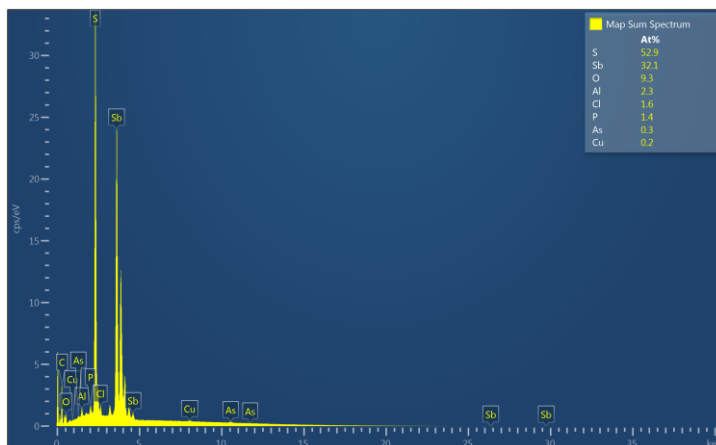
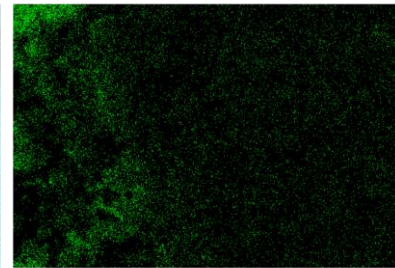
S Kα1



Sb Lα1



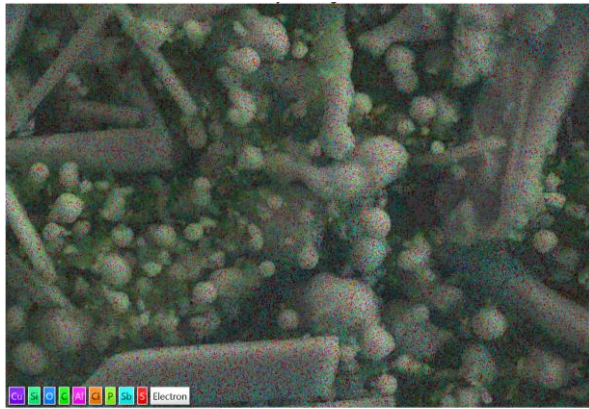
C Kα1\_2



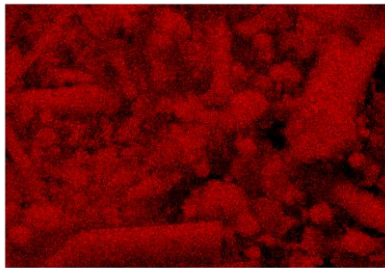
**Figure S12.** SEM/EDX maps of the orange material precipitated in Ar from  $Sb_2S_3$  dissolved in  $[P_{66614}]Cl$ . Sb and S show homogeneous distribution in the agglomerates in the ratio of Sb : S; 32.1 : 52.9 (At %). Carbon content comes from the carbon pad.



### Precipitated in air after DSC

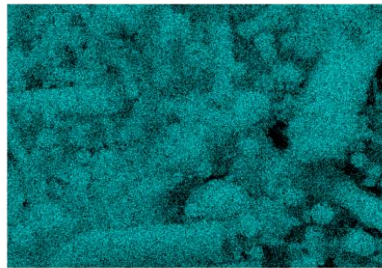


S K $\alpha$ 1



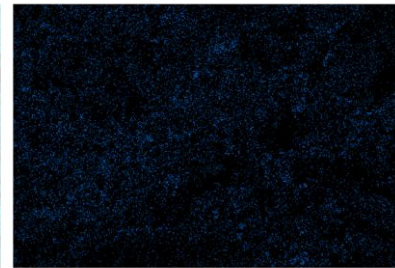
25µm

Sb L $\alpha$ 1

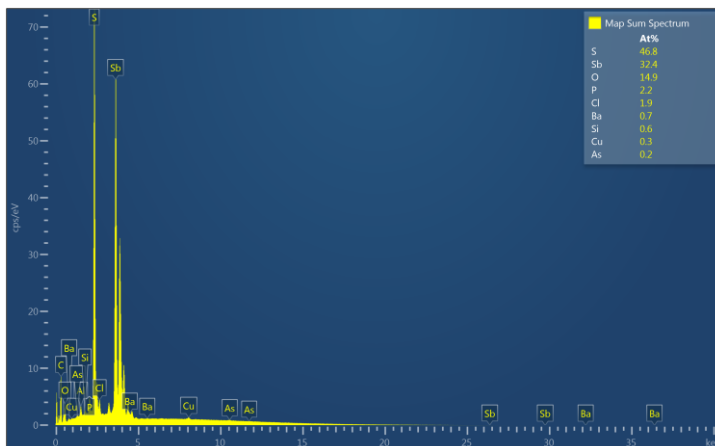


25µm

O K $\alpha$ 1



25µm



**Figure S13.** SEM/EDX maps of the in air precipitated sample after DSC. Sb and S show homogeneous distribution in the agglomerates in the ratio of Sb : S; 32.4 : 46.8 (At %). Carbon content comes from the carbon pad. Carbon and oxygen are equally distributed in the measuring range.

Precipitated in Ar after DSC

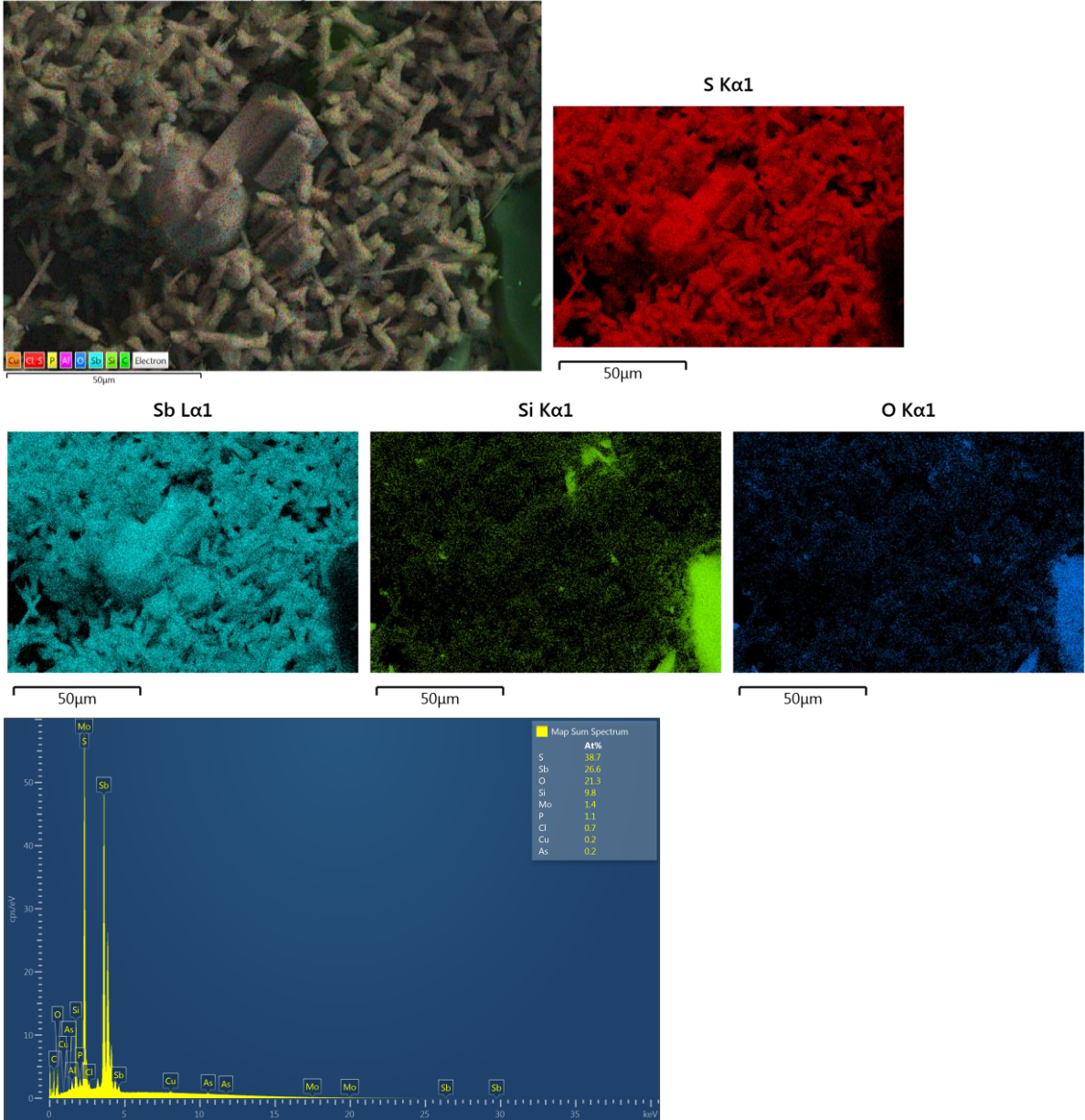
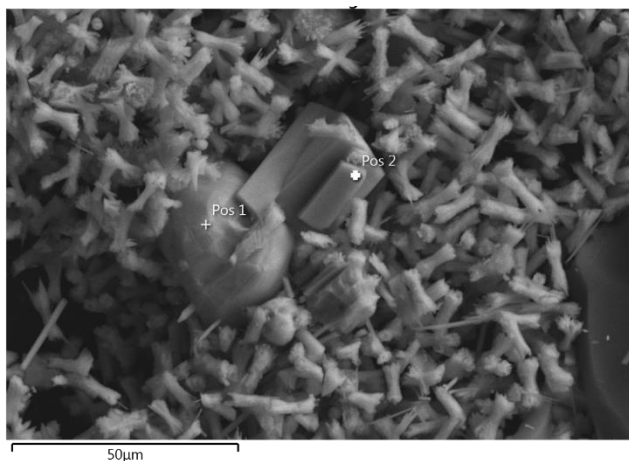


Figure S14. SEM/EDX maps of the in Ar precipitated sample after DSC. Sb and S show homogeneous distribution in the agglomerates in the ratio of Sb : S; 26.6 : 38.7 (At %). The silicon and oxygen can be addressed by quartz glass pieces of the DSC ampoule.

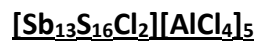




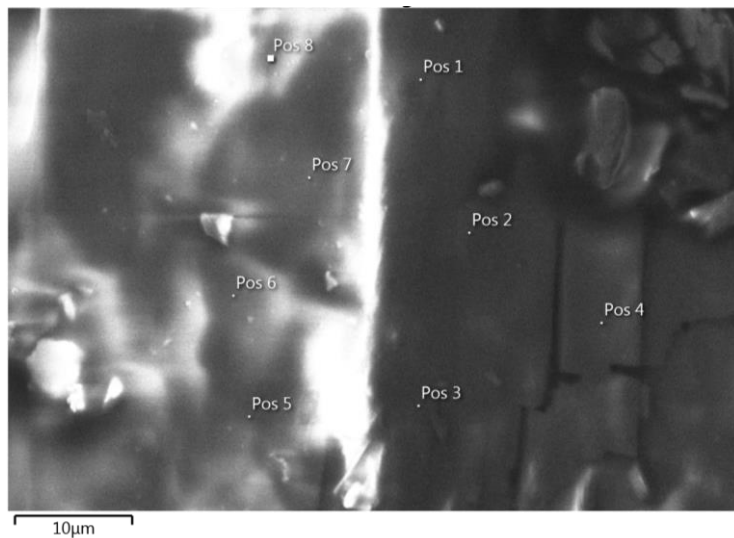
**Figure S15.** SEM image of the in Ar precipitated sample after DSC with the marked positions for EDX. The atomic ratios are shown in table S1.

**Table S1.** SEM/EDX point spectra of an crystal of the in Ar precipitated sample after DSC. Dividing the average Sb and S ratios gives a ratio of Sb : S of 1 : 1.50(5)

Spectrum Label	Pos 1	Pos 2			
O		3.74			
S	58.35	57.42			
Cl	0.63				
Mo	2.86				
Sb	38.16	38.84			
Total	100.00	100.00			
Statistics	O	S	Cl	Mo	Sb
Max	3.74	58.35	0.63	2.86	38.84
Min	3.74	57.42	0.63	2.86	38.16
Average		57.89			38.50
Standard Deviation		0.66			0.48



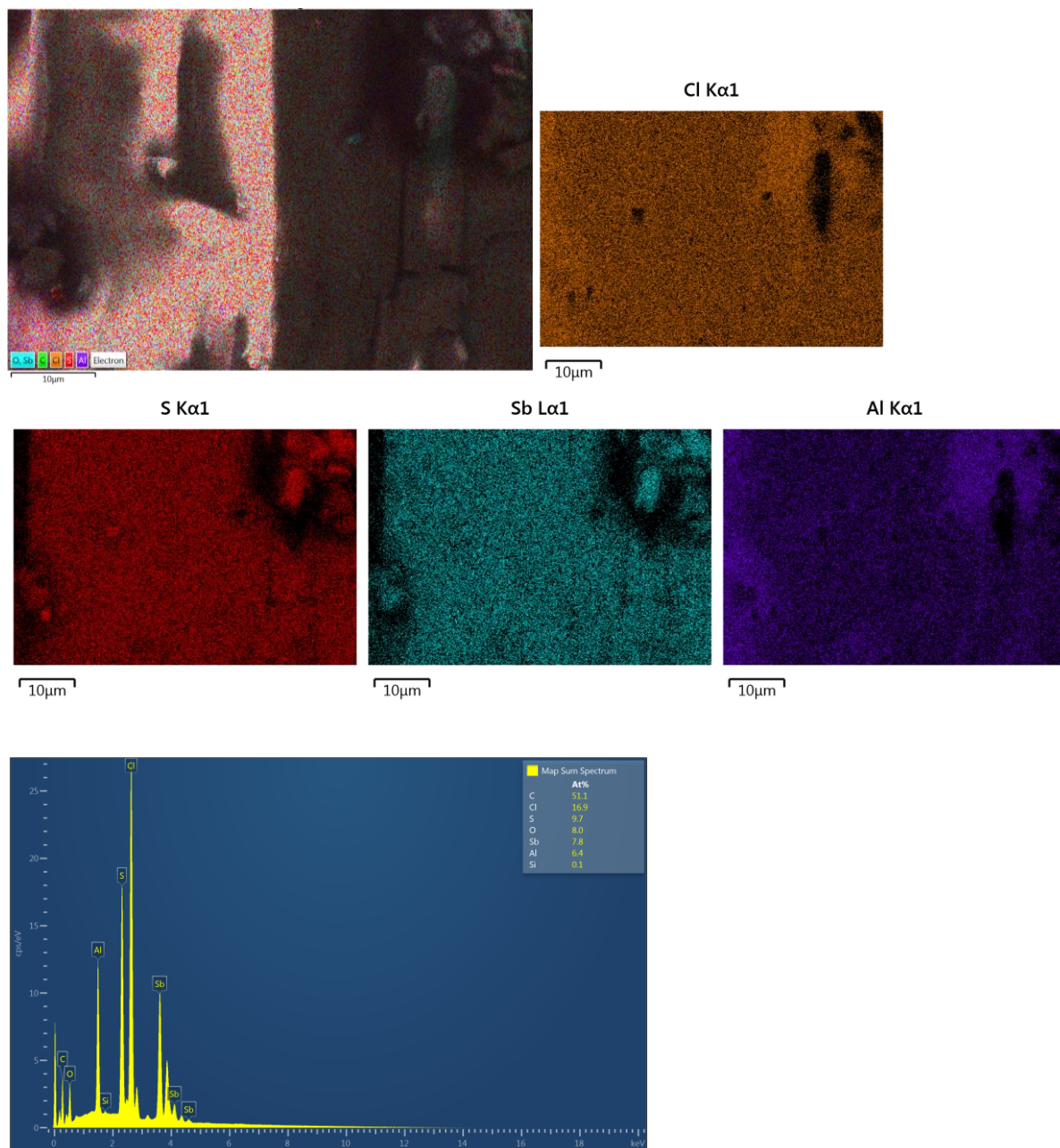
**SEM/EDX characterization:**



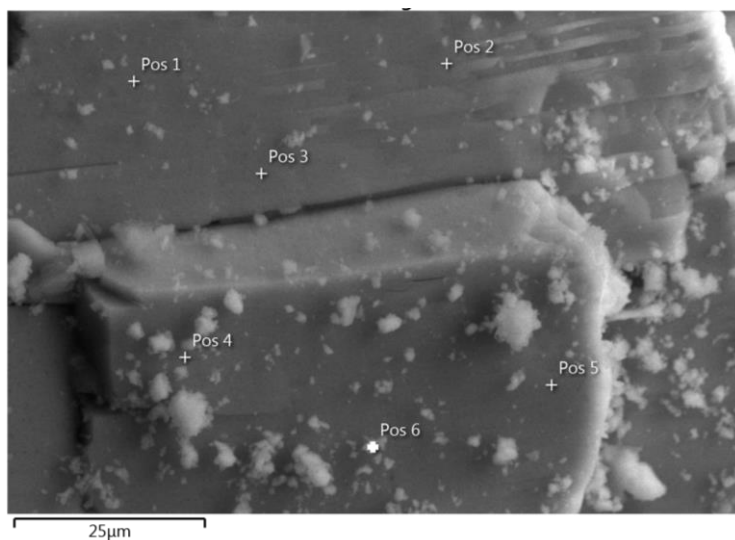
**Figure S16.** SEM image with marked positions for EDX measurements, where the atomic ratios are shown in table S3.

**Table S2.** Atomic ratios of the EDX measurements shown in figure S16. The ratio of Sb : S; 9.80 : 12.2 (At %) fit well to the ratio of 13 : 16 from the SCXRD structure solution.

Spectrum Label	Pos 1	Pos 2	Pos 3	Pos 4	Pos 5	Pos 6	Pos 7	Pos 8
C	51.84	50.12	53.49	51.10	51.24	46.46	48.95	49.82
O	6.42	5.76	7.18	5.24	6.37	5.66	5.99	4.62
Al	5.28	5.22	5.28	5.19	5.34	5.56	5.44	5.36
S	11.58	12.45	10.47	12.31	11.53	13.66	12.69	12.94
Cl	15.52	16.35	15.10	16.23	16.21	17.87	16.83	16.98
Sb	9.36	10.09	8.49	9.94	9.30	10.79	10.10	10.28
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Statistics	C	O	Al	S	Cl	Sb		
Max	53.49	7.18	5.56	13.66	17.87	10.79		
Min	46.46	4.62	5.19	10.47	15.10	8.49		
Average	50.38	5.90	5.33	12.20	16.38	9.80		
Standard Deviation	2.10	0.78	0.12	0.99	0.86	0.71		



**Figure S17.** SEM/EDX maps of one of the crystals. Sb and S show homogeneous distribution in the ratio of Sb : S; 7.8 : 9.7 (At %), which fits well to the ratio 13 : 16 from SCXRD.



**Figure S18.** SEM image with marked positions for EDX measurements, where the atomic ratios are shown in table S4.

**Table S3.** Atomic ratios of the EDX measurements shown in figure S16. The ratio Sb : S : Cl : Al are 0.83(2) : 1 : 1.60(3) : 0.32(1) (At %) fit well to the ratio of 13 : 16 : 22 : 5 from the SCXRD structure solution.

Spectrum Label	Pos 1	Pos 2	Pos 3	Pos 4	Pos 5	Pos 6
C	53.58	57.76	55.59	61.07	62.08	63.12
O	5.03	3.52	4.45	5.10	3.75	10.93
Al	3.50	2.98	3.27	2.61	2.80	3.15
S	11.06	10.35	10.68	9.30	8.94	6.74
Cl	17.84	16.82	17.22	13.95	14.65	10.85
Sb	9.01	8.58	8.80	7.96	7.78	5.22
Total	100.00	100.00	100.00	100.00	100.00	100.00
Statistics	C	O	Al	S	Cl	Sb
Max	63.12	10.93	3.50	11.06	17.84	9.01
Min	53.58	3.52	2.61	6.74	10.85	5.22
Average	58.87	5.46	3.05	9.51	15.22	7.89
Standard Deviation	3.83	2.75	0.32	1.58	2.62	1.39

**Table S4.** Crystal structure determination and crystallographic data for  $[\text{Sb}_{13}\text{S}_{16}][\text{AlCl}_4]_5$ 

Identification code	(Sb <sub>13</sub> S <sub>16</sub> Cl <sub>2</sub> )[AlCl <sub>4</sub> ] <sub>5</sub>	
Empirical formula	Al <sub>5</sub> Cl <sub>22</sub> S <sub>16</sub> Sb <sub>13</sub>	
Formula weight	3010.51	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.8886(10) Å b = 19.043(2) Å c = 21.125(2) Å	α = 63.884(2)° β = 80.322(2)° γ = 88.699(2)°
Volume	3159.7(6) Å <sup>3</sup>	
Z	2	
Density (calculated)	3.164 Mg/m <sup>3</sup>	
Absorption coefficient F(000)	7.003 mm <sup>-1</sup> 2716	
Crystal size	0.056 x 0.042 x 0.032 mm <sup>3</sup>	
Theta range for data collection	1.940 to 32.623°	
Index ranges	-13<=h<=13, -28<=k<=28, -32<=l<=31	
Reflections collected	127692	
Independent reflections	23016 [R(int) = 0.0319]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7464 and 0.5687	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	23016 / 0 / 505	
Goodness-of-fit on F <sup>2</sup>	1.308	
Final R indices [I>2sigma(I)]	R1 = 0.0272, wR2 = 0.0265	
R indices (all data)	R1 = 0.0442, wR2 = 0.0273	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.291 and -1.063 e.Å <sup>-3</sup>	

**Table S5.** Atomic coordinates ( $\times 10^4$ ) and displacement parameters ( $\times 10^3 \text{ \AA}^2$ ) for  $[\text{Sb}_{13}\text{S}_{16}\text{Cl}_2][\text{AlCl}_4]_5$ .  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor. The exponent of the anisotropic displacement factor takes the form:  $2\pi^2[h^2 a^{*2}U_{11} + \dots + 2hka^*b^*U_{12}]$

Atom	x	y	z	$U(\text{eq})$	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
Sb(1)	6572(1)	514(1)	3947(1)	29(1)	35(1)	20(1)	31(1)	-10(1)	-4(1)	0(1)
Sb(2)	4472(1)	1922(1)	2479(1)	32(1)	32(1)	31(1)	42(1)	-20(1)	-19(1)	10(1)
Sb(3)	8562(1)	2063(1)	2149(1)	32(1)	28(1)	40(1)	25(1)	-14(1)	-2(1)	2(1)
Sb(4)	6373(1)	2667(1)	3580(1)	25(1)	33(1)	21(1)	25(1)	-12(1)	-8(1)	3(1)
Sb(5)	3967(1)	3767(1)	4431(1)	28(1)	23(1)	33(1)	27(1)	-15(1)	2(1)	2(1)
Sb(6)	8025(1)	4063(1)	4171(1)	29(1)	24(1)	30(1)	37(1)	-19(1)	-7(1)	-3(1)
Sb(7)	6195(1)	5006(1)	2542(1)	27(1)	26(1)	21(1)	28(1)	-5(1)	-4(1)	-2(1)
Sb(8)	5421(1)	6358(1)	674(1)	27(1)	25(1)	28(1)	27(1)	-10(1)	-10(1)	1(1)
Sb(9)	9239(1)	5943(1)	923(1)	30(1)	21(1)	25(1)	40(1)	-15(1)	0(1)	3(1)
Sb(10)	7191(1)	7353(1)	1579(1)	28(1)	29(1)	24(1)	31(1)	-15(1)	0(1)	-3(1)
Sb(11)	4363(1)	8010(1)	2711(1)	30(1)	24(1)	30(1)	34(1)	-14(1)	0(1)	-3(1)
Sb(12)	8312(1)	8326(1)	2744(1)	30(1)	28(1)	34(1)	37(1)	-21(1)	-13(1)	7(1)
Sb(13)	6708(1)	9651(1)	1098(1)	35(1)	43(1)	23(1)	33(1)	-7(1)	-3(1)	1(1)
S(1)	4420(1)	1428(1)	3770(1)	30(1)	30(1)	25(1)	36(1)	-15(1)	-1(1)	0(1)
S(2)	6515(1)	1123(1)	2298(1)	33(1)	40(1)	29(1)	37(1)	-21(1)	-9(1)	7(1)
S(3)	8754(1)	1487(1)	3410(1)	30(1)	28(1)	31(1)	27(1)	-10(1)	-7(1)	-1(1)
S(4)	6498(1)	2995(1)	2256(1)	28(1)	42(1)	20(1)	23(1)	-8(1)	-9(1)	1(1)
S(5)	4095(1)	3469(1)	3412(1)	27(1)	26(1)	28(1)	30(1)	-14(1)	-10(1)	2(1)
S(6)	6110(1)	2970(1)	4881(1)	28(1)	28(1)	31(1)	24(1)	-10(1)	-6(1)	-1(1)
S(7)	8186(1)	3978(1)	2997(1)	26(1)	23(1)	28(1)	26(1)	-11(1)	-2(1)	1(1)
S(8)	5875(1)	4963(1)	3851(1)	36(1)	33(1)	26(1)	54(1)	-23(1)	-5(1)	3(1)
S(9)	6865(1)	5130(1)	1154(1)	35(1)	29(1)	25(1)	55(1)	-22(1)	-7(1)	1(1)
S(10)	8513(1)	5934(1)	2142(1)	29(1)	25(1)	26(1)	28(1)	-5(1)	-7(1)	-4(1)
S(11)	7896(1)	7106(1)	239(1)	32(1)	30(1)	29(1)	26(1)	-3(1)	-4(1)	-3(1)
S(12)	4855(1)	6546(1)	1788(1)	29(1)	23(1)	29(1)	38(1)	-19(1)	1(1)	-1(1)
S(13)	6636(1)	7135(1)	2889(1)	29(1)	34(1)	22(1)	30(1)	-10(1)	-5(1)	2(1)
S(14)	5054(1)	8452(1)	1398(1)	30(1)	34(1)	29(1)	32(1)	-16(1)	-11(1)	5(1)
S(15)	8977(1)	8918(1)	1449(1)	35(1)	29(1)	35(1)	39(1)	-16(1)	1(1)	-3(1)
S(16)	5918(1)	8983(1)	2805(1)	32(1)	29(1)	35(1)	45(1)	-27(1)	-11(1)	8(1)
Cl(1)	6489(1)	665(1)	5028(1)	43(1)	51(1)	47(1)	27(1)	-12(1)	-6(1)	-10(1)
Cl(2)	7412(1)	9797(1)	-82(1)	74(1)	85(1)	89(1)	31(1)	-15(1)	1(1)	0(1)
Al(1)	482(1)	1793(1)	5270(1)	26(1)	25(1)	27(1)	25(1)	-10(1)	-5(1)	2(1)
Cl(11)	153(1)	2709(1)	4256(1)	45(1)	45(1)	49(1)	28(1)	-6(1)	-9(1)	15(1)
Cl(12)	2671(1)	2039(1)	5468(1)	50(1)	37(1)	55(1)	43(1)	-4(1)	-14(1)	-15(1)
Cl(13)	604(1)	710(1)	5232(1)	52(1)	58(1)	41(1)	68(1)	-33(1)	-17(1)	9(1)
Cl(14)	-1251(1)	1859(1)	6058(1)	50(1)	46(1)	63(1)	37(1)	-21(1)	0(1)	18(1)
Al(2)	2278(1)	7885(1)	-140(1)	26(1)	25(1)	27(1)	24(1)	-9(1)	-4(1)	-1(1)
Cl(21)	1285(1)	7266(1)	967(1)	42(1)	42(1)	53(1)	26(1)	-15(1)	1(1)	-13(1)
Cl(22)	2248(1)	7026(1)	-538(1)	42(1)	56(1)	40(1)	36(1)	-21(1)	-10(1)	3(1)
Cl(23)	4586(1)	8239(1)	-233(1)	47(1)	27(1)	51(1)	53(1)	-15(1)	-7(1)	-7(1)
Cl(24)	965(1)	8842(1)	-659(1)	51(1)	40(1)	36(1)	66(1)	-11(1)	-19(1)	9(1)
Al(3)	2147(1)	4018(1)	1425(1)	29(1)	25(1)	31(1)	34(1)	-15(1)	-8(1)	5(1)
Cl(31)	1450(1)	3037(1)	2436(1)	46(1)	52(1)	36(1)	40(1)	-13(1)	3(1)	1(1)
Cl(32)	2606(1)	5005(1)	1611(1)	47(1)	48(1)	39(1)	58(1)	-25(1)	-10(1)	-5(1)
Cl(33)	319(1)	4228(1)	843(1)	56(1)	50(1)	55(1)	84(1)	-38(1)	-42(1)	20(1)
Cl(34)	4149(1)	3774(1)	870(1)	56(1)	39(1)	95(1)	44(1)	-40(1)	-6(1)	19(1)
Al(4)	1128(1)	6017(1)	3610(1)	27(1)	25(1)	28(1)	29(1)	-15(1)	-3(1)	1(1)
Cl(41)	1064(1)	7077(1)	2660(1)	39(1)	47(1)	35(1)	33(1)	-13(1)	-10(1)	4(1)
Cl(42)	2930(1)	6127(1)	4117(1)	41(1)	38(1)	52(1)	42(1)	-25(1)	-14(1)	-1(1)
Cl(43)	1657(1)	5064(1)	3343(1)	42(1)	50(1)	36(1)	49(1)	-28(1)	-8(1)	6(1)
Cl(44)	-1030(1)	5800(1)	4290(1)	50(1)	33(1)	56(1)	57(1)	-26(1)	10(1)	-2(1)
Al(5)	1669(1)	243(1)	2386(1)	30(1)	25(1)	34(1)	36(1)	-19(1)	-10(1)	4(1)
Cl(51)	-709(1)	-107(1)	2793(1)	40(1)	24(1)	47(1)	64(1)	-37(1)	-8(1)	5(1)
Cl(52)	2860(1)	204(1)	3189(1)	46(1)	48(1)	50(1)	40(1)	-16(1)	-18(1)	-11(1)
Cl(53)	2551(1)	-526(1)	1942(1)	46(1)	46(1)	60(1)	47(1)	-34(1)	-15(1)	26(1)
Cl(54)	1901(1)	1420(1)	1588(1)	54(1)	67(1)	40(1)	45(1)	-8(1)	-17(1)	3(1)



**Table S6.** Atomic distances d (Å) and angels (°) in [Sb<sub>13</sub>S<sub>16</sub>Cl<sub>2</sub>][AlCl<sub>4</sub>]<sub>5</sub>.**Interatomic distances:**

Sb(1)-Cl(1)	2.4123(7)	Sb(10)-S(14)	2.7373(6)
Sb(1)-S(3)	2.4607(6)	Sb(10)-S(10)	2.7568(6)
Sb(1)-S(1)	2.5188(6)	Sb(10)-S(11)	3.0308(7)
Sb(1)-S(2)	3.1609(7)	Sb(10)-S(15)	3.2901(7)
Sb(1)-Cl(1)#1	3.3380(7)	Sb(11)-S(16)	2.4335(6)
Sb(1)-Sb(3)	3.7917(3)	Sb(11)-S(14)	2.4848(7)
Sb(2)-S(2)	2.4320(6)	Sb(11)-S(13)	2.5584(6)
Sb(2)-S(1)	2.4537(7)	Sb(11)-Cl(53)#5	3.1149(7)
Sb(2)-S(4)	2.5834(6)	Sb(11)-Sb(12)	3.5920(4)
Sb(2)-Cl(52)	3.1906(7)	Sb(12)-S(15)	2.4246(7)
Sb(2)-Cl(31)	3.3734(7)	Sb(12)-S(16)	2.4578(6)
Sb(2)-Sb(3)	3.5785(5)	Sb(12)-S(13)	2.6164(6)
Sb(3)-S(3)	2.4289(6)	Sb(12)-Cl(51)#6	3.1773(7)
Sb(3)-S(2)	2.4636(7)	Sb(12)-Cl(41)#3	3.4114(7)
Sb(3)-S(4)	2.5761(6)	Sb(12)-Cl(12)#4	3.4818(8)
Sb(3)-Cl(22)#2	3.2677(7)	Sb(13)-Cl(2)	2.3563(8)
Sb(3)-Cl(54)#3	3.4114(8)	Sb(13)-S(15)	2.4552(7)
Sb(3)-Cl(31)#3	3.4833(7)	Sb(13)-S(14)	2.5169(6)
Sb(4)-S(5)	2.4856(6)	Sb(13)-S(16)	3.2049(7)
Sb(4)-S(4)	2.5677(6)	Sb(13)-Cl(24)#7	3.2646(7)
Sb(4)-S(7)	2.6744(6)	Cl(1)-Sb(1)#1	3.3380(7)
Sb(4)-S(1)	2.8042(6)	Al(1)-Cl(13)	2.0986(10)
Sb(4)-S(6)	3.0198(7)	Al(1)-Cl(14)	2.1145(9)
Sb(4)-S(3)	3.1463(7)	Al(1)-Cl(11)	2.1486(9)
Sb(5)-S(5)	2.4415(6)	Al(1)-Cl(12)	2.1599(9)
Sb(5)-S(6)	2.4582(6)	Cl(11)-Sb(6)#8	3.1214(7)
Sb(5)-S(8)	2.5656(6)	Cl(12)-Sb(12)#4	3.4819(8)
Sb(5)-Cl(12)	3.1520(7)	Al(2)-Cl(24)	2.1127(9)
Sb(5)-Cl(43)	3.4583(7)	Al(2)-Cl(23)	2.1246(9)
Sb(5)-Sb(6)	3.5753(5)	Al(2)-Cl(21)	2.1364(9)
Sb(6)-S(6)	2.4559(6)	Al(2)-Cl(22)	2.1449(9)
Sb(6)-S(8)	2.5126(6)	Cl(21)-Sb(9)#8	3.1839(7)
Sb(6)-S(7)	2.5361(6)	Cl(22)-Sb(3)#2	3.2677(7)
Sb(6)-Cl(11)#3	3.1214(7)	Cl(24)-Sb(13)#7	3.2647(7)
Sb(6)-Cl(42)#4	3.4293(7)	Al(3)-Cl(34)	2.1199(9)
Sb(7)-S(10)	2.5187(6)	Al(3)-Cl(33)	2.1263(9)
Sb(7)-S(7)	2.5783(6)	Al(3)-Cl(31)	2.1281(9)
Sb(7)-S(8)	2.6978(7)	Al(3)-Cl(32)	2.1434(10)
Sb(7)-S(9)	2.7946(7)	Cl(31)-Sb(3)#8	3.4832(7)
Sb(7)-S(12)	2.9875(6)	Cl(33)-Sb(9)#8	3.4498(8)
Sb(7)-S(5)	3.1286(6)	Cl(34)-Sb(8)#2	3.3398(8)
Sb(8)-S(11)	2.4546(6)	Al(4)-Cl(44)	2.1215(9)
Sb(8)-S(12)	2.5004(6)	Al(4)-Cl(42)	2.1330(9)
Sb(8)-S(9)	2.5211(6)	Al(4)-Cl(41)	2.1386(9)
Sb(8)-Cl(32)	3.3104(7)	Al(4)-Cl(43)	2.1443(9)
Sb(8)-Cl(34)#2	3.3398(8)	Cl(41)-Sb(12)#8	3.4114(7)
Sb(8)-Cl(23)	3.3716(8)	Cl(42)-Sb(6)#4	3.4291(7)
Sb(9)-S(11)	2.4615(6)	Al(5)-Cl(54)	2.1191(10)
Sb(9)-S(9)	2.4946(6)	Al(5)-Cl(52)	2.1194(9)
Sb(9)-S(10)	2.5382(7)	Al(5)-Cl(53)	2.1324(9)
Sb(9)-Cl(21)#3	3.1839(7)	Al(5)-Cl(51)	2.1446(9)
Sb(9)-Cl(33)#3	3.4498(8)	Cl(51)-Sb(12)#9	3.1773(7)
Sb(10)-S(12)	2.4714(6)	Cl(53)-Sb(11)#10	3.1149(7)
Sb(10)-S(13)	2.5714(7)	Cl(54)-Sb(3)#8	3.4114(8)

**Angles:**

Cl(1)-Sb(1)-S(3)	87.28(2)	Cl(1)-Sb(1)-Sb(3)	121.233(17)
Cl(1)-Sb(1)-S(1)	82.99(2)	S(3)-Sb(1)-Sb(3)	38.852(14)
S(3)-Sb(1)-S(1)	99.24(2)	S(1)-Sb(1)-Sb(3)	85.015(15)
Cl(1)-Sb(1)-S(2)	154.32(2)	S(2)-Sb(1)-Sb(3)	40.235(12)
S(3)-Sb(1)-S(2)	78.799(18)	Cl(1)#1-Sb(1)-Sb(3)	150.187(12)
S(1)-Sb(1)-S(2)	78.148(18)	S(2)-Sb(2)-S(1)	95.43(2)
Cl(1)-Sb(1)-Cl(1)#1	79.76(2)	S(2)-Sb(2)-S(4)	89.29(2)
S(3)-Sb(1)-Cl(1)#1	166.706(19)	S(1)-Sb(2)-S(4)	88.716(19)
S(1)-Sb(1)-Cl(1)#1	76.375(19)	S(2)-Sb(2)-Cl(52)	76.48(2)
S(2)-Sb(1)-Cl(1)#1	112.009(16)	S(1)-Sb(2)-Cl(52)	74.799(18)

S(4)-Sb(2)-Cl(52)	156.837(18)	S(10)-Sb(7)-S(9)	84.375(19)
S(2)-Sb(2)-Cl(31)	168.67(2)	S(7)-Sb(7)-S(9)	93.154(18)
S(1)-Sb(2)-Cl(31)	94.949(18)	S(8)-Sb(7)-S(9)	173.369(18)
S(4)-Sb(2)-Cl(31)	95.47(2)	S(10)-Sb(7)-S(12)	78.870(19)
Cl(52)-Sb(2)-Cl(31)	101.960(19)	S(7)-Sb(7)-S(12)	160.566(17)
S(2)-Sb(2)-Sb(3)	43.380(15)	S(8)-Sb(7)-S(12)	98.349(18)
S(1)-Sb(2)-Sb(3)	90.769(14)	S(9)-Sb(7)-S(12)	81.984(17)
S(4)-Sb(2)-Sb(3)	46.003(14)	S(10)-Sb(7)-S(5)	159.093(17)
Cl(52)-Sb(2)-Sb(3)	116.768(14)	S(7)-Sb(7)-S(5)	78.685(18)
Cl(31)-Sb(2)-Sb(3)	140.963(13)	S(8)-Sb(7)-S(5)	81.203(17)
S(3)-Sb(3)-S(2)	95.06(2)	S(9)-Sb(7)-S(5)	104.375(17)
S(3)-Sb(3)-S(4)	94.10(2)	S(12)-Sb(7)-S(5)	120.743(17)
S(2)-Sb(3)-S(4)	88.77(2)	S(11)-Sb(8)-S(12)	94.46(2)
S(3)-Sb(3)-Cl(22)#2	169.652(19)	S(11)-Sb(8)-S(9)	87.73(2)
S(2)-Sb(3)-Cl(22)#2	82.756(19)	S(12)-Sb(8)-S(9)	98.20(2)
S(4)-Sb(3)-Cl(22)#2	75.772(18)	S(11)-Sb(8)-Cl(32)	163.805(19)
S(3)-Sb(3)-Cl(54)#3	96.628(19)	S(12)-Sb(8)-Cl(32)	79.644(18)
S(2)-Sb(3)-Cl(54)#3	106.38(2)	S(9)-Sb(8)-Cl(32)	78.306(19)
S(4)-Sb(3)-Cl(54)#3	160.504(17)	S(11)-Sb(8)-Cl(34)#2	88.12(2)
Cl(22)#2-Sb(3)-Cl(54)#3	93.689(17)	S(12)-Sb(8)-Cl(34)#2	173.697(18)
S(3)-Sb(3)-Cl(31)#3	69.136(17)	S(9)-Sb(8)-Cl(34)#2	87.63(2)
S(2)-Sb(3)-Cl(31)#3	164.046(19)	Cl(32)-Sb(8)-Cl(34)#2	99.337(19)
S(4)-Sb(3)-Cl(31)#3	94.372(19)	S(11)-Sb(8)-Cl(23)	75.018(18)
Cl(22)#2-Sb(3)-Cl(31)#3	113.181(16)	S(12)-Sb(8)-Cl(23)	87.438(19)
Cl(54)#3-Sb(3)-Cl(31)#3	74.480(18)	S(9)-Sb(8)-Cl(23)	162.256(17)
S(5)-Sb(4)-S(4)	83.998(19)	Cl(32)-Sb(8)-Cl(23)	119.358(17)
S(5)-Sb(4)-S(7)	89.74(2)	Cl(34)#2-Sb(8)-Cl(23)	87.677(19)
S(4)-Sb(4)-S(7)	81.443(18)	S(11)-Sb(9)-S(9)	88.18(2)
S(5)-Sb(4)-S(1)	86.52(2)	S(11)-Sb(9)-S(10)	97.90(2)
S(4)-Sb(4)-S(1)	81.831(18)	S(9)-Sb(9)-S(10)	90.49(2)
S(7)-Sb(4)-S(1)	163.152(18)	S(11)-Sb(9)-Cl(21)#3	80.25(2)
S(5)-Sb(4)-S(6)	80.976(18)	S(9)-Sb(9)-Cl(21)#3	156.484(19)
S(4)-Sb(4)-S(6)	157.290(18)	S(10)-Sb(9)-Cl(21)#3	71.136(17)
S(7)-Sb(4)-S(6)	81.558(17)	S(11)-Sb(9)-Cl(33)#3	137.73(2)
S(1)-Sb(4)-S(6)	113.967(17)	S(9)-Sb(9)-Cl(33)#3	72.269(19)
S(5)-Sb(4)-S(3)	159.056(18)	S(10)-Sb(9)-Cl(33)#3	118.763(19)
S(4)-Sb(4)-S(3)	79.080(17)	Cl(21)#3-Sb(9)-Cl(33)#3	129.156(18)
S(7)-Sb(4)-S(3)	99.766(18)	S(11)-Sb(9)-Sb(8)	43.865(14)
S(1)-Sb(4)-S(3)	78.983(18)	S(9)-Sb(9)-Sb(8)	45.415(15)
S(6)-Sb(4)-S(3)	118.696(16)	S(10)-Sb(9)-Sb(8)	88.085(14)
S(5)-Sb(5)-S(6)	94.49(2)	Cl(21)#3-Sb(9)-Sb(8)	117.226(15)
S(5)-Sb(5)-S(8)	98.92(2)	Cl(33)#3-Sb(9)-Sb(8)	112.999(12)
S(6)-Sb(5)-S(8)	87.27(2)	S(12)-Sb(10)-S(13)	90.304(19)
S(5)-Sb(5)-Cl(12)	89.55(2)	S(12)-Sb(10)-S(14)	79.93(2)
S(6)-Sb(5)-Cl(12)	71.510(19)	S(13)-Sb(10)-S(14)	83.712(18)
S(8)-Sb(5)-Cl(12)	157.764(19)	S(12)-Sb(10)-S(10)	84.34(2)
S(5)-Sb(5)-Cl(43)	74.376(18)	S(13)-Sb(10)-S(10)	83.608(18)
S(6)-Sb(5)-Cl(43)	163.818(18)	S(14)-Sb(10)-S(10)	159.694(18)
S(8)-Sb(5)-Cl(43)	83.020(19)	S(12)-Sb(10)-S(11)	82.087(18)
Cl(12)-Sb(5)-Cl(43)	119.150(17)	S(13)-Sb(10)-S(11)	163.557(17)
S(5)-Sb(5)-Sb(6)	93.187(14)	S(14)-Sb(10)-S(11)	109.055(18)
S(6)-Sb(5)-Sb(6)	43.292(14)	S(10)-Sb(10)-S(11)	81.160(18)
S(8)-Sb(5)-Sb(6)	44.648(14)	S(12)-Sb(10)-S(15)	152.548(18)
Cl(12)-Sb(5)-Sb(6)	114.775(13)	S(13)-Sb(10)-S(15)	79.019(17)
Cl(43)-Sb(5)-Sb(6)	124.200(13)	S(14)-Sb(10)-S(15)	73.848(18)
S(6)-Sb(6)-S(8)	88.51(2)	S(10)-Sb(10)-S(15)	118.942(18)
S(6)-Sb(6)-S(7)	96.625(19)	S(11)-Sb(10)-S(15)	113.937(16)
S(8)-Sb(6)-S(7)	89.16(2)	S(16)-Sb(11)-S(14)	98.58(2)
S(6)-Sb(6)-Cl(11)#3	82.64(2)	S(16)-Sb(11)-S(13)	88.83(2)
S(8)-Sb(6)-Cl(11)#3	159.38(2)	S(14)-Sb(11)-S(13)	89.292(19)
S(7)-Sb(6)-Cl(11)#3	73.505(18)	S(16)-Sb(11)-Cl(53)#5	81.42(2)
S(6)-Sb(6)-Cl(42)#4	70.015(18)	S(14)-Sb(11)-Cl(53)#5	71.539(18)
S(8)-Sb(6)-Cl(42)#4	87.921(19)	S(13)-Sb(11)-Cl(53)#5	156.758(19)
S(7)-Sb(6)-Cl(42)#4	166.387(16)	S(16)-Sb(11)-Sb(12)	43.010(14)
Cl(11)#3-Sb(6)-Cl(42)#4	106.276(17)	S(14)-Sb(11)-Sb(12)	88.687(15)
S(6)-Sb(6)-Sb(5)	43.343(14)	S(13)-Sb(11)-Sb(12)	46.711(15)
S(8)-Sb(6)-Sb(5)	45.854(15)	Cl(53)#5-Sb(11)-Sb(12)	117.753(15)
S(7)-Sb(6)-Sb(5)	87.989(13)	S(16)-Sb(11)-Sb(10)	101.287(16)
Cl(11)#3-Sb(6)-Sb(5)	120.633(14)	S(14)-Sb(11)-Sb(10)	46.850(14)
Cl(42)#4-Sb(6)-Sb(5)	80.448(11)	S(13)-Sb(11)-Sb(10)	43.158(14)
S(10)-Sb(7)-S(7)	81.94(2)	Cl(53)#5-Sb(11)-Sb(10)	118.243(14)
S(10)-Sb(7)-S(8)	89.185(19)	Sb(12)-Sb(11)-Sb(10)	64.587(7)
S(7)-Sb(7)-S(8)	84.357(19)	S(15)-Sb(12)-S(16)	96.62(2)

S(15)-Sb(12)-S(13)	96.74(2)	Sb(11)-S(13)-Sb(10)	93.95(2)
S(16)-Sb(12)-S(13)	87.01(2)	Sb(11)-S(13)-Sb(12)	87.91(2)
S(15)-Sb(12)-Cl(51)#6	92.03(2)	Sb(10)-S(13)-Sb(12)	98.323(19)
S(16)-Sb(12)-Cl(51)#6	75.269(19)	Sb(11)-S(14)-Sb(13)	98.02(2)
S(13)-Sb(12)-Cl(51)#6	161.033(17)	Sb(11)-S(14)-Sb(10)	91.676(19)
S(15)-Sb(12)-Cl(41)#3	83.651(19)	Sb(13)-S(14)-Sb(10)	101.20(2)
S(16)-Sb(12)-Cl(41)#3	166.101(18)	Sb(12)-S(15)-Sb(13)	101.48(2)
S(13)-Sb(12)-Cl(41)#3	79.176(19)	Sb(12)-S(15)-Sb(10)	85.332(18)
Cl(51)#6-Sb(12)-Cl(41)#3	118.629(16)	Sb(13)-S(15)-Sb(10)	88.761(19)
S(15)-Sb(12)-Cl(12)#4	165.65(2)	Sb(11)-S(16)-Sb(12)	94.51(2)
S(16)-Sb(12)-Cl(12)#4	75.415(19)	Sb(11)-S(16)-Sb(13)	82.861(18)
S(13)-Sb(12)-Cl(12)#4	94.758(17)	Sb(12)-S(16)-Sb(13)	82.577(18)
Cl(51)#6-Sb(12)-Cl(12)#4	74.519(17)	Sb(1)-Cl(1)-Sb(1)#1	100.24(2)
Cl(41)#3-Sb(12)-Cl(12)#4	106.989(16)	Cl(13)-Al(1)-Cl(14)	116.08(4)
Cl(2)-Sb(13)-S(15)	91.27(3)	Cl(13)-Al(1)-Cl(11)	110.63(4)
Cl(2)-Sb(13)-S(14)	89.19(3)	Cl(14)-Al(1)-Cl(11)	106.97(4)
S(15)-Sb(13)-S(14)	94.38(2)	Cl(13)-Al(1)-Cl(12)	106.59(4)
Cl(2)-Sb(13)-S(16)	165.02(3)	Cl(14)-Al(1)-Cl(12)	108.53(4)
S(15)-Sb(13)-S(16)	78.986(19)	Cl(11)-Al(1)-Cl(12)	107.76(4)
S(14)-Sb(13)-S(16)	80.343(18)	Al(1)-Cl(11)-Sb(6)#8	121.09(3)
Cl(2)-Sb(13)-Cl(24)#7	88.25(3)	Al(1)-Cl(12)-Sb(5)	111.94(3)
S(15)-Sb(13)-Cl(24)#7	82.83(2)	Al(1)-Cl(12)-Sb(12)#4	100.24(3)
S(14)-Sb(13)-Cl(24)#7	176.170(19)	Sb(5)-Cl(12)-Sb(12)#4	113.95(2)
S(16)-Sb(13)-Cl(24)#7	101.624(18)	Cl(24)-Al(2)-Cl(23)	112.31(4)
Cl(2)-Sb(13)-Sb(11)	129.43(2)	Cl(24)-Al(2)-Cl(21)	109.92(4)
S(15)-Sb(13)-Sb(11)	87.589(16)	Cl(23)-Al(2)-Cl(21)	109.29(4)
S(14)-Sb(13)-Sb(11)	40.670(14)	Cl(24)-Al(2)-Cl(22)	112.10(4)
S(16)-Sb(13)-Sb(11)	39.758(11)	Cl(23)-Al(2)-Cl(22)	108.77(4)
Cl(24)#7-Sb(13)-Sb(11)	141.382(15)	Cl(21)-Al(2)-Cl(22)	104.11(4)
Sb(2)-S(1)-Sb(1)	100.86(2)	Al(2)-Cl(21)-Sb(9)#8	100.22(3)
Sb(2)-S(1)-Sb(4)	93.039(19)	Al(2)-Cl(22)-Sb(3)#2	106.50(3)
Sb(1)-S(1)-Sb(4)	91.83(2)	Al(2)-Cl(23)-Sb(8)	90.72(3)
Sb(2)-S(2)-Sb(3)	93.93(2)	Al(2)-Cl(24)-Sb(13)#7	102.70(3)
Sb(2)-S(2)-Sb(1)	85.482(18)	Cl(34)-Al(3)-Cl(33)	110.92(4)
Sb(3)-S(2)-Sb(1)	83.794(18)	Cl(34)-Al(3)-Cl(31)	109.68(4)
Sb(3)-S(3)-Sb(1)	101.69(2)	Cl(33)-Al(3)-Cl(31)	107.65(4)
Sb(3)-S(3)-Sb(4)	87.905(18)	Cl(34)-Al(3)-Cl(32)	109.93(4)
Sb(1)-S(3)-Sb(4)	85.201(19)	Cl(33)-Al(3)-Cl(32)	110.51(4)
Sb(4)-S(4)-Sb(3)	98.74(2)	Cl(31)-Al(3)-Cl(32)	108.09(4)
Sb(4)-S(4)-Sb(2)	95.824(19)	Al(3)-Cl(31)-Sb(2)	100.40(3)
Sb(3)-S(4)-Sb(2)	87.83(2)	Al(3)-Cl(31)-Sb(3)#8	103.97(3)
Sb(5)-S(5)-Sb(4)	98.85(2)	Sb(2)-Cl(31)-Sb(3)#8	103.89(2)
Sb(5)-S(5)-Sb(7)	85.980(18)	Al(3)-Cl(32)-Sb(8)	124.84(3)
Sb(4)-S(5)-Sb(7)	90.694(19)	Al(3)-Cl(33)-Sb(9)#8	97.43(3)
Sb(6)-S(6)-Sb(5)	93.37(2)	Al(3)-Cl(34)-Sb(8)#2	127.89(3)
Sb(6)-S(6)-Sb(4)	87.461(18)	Cl(44)-Al(4)-Cl(42)	111.58(4)
Sb(5)-S(6)-Sb(4)	85.540(17)	Cl(44)-Al(4)-Cl(41)	108.26(4)
Sb(6)-S(7)-Sb(7)	94.148(19)	Cl(42)-Al(4)-Cl(41)	109.56(4)
Sb(6)-S(7)-Sb(4)	93.843(18)	Cl(44)-Al(4)-Cl(43)	111.09(4)
Sb(7)-S(7)-Sb(4)	99.83(2)	Cl(42)-Al(4)-Cl(43)	106.12(4)
Sb(6)-S(8)-Sb(5)	89.50(2)	Cl(41)-Al(4)-Cl(43)	110.23(4)
Sb(6)-S(8)-Sb(7)	91.83(2)	Al(4)-Cl(41)-Sb(12)#8	117.32(3)
Sb(5)-S(8)-Sb(7)	93.38(2)	Al(4)-Cl(42)-Sb(6)#4	117.78(3)
Sb(9)-S(9)-Sb(8)	89.78(2)	Al(4)-Cl(43)-Sb(5)	112.23(3)
Sb(9)-S(9)-Sb(7)	89.58(2)	Cl(54)-Al(5)-Cl(52)	106.43(4)
Sb(8)-S(9)-Sb(7)	91.59(2)	Cl(54)-Al(5)-Cl(53)	110.69(4)
Sb(7)-S(10)-Sb(9)	95.13(2)	Cl(52)-Al(5)-Cl(53)	112.03(4)
Sb(7)-S(10)-Sb(10)	100.51(2)	Cl(54)-Al(5)-Cl(51)	109.50(4)
Sb(9)-S(10)-Sb(10)	92.497(18)	Cl(52)-Al(5)-Cl(51)	112.21(4)
Sb(8)-S(11)-Sb(9)	92.11(2)	Cl(53)-Al(5)-Cl(51)	106.01(4)
Sb(8)-S(11)-Sb(10)	85.551(18)	Al(5)-Cl(51)-Sb(12)#9	113.41(3)
Sb(9)-S(11)-Sb(10)	87.724(19)	Al(5)-Cl(52)-Sb(2)	102.27(3)
Sb(10)-S(12)-Sb(8)	97.89(2)	Al(5)-Cl(53)-Sb(11)#10	124.71(3)
Sb(10)-S(12)-Sb(7)	95.61(2)	Al(5)-Cl(54)-Sb(3)#8	99.00(3)
Sb(8)-S(12)-Sb(7)	87.622(18)		