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## ARTICLE

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Synthesis and structure determination of  $\text{AgScP}_2\text{Se}_6$ ,  $\text{AgErP}_2\text{Se}_6$  and  $\text{AgTmP}_2\text{Se}_6$ 

A. Pfitzner\*, S. Seidlmayer

*Dedicated to Professor Reinhard Nesper on the the Occasion of his 60<sup>th</sup> birthday***Keywords:** Selenium; Silver; Scandium; Thulium; Layered Compounds

$\text{AgScP}_2\text{Se}_6$ ,  $\text{AgErP}_2\text{Se}_6$  and  $\text{AgTmP}_2\text{Se}_6$  were obtained by high temperature reaction of stoichiometric amounts of the elements in evacuated silica ampoules. They crystallize in the space-group  $P\bar{3}1c$ ,  $Z = 2$ , with the lattice constants  $a = 6.463$  (1) Å,  $c = 13.349$  (1) Å ( $\text{AgScP}_2\text{Se}_6$ ),  $a = 6.578$  (1) Å,  $c = 13.410$  (2) Å ( $\text{AgErP}_2\text{Se}_6$ ), and  $a = 6.567$  (1) Å,  $c = 13.422$  (1) Å ( $\text{AgTmP}_2\text{Se}_6$ ). The crystal structures of  $\text{AgScP}_2\text{Se}_6$  and  $\text{AgErP}_2\text{Se}_6$  were refined from merohedrally twinned crystals.

No twinning was observed for  $\text{AgTmP}_2\text{Se}_6$ . The crystal structures are isotypic and can be described as a variant of the  $\text{CdI}_2$  structure type. Thus, selenium atoms are hexagonally close packed. The octahedral voids of every second layer are occupied by Ag,  $M^{3+}$ , and  $\text{P}_2$  dumbbells in an ordered fashion. The crystal structures are isotypic to  $\text{AgScP}_2\text{S}_6$  and  $\text{AgInP}_2\text{S}_6$ .

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## Introduction

Hexachalcogenohypodiphosphates have attracted some attention in the past due to their interesting physical and chemical properties.  $\text{Ni}_2\text{P}_2\text{S}_6$  for example has been investigated for its potential use in high-energy lithium batteries [1-4], and  $\text{Sn}_2\text{P}_2\text{S}_6$  for its strong non-linear-optical effects [5]. Hexachalcogenohypodiphosphates form a vast family of compounds, which can roughly be divided into two major groups. One group, exhibiting layered structures and a second group crystallizing in three-dimensional (3D) network structures. Each group can be further subdivided into subgroups based on the structure type, the elemental composition or the oxidation state of the metal ions. The most investigated subgroups are the  $M_2\text{P}_2Q_6$  compounds ( $Q = \text{S}, \text{Se}$ ), where  $M$  denotes a divalent metal cation as Fe, Ni, Cd, Zn, Mn, Sn, Pb, Ba or Sr, and the group  $M^I M^{III} \text{P}_2 Q_6$ , where  $M^I$  is a monovalent cation as Na, K, Ag or Cu, and  $M^{III}$  is a trivalent cation as Cr, V, Al or In.  $M_2\text{P}_2Q_6$  compounds containing transition metals crystallize with layer structures. On the other hand divalent maingroup metal containing hexachalcogenohypodiphosphates tend to form 3D network structures. The transition metal layered structures were investigated for their capability to intercalate various guest species into the van der Waals-gaps [6-9]. Among the 3D network structures  $\text{Sn}_2\text{P}_2\text{S}_6$  is the most intriguing example; the investigations focus on its strong NLO properties [10-12]. In the  $M^I M^{III} \text{P}_2 Q_6$  family, the alkali metal compounds usually form 3D network structures, e.g.,  $\text{NaV}_{0.84}\text{P}_2\text{S}_6$  or  $\text{KSbP}_2\text{S}_6$  [13, 14]. When  $M^I$  is a monovalent transition metal ( $\text{Cu}^{1+}$ ,  $\text{Ag}^{1+}$ ) often layered structures are observed, e.g., for  $\text{AgScP}_2\text{S}_6$  [15],  $\text{AgInP}_2\text{S}_6$  [16],  $\text{CuCrP}_2\text{S}_6$  and  $\text{CuInP}_2\text{S}_6$  [17]. Only a general trend is observable for structure prediction, the larger the cations the more pronounced is the tendency to form 3D-network structures. Obviously this is not satisfactory and more systematic structural studies need to be done and more dedicated factors for structure type formation have to be found.

## Results and Discussion

$\text{AgScP}_2\text{S}_6$ ,  $\text{AgErP}_2\text{S}_6$ , and  $\text{AgTmP}_2\text{S}_6$  are isotopic to  $\text{AgScP}_2\text{S}_6$  [15]. The crystal structures can be derived from the  $\text{CdI}_2$  structure type. Herein, their structures will be discussed by using  $\text{AgScP}_2\text{S}_6$  as a representative. The description holds for the Er and the Tm compound just by replacing Sc by the respective rare earth metals.

Selenium atoms form a close packed hexagonal arrangement in which the octahedral voids of every second layer are occupied in an ordered way by silver, scandium and  $\text{P}_2$  dumbbells. The  $M\text{Se}_6$ -octahedra and  $\text{P}_2\text{Se}_6$ -octahedra within a layer are connected via common edges and form a triangular lattice with an AB-type layer stacking, see Figure 1.

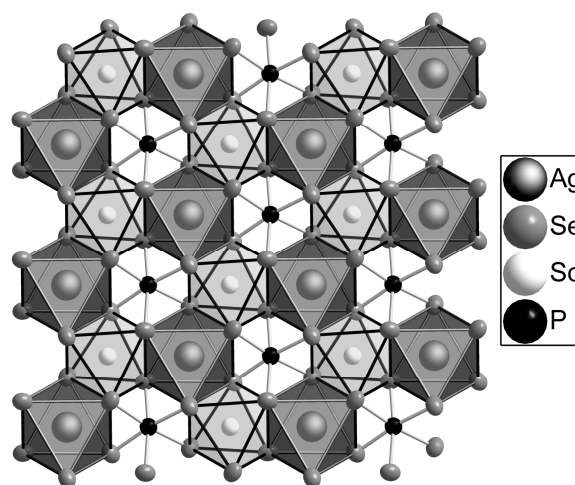
In all compounds the  $[\text{P}_2\text{Se}_6]^{4-}$ -anion has staggered conformation, which is typically observed in hexachalcogenohypodiphosphates. The torsion angles (Se-P-P-Se), se-

lected bond angles and bond lengths  $d(\text{P-P})$  and  $d(\text{P-Se})$  are listed in Table 1.

The P- $\text{PSe}_3$ -tetrahedra have bond angles close to the ideal tetrahedron angle with values ranging from  $110.90^\circ$  to  $111.64^\circ$  for the angles (Se-P-Se) and  $107.20^\circ$  to  $108.00^\circ$  for the angles (P-P-Se). The bond lengths  $d(\text{P-P})$  are close to the usual value for P-P single bonds of  $2.21 \text{ \AA}$ . The distances  $d(\text{P-Se})$  are equal within the threefold standard deviations and vary between  $2.188 \text{ \AA}$  in  $\text{AgErP}_2\text{Se}_6$ , and  $2.192 \text{ \AA}$  in  $\text{AgTmP}_2\text{Se}_6$  and  $\text{AgScP}_2\text{Se}_6$ , respectively.

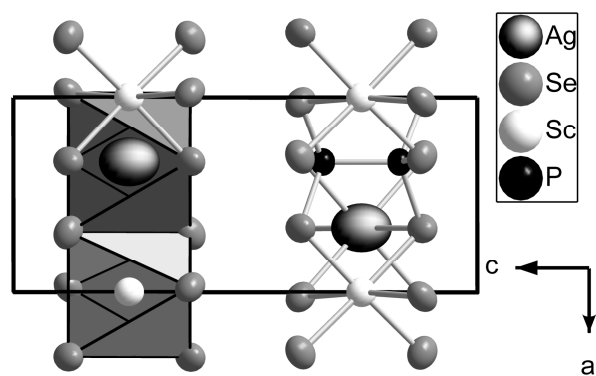
The Ag atoms are sixfold coordinated in slightly distorted octahedra with distances  $d(\text{Ag-Se})$  of  $2.899 \text{ \AA}$  and  $2.909 \text{ \AA}$ . The angles (Se-Ag-Se) deviate only slightly from  $90^\circ$ . The large displacement ellipsoids for the silver atoms are commonly observed in  $\text{AgMP}_2\text{S}_6$  compounds and generally for  $d^{10}$ -ions (e.g., Ag, Cu, Au, Zn, Cd, Hg). They are due to a second order Jahn-Teller-effect. [18, 19]

The  $M^{III}$  atoms are also sixfold coordinated. However, the octahedra  $M^{III}\text{Se}_6$  show slightly larger distortions than the octahedra  $\text{AgSe}_6$ . The corresponding distances are  $d(\text{Sc-Se}) = 2.709 \text{ \AA}$ ,  $d(\text{Tm-Se}) = 2.825 \text{ \AA}$ , and  $d(\text{Er-Se}) = 2.834 \text{ \AA}$ , see Table 2. These distances compare well to those observed in binary selenides of Sc, Er, and Tm.



**Figure 1.** View on (001) of the crystal structure of  $\text{AgScP}_2\text{Se}_6$ . The cations in the octahedral voids of the Se layers are trigonally ordered. Ellipsoids enclose 99% probability.

The height of the van der Waals-gap is about  $3.15 \text{ \AA}$  whereas the height of the Se double layers which are occupied by the cations is about  $3.55 \text{ \AA}$ . The cations in the  $a$ - $b$ -layers are ordered occupying edge-sharing octahedra, forming a triangular honeycomb lattice. Thus distances between equal cations are optimized. The crystal structure of  $\text{AgScP}_2\text{Se}_6$  is displayed in Figures 1 and 2.



**Figure 2.** View on the *a-c* plane of  $\text{AgScP}_2\text{Se}_6$ . The van der Waals-gap has a height of about 3.15 Å. Coordination octahedra are drawn for one Ag and one Sc atom. Ellipsoids represent 99% probability.

**Table 1** Bond lengths and angles in the  $[\text{P}_2\text{Se}_6]^{4-}$ -units

Compound	Torsion angles (Se-P-P-Se) in °	Angles (P-P-Se), (Se-P-Se) in °	<i>d</i> (P-P) in Å	<i>d</i> (P-Se) in Å
$\text{AgScP}_2\text{Se}_6$	172.21(6)	107.20(2)	2.205(3)	2.192(1)
	52.20(8)	111.64(2)		
$\text{AgErP}_2\text{Se}_6$	-67.81(8)	107.77(1)	2.230(2)	2.188(1)
	56.87(6)	111.11(1)		
	-63.14(5)			
$\text{AgTmP}_2\text{Se}_6$	176.5(1)	108.00(3)	2.215(6)	2.192(2)
	56.5(1)	110.90(3)		
	-63.5(1)			

**Table 2** Selected bond lengths and angles for  $\text{AgSe}_6$  and  $M^{\text{III}}\text{Se}_6$  octahedra

Compound	Angles (Se-Ag-Se) in °	Angles (Se- $M^{\text{III}}$ -Se) in °	<i>d</i> (Ag-Se) in Å	<i>d</i> ( $M^{\text{III}}$ -Se) in Å
$\text{AgScP}_2\text{Se}_6$	87.30(2)	87.30(2)	2.899(1)	2.709(1)
	92.31(2)	92.31(2)		
	93.09(2)	93.09(2)		
	179.43(2)	179.43(2)		
$\text{AgErP}_2\text{Se}_6$	86.36(1)	84.61(1)	2.909(1)	2.834(1)
	91.84(1)	92.11(1)		
	95.49(1)	98.90(1)		
	177.35(1)	174.97(1)		
$\text{AgTmP}_2\text{Se}_6$	86.30(2)	84.35(2)	2.909(1)	2.825(1)
	93.10(2)	92.40(3)		
	95.34(2)	99.12(3)		
	177.64(2)	175.01(2)		

## Conclusions

$\text{AgScP}_2\text{Se}_6$ ,  $\text{AgErP}_2\text{Se}_6$ , and  $\text{AgTmP}_2\text{Se}_6$  are three new hexaselenohypodiphosphates with a layered structure of the  $\text{AgInP}_2\text{S}_6$  structure type.  $\text{AgScP}_2\text{Se}_6$  has an optical bandgap of 1.55 eV. Preliminary impedance spectroscopic investigations show no silver ion conductivity.

## Experimental Section

### Synthesis

$\text{AgScP}_2\text{Se}_6$  was prepared by annealing stoichiometric amounts of the pure elements in evacuated silica ampoules at 650 °C for 17 days. The phase pure product was obtained as a brownish powder and very small hexagonal shaped orange platelets. X-ray powder diffraction data of the bulk material confirmed its purity.

$\text{AgErP}_2\text{Se}_6$  was obtained by annealing stoichiometric amounts of the pure elements in evacuated silica ampoules at 600 °C for 4 days. The product was a mixture of a dark brown powder and small dark-brown platelets. Phase pure  $\text{AgErP}_2\text{Se}_6$  was not be obtained. Powder diffraction data showed small amounts of  $\text{Er}_2\text{Se}_3$  as a by-product.

$\text{AgTmP}_2\text{Se}_6$  results from annealing stoichiometric amounts of the pure elements in evacuated silica ampoules at 550 °C for 5 days. The product was a dark-brown powder and dark-brown small platelets.  $\text{Tm}_2\text{Se}_3$  and  $\text{Ag}_4\text{P}_2\text{Se}_6$  always were found as by-products.

### Powder Diffraction

X-ray powder diffraction data were recorded on a STOE STADI P diffractometer with a Ge(111) monochromator providing  $\text{Cu-K}\alpha_1$  radiation. Lattice constants were refined from the powder patterns and then used for the single crystal structure refinements in the last cycles, see Table 3.

### Single-Crystal X-Ray Diffraction

Single crystals suitable for crystal structure determination were mounted on glass fibres and checked for their quality by Weissenberg photographs. X-ray diffraction intensities were collected on a STOE IPDS at 293(2) K using  $\text{Mo-K}\alpha$  radiation. The measurements were performed in an incremental scanning mode. Data were corrected for Lorentz and polarization effects. Absorption was numerically corrected after an optimisation of the crystal shape with the X-SHAPE [20] routine. Further details of the measurements and basic crystallographic information are collected in Table 3. The program package WinGX [21], using SHELXS-97 and SHELXL-97 [22, 23] was used for solving and refining the crystal structures. The refined atom positions and equivalent isotropic displacement parameters are printed in Table 4.

For the structure solution and refinement of  $\text{AgScP}_2\text{Se}_6$  and  $\text{AgErP}_2\text{Se}_6$  merohedral twinning according to a twinning matrix  $\begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix}$  had to be taken into account. Thus, the *R*-values dropped from  $wR_2 = 0.109$  to  $wR_2 = 0.076$  ( $\text{AgScP}_2\text{Se}_6$ ) for a twin ratio of 0.967 : 0.033. For  $\text{AgErP}_2\text{Se}_6$  the initial *R*-value  $wR_2 = 0.614$  dropped significantly to  $wR_2 = 0.036$  with a refined twin ratio of 0.742 : 0.258. No twinning was observed for  $\text{AgTmP}_2\text{Se}_6$ .

**Table 3** Crystallographic data and structure refinement summary for AgScP<sub>2</sub>Se<sub>6</sub>, AgErP<sub>2</sub>Se<sub>6</sub>, and AgTmP<sub>2</sub>Se<sub>6</sub>. Lattice constants were taken from powder data refinements.

Empirical formula	AgScP <sub>2</sub> Se <sub>6</sub>	AgErP <sub>2</sub> Se <sub>6</sub>	AgTmP <sub>2</sub> Se <sub>6</sub>
Formula weight in g/mol	688.54	810.83	812.50
Temperature in K		298(2)	
Wavelength in Å		0.71073 (Mo K $\alpha$ )	
Crystal system		trigonal	
Space group	$P\bar{3}1c$	$P\bar{3}1c$	$P\bar{3}1c$
a in Å	6.463(1)	6.578(1)	6.567(1)
c in Å	13.349(1)	13.410(2)	13.422(2)
Volume in Å <sup>3</sup>	482.93(3)	502.6(1)	501.27(9)
Z	2	2	2
Calculated density in g/cm <sup>3</sup>	4.735	5.358	5.383
$\mu$ in mm <sup>-1</sup>	25.594	32.210	32.773
Crystal colour and shape	orange-brown, platelet	dark-brown, platelet	dark-brown, platelet
Crystal size in mm <sup>3</sup>			
Diffractometer Type		STOE IPDS I	
Index ranges,	$-8 \leq h \leq 8$ $-8 \leq k \leq 8$ $-17 \leq l \leq 17$	$-8 \leq h \leq 8$ $-8 \leq k \leq 8$ $-16 \leq l \leq 16$	$-8 \leq h \leq 7$ $-8 \leq k \leq 8$ $-17 \leq l \leq 17$
Min. $\theta$ in °	3.05	3.04	3.04
Max. $\theta$ in °	27.88	26.69	27.85
Reflections (collected / unique)	5119 / 394	6968 / 362	4480 / 407
$R_{int}$ , $R_{\sigma}$	0.0841, 0.0335	0.0432, 0.0142	0.1242, 0.0538
Data / restraints / parameters	394 / 0 / 19	362 / 0 / 19	407 / 0 / 17
Goof	1.152	1.167	1.025
$R_1$ , $wR_2$ (all reflections)	0.0453, 0.0764	0.0196, 0.0356	0.0580, 0.0785
Extinction coefficient	0.0087(11)	0.0019(3)	-
twin matrix	$\bar{1}00, 0\bar{1}0, 001$	$\bar{1}00, 0\bar{1}0, 001$	-
twin fractions	0.967(2):0.033(2)	0.742(2):0.258(2)	-

**Table 4** Refined atomic parameters and equivalent isotropic displacement parameters  $U_{Eq}$  of the title compounds.

Compound	Atom	Wyckoff position	x	y	z	$U_{Eq}$ in Å <sup>2</sup>
AgScP <sub>2</sub> Se <sub>6</sub>	Ag1	2d	$\frac{2}{3}$	$\frac{1}{3}$	$\frac{1}{4}$	0.0515(5)
	Sc1	2a	0	0	$\frac{1}{4}$	0.0167(3)
	Se1	12i	0.0229(1)	0.33077(8)	0.11885(5)	0.0154(5)
	P1	4f	$\frac{1}{3}$	$\frac{2}{3}$	0.1674(2)	0.0130(5)
AgErP <sub>2</sub> Se <sub>6</sub>	Ag1	2d	$\frac{2}{3}$	$\frac{1}{3}$	$\frac{1}{4}$	0.0643(4)
	Er1	2a	0	0	$\frac{1}{4}$	0.0184(1)
	Se1	12i	0.02183(6)	0.34516(6)	0.11705(3)	0.0209(1)
	P1	4f	$\frac{1}{3}$	$\frac{2}{3}$	0.1669(1)	0.0148(3)
AgTmP <sub>2</sub> Se <sub>6</sub>	Ag1	2d	$\frac{2}{3}$	$\frac{1}{3}$	$\frac{1}{4}$	0.0688(8)
	Tm1	2a	0	0	$\frac{1}{4}$	0.0244(3)
	Se1	12i	0.0216(2)	0.3439(1)	0.11704(7)	0.0270(3)
	P1	4f	$\frac{1}{3}$	$\frac{2}{3}$	0.1675(3)	0.0206(8)

### UV-Vis spectroscopy

UV-Vis data of AgScP<sub>2</sub>Se<sub>6</sub> were collected on a Bruins instruments Omega 20 two-wave photometer in remission mode. The obtained data were transformed into absorption spectra by using the Kubelka-Munk function. The value for the band-gap was extrapolated by linear regression of the absorption edge kink. The extrapolated band-gap is 1.55 eV.

### Thermal analysis

DTA measurements were performed in sealed thin walled silica ampoules on a SETARAM TG-DTA 92-16.18.

The measurements showed peritectic decomposition for the compounds under discussion. Decomposition temperatures are 654 °C (AgErP<sub>2</sub>Se<sub>6</sub>), 657 °C (AgTmP<sub>2</sub>Se<sub>6</sub>), and 711 °C (AgScP<sub>2</sub>Se<sub>6</sub>), respectively. After the initial heating a second peak is observed in all samples at 569-572 °C which is attributed to Ag<sub>4</sub>P<sub>2</sub>Se<sub>6</sub> which could be detected in the powder diffraction patterns.

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