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Research Article

Phase Equilibria in the $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ Quasiternary System

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Abstract

For the first time the phase equilibrium of the $Tl_4P_2Se_6$–$TlInP_2Se_6$ binary and $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ ternary systems were investigated by the methods of physical-chemical analysis (DTA, XRD, MSA) and mathematical design in multi-component system. It has been established that the $Tl4P_2Se_6$–$TlInP_2Se_6$ system belongs to the eutectic type and characterized by the formation of boundary solid solutions on the basis of complex compounds. The space phase diagrams, projection of liquidus surface of the $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ system were constructed. Ternary system characterized by two invariant processes – eutectic (L ↔ mtm$Tl_4P_2Se_6$ + $TlInSe_2$ + htm$TlInP_2Se_6$,705K) and peritectic (L + hhtm$Tl_4P_2Se_6$ ↔ $TlInSe_2$ + htm$TlInP_2Se_6$,739K). On the basis of complex selenophosphate compounds takes place metatectic, eutectoid ($Tl_4P_2Se_6$) and two peritectoid ($TlInP_2Se_6$) processes, respectively. Specifics of crystal structure of complex compounds were given from the position of the second coordination surrounded of atoms in cationic and anionic sublattice

Keywords: Selenophosphate; Phase diagram; Liquidus projection; Solid solution; Crystal structure

Introduction

Creation of new materials with a complex of predicted properties is the most important task of the modern inorganic science. The solution of these problems is based on the obtaining of new substances by changing of the composition by iso- and heterovalent substitution of the structures, the formation of solid solutions, composite eutectic (peritectic) phase. The study of the physical-chemical interaction of multi-component systems allows us to determine the phase formation patterns according to the composition and temperature, to determine the boundary concentration of solid solutions, to find coordinates of invariant transformations, to select the rational composition and obtain technological mode of growth qualitative single crystals, to consider the laws of “composition – crystalline structure – properties”.

Perspective compounds that are widely used in production of working elements for semiconductor IR and laser technology, thermal generation, solar power, are materials based on complex chalcogenide compounds [1-4]. Special attention is paid to compounds of the $M_2P_2Se_6$ type [5-12]. Modification of the composition of $M_2P_2Se_6$ type compounds by isovalent substitutions of the chalcogen S→Se, which form the sublattice of the anionic group $[P_2X_6]^{4-}$, as well as Sn^{2+}→Pb^{2+}, heterovalent substitutions $2Sn^{2+}$→$4M^{1+}$ (M1 – K^+, Na^+, Rb^+, Tl^+, Ag^+, Cu^+), $2Sn^{2+}$→$M1^+$+$M2^{3+}$ (M2 – In^{3+}, Sb^{3+}, Bi^{3+}, Fe^{3+}) leads to the formation of new composition with different structure of cationic sublattice, which is accompanied by a change in crystal-chemical parameters [13].

Study of physical-chemical interaction in the Tl_2Se–In_2Se_3–“P_2Se_4” system showed that intermediate complex selenides which melts congruently In_2Se_3 (655K) [14], $TlInSe_2$ (1023 K) [14], $Tl_4P_2Se_6$ (758K) [15-17], $In_4(P_2Se_6)_3$ (880K) [16], $TlInP_2Se_6$ (875K) [16,18] form five quasi-binary eutectic type sections with formation of limited solid solution. Quasi-binary sections divided initial Tl_2Se–In_2Se_3–“P_2Se_4” ternary system on secondary quasiternary systems [13], one of them is $TlInSe_2$–$Tl_4P_2Se_6$–$TlInP_2Se_6$.

Materials and Methods

$TlInSe_2$, $Tl_4P_2Se_6$, $TlInP_2Se_6$ ternary compounds were prepared by the single-temperature method from stoichiometric amounts of the initial Tl_2Se binary compound and elementary In, P, Se in evacuated quartz containers. Synthesis of compounds was carried out with high-purity elements (more than 99.99wt.%). The highest synthesis temperatures were for $TlInSe_2$ – 1073K, $Tl_4P_2Se_6$ – 893K, $TlInP_2Se_6$ – 853K, respectively. Speed of heating and cooling were 25-30K per hour. Maximum temperature for synthesis of binary and ternary alloys was 1073K. After thermal treatment at highest temperature (823K) for 48h the samples were slowly cooled (250K per hour) down to 573K and homogenized at this temperature for 336h. Subsequently the ampoules were quenched into cold water.

The phase equilibria in the ternary system were investigated by the differential thermal (DTA, a chromel-alumel thermocouple, with an accuracy of ±5K), X-ray powder diffraction (DRON-3-13 diffractometer, Cu Kα radiation, Ni filter), microstructure (MSA, metallographic microscope Lomo Metam R1) analyses in combination with the simplex method of mathematical modeling of phase equilibria in multi-component systems [19,20]. Crystal structure calculation was carried out with program WinCSD [21].

Results and Discussion

$Tl_4P_2Se_6$–$TlInP_2Se_6$ quasibinary system

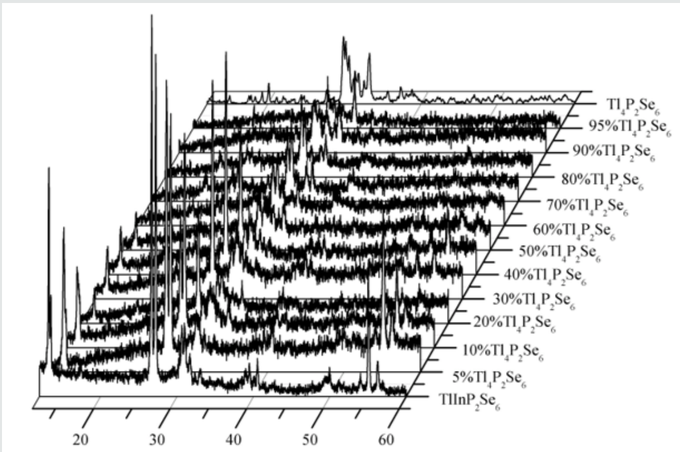

Figure 1: Results of X-ray analysis of the $Tl_4P_2Se_6$–$TlInP_2Se_6$ system.

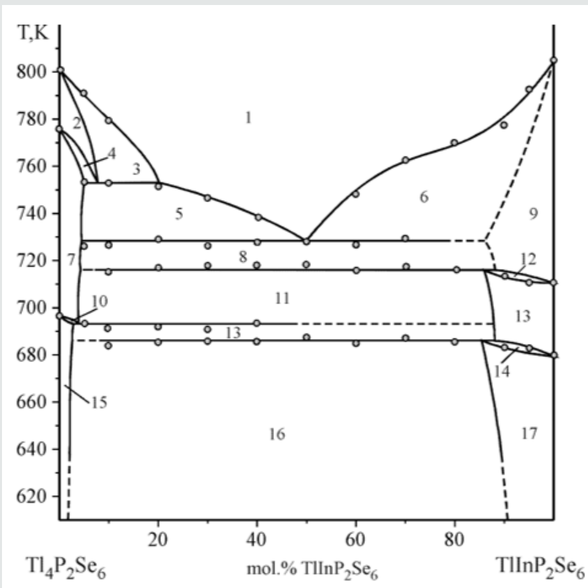

Figure 2: Phase diagram of the $Tl_4P_2Se_6$–$TlInP_2Se_6$ system.

1–L, 2–htm$Tl_4P_2Se_6$, 3–L+htm$Tl_4P_2Se_6$, 4–htm$Tl_4P_2Se_6$+mtm$Tl_4P_2Se_6$, 5–L+mtm $Tl_4P_2Se_6$, 6–L+htm$TlInP_2Se_6$, 7–mtm $Tl_4P_2Se_6$, 8–mtm $Tl_4P_2Se_6$+htm$TlInP_2Se_6$, 9–htm$TlInP_2Se_6$, 10–mtm $Tl_4P_2Se_6$+ltm $Tl_4P_2Se_6$, 11–mtm $Tl_4P_2Se_6$+mtm$TlInP_2Se_6$, 12–htm$TlInP_2Se_6$+mtm$TlInP_2Se_6$, 13–mtm$TlInP_2Se_6$, 14–mtm$TlInP_2Se_6$+ltm$TlInP_2Se_6$, 15–ltm $Tl_4P_2Se_6$, 16–ltm $Tl_4P_2Se_6$+ltm$TlInP_2Se_6$, 17–ltm$TlInP_2Se_6$

Results of X-ray investigation and phase diagram of the $Tl_4P_2Se_6$–$TlInP_2Se_6$ system are presented in Figures 1 & 2. In this system are formed γ–, γ'– γ''– solid solutions based on low-, middle-, high-temperature polymorphic modifications (*ltm-, mtm-, htm-*) of $TlInP_2Se_6$ and ε–, ε'–, ε'– solid solutions based on low-, middle-, high-temperature polymorphic modifications of $Tl_4P_2Se_6$, respectively. $Tl_4P_2Se_6$–$TlInP_2Se_6$ system belongs to the Rozeboom V type and is characterized by the invariant eutectic processes L ↔ mtm $Tl_4P_2Se_6$ + htm$TlInP_2Se_6$. The coordinates of the eutectic point correspond to 50mol.% $TlInP_2Se_6$ at 729K. The metatectic process htm$Tl_4P_2Se_6$ ↔ L + mtm$Tl_4P_2Se_6$ and eutectictoid process based on the polymorphic transformation of the ternary compound $Tl_4P_2Se_6$ are observed at 750K and 695K, respectively. Two peritectictoid processes htm$TlInP_2Se_6$ ↔ mtm$TlInP_2Se_6$ and mtm$TlInP_2Se_6$ + ltm$Tl_4P_2Se_6$ ↔ ltm$TlInP_2Se_6$ based on the polymorphic transformation of $TlInP_2Se_6$ takes place at 715K and 685K. At 573K the existence of the solid solutions range of low-temperature polymorphic modification of $TlInP_2Se_6$ is less than 7mol.%, for ltm$Tl_4P_2Se_6$ – do not exceed than 5mol.% [22,23].

$TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ quasiternary system

A perspective view and the projection of the liquidus surface of the $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ system are shown in Figures 3 & 4, respectively. The points B, C and D, which are located on the edges of triangular prism, represent the melting temperature of the ternary selenophosfates $TlInSe_2$ (1029K), $Tl_4P_2Se_6$ (789K), $TlInP_2Se_6$ (875K). The points C', C'', D' and D'' represent the temperature of polymorphic transformation of $TlInP_2Se_6$ and $TlInP_2Se_6$ compounds. The liquidus of the ternary system consists of four primary crystallization areas: $TlInSe_2$(B)–e4–E2–U3–e5–$TlInSe_2$(B) (β phase), $Tl_4P_2Se_6$(C)–e5–U3–e6–m3–$Tl_4P_2Se_6$(C) (ε'' phase), $TlInP_2Se_6$(D)–e4–E2– $TlInP_2Se_6$(D) (γ'' phase) and m3–U3–E2–e6–m3 (ε' phase). The fields of primary crystallization are divided by monovariant lines e4–E2 (process L↔ $TlInSe_2$ + htm$TlInP_2Se_6$), e5–U3 (process L↔ $TlInSe_2$ + htm$Tl_4P_2Se_6$), m3–U3 (process htm$Tl_4P_2Se_6$ ↔ L + mtm$Tl_4P_2Se_6$), e6–E2 (process L↔ mtm$Tl_4P_2Se_6$ + htm$Tl_4P_2Se_6$), U3–E2 (process L↔ $TlInSe_2$ + mtm$Tl_4P_2Se_6$) which cross at the ternary invariant peritectic points U3 (17mol.% $TlInSe_2$, 62mol.% $Tl_4P_2Se_6$, 21mol.% $TlInP_2Se_6$, 739K) and ternary invariant eutectic points E2 (22mol.% $TlInSe_2$, 46mol.% $Tl_4P_2Se_6$, 32mol.% $TlInP_2Se_6$, 705K). In the subliquidus part two surfaces depict the monovariant metatectic process at 739K (b8–U3–c8–b8) based on the polymorphic transformation between the middle- and high-temperature modifications of the ternary compound $Tl_4P_2Se_6$ (739K) and invariant eutectic process at 705K (b7–c7–d7–b7). In subsolidus part three surfaces describe the invariant eutectoid process at 675K (b5–c5–d5–b5) based on the polymorphic transformation mtm$Tl_4P_2Se_6$ ↔ ltm$Tl_4P_2Se_6$, invariant peritectoid process at 685K (b6–c6–d6–b6) based on the polymorphic transformation htm$TlInP_2Se_6$ ↔ mtm$TlInP_2Se_6$ and invariant peritectoid process at 642K (b4–c4–d4–b4) based on the polymorphic transformation mtm$TlInP_2Se_6$ ↔ ltm$TlInP_2Se_6$. At temperature below for 705K all alloys are in solid state phase. New complex compounds were not observed in the ternary system. The types and temperature of the processes in the $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ quasiternary system are shown in Table 1.

Table 1: Types, temperature of the processes in the $TlInSe_2$–$TlInP_2Se_6$– $Tl_4P_2Se_6$ system.

Types	Processes	Temperature,K
melting of $TlInSe_2$	B: $TlInSe_{2(sol)}$ ↔$TlInSe_{2(liq)}$	1029
melting of $Tl_4P_2Se_6$	C: $Tl_4P_2Se_{6(sol)}$ ↔$Tl_4P_2Se_{6(liq)}$	789
polymorphic transformation of $Tl_4P_2Se_6$	C': htm$Tl_4P_2Se_6$↔mtm$Tl_4P_2Se_6$	775
polymorphic transformation of $Tl_4P_2Se_6$	C'': mtm$Tl_4P_2Se_6$↔ltm$Tl_4P_2Se_6$	698
melting of $TlInP_2Se_6$	D: $TlInP_2Se_{6(sol)}$ $TlInP_2Se_{6(liq}$	875
polymorphic transformation of $TlInP_2Se_6$	D': htm$TlInP_2Se_6$↔mtm$TlInP_2Se_6$	711
polymorphic transformation of $TlInP_2Se_6$	D'': mtm$TlInP_2Se_6$↔ltm$TlInP_2Se_6$	680
binary invariant eutectic	e4: L↔ $TlInSe_2$ + htm$TlInP_2Se_6$	774
binary invariant eutectic	e5: L↔ $TlInSe_2$ + htm$Tl_4P_2Se_6$	776
binary invariant eutectic	e6: L↔ mtm$Tl_4P_2Se_6$ + htm$TlInP_2Se_6$	729
binary invariant metatectic	m3: htm $Tl_4P_2Se_6$↔L + mtm$Tl_4P_2Se_6$	756
monovariant eutectic	e4–E2: L↔ $TlInSe_2$ + htm$TlInP_2Se_6$	774–705
monovariant eutectic	e5–U3: L↔$TlInSe_2$ + htm$Tl_4P_2Se_6$	776–705
monovariant eutectic	e6–E2: L↔ mtm$Tl_4P_2Se_6$ + htm$TlInP_2Se_6$	729–705
monovariant metatectic	m3–U3: htm$Tl_4P_2Se_6$↔L + mtm$Tl_4P_2Se_6$	756–739
monovariant peritectic	U3–E2: L + htm$Tl_4P_2Se_6$↔ $TlInSe_2$	739–705
ternary invariant peritectic	U3: L + htm$Tl_4P_2Se_6$↔ $TlInSe_2$ + mtm$Tl_4P_2Se_6$	739
ternary invariant eutectic	E2: L↔ $TlInSe_2$ + mtm$Tl_4P_2Se_6$ + htm$TlInP_2Se_6$	705

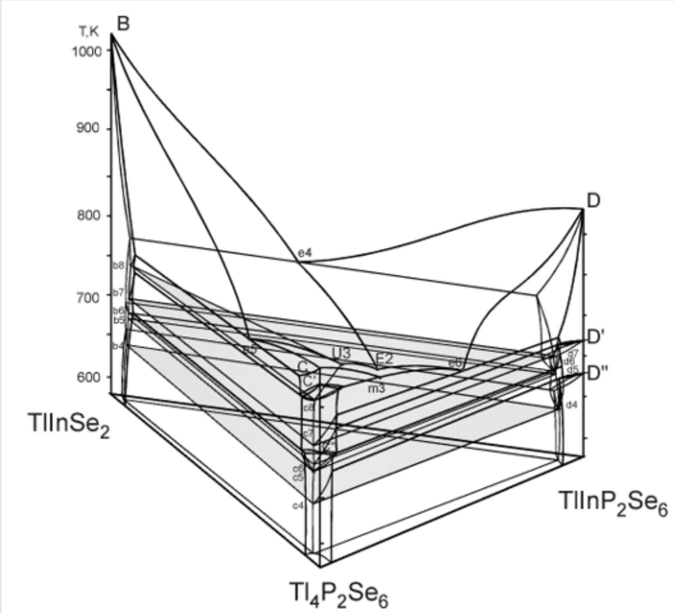

Figure 3: Perspective view of the $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ system.

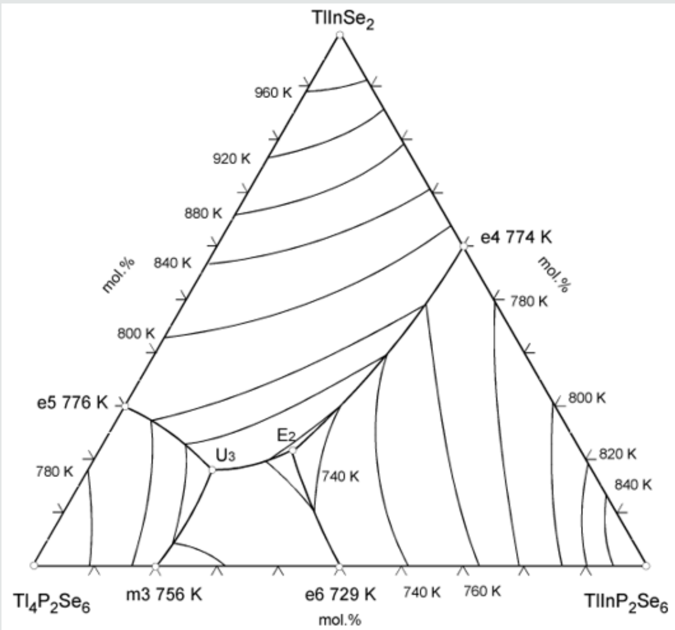

Figure 4: Liquidus surface of the $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ system.

Crystal-chemistry of the $TlInSe_2$, $TlInP_2Se_6$ and $Tl_4P_2Se_6$ compounds

Table 2: Lattice parameters of $TlInSe_2$, $TlInP_2Se_6$ and $Tl4P_2Se_6$ complex compounds.

Copmound	Lattice Parameters
$TlInSe_2$ [17]	SG I4/mcm, a=8.064 (4), c=6.833 (4) Å
$TlInP_2Se_6$ [16]	SG P$\bar{1}$, a =6.431(14), b =7.500(16), c =12.124(3) Å α=100.55(5)°, β=93.74(5)°, γ=113.45(4)°
$Tl_4P_2Se_6$ [17]	SG *P12₁/c1, a=12.239(2), b=9.055(2), c=12.328(2) Å, β=98.83(1)*

Crystal-structure studies of $TlInSe_2$, $TlInP_2Se_6$ and $Tl_4P_2Se_6$ complex chalcogenides were carried out by a powder method, refinement of the structural parameters – by the Rietveld method. The lattice parameters of initial $TlInSe_2$, $TlInP_2Se_6$ and $Tl_4P_2Se_6$ compounds are shown in Table 2.

The second coordination surrounding (SCS) of the anions atoms indicates the diversity of anionic sublattices in the compounds of the system. In the structure of the $TlInSe_2$ compound individual selenium atoms form anionic sublattice. SCS of atoms Se formed defective (-1) hexagonal analogue of the cubo-octahedron (Figure 5). The defect in anionic sublattice indicates covalent type of the cation – anion bonds.

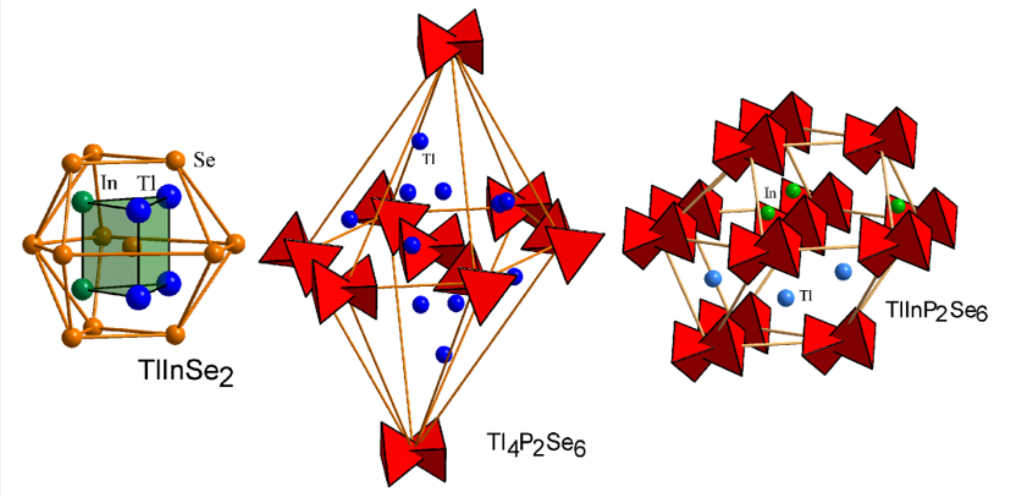

Figure 5: Second coordination surrounding (SCS) of anionic atoms in the structure of $TlInSe_2$, $TlInP_2Se_6$ and $Tl_4P_2Se_6$ compounds.

The crystal analysis of $Tl_4P_2Se_6$ and $TlInP_2Se_6$ complex compounds showed that in the structure of both compounds can be isolated anionic group of atoms $[P_2Se_6]^{4-}$ in the form of two fused tetrahedrons (occupied the nodes of the anionic sublattice). In the structure of the $TlInP_2Se_6$ compound atoms In are displaced in the tetrahedral cavity and located on the verge of tetrahedral and octahedral cavities, atoms Tl are displaced into octahedral cavities. Atoms of cations are absent in the gap between layers of anionic groups $[P_2Se_6]^{4-}$. SCS in the form of a hexagonal analogue of the cubo-octahedron for $TlInP_2Se_6$ compound indicate that the anions are dense packing in this compound. With an increase in the number of cations atoms per anionic group in $Tl_4P_2Se_6$ compound atoms Tl are displaced into tetrahedral cavities and evenly distributed in space. SCS in the form of a hexagonal bipiramide for $Tl_4P_2Se_6$ compound indicates the primitive packing of anion atoms.

Conclusion

Differential thermal, X-ray phase, microstructure analyses and mathematical modeling of phase equilibria in the multi-component systems were used to construct the of the $Tl_4P_2Se_6$–$TlInP_2Se_6$ binary system, perspective view and liquidus surface projection of the $TlInSe_2$–$TlInP_2Se_6$–$Tl_4P_2Se_6$ ternary system. The character of the monovariant processes, the temperatures and coordinate of the invariant processes in the ternary system were determined. In this system there exists ternary invariant peritectic process (U3: 17mol.% $TlInSe_2$, 62mol.% $Tl_4P_2Se_6$, 21mol.% $TlInP_2Se_6$, 739K) and ternary invariant eutectic process (E2: 22mol.% $TlInSe_2$, 46mol.% $Tl_4P_2Se_6$, 32mol.% $TlInP_2Se_6$, 705K). Also, in the system take places monovariant metatectic process mtm$Tl_4P_2Se_6$ ↔ htm$Tl_4P_2Se_6$ (739K), invariant eutectoid process mtm$Tl_4P_2Se_6$ ↔ ltm$Tl_4P_2Se_6$ (675K), invariant peritectoid process htm$TlInP_2Se_6$ ↔ mtm$TlInP_2Se_6$ (685K) and invariant peritectoid process mtm$TlInP_2Se_6$ ↔ ltm$TlInP_2Se_6$ (642K). The existence of solid solutions of the ternary compounds $TlInSe_2$, $TlInP_2Se_6$, $Tl_4P_2Se_6$ was established. In the ternary system, the components of which are compounds with different types of anionic sublattices, it was necessary to wait for the formation of new phases with a layered structure: separate atoms of selenium – paired tetrahedrons. But the results of physical-chemical investigations showed that the new complex compounds were not formed in the ternary system.

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