**REPUBLIC OF AZERBAIJAN**

*On the rights of the manuscript*

# ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

# INVESTIGATION OF THE INTERACTION CONDITIONS OF ARSENIC(V) SULFIDE WITH THE SALTS OF $Cu^{+}$, $Ag^{+}$, $Tl^{+}$, $Cu^{+2}$, $Pb^{+2}$ IN AQUEOUS AND ORGANIC MEDIUM, PHYSICOCHEMICAL STUDY OF THE OBTAINED COMPOUNDS

Speciality: 2303.01 – Inorganic chemistry

Field of science: Chemistry

Applicant: **Huseyn Abulfaz Imanov**

**NAKHCHIVAN – 2022**

The work was performed at the laboratory "Chemistry and technology of mineral raw materials" of the Institute of Natural Resources of Nakhchivan Branch of ANAS.

**Scientific supervisor:** D.Sci. Chem.,
**Bayram Zulfugar Rzayev**

**Official opponents:** D.Sci. Chem., professor
**Yasin Nagy Babayev**
D.Sci. Chem., professor
**Imir Ilyas Aliyev**
Ph.D. Chem. Ass.Dr.
**Gulbuta Gulbala Shafagatova**

Dissertation council FD 1.24 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Nakhchivan branch of the Azerbaijan National Academy of Sciences.

Chairman of the Dissertation council: The corresponding member of ANAS, D.Sci. Chem., professor **Tofig Abbasali Aliyev**

Scientific secretary of the Dissertation council: Ph.D. Chem. Ass.Dr. **Gorkhmaz Mansur Huseynov**

Chairman of the scientific seminar: D.Sci. Chem., professor **Akif Shikhan Aliyev**

## GENERAL CHARACTERISTICS OF WORK

**The relevance and usage rate of the topic.** The intensive development of electrical engineering, nanotechnology and computer technology in modern times is directly related to the development of technological methods for the synthesis and application of semiconductor materials. The achievements in this direction play a decisive role in the development of civilizations. In order to meet the demand for binary and triple semiconductor compounds in industry and technology, there is a great need to improve semiconductor materials and develop new, simpler methods for obtaining nanoparticles and thin films.

Thin layers of arsenic binary compounds ($As_2S_3$, $As_2S_5$, $As_2Se_3$, $As_2Te_3$) from glassy chalcogenide semiconductor materials are successfully used in waveguides as nonlinear optical devices for all optical signal processors. In addition, optical fiber is produced from arsenic binary compounds with sulfur, and researchers are studying the technological conditions for the production of integrated optical elements based on thin layers of these compounds.

It is widely used in the manufacture of parts for optical devices, teletechnics and microelectronics from chemically resistant semiconductor compounds synthesized from the Cu-As-S system with optoelectronic properties. The copper(I) thioarsenate compound has recently been used as a promising single-junction solar absorber in solar cells.

Thio compounds of silver containing $AgAsS_2$ $Ag_3AsS_3$ and $Ag_3AsS_4$ are among the important functional materials of modern technology. Obtained compounds and glasses from the Ag-As-S system widely used in the preparation of valuable semiconductor, photo- and ferroelectric materials, solid electrolytes, electrochemical sensors, electrochemical screens, etc.

Materials obtained from thallium chalcogenides and their solid, liquid and glass derivatives with other elements have great potential for industrial use. Arsenic triple chalcogenides in the Tl-As(V)-S system are promising as functional materials in modern

electronics engineering. The multi-component phases of these compounds have photoelectric, thermal, ferroelectric, optoacoustic and other important properties.

The glasses of thio compounds obtained from the Pb-As(V)-S system are promising materials for a number of potential applications in the fields which are far-infrared optics, chemical sensors, nonlinear photonics, etc. Recently, the electronic properties of amorphous thin layers obtained from the Pb-As(V)-S system allow their application in electronic and opto-electronic devices.

Taking into account the possibility of applying the results obtained in the study, because the methods of synthesis of triple compounds from the $Me^{+n}$-$As_2S_5$ ($Me^{+n}$ = $Cu^{+}$, $Cu^{+2}$, $Ag^{+}$, $Tl^{+}$, $Pb^{+2}$) system in aqueous and organic medium have not been developed, the conditions for their acquisition are not studied and have been rarely found in literary materials, the development of new and simple sedimentation methods, determination of the characteristics of chemical interactions in this system, selection of optimal conditions and physico-chemical study of obtained thio compounds have scientific and practical importance and ensures the relevance of the dissertation.

**Object and subject of research.** In the presented dissertation work, the object of research was triple compounds synthesized in aqueous and organic environment on the basis of arsenic(V) sulfide and $Cu^{+}$, $Cu^{+2}$, $Ag^{+}$, $Tl^{+}$, $Pb^{+2}$ salts, and extensive research and laboratory work was carried out to obtain them. Arsenic(V) sulfide was synthesized as the initial material for the study. Physico-chemical properties of thio compounds obtained during the researches were studied and their areas of application were determined. The subject of the study was the study of the interaction conditions of arsenic(V) sulfide and salts of copper, silver, thallium, lead by the method of precipitation in solution.

**Aims and objectives of the research.** As an alternative to the ampoule synthesis method the aim is to develop various methods for obtaining arsenic(V) sulfide in aqueous and organic medium, and then to develop methods for the study of synthesis conditions of triple

sulfides in aqueous and organic medium based on arsenic(V) sulfide with the copper, silver, thallium, lead salts. In addition, one of the aims of the work was to develop new methods for obtaining nano- and microparticles of synthesized compounds. At the same time, research has been conducted to obtain thin layers of compounds.

In order to achieve the purpose of the research, the following issues are addressed:

- Development of a new method for obtaining arsenic(V) sulfide from the interaction of sodium metaarsenite with thioacetamide;
- Investigation of conditions for obtaining thin layer, nano- and microparticles of $As_2S_5$ in aquatic and organic medium;
- Investigation of the obtaining conditions for $Cu_3AsS_4$-containing thio compounds from the interaction of $As_2S_5$ and CuCl in aquatic and organic medium;
- Study of the synthesis conditions of thio compounds in an aqueous medium on the basis of arsenic(V) sulfide and copper(II) chloride;
- Investigation properties and obtaining conditions of silver thioarsenate based on arsenic(V) sulfide and silver nitrate in aquatic and organic medium;
- Study of obtaining conditions for $Tl_3AsS_4$-containing thioarsenate in aqueous and organic medium on the basis of arsenic(V) sulfide and thallium(I) nitrate;
- Study of the synthesis conditions of arsenic triple thio sulfides from Pb-As-S systems in aquatic environment and study of the properties of the obtained samples.

**The research methods.** Research related to the dissertation work was carried out by differential thermal, thermogravimetric, X-ray, elemental, calorimetric, ultraviolet spectroscopy and scanning electron microscopy analysis methods, which have great importance in modern times for physical and chemical analysis. Differential thermal and thermogravimetric analysis of the synthesized samples was carried out on NETZSCH STA 449F3. In addition, the Термоскан-2 device was used for differential thermal analysis. X-ray

phase analysis of the obtained thio compounds was performed on a D2 PHASER Bruker (CuKα, 2θ, 20-80 degrees) diffractometer. The HITACHI TM3000 scanning electron microscope was used to study the morphology and structure of the samples. Determination of the amount of arsenic that are filtered during the synthesis of compounds and transferred to the solution during the chemical analysis of samples was carried out with a photocolorimeter device КФК-2МП УХЛ4.2. The optical absorption curve of the obtained thin layers was plotted using U-5100 ultraviolet spectrophotometer. Both qualitative and quantitative composition of compounds has been determined in the JSM-6610LV SEM – Oxford Instrument element analyzer device.

**The main provisions of the defense.**

- Obtaining nano-particles and a thin layer of $As_2S_5$;
- The study of the obtaining conditions and physicochemically investigation for thioarsenates from $Cu^+$ and $Cu^{2+}$-As(V)-S systems in aquatic and organic medium;
- Investigation of the synthesis conditions for silver thioarsenate in aqueous and organic medium from the Ag-As-S system and obtaining its nano- and microparticles, physicochemically investigation;
- Investigation of the synthesis conditions for triple thio compounds in aqueous and organic medium from the Tl-As-S system and obtaining its nano- and microparticles, physicochemically investigation;
- Investigation of the synthesis conditions for triple compounds in aqueous medium from the Pb-As-S system and obtaining its nano- and microparticles, physicochemically investigation;
- Study of the synthesis conditions of thio compounds in the aquatic environment from the system $Pb(CH_3COO)_2–Na_3AsO_4–H_2S–H_2O$, obtaining their nano- and microparticles and physicochemical research.

**Scientific novelty of the research.** The following new scientific results were obtained in the dissertation work:

1. A thin layer of arsenic(V) sulfide was obtained by stencil printing method.
2. Obtaining methods for triple thio compounds from the interaction of arsenic(V) sulfide with water-soluble salts of copper(I) and copper(II) in water and organic media were developed, synthesis conditions were studied, and nano- and microparticles were obtained.
3. The characteristic of physicochemical interactions of silver nitrate with arsenic(V) sulfide in aqueous and organic media was determined, easy methods for obtaining relevant triple compounds were developed, and micro- and nanoparticles were obtained.
4. The characteristic of physical and chemical interactions of arsenic(V) sulfide and thallium nitrate in aquatic and organic media was determined, easy methods for obtaining relevant triple compounds were developed, nanoparticles were obtained and the main parameters characterizing the compounds were determined.
5. Obtaining methods for triple compounds from the interaction of arsenic(V) sulfide and lead(II) acetate in the aquatic environment were developed, synthesis conditions were studied and physical and chemical parameters were determined.

**Theoretical and practical significance of the research.** Compared with the ampoule synthesis method, the theoretical significance of the results of the dissertation is the development of a new, simpler method of synthesis of $As_2S_5$ in aqueous and organic medium and study of the obtaining conditions for triple thio compounds based on $As_2S_5$ and the $Cu^+$, $Cu^{+2}$, $Ag^+$, $Tl^+$, $Pb^{+2}$ salts. The results obtained regarding with the physicochemical properties of the obtained thio compounds have made a significant contribution to the chemistry and materials science of triple metal chalcogenides.

The practical significance of the dissertation is the advantages of the methods of obtaining thio compounds synthesized from the system Me-$As_2S_5$ (Me = $Cu^+$, $Cu^{+2}$, $Ag^+$, $Tl^+$, $Pb^{+2}$) in aqueous and

organic medium, their production technology is very simple and the processes are performed at low temperatures (293-363 K), allowing to obtain large quantities of substances due to require less energy and labor. The method can be used to obtain thin films and nanostructures. In the aquatic and organic environmetn obtained $As_2S_5$ and synthesized triple thio compounds based on $As_2S_5$ and $Cu^{+}$, $Cu^{+2}$, $Ag^{+}$, $Tl^{+}$, $Pb^{+2}$ salts are semiconductor materials and can be widely used and applied in nonlinear optical devices, optical fiber production, production of optical elements, manufacture of parts of optical devices, teletechnics and microelectronics, solar cells, electronic, opto-electronic, optoacoustic devices, chemical sensors.

**Approbation and implementation of research.** The main results of the dissertation were presented at the following scientific conferences, seminars and published in journals: Akademik M.Nağıyevin 110 illik yubileyinə həsr edilmiş "Nağıyev qirayətləri".Beynalxalq konfransı, (Baku/Azerbaijan 30-31 October, 2018), "Kimyanın yıldızlı 100. Yılı" 31st International Chemistry Conference (Yildiz Technical University, Istanbul/Turkey 10-13 September 2019), "Actual problems of chemical engineering" mövzusunda Azərbaycan Dövlət Neft və Sənaye Universitetinin 100 illiyinə həsr olunmuş beynəlxalq konfrans, (Baku/Azerbaijan 24-25 December, 2020), Научные тенденции в эпоху стремительного развития технологий, I научно-практической конференции, (Samara/Russia, 5 Mart, 2022), The latest problems of modern science and practice, I International Scientific and Practical Conference, (Boston/USA 11-14 January, 2022), Современные технологии: тенденции и перспективы развития, III Международная научно-практическая конференция, (Petrozavodsk/Russia 10 January, 2022), Journal of the Turkish Chemical Society Section A: Chemistry, (Istanbul/Turkey 2021, 8(2), p. 527-534), Journal of the Turkish Chemical Society Section B: Chemical Engineering, (Istanbul/Turkey 2020, p. 35-40), Nakhchivan Branch of ANAS Scientific Works, (Nakhchivan/Azerbaijan 2020, №2 p.52-55), Nakhchivan Branch of ANAS Scientific Works (Nakhchivan/Azerbaijan 2020, №4 p.51-55), Nakhchivan Branch of ANAS Scientific Works

(Nakhchivan/Azerbaijan 2018, №2 p.58-62), Nakhchivan State University Scientific Works, (Nakhchivan/Azerbaijan 2018, №92, p.184-188), Nakhchivan Branch of ANAS Scientific Works (Nakhchivan/Azerbaijan 2021, №4 p.32-39), Journal of Chemical Problems of the Institute of Catalysis and Inorganic Chemistry of ANAS (Baku/Azerbaijan 2021, №3 p.135-142), Science and world (Volgograd/Russia 2022, №1 p.17-20), $As_2S_5$ ağır metal duzlarının qarşılıqlı təsirindən tiobirləşmələrin alınması AMEA Naxçıvan Bölməsi Təbii Ehtiyatlar İnstitutunu elmi seminarında (Nakhchivan/Azerbaijan 28 January, 2022).

Including 6 conference materials 9 articles 4 of which are not co-authored, were published in international index journals regarding the dissertation. Published scientific articles and theses fully cover the content of the dissertation.

**Name of the organization where the dissertation work is carried out.** The dissertation work was carried out in the laboratory "Chemistry and technology of mineral raw materials" of the Institute of Natural Resources of Nakhchivan Branch of ANAS.

**Personal participation of the author.** The applicant is responsible for solving issues in the dissertation, experimental implementation of research, analysis based on samples obtained from laboratory experiments, analysis of the results obtained by the methods of physical and chemical analysis, writing theses and articles, dissertations.

**Volume and structure of the dissertation.** The dissertation consists of an introduction, 5 chapters, results and a list of references. The total volume of the dissertation consists of 166 pages, 75 figures, 30 tables and 160 titles of literature. The total volume of the work (excluding pictures, tables, graphs and bibliography) was 205767 characters.

## THE MAIN CONTENT OF THE WORK

**In the introductory** part of the dissertation, the relevance and usage rate of the topic is noted, detailed information is given about the

object, subject, purpose and objectives of the research, main provisions, research methods, scientific novelty, theoretical and practical significance, approbation, application, structure and scope.

**In the first chapter,** scientific researches on As-S, Ag-As-S, Tl-As-S, Cu (I)/Cu(II) -As-S, Pb-As-S system, as well as literature materials on the obtaining conditions of binary and triple compounds that are synthesized from these systems are noted. In addition, the literature on the physical and chemical properties of these compounds was analyzed and systematized.

**The second chapter** contains scientific information on chemically pure reagents used for laboratory experiments, chemical analysis methods, experimental devices, their working principle and their importance for physical and chemical analysis.

**The third chapter** presents the results of research on the obtaining conditions of $As_2S_5$ in aqueous and organic medium, obtaining its thin layer and the synthesis condition of thio compounds from the interaction of $As_2S_5$ with water-soluble salts of copper, thallium and silver in water. In addition, at the end of the chapter, the results of the solubility properties of the obtained compounds in acid and alkaline solutions of certain concentrations are noted.

**Investigation of the obtaining conditions of $As_2S_5$ compound in water and organic media.** $As_2S_5$ was obtained in aqueous medium on the basis of sodium metarsenite and hydrogen sulfide, sodium metarsenite and thioacetamide, and in organic medium on the basis of sodium arsenate and thioacetamide at a temperature of 273-283 K for 2, 4 and 10 hours, respectively. The individuality of the obtained $As_2S_5$ sample was confirmed by X-ray phase (XRD), differential thermal (DTA), thermogravimetric (TG), chemical, scanning electron microscope (SEM) and element analysis methods. Chemical analysis of the obtained samples was carried out. The content of arsenic in the samples was determined by the colourimetric method and the sulfur were determined by titration with barium nitrate. The results confirmed that the samples corresponded to the $As_2S_5$ formula.

It was found that arsenic(V) sulfide has a maximum yield in the range of pH=0-2. When the pH>2, its yield decreases, and then the yield of arsenic(III) sulfide increases. It was found that when the pH=5, only arsenic(III) sulfide compounds are present in the system. In aqueous solution, the arsenic (V) sulfide compound is formed in the temperature range of 273-283 K.

According to the analysis of TG (NETZSCH STA 449F3), it was determined that the sulfur and arsenic in the samples were completely evaporated due to oxidation and sublimation in the temperature range 573-873 K.

Differential thermal analysis of $As_2S_5$ compound (Термоскан-2) was performed. Three endothermic effects were observed in the differential thermal analysis of the $As_2S_5$ compound: the endothermic effect at 364 K corresponds to the peritectic conversion temperature of the $As_2S_5$ compound, and the endothermic effects at 386 K and 582 K correspond to the melting temperatures of the S and $As_2S_3$ compounds, respectively. The DTA results show that the As2S5 compound is resistant to temperatures up to 364 K.

Elemental analysis of the obtained compound (JSM-6610LV SEM – Oxford Instrument) was performed to determine the stoichiometric composition of $As_2S_5$. Based on the results, the weight (As-28.53%, S-71.47%) and atomic (As-28.53%, S-71.47%) ratios of arsenic and sulfur in the compounds were determined.

The composition of arsenic (V) sulfide sediment was studied by X-ray phase (2D PHASER “Bruker”) analysis method. Based on the XRD results, it was determined that the $As_2S_5$ compound obtained in aqueous and organic medium is amorphous.

The formation of micro- and nanoparticles of $As_2S_5$ in an organic medium was observed and images of micro- and nanoparticles obtained at different temperatures were taken (TM-3000 Hitachi electron microscope) (Figure 1). When studying the effect of temperature on the formation, growth and formation of nanoparticles of $As_2S_5$, it was found that the problem of its decomposition arises during the production of $As_2S_5$ at high temperatures. For this reason, it is recommended to obtain it at room temperature and below.

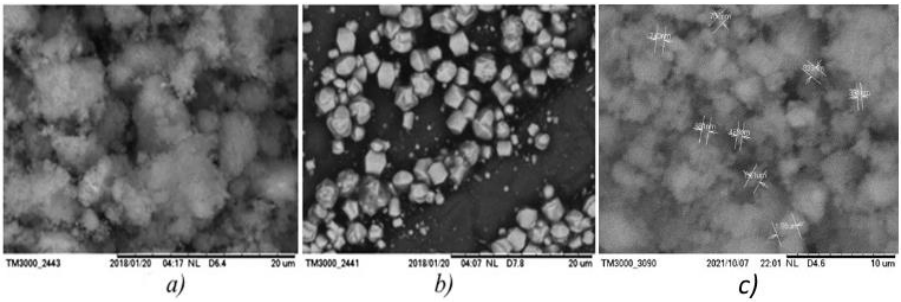



**Figure 1. SEM (a-283 K (water), b-353K (water), c-283 K (organic)) images of the $As_2S_5$ compound**

The thin film of $As_2S_5$ is made by stencil printing. The absorption spectrum of the thin layer of the compound was recorded on a U-5100 Hitachi ultraviolet spectrophotometer, the dependencies were constructed on the basis of the obtained values, and the value of the forbidden zone of the compound was determined to be $E_g^0$ = 2.55 eV (Figure 2).

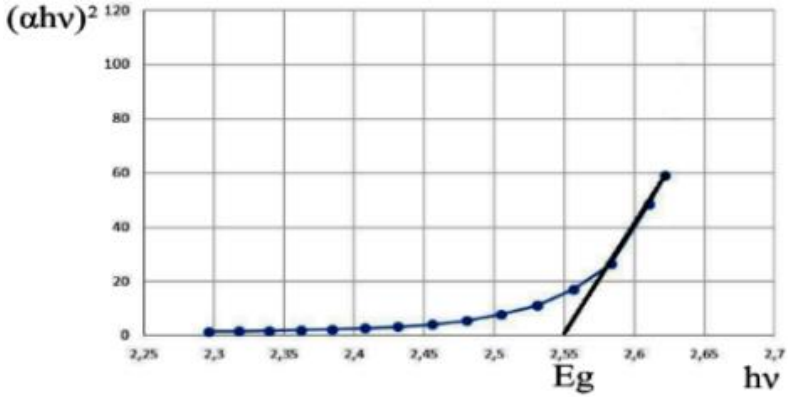



**Figure 2. Absorption spectrum of a thin film of $As_2S_5$**

**Investigation of chemical interactions in the $As_2S_5$–CuCl–$H_2O$ system.** The interaction condition of $As_2S_5$ and CuCl compounds in the aquatic environment has been studied. The equations of the reactions can be summarized as follows:

$$CuCl+2NH_4OH \rightarrow [Cu(NH_3)_2]Cl+2H_2O \quad (1)$$

$$4As_2S_5+15[Cu(NH_3)_2]Cl+27H_2O \rightarrow 5Cu_3AsS_4+3H_3AsO_4+15NH_4Cl+15NH_4OH \quad (2)$$

To study the obtaining conditions of the $Cu_3AsS_4$ compound, the dependence of the yield of the compound on the pH and concentration of the medium was studied. It was found that the maximum yield of $Cu_3AsS_4$ is observed in the range of pH=5-7 and in the mixture of primary components in the ratio of $As_2S_5$:CuCl=4:15.

Chemical analysis of the composition of the compound by iodametric, molybdate, barium nitrate titration and gravimetric methods was carried out and it was determined that the synthesized compound corresponds to the formula $Cu_3AsS_4$.

The DTA of the sample was obtained and it was found that the $Cu_3AsS_4$ compound undergoes polymorphic conversion at 633 K and melts at 963.1 K.

Under optimal conditions and in the aquatic environment, the individuality of the $Cu_3AsS_4$ compound was confirmed by the X-ray phase analysis method (2D PHASER "Bruker", $CuK_\alpha$, 2θ, 20-80 degrees.). The $Cu_3AsS_4$ compound was pulverized and its diffractogram was drawn after thermal processed in a vacuum ((~$10^{-2}$ Pa) for one hour at a temperature of 513 - 543 K (Figure 3).

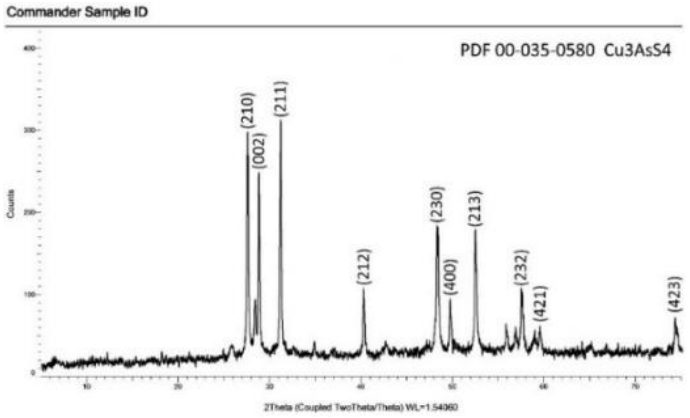



**Figure 3. Diffractogram of $Cu_3AsS_4$ compound**

The values of the intensity maxima in the diffractogram corresponded to the values of PDF 00-035-0580.

To determine the stoichiometric composition of the $Cu_3AsS_4$ compound, elemental analysis of the composition of the obtained compound was performed, energy-dispersion spectrum was drawn. According to the results obtained, the weight (Cu-48.53%, As-18.62%, S-32.85%) and atomic (Cu-49.53%, As-12.18%, S-38.29%) ratios of copper, arsenic and sulfur in the compound were determined, and the simple formula was determined to be $Cu_3AsS_4$.

**Determination of the nature of the interaction of $As_2S_5$ and $TlNO_3$ in aqueous media.** The interaction condition of $As_2S_5$ and $TlNO_3$ in the aquatic environment has been investigated. The equation of the reaction can be summarized as follows:

$$4As_2S_5+15TlNO_3+12H_2O\rightarrow 5Tl_3AsS_4+3H_3AsO_4+15HNO_3 \quad (3)$$

The process was carried out by mixing in a magnetic stirrer for 3 hours and keeping the pH of the medium at 4-5. After the reaction, the precipitate was filtered and washed with distilled water. In order to homogenize the obtained compound, ultra-pure water was re-added to the sediment and stored in a microwave oven at a temperature of 343-353 K for 96 hours. After homogenization, the precipitate was filtered again and washed with ultra-clean water and then with ethanol. The sediment was dried at 353 K under vacuum (~$10^{-1}$ Pa).

The thermodynamic values of enthalpy, entropy and Gibbs energy of the final equation of the synthesis reaction (3) of $Tl_3AsS_4$ were determined (Table 1).

**Table 1**

**Thermodynamic functions of final equation (3) of the reaction**

| $\Delta G^0_{298}$ | $\Delta H^0_{298}$ | $\Delta S^0_{298}$ |
|---|---|---|
| kC/mol | | C/(mol·K) |
| - 21.97 | - 206.25 | - 2867.95 |

Chemical analysis of the synthesized compound was carried out by bromatometric, molybdate, barium nitrate titration and gravimetric methods. Based on the results of the analysis, it was determined that the synthesized compound corresponds to the formula $Tl_3AsS_4$.

Thermogravimetric analysis was performed to determine the stoichiometric composition of the obtained $Tl_3AsS_4$ compound. As and S in the sample heated to a temperature of 1173 K were completely evaporated as a result of oxidation, and the remaining residue was $Tl_2O$. As a result of the analysis, taking into account the total losses and the amount of residue left in the end, it was determined that the formula of the compound corresponds to $Tl_3AsS_4$.

The DTA results of the obtained $Tl_3AsS4$ compound are given in figure 4.

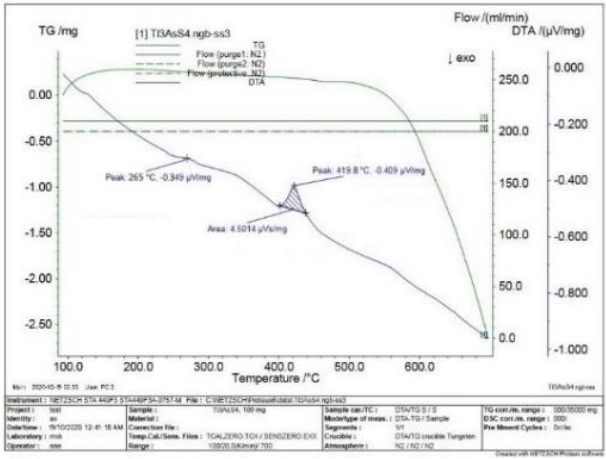



**Figure 4. DTA curve of $Tl_3AsS_4$ compound**

Thermal effects were observed in the DTA curve of $Tl_3AsS_4$ at temperatures of 538 K and 692.8 K. The weak thermal effect observed at a temperature of 538 K can be considered as the softening or polymorphic conversion temperature of the $Tl_3AsS_4$ compound. Thermal temperature at 692.8 K corresponds to its melting temperature.

The $Tl_3AsS_4$ compound was thermally processed in a vacuum (~$10^{-2}$ Pa) at 493 K for 2 hours and its composition was studied by X-ray phase (2D PHASER "Bruker") analysis method (Figure 5).

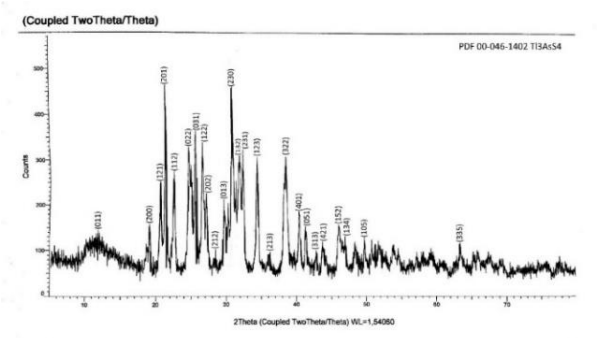



**Figure 5. Diffractogram of $Tl_3AsS_4$ compound**

The analysis showed that the composition of the sediment consists of $Tl_3AsS_4$ compound, and the value of the intensity maxima in the diffractogram corresponds well to the value of PDF 00-046-1402.

The morphology of the $Tl_3AsS_4$ compound obtained in an aqueous medium at 333-343 K was examined under a HITACHI TM3000 microscope and it was determined that the sediment obtained from the solution was composed of micro- and nanoparticle aggregates.

Elemental analysis of the obtained compound was performed to determine the stoichiometric composition of the $Tl_3AsS_4$ compound. According to the results, the mass and atomic ratios of thallium, arsenic and sulfur in the compounds were determined to be 75.25%, 9.29%, 15.46% and 37.79%, 12.72% and 49.48%, respectively. Based on the results of elemental analysis, it was determined that the simple formula of the compound obtained in the aqueous medium is well suited to $Tl_3AsS_4$.

The pycnometric method was used to determine the density of the sample. The density of the obtained $Tl_3AsS_4$ compound was 5.34 g/cm$^3$.

**Investigation of the obtaining conditions of the silver thioarsenate on the basis of arsenic (V) sulfide and silver nitrate in water medium.** The $Ag_3AsS_4$ compound was synthesized in the aquatic environment by the interaction of $As_2S_5$ and $AgNO_3$. The reaction mixture was stirred in a magnetic stirrer for 45-60 minutes, and the pH of the medium was kept at 4-5. The sediment was filtered and washed with distilled water and ethanol at the end of the process. It was placed in a microwave oven for homogenization at a temperature of 333-343K for 48 hours by re-adding water to the washed sediment. At the end of the process, the sediment was re-filtered, first washed with distilled water and then with ethanol. The cleaned sediment was vacuum dried at a temperature of 353 K.

The amount of silver, arsenic and sulfur in the compound obtained in the aquatic environment was determined by titration methods with faience, molybdate and barium nitrate, and the results are shown in the following table (Table 2).

**Table 2**

**Chemical analysis of silver thioarsenate compound**

| The amount of sample, q | The amount of elements, q | | | | | |
|---|---|---|---|---|---|---|
| | Ag | | As | | S | |
| | Theor. | Exper. | Theor. | Exper. | Theor. | Exper. |
| 0.526 | 0.323 | 0.320 | 0.075 | 0.070 | 0.128 | 0.121 |

Based on the results of chemical analysis, the composition of the synthesized sample was determined to correspond to the formula $Ag_3AsS_4$.

The thermodynamic values of enthalpy ($\Delta H^0_{298}$= -954.15 kC/mol), entropy ($\Delta S^0_{298}$= 3979.59 C/(mol·K)) and Gibbs energy ($\Delta G^0_{298}$= - 2139.89 kC/mol) of the final equation of the reaction (4) of the synthesis of silver thioarsenate compounds were determined.

$$4As_2S_5+15AgNO_3+12H_2O\rightarrow 5Ag_3AsS_4+3H_3AsO_4+15HNO_3 \qquad (4)$$

The stoichiometric composition of the compound was determined by thermogravimetric (NETZSCH STA 449F3) analysis (Figure 6).

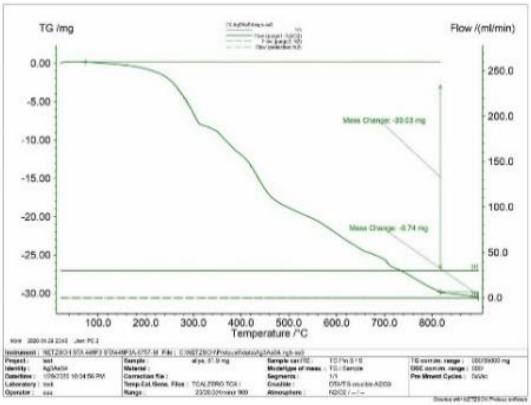



**Figure 6. Thermogram of silver thioarsenate compound**

As can be seen from the thermogram, the losses in the sample were due to the oxidation and sublimation of sulfur and arsenic. The weight of the residue after cooling was 51.13 mg, which corresponds to the proportion of silver. Based on these results, it was confirmed that the simple formula of the compound is $Ag_3AsS_4$.

Differential thermal analysis of the obtained $Ag_3AsS_4$ compound was performed. According to the DTA results, exothermic effects were observed at 572.5 K, 724.9 K and endothermic effects at 888.2 K. The melting point of $Ag_3AsS_4$ was determined to be 888.2 K.

X-ray phase analysis of the sample was performed on a 2D PHASER Bruker powder diffractometer. The results of the analysis showed that the main component (89.5%) consists of $Ag_3AsS_4$. The value of the intensity maxima in the diffractogram corresponds well to the value of PDF 01-089-1370.

The micromorphology of the silver thioarsenate compound obtained at a temperature of 343 K and in an aqueous medium was studied under a scanning electron microscope HITACHITM 3000 (Figure 7).

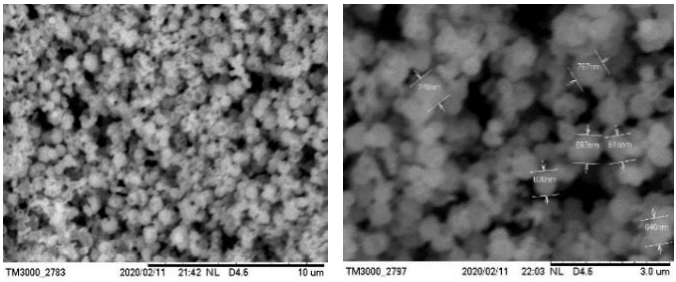



**Figure 7. SEM image of $Ag_3AsS_4$ compound**

The SEM analysis revealed that the silver thioarsenate compound consisted of spherical particles varying in size from 500 to 800 μm, and no other phase particles were observed between the particles.

Elemental analysis of the obtained compound was performed to determine the composition of the $Ag_3AsS_4$ compound (Figure 8).

| Element | Weight% | Atomic% |
|---|---|---|
| S | 23.83 | 48.93 |
| As | 15.69 | 15.16 |
| Cu | 60.48 | 35.91 |
| Cəm | 100.00 | |

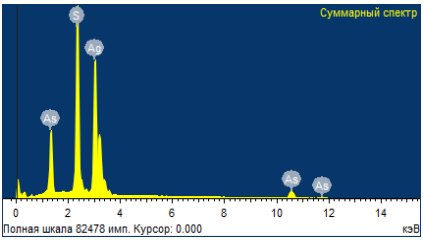



**Figure 8. Elemental analysis of $Ag_3AsS_4$**

Based on the results of the element analysis, the weight and atomic ratios of silver, arsenic and sulfur in the compounds were determined. Based on the mass and atomic values of silver, arsenic and sulfur, it was determined that the simple formula of the compound obtained in the aquatic environment corresponds to $Ag_3AsS_4$.

The pycnometric method was used to determine the density of the sample. The density of the obtained thio compound was 3.96 $g/cm^3$.

**Investigation of solubility conditions of thio compounds in acid and alkaline solutions.** The solubility of $Ag_3AsS_4$, $Cu_3AsS_4$,

$Tl_3AsS_4$ and $Pb_3As_4S_9$ compounds in 1 M concentration solid acid and alkaline solutions at different time intervals was studied. As a result of experiments, it became clear that the amount of soluble thio compounds in 1 M of hydrochloric and sulfuric acid remains constant regardless of the time. At the same time, the solubility of samples in 1 M concentrate of nitric acid, potassium hydroxide and ammonium hydroxide solution in experiments carried out for 1, 5, 8 and 24 hours increased.

At the same time, it should be noted that when thioarsenates react with solid potassium hydroxide and ammo hydroxide solutions, the substance decomposes, arsenic(V) sulfide obtained during decomposition reacts with potassium hydroxide to form $K_3AsO_2S_2$ and $K_2S$. Based on this, it can be written that the reactions proceed according to the following equations:

$$2Me_3AsS_4 + 8KOH \rightarrow 3Me_2S + 2K_3AsO_2S_2 + K_2S + 4H_2O \quad (5)$$
$$2Me_3AsS_4+8NH_4OH\rightarrow3Me_2S+2(NH_4)_3AsO_2S_2+(NH_4)_2S+4H_2O \quad (6)$$

When solid nitric acid was added to the synthesized thio compounds, decomposition of thio compounds was observed.

**In the fourth chapter** presents the results of the acquisition conditions of thio compounds from $As_2S_5$-$CuCl_2$-$H_2O$, $As_2S_5$-$Pb(CH_3COO)_2$-$H_2O$, $As_2S_5$-$Pb(CH_3COO)_2$-$H_2S$-$H_2O$ systems and their stoichiometric composition and individuality which are confirmed by physical and chemical analysis methods.

**Investigation of the interaction condition of $As_2S_5$ and $CuCl_2$ in the aquatic environment.** The acquisition conditions of copper(II) thioarsenate from the interaction of arsenic(V) sulfide and copper(II) chloride in the aquatic environment have been studied. The equation of the reaction can be summarized as follows:

$$8As_2S_5+15CuCl_2+24H_2O\rightarrow5Cu_3(AsS_4)_2+6H_3AsO_4+30HCl \quad (7)$$

The reaction mixture was stirred in a magnetic stirrer for 180 minutes, and the pH of the medium was kept at 4-5. At the end of the

process, the sediment was filtered, washed with distilled water and ethanol, and vacuum-dried at 353 K.

Thermodynamic parameters of the reaction were determined based on the final equation of the reaction (7) carried out in the direction of obtaining the compound $Cu_3(AsS_4)_2$ (Table 3).

**Table 3**

**Thermodynamic functions of final equation (7) of the reaction**

| $\Delta G^0_{298}$ | $\Delta H^0_{298}$ | $S^0_{298}$ |
|---|---|---|
| kC/mol | | C/(mol·K) |
| 303.54 | 56.2 | -830 |

Thermogravimetric and differential thermal analysis of the sample was performed on a thermo-analyzer NETZSCH STA 449F3. The melting temperature of the thio-compound corresponded to the melting point of $Cu_3AsS_4$, 964.3 K.

The composition of the sample was studied by the X-ray phase analysis (2D PHASER "Bruker") method. It was found that the sample consists of a compound of $Cu_3AsS_4$. The values of the intensity maxima on the diffractogram were in good compliance with the standards ($Cu_3AsS_4$ – PDF 01-075-0637, CuS – PDF 00-006-0464).

The stoichiometric composition of the thio compound obtained by the interaction of $As_2S_5$ and $CuCl_2$ in the aqueous medium was specified by elemental analysis. Based on the analysis of the weight (Cu-52.38%, As-13.94%, S-33.68%) and atomic (Cu-38.69%, As-10.23%, S-51.08%) ratio of copper, arsenic and sulfur in the compound, it was determined that its simple formula is well suited to $Cu_3AsS_4$.

The pycnometric method was used to determine the density of the sample. The density of the obtained thio compound was 3.59 $g/cm^3$.

**Investigation of the interaction conditions of $As_2S_5$ and $Pb(CH_3COO)_2$ compounds in the aquatic environment.** The interaction of titrated (mg/ml) $Pb(CH_3COO)_2$ solution with freshly precipitated $As_2S_5$ was used to investigation of obtaining condition of

the $Pb_3(AsS_4)_2$ compound. Studies have shown that the optimal conditions for the synthesis of the compound $Pb_3(AsS_4)_2$, which is intended to be obtained by reaction - pH 2-6, temperature 323-353 K and time 60-90 minutes. According to the reaction equation, 3/8 of the arsenic has been passed to the filtrate.

Thermodynamic parameters of the reaction were determined based on the final equation of the reaction which is the synthesis of the intended $Pb_3(AsS_4)_2$ compound (Table 4).

$$8As_2S_5+15Pb(CH_3COO)_2+24H_2O\rightarrow 5Pb_3(AsS_4)_2+6H_3AsO_4+30CH_3COOH \quad (8)$$

$$8As_2S_5+15Pb^{2+}+24H_2O\rightarrow 5Pb_3(AsS_4)_2+6AsO_4^{3-}+48H^+ \quad (9)$$

**Table 4**

**Thermodynamic functions of the final equation of the reaction**

| $\Delta G^0_{298}$ | $\Delta H^0_{298}$ | $S^0_{298}$ |
|---|---|---|
| kC/mol | | C/(mol·K) |
| 405.75 | - 263.85 | - 2247.1 |

To determine the stoichiometric composition of the obtained compound, TG analysis was performed on a dervatograph. According to the results of the analysis, the total loss corresponded to the amount of arsenic and sulfur in the sample, and the residue in the crucible to PbO.

Differential thermal analysis of the synthesized thiol compound was carried out on the NETZSCH STA 449F3 thermoanalysis device. According to DTA, the melting point of the compound was determined to be 726.2 K. Contrary to the TG analysis, the compound obtained by DTA results was determined to be $Pb_3As_4S_9$.

The composition of the sample obtained on the basis of the interaction of arsenic(V) sulfide with lead(II) acetate was investigated by the X-ray phase analysis (2D PHASER “Bruker”) method. It was found that the sample consisted of lead(II) thioarsenite and a small amount of lead sulfide. The results of the intensity maxima in the

diffractogram corresponded to the values PDF 00-024-1469 and PDF 00-005-0592.

The results of elemental analysis of arsenic(V) sulfide and lead(II) acetate-based compounds are given. Based on the results of the analysis, the weight (Pb-52.63%, As-24.24%, S-23.16%) and atomic (Pb-19.73%, As-25.42%, S-54.85%) ratios of lead, arsenic and sulfur in the compounds were determined. According to the values of mass and atomic ratios, the simple formula of the compound obtained in the aqueous medium was determined to be $Pb_3As_4S_9$.

The pycnometric method was used to determine the density of the sample. The density gravity of the obtained thio compound was 4.58 $g/cm^3$.

**Investigation of physical and chemical interaction conditions in the $Pb(CH_3COO)_2$–$Na_3AsO_4$–$H_2S$–$H_2O$ system.** $Pb(CH_3COO)_2$ and $Na_3AsO_4$ solutions have been used as primary components to study the physicochemical interactions of the $Pb(CH_3COO)_2$-$Na_3AsO_4$-$H_2S$-$H_2O$ system in the aquatic environment. A white precipitate of lead arsenate was formed from the interaction of 0.1 M solutions of the initial components in different molar ratios. It is planned to obtain lead thioarsenate compound by releasing hydrogen sulfide gas from this mixture. The precipitation process was completed in cold conditions at a temperature of 276 - 283 K and within 60 minutes. The heat treatment of the synthesized sediments was carried out in vacuum (~$10^{-2}$ Pa) in the temperature range of 373-523 K.

The obtained $Pb_2As_2S_5$, $PbAs_2S_4$, $Pb_9As_4S_{15}$ thio compounds were analyzed in the nitrogen stream by DTA analysis and it was determined that the compounds melted at 781 K, 727.2 K and 822.1 K, respectively.

Chemical analysis of $Pb_2As_2S_5$, $PbAs_2S_4$ and $Pb_9As_4S_{15}$ samples obtained from $Pb(CH_3COO)_2$–$Na_3AsO_4$–$H_2S$–$H_2O$ system was performed by the dichromatometric, molybdate, barium nitrate titration methods. Based on the results of chemical analysis of the obtained thio compounds, it was determined that the composition of

the compounds corresponds to the formulas $Pb_2As_2S_5$, $PbAs_2S_4$ and $Pb_9As_4S_{15}$.

X-ray phase analysis was performed to confirm the individuality of the synthesized thio compounds. The results of the intensity maxima of $Pb_2As_2S_5$, $PbAs_2S_4$ and $Pb_9As_4S_{15}$ in the diffractogram corresponded to the values of PDF 00-010-0453, PDF 01-076-1879, PDF 00-044-1412, respectively.

**The fifth chapter** describes the obtaining conditions of $Cu_3AsS_4$, $Tl_3AsS_4$, $Ag_3AsS_4$ compounds synthesized from the $Me^+$-$As_2S_5$ ($Me^+ = Cu^+$, $Tl^+$, $Ag^+$) system in an organic medium and the results of their physicochemical analysis.

**Study of physicochemical interactions condition between $As_2S_5$ and CuCl salts in ethylene glycol medium.** Interaction conditions of $As_2S_5$ and CuCl compounds in ethylene glycol medium have been studied. The reaction mixture was stirred at pH 8-9 for 3 hours using a magnetic stirrer, then the formed dark brown sediment is filtered, first washed with distilled water and then with ethanol.

Chemical and TG analysis of the compound (NETZSCH STA 449F3) was performed (Figure 9).

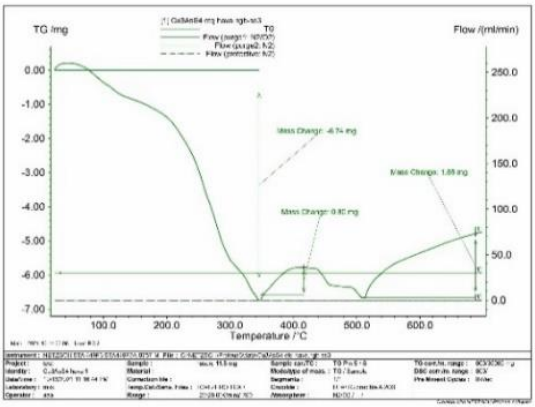



**Figure 9. Thermogravimetric analysis of $Cu_3AsS_4$**

The total mass loss at 793 K due to oxidation of sulfur and arsenic in the air stream was 6.86 mg. Based on the total loss and

residual content of the sample, it was determined that the composition of the compound corresponds to the formula of the $Cu_3AsS_4$ compound.

Differential thermal analysis of the synthesized sample (NETZSCH STA 449F3) was performed (Figure 10). The results of the analysis showed that the copper(I) thioarsenate compound undergoes a polymorphic transformation at 578 K. The subsequent increase in temperature corresponds to the formation of $Cu_3AsS_4$. The melting temperature of the sample was 965.5 K.

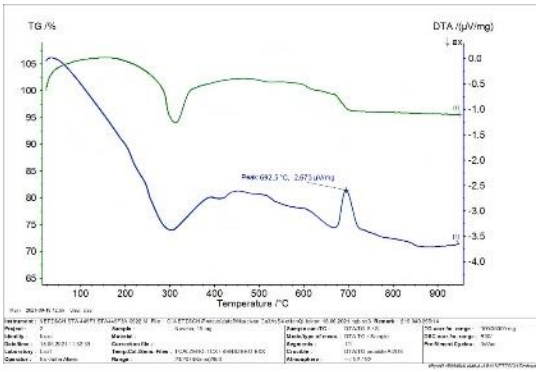



**Figure 10. DTA curve of $Cu_3AsS_4$ compound**

SEM analysis of $Cu_3AsS_4$ compound obtained at 353 K in ethylene glycol medium was carried out and it was determined that the obtained precipitate formed a cotton-like shape and was composed of nanoparticle aggregates (Figure 11).

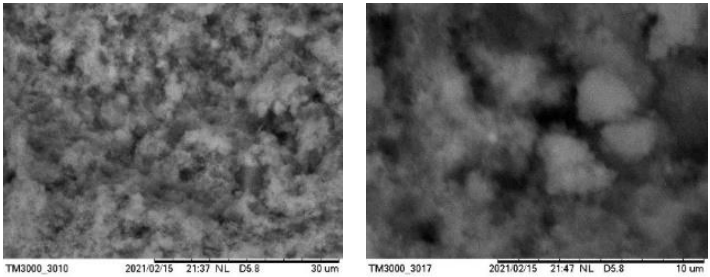



**Figure 11. SEM image of $Cu_3AsS_4$ compound**

The weight (Cu-49.91%, As-16.18%, S-33.91%) and atomic (Cu-38.15%, As-10.49%, S-51.36%) ratios of copper, arsenic, and sulfur in the sample were determined by elemental analysis. According to the results of the analysis, it was determined that the simple formula of the compound obtained in ethylene glycol medium is $Cu_3AsS_4$.

**Study of physical and chemical interaction conditions in solvothermal conditions in the system $As_2S_5$ – $TlNO_3$ – $C_2H_6O_2$.** The characteristics of physicochemical interactions in ethylene glycol medium based on $As_2S_5$ and $TlNO_3$ were studied. Using 0.1 M ammonium hydroxide, the pH of the medium was maintained at 7-8 and the reaction mixture was mixed with a magnetic stirrer at 358 K for 3 hours. At the end of the process, the sediment was filtered and washed first with distilled water and then with ethyl alcohol.

It was confirmed by chemical analysis methods (bromatometric, molybdate, barium nitrate titration) that the composition of the sample synthesized corresponds to the formula $Tl_3AsS_4$.

Differential thermal analysis of $Tl_3AsS_4$ compound obtained in ethylene glycol medium (NETZSCH STA 449F3) was performed (Figure 12).

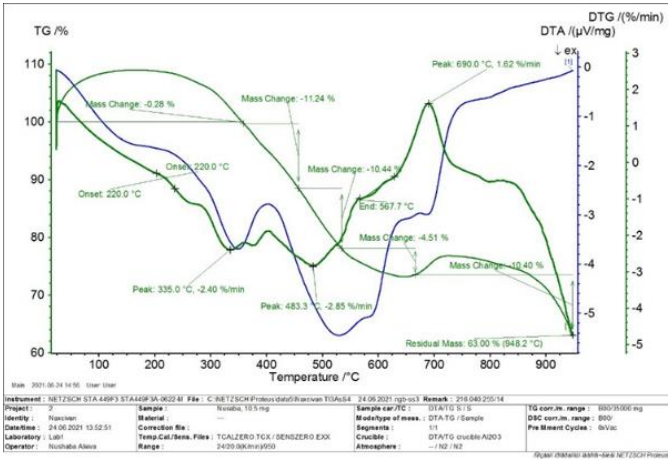



**Figure 12. DTA curve of $Tl_3AsS_4$ compound**

It is clear from the differential thermal analysis curve that the melting of the $Tl_3AsS_4$ compound occurred at a relatively low temperature (~683 K).

The $Tl_3AsS_4$ sample was heat-treated at 523-543 K (at ~$10^{-2}$ Pa vacuum) for 2 h and its composition was investigated by X-ray phase analysis (2D PHASER "Bruker") (Figure 13).

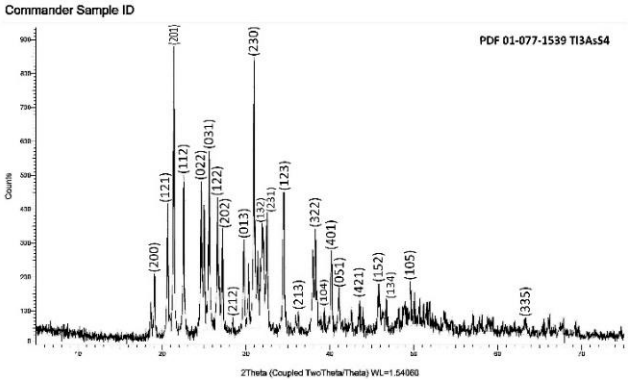



**Figure 13. Diffractogram of $Tl_3AsS_4$ compound**

The results of the analysis corresponded to the value of PDF 01-077-1539 and confirmed its individuality.

The micromorphology of the $Tl_3AsS_4$ compound obtained in an ethylene glycol medium at 353 K was studied under a HITACHI TM3000 microscope (Figure 14).

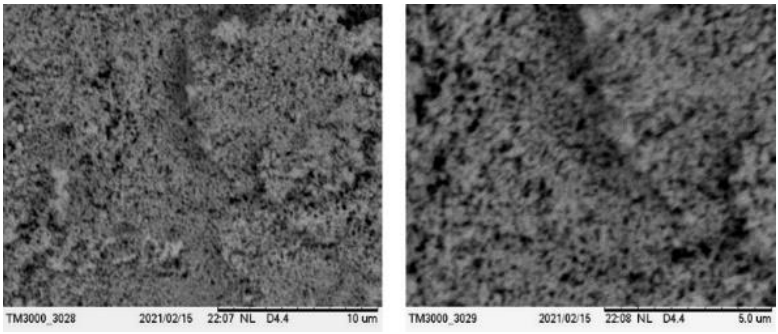



**Figure 14. SEM images of $Tl_3AsS_4$ compound**

According to the SEM analysis, it is clear that the sediment was taken from the solution is composed of nanoparticle aggregates.

**Investigation of the synthesis condition of $Ag_3AsS_4$ compounds based on $As_2S_5$ and $AgNO_3$ in ethylene glycol medium.** $Ag_3AsS_4$ was obtained under hydrothermal conditions in an organic medium based on the interaction of $AgNO_3$ and $As_2S_5$. In this case, the concentration of hydrogen ions in the solution was in the range of pH = 5-7.

The composition of the synthesized sample was examined by chemical analysis (fayans, molybdate, barium nitrate titration) and it was determined that it corresponds to the formula $Ag_3AsS_4$.

Differential thermal analysis of silver thioarsenate compounds synthesized in ethylene glycol medium was performed. According to the results of the analysis, exothermic effects were observed at 577.7 K and endothermic effects at 890.4 K. It was determined that the melting point of the silver thioarsenate compound corresponds to 890.4 K.

The composition of the silver thioarsenate compound was determined by X-ray phase analysis and the results of the analysis are shown in figure 15.

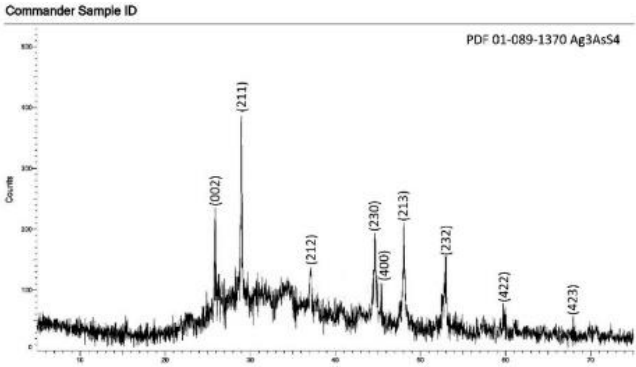



**Figure 15. X-ray phase analysis of $Ag_3AsS_4$ compound**

As a result of the analysis, it was found that the main component of the compound obtained from a mixture of primary

components in a ratio of 4:15 in ethylene glycol medium is a silver thioarsenate compound. The results of the X-ray phase analysis of the silver thioarsenate compound were in accordance with PDF 01-089-1370 and confirmed their individuality.

The micromorphology of the silver thioarsenate compound obtained at a temperature of 343 K was studied under a HITACHI TM3000 scanning electron microscope (Figure 16).

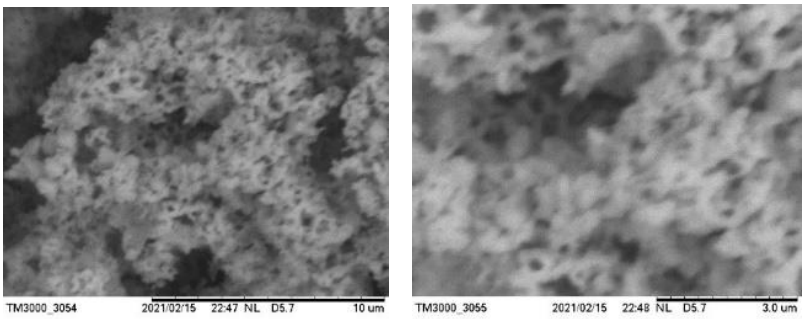



**Figure 16. SEM image of $Ag_3AsS_4$ at 10 and 3 μm area**

The SEM analysis showed that the particles in the silver thioarsenate compound obtained in the ethylene glycol medium had a dense interlocking structure, and no other phase particles were observed in the 10 and 3 μm areas.

Elemental analysis of the obtained sample was carried out and it was concluded that the compound obtained in ethylene glycol medium according to the values of weight and atomic ratios of silver, arsenic, sulfur corresponds well to the formula $Ag_3AsS_4$ (Figure 17).

| Element | Weight% | Atomic% |
|---|---|---|
| S | 24.03 | 52.46 |
| As | 13.82 | 11.83 |
| Ag | 62.15 | 35.71 |
| Cəm | 100.00 | |

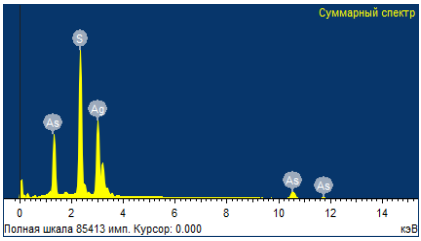



**Figure 17. Elemental analysis of $Ag_3AsS_4$ compound**

## RESULTS

1. Interactions between $NaAsO_2$ and $H_2S$, $NaAsO_2$ and $C_2H_5NS$, $Na_3AsO_4$ and $C_2H_5NS$ in water and organic media were investigated and $As_2S_5$ compounds were obtained at 2, 4 and 10 hours, respectively. The individuality of the synthesized compounds was confirmed by RFA, DTA, TG, chemical, SEM and element analysis methods. According to the results of the analysis, it was determined that the $As_2S_5$ compound obtained in the temperature range of 273-283 K is amorphous state. As a result of the DTA of the compound, $As_2S_5$ is as stable as a temperature of 364 K. The results of the TG analysis method revealed that this compound decomposes at a temperature of T>363 K. The weight and atomic ratios of arsenic and sulfur were determined by the physicochemical analysis methods in the composition of the compound and determined the stoichiometric composition of the compound. According to the results of SEM analysis, $As_2S_5$ particles obtained at temperatures of 283 K and 353 K were cotton-shaped and partially cubic-shaped, respectively. In addition, nanoparticles and a thin film of $As_2S_5$ were obtained, the absorption spectrum was drawn, and the width of the band gap was determined to be $Eg^0 = 2.55$ eV.

2. The nature of the physicochemical interaction between CuCl and $As_2S_5$ in aqueous and organic media was studied and the $Cu_3AsS_4$ compound was synthesized within 3 hours. Individuality and stoichiometric composition of the compound were confirmed by physicochemical analysis methods. The reaction equation of obtaining of $Cu_3AsS_4$ compound was compiled, the thermodynamic parameters were calculated. The melting of $Cu_3AsS_4$ synthesized in aqueous medium at a temperature of 963.1 K and the melting of the compound obtained in an organic medium at a temperature of 965.5 K was determined as a result of DTA. The micromorphology of the compound obtained in water and organic medium at a temperature of 323-363 K was studied and it was determined that the compound is composed of micro- and nanoparticles. The maximum yield of the compound obtained in the aqueous medium was in the range of 5-7

pH, and the maximum yield of the compound synthesized in the organic medium was in the range of 6-8 pH. Weight and atomic ratios of copper, arsenic and sulfur in the obtained $Cu_3AsS_4$ compound were determined.

3. The interaction condition of $CuCl_2$ and $As_2S_5$ in the aquatic environment was studied and a thiocompound was synthesized in 180 minutes. The individuality and stoichiometric composition of the obtained thiocompound were studied by the physicochemical analysis methods. In contrast to the TG analysis, the composition of the thio compound based on the results of other analytical methods was confirmed to be composed of $Cu_3AsS_4$.

4. The nature of the physicochemical interaction on the $TlNO_3$–$As_2S_5$–$H_2O/C_2H_6O_2$ system was investigated and the $Tl_3AsS_4$ compound was synthesized within 3 hours. The individuality and stoichiometric composition of $Tl_3AsS_4$ compound based on $TlNO_3$ and $As_2S_5$ in the temperature range 348-358 K at the ratio of 4:15 mol were confirmed by RFA, DTA, TQ, chemical, SEM and element analysis methods. The weight and atomic ratios of Tl, As and S in the compound have been determined. The micromorphology of the $Tl_3AsS_4$ compound was studied and it was determined that the compound consists of nanoparticles. thermodynamic values were calculated based on the reaction equation for the acquisition of $Tl_3AsS_4$. Obtaining $Tl_3AsS_4$ in aqueous medium was found to be in the range of 4-5, and in organic medium in the range of 7-8. According to the DTA, the melting point of the thallium(I) thioarsenate compound obtained in the aqueous medium was 692.8 K, and that of the compound synthesized in the organic medium was 688 K.

5. Physico-chemical effects of the Ag–As–S system in aquatic and organic environments have been studied and $Ag_3AsS_4$, a triple thiol compound of arsenic, has been synthesized under defined optimal conditions. The individuality of the compound obtained was confirmed based on the results of RFA and TQ analysis. $Ag_3AsS_4$ obtained in aqueous and organic medium was found to melt at 888.2 K and 890.4 K temperatures, respectively, according to DTA. The SEM analysis revealed that the $Ag_3AsS_4$ compound obtained in the

aqueous medium consisted of particles with particle sizes in the range of 500-900 nm, while the $Ag_3AsS_4$ compound obtained in the organic medium consisted of particles with a dense mesh structure connected to each other. The reaction equation for the acquisition of silver thioarsenate was compiled, thermodynamic parameters were calculated and the density was determined by the pycnometric method. According to the results of the chemical analysis, the formula of the compound was specified according to the weight of the obtained sample and the amount of filtered arsenic. The elemental composition of the $Ag_3AsS_4$ compound was determined according to the results of the elemental analysis of the compound.

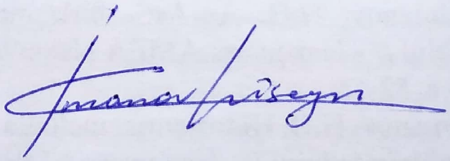
6. The interaction condition of $Pb(CH_3COO)_2$ and $As_2S_5$ in aqueous medium was studied and $Pb_3As_4S_9$ was synthesized. The optimal conditions for the synthesis of thio compounds were determined to be pH 2-6, temperature 323-353 K and time 60-90 minutes. The individuality of the $Pb_3As_4S_9$ compound was determined by physico-chemical analysis methods. The melting point of $Pb_3As_4S_9$ was determined to be 726.2 K based on the DTA results of the synthesized thio compound. The micromorphology of the obtained compound was studied and it was confirmed that there is adhesion between the particles of the compound and that it consists of nanoparticles. The weight and atomic ratios of the compound obtained in the aqueous medium were determined based on the element analysis, and it was determined that the simple formula of the compound was corresponded to $Pb_3As_4S_9$.

7. The interaction condition of $Pb(CH_3COO)_2$ and $Na_3AsO_4$ in aqueous medium at certain mole ratios, at a temperature of 276 - 283 K and for 60 minutes was studied and $Pb_2As_2S_5$, $PbAs_2S_4$, $Pb_9As_4S_{15}$ compounds were synthesized. The stoichiometric composition of each obtained thio compound was determined by physical and chemical analysis methods, and its individuality was confirmed. According to the DTA results, the melting temperatures of $Pb_2As_2S_5$, $PbAs_2S_4$, $Pb_9As_4S_{15}$ compounds were determined to be 781 K, 727.2 K and 822.1 K, respectively.

## THE MAIN RESULTS OF THE DISSERTATION HAVE BEEN PUBLISHED IN THE FOLLOWING SCIENTIFIC WORKS


1. İmanov, H.Ə. Su mühitində arsen(V) sulfidin alınması şəraitinin araşdırılması // - Naxçıvan: AMEA Naxçıvan Bölməsi Xəbərlər, - 2018, № 2, - s. 58-62.
2. Hüseynov, Q.M., İmanov, H.Ə., Məmmədova, S.H. Natrium-metarsenit və tioasetamid əsasında arsen(V) sulfidin alınması şəraitinin tədqiqi // - Naxçıvan: NDU Elmi Əsərlər, - 2018, № 92, - s. 184-188.
3. İmanov, H.Ə., Hüseynov, Q.M. Hidrokimyəvi metodla arsen(V) sulfidin alınması şəraitinin tədqiqi // Nağıyev Qiraətləri Elmi Konfrans, - Bakı: AMEA Kataliz və Qeyri-Üzvi Kimya İnstitutu, - 30-31 oktyabr, - 2018, - s. 276.
4. İmanov, H.Ə., Hüseynov, Q.M. Hidrokimyasal yöntemle $Pb(CH_3COO)_2$-$Na_3AsO_4$-$H_2S$-$H_2O$ isteminden üçlü sülfürlerin edinim koşullarının araştırılması // 31. Ulusal Kimya Kongresi, - İstanbul: Türkiyə Kimya Dərnəyi, - 10-13 sentyabr, - 2019, - s. 284.
5. İmanov, H.Ə., Hüseynov, Q.M. Investigation of obtaining condition of $Tl_3AsS_4$ compound in the aquatic environment // Kimya mühəndisliyinin aktual problemləri, Azərbaycan Dövlət Neft və Sənaye Universitetinin 100 illiyinə həsr olunmuş beynəlxalq konfrans, - Bakı: Azərbaycan Dövlət Neft və Sənaye Universiteti, - 24-25 dekabr, - 2020, - s. 247-250.
6. Imanov, H.A., Huseynov, G.M. Investigation of acquisition of triple sulfurs from $Pb(CH_3COO)_2$-$Na_3AsO_4$-$H_2S$-$H_2O$ system by hydrochemical method // Journal of the Turkish Chemical Society Section B: Chemical Engineering, - 2020, 3, № 2, - p. 35-40.
7. İmanov, H.Ə. $Ag_3AsS_4$ birləşməsinin alınması və xassələrinin tədqiqi // - Naxçıvan: AMEA Naxçıvan Bölməsi Xəbərlər, - 2020, № 2, - s. 52-55.
8. İmanov, H.Ə. Hidrotermal metodla $Cu_3AsS_4$ birləşməsinin alınması şəraitinin tədqiqi // - Naxçıvan: AMEA Naxçıvan Bölməsi Xəbərlər, - 2020, № 4, - s. 51-55.

9. Imanov, H.A., Huseynov, G.M. Investigation of the acquisition conditions of $Tl_3AsS_4$ compound in the aquatic environment // Journal of the Turkish Chemical Society Section A: Chemistry, - 2021, 8, № 2, - p. 527-534.
10. İmanov, H.Ə., Hüseynov, Q.M. Etilenqlikol mühitində $Cu_3AsS_4$ birləşməsinin alınması və xassələrinin tədqiqi // - Naxçıvan: AMEA Naxçıvan Bölməsi Xəbərlər, - 2021, № 4, - s. 32-39.
11. Imanov, H.A. and Huseynov, G.M., Study of the conditions for the acquisition of $Tl_3AsS_4$ in ethylene glycol // - Bakı, Kimya Problemləri, - 2021, 3, № 19, - p. 135-142.
12. Imanov, H.A. Investigation of conditions for obtaining $Ag_3AsS_4$ from system $As_2S_5$-$AgNO_3$-$C_2H_6O_2$ // - Volgograd: Science and world, - 2022, 101, № 1, - p 17-20.
13. Imanov, H.A. Investigation of the conditions for the acquisition of triple thio-compounds from the interaction of $As_2S_5$ and $CuCl_2$ // Научные тенденции в эпоху стремительного развития технологий, I научно-практической конференции, - Самара: - 5 марта, - 2022, - с 20-22.
14. Imanov, H.A. Investigation of the conditions for obtaining a triple thio-compound from the $Pb(CH_3COO)_2$-$As_2S_5$-$H_2O$ system // The latest problems of modern science and practice, I International Scientific and Practical Conference, - Boston: - 11-14 January, - 2022, - p. 84-87.
15. Imanov, H.A. Investigation of the conditions for the acquisition of $Ag_3AsS_4$ in ethylene glycol medium // Современные технологии: тенденции и перспективы развития, III Международная научно-практическая конференция, - Петрозаводск: МЦНП Новая наука, - 10 января, - 2022, - с. 232-236.



The defense will be held on "7" June 2022 at 11$^{00}$ at the meeting of the Dissertation council FD 1.24 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Nakhchivan Branch of the National Academy of Sciences of Azerbaijan.

Address: AZ7000, Nakhchivan city, Heydar Aliyev avenue, 76.

Dissertation is accessible at the Scientific Library of the Nakhchivan Branch of ANAS.

Electronic versions of dissertation and its abstract are available on the official website of the Nakhchivan Branch of ANAS (http://ameanb.az).

Abstract was sent to the required addresses on "4" May 2022

Signed for print: 26.04.2022
Paper format: $60\times90^{1/16}$
Volume: 42547
Number of hard copies: 20