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Д.В. Сокольский атындағы «Жанармай,  
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# Х А Б А Р Л А Р Ы

## ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
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## NEWS

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## SYNTHESIS OF THALLIUM SULPHATE IN A SULFURIC ACID SOLUTION AT POLARIZATION BY CURRENT

**Abstract.** Nowadays, the one of the actual problems is the recycling of metal waste and its provision to consumers. However, it is known that there are a number of difficulties with the recycling of thallium waste. By using new recycling methods, it's possible to increase thallium resources and lower prices for thallium-derived products. Therefore, some work was carried out to extract its compounds from thallium waste by studying the electrochemical properties of thallium in our research work. The experimental results showed that thallium electrodes dissolve in an aqueous solution of sulfuric acid under alternating current polarization with the formation of thallium (I) sulfate.

In this work, the kinetics of electrochemical oxidation-reduction of thallium in aqueous solutions of sulfuric acid is studied by potentiometry. To characterize the mechanism of thallium electrooxidation, the following parameters were calculated: the order of the reaction and the effective activation energy of the process. The impact of electrolysis modes: current density, concentration and temperature of the electrolyte on the dimensional characteristics of the obtained thallium (I) sulfate was investigated.

**Key words:** potentiodynamic polarization curves, electrochemical behavior, thallium, sulfuric acid, current density.

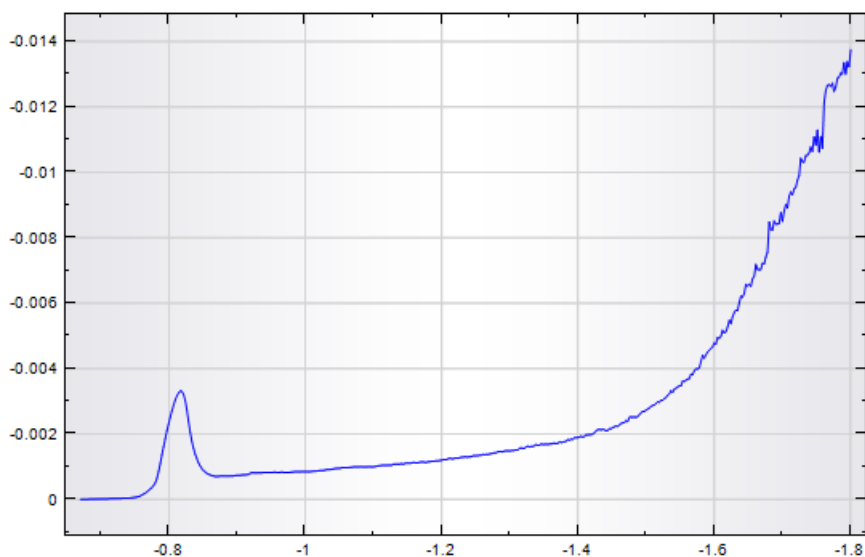
Most metals discover new uncharacteristic for them properties of electrodisolution during alternating current polarization. Particularly, the rate of dissolution of a metal can significantly increase or, on the contrary, noticeably decrease in comparison with its dissolution during anodic direct current polarization. Differences are also observed in the oxidation states of the released ions in the modes of alternating and direct current polarization.

Although thallium is chemically similar to heavy metals (lead, silver, gold), some of its properties resemble alkali metals. Since the ionic radii of thallium and alkali metal monovalent ions are very close to each other, thallium monovalent compounds tend to form isomorphic crystals mixed with alkali metal compounds. Obviously, the study of the property of an element, in the property of which such a variety is observed, is always of interest. Thallium in the elemental state is widely used in the production of many radioactive isotopes ( $\text{Hg}^{203}$ ), as well as its own isotopes ( $\text{Tl}^{204}$ ,  $\text{Tl}^{203}$ ), in many studies of analytical chemistry. A standard solution of monovalent sulfate thallium permanganate can be titrated in the presence of dichromate; it is most often used in analyzes for the content of steel [1-2]. And the  $\text{TlNO}_3$  solution is used when titrating the excess of the reagent after the precipitation of potassium, ammonium and organic bases with sodium tetraphenylboron salt.

Thallium dissolves in acids at a slow rate. In this regard, works devoted to the study of the mechanism of electrochemical dissolution and the regularities of dissolution of a thallium electrode in sulfuric acid solution were carried out by the potentiometric method by taking polarization curves and industrial alternating current polarization with a frequency of 50 Hz [3-13].

Potentiometric curves were recorded on an Autolab-30 potentiostat. The measurements were carried out in a three-electrode cell relative to a silver chloride electrode ( $E_0 = +0.203$  V). Platinum served as an auxiliary electrode. A standard solution of sulfuric acid was prepared according to the procedure described in [14]. Voltammograms, in mainly obtained in the temperature range of 20-25 ° C. Analysis of polarization curves gives a complete characteristic of electrode processes [15-16]. The method of recording polarization curves is based on establishing a relationship between the polarization current and individual values of the electrode potential. To clarify the mechanism of processes occurring during alternating current polarization, the electrodes were subjected to cyclic polarization (solution concentration 100 g / L, sweep rate 50 mV / s), i.e., the curves were recorded first in the cathode-anode direction, then anode-cathode. Thus, to a certain extent, a change in direction current occurring during the passage of alternating current.

Cyclic polarization curves to a certain extent reflect the situation when the metal is alternately either the anode or the cathode.



100 g/l; H<sub>2</sub>SO<sub>4</sub>; v = 50 mV/c; t=25°C

Figure 1 - Cathodic potentiodynamic polarization curves on a thallium electrode



100 g/l; H<sub>2</sub>SO<sub>4</sub>; v = 50 mV/c; t=25°C

Figure 2 - Anodic potentiodynamic polarization curves on a thallium electrode

Figure 1 shows the cathode potentiodynamic polarization curve. When the potential is shifted to the anode region within the "minus" 0.8-0.85 V, a maximum current is observed, corresponding to the thallium reduction current. Constant maintenance of the electrolyte temperature during the experiment ensures a uniform passage of the ionization process, i.e., a slow course of electrochemical processes. In this zone, only the reduction current of  $Tl^+$  ions is recorded. Further, gas hydrogen is released on the electrode surface.

As can be seen in figure 2, at a potential sweep rate of 50 mV / s, an anode current is recorded at a potential "minus" of 0.2 V. This corresponds to the thallium oxidation process. At the "plus" potential, the oxygen reduction current is observed. The results showed that at very low potentials, thallium oxidation is accompanied by the reduction of hydrogen and oxygen ions.



100 g/l;  $H_2SO_4$ ;  $v = 50$  mV/c;  $t=25^\circ C$

Figure 3 – Cathode-anode potentiodynamic cyclic polarization curves on a thallium electrode



100 g/l;  $H_2SO_4$ ;  $v = 50$  mV/c;  $t=25^\circ C$

Figure 4 – Anodic-cathodic potentiodynamic cyclic polarization curves on a thallium electrode



Figures 3-4 show cyclic cathode-anodic and anode-cathodic potentiodynamic polarization curves recorded on a thallium electrode with a sweep rate of 50 mV / s in a sulfuric acid solution. As can be seen from Figure 3, when the potential value shifted in the direction of the cathode within the "minus" 0.85-0.9 V range, waves were recorded indicating the progress of the thallium reduction reaction and the corresponding reaction of the hydrogen ion reduction from "minus" 0.9 V.

When the value of the potential is shifted in the direction of the anode-cathode at a "minus" of 0.75 V, a wave of current oxidation is observed, from the result it is seen that the thallium oxidation reaction is difficult:



Then the metal is completely passivated. It is known from the literature [17,18] that the electrochemical anodic dissolution of thallium in the presence of OH ions in aqueous solutions is complicated by the formation of dark brown thallium oxide formed on the anode surface. In this case, the rate of reduction of hydrogen ions on the thallium surface increases:



In order to comprehensively study the electrochemical properties of thallium in a sulfuric acid solution, industrial alternating current electrolysis was carried out.

The experiments were carried out in an electrolyzer with a capacity of 300-400 ml. Sulfuric acid solutions of various concentrations were used as the electrolyte. The electrodes are made of 99.99% pure thallium plates. The magnitude of the alternating current in each phase was controlled by rheostats. The number of thallium ions transferred into the solution during polarization with alternating current was determined from the decrease in the weight of the electrodes, as well as from the concentration of metal ions in the solution.

When a thallium electrode is polarized with an alternating current in a sulfuric acid medium, thallium is oxidized in the anodic half-period and, according to reaction (1), goes into solution in the form of a monovalent ion:



Thallium, which has a negative potential according to reaction 2, reacts with hydrogen ions, forming additional thallium ions and releasing gas hydrogen:



Thallium ions formed in solution interact with sulfate ions, resulting in the formation of the compound  $\text{Tl}_2\text{SO}_4$ :



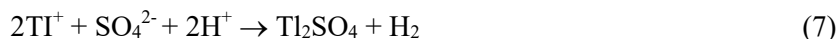
The influence of the current density on the current efficiency of the formation of thallium (I) ions was studied in the range of 1000-8000 A / m<sup>2</sup>. The maximum output value of the formation of thallium (I) ion by current is 56.7% at 8000 A / m<sup>2</sup> (figure 5). With an increase in the current density, the current efficiency of thallium ions (I) also increases. The dissolution current efficiency of thallium ions (I) was calculated for the anodic half-period of the alternating current in each phase. This is due to the fact that when thallium electrodes are in the anodic half-cycle of an alternating current, they dissolve, forming a monovalent ion, and in the cathodic half-cycle, a discharge of hydrogen ions occurs on the surface of the electrodes.

At high current densities in the cathodic half-period, the rate of reduction of hydrogen ions on the thallium surface increases:



The influence of the electrolyte concentration on the current efficiency of thallium (I) ions formed during the polarization of thallium electrodes with alternating current in a sulfuric acid solution was investigated in the range of 2.0-14.0 mol / l (figure 6).

With alternating current polarization, the initial concentration of sulfuric acid has a significant effect on the formation of thallium (I) sulfate. The maximum current efficiency of the formation of  $Tl^+$  ions is observed at 14.0 mol / L and is ~ 51%. Due to the occurrence of the supersaturation process at high concentrations of the solution, during the anodic half-period of the alternating current, an oxide layer does not form on the electrode surface, as a result of which the current efficiency of the formation of thallium sulfate increases:



However, it is known that the processes of electrochemical dissolution in more saturated solutions are carried out with some difficulties. And this increases the proportion of the process taking place in the cathodic half-period.

The acidity of the solution has a great influence on the electrochemical dissolution of thallium in a 14.0 mol / l sulfuric acid solution. The presence of a sufficient amount of acid concentration does not allow the formation of an oxide layer on the metal surface, due to which the metal with a negative potential, freed from the protective layer, intensively dissolves with the formation of its own ion.

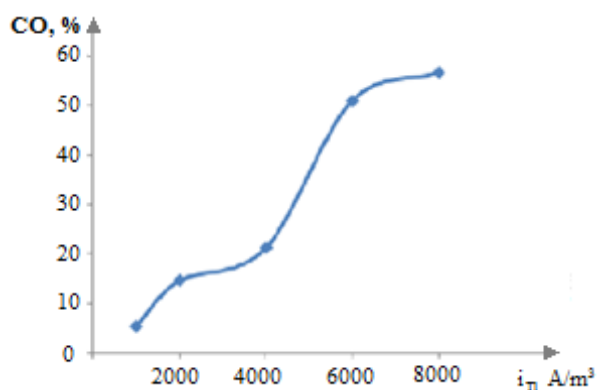


Figure 5 – Influence of the current density at the titanium electrode on the current efficiency of thallium (I) ions:  
 $C_{H_2SO_4} = 14.0$  mol / l;  $\tau = 0.5$  hour;  $t = 20$  °C

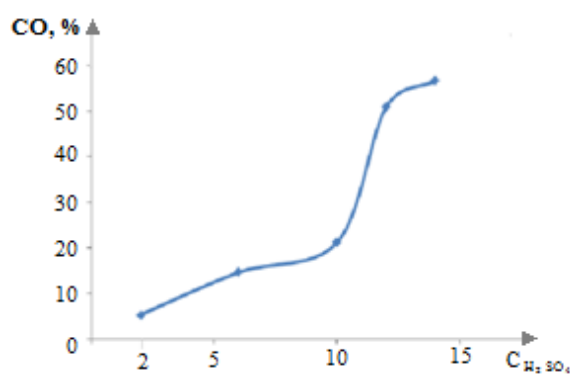


Figure 6 – Effect of electrolyte concentration on the current efficiency of thallium (I) ions:  
 $i = 6000$  A/m<sup>2</sup>;  $\tau = 0,5$  час;  $t = 20$  °C

The order of the thallium oxidation reaction is determined, which corresponds to a value of 1.5. This proves that the electrolyte concentration affects the electrode process.

The influence of the temperature of the electrolyte (sulfuric acid) on the dissolution of thallium electrodes during polarization with alternating current, the current density of 6000 A / m<sup>2</sup> was studied in the range of 20-80 °C (figure 7). As the electrolyte temperature rises, the thickness of the oxide layer formed on the electrode surface decreases. Due to the dissolution of the oxide layer on the electrode surface, the current efficiency of  $Tl^+$  ions sharply increases from 39% to 70%. A further increase in temperature does not affect the current efficiency. Explaining this phenomenon requires special research.

Based on the results of experiments devoted to the study of the effect of the solution temperature on the current efficiency of thallium (I) ions, the value of the activation energy was calculated using the Arrhenius equation. The activation energy of thallium ions is 10.07 kJ / mol. This indicates that the main electrode process takes place in a diffusion mode.

The effect of the duration of electrolysis on the current efficiency and the formation of  $Tl^+$  ions upon polarization of thallium electrodes with alternating current in a sulfuric acid solution is shown in figure 8. With an increase in the electrolysis time, the current efficiency of  $Tl^+$  ions also gradually decreases and eventually reaches a minimum value. Since with an increase in the duration of electrolysis, the concentration of sulfate ions in the solution gradually decreases, the output of thallium sulfate by current decreases accordingly. Apparently, at this time, along with the reaction of the formation of an oxide layer on the electrode surface, a thin thallium sulfate layer covers the electrode surface and inhibits thallium dissolution with the formation of a monovalent ion.

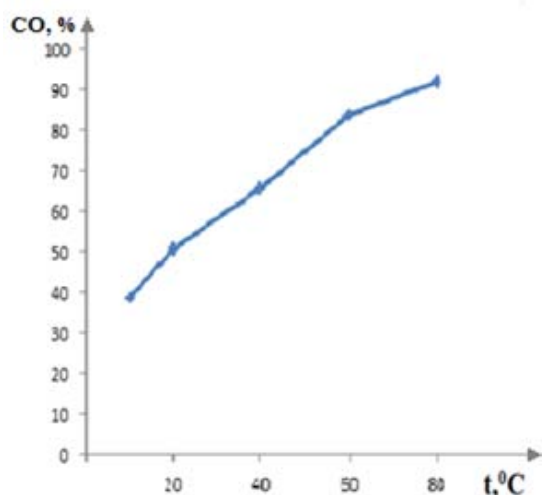


Figure 7– Influence of the electrolyte temperature on the current efficiency of thallium (I) ions:  
 $i = 6000 \text{ A/m}^2$ ;  $\tau = 0,5 \text{ час}$ ;  $C_{\text{H}_2\text{SO}_4} = 9,0 \text{ mol / l}$

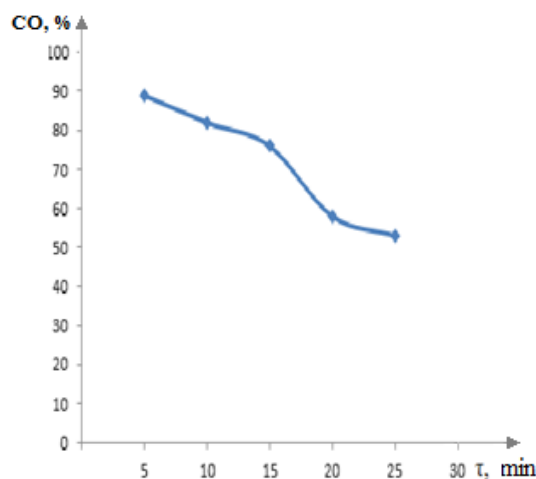


Figure 8 – The effect of electrolysis duration on the current efficiency of thallium (I) ions:  
 $i = 6000 \text{ A/m}^2$ ,  $C_{\text{H}_2\text{SO}_4} = 14,0 \text{ mol / l}$ ,  $t = 20 \text{ }^\circ\text{C}$

The electrochemical behavior of thallium was studied in an aqueous solution of sulfuric acid by removing polarization curves and polarization with an alternating current of 50 Hz. The obtained polarization curves show that thallium hardly dissolves at direct current. When electrolyzed by alternating current, a white precipitate of thallium (I) sulfate is formed as a product. The conducted studies allow us to develop electrochemical effective methods for the synthesis of inorganic salts of this metal by polarizing thallium electrodes with alternating current [19,20].

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### АЙНЫМАЛЫ ТОКПЕН ПОЛЯРИЗАЦИЯЛАУ АРҚЫЛЫ КҮКІРТ ҚЫШҚЫЛЫ ЕРІТІНДІСІНДЕ ТАЛЛИЙ СУЛЬФАТЫН СИНТЕЗДЕУ

**Аннотация.** Айнымалы токпен поляризациялағанда металдардың көпшілігі электрохимиялық ерудің жаңа сипаттамалық емес қасиеттерін анықтайды. Атап айтқанда, металдың еру жылдамдығы тұрақты токпен анодты поляризация кезінде оның еру үдерісімен салыстырғанда едәуір артуы немесе керісінше айтарлықтай төмендеуі мүмкін. Айнымалы және тұрақты токтың поляризация режимінде түзілетін иондардың тотығу дәрежесінде де айырмашылықтар бар.

Қазіргі кездегі өзекті мәселелердің бірі – металл қалдықтарын қайта өңдеп, тұтынушыларға ұсыну. Дегенмен таллий қалдықтарын өңдеудің бірқатар қиыншылықтары бар екендігі белгілі. Қалдықты өңдеудің жаңа әдіс-тәсілдерін тауып, оны пайдалану арқылы таллий ресурстарын көбейтуге және таллийден алынған өнімдер бағасын төмендетуге болады. Сондықтан да жұмысымызда таллийдің электрохимиялық қасиеттерін зерттеу арқылы таллий қалдықтарынан оның қосылыстарын алу бағытында жұмыстар жүргізілді. Тәжірибе нәтижелері күкірт қышқылының сулы ерітіндісінде айнымалы токпен поляризацияланған таллий электродтарының таллий (I) сульфатын түзе еритіндігін көрсетті.

Бұл жұмыста потенциалметрия әдісі арқылы күкірт қышқылы ерітіндісінде таллийдің электрохимиялық тотығу-тотықсыздану кинетикасы зерттелді. Таллийдің электрохимиялық тотығу-тотықсыздану кинетикасын анықтау үшін келесі параметрлер есептелінді: реакция реті, эффективті активтендіру энергия (Еэф). Электролиз режимдерінің: ток тығыздығы, электролит концентрациясы және температурасы, электролиз ұзақтығы зерттелді.

**Түйін сөздер:** потенциодинамикалық поляризациялық қисықтар, электрохимиялық еру, таллий, күкірт қышқылы, ток тығыздығы.

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### СИНТЕЗ СУЛЬФАТА ТАЛЛИЯ В РАСТВОРЕ СЕРНОЙ КИСЛОТЫ ПРИ ПОЛЯРИЗАЦИИ ПЕРЕМЕННЫМ ТОКОМ

**Аннотация.** Одна из актуальных проблем на сегодняшний день – переработка металлических отходов и предоставление их потребителям. Однако известно, что существует ряд сложностей с переработкой таллиевых отходов. Используя новые методы переработки отходов, можно увеличить ресурсы таллия и снизить цены на продукты, полученные из таллия. Поэтому в нашей исследовательской работе были проведены работы по извлечению его соединений из отходов таллия путем изучения электрохимических свойств таллия. Результаты эксперимента показали, что в водном растворе серной кислоты при поляризации переменным током таллиевые электроды растворяются с образованием сульфата таллия (I).

В данной работе методом потенциометрии исследована кинетика электрохимического окисления-восстановления таллия в водных растворах серной кислоты. Для характеристики механизма электроокисления таллия рассчитаны следующие параметры: порядок реакции и эффективная энергия активации процесса (Еэф). Исследовано влияние режимов электролиза: плотности тока, концентрации и температуры электролита, продолжительность электролиза на размерные характеристики полученного сульфата таллия (I).

Потенциометрические кривые снимались на потенциостате "Autolab-30". Измерения проводились в трехэлектродной ячейке относительно хлорсеребряного электрода ( $E^0 = +0,203$  В). Вспомогательным электродом служила платина.

**Ключевые слова:** потенциодинамические поляризационные кривые, электрохимическое поведение, таллий, серная кислота, плотность тока.

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