IRSTI 61.01.91 https://doi.org/10.58805/kazutb.v.1.26-841

TESTING A NEW TECHNOLOGY OF PROCESSING COPPER ELECTROLYTE TO OBTAIN NICKEL SULFATE

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Complex processing of electrolyte is one of the priority tasks of copper producers. In this work, in order to obtain high-quality copper products and nickel sulfate, the following methods were used: neutralization, demineralization, extraction of impurities (As, Sb, Bi) with pseudo-brookite, additional oxidation of iron to trivalent, neutralization of the solution with nickel carbonate with the release of insoluble compounds of copper, iron and zinc and crystallization of nickel sulfate. The advantage of using basic copper sulfate as a neutralizing agent is the prevention of contamination of the electrolyte with foreign components, as well as its high chemical activity. The quantities required of basic copper sulfate for the implementation of the technology are formed at subsequent stages of processing. High-quality copper sulfate is obtained. Basic copper sulfate is also a raw material for obtaining copper oxide. The removal of such impurities as arsenic, antimony and bismuth is carried out using pseudo-brookite as their extractant. To prevent the ingress of divalent iron into the products, it was oxidized to trivalent by introducing calculated amounts of hydrogen peroxide. The working solution was purified from iron, zinc and residual copper by introducing phosphoric acid and nickel carbonate. Nickel sulfate was isolated from the solution by crystallization, its average yield was 71.6%. Identification of intermediate and target products was carried out by IR spectroscopy and laser atomic emission spectroscopy. The test results showed the efficiency of this method of processing copper electrolyte, aimed at expanding the range of products based on copper and nickel.

Keywords: copper electrolyte, pseudobrookite, nickel sulfate, deep decontamination, copper sulfate, neutralization, crystallization

НИКЕЛЬ СУЛЬФАТЫН АЛУ ҮШІН МЫС ЭЛЕКТРОЛИТІН ҚАЙТА ӨҢДЕУДІҢ ЖАҢА ТЕХНОЛОГИЯСЫН СЫНАУ

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Электролитті кешенді өңдеу мыс өндірушілердің басым міндеттерінің бірі болып табылады. Бұл жұмыста жоғары сапалы мыс өнімдері мен никель сульфатын алу үшін келесі әдістер қолданылды: бейтараптандыру, мыссыздандыру, қоспаларды (As, Sb, Bi) псевдобрукитпен экстракциялау, темірді үш валентті темірге дейін тотықтыру, темір, мырыш және мысты ерімейтін қосылыстар түрінде шығару үшін ерітіндіні никель карбонатымен бейтараптандыру және никель сульфатын кристалдандыру. Бейтараптандырушы реагент ретінде негізгі мыс сульфатын пайдаланудың артықшылығы электролиттің бөгде компоненттермен ластануын болдырмау, сонымен қатар оның жоғары химиялық белсенділігі болып табылады. Технологияны жүзеге асыруға оның қажетті мөлшерлері өңдеудің келесі кезеңдерінде қалыптасады. Жоғары сапалы мыс купоросын өндіруді қамтамасыз етеді.

Негізгі мыс сульфаты да мыс оксидін алу үшін шикізат болып табылады. Мышьяк, сурьма және висмут сияқты қоспаларды жою үшін экстрагент ретінде псевдобрукитт қолданылды. Екі валентті темірдің өнімдерге енуіне жол бермеу үшін оны сутегі асқын тотығының есептік мөлшерін енгізу арқылы үш валентті темірге дейін тотықтырдық. Жұмыс ерітіндісін темірден, мырыштан және мыс қалдықтарынан тазартуға фосфор қышқылы мен никель карбонатын енгізу арқылы қол жеткізілді. Никель сульфаты ерітіндіден кристалдану арқылы бөлініп алынды, оның орташа шығымы 71,6% құрады. Аралық және мақсатты өнімдерді анықтау ИҚ-спектроскопия және лазерлік атомдық эмиссиялық спектроскопия көмегімен жүзеге асырылды. Сынақ нәтижелері мыс пен никель негізіндегі өнімдердің ассортиментін кеңейтуге бағытталған мыс электролитін өңдеудің бұл әдісінің тиімділігін көрсетті.

Түйін сөздер: мыс электролиті, псевдобрукит, никель сульфаты, мыс электролитін терең мыссыздандыру, мыс купоросы, бейтараптау, кристаллдау.

ИСПЫТАНИЕ НОВОЙ ТЕХНОЛОГИИ ПЕРЕРАБОТКИ МЕДНОГО ЭЛЕКТРОЛИТА ДЛЯ ПОЛУЧЕНИЯ СУЛЬФАТА НИКЕЛЯ

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Комплексная переработка электролита одна из приоритетных задач производителей меди. В данной работе с целью получения качественной медной продукции и сульфата никеля использованы метод нейтрализации, обезмеживание, экстракция примесей (As, Sb, Bi) псевдобрукитом, доокисление железа до трехвалентного, нейтрализация раствора карбонатом никеля с выделением нерастворимых соединений меди, железа и цинка и кристаллизации сульфата никеля. Преимуществом использования основного сульфата меди в качестве нейтрализующего реагента является недопущение загрязнения электролита посторонними компонентами, а также его высокая химическая активность. Необходимые его количества для реализации технологии образуются на последующих стадиях переработки. Обеспечивается получение качественного медного купороса. Основной сульфат меди является также сырьем для получения оксида меди. Вывод таких примесей, как мышьяк, сурьма и висмут осуществлен применением псевдобрукита в качестве их экстрагента. Для предупреждения попадания двухвалентного железа в продукты, его окисляли до трехвалентного введением расчетных количеств пероксида водорода. Очистка рабочего раствора от железа, цинка и остаточного содержания меди достигалась введением фосфорной кислоты и карбоната никеля. Сульфат никеля выделен из раствора методом кристаллизации, его средний выход составил 71,6%. Идентификация промежуточных и целевого продуктов проведена методами ИК-спектроскопии и лазерной атомно-эмиссионной спектроскопии. Результаты испытаний показали эффективность данного способа переработки медного электролита, направленной на расширение ассортимента продукции на основе меди и никеля.

Ключевые слова: медный электролит, псевдобрукит, сульфат никеля, глубокое обезмеживание, медный купорос, нейтрализация, кристаллизация

Introduction. Processed technological solutions of electrolytic refining of copper contain significant amounts of copper and nickel sulfates, sulfuric acid, arsenic and other components of copper electrolyte. To avoid the concentration of impurities in the electrolyte, part of the working solution is removed from the process for processing. The process of processing copper electrolyte is aimed at creating conditions for the selective release of harmful and undesirable impurities and ensuring the effective separation of valuable components for their further use in the technological cycle of copper production and obtaining marketable products. Analysis of existing developments [1-11] indicates a number of significant shortcomings in the known technological developments for the removal of arsenic from copper electrolyte and further processing of the solution, such as contamination of the electrolyte with foreign components, loss of copper in the form of copper-arsenate cakes, etc., due to which some of the proposed technologies have not been implemented.

To eliminate copper losses and obtain nickel sulfate, we have developed a new method for processing copper electrolyte [12]. The electrolyte processing process consists of four main stages, followed by vacuum filtration and crystallization of the final product:

- neutralization and deep de-copperization of copper electrolyte;

- extraction of arsenic, antimony and bismuth into the solid phase with pseudobrookite;

- additional oxidation of ferrous iron to ferric;

- deep neutralization of the working solution of carbon nickel with the release of insoluble compounds, iron and zinc, and crystallization of nickel sulfate.

According to the technological scheme, the process of processing copper electrolyte with obtaining nickel sulfate as a target product consists of four main stages:

This stage is carried out under the conditions accurately determined by the authors [12, 13]. The copper electrolyte is preliminarily diluted with water by two times. The latter neutralization is carried out by introducing a copper (II) oxide weighed portion in a molar ratio of CuO : Cu = 3:1 with constant stirring for four hours at a temperature of 98^{0}C. The basic copper sulphate formed after separation is used to neutralize the electrolyte following portions. This operation is carried out at a temperature of 85^{0}C with constant stirring for 15 minutes until the pH 1.3-1.5 reached. The basic copper sulfate is taken in a molar ratio to sulfuric acid of 2:1. Electrolyte samples are taken throughout the experiment and the solution pH is determined until the required value is reached.

The resulting copper-nickel mother liquors are re-diluted with water 2 times and subjected to deep de-curing with copper (II) oxide (CuO:Cu = 3:1) at 98^{0}C for four hours with constant stirring.

2. The arsenic, antimony and bismuth extraction into the solid phase with the help of pseudobrukite is carried out in accordance with the method developed by us earlier [14].

The process is carried out in a thermostated cell at a temperature of 60^{0}C with a given sulfuric acid concentration at constant stirring for one hour. A pseudobrukite weighed portion is taken equal to the ratio of the precipitant to arsenic 1:1 and served in two portions (the precipitant (DRP) dosage ratio is equal to 2). The hot solutions are then filtered out. The precipitate is separated from the resulting solution.

3. Additional oxidation of ferrous iron to ferric iron is carried out according to the conditions strictly specified by the authors [15]: a 50% solution of hydrogen peroxide is used as an oxidizing reagent, which is introduced into solutions at a temperature of 55^{0}C, the oxidation process takes 1 hour with constant stirring. The amount of hydrogen peroxide required to oxidize all Fe^{2+} ions to Fe^{3+} is calculated from the reaction equation:

$$H_2O_2 + Fe^{2+} + H_2 \rightarrow Fe^{3+} + 2H_2O \qquad (1)$$

4. Deep neutralization of the working solution with nickel carbonate with the release of insoluble compounds of copper, iron and zinc, and crystallization of nickel sulfate.

Before deep neutralization of the electrolyte with nickel carbonate, it is necessary to transfer iron, zinc and the residual content of copper in solutions into a solid phase.

For this purpose, an 87% solution of orthophosphoric acid in a predetermined amount is injectedinto the working solutions. Orthophosphoric acid in a stoichiometric ratio is injected to bind impurities into sparingly soluble phosphates according to the reaction equation:

$$3Me^{2+} + 3NiCO_3 + 2H_3PO_4 \rightarrow Me_3(PO_4)_2 \downarrow + 3Ni^{2+} + 3CO_2 \uparrow + 3H_2O \qquad (2)$$

whereas – Me^{2+}- ions $Cu^{2+}$and Zn^{2+}.

With the participation of the Fe^{3+} ion, the reaction proceeds according to the following equation:

$$2Fe^{3+} + 3NiCO_3 + 2H_3PO_4 \rightarrow 2FePO_4 \downarrow + 3Ni^{2+} + 3CO_2 \uparrow + 3H_2O \qquad (3)$$

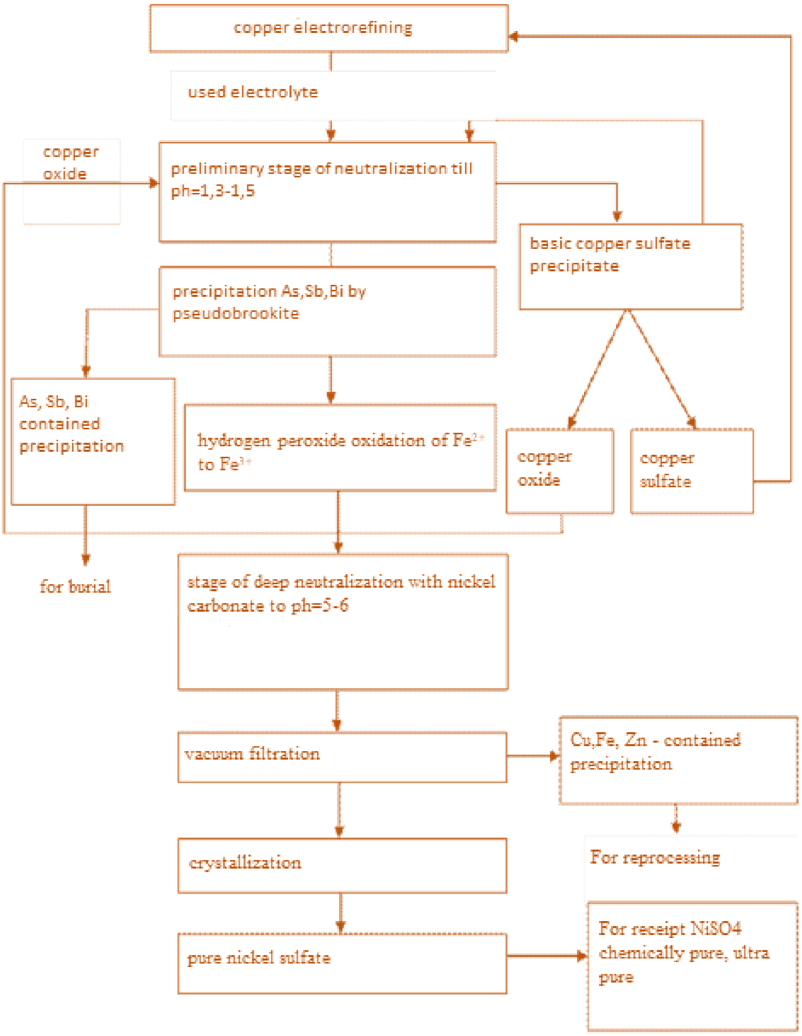

Fig. 1 - Technological scheme of the integrated processing of copper electrolyte to obtain nickel sulfate

Then the solutions are heated to 85^0C with stirring. Water is added in an amount calculated from the reaction equation and heated further to 90^0C. A nickel carbonate predetermined amount is added to the heated solutions in portions.

The solutions are constantly stirred and maintained at a temperature of 90^0C for one and a half hours. When adding each portion, it is necessary to take a sample and measure the solutions pH. Measurements of pH are carried out until the value of 5.5-5.9 is set. With each pH measurement, the samples are cooled to a temperature of $23\text{-}25^0$C. Then the solutions are maintained at a temperature of 90^0C. The authors of [15] found required conditions under which precipitates of iron, copper and zinc insoluble salts are formed, namely pH = 5.5-5.9.

At the process end, the solutions are subjected to vacuum filtration in a hot state. Unreacted nickel carbonate as well as copper, iron and zinc salts insoluble precipitates remain on the filter. The solutions pH is brought to values of ≈ 1.99-2.02 with the help of adding the calculated amounts of concentrated sulfuric acid. The authors of [15] established that this pH limit is the conditions under which the nickel sulfate crystallization occurs. Further, the investigated solutions are cooled to a temperature of $20\text{-}23^0$C and left to stay aside for the nickel sulfate crystallization.

The purpose of this work is to test a new technology of processing of copper electrolyte with the removal of copper in the form of products assalts and hydroxides in large-scale laboratory conditions, and obtain a new product - nickel sulfate in accordance with the technological scheme developed by us (Figure 1).

Materials and methods. The object of large-scale laboratory tests is the technological copper-containing sulfuric solution of the corporation ”KAZAKHMYS SMELTING” (Republic of Kazakhstan, city of Balkhash). The quantitative content of the main components of the process solution (Cu, Ni, H_2SO_4, As, Sb, Bi, Fe, Zn) was determined on a SPEKS SSP-705-4 scanning spectrophotometer, as well as on a laser atomic emission spectrometer SPEKS LAES MATRIX CONTINUUM (Closed Joint Stock «Company Spectroscopic Systems» Russian Federation, 2016) , the results of which are presented in Table 1.

Large-scale laboratory tests were carried out with a volume of the working solution equal to 500 ml.

Table 1 - Electrolytecomposition

Component	Cu	Ni	H2SO4	As	Sb	Bi	Fe	Zn
g/l	51.20	16.98	95.88	14.04	8.63	5.78	9.42	9.17

Table 2 - Results of the copper electrolyte neutralization process

$(Cu_3(OH)_4SO_4:H_2SO_4) = 2:1$; $t = 85^0C$; $\tau = 15$ min)

pH of working solution		H_2SO_4(g/l)	Cu(g/l)	Ni(g/l)	As(g/l)
Before experiment	After experiment				
0.08	1.31	2.79	31.0	16.70	14.03

Results and discussion. Based on known methods for processing copper electrolyte [12,14], 500 ml of water was added to a copper electrolyte with a volume of 500 ml, and treated with 96 g of copper (II) oxide in a molar ratio CuO:Cu = 3:1. The reaction mass was heated in a thermostated cell to 98^0C with constant stirring for four hours. The solution volume was decreased by 2 times at the end of the neutralizing process. The solution was filtered in a hot state, resulting in a precipitate of basic dark green copper sulfate weighing 406.59 g on the filter. Further, basic copper sulfate obtained in an amount of 348.3 g was used for subsequent neutralization of copper electrolyte with a volume of 500 ml for each experiment.

The neutralization of free sulfuric acid was carried out for 15 min in a thermostated cell at a temperature of 85^{0}C. The solution pH was monitored during the experiment until its values reached 1.31. Hot solutions were subjected to vacuum filtration, and then cooled to a temperature of 20^{0}C. Copper sulfate weighing ≈ 43.8 g was obtained in each experiment. The solutions were analyzed for the residual content of sulfuric acid, copper, nickel, and arsenic. The average values of the investigation results are shown in Table 2.

The obtained copper-nickel mother liquors were re-diluted with water 2 times and subjected to de-curing with copper (II) oxide. Width of the confidence interval according to the Cochran criterion for copper content Δ= 0.18δ = 2Δ.

The resulting basic copper sulfate weighing 24.99 g was used to neutralize the next portion of the electrolyte. The main copper sulfate is also a raw material for the production of copper oxide, copper sulfate. The resulting solutions, after neutralization and de-curing of the electrolyte, were analyzed for the residual content of sulfuric acid, copper, nickel, and arsenic. The average values of the analysis results are shown in Table 3.

Table 3 - Copper electrolyte dressing from copper results

(CuO:Cu= 3:1; t = 98^{0}C; τ = 4 hour)

pH of working solution		H_2SO_4(g/l)	Cu(g/l)		Ni(g/l)	As(g/l)
Before experiment	After experiment		Before experiment	After experiment		
1.31	2.37	0.21	31.0	0.79	16.5	14.01

Copper-bearing sediments were identified by IR spectroscopic analysis (Figure 2). According to reference data [15], intense absorption bands at 1106-1366 cm^{-1} and 451-621 cm^{-1} are related to the sulfate ion. Also, a band in the region of 3140 - 3433 cm^{-1} is characteristic of stretching vibrations of OH-groups, and absorption bands at 1340-1627 cm^{-1} associated with the presence of water molecules in a highly hydrated sediment. Based on the data of the IR spectra of the precipitate and the results of photometric analysis for copper, it was established that the resulting precipitate is the main copper sulfate $Cu_3(OH)_4SO_4$.

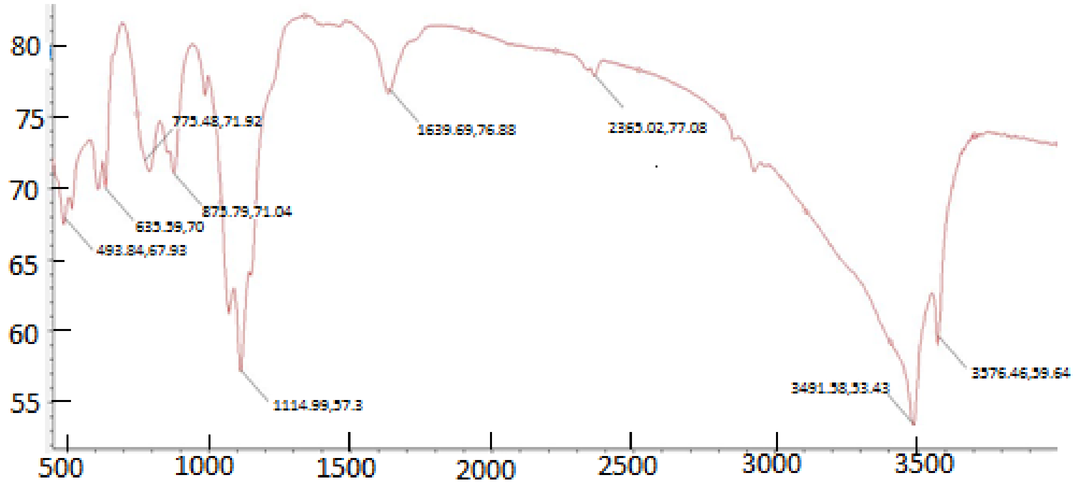

Fig. 2 - IR spectrogram of a copper-bearing precipitate

Additionally, the analysis for the content of impurities in the resulting sediment was carried out on a laser atomic emission spectrometer SPEX LAES MATRIX CONTINUUM (Figure 3). As can be seen from the spectrogram, intense green peaks are observed, which correspond to copper ions. The presence of such impurities as iron, zinc, arsenic, antimony, bismuth in the composition of the precipitate is not observed on the spectrogram. Thus, the resulting basic copper sulfate does not contain foreign impurities of other metals and is a chemically pure compound.

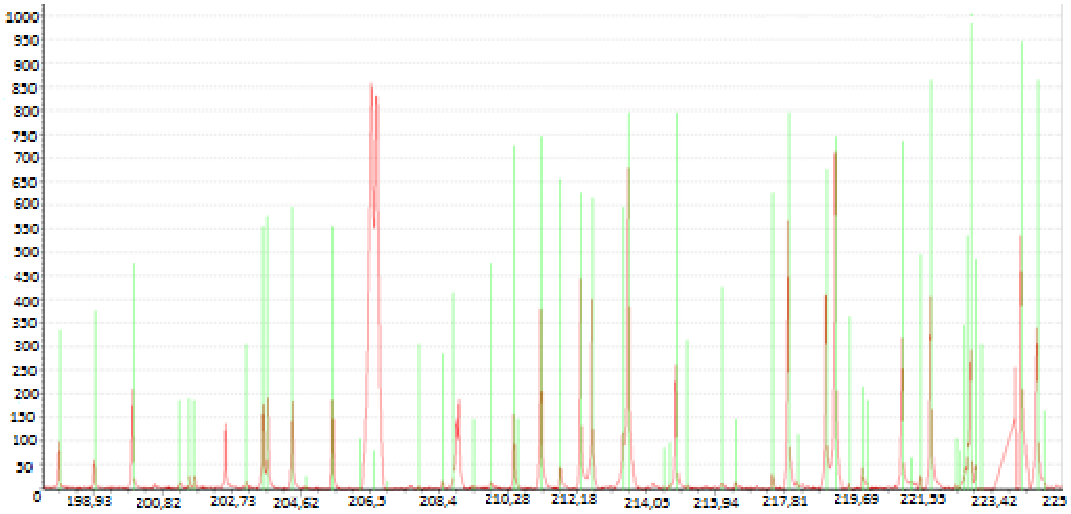

Fig. 3 – Laser spectrogram of copper-bearing sediment

The deposition of arsenic, antimony and bismuth with pseudobrookite was carried out in a thermostated cell at a temperature of 60^0C, with a given concentration of sulfuric acid with constant stirring for one hour. We used the resulting solutions with a volume of 182 ml each. A weighed portion of pseudobrookite was taken equal to the ratio of the precipitant to arsenic 1:1 in the amount of 14.01 g and fed in two portions (DRP= 2). Then the solution was filtered while hot, the resulting filtrates with a volume of 165 ml were analyzed for the residual arsenic content, antimony and bismuth in them. The results of analyzes for the residual content of antimony, bismuth arsenic, shown in Table 4, showed that their content in solutions is reduced to trace amounts, i.e., the resulting solutions are almost completely purified from them.

For the oxidation of Fe^{2+} ions to Fe^{3+}, 3.1 ml of hydrogen peroxide was introduced into solutions with a volume of 158 ml of each experiment. Then the solutions were stirred for one hour in a thermostated cell at a temperature of 50-55^0C. At the end of the experiments, the solutions were cooled and analyzes were carried out for the total iron content and for Fe^{3+}, the results of all analyzes of the experiments were averaged (g/l): $C(Fe_{tot})$ = 6,035; $C(Fe^{3+})$ = 6,030. Under these conditions, complete oxidation of Fe^{2+}toFe^{3+}ions is achieved.

In order to purify working solutions from ions of copper, iron, zinc ($C(Zn^{2+})$ = 6.7 g/l; $C(Fe^{3+})$ = 6.03 g/l; $C(Cu^{2+})$ = 0.79 g/l) a solution of phosphoric acid in the amount of 13.8 mlwere added in them.

Then the solutions were heated with stirring to 85^0C, added H_2O in an amount of 25.5 ml, and the solutions were heated to 90^0C. Nickel carbonate weighing 12.62 g was added to the heated solution in portions. The solutions were constantly stirred and kept at a temperature of 90^0C for 1.5 hours. With the addition of each portion of nickel carbonate, a sample was taken and the pH of the solutions was measured. Nickel carbonate was added until a pH of about 5.98 was reached.

Insoluble precipitates and unreacted nickel carbonate were separated by vacuum filtration. 0.1 ml of concentrated sulfuric acid was added to the filtrate, after the addition of which the pH of the solutions reached a value of ≈ 2.39.

At the end of the process, the solution was subjected to vacuum filtration while hot. Insoluble sediments weighing 2.19 g remained on the filter. Then the test solution was cooled to a temperature of 20-23^{0}C. Crystals of nickel sulfate began to grow in the solution on the seventh day. The crystals were separated from the working solution by filtration, washed with distilled water, dried, and weighed. As a result, we got 66.35 g heptahydrate nickel sulfate, on average in each experiment.

Table 4 – The results of the precipitation of arsenic, antimony and bismuth by pseudobrookite(*t* = 60 ^{0}C; *τ* = 1 hour; DRP= 2)

№ Experiment	Fe2TiO5:As	Concentration after experiment, (g/l)			Degree of precipitation, %		
		As	Sb	Bi	As	Sb	Bi
1	0.5:1	0.070	0.0023	0.0011	99.15	99.85	99.88
2	0.8:1	0.032	0.0016	0.0005	99.67	99.89	99.90
3	1:1	Traces	Traces	Traces	99.99	99.99	99.99
4	1.2:1	Traces	Traces	Traces	99.99	99.99	99.99
5	1.5:1	Traces	Traces	Traces	99.99	99.99	99.99

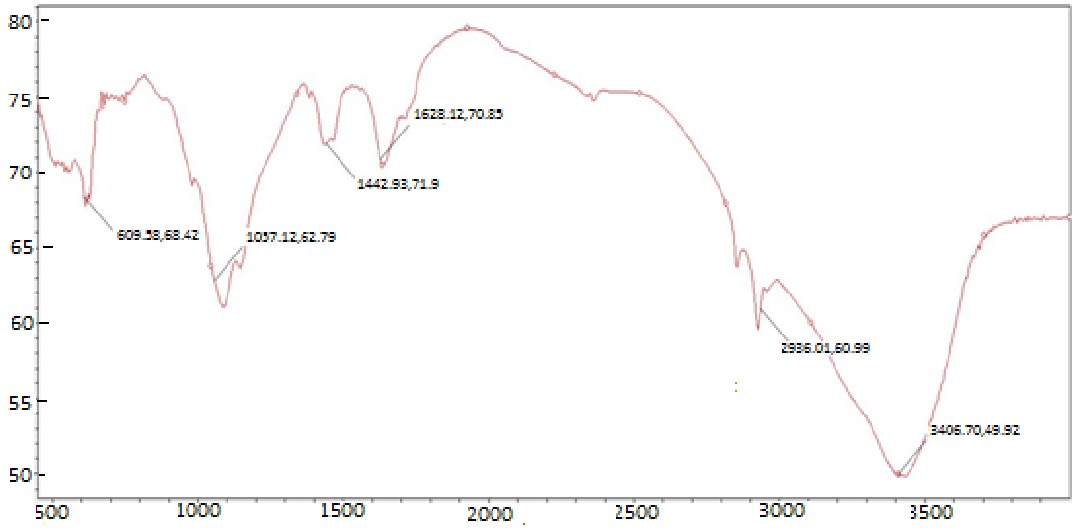

Fig. 4 - IR spectra of crystals of the obtained nickel sulfate

Calculated the average yield of the target product of nickel sulfate. The theoretical mass of the target product according to calculations from the reaction equations:

$$NiCO_3 \rightarrow NiO + CO_2 \uparrow \quad (4)$$

$$NiO + H_2SO_4 \rightarrow NiSO_4 + H_2O \quad (5)$$

$$NiSO_4 + 7H_2O \rightarrow NiSO_4 \cdot 7H_2O \quad (6)$$

was 92.59 g. The average yield of the target product of nickel sulfate was 71.66% of the theoretically possible.

Analysis of the obtained IR spectra of nickel sulfate crystals (Figure 4) confirmed the absence of copper, iron and zinc impurities in nickel sulfate crystals. In the figure, one can see intense bands at 1106-1366 cm^{-1} and 451-621 cm^{-1}, which corresponds to the absorption bands of sulfate ions; in addition, bands are observed in the region of 1340-1670 cm^{-1}, characteristic of the presence of crystallized water in a highly hydrated sediment. It is also possible to observe low-intensity absorption bands in the region of 3440-3470 cm^{-1} characteristic of stretching vibrations of OH-groups, and an insignificant absorption band at 2830-2850 cm^{-1}related to the carbonate ion [15]. That is, the obtained crystals were confirmed to be nickel sulfate ($NiSO_4$•$7H_2O$).

The nickel sulfate obtained as a result of large-scale laboratory tests, as noted earlier, does not contain metal impurities, and can be used as a raw material for obtaining nickel sulfate of reagent grade, special grade, which in turn confirms the technological significance of the tests carried out and the effectiveness of the technological scheme of the new technology for processing copper electrolyte (Figure 1).

Conclusion. The large-scale laboratory tests have shown the efficiency of the new technology of processing copper electrolyte and the possibility, along with obtaining marketable copper products: basic copper sulfate, copper oxide, copper sulfate, to expand the range of products of vitriol processing based on nickel - nickel sulfate. The results of large-scale laboratory tests can serve as initial data for planning and conducting subsequent production tests of a new technology of processing copper electrolyte.

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