**Methods for Processing Precipitates Formed During the Treatment of Uranium-Containing Solutions**

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**Abstract.** This article discusses the influence of phosphates on uranium processing processes and the method of processing uranium-phosphate precipitates. The sorption process using strong basic anion exchangers is widely used for the extraction of uranium from productive solutions obtained from underground leaching. In addition to uranium, the solution entering the sorption process contains additional impurities. In this case, cations of alkali and alkaline earth metals, as well as divalent ions of copper, iron, cobalt, and manganese, are not sorbed. A number of anions, on the contrary, are well sorbed by anion exchangers and are sorbed by the separated uranium ion. These include sulfate, nitrate, chloride, fluoride, and phosphate anions. These anions are classified as depressant additives. There are ions similar to strongly basic anion exchangers, which over time accumulate in anion exchangers, damaging them, and also reduce the degree of extraction of minerals, in particular uranium.

**Keywords:** underground leaching, uranium-containing productive solution, depressants, sorption, desorption, phosphate ion, sulfuric acid, precipitate.

**INTRODUCTION**

The development of uranium mining based on physicochemical technologies in the Kyzylkum region is inextricably linked with the general trend in the global mining industry and occurs against the backdrop of continuous changes in economic conditions and demand in the raw materials market. Along with the production of other rare metals, the extraction, processing, and further use of uranium also have a significant impact on our economy. Currently, there are many unresolved problems in the technology of uranium production. One of these problems is the negative influence of various ions on the sorption and desorption process of uranium. During the selective leaching of ore with sulfuric acid, hexavalent uranium, trivalent iron, vanadium, phosphorus, arsenic, molybdenum, chloride ions, nitrate ions, and others can be found in solution as anion exchangers. In addition, the sulfuric acid itself can exist in solution in the form of sulfate or bisulfate ions. All these anions, as a rule, can be in the form of additional salts - calcium, magnesium, sodium, potassium, aluminum, cobalt, copper, titanium, etc., which, since they are not characterized by the formation of complex sulfates, remain completely in the aqueous phase, but high concentrations of anions are an inhibitor of sorption uranium extraction [1, 2].

**METHODS**

The increase in deposits related to the development of various unfavorable minerals within uranium mineralization results in the presence of additional elements, including unwanted ones. These elements, which have not previously been encountered in ore mining practices, appear alongside uranium in the productive solutions during underground leaching [3, 4].

The mass concentration of phosphorus in technological solutions was determined using a photometric method. This measurement procedure is applied to technological solutions, and the photometric method was used to measure the mass concentration of phosphorus in the range from 0.001 g/dm³ to 0.6 g/dm³. The amount of phosphorus compounds in the solutions ranges from 0.1 to 0.13 g/l. Phosphorus compounds comprising 0.1–0.12 g/l of phosphorus are sorbed together with uranium on strongly basic anion exchangers (BО020) [5, 6].

The phosphorus content in the anionite reaches 2-6 kg/t, which introduces the process of nitrate desorption of uranium from uranium phosphate precipitates containing 25-50% uranium into the workflow. Currently, in industry, there is no method for the highly selective, i.e., 100% preliminary, elution of phosphorus ions from uranium-saturated anionites. During processing, the efficiency of selective elution of the formed phosphorus compounds via sulfuric acid selective elution is 85-90%. This process transfers the uranium phosphate precipitation step from the desorption stage to the final product storage stage [7, 8]. The phosphorus ions remaining in the anionite alongside uranium are desorbed, enriched during the precipitation stage, and sent to the Hydrometallurgical Complex as a final product.

Before treatment with gelatin, the chemical composition of the precipitate from the final product was determined by chemical analysis, revealing the presence of the following elements, %: uranium—50-60; phosphorus—5-6; loss on ignition—15-20%. This chemical composition of the separated precipitate corresponds to the formula NH₄UO₂PO₄·3H₂O and is considered to be the mineral uramphite, i.e., a hydrated phosphate of uranium and ammonium.

In production conditions, precipitates of uranyl and ammonium phosphates accumulate in containers for storing finished products. During the processing of the finished product with gelatin, silicon-containing precipitates enter the same container along with it. As a result, a mixture of uranium phosphate and silicon-containing precipitates accumulates in containers for storing the finished product. The analysis of this precipitate mixture is presented in Table 1.

**TABLE 1.** Chemical composition of the precipitate

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Component composition, %** | | | | | | | |
| **U** | **Р** | **SiО2** | **Fе** | **Аl2О3** | **SО4** | **NО3** | **others** |
| 28,4 | 2,5 | 34,2 | - | - | - | - | 34,9 |
| 32,1 | 5,1 | 29,7 | - | - | - | - | 33,1 |
| 45,5 | 4,4 | 8,0 | - | 2,0 | 11,4 | - | 28,7 |
| 44,8 | 3,8 | 9,2 | - | - | 15,6 | - | 26,6 |
| 42,1 | 6,0 | 13,5 | 0,7 | - | 3,2 | - | 34,5 |
| 41,6 | 5,4 | 11,5 | 0,7 | - | 9,1 | 2,1 | 29,6 |

The high content of uranium in these deposits necessitates their conversion into finished products during processing.

Phosphorus-containing uranyl compounds constitute a very wide group of compounds. A group of uranium phosphate minerals with a common formula is known in nature: Mх(UО2)у(ХО4)z\*nH2О.

Here: M=Nа+,K+,Сu+2,Mg+2,Са+2,Аl+3,Mn+2,Fе+2, Аs, V

(1)

In the uranium extraction technology during the reprocessing process, practically insoluble ammonium phosphate uranyl is used for precipitation in water and diluted acids.

(2)

Due to the low precipitate value of the complex (1.9-2.5), effective purification of uranium from numerous elements is achieved.

The ability of uranium phosphate compounds to interact with various classes of extractants and sorbents is determined by the ionic state of uranium in solutions. A wide-ranging study to determine the stability of uranium phosphate complexes in the concentration range of 0.01-10 M phosphate ions was carried out by Teymer. The stability constants for the complexes were found to be equal:

; (3)

; (4)

; (5)

It is reported that uranyl compounds in orthophosphoric acid solutions exist mainly in the form of positively charged complexes [5]. As the concentration of phosphoric acid increases, the number of neutral and negative ion complexes decreases. The formation of uranyl complexes is demonstrated within the nitric acid concentration range from 0.01 to 10 M.

(6)

Stability constants are 21.6 and 13.8, respectively. The species and the di-phosphates with potassium and ammonium were studied. Based on the provided data, the solubility products of the uranium-containing complexes:

; (7)

;(8)

;(9)

We are primarily interested in uranium phosphate complexes containing ammonium ions. Under laboratory conditions and in nature, crystalline ammonium uranyl phosphate is found with three water molecules—the mineral uramphite: NH₄UO₂PO₄·3H₂O.

*Method of selective leaching of uranium from phosphorus precipitates with soda and precipitation of phosphorus compounds with divalent iron*

The following optimal regime of uranium leaching from phosphorus deposits can be provided:

а) dissolution of phosphorus deposits;

- the initial soda content in the solutions is 100-150% g/l;

- selective dissolution ratio-3:1;

- selective dissolution temperature-40-50оС;

- selective dissolution duration-4 hours;

- the number of leaching stages for obtaining uranium content less than 0.05% is -5;

b) phosphorus ion deposition;

- reagent-precipitating iron sulfuric acid solution. Selected from the groups of precipitating reagents: magnesium, aluminum, and iron oxide;

- excess of the precipitating reagent in the phosphorus composition - at least three;

- precipitation time is 2-3 hours (see Table 2).

**TABLE 2.** Dependence of uranium-containing sediment composition on the number of implemented   
selective leaching-precipitation cycles

|  |  |  |  |
| --- | --- | --- | --- |
| **Number of cycles completed** | **Precipitate composition, %** | | |
| **U** | **Fе** | **Р** |
| **III** | 0,3-0,5 | 40-41 | 2,0-1,5 |
| **IV** | 0,15-0,11 | 62-65 | 1,5-1,3 |
| **V** | 0,05-0,04 | 69-70 | 1,1-1.0 |

As a result of treating the carbonate solution containing uranium with an iron sulfate solution, a mixed precipitate with divalent iron, phosphorus compounds, and carbonate ions is formed. The composition of the initial precipitates by dry mass, depending on the number of leaching-precipitation cycles, is presented in Table 2.

The moisture content of the precipitate constitutes 50-70%. The composition of the precipitate (in terms of dry weight) in kg/l is: Radium n10⁻¹², Polonium n10-11, which exceeds the permissible level (PL) and requires the construction of special waste storage facilities. A precipitate containing 65-70% iron can also be selectively leached with sulfuric acid, followed by oxidation, where divalent iron is used as an oxidant to enhance the in-situ leaching process of uranium.

**TABLE 3.** Composition of gelatin

|  |  |  |
| --- | --- | --- |
| **glycine** | NH2СH2СООH | 54% |
| **Proline** | С5H5NHСООH | 45% |
| **Oxyproline** | 5H5NHОNСООH | 14% |
| **Glutamic acid** | HООС—СH2—СH2—СH(NH2)СООH | 12% |
|  | NH2(NH)С—NH(СH2)3—СHNH2СООH | 9% |
| **Lysine** | СH2—(СH)2(NH2)—СH(NH2)—СООH | 5% |

Technical gelatin is a product of the recycling of organic residues and is a mixture of various amino acids, the composition of which depends on the method of its preparation (see Table 3):

Primarily first-grade technical gelatin was used in the work.

**RESULTS AND DISCUSSION**

*Selective Leaching of Uranium Phosphate Precipitates with Sodium and Ammonium Bifluoride Solutions*

To carry out the process of selectively leaching phosphorus and minor silicon compounds from uranium-containing precipitates, they were first treated with a sodium hydroxide solution. Experimental work on the alkaline leaching of uranium from the uranium phosphate precipitate was conducted by stirring 10 g of the precipitate with 100 ml of NaOH solution for 4 hours on a magnetic stirrer. The initial composition of the precipitate was determined to be 29% uranium and 4% phosphorus. After selective leaching, the residue was filtered, and the quantity of uranium and phosphorus ions in the filtrate and the precipitate was determined after washing with technical water. The experimental results are presented in Table 4.

**TABLE 4.** Results of treating uranium-containing precipitates with sodium hydroxide solutions of varying concentrations

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **NaOH concentration, g/l** | **Filtrate** | | | **Precipitate** | |
| **U, g/l** | **Р, g/l** | **U, %** | | **Р, %** |
| **10** | 0,012 | 0,025 | 33,32 | | 3,72 |
| **30** | 0,018 | 0,177 | 44,63 | | 4,2 |
| **50** | 0,056 | 3,04 | 50,58 | | 1,52 |
| **70** | 0,280 | 3,74 | 52,7 | | 0,176 |
| **90** | 0,490 | 3,9 | 53,5 | | 0,068 |
| **100** | 0,47 | 3,8 | 55,93 | | 0,041 |
| **120** | 0,50 | 3,63 | 33,7 | | 0,041 |

The experimental results showed that increasing the amount of sodium hydroxide in the solutions from 50 g/L to 100 g/L led to an increase in the efficiency of the phosphorus selective leaching process by 30-35% (from 60% to 95-96%).

A series of laboratory experiments were conducted with an ammonium bifluoride solution to completely separate silicon compounds from the precipitate. The process of extracting silicon compounds was carried out in two stages. The interaction between the solution and the precipitate lasted 2 hours. The initial composition of the precipitate was (%): uranium-28; phosphorus-3.36; and silicon compounds-35. The water-soluble uranium was washed from the precipitate. Afterwards, the first stage of selective leaching of silicon compounds was carried out using solutions of the following composition:

А) 100 g/l NаОH;

B) 100 g/l NаОH + 20 g/l Ammonium bifluoride;

V) 100g/l NаОH + 50 g/l Ammonium bifluoride,

The volumetric ratio of the precipitate to the solution was 1:3. After a 2-hour interaction period, the pulp was filtered, and the precipitate was analyzed for its uranium and phosphorus content. The results of the laboratory experiments after the first stage of selective leaching of the precipitate are presented in Table 5.

**TABLE 5.** Results of the selective leaching of uranium-containing precipitates with alkaline solutions by   
adding ammonium bifluoride solution (Stage I)

|  |  |  |  |
| --- | --- | --- | --- |
| **Leaching solution, g/l** | **Precipitate, %** | | |
| **U** | **Р** | **SiО2** |
| **100NаОH** | 39,5 | 0,365 | 21,96 |
| **100NаОH+20NH4F\*HF** | 36,3 | 0,40 | 29,45 |
| **100NаОH+50NH4F\*HF** | 33,8 | 0,48 | 30,22 |

After the first stage of processing, the silicon compounds in the precipitate were subjected to repeated selective leaching under the same conditions as described above. The experimental results are presented in Table 6. The data presented in Table 6 show that in an alkaline medium, ammonium bifluoride does not significantly affect the process of separating fine silicon compounds during the uranium phosphate precipitation process.

**TABLE 6.** Results of the selective leaching of uranium-containing precipitates with   
alkaline solutions by adding ammonium bifluoride solution (Stage II)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Leaching solution, g/l** | **Precipitate, %** | | | **Precipitate, %** | | |
| **U** | **Р** | **SiО2** | **U** | **Р** | **SiО2** |
| **100NаОH** | 53,52 | 0,098 | 10,13 | 0,154 | 2,87 | 16,8 |
| **100NаОH+20NH4F\*HF** | 55,4 | 0,148 | 10,32 | 0,138 | 3,2 | 18,7 |
| **100NаОH+50NH4F\*HF** | 43,84 | 0,123 | 6,16 | 0,140 | 3,46 | 20,8 |

In the next stage of the laboratory experiments, the effect of temperature on the selective leaching process of uranium phosphate precipitates in a 10% caustic soda solution was determined. The three-stage selective leaching process was carried out at temperatures of 20, 40, and 60°C, with a L:S (Liquid-to-Solid) ratio of 1:3, and an interaction time of 3 hours per stage. After each stage, the solution was filtered and replaced with a fresh portion of the 10% sodium alkali solution; the filtrate and the precipitate were analyzed for their content of uranium, phosphorus, and silicon compounds.

The separation of phosphorus and silicon compounds from the uranium-phosphorus precipitates proceeded most efficiently at 40°C. An increase in temperature to 70°C led to the formation of gel-like compounds that were difficult to filter.

Based on the conducted research, the following conclusions can be drawn: A 10% caustic soda solution was used for the selective leaching of silicon and phosphorus compounds from the uranium-phosphorus precipitate. The leaching process was conducted over 4 hours with a precipitate-to-solution volume ratio of 1:10.

The addition of ammonium bifluoride to the leaching solution did not show a significant effect on the process of extracting silicon compounds from the studied precipitate. The leaching process is considered most efficient when carried out at 40°C

*Selective leaching of uranium from uranium-containing precipitates with sulfuric acid*

For the selective leaching of precipitates consisting of uranium phosphate and silicon with sulfuric acid (H₂SO₄), the composition of the precipitate sample is given in Table 7.

**TABLE 7**. Composition of the precipitate consisting of uranium, P, and Si compounds, %

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **U** | **Р** | **Аl** | **SiО2** | **SО42-** | **Other** |
| 43,5 | 7,3 | 0,63 | 12,0 | 3,5 | 33,07 |

In the selective leaching of uranium phosphate precipitates, the process was carried out to determine the optimal concentration of H₂SO₄, with a L:S ratio of 1:10, periodic stirring over 2 hours, and sulfuric acid amounts of 50, 100, 150, and 200 g/l. The effect of the sulfuric acid amount on the dissolution of the precipitates is presented in Table 8.

**TABLE 8.** Effect of H₂SO₄ concentration on the selective leaching of uranium from uranium phosphate precipitate

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sulfuric acid concentration, %** | **Filtrate, g/l** | | **Precipitate, %** | |
| **U** | **Р** | **U** | **Р** |
| **5%** | 10,2 | 1,78 | 36,5 | 4,82 |
| **10%** | 17,5 | 1,88 | 25,78 | 3,2 |
| **15%** | 24,4 | 2,66 | 7,7 | 1,0 |
| **20%** | 24,4 | 2,66 | 5,8 | 0,75 |

Table 8 shows that the maximum dissolution of the precipitate was observed at a sulfuric acid concentration of 15-20%. For the economical and efficient use of H₂SO₄ in subsequent experiments on processing uranium phosphate precipitates, based on dissertation research, a 15% H₂SO₄ solution was used. Based on these parameters, the L:S ratio for the complete dissolution of the precipitate was determined. The experimental results are presented in Table 9.

The experimental data presented in Table 8 indicate that using a 15% H₂SO₄ solution with an L:S ratio of 1:10 is efficient for dissolving the precipitate.

**TABLE 9.** Effect of the Solid:Liquid (S:L) ratio on the leaching process of uranium-containing precipitates

|  |  |  |  |
| --- | --- | --- | --- |
| **S:L** | **Filtrate, g/l** | **Precipitate, %** | |
| **U** | **Р** | **U** |
| **1:5** | 39,4 | 5,69 | 3,63 |
| **1:10** | 23,7 | 3,85 | 0,980 |
| **1:20** | 13,5 | 2,22 | 0,600 |
| **1:30** | 8,4 | 1,31 | 0,383 |
| **1:50** | 5,85 | 0,785 | 0,327 |

A technological scheme for treating the settled uranium phosphate precipitates with sulfuric acid was developed. According to it, under static conditions with an L:S ratio of 1:10, periodic stirring for 2 hours, and sulfuric acid concentrations of 50, 100, 150, and 200 g/l. The composition of the studied precipitate was (%): uranium - 42.75; phosphorus - 6.375; aluminum - 0.54; silicon dioxide - 11.2; sulfate ion - 3.3. The effect of sulfuric acid concentration on the dissolution of the precipitate is presented in Table 10.

**TABLE 10.** Effect of sulfuric acid concentration on the dissolution of precipitates consisting of uranium   
and phosphorus compounds

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sulfuric acid concentration, %** | **Filtrate, g/l** | | **Precipitate, %** | |
| **U** | **Р** | **U** | **Р** |
| **5** | 9,8 | 1,93 | 35,9 | 4,72 |
| **10** | 16,9 | 1,93 | 24,86 | 3,146 |
| **15** | 23,4 | 2,77 | 7,6 | 0,956 |
| **20** | 23,4 | 2,77 | 5,78 | 0,634 |

According to the data presented in Table 9, the optimal regime should be considered treating the precipitate with 15-20% sulfuric acid, where the maximum dissolution of the precipitate occurs. Under similar conditions, the S:L ratio at which the most complete dissolution of the precipitate occurs was determined. The results of the laboratory experiments are presented in Table 11.

**TABLE 11.** Effect of the Solid-to-Liquid (S:L) ratio on the dissolution process of uranium phosphate precipitates

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S:L** | **Filtrаte, g/l** | | **Precipitate, %** | |
| **U** | **Р** | **U** | **Р** |
| **1:5** | 39,4 | 5,69 | 3,63 | 3,95 |
| **1:10** | 23,7 | 3,85 | 0,980 | 1,9 |
| **1:20** | 13,5 | 2,22 | 0,600 | 1,11 |
| **1:30** | 8,4 | 1,31 | 0,383 | 0,95 |
| **1:50** | 5,85 | 0,785 | 0,327 | 0,65 |

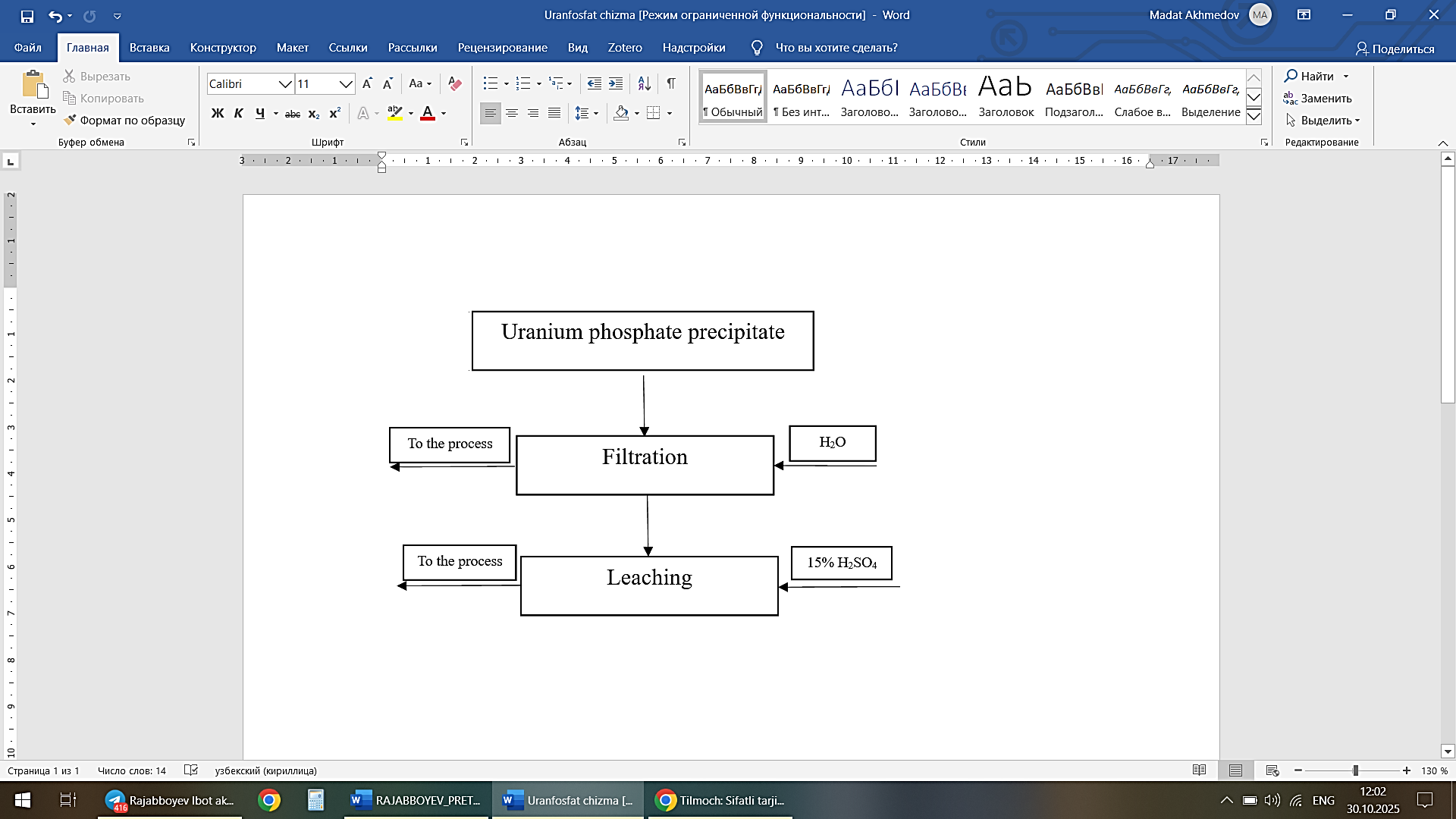
The experimental data presented in Table 11 indicate that for dissolving the precipitate, a 15% sulfuric acid solution with an L:S ratio of 1:10 is used. The uranium-containing solutions obtained as a result of dissolving the uranium phosphate precipitates have high acidity: 140-150 g/l H₂SO₄.

The method of processing uranium phosphate precipitates with a sulfuric acid solution is the most efficient and includes the following steps (see Figure 1):

1. Uranium phosphate precipitates are sent from the product storage tanks to a filter press, and the water-soluble uranium is washed with water;

2. Then, the uranium phosphate precipitates are dissolved in 15% sulfuric acid at a solution-to-precipitate volume ratio of approximately 10:1 with stirring for 2–4 hours;

3. Afterwards, the sulfuric acid solution, containing up to 20-25 g/l of uranium and 3-4 g/l of phosphorus, is left to settle for 1-2 days;



**FIGURE 1.** Technological scheme for the selective leaching of uranium phosphate precipitates

4. The clarified solution is returned to the technological process.

The selective leaching of uranium from the precipitates with sulfuric acid is repeated another 2-3 times until the uranium content in them is reduced to below 0.05%.

**CONCLUSION**

The selective leaching of uranium phosphate precipitates with a sulfuric acid solution is considered the most optimal.

Uranium phosphate precipitates are transferred from finished product tanks to a filter press, and water-soluble uranium is washed with water;

The uranium phosphate precipitates are then dissolved in 15% sulfuric acid at a solution-to-precipitate volume ratio of approximately 10:1, with stirring for 2–4 hours;

The sulfuric acid solution, containing up to 20–25 g/L uranium and 3–4 g/L phosphorus, is then left to settle for 1–2 days.

The selective leaching of uranium from the precipitate using sulfuric acid is repeated an additional 2–3 times until the uranium content in the precipitates decreases to below 0.05%. After treatment with the sulfuric acid solution, the productive solutions from the precipitate are sent for precipitation.

At the same time, the increase in the uranium and phosphorus content in the solution is insignificant and does not exceed 10%—this hardly changes the equilibrium saturation values of the anionite for uranium and phosphorus. In other words, the composition of the phosphorus compounds in the saturated anionite obtained from the process remains almost unchanged.

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