

## PHYSICAL AND CHEMICAL FOUNDATIONS OF THE TECHNOLOGY FOR OBTAINING URANIUM OXIDES

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The article presents the results of industrial testing of processes for obtaining high-purity uranium compounds, including extraction, concentration, and purification of uranium from a mixture of nitric and phosphoric acids, solid-phase stripping, and reduction of U(VI) to U(IV). It is shown that the combination of three different extraction lines for the concentration and purification of uranium from accompanying valuable elements and radioactive isotopes makes it possible to obtain a natural uranium concentrate with a low content of impurities, which determine the economics of uranium hexafluoride production. The processes of drying and roasting of crystals of ammonium uranyl tricarbonate after solid-phase re-extraction in an atmosphere of reducing gases of ammonia and hydrogen make it possible to obtain uranium dioxide suitable for fluorination to obtain tetra and uranium hexafluoride.

### INTRODUCTION

The production of uranium, as the first fuel for nuclear power plants, appeared in the 1950s. It successfully solved the problem of processing low-grade uranium ores with a high extraction of uranium and its compounds. Together with the production of uranium, a new assessment of its quality appeared – the nuclear purity, which limits the content of impurities with a high thermal neutron capture cross-section to a limit that does not interfere with the fission of the <sup>235</sup>U isotope nuclei [1].

The technology for the production of uranium and its compounds in Ukraine began to develop rapidly in the 1960s when the processes of sorption and extraction of uranium compounds were first developed and introduced into the industry. They made it possible to increase the extraction of uranium to 99.5% and ensured its purity at the level of 99.9% [2]. The main reagents for processing uranium ores were sulfuric and nitric acids, sodium carbonate. Subsequently, a mixture of sulfuric and nitric acids was used for the processing of phosphorites [2].

The sulfuric acid method of uranium ores processing is the main in the uranium industry. Along with the advantages, low cost, and availability of sulfuric acid, produced at uranium plants, this method has a drawback – lime which is used for neutralization, forms gypsum dumps. This significantly increases the volume of waste pulp and the area of their storage [1, 3].

Nitric acid leaching of uranium ores made it possible to achieve the highest recovery of uranium and accompanying elements. This technology has not found wide application due to the higher than sulfuric acid cost, and the need to create an associated production line for the utilization of nitrate ions. Associated production of fertilizer from reagents used for the processing of uranium and rare metals was more profitable than the direct production of saltpeter from Na<sub>2</sub>CO<sub>3</sub> and HNO<sub>3</sub> [2, 3].

The use of nitric acid in the uranium technology was developed in Ukraine only at the Prydniprovsky chemical plant (PChP) [3]. At the same time, nitric acid was

utilized in the production of ammonium or sodium nitrates. This significantly reduced the cost of uranium.

The quality of uranium oxide concentrate (U<sub>3</sub>O<sub>8</sub>) with a U content of ≥ 84% required additional costly operations to reduce U(VI) to U(IV) in a gaseous environment with hydrogen or membrane electrolyzers with purification of UF<sub>4</sub> from impurities that prevent its conversion into UF<sub>6</sub> [4].

For the extraction of uranium from sulfuric acid media, mixtures of extractants were used: a solution of tributyl phosphate (TBP) in kerosene, di-(2-ethylhexyl) phosphoric acid (D2EHPA) with TBP, trialkyl amine, etc. [5]. This also made it possible to extract such valuable elements as molybdenum, vanadium, iron, scandium, etc. The process of extracting uranium from nitric acid media was first studied at the PChP, some results are presented in [3].

The process of re-extraction of uranium from the extractant is of great importance for obtaining pure uranium oxides. Ammonium carbonate is usually used as the main stripping agent. The study of the quality of crystals of ammonium uranyl tricarbonate (AUTC) in the sulfuric acid extraction version was carried out in [6]. A feature of that work was the circulation of carbonate mother liquor after the separation of AUTC crystals, which worsened the quality of U<sub>3</sub>O<sub>8</sub>. The low concentration of uranium (15 g/L) in the saturated extractant and ammonium carbonate (150 g/L) did not allow larger AUTC crystals to be obtained. The process of crystal calcination was carried out under rarefaction, which led to the oxidation of U(IV) to U(VI).

The types of extractors used have a key influence on the efficiency of the extraction process. They have evolved from box-type extractors to centrifugal apparatus. The first studies of extraction processes began in the 1960s based on bulky mixer-settlers with low kinetics and low specific productivity. In the 1970s, pulsating columns were developed, as a result, several apparatuses were created for the implementation of rare metal extraction processes [7]. Large volumes of flammable-explosive extractant in the cycle required serious costs to ensure production safety. This led to the

creation of more efficient devices of a new design: vibro- and pneumatic extractors [8]. The latest achievement in extraction technology is centrifugal extractors, which improve the kinetics of the process and reduce the contact time of the phases to seconds. This made it possible to intensify the extraction process, reduce the volume of the extractant, and implement the extraction process without the formation of a third phase [9].

The purpose of industrial testing was to optimize the parameters of the processes of extraction, re-extraction, drying and calcination, reduction of U(VI) to U(IV). To do this, it was necessary to study the processes of selective extraction of uranium from its various

compounds with purification from impurities to a purity of 99.9%, obtaining uranium dioxide with a bulk density of 2.7...2.9 g/cm<sup>3</sup>. For the processing of ore solutions of various chemical compositions, it was necessary to optimize the technological scheme to ensure a high extraction of uranium and its purification from thorium, molybdenum, and vanadium.

## MATERIALS AND METHODS

To carry out experiments on the extraction purification of uranium, two compositions of working solutions were obtained, the chemical composition of which (in g/L) is given in Table 1.

Table 1

Chemical composition of the studied solutions

Name	HNO <sub>3</sub>	H <sub>3</sub> PO <sub>4</sub>	U	Th	Fe <sup>3+</sup>	Si	NaNO <sub>3</sub>
Nitrophosphate solution of uranium	≥ 100	60...80	4.5...9.0	0.3	≤ 14	0.1	–
Nitric acid commercial solution of uranium from a concentrate of the “yellowcake” type	60...90	–	40...60	–	≤ 5.0	0.15	≥ 200

The first solution was obtained during the processing of uranium-rare-earth phosphorites from the Melovoye deposit (Kazakhstan). The second solution was obtained by processing a rich concentrate of the “yellowcake” type under the nitric acid scheme.

To recover uranium into the organic phase, a mixture of TBP and D2EHPA in kerosene was used. A rich uranium solution containing up to 50 g/L of uranium and an extractant partially saturated with uranium enters the first extraction line after contact with a poor phosphate solution in the second line. During the process, the extractant was saturated with uranium and sent to the solid-phase re-extraction of uranium by a solution of (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>. After re-extraction, the extractant was treated with 45% nitric acid to saturation up to 500 g/L in HNO<sub>3</sub> and returned to the cycle.

For industrial testing, three-section box extractors with a volume of 15 m<sup>3</sup> with a mechanical drive were used in all trials. For extraction from the phosphate solution, an extraction column with a diameter of

600 mm and a volume of 4 m<sup>3</sup> was used in parallel. A cascade of vibroextractors with a diameter of 1200 mm and a volume of 0.9 m<sup>3</sup> made of stainless steel was used in parallel to extract uranium from a nitric acid solution.

The raffinate of the second stage of extraction, after the treatment, was sent to the processing of phosphoric acid with the production of complex fertilizers of the “nitrophos” type. The raffinate of the first line was sent to the production of sodium nitrate.

The mother liquor after crystallization of AUTC contains ammonium nitrate, uranium with a concentration corresponding to its solubility at an excessive carbonate content of the process. It was sent to prepare a carbonate solution for the stripping of uranium.

In the process of solid-phase re-extraction, AUTC crystals with a size of more than 100 μm were formed, which ensured the required quality of uranium oxide in terms of the content of impurities after washing, the required granulometric composition, and an increased bulk density of UO<sub>2</sub>.

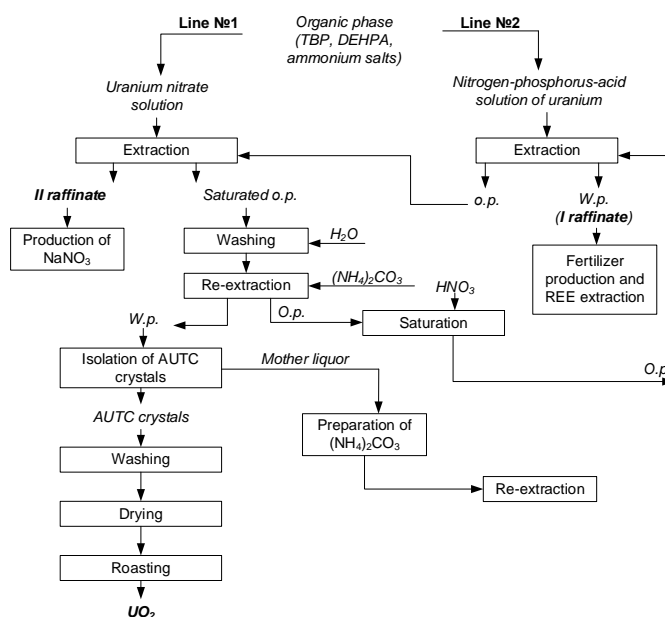


Fig. 1. A scheme of industrial testing of the technology for producing uranium dioxide

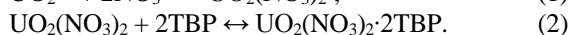
The AUTC crystals were washed, hydrocycloned, dried in a furnace at a temperature of 350 °C, and entered the VGPT-8 furnace, where the modes of roasting under vacuum and overpressure were studied.

Industrial tests were carried out according to the following block diagram (Fig. 1).

NH<sub>3</sub> and CO<sub>2</sub> were captured in the scrubber to form a mixture of bicarbonate and ammonium carbonate, which was returned to the stripping cycle. The uranium oxide concentrate after roasting was analyzed for the content of impurities according to TU 85.1981-89. The granulometric composition and bulk density were determined before and after shaking. During the tests, the saturation of the extractant with uranium, its content in the raffinate were analyzed, the optimal ratio of the water and organic phases (w.p. and o.p.), as well as the duration of each process, were determined.

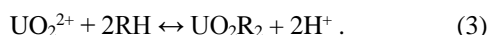
## RESULTS AND DISCUSSION

After laboratory studies, a solution containing (80±20) g/L TBP and (30±10) g/L D2EHPA in hydrocarbon solvent kerosene was chosen as an extractant. The choice of the extractant was determined by the chemical composition of commercial uranium solutions. Uranium in solution No. 1 was in the form of the compound UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>, which dissociated into the UO<sub>2</sub><sup>2+</sup> cation and the nitrate anion. The process of uranium extraction by TBP was accompanied by the solvation of neutral UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> molecules by two TBP molecules and was described by the equations:



Uranyl nitrate in a mixture of nitric and phosphoric acids at a concentration of 90 and 80 g/L, respectively, was in a more dissociated form than in a mixture of nitric acid and sodium nitrate. D2EHPA was used to extract it.

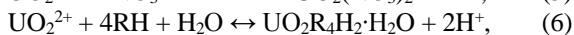
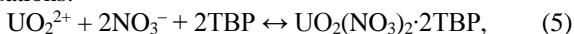
For complete recovery of uranium, which was present in solution in the form of the UO<sub>2</sub><sup>2+</sup> cation and the neutral UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> molecule, TBP was mixed with D2EHPA. In this case, the extraction of uranium was described by the equation:



Taking into account the participation of water molecules in the reaction, the equation gained the following form:



The overall process of uranium extraction with a mixture of TBP and D2EHPA was described by the equations:



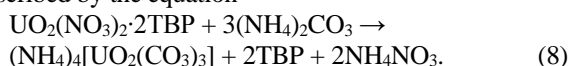
During the industrial tests, the phenomenon of the formation of a solid phase, the so-called "beard", regularly appeared. The results of studying the causes of this undesirable process were presented in [6].

The presence of impurities of iron, silicon, titanium, zirconium after the extraction process led to emulsification and interaction of the extractant with the hydrolysis products of TBP: mono- and dibutyl phosphoric acids. The third phase required its withdrawal from the cycle to stabilize the efficiency of the extraction process. It was found that the third phase is at the

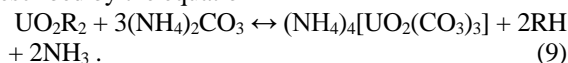
interface and can grow in height in box extractors. Its removal from the system once per month with a volume of up to 1.5 m<sup>3</sup> did not violate the efficiency of the extraction process.

The process of returning the mother liquor to the stripping cycle was also tested. This led to the accumulation of impurities, especially thorium, which worsened the quality of the commercial product. To remove thorium and impurities, the mother liquor was dissolved in nitric acid, which required the organization of an additional process of standalone extraction for the removal of uranium and thorium. The gas cleaning dust and the UO<sub>2</sub> fraction > 2 mm were sent for dissolution in nitric acid.

The process of uranium re-extraction from TBP is described by the equation



The process of uranium re-extraction from D2EHPA is described by the equation



In fact, three mechanisms are realized in both processes: solvation, cation exchange, and dehydration with the formation of a mixed complex.

The uranium nitric-phosphate solution contained the largest amount of impurities elements. The ratio of uranium to phosphorus, thorium, REE, and iron was 1:8, 20:1, 1:3, 1:2, respectively, which could lead to an increase in their concentration in the commercial product. Under TU 95.1981-89, the content of thorium and rare earth elements in uranium oxide should not exceed 0.005% and 1·10<sup>-5</sup>%, respectively, which was ensured by a three-stage extraction and constant removal of impurities with the mother liquor from the cycle through standalone extraction.

Taking into account the lower concentration of uranium in the nitric-phosphoric acid solution than in the nitric acid solution, the process of extracting uranium from the phosphorus-containing solution on the second line was used first in the course of the extractant at the ratio of the organic and aqueous phases O:W = 3:1. This ensured the degree of uranium extraction of 99.7% and the conversion of REE, P<sub>2</sub>O<sub>5</sub>, Fe, and radionuclides (<sup>232</sup>Th, <sup>227</sup>Ac, <sup>210</sup>Po, <sup>210</sup>Pb) into raffinate, which formed insoluble compounds with the phosphate ion. The raffinate with a uranium content of ≤ 30 g/L was supplied for the extraction of rare earth elements and the utilization of phosphorus in the form of fertilizers.

The capacity of the extractant for uranium after the second line did not exceed 18 g/L, and for its maximum saturation with uranium (up to 45 g/L), it was sent to the first line, where it contacted with a rich nitrate solution of uranium and sodium at a ratio of O:W = 1:1. At the same time, the main impurities (Fe, Al, Ca) were displaced into the raffinate, which was supplied to the production of sodium nitrate.

The extractant after stripping was washed with water at O:W = 3:1 and saturated with nitric acid up to 50 g/L. It returned to the cycle for standalone extraction. After the extraction of uranium and thorium at O:W = 8:1, the extractant was saturated with uranium to a content of 10 g/L, while the extraction of uranium was 99.9%.

The optimal phase ratio in the extraction process on the first line was 1:1, the number of stages was 8, which made it possible to ensure the content of uranium in

raffinate was less than 30 mg/L. The results of the uranium extraction process testing are shown in Fig. 2.

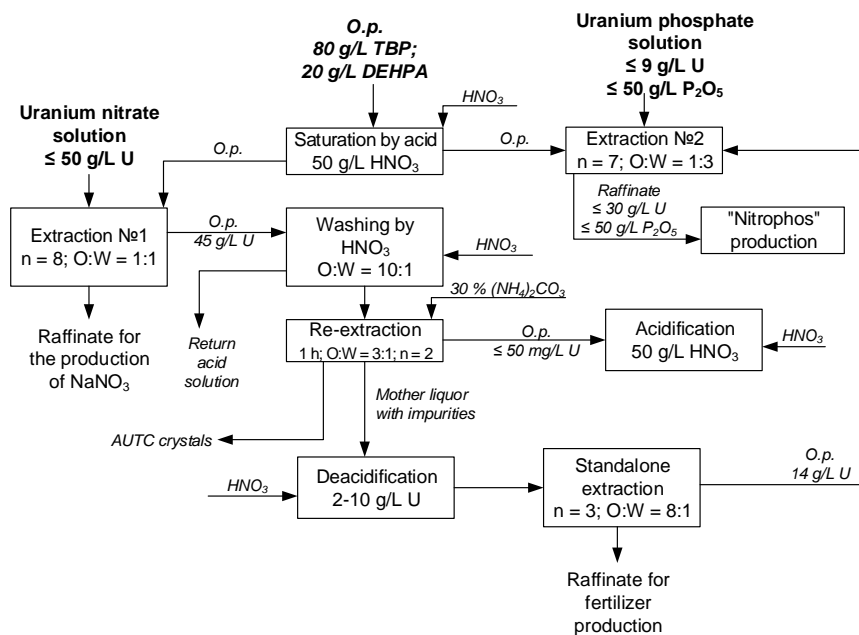


Fig. 2. A scheme of uranium purification by extraction

The technological parameters of extraction processes are shown in Table 2.

Table 2

Technological mode of the extraction process

Name	Uranium concentration, g/L			Number of steps	O:W	Note
	in the water phase	in the extractant	in raffinate			
Standalone uranium extraction	2.0...10.0	$\leq 10.0$	$\leq 0.01$	3	1:(5...10)	–
Extraction of uranium from a phosphate solution	$\leq 7.0$	10.0...14.0	$\leq 0.03$	7	1:(2...3)	The formation of the third phase is observed
Extraction of uranium from a nitric acid solution	$\geq 46.0$	45.0	$\leq 0.03$	8	1:1	

The phase ratio at the standalone extraction stage varied depending on the concentration of uranium in the mother liquor, the amount of dust after calcination, and the coarse fraction  $\text{UO}_2 > 2 \text{ mm}$ , within 2...10 g/L, or due to different impurity content and the need for maximum recovery of uranium and thorium.

The dependence of the capacity of the extractant on the initial concentration of uranium is shown in Fig. 3.

The isotherm showed that 3 stages are sufficient for the complete extraction of uranium. At the same time, the uranium content in the ammonium nitrate solution, which was sent to the production of fertilizers, is less than 10 mg/L. The isotherm of uranium extraction from a phosphate solution confirmed the need for 7 extraction steps, which made it possible to quite fully extract thorium from the extractant since it formed sparingly soluble compounds with a phosphate ion, the content of which was 35...50 g/L in terms of  $\text{P}_2\text{O}_5$ .

The saturation of the extractant with uranium in the first line was  $\leq 45 \text{ g/L}$ , which ensured that the uranium

content in the raffinate (a nitrate solution of sodium nitrate) was no more than 30 mg/L.

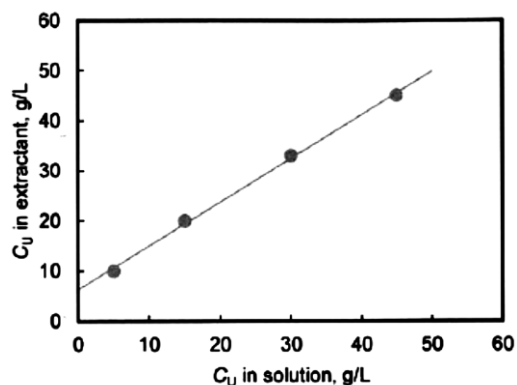


Fig. 3. Uranium extraction isotherm

For additional purification of uranium from impurities, the saturation of the extractant for uranium was increased to 70 mg/g, but this required an additional extraction process before the recovery of uranium from the raffinate to prevent its entry into sodium nitrate.

For solid-phase re-extraction of uranium and thorium, a 30% solution of  $(\text{NH}_4)_2\text{CO}_3$  was used at O:W = 3:1. The process was carried out for 2 h at a temperature of 20...25 °C. AUTC crystals after filtration were classified in hydrocyclones with a diameter of 100 mm with a taper of 15% to separate small crystals contaminated with impurities, as well as oxides and carbonates of divalent metals. The extraction of uranium into AUTC was more than 99.5%.

A 30% ammonium carbonate solution was chosen for the stripping of uranium from a saturated extractant. The process was carried out at a ratio of O:W = 3:1 for 1 h. This made it possible to obtain large, well-filtered AUTC crystals. The excess concentration of  $(\text{NH}_4)_2\text{CO}_3$  was up

to 50 g/L, which made it possible to achieve almost complete conversion of uranium into AUTC and the transformation of metal impurities into insoluble carbonates or hydroxides. The latter formed a lighter phase than AUTC crystals, so they were easily separated during classification.

To separate large AUTC crystals and metal impurities precipitate, the hydrocyclone method was used, which was implemented in three stages by countercurrent during washing of AUTC crystals with water and an ammonium carbonate solution on hydrocyclones. The results of industrial tests made it possible to implement the following technological scheme (Fig. 4).

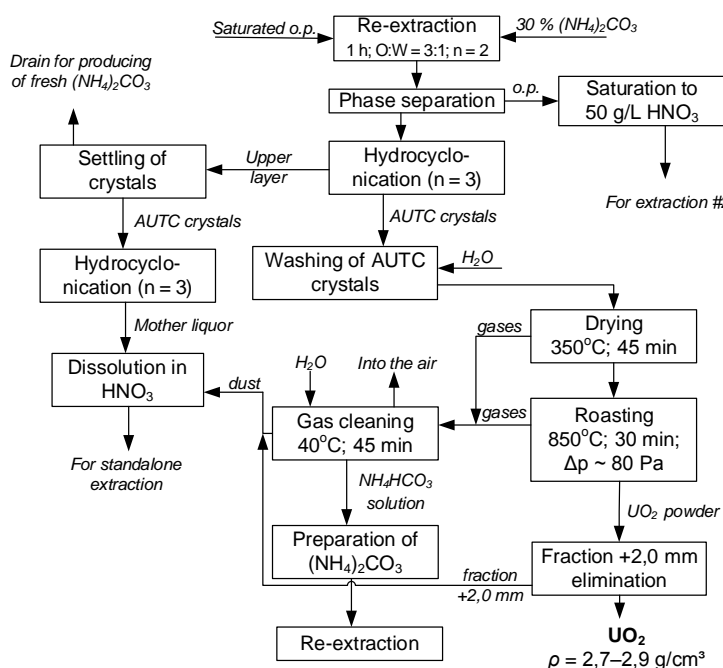
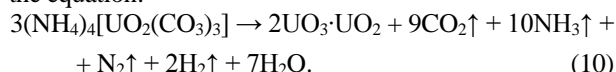
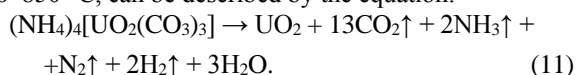


Fig. 4. A scheme of uranium dioxide obtaining

A mother liquor with a uranium content of 1...2 g/L containing an excess of  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{NH}_4\text{HCO}_3$  was treated with nitric acid. A solution with excess acidity up to 50 g/L was sent to standalone extraction at O:W = 8:1 to recover uranium and thorium. The raffinate containing up to 200 g/L of  $\text{NH}_4\text{NO}_3$  was supplied to the production of “nitrophos” fertilizer. The drying temperature of AUTC crystals was maintained at 350 °C in furnaces rotating at a speed of 30 rpm. Then the crystals were roasted at a temperature of  $(800\pm 50)$  °C. The roasting of AUTC crystals under vacuum with air access increased the uranium concentration to 85%, which corresponded to the formula  $2\text{UO}_3 \cdot \text{UO}_2$ . The process can be described by the equation:



The process of roasting at an excess pressure of reducing gases up to 80 Pa, formed during the decomposition of AUTC in the temperature range of 800–850 °C, can be described by the equation:



The size of AUTC crystals makes it possible, after calcination, to obtain uranium dioxide powder with a particle size of up to 2 mm and a density after tapping of 2.7...2.9 g/cm<sup>3</sup>. Chemical analysis of uranium oxide for the content of critical impurities showed their lesser content than according to TU 95.1981-89.

It was of practical interest to compare the purity of  $\text{UO}_2$  and  $\text{UF}_4$ . The standard chemical composition of  $\text{UF}_4$  to obtain  $\text{UF}_6$  is as follows, %:  $\text{UF}_4 \geq 96$ ;  $\text{UO}_2\text{F}_2 - 2.0$ ;  $\text{UO}_2 - 1.8$ ; Th < 0.005; Ni < 0.0035; Cr < 0.009; Mn < 0.0001; Cd < 0.00005; B < 0.00001; Mo < 0.001; V < 0.001. Uranium dioxide obtained by the developed technology with the use of extraction, re-extraction and roasting processes corresponded in quality to  $\text{UF}_4$ , which made it possible to recommend it for direct hydrofluorination.

## CONCLUSIONS

1. Industrial tests of the extraction refining of uranium made it possible to optimize the parameters of the technology for uranium dioxide obtaining, meeting the requirements for the production of  $\text{UF}_6$  without additional purification.

2. During the tests, a complex extraction scheme for the processing of uranium-containing solutions of various compositions with the utilization of all reagents in the form of fertilizers was developed.

3. A mixture of TBP and D2EHPA in kerosene was tested as an extractant, which made it possible to efficiently purify uranium from thorium and impurities with a large thermal neutron capture cross-section.

4. During the solid-phase re-extraction of uranium with ammonium carbonate, large AUTC crystals were obtained, which made it possible to effectively separate them from impurities and obtain large particles of uranium dioxide up to 2 mm in size with a high bulk density of up to 2.9 g/cm<sup>3</sup>.

5. The combination of drying and roasting of AUTC crystals maintaining the excess pressure of hydrogen-containing gases within the range of up to 0.8 kPa, made it possible to completely reduce U(VI) to U(IV).

6. Obtaining UO<sub>2</sub> in the refining process eliminates the costly hydrogen reduction of U<sub>3</sub>O<sub>8</sub> to UO<sub>2</sub>, which is a prerequisite of UF<sub>6</sub> production. This allows to significantly reduce the cost of uranium hexafluoride production.

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## ФІЗИКО-ХІМІЧНІ ОСНОВИ ТЕХНОЛОГІЇ ОТРИМАННЯ ОКСИДІВ УРАНУ

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Викладено результати промислових випробувань процесів отримання високочистих сполук урану, що включають екстракцію, концентрування та очищення урану із суміші азотної та фосфорної кислоти, твердофазну реекстракцію та відновлення U(VI) до U(IV). Показано, що поєднання трьох різних екстракційних ліній концентрування та очищення урану від супутніх цінних елементів та радіоактивних ізотопів дозволяє отримувати концентрат природного урану з низьким вмістом домішок, що визначають економіку виробництва гексафториду урану. Процеси сушіння та прокалювання кристалів амонію уранілтрикарбонату після твердофазної реекстракції в атмосфері відновлювальних газів аміаку та водню дозволяють отримати двоокис урану придатну для фторування з одержанням тетра- та гексафториду урану.

## ФИЗИКО-ХИМИЧЕСКИЕ ОСНОВЫ ТЕХНОЛОГИИ ПОЛУЧЕНИЯ ОКСИДОВ УРАНА

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Изложены результаты промышленных испытаний процессов получения высокочистых соединений урана, включающих экстракцию, концентрирование и очистку урана из смеси азотной и фосфорной кислоты, твердофазную реэкстракцию и восстановление U(VI) до U(IV). Показано что сочетание трех различных экстракционных линий концентрирования и очистки урана от сопутствующих ценных элементов и радиоактивных изотопов позволяет получать концентрат природного урана с низким содержанием примесей, определяющих экономику производства гексафторида урана. Процессы сушки и прокалки кристаллов аммония уранилтрикарбоната после твердофазной реэкстракции в атмосфере восстановительных газов аммиака и водорода позволяют получить двуокись урана пригодную для фторирования с получением тетра- и гексафторида урана.