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EXTRACTION PROPERTIES OF SYNTHESIZED FLUORINATED ORGANOPHOSPHORUS COMPOUNDS

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ABSTRACT

This work presents the results of studies of extraction properties in relation to the uranium of bis(2,2,2-trifluoroethyl)diallylamido phosphite, bis(2,2,2-trifluoroethyl)dimethylamido phosphite, and bis(N,N-dialamido)isopropyl phosphite synthesized by us earlier. Tests were carried out in a factory laboratory, as the initial solutions used nitrate solution prepared from uranium oxide (C_U = 10.30 g/dm^3, C_{HNO3} = 56.40 g/dm^3) and uranium sulfate solution prepared from the chemical concentrate of natural uranium and deoxidized with sulfuric acid (C_U = 10.60 g/dm^3, C_{H2SO4} = 25.10 g/dm^3). It was proved that the synthesized acyclic amido phosphites can effectively extract uranium from these technological solutions with a proper selection of technological parameters, and the degree of uranium extraction may vary from 34.43 to 95.94%.

Keywords: Amidophosphites, Polyfluoroalkyl, Allyl, Extractants, Extraction, Uranium-Containing Solutions, Uranium Extraction, Phosphorus Compounds.

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INTRODUCTION

Organic phosphorus compounds are being actively researched by leading scientists around the world due to the high reactivity of these compounds and the wide range of their practically useful properties.[1-18] Currently, tributyl phosphate is widely used in hydrometallurgy for the extraction of uranium from acidic production solutions, which forms a complex with uranyl salt that readily passes into the organic phase.[19] New possibilities for the determination and isolation of uranium have been opened by the appearance of organophosphorus extractants, including neutral or acid esters of phosphoric acids or their functional derivatives with amino groups.[20-23,] For example, bifunctional derivatives of amido phosphonates have shown high extraction efficiency with respect to uranium.[24] There is also data on the use of butyl-1-[N,N-bis(2-ethylhexyl)carbamoyl]nonyl phosphonic acid (DEHCNPB) in uranium extraction. This extracting molecule shows high affinity and selectivity for uranium(VI) versus the other elements present in phosphoric acid (Al, Ti, V, etc.).[25-28] The work[29] investigated the effect of amido phosphonate ligand (DEHCEBP) impregnated on the mesoporous silica surface on the selectivity and efficiency in the solid-phase extraction of uranium in acid solutions. It was shown that the synthesized materials were highly selective with respect to U compared to Fe and Mo. Such high efficiency and selectivity with respect to actinoids of such extractants are explained by the simultaneous presence in the structure of these compounds of the main nucleophilic centers of various "softness" and "hardness", such as oxygen, nitrogen, and phosphorus atoms.[30] Amidophosphites are of particular interest among phosphorus-containing compounds since the presence of a phosphorus atom in the trivalent state promotes the formation of complex compounds with soft metals.[31] Taking into account the current lack of systematic data on the synthesis and implementation in practice of principally new nitrogen-containing organophosphorus compounds, as well as the volume of mined minerals in Kazakhstan, the creation of domestic extractants of heavy metals with improved technical characteristics is of great importance in the development of the metallurgical complex. In this paper, we present the results of test studies on the

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extraction properties of some amidophosphites synthesized by us[32] in uranium extraction from nitric acid or sulfuric acid solutions of commercial desorbates of hydrometallurgical production in Kazakhstan.
The aim of these investigations was to study the extraction ability of amidophosphites with different functional groups (polyfluoroalkyl, allyl, and amide) in parameters as close to the production ones as possible.

EXPERIMENTAL

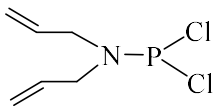
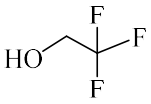
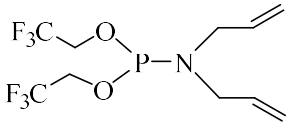
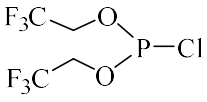
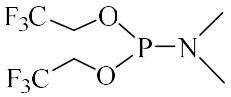
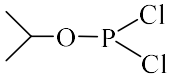
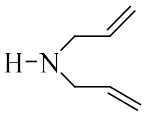
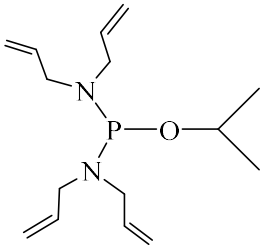
Reagents for Synthesis

2,2,2-Trifluoroethanol (> 99%), 2-propanol (≥99.5%), diallylamine (99%), dimethylamine (99%), dichloromethane (> 99.9%), phosphorus trichloride (>99%), triethylamine (≥99.5%), were commercial reagents prepared by Sigma Aldrich.

Synthesis Methods

The synthesis of a larger batch of target amidophosphites was carried out according to our previously proposed method[32] scaled up fivefold. Bis(2,2,2-trifluoroethyl)diallylamidophosphite 3a was synthesized from *N, N*-dialylamidodichlorophosphite 1a, and 2,2,2-trifluoro-1-ethanol. Bis(2,2,2-trifluoroethyl)dimethylamidophosphite 3b was obtained by the reaction of bis(2,2,2-trifluoroethyl)chlorophosphate 1b with dimethylamine 2b. The interaction of isopropyl dichlorophosphite 1c with *N, N*-dialylamine 2c gave bis(*N, N*-dialylamido)isopropylphosphite 3c (Table-1).
The starting *N, N*-diallilamidodichlorophosphite **1a** was obtained from *N, N*-diallilamine, and phosphorus(III) chloride according to our improved method.[33] Bis(2,2,2-trifluoroethyl)chlorophosphite **1b** was synthesized by interaction of bis(2,2,2-trifluoroethyl)phosphonate with phosphorus(V) chloride and isopropyl dichlorophosphite 1c was obtained by reaction of 2-propanol with phosphorus(III) chloride.[33]

Table-1: Synthesis of Amidophosphites 3a, b, and Diamidophosphite 3c

Starting reagents		Amidophosphites	Yield, %
 **1a**	 **2a**	 **3a**	60
 **1b**	 **2b**	 **3b**	42
 **1c**	 **2c**	 **3c**	73

The obtained starting and target compounds were identified by NMR and IR spectroscopy on Bruker DPX 400, Bruker AV-400, and Bruker IFS 25 spectrometers. The spectral characteristics of the synthesized compounds 1a-c, and 3a-c are identical to those in the literature.[32]

Materials and Methods of Extraction Experiments

The synthesized bis(2,2,2-trifluoroethyl)diallylamido phosphite (3a), bis(2,2,2-trifluoroethyl)dimethylamido phosphite (3b), bis(*N, N*-dialylamido)isopropyl phosphite (3c) were used as extractants. The study of the extraction properties of the synthesized amidophosphites 3a-c with respect to uranium was carried out under the conditions of the factory laboratory in two regimes. The first regime is uranium extraction from a nitric acid solution, and in the second regime, the extraction of uranium was

carried out from a sulfuric acid solution. Uranium oxide (U_3O_8) was used to prepare the nitric acid solution; the content of uranium and acid in the prepared nitric acid solution is U - 10.3 g/dm^3, HNO_3 - 56.4 g/dm^3. A chemical concentrate of natural uranium (CCNU) was used to prepare the sulfuric acid solution; the content of uranium and acid in the prepared sulfuric acid solution is U - 10.6 g/dm^3; H_2SO_4 - 25.1 g/dm^3. The procedure for extracting uranium was as follows: a measured amount of synthesized extractant and prepared uranium-containing solution was poured into an extraction vessel, and mixed with a magnetic stirrer. The internal temperature was 23°C, contact time of phases was 20 minutes. The ratio of the volumes of the aqueous and organic phases was kept at 1: 10. After the specified time, the phases were drained into a separating funnel to separate the organic phase from the aqueous phase. The volumes, color, and nature of the phases were determined. The uranium concentration was determined according to the method.[34]

Extraction efficiency was evaluated by the degree of metal extraction by the extractant, which was determined by the formula:

$$\%E = \frac{100D}{D+(V/V\prime)},$$

Where D - partition coefficient is determined by the ratio of the concentration of the extracting agent in the organic phase to the concentration of the substance in the aqueous phase after the occurrence of equilibrium (C/C'), and V and V' - the volume of the aqueous and organic phases, respectively.

RESULTS AND DISCUSSION

The results of the successful testing of larger batches of the synthesized amidophosphites for their extraction properties with respect to uranium under plant laboratory conditions are presented below. Testing the extraction activity of bis(2,2,2-trifluoroethyl)diallylamidophosphite 3a. During the extraction of uranium from nitric acid solution with amidophosphite 3a the following was observed: the color of the organic phase turned bright yellow, the boundary of phase separation was clear and pronounced, and emulsion formation was not observed. The degree of uranium extraction from the nitric acid solution by the studied extractant was 94.60 %. The uranium content in the organic phase and in the mother liquor was 95.78 and 0.59 g/dm^3 respectively. The same extractant for the extraction of uranium from sulfuric acid solution showed slightly lower efficiency, the degree of uranium extraction was 83.69%. The uranium content in the organic phase and mother liquor were 136.00 and 1.06 g/dm^3 respectively (Table-2).

Table-2: The Composition of the Aqueous and Organic Phases in the Extraction of Uranium from Nitrate and Sulfate Solutions with Amidophosphite 3a

The aqueous phase of the solution			The organic phase of the solution		Uranium extraction efficiency from the solution, %
U, g/dm^3	HNO_3, g/dm^3	V, cm^3	U, g/dm^3	V, cm^3	
0.59	56.40	35.20	95.78	≈ 3.80	94.60
U, g/dm^3	H_2SO_4, g/dm^3	V, cm^3	U, g/dm^3	V, cm^3	83.69
1.06	25.10	55.00	136.00	2.20	

Testing the extraction activity of bis(2,2,2-trifluoroethyl)dimethylamidophosphite 3b. When uranium was extracted from the nitric acid solution of amidophosphite 3b, a change in the color of the organic phase from colorless to yellow was observed, and the third phase was not formed A similar picture is obtained when uranium is extracted from a sulfuric acid solution. Studies have shown that this extractant has a high extractability with respect to uranium, the degree of extraction of uranium from a nitric acid solution was 92.64%. At the same time, uranium in the organic phase was contained in an amount of 93.54 g/dm^3, and in the mother liquor – 0.77 g/dm^3 (Table-3). The process of uranium extraction by amidophosphite 3b from sulfuric acid solution was also quite effective; the degree of uranium extraction, in this case, was 86.24 %. The content of uranium in the organic phase and in the mother, liquor was 126.10 g/dm^3 and 1.36 g/dm^3, respectively (Table-3).

Testing the extraction activity of bis(*N, N*-diallamido)isopropylphosphite 3c. During the interaction of the extractant 3c with acidic uranium-containing solutions there was a stratification of the system into two phases, without the formation of a third phase, and the boundary of phase separation was distinct and the

color of the organic phase turned bright yellow. The results showed that amidophosphite 3c can extract uranium with high efficiency from both nitric and sulfuric acid solutions. The degree of uranium extraction from nitric acid solution was - 95.94 %. The content of uranium in the organic phase and in the mother liquor is 96.89 g/dm^3 and 0.42 g/dm^3 respectively. The degree of uranium extraction from the sulfuric acid solution was 34.43%. The uranium content in the organic phase and in the mother, liquor was 60.00 and 6.60 g/dm^3 respectively (Table-4).

Table -3: The Composition of the Aqueous and Organic Phases during the Extraction of Uranium from Nitrate and Sulfate Solutions with Amidophosphite 3b

The aqueous phase of the solution			The organic phase of the solution	
U, g/dm^3	HNO_3, g/dm^3	V, cm^3	U, g/dm^3	V, cm^3
0.77	56.40	44.30	93.54	≈ 3.80
U, g/dm^3	H_2SO_4, g/dm^3	V, cm^3	U, g/dm^3	V, cm^3
1.36	25.10	34.00	126.10	2.30

Table-4: The Composition of the Aqueous and Organic Phases during the Extraction of Uranium from Nitrate and Sulfate Solutions with Amidophosphite 3c

The aqueous phase of the solution			The organic phase of the solution	
U, g/dm^3	HNO_3, g/dm^3	V, cm^3	U, g/dm^3	V, cm^3
0.42	56.40	48.70	96.89	≈ 5.00
U, g/dm^3	H_2SO_4, g/dm^3	V, cm^3	U, g/dm^3	V, cm^3
6.60	25.10	52.00	60.10	3.00

On the basis of experimental data, diagrams of the dependence of the degree of extraction and the distribution coefficient on the nature of acidity of the uranium-containing solution are prepared (Fig.-1 and 2). Testing of the extraction properties of the synthesized acyclic amidophosphites led to the following results. Amidophosphite, which does not contain a fluoroalkyl group, showed remarkable extraction abilities in the nitric acid extraction of uranium (about 95.94%), whereas with sulfuric acid extraction, the efficiency of the extractant sharply decreased (only 34.43%) (Fig.-1). The introduction of fluoroalkyl groups into the composition of amidophosphites increases the stability of the extractant in uranium-containing sulfuric acid solutions. For example, the use of amidophosphites containing fluoroalkyl groups during the extraction of uranium from sulfuric acid solutions of commercial desorbate increased the degree of extraction to 83.69%, while at the same time, the efficiency of extraction from nitric acid uranium-containing solutions remained at the same level (up to 94.60%). In general, the recovery efficiency of uranium from nitric acid solutions is higher than from sulfuric acid solutions for all three amidophosphites used in the test (Fig.-1).

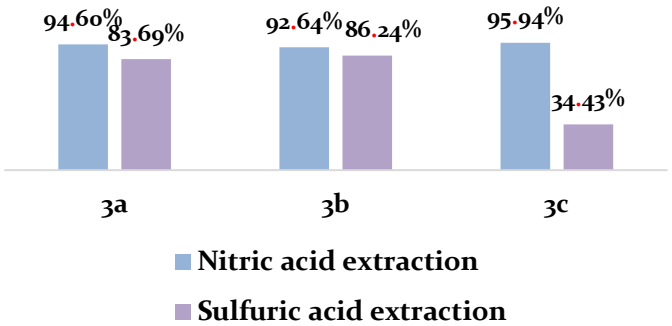


Fig.-1: Uranium Extraction Degree E % Diagram for Compounds (3a-c)

Figure-2 shows the values of distribution coefficients of uranium between the organic and aqueous phases during extraction from nitric acid and sulfuric acid solutions. During the extraction of uranium from the solution prepared from U_3O_8 the distribution coefficients are significantly higher than during the extraction of uranium from the solution prepared from CCNU. The distribution coefficient reaches its

maximum value during the extraction of uranium with amidophosphite 3c as the extracting agent from nitric acid solution (230.6) (Fig.-2).

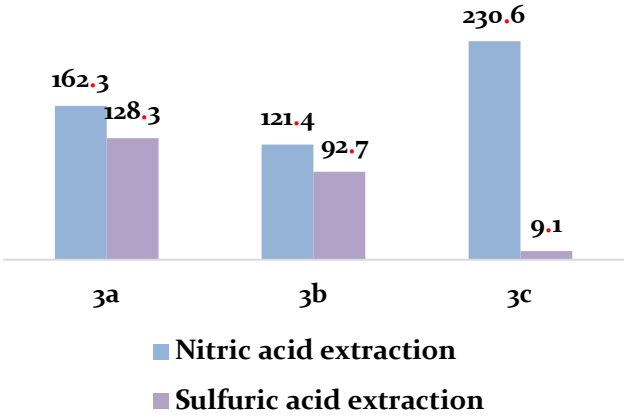


Fig.-2: Uranium Partition Coefficient D_U Diagram for Compounds (3a-c)

It should be added that the technological application of compounds (3a-c) is not difficult in any way, since they do not dissolve in these acidic uranium-containing solutions, are characterized by a clear, pronounced separation boundary of the organic and aqueous phases, and the absence of the formation of an emulsion (third phase) during the extraction process. The extractability of the reagents depends to a large extent on the nature of their substituents (polyfluoroalkyl, allyl, and amide groups). The synthesized amidophosphites have high extraction characteristics apparently due to the presence of various centers of "hardness" and "softness" (phosphorus, nitrogen, oxygen), which contribute to the formation of complex compounds with uranyl ions. In addition, the presence of double bonds causes the formation of additional sigma and π-complexes, which also leads to an increase in the extraction ability of amidophosphites. The introduction of polyfluoroalkyl groups increases the lipophilicity and hydrophobicity of the extractants due to the change in their hydrophilic-hydrophobic balance. At the same time, the noncombustibility of the extractants increases, which allows the use of flammable organic solvents as diluents. With the prevalence of acidic uranium extraction in the hydrometallurgical industry of Kazakhstan, the ability of obtained extractants to work in sulfuric, nitric, and chlorine-acid solutions is an undeniable advantage, while the efficiency of commercial extractants is greatly reduced in more acidic industrial uranium-containing solutions. Based on the results presented, the innovative extractants developed by us can be recommended for use and find wide application in the extraction processes of extraction, concentration, and separation of metals - uranium and associated metals in the hydrometallurgical industry not only in Kazakhstan but also in other countries. Further research will focus on the study of selectivity and the impact of varying the parameters of the recovery process and the separation of metals on the extraction ability of these compounds.

CONCLUSION

The results of our studies have shown that all acyclic amidophosphites bis(2,2,2-trifluoroethyl)diallylamidophosphite 1, bis(2,2,2-trifluoroethyl)dimethylamidophosphite 2, bis(*N, N*-diallamido)isopropylphosphite 3 show high efficiency in the process of uranium extraction from nitric acid or sulfuric acid solutions of Kazakhstan hydrometallurgy. The degree of extraction in relation to uranium varies from 35.2 to 96.3%.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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