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**ABSTRACT**

of the dissertation for the degree of Doctor of Philosophy

**SYNTHESIS OF HIGH PURITY AMMONIUM SALTS FOR  
LOW BACKGROUND MEASUREMENTS, SORPTION OF  
HEAVY METALS FROM SALT SOLUTIONS ON ION  
EXCHANGE ELASTOMERS**

Speciality: 2305.01 – Nuclear chemistry

Field of science: Chemistry

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## GENERAL CHARACTERISTICS OF THE WORK

**Relevance of the topic and degree of development.** Pioneering laboratories built for low-background measurements intensively carried out fundamental physical research: the search for dark matter, the study of neutrinoless double beta decay, neutrino registration etc. Some of these experiment are CUPID-Mo, which studies the double beta decay on molybdenum crystals and the EDELWEISS experiments studies the search for dark matter in an underground laboratory in Modane, France.<sup>1,2</sup> The level of radioactive background is the main indicator for experiments studying rare natural phenomena. In these experiments, various ways are sought to weaken or even eliminate each of the sources cause the formation of the radioactive background. One of the most important components of successful research in this field is the acquisition of ultrapure substances - materials purified from possible radioactive impurities and the use of highly purified substances in further research.

Fluxes are often used in low-background experiments in the manufacture of electronic devices. Literature data indicates radioactive isotopes of the elements U, Th, and K, which pose problems for low-background studies, have been found in commercially available solders.<sup>3</sup> Therefore, it is an actual issue to determine the existing mixtures in trace amounts in the composition of flux materials and to develop a methodology for purchasing fluxes in accordance with the requirements for conducting low-background experiments (the amount of radioactive mixtures should not exceed 1  $\mu\text{Bq/kg}$  of composition).

There is also a great need for ultrapure substances in the field of nuclear medicine. Nowadays, diagnostic and therapeutic procedures

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<sup>1</sup> Armengaud E. Performance of the EDELWEISS-III experiment for direct dark matter searches // E. Armengaud, Q. Arnaud, C. Augier, et al. JINST. - 2017. - vol. 12. - № 8. - p. 08010

<sup>2</sup> Armengaud E. The CUPID-Mo experiment for neutrinoless double-beta decay: performance and prospects // E. Armengaud, C. Augier, AS. Barabash et.al. Eur. Phys. J. -2020. C 80:44, p 1-15.

<sup>3</sup> Akerib D.S. Production of solder and flux with low radioactivity // D.S, Akerib, A. D. Silva, D.L. Herli et al. Nucl.Instrum. Meth. -1997. vol 400, issue 1, p. 181-183.

using radiopharmaceuticals are being improved and widely applied. Irradiation of tumors using external radiation sources does not show sufficient selectivity in the destruction of cancer cells, moreover has a cytotoxic effect. "Target therapy and diagnostics" fields of nuclear medicine are being actively developed in order to ensure delivery of the radioisotope directly to tumor cells. The part responsible for the targeted delivery of the radiopharmaceutical to the cell is called "Vector", the part that ensures the connection of the radioisotope with the active site is called "Chelator" and the part responsible for the destruction of the cell is called "Nuclide". In order to bind the radionuclide with the peptide chelator, in most cases buffer salt solutions are used. In order to obtain a radiopharmaceutical with high radioactivity, salt solutions containing minimum metal cations are needed.

Ammonium salts can be used in the processes of soldering the details of electronic circuit devices used in low-background measurements as well as in synthesis of radiopharmaceuticals in the field of nuclear medicine. Ammonium salt solutions used in the vector part of the radiopharmaceutical are biologically and chemically harmless due to their compatibility and easy excretion in the human body.

In general, the physical and chemical properties of impurities are close to the original substance they contain, otherwise they would be easily separated in a process of purification of the original substance. Purification of any substance from micro-mixtures is a complex chemical challenge. The use of special purification methods is required for production of high-purity substances to reduce the amount of metal mixtures from  $10^{-6}$  by at least one order of magnitude. Heating devices are used to purify salts using traditional distillation and cooling crystallization methods. This may cause the material being purified to decompose due to temperature. Sorption or mechanical capture of mixed ions and molecules separated by crystals of the main substance during the purification of salts by the method of cooling crystallization is also not excluded. Therefore, it would be crucial to develop the method of synthesis of salts from partially volatile compounds. High purity precursors can be obtained by sub-distillation. It is possible to

synthesize such precursors from ammonia with chloride or acetic acid. To study the models of multi-element sorption in salt solutions on adsorbents is very important in order to allow for high-order purification of ammonium salts from radionuclide and other heavy metal mixtures.

**Object and subject of the research.** Synthesized ammonium chloride and ammonium acetate salts of special purity, which are used as a medium in nuclear medicine and as a solder in low-background research, were taken as the research object. The subject of the dissertation is research of contaminations in ammonium chloride and ammonium acetate salts, reagents used for their synthesis, in the materials (systems) of containers used during synthesis and for storing substances by radioisotope and elemental analysis methods.

**Goals and objectives of the dissertation work.** The aim of the dissertation work is to obtain ammonium chloride and ammonium acetate salts of high-purity, which are buffer components for nuclear medicine and low-background research, to study the sorption behaviour of mixtures in salt media on cation-exchange and anion-exchange sorbents.

**In order to achieve the goal of the dissertation work was planned to implement the following:**

- analysis of reagents and materials used in the dissertation work by various spectrometric methods;
- purification of hydrochloric acid from radioactive and other heavy metal mixtures, determination of metal micromixtures in acid by mass and atomic emission spectrometer;
- purification of acetic acid from radioactive and other heavy metal mixtures, determination of metal micromixtures in acid by neutron activation analysis;
- synthesis of ammonium chloride salt and analysis of the synthesized compound by various spectrometric methods;
- synthesis of ammonium acetate salt and analysis of the synthesized compound by various spectrometric methods;
- determination of the distribution coefficient of 60 elements in the ammonium chloride solution on the ion-exchange sorbent;

- determination of distribution coefficient of 60 elements in ammonium acetate solution on ion-exchange sorbent.

### **Research methods:**

Primary precursors were purified from radioactive and other heavy metal mixtures by the sub-distillation method. The speed of purification was regulated as 5 ml/h. The method of synthesis of ammonium salts consists of purification of hydrochloric and acetic acids, transfer of ammonia gas through the acid (bubbling), evaporation and crystallization of the saturated salt solution under vacuum conditions and at room temperature.

An inductively plasma mass spectrometer and an inductively plasma atomic emission spectrometer were used to estimate the element composition of the chemical reagents used in the dissertation work. The spectrometers are capable of determining more than 60 elements in the range of 0.1 – 10,000 ng/l.

Neutron activation analysis was performed in a research reactor with a nominal power of 10 MW and using enriched uranium-235 (19.7%) fuel at the Institute of Nuclear Physics of the National Academy of Sciences of Uzbekistan. The maximum neutron density in the experimental channel: for fast neutrons ( $E_n = 0.821$  MeV) in the active zone -  $5 \times 10^{13}$  neutrons / ( $\text{cm}^2 \times \text{sec}$ ), for thermal neutrons in the reflector -  $1.2 \times 10^{14}$  neutrons / ( $\text{cm}^2 \times \text{sec}$ ). Samples and standards were irradiated for 5 h. The irradiated samples were analyzed by gamma spectrometer within 1 hour.

A 210  $\text{cm}^3$  coaxial detector "Gentiane" was used for low background measurements. The sensitivity of the detector makes it possible to measure activity of U/Th elements below 1 mBq. The registration efficiency was ensured using Monte Carlo method. The measurement of samples continued within several months.

As a model system for comparative analysis the distribution coefficients study using the solutions prepared on the basis of multi-element standards, the content of radionuclides in which is artificially controlled and Dowex sorbents added to these solutions, was employed. 2 ml of each standard (radionuclidic solution) was taken and evaporated until pure dry salt was obtained. Dry salted standards were dissolved in high purity ammonium salt solutions and were used for

comparative analysis. 1 ml of the obtained solutions was added to 50 mg of cation or 50 mg of anion-exchange Dowex sorbent. The samples were mixed by shaking for 24 h in order to achieve sorbent and solution equilibrium. After reaching equilibrium, 0.5 ml of each sample was taken and diluted to 5 ml then elemental contents were analyzed by inductive plasma mass spectroscopy (ICP-MS) and inductive plasma atomic emission spectroscopy (ICP-AES) methods.

### **Basic provisions for protection**

- estimation of the purity of the primary precursor and plastic materials used in the dissertation work;
- estimation of the purification of mixtures of radioactive and other metals mixtures from chloride and acetic acid;
- estimation of the purity of the synthesized ammonium chloride salt;
- estimation of the purity of the synthesized ammonium acetate salt;
- study of adsorption of elements from ammonium chloride salt solution on ion-exchange sorbent;
- study of adsorption of elements from ammonium acetate salt solution on ion-exchange sorbent.

### **Scientific novelty of the research:**

- the method of production of high-purity ammonium chloride and ammonium acetate salts was developed;
- the possibility of using the synthesized high-purity ammonium chloride and ammonium acetate salts as a fluxing reagent for low-background experiments was estimated by different spectroscopic methods;
- the possibility of using synthesized high-purity ammonium chloride and ammonium acetate salt solutions as a buffer substance for the production of radiopharmaceuticals was estimated by different spectroscopic methods;
- for the first time, distribution coefficients of 60 elements on ion-exchange sorbents at different concentrations of ammonium chloride solution were determined.
- for the first time, distribution coefficients of 60 elements on ion-exchange sorbents at different concentrations of ammonium acetate

solution were determined.

**Theoretical and practical significance of the research.** The obtained results stimulate to enrich the important information base on such fields as radiochemistry, nuclear medicine, medical chemistry, nuclear chemistry, ecology, geology, electronics, radiation materials science.

The results of scientific research can be used in the following areas:

- produced high-purity ammonium salts can be used as fluxing material for soldering details of electronic devices used in low-background measurements and a buffer solution when chelating radionuclide in the field of nuclear medicine.

The results obtained by studying the sorption of numerous elements from ammonium salt solutions on ion-exchange adsorbents can be used for the purification of ammonium chloride and ammonium acetate salts by chromatographic methods, for the separation of various radionuclides and the acquisition of high-purity substances to be used in low-background experiments.

**Approval and implementation of work.** The results of the dissertation work were reported and discussed at the following international conferences and published in the materials of those conferences:

IX Российская конференция с международным участием. Радиохимия 2018. 17-21 сентября 2018 г., Санкт-Петербург, Российская Федерация.

LXIX International conference NUCLEUS-2019 on nuclear spectroscopy and nuclear structure 1–5 July 2019. Dubna, Russia.

LXX International Conference NUCLEUS-2020. 12 – 17 October 2020, Saint Petersburg, Russia.

LXXII International Conference «NUCLEUS-2022» 11 – 16 July 2022, Moscow, Russia.

X Российская конференция с международным участием Радиохимия 2022. 26-30 сентября 2022 г., Санкт - Петербург, Российская Федерация.

**Publications:** The results of the research presented in 12 scientific papers (7 articles and 5 conference materials) were published



in foreign and domestic scientific journals and conference materials.

**Name of the organization the dissertation work was performed:**

The dissertation work was carried out in 2017-2022 at the Institute of Radiation Problems of the Ministry of Science and Education of the Republic of Azerbaijan and at the Joint Institute for Nuclear Research in Dubna, Russian Federation.

**Structure and scope of work:** The presented dissertation consists of 28751 symbols for the introduction, 50935 symbols for chapter I, 36378 symbols for chapter II, 42635 symbols for chapter III, 42269 symbols for chapter IV and 2665 symbols for the results, a total of 203633 symbols. The dissertation consists of 162 pages, of which 101 pages of text, 39 images and graphs, including 16 tables.

**Author's personal contribution** consists of: Search and analysis of literature on the research topic, research planning, making the "Clean Room" ready for use ; carrying out of experimental work - synthesis of ammonium salts, analysis of the content of metals and radionuclides in primary precursors and ammonium salts using low-background gamma spectrometry, neutron activation analysis, mass and atomic emission spectrometry, study of adsorption of 60 elements from ammonium chloride and ammonium acetate solutions on cation and anion exchange sorbents.

## **MAIN CONTENT OF THE RESEARCH:**

**The introduction part included:** the relevance of the dissertation work, the importance of conducted systematic studies and planned experiments in order to achieve a defined goal, the innovations of scientific results, the experimental and practical significance of the work, the methods used in the dissertation work, important provisions, approval of the work, the personal contribution of the author and the brief content of the chapters.

**The first chapter** provides an overview of published scientific literature about materials on the classification of chemical substances according to their purity, methods of distribution and determination of

radioactive mixtures in various substances, methods of purification of substances from radionuclides and other heavy metal mixtures, production of ammonium salts and scope of their application.

An analysis of the scientific literature reports that the amount of radioactive elements (U, Th, etc.), di, tri and tetravalent metals mixtures in the substances used in low-background experiments and in the processes of production of radiopharmaceuticals is of utmost importance<sup>4</sup>. The first chapter shows that high-purity reagents available from the commercial network do not meet the requirements of low-background experiments and nuclear medicine. In the chapter, the importance of mass and gamma spectrometry, neutron activation analysis used for low-background measurements in the determination of mixtures in the substance, the sensitivity of the devices, the limit of determination, the comparative analysis of the minimum quantities that can be determined of the mixtures were carried out.

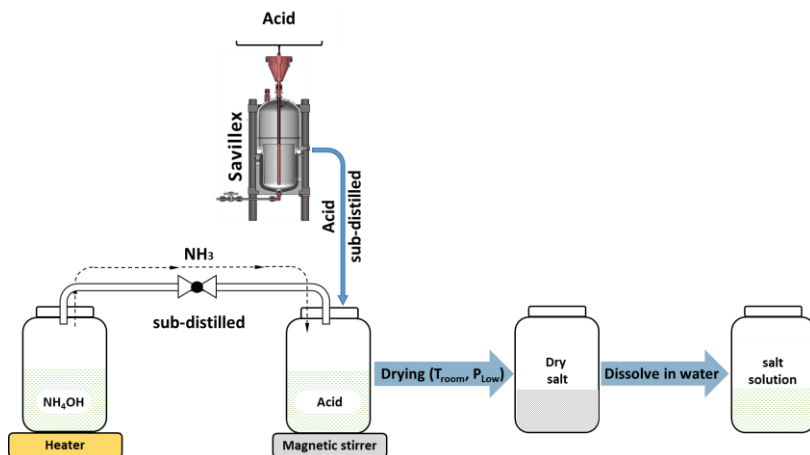
A study of scientific literature materials about “the study of methods for purifying substances from radionuclides and other mixtures of heavy metals” and “the production of ammonium salts” shows that although the determination of some parameters in the production of ammonium salts has been patented, methods for its production of high purity have not been systematically studied. Therefore, the synthesis of ammonium salts that meet high purity standards for low-background experiments and nuclear medicine is still an urgent problem.

**The second chapter** describes in detail the experiments and the synthesis method of ammonium salts. The synthesis of ammonium salts was carried out in a "Clean Room" built in order to minimize the presence of extraneous polluting factors in the experimental zone at the Joint Institute for Nuclear Research in Dubna, Russian Federation. The synthesis process includes stages such as purification of hydrochloric and acetic acids, obtaining saturated solutions of ammonium chloride and ammonium acetate salts by reaction of acid residues with ammonia, obtaining solid ammonium salts under vacuum conditions

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<sup>4</sup> Irina V. The importance of high specific radioactivity in the performance of <sup>68</sup>Ga-labeled peptide // V. Irina, G.J. Beyer, P. E. Bergström et al. Nucl. Med. Biol. 2008. vol. 35, p. 529–536.

and making up of salt solutions (Fig.1).



**Figure 1.** Scheme for the synthesis of ammonium salts in a clean room.

An inductive plasma mass spectrometer (X-7, Thermo Elemental, USA) and an atomic emission spectrometer (ICAP-6500, Thermo scientific, USA) were used to estimate the elemental composition of the samples in the dissertation work. Sample preparation was carried out at the Institute of High Purity Materials and Problems of Microelectronics Technologies (Chernogolovka, Russia). These spectrometers are capable of determining many elements (rare earth metals, U, Th, etc.) with a minimum detection limit of 0.1-5 ng/l, some elements (K) within 10-50  $\mu\text{g/l}$ , in total more than 60 elements. Samples were diluted using 5% nitric acid and deionized water. In addition, standard solution of indium was used to reduce the matrix effect of the spectrometer during sample preparation.

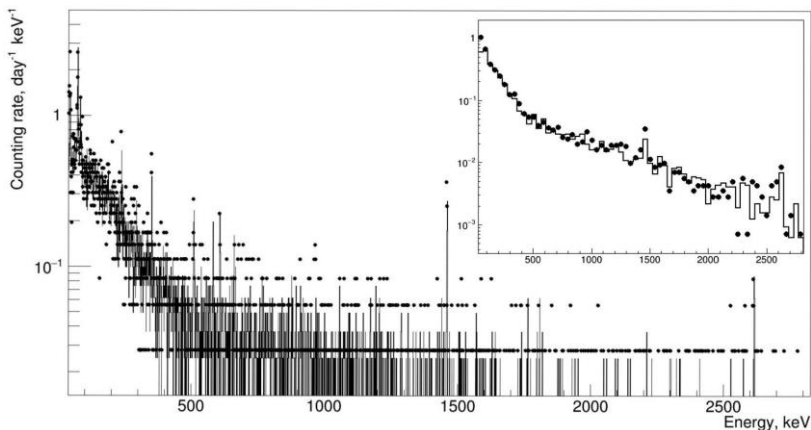
Elemental analysis of the samples by neutron activation method was carried out in the research reactor. Irradiation of the samples was carried out in the vertical channel of the reactor (WWR-SM) with a neutron flux of  $5 \times 10^{13} \text{ n}^0/\text{cm}^2 \times \text{s}$ . The samples to be analyzed together with the standards were wrapped in aluminum foils and placed in quartz ampoules. The ampoules were irradiated for 5 hours in the reactor in special metallic containers. The determination time of

radionuclides with a short half-life in samples and standard materials was 900 sec. and 100-600 sec. respectively. The determination time of radionuclides with a long half-life was 3600 sec. after three days of irradiation was analyzed using gamma spectrometer. Processing of spectra was carried out using Genie-2000 software.

Purity of ammonium chloride salt was estimated by performing analyzes in spectrometers used in low background experiments. The measurements made with the Gentiane detector- part of the ultra-low-background HPGe spectrometer of EDELWEISS experiment<sup>1</sup>, which studies rare natural phenomena in an underground laboratory (Modan, France). The natural radioactive background caused by the laboratory walls was screened due to active muon veto, polyethylene and lead sheets. The main radioactive background caused by natural  $\beta$  and  $\gamma$  rays was reset due to the natural capabilities of the germanium detector. The sensitivity of the detector makes it possible to measure the activities of elements of the U/Th series below 1 mBq. The amount of measured samples was taken as 50-100 grams. Before the spectrometric analysis of salts, the spectrometric background was measured and calibrated for 77.7 days. Gamma spectrometric analysis of ammonium chloride salt was performed for 34.6 days (Figure 2). The efficiency of recording was performed using the Monte Carlo method.

The method of determining the distribution coefficient of 60 elements on cation and anion-exchange sorbents at different concentrations of ammonium chloride and ammonium acetate solutions was developed. Multi-element standards were used in the development of the method. 2 ml of each standard was taken and evaporated until pure dry salt was obtained and diluted until 2 ml of 0.1 M ammonium solution was obtained. 0.25 ml of the initial solutions were taken in order to prepare 3 ml of ammonium solution of different concentrations. Separately, 1 ml of "mother" solution was added to 50 mg of cation or 50 mg of anion-exchange sorbent. As a result, each sample with ammonium solution contained mass of 100  $\mu\text{g}/\text{l}$  of multi-element standard. The samples were mixed by shaking for 24 h in order to achieve sorbent and solution equilibrium. After reaching equilibrium, 0.5 ml of each sample was taken and diluted to 5 ml with 0.1 ml of 5%

nitric acid and 0.05 ml of indium standard. The element composition of diluted samples was studied by ICP-MS and ICP-AES methods.



**Figure 2.** Comparison of the energy spectra of  $\text{NH}_4\text{Cl}$  salt, (dots) with the background (line), measured on a low-background gamma spectrometer synthesized in clean rooms. Same data with a wider bin ( $\sim 40$  keV) (Modan, France).

**In the third chapter,** the purity of the synthesized ammonium chloride and ammonium acetate salts, as well as the vessels used in the synthesis of these salts and the purified precursors, was estimated by low-background gamma-spectroscopy, INAA, ICP-MS, ICP-AES methods.

In order to estimate the purity of the plastic containers used for the purification and synthesis of substances in the thesis work, the amount of mixtures was analyzed by the INAA method. The activation products of light elements such as C, H, O, N, F, which are the basis of plastics, have a very low half-life and decay faster than the activation products of the investigated U and Th radionuclides. Therefore, the INAA method is an ideal method for determining Th and U elements in plastic matrices. The amount of Th in plastics was determined based on the 311.9 keV gamma lines of the  $^{233}\text{Pa}$  isotope with a half-life of 26.9 days, the amount of U

was determined based on the 228 keV gamma lines of the  $^{239}\text{Np}$  isotope with a half-life of 2.3 days. The maximum amount of uranium was determined in perfluoroalkoxy alkane plastic (PFA) material ( $4.3 \times 10^{-10}$  g/g), which corresponds to 5.3 mBq/kg activity, and the maximum amount of thorium was determined in polypropylene (PP) material ( $8.8 \times 10^{-10}$  g/g) which corresponds to an activity of 3.57 mBq / kg. In Nalgen brand polypropylene, K which is the main source of gamma background for low background experiments was several times higher than in other brands. It was noticeable that in both brands of PP samples, the amount of Na element was higher than in PFA material. The element Na can result in unnecessary reactions with various particles (e.g., alpha particles) at low energies, which can lead to false signals that can affect the background. The amount of Fe was approximately the same for the three brands of plastic. The amount of Zn was higher in PP than in PFA material. The amount of Fe and Zn elements is important in the substances used for the synthesis of radiopharmaceuticals. The amount of La in the PFA material was 10 times and the amount of Cs was approximately 5 times less than the PP material. The amount of Rb in "Apexlab" brand polypropylene was 5 times more than other brands.

The element composition of  $\text{NH}_4\text{OH}$  solution was determined by the INAA method. The amount of most elements in the solution was below the detection limit of the spectrometer. The amount of uranium ( $<1.20 \times 10^{-11}$  g/g) and thorium ( $<1.67 \times 10^{-11}$  g/g) in the solution was below the detection limit of the spectrometer, accordingly, the measured activity of the  $^{238}\text{U}$  isotope was 148  $\mu\text{Bq/kg}$ , the activity of  $^{235}\text{U}$  was 7  $\mu\text{Bq/kg}$ , the activity of  $^{232}\text{Th}$  was lower than 68  $\mu\text{Bq/kg}$  respectively. In order to minimize the amount of unnecessary heavy metal impurities such as Ag, Au, which can be activated by neutron, the solution was evaporated at a temperature below the boiling point ( $50^\circ\text{C}$ ).

The purity of deionized and bidistilled water used in the synthesis process was analyzed and estimated by ICP-MS and ICP-AES methods. The concentration of trace elements in water samples was below the detection limit of the mass spectrometer. The amount

of K, Th and U was  $> 8 \mu\text{g/l}$ ,  $> 2 \text{ ng/l}$ ,  $>0.4 \text{ ng/l}$  and their respective activities were  $^{40}\text{K}$ ;  $>248 \mu\text{Bq/l}$ ,  $^{232}\text{Th}$ ;  $>12 \mu\text{Bq/l}$ ,  $^{235}\text{U}$ ;  $> 0.2 \mu\text{Bq/l}$  and  $^{238}\text{U}$ ;  $> 4.93 \mu\text{Bq/l}$ . The use of the analyzed water was accepted in the experiments, which meets the purity requirements.

In the dissertation work, the purification of hydrochloric acid from radionuclides and other metal mixtures was estimated by ICP-MS and ICP-AES methods.

The acid was successively sub-distilled for 3 times. The amount of elements were approximately the same in all three periods. As a result of the use of the sub-distillation method, the amount of mixtures containing hydrochloric (OSCh RU GOST 20-4) acid has significantly decreased. After purification of the acid by sub-distillation, the amount of potassium decreased from  $199 \mu\text{g/l}$  to the minimum detection limit ( $< 8 \mu\text{g/l}$ ). The amount of uranium (from  $10 \text{ ng/l}$  to  $<0.4 \text{ ng/l}$ ) and thorium (from  $40 \text{ ng/l}$  to  $2 \text{ ng/l}$ ) also decreased to the minimum detection limit. The activity of  $^{235}\text{U}$  and  $^{232}\text{Th}$  for sub-distilled acid was  $4.93 \mu\text{Bq/l}$  and  $8.12 \mu\text{Bq/l}$ , respectively. The decrease in the concentration of elements such as Hf, Au, Be, Al, Cd and lanthanoids, as well as natural radioactive isotopes La, Rb, In, Co, etc. was recorded.

After the sub-distillation process, the amount of Fe, which hinders the production of radiopharmaceuticals with high specific activity, decreased by 54 times, Zn by 38 times, Cr by 17 times and Al by more than 25 times. According to the obtained result, the synthesis of ammonium chloride with acid purified by sub-distillation method is more suitable for the purpose.

The purification of acetic acid from trace metal impurities before and after sub-distillation method was estimated by INAA method (Table 1). The purification process was effective in the separation of metal impurities (U and K), which are the main background source of low-background experiments. The amount of K was decreased from  $1.8 \times 10^{-8} \text{ g/g}$  to  $2.0 \times 10^{-9} \text{ g/g}$ , the amount of uranium was decreased from  $5.8 \times 10^{-12} \text{ g/g}$  to  $3.4 \times 10^{-13} \text{ g/g}$ . A decrease in the amount of elements with natural radioactive isotopes, such as Co, La, Sm, Rb, which are the components of the radioactive background, was recorded as a result of purification by

the sub-distillation method. The amount of elements such as Hf and Au also dropped below the minimum detection limit. After purifying the acid by sub-distillation method, a decrease in the concentrations of metals such as Fe, Zn, Mo, Cr, which prevent the binding of radionuclides to chelator was recorded as well.

**Table 1.**  
**Elemental composition of acetic acid (RU GOST-61-75 and purified by sub-distillation) determined by the INAA method.**

Element	Radio-nuclide	Half-life	The energy of $\gamma$ lines keV	Amount and determination limit, g/g	
				CH <sub>3</sub> COOH RU GOST-61-75	CH <sub>3</sub> COOH Sub-distilled
As	<sup>76</sup> As	26.2 h	559.1	$2.8 \times 10^{-12}$	$2.7 \times 10^{-12}$
Au	<sup>198</sup> Au	2.6 d	411.8	$2.7 \times 10^{-14}$	$< 2.0 \times 10^{-14}$
Br	<sup>82</sup> Br	1.47 d	554.3	$1.0 \times 10^{-9}$	$3.4 \times 10^{-11}$
Ca	<sup>47</sup> Ca	4.54 d	1297.1	$9.7 \times 10^{-8}$	$< 2.3 \times 10^{-8}$
Co	<sup>60</sup> Co	5.22 year	1332.5	$1.8 \times 10^{-10}$	$1.2 \times 10^{-11}$
Cr	<sup>51</sup> Cr	27.7 d	320.1	$1.5 \times 10^{-8}$	$1.2 \times 10^{-10}$
Eu	<sup>152</sup> Eu	13.5 year	121.7	$4.9 \times 10^{-11}$	$< 1.0 \times 10^{-12}$
Fe	<sup>59</sup> Fe	44.5 d	1099.2	$4.5 \times 10^{-7}$	$3.5 \times 10^{-9}$
Hf	<sup>181</sup> Hf	42.4 d	133.0	$1.0 \times 10^{-11}$	$< 5.0 \times 10^{-12}$
K	<sup>42</sup> K	12.3 h	1524.6	$1.8 \times 10^{-8}$	$2.0 \times 10^{-9}$
La	<sup>140</sup> La	1.7 d	487.0	$2.4 \times 10^{-12}$	$8.5 \times 10^{-13}$
Mo	<sup>99</sup> Mo	2.75 d	140.5	$1.0 \times 10^{-9}$	$1.1 \times 10^{-11}$
Na	<sup>24</sup> Na	14.9 h	1368.6	$5.0 \times 10^{-7}$	$2.1 \times 10^{-9}$
Rb	<sup>86</sup> Rb	18.7 d	1076.6	$2.4 \times 10^{-11}$	$< 2.0 \times 10^{-11}$
Sb	<sup>122</sup> Sb	2.7 d	564.2	$7.5 \times 10^{-13}$	$1.0 \times 10^{-12}$
Se	<sup>75</sup> Se	119.7 d	279.5	$9.8 \times 10^{-9}$	$4.5 \times 10^{-9}$
Sm	<sup>153</sup> Sm	1.94 d	103.1	$2.2 \times 10^{-12}$	$5.2 \times 10^{-13}$
Th	<sup>233</sup> Pa	26.9 d	311.9	$< 1.5 \times 10^{-12}$	$< 1.5 \times 10^{-12}$
U	<sup>239</sup> Np	2.3 d	228.0	$5.8 \times 10^{-12}$	$3.4 \times 10^{-13}$
Zn	<sup>69m</sup> Zn	13.7 h	438.6	$4.6 \times 10^{-8}$	$6.0 \times 10^{-10}$

**Note:** “<” below detection limit.



Low background gamma spectroscopy, INAA ICP-MS and ICP-AES methods were used to estimate the purity of NH<sub>4</sub>Cl salt synthesized in a clean room. The obtained results were compared with the analogues of salts manufactured in the RF and USA. The level of radiation activity (contamination) of commercial available ammonium chloride salt produced in the Russian Federation was higher than the level of background radiation of the low-background gamma spectrometry. Determined rate of radiation by gamma spectrometry analysis of the sample was 5 times higher than the rate of radiation from the background.

**Table 2.**

**Low background gamma spectrometric analysis of synthesized ammonium chloride salt.**

Nuclide	$\gamma$ - lines, keV	Minimum detection limit (DL) of activity, mBq/kg	NH <sub>4</sub> Cl (synthesized)
Pb-210	46	91	< DL
Pb-212	239	2	< DL
Pb-214	242	10	< DL
Pb-214	352	5	< DL
Bi-214	609	5	< DL
Ac-228	911	5	< DL
K-40	1461	77	< DL
Bi-214	1765	10	< DL
Tl-208	2614	5	< DL

**Note:** “<” below detection limit.

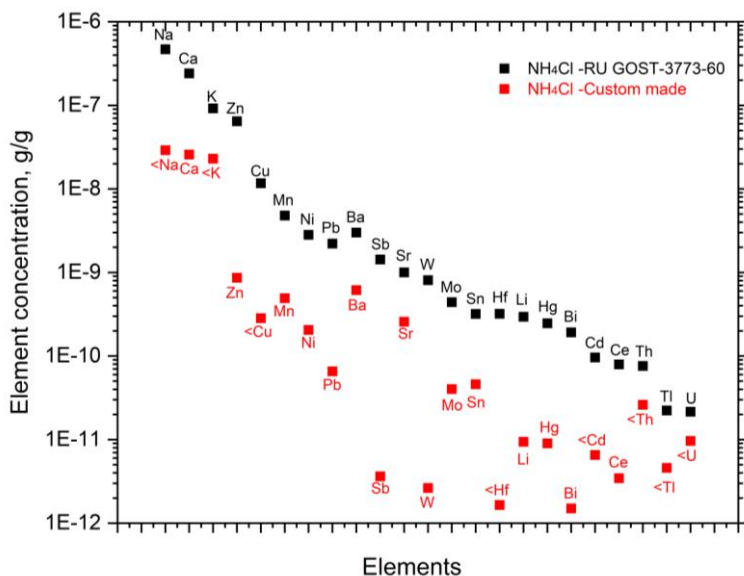
In the synthesized salt (Table 2), values above the internal background of the detector were not observed and the activity of radioisotopes was below the minimum detection limit. Therefore, it can be estimated that the purity of the synthesized salt has met to the requirements of low-background experiments.

The elemental composition of ammonium chloride salt synthesized in "clean room" conditions was studied by INAA method. According to the result, the amount of U, Th and K elements was below the minimum detection limit of the spectrometer. Thus, the amount of Th element was lower than  $1.0 \times 10^{-10}$  g/g, which corresponds to the 0.4 mBq/kg activity of Th - 232 isotope and the amount of U element was lower than  $1.0 \times 10^{-10}$  g/g, which corresponds to an activity of 1.2 mBq/kg. As a result, a high amount of any background element has not been determined.

Purity of synthesized ammonium chloride salt solution (5 M) and commercial analogue was analyzed by ICP-MS and ICP-AES method (Fig. 3). While comparing the element content of both solutions, it is noticeable that the salt solution synthesized in a clean room is cleaner than the commercial analogue produced in Russia.

According to the figure, the amount of elements such as Na, Ca, K, Zn in the salt solution obtained from the commercial network is the main quantity of the metal mixtures. An excessive decrease in the amount of these elements was observed in the solution obtained in the clean room. Although the amount of potassium, uranium, and thorium elements in the salt solution produced in Russia was determined, the amount of these elements in the salt solution synthesized in the clean room was below the minimum determination limit. K;  $< 23 \times 10^{-9}$  g/g, Th;  $< 2 \times 10^{-11}$  g/g, U;  $< 1 \times 10^{-11}$  g/g and activities respectively of  $^{40}\text{K}$ ;  $< 0,7$  mBq/kg,  $^{232}\text{Th}$ ;  $< 0,08$  mBq/kg and  $^{238}\text{U}$ ;  $< 0,1$  mBq/kg.

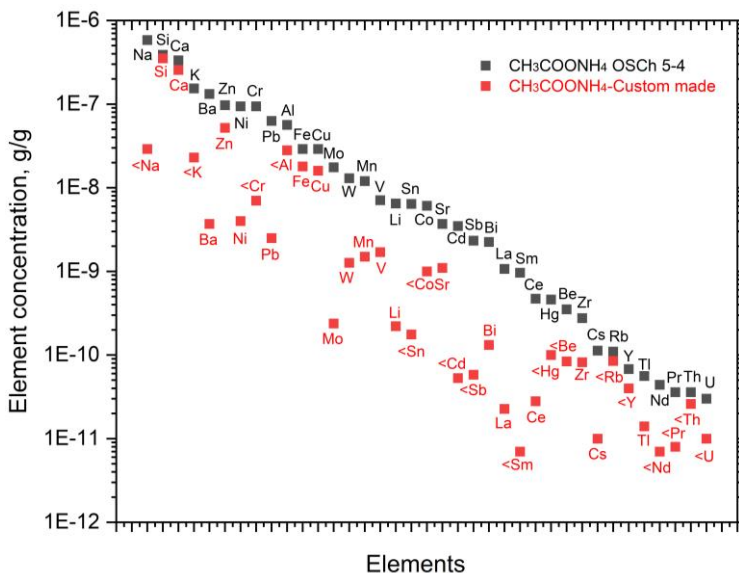
The concentration of information-carrying elements mainly related to tectonic changes, such as Sr and Pb, in the solution obtained in the "clean room" was lower than the commercial analogue. It is an indicator the purity of the natural isotope Pb-210 and the radioisotope Sr-90 in the solution obtained in the "clean room". In addition, a decrease in the amount of Cd and lanthanoids, which have a high neutron capture ability, was achieved.



**Figure 3.** Determination of elemental composition of NH<sub>4</sub>Cl salt solutions (5 M) by inductive plasma mass spectroscopy (ICP-MS) and inductive plasma atomic emission spectroscopy (ICP-AES). “<”- below detection limit.

Elemental composition of ammonium solution (5M) prepared from synthesized ammonium acetate salt in clean room was determined by ICP-MS and ICP-AES methods. In order to compare the obtained results, the element composition of the commercial analog (CH<sub>3</sub>COONH<sub>4</sub> - OSCH-5-4) was analyzed by the same method (Fig. 4). According to the figure, the ammonium acetate salt solution synthesized in the clean room was purer than the commercial analogue. The amount of U, Th and K elements in the salt solution produced in Russia was registered (K:  $154 \times 10^{-9}$  g/g, Th:  $3.6 \times 10^{-11}$  g/g, U;  $3 \times 10^{-11}$  g/g). In the solution synthesized in a clean room, the amount of these elements was lower than the minimum determination limit of the mass spectrometer K;  $< 23 \times 10^{-9}$  g/g, Th:  $< 2 \times 10^{-11}$  g/g, U;  $< 1 \times 10^{-11}$  g/g and

corresponding specific activities  $^{40}\text{K}$ ; < 0.7 mBq/kg,  $^{232}\text{Th}$ ; < 0.08 mBq/kg and  $^{238}\text{U}$ ; < 0.1 mBq/kg.



**Figure 4.** Determination of elemental composition of  $\text{CH}_3\text{COONH}_4$  salt solutions (5 M) by inductive plasma mass spectroscopy (ICP-MS) and inductive plasma atomic emission spectroscopy (ICP-AES). “<”- below the detection limit.

The low concentrations of elements such as Na, Pb, Cs, Co, Sr, etc. in solution indicator of the possibility of cleaning technogenic elements from salts. A decrease in the concentration of these elements indicates a decrease in their isotopes ( $\text{Pb-210}$ ,  $\text{Cs-137}$ ,  $\text{Co-60}$ ,  $\text{Sr-90}$ , etc.). The decrease in concentration of Na in the solution deserves a special attention. A decrease in the Na element is an indicator of an increase in the overall chemical purity of the salt. In addition, a decrease in the concentration of Cd and lanthanoids with high neutron capture properties was observed.

The amount of Fe, Zn, Ba, Pb, Sn, Al, Cr, Hf and many other elements of interest in the field of nuclear medicine in the ammonium acetate solution obtained in the "clean room" was lower than in the salt solution produced in the Russian Federation.

**In the fourth chapter**, the adsorption of different groups of elements at different concentrations of ammonium chloride and ammonium acetate solution on strong acid-based cation (Dowex 50W-X8) and strong alkali-based anion-exchange (Dowex 1-X8) sorbents was studied. This group of elements includes more than 60 elements of the group of alkali metals, alkaline earth metals, lanthanoids, transition metals, actinoids, platinum group transition metals, post-transition elements.

The distribution coefficient ( $K_d$ ) was determined by the following formula:

$$K_d = \frac{C_1}{C_2} = \frac{C_B - C_A / W}{C_A / V}$$

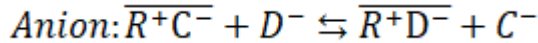
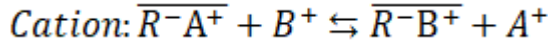
$C_1$  - concentration of elements in cation or anion-exchange sorbents,  $C_2$  - concentration of elements in ammonium solution.  $C_A$  - concentration of element in ammonium solution after equilibrium,  $C_B$  - the concentration of the element in the ammonium solution before equilibrium,  $W$  - weight of sorbent,  $V$  - volume of solution.

In ion-exchange processes, the mechanism between the ion-exchange sorbent and the solution is characterized by two principles:

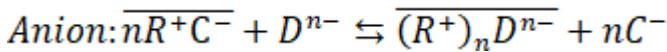
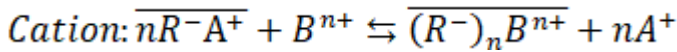
- the process is reversible;
- exchange reactions are based on equivalence and correspond to the principle of electro-neutrality.

The exchange process between the ion-exchange sorbent and the ions in the solution can be carried out only if the ion absorbed by the ion-exchange sorbent is different from the ion

desorbed from the sorbent. The exchange reaction of identically charged ions can be written as:



The exchange reaction of differently charged ions can be written as:



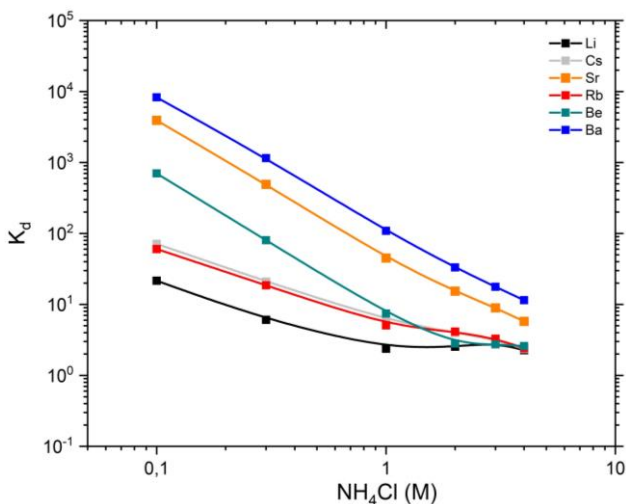
R – ion-exchanger; A+, B+, C– and D- are exchangeable ions.

The sorption of elements in solutions of ammonium chloride of various concentrations on cation and anion exchange sorbents are explained in detail in the following texts.

The decreases of distribution coefficient was observed as the concentration of ammonium ions increases for the alkali, alkaline-earth and rare earth elements on cation-exchange sorbent (Figure 5, 6). The reason for this trend was more diffused (or adsorbed)  $\text{NH}_4^+$  ions on the sorbent as the concentration of the solution increases.

As the size of the atomic radius of the elements with the same charge of the three mentioned groups increases, a partial increase in the distribution coefficient is observed. This pattern is related to the different hydration of ions.

The more the cations are hydrated, the less they are captured by the sorbent. So, highly hydrated ions migrate in the direction with more water molecules, from the sorbent to the solution.



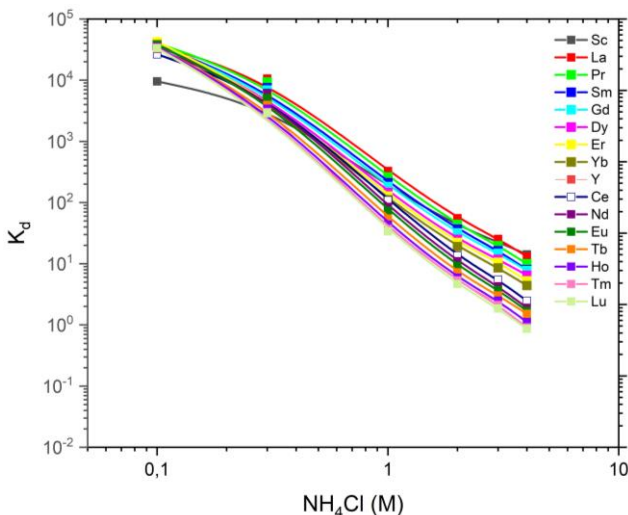
**Figure 5.** Distribution coefficient ( $K_d$ ) of alkaline and alkaline earth metals on cation-exchange sorbent (Dowex 50W-X8  $\text{NH}_4^+$ -form) at different concentrations (0.1, 0.3, 1, 2, 3 and 4 M) of  $\text{NH}_4\text{Cl}$  solution.

For example, Li hydrates more than other alkali metals and the size of the hydrated ion is larger than other cations. Therefore, it has less adsorption capacity than other alkali metals. In the group of alkaline-earth metals, this trend and determined distribution coefficients change like  $\text{Be} < \text{Sr} < \text{Ba}$ . The specified group of elements are not sorbed by the anion-exchange sorbent. They are present in the solution only in the form of positive ions.

The ionic radius of the rare earth metals are very close to each other and they have almost the same sorption values. Therefore, it is very difficult to separate the elements of this group. The values of the distribution coefficients in the  $10^5$  order of magnitude in solid solutions for rare earth elements were calculated.

The distribution of unequally charged ions in the cation-exchange sorbent depends on the concentration of the solution. The diluter the solution stands, the more highly charged ions are

exchanged with the cation-exchange sorbent. A doubly-charged ion entering the cation-exchange sorbent sends two singly-charged ions (or a triply- charged ion sends three singly charged ions) back into the solution, and the diluter the solution, the more likely this exchange is to occur. This trend is called electroselectivity. Electroselectivity in a given system varies as: rare earth metals > alkaline-earth metals > alkali metals.

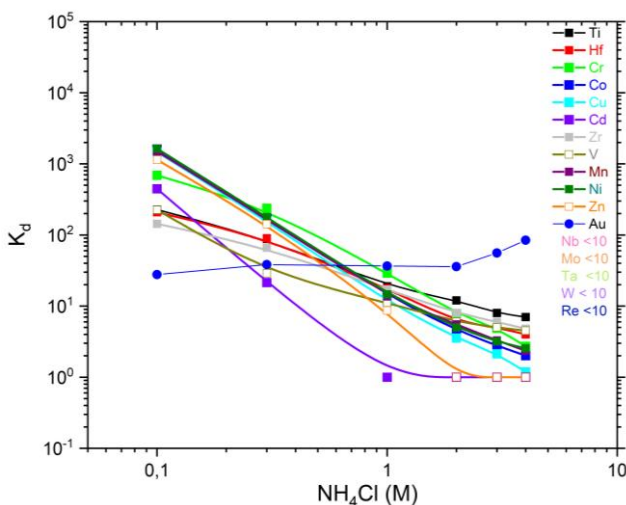


**Figure 6.** Distribution coefficient ( $K_d$ ) of rare earth metals on cation-exchange sorbent (Dowex 50W-X8  $\text{NH}_4^+$ - form) at different concentrations (0.1, 0.3, 1, 2, 3 and 4 M) of  $\text{NH}_4\text{Cl}$  solution.

For most transition metals, the value of the distribution coefficient in the cation-exchange sorbent decreases with the increase in the concentration of the ammonium chloride solution (Figure 7). This is mainly manifested in divalent and trivalent elements. Elements with 4, 5, 6 and 7 valences are relatively weakly adsorbed on the sorbent and the value of the distribution coefficient of most of them is lower than 10. Mo and V elements form chlorides which quickly hydrolyzed in chlorinated solutions,



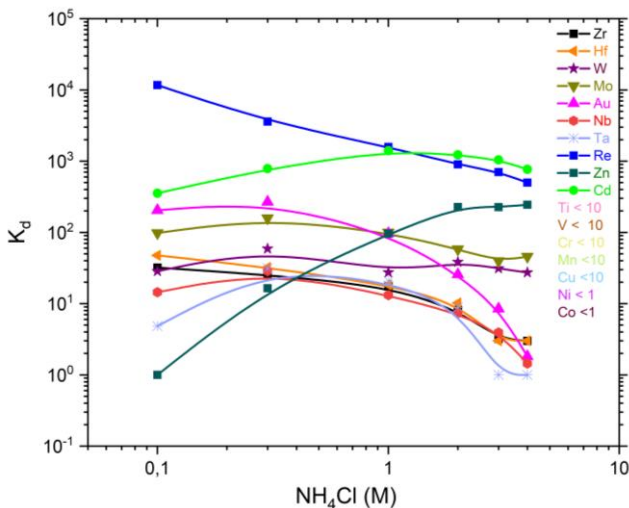
so chlorides weaken the adsorption of these elements to the sorbent. Cr (III) forms a +2 charged ion in an aqueous solution of ammonium chloride and it behaves like the elements of the second group. Relatively weak adsorption of Re element on cation-exchange sorbent is explained by the formation of coordination compound  $[\text{NH}_4][\text{ReO}_4]$ . Zn and Cd elements form +2 charged hydrocations  $\text{Me}(\text{OH}_2)_6^{2+}$  and positive  $\text{Me}^{2+}$ ,  $[\text{MeCl}]^+$  ions in aqueous solutions.



**Figure 7.** Distribution coefficient ( $K_d$ ) of transition metals on cation-exchange sorbent (Dowex 50W-X8  $\text{NH}_4^+$ - form) at different concentrations (0.1, 0.3, 1, 2, 3 and 4 M) of  $\text{NH}_4\text{Cl}$  solution.

The weak adsorption of 2 and 3 valent elements was observed on anion-exchange sorbent at the solution, on the other hand the adsorption of Zn and Cd elements on the anion-exchange sorbent increases as the concentration of the solution increases (Figure 8). This is due to their formation of stable  $[\text{MeCl}_3]^-$  and  $[\text{MeCl}_4]^{2-}$  anions in solid ammonium chloride solution. Adsorption of Au element on anion-exchange sorbent was 10

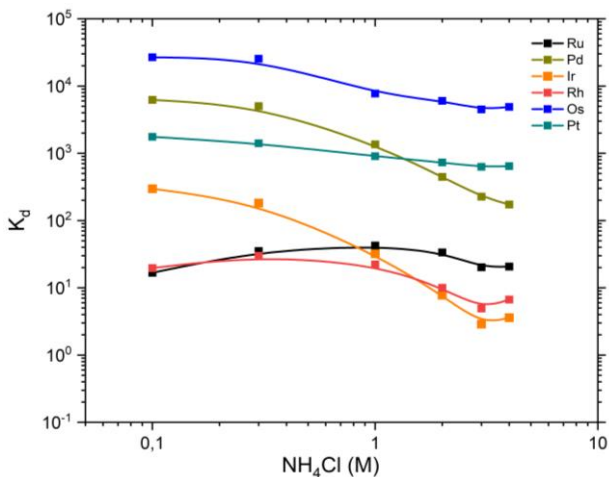
times higher than on cation-exchange sorbent. This can be explained by the fact that gold forms a stable  $[\text{AuCl}_4]^-$  anion in solution.



**Figure 8.** Distribution coefficient ( $K_d$ ) of transition metals on anion-exchange sorbent (Dowex 1-X8Cl<sup>-</sup> form) at different concentrations (0.1, 0.3, 1, 2, 3 and 4M) of  $\text{NH}_4\text{Cl}$  solution.

The platinum group metals were weakly adsorbed on cation-exchange sorbent in ammonium chloride solution ( $K_d < 10$ ), while they have shown high adsorption values on anion-exchange sorbent (Figure 9).

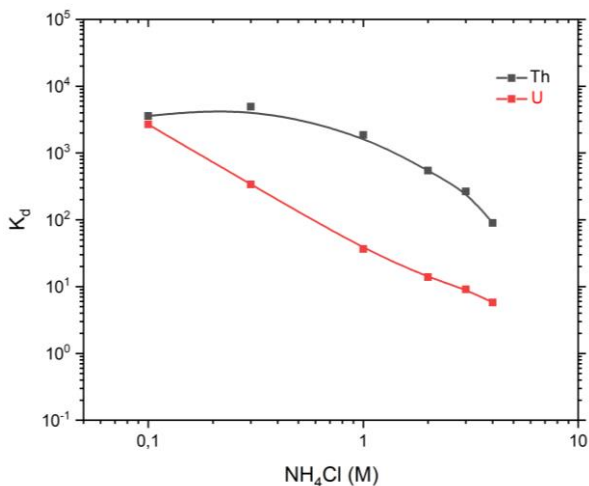
Elements with oxidation state III from platinum group metals form complexes with chloride ions in aqueous solutions. In aqueous solutions, in the absence of any external influences, platinum and palladium show predominantly oxidation state IV, and their six-coordination number allows the formation of negatively charged complexes.



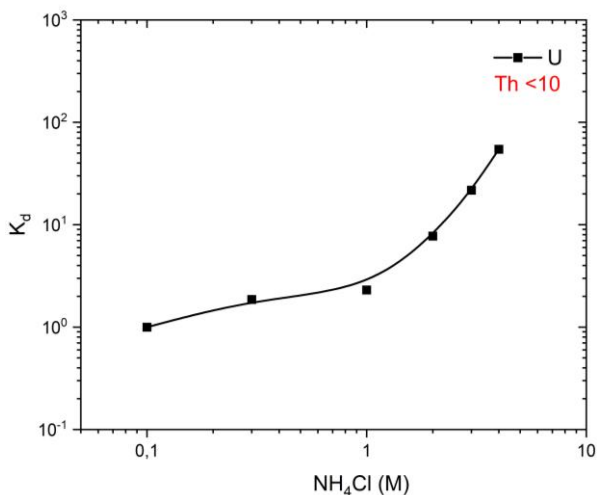
**Figure 9.** Distribution coefficient ( $K_d$ ) of platinum group metals on anion-exchange sorbent (Dowex 1-X8Cl<sup>-</sup> form) at different concentrations (0.1, 0.3, 1, 2, 3 and 4M) of  $\text{NH}_4\text{Cl}$  solution.

As the concentration of the ammonium chloride solution increases, the distribution coefficient of U and Th actinoids decreases on the cation-exchange sorbent (Fig. 10). Adsorption of uranium and adsorption of 2+ charged ions are compatible. This is explained by the presence of uranium in the solution in the form of uranyl ( $\text{UO}_2^{2+}$ ).

Th demonstrates weak sorption in the anion-exchange sorbent ( $K_d < 10$ ). The distribution coefficient of the U element in the anion-exchange sorbent is observed to increase with the increase in the concentration of the solution. This can be explained by the formation of  $\text{UO}_2\text{Cl}_3^-$  and  $[\text{UO}_2\text{Cl}_4]^{2-}$  ions as the concentration of  $\text{Cl}^-$  ions increases in the solution. A somewhat weak sorption of Th in anion-exchange sorbent can be explained by the formation of negatively charged complexes that may be formed as a result of hydrolysis (Figure 11).



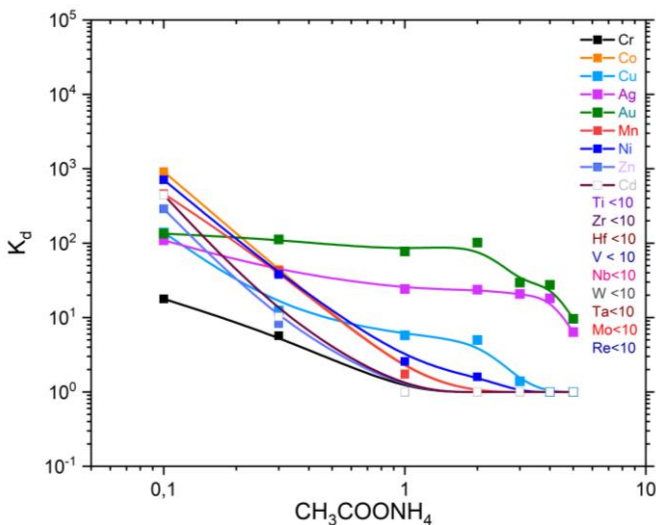
**Figure 10.** Distribution coefficient ( $K_d$ ) of actinoids on cation-exchange sorbent (Dowex 50W-X8  $\text{NH}_4^+$  - form) at different concentrations (0.1, 0.3, 1, 2, 3 and 4 M) of  $\text{NH}_4\text{Cl}$  solution.



**Figure 11.** Distribution coefficient ( $K_d$ ) of actinoids on anion-exchange sorbent (Dowex 1-X8  $\text{Cl}^-$  - form) at different concentrations (0.1, 0.3, 1, 2, 3 and 4M) of  $\text{NH}_4\text{Cl}$  solution.

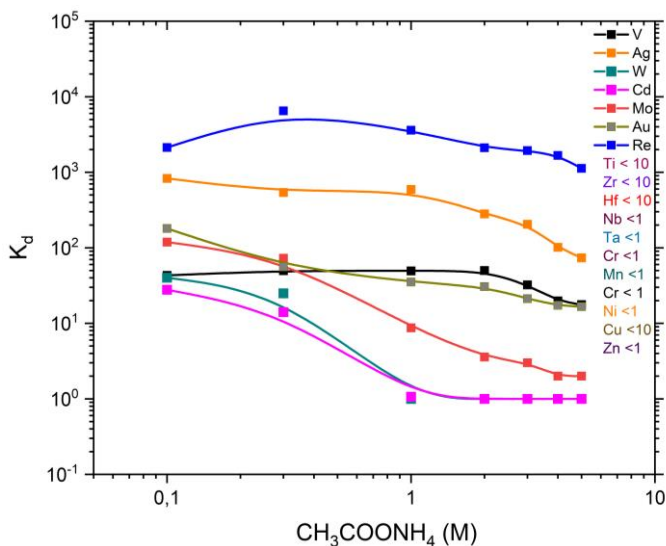
The sorption tendencies of alkali, alkaline earth and rare earth metals on cation and anion exchange sorbents in ammonium acetate solution were similar to those in ammonium chloride solution. Elements of all three groups were well sorbed from aqueous solution of ammonium acetate on cation-exchange sorbent. As the concentration of the solution increased, the values of the distribution coefficient decreased. On the anion-exchange sorbent, this group of elements was not sorbed from the solution to the sorbent.

Transition metals Au and Ag show a similar sorption pattern on cation-exchange sorbent (Figure 12). These elements were sorbed by the sorbent at all concentrations of the solution. The processes of sorption of Mn, Co, Ni, Cu, Zn and Cd elements have the same trend.



**Figure 12.** Distribution coefficient ( $K_d$ ) of transition group elements on cation-exchange sorbent (Dowex 50W-X8  $\text{NH}_4^+$ -form) at different concentrations (0.1, 0.3, 1, 2, 3, 4 and 5 M) of  $\text{CH}_3\text{COONH}_4$  solution.

High sorption values at low concentrations of the solution decrease as the concentration of the solution increases. These elements form hydrocations ( $\text{Me}(\text{H}_2\text{O})_2^+$ ) in aqueous solutions. Also, these elements are known to form mono- and bidentate cations in acetate medium. Chromium has significant distribution coefficient values only at concentrations of 0.1-0.3 M. At other concentrations, this element was not captured in the sorbent. Ti, Sr, Hf, V, Nb, Ta, Mo, V and Re elements were weakly sorbed on the sorbent.  $K_d$  values for these elements were below 10. Ti, Sr, Hf elements are hydrolyzed in solution and form aquacomplexes. The processed complexes reduce the sorption of those elements in the sorbent. Ti, Sr and Hf elements were weakly sorbed on anion-exchange sorbent ( $K_d < 10$ ).



**Figure 13.** Distribution coefficient ( $K_d$ ) of transition group elements on anion-exchange sorbent (Dowex 1-X8  $\text{CH}_3\text{COO}^-$  - form) in different concentrations (0.1, 0.3, 1, 2, 3, 4 and 5 M) of  $\text{CH}_3\text{COONH}_4$  solution.

Hydrolysis processes for these elements in ammonium acetate solution were very characteristic. At all concentrations of ammonium acetate solution, element V was captured in the sorbent (Figure 13).

It is known that vanadium can form  $[(VO)_2(OH)_5]^-$  and  $[(VO)_2(OH)_6]^{2-}$  anion-complexes in solution at  $pH > 5$  values. Unlike vanadium, Nb and Ta do not form negative ions in the solution, so they were not sorbed on the sorbent. At low concentrations of the solution, Ag was significantly sorbed in the solution. It is known that silver acetate has a slight solubility in aqueous solutions. The sorption of Ag on the sorbent can be explained to the formation of neutral forms of silver acetate. The distribution coefficient of Cr, Mn, Co, Ni and Zn in acetate solution is less than 1. In diluted ammonium acetate solution, Mo and W elements are well captured on anion exchange sorbent. The high adsorption of the Re element in the sorbent at all concentrations of the ammonium acetate solution can be explained by the good capture of the  $[ReO_4]^-$  ion formed in aqueous solutions. At low concentrations of the solution, the value of the distribution coefficient of element reaches  $10^3$ .

## MAIN CONCLUSION

1. The concentration of mixed elements, including Th and U radionuclides, in ammonium chloride and ammonium acetate salts synthesized for nuclear medicine and low-background research with an improved method of obtaining pure substances was studied by neutron activation, gamma spectroscopy, atomic emission and mass spectrometric methods. Synthesized salts are purer than their commercial analogues. Synthesized salts that meet modern requirements in terms of purity were recommended by the Joint Institute for Nuclear Research for use in relevant technical fields and salt samples were presented for application.

2. The distribution of polluting metals and radioactive mixtures in deionized and bidistilled water used for the synthesis of ammonium salts, in ammonium alkali, hydrochloric and acetic acids used as initial

precursors purified by sub-distillation, was studied. In the purified precursors, the reduction of amount of Th, U and other radionuclides to 1 ng/l was determined.

3. The purity of the plastic containers used in the process of synthesis of ammonium salts and for storing solutions was investigated by neutron activation method and was determined trace amount for 25 elements, including Th and U in concentrations of 0.1  $\mu\text{g}/\text{kg}$  - 1.0 mg/kg. Trace amounts of radionuclide atoms in the polymer material of the containers used in the experiments are attached to the polymer molecules by chemical bonds. The non-transfer of impurities into solutions has been confirmed by analysis and evidential measurements.

4. It was determined that alkaline, alkaline-earth metals and rare earth elements are sorbed in Dowex 50W-X8 cation-exchange sorbent as they are in the form of positive ions in ammonium chloride and ammonium acetate solutions. The adsorption capacity of cations increases as the charge of cation ions increases.

5. As the valence of transition elements increases, the adsorption capacity of elements decreases on cation-exchange sorbents and the sorption capacity increases on anion-exchange sorbent. Platinum group elements have weak sorption property on Dowex 50W-X8 cation-exchange sorbent and high sorption property on Dowex 1-X8 anion-exchange sorbent. Non-metals form strong covalent bonds in ammonium chloride and ammonium acetate solutions and therefore poorly sorbed by ion-exchange sorbents.

6. Of the most common actinides in nature, thorium is less sorbed by anion-exchange sorbents from both solutions than uranium. On the other hand, thorium is sorbed better than uranium by a cation exchange sorbent.

7. The results obtained as a studying the adsorption of elements from solutions of ammonium chloride and ammonium acetate salts to ion-exchange sorbents can be recommended for the use in selective separation and purification of those elements.



## LIST OF PUBLISHED SCIENTIFIC WORKS ON THE TOPIC OF THE DISSERTATION

1. **Mirzayev N.**, Marinov G., Marinova A., Mammadov Kh.F., Karandashev V. K., Filosofov D. V. Sorption of 60 elements on cation exchange resins in ammonium acetate solutions // Journal of Radiation Researches – 2018, vol. 5, № 2, p. 165-172
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The defense will be held on 27 September 2024 at 14<sup>00</sup> at the meeting of the Dissertation council FD 1.22 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at Institute of Radiation Problems of the Ministry of Science and Education.

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