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Modern Methods of Separation and Determination of Radioactive Elements*

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Abstract

A review is given of the new techniques of separation and determination of radioactive elements developed in recent years. Great progress has been achieved in obtaining and stabilizing actinides in unusual oxidation states, both high and low. On this basis a highly efficient extraction and sorption technique for the separation of elements is proposed. Application of bidentate phosphoro-organic reagents open up new prospects in the separation of elements in the trivalent state.

Achievements in the development and application of luminescent and coulometric methods of actinide element determinations are considered. The techniques of determination of some radioactive elements in natural samples are discussed.

Introduction

From the acknowledged 110 elements of the Mendeleev Periodic System only about 30 elements lack stable isotopes and are radioactive. The chemical properties of transuranium elements (TUE) have recently been most intensively studied. This is mainly due to the rapid growth of nuclear energetics and the increasing use of TUE in various fields of science and engineering.

Two principal trends in the progress of TUE analytical chemistry can be noted. First, to solve the group of problems connected with environmental control, determination of TUE (especially plutonium and americium in various natural samples) and methods of analysis with record low detection limits have been developed. These are mainly radiometric methods combined with preliminary chemical isolation and concentration, as well as luminescence methods. The other trend consists in the development of simple, reliable, precise and selective techniques for determination of the elemental and nuclide composition of irradiated targets, technological solutions and products, on the basis of various analytical methods.

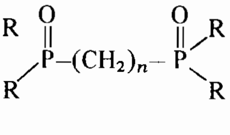
Separation Methods

Modern analytical methods, particularly when used in combination with computer data-processing, make it possible to determine directly individual isotopes of TUE in various materials. Preliminary isolation, preconcentration and separation from other elements is required to solve numerous practical problems of TUE determination. Extraction and chromatography, extraction-chromatography, precipitation and coprecipitation, paper chromatography, sublimation and electrophoresis are used for these purposes. Some problems in the methods of TUE separation and determination have been systematized in surveys [1–3] and monographs [4, 5].

Liquid Extraction

The extraction methods of TUE isolation and separation are widely applied both in the analytical chemistry of these elements and in the technology of their production. Extractants of different classes are applied for TUE extraction isolation and separation: organophosphorus compounds, amines, chelate-forming reagents and their mixtures (synergetic extraction) [6].

The most successful investigations of recent years have been into TUE extraction from strong acid solutions, particularly aimed at separation of the long-living americium and curium isotopes from the waste radioactive solutions subjected to disposal. This is a complicated radiochemical task, since these solutions are strongly radioactive and contain large amounts of salts and acids. Most of the presently known extractants cannot solve this task, except for the neutral bi- and polydentate organophosphorus extractants containing either two ≡P=O groups, or one ≡P=O group and one =C=O group. The properties of these reagents



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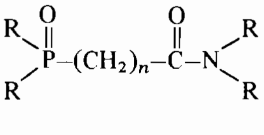

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for the extraction of different metals have recently been considered in detail by Schulz and Navratil [7]. The wide possibilities of varying the structure of those compounds (the number and nature of the functional groups, the nature and structure of the bridge between the functional groups, and one substituents at phosphorus and nitrogen atoms) allow one to obtain reagents with certain properties.

The most intensive work with these reagents is carried out in the Soviet Union in our institute and in the U.S.A. (the Argonne National Laboratory); recently several papers on this subject have appeared in Japan, Czechoslovakia, France and India.

In our laboratory more than 60 phosphorus- and phosphorus–nitrogen-containing bi- and polydentate neutral reagents have been studied. The effect of their structure (molecule conformation, nature of the substituents at the atoms of phosphorus and nitrogen and in the methylene bridge, and the composition of the bridge) on the extraction capacity, selectivity and solubility has also been investigated [8,9]. All the reagents studied may conventionally be divided into three groups:

The first group combines phosphorus-containing bi- and tridentate reagents with linear bridges between the P=O groups and their derivatives, in which the hydrogen atom in the bridge is substituted by another atom or a radical. The reagents of that group are characterized by the highest extraction capacity and a high radiation stability. Reagents with phenyl substituents at the phosphorus atoms and with a methylene (or vinylene) bridge between the functional groups should especially be stressed. With the help of these reagents, methods have been worked out of TPE extraction isolation from strong acid salt-containing solutions with simultaneous concentration by 20–100 times. The reagents are of typical group character and are not selective [8].

The second group consists of the bi-, tri- and tetradentate phosphorus-containing reagents, in which the bridge between the functional groups is composed of chains of *ortho*- and *meta*-xylene, mesitylene, durene and other methyl-substituted aromatic compounds. The major property of those reagents consists in their high selectivity, which reveals itself in relation to the elements, depending on the location of the functional groups in the aromatic ring of the bridge. Thus, at the *ortho*-position of the phosphenylmethyl groups, the U(VI), Pu(IV)/Am(III) separation factor exceeds 10^4; at the *meta*-position the Eu/Am separation factor exceeds 5, and with an increase in acidity it reaches 10–12 [10].

The phosphorus- and nitrogen-containing reagents which make up the third group (dialkyl(diaryl) dialkylcarbamoylmethyl phosphine oxides) have quite a high extraction capacity, although they are in some ways inferior to the first group of reagents. Their priority however, consists in their higher solubility in organic diluents and their relative simplicity in synthesis [9]. It is interesting that this group of compounds is characterized by the so-called 'phenyl effect', which had earlier been observed only in the extraction by alkylenediphosphine dioxides, and which consists of an increase in the extraction capacity of the reagents on the replacement of the alkyl substituents at phosphorus atoms with the more electronegative phenyl ones [11]. Therefore the phenyl-containing extractants become the best ones in this group. These reagents are non-selective, as well as the alkylenediphosphine dioxides. The application of these reagents to the group extraction concentration of transplutonium elements from strong acidic salt-containing solutions increases the concentration factor by up to 50 [9].

The main drawback of all the mentioned reagents is the low solubility of their complexes with metals in aliphatic hydrocarbons. It has lately been shown that the addition of TBP to bifunctional reagents allows use of these diluents as well. Besides, TBP addition increases the solubility of bidentate reagents and their metal complexes in all the diluents.

The application of alkaline solutions is worthwhile for the TUE group isolation and concentration. The extraction of metals from such solutions had not been studied until recently. Our investigations have shown that TUE and many other elements can be extracted from alkaline solutions in the presence of the complex-forming reagents by extractants of various classes: quaternary ammonium bases, amines, alkylpyrocatechols, alkyl derivatives of amine-alcohols and β-diketonates [12]. Some of the extractants studied, especially alkylpyrocatechols and α-oxy-5-alkylbenzyldiethanolamine, are characterized by a high extraction capacity in relation to TPE, since with their help it is possible to isolate effectively those elements from the alkaline and carbonate solutions with a concentration of the order of 5–6 M (Fig. 1). The efficiency of the extraction isolation of TUE depends on the nature of the complex-forming reagents capable of keeping elements in alkaline solutions in the soluble form. The role of the complex-forming reagents is sometimes reduced to keeping the hydrolyzable elements in the soluble form in alkaline solutions (α-hydroxycarboxylic acids, carbonate ions), which is essential, since they remain in the aqueous phase during the extraction. In other cases, complex-forming reagents (aminopolyphosphonic acids) pass into the organic phase together with the metal. Therefore, depending on the conditions, the elements may be extracted from alkaline and carbonate solutions in the form of ion associates, the anionic part of which contains either hydroxo-complexes of the corresponding metals, or their compounds with the complex-forming ligand, while the cationic part is the extractant. They can

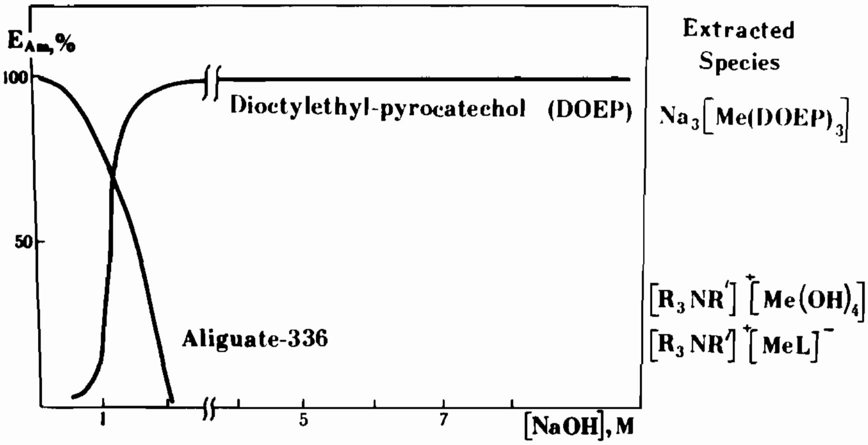

Fig. 1. Dependence on the concentration of NaOH of the extraction of Am(III) by 0.04 M DOEP in toluene and 0.2 M Aliquat 336 in xylene.

also be extracted in the form of the coordinationally saturated and hydrolyzed β-diketonates.

Methods of extraction and extraction chromatography [1] in alkaline and carbonate solutions are used to solve a number of practical tasks [13]. First, under certain conditions with the help of all the extractants studied, one can quantitatively isolate and concentrate the elements capable of existing in alkaline solutions in the form of various complex compounds. Secondly, the group concentration of tervalent TPE is achieved in these systems with simultaneous isolation from the elements in other oxidation states as well as from iron (separation factor Am(III)/U(VI) = 2×10^3; Pu(IV)/Am(III) > 1×10^2; Ce(IV)/Am(III) = 4×10^2; Fe(III)/Am(III) = 1×10^3; Am(III)/Cs(I) = 2×10^3). The selectivity of extraction may be increased by varying the alkaline concentration and the time of phase contact. That will allow separation of TPE from some rare earth fission products (Ce/Am = 3×10^2; Eu/Am = 70) and the intragroup separation of the TPE (Bk/Am = 4×10^2). The prior method for separation of tervalent americium and curium in carbonate media is extraction chromatography, since separation is increased due to the differences in the values of the mass-transfer coefficients, as well as to the equilibrium of the process.

The best separation is obtained by using the methods based on various oxidation states of the separated elements. Almost all the transplutonium elements (except Lr) can exist in solution in ‘unusual’ oxidation states from 1+ up to 7+. The possibility of oxidation or reduction of the M^{3+} ion to a higher or a lower oxidation state, and its stability in solution, are known to be determined by the value of the oxidation potential of the corresponding redox couple. It is quite easy to obtain such ions as Am(V), Am(VI), Bk(IV), Md(II), No(II) in dilute solutions of mineral acids. In other cases the values of the potentials of the M(IV)/M(III) couples are too great, and those of M(III)/Me(II) are too small, and neither oxidation nor reduction are possible. However, in the presence of strong complex-forming reagents, which form more stable complexes with M^{4+} ions, the oxidation potential of the M(IV)/M(III) redox couple is decreased to such an extent that oxidation becomes possible.

Systematic investigation of TPE properties in solution has resulted in the creation of quite a number of practically convenient methods for the preparation and stabilization of Am(IV) and Bk(IV), Am(V) and Am(VI), Fm(II) and Md(I) [14]. This has expanded their application in analytical practice and technology, and primarily in the extraction and sorption methods of isolation and separation of those elements. Thus, for example (Fig. 2), the extraction of tetravalent americium from nitric and sulphuric acid solutions containing potassium phosphotungstate with the high-molecular secondary amine solution gives a high degree of separation from the other TPE in the tervalent state and from the fission elements [15]. The tervalent curium is separated from americium by 1-phenyl-3-methyl-4-benzoylpyrazol-5-one (PMBP) extraction after the electrochemical oxidation of the latter up to the hexavalent state in sodium pyrophosphate solution with pH = 10 has been conducted. The coefficient of curium separation from americium during one extraction cycle is $\sim 10^3$ (Table I).

Sorption

Sorption methods are widely used for the isolation of radioactive elements, recovery from irradiated materials, and separation from fission products and miscellaneous impurities aimed at obtaining extremely pure samples of these elements for medical purposes and physical investigations, as well as the separation of TPE from different natural materials for

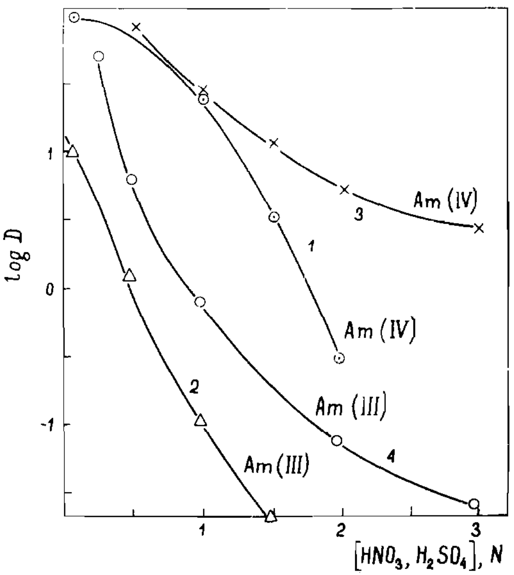

Fig. 2. The extraction of Am(III) and Am(IV) by 3% dioctylamine in dichloroethane from nitric (1, 2) and sulphuric (3, 4) acids containing 10^{-3} M $K_{10}P_2W_{17}O_{61}$.

quantitative determination. These methods have a number of advantages. They are effective for TPE separation and they are simple when the process is carried out at a distance with the highly radioactive sample in shielded hot boxes and cells. Unfortunately, at present, the choice of the optimum conditions for sorption separation is still made experimentally, although methods of mathematical modelling of these processes have been developed.

Among numerous sorbents used for radioanalytical purposes, a new type of sorbent–solid extractants (TVEX) has successfully been studied. It was obtained by introducing an extractant into the polymer matrix during its synthesis. TVEX's properties are characterized by a high content of the extractant in granules, a regular spheric form, simplicity of column preparation and satisfactory reproducing properties; all that makes them appropriate for analytical as well as preparatory purposes. The behaviour of uranium, plutonium and TPE (including TPE in various oxidation states) has been studied, as well as the conditions of separation of those elements, their group separation from the fission product on the TVEXs with TBP, TOPO, HDEHP and TOA, etc. [16, 17].

Among the inorganic sorbents, zirconium phosphate has gained the most wide use in TUE separation. This sorbent has been used for americium and curium separation, selective isolation of berkelium and for other purposes [18].

The application of aqueous–alcohol solutions has made it possible to essentially enhance the efficiency of separation of radioactive elements on ionites [19]. On the basis of systematic study of the behaviour of most TU and fission elements on cationites and anionites in the aqueous–alcohol solutions of nitric, chloric, hydrochloric, sulphuric and phosphoric acids, as well as in solutions of salts and strong complex-forming reagents, fairly effective and practically convenient methods of isolation and concentration of many elements from large amounts of solutions of almost any composition have been worked out. In a number of cases high coefficients of separation of the elements with similar properties have been achieved; for example, a number of TUE with the use of their consecutive eluation from the anionite with different aqueous–organic solutions (Fig. 3).

Especially effective TUE separation on ionites can be expected when the unusual oxidation states of these elements are used. However, detailed investigations in this direction have not been conducted. The first results have been obtained on americium (Fig. 4) and berkelium (Fig. 5) separation from other TPE with the use of their highest oxidation states [20], and on the isolation of the short-living mendeleevium isotopes in the 2+ oxidation state from irradiated targets [21] (Fig. 6).

Separation in the Gaseous Phase

The interest in the volatile compounds of the radioactive elements is caused by the possibility of using them for the development of new methods of separation of radionuclides, of mass-spectrometric separation of isotopes, for obtaining and purifying metals, and for obtaining various kinds of coatings. The most pronounced results have been obtained from the synthesis and study of the properties of the volatile actinide β-diketonates. β-Diketonates of thorium, uranium, neptunium and plutonium, includ-

TABLE I. Separation of Am and Cm from 0.1 M Pyrophosphate Solution at pH = 10 by Extraction with 0.1 M Solution of PMBP in Chloroform

Element	Taken		Found in aqueous phase		Found in organic phase		
	Oxidation state	µg	Oxidation state	µg	Oxidation state	µg	%
Am	VI	241.2	V	227.0	III	14.2	5.8
Cm	III	25	III	0.04	III	25	98.4

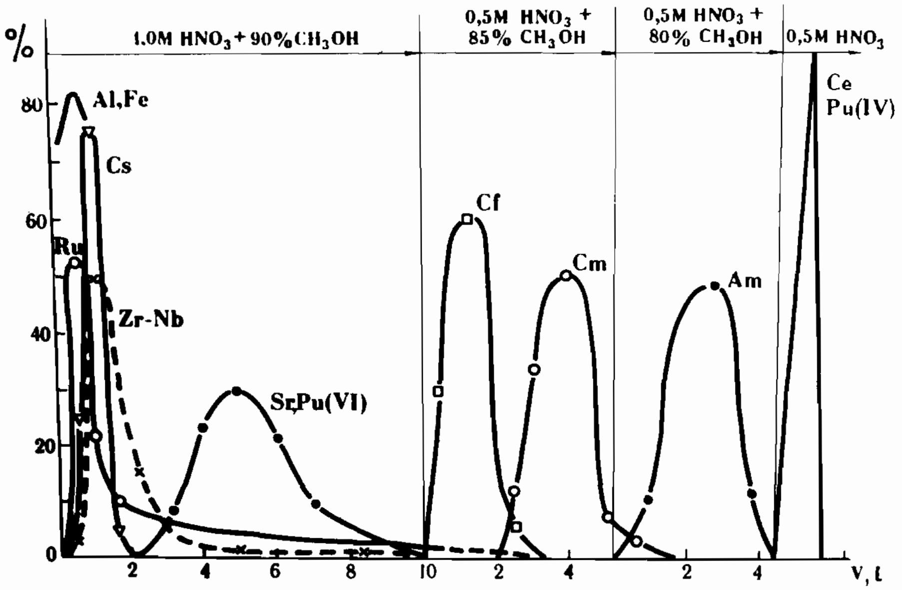

Fig. 3. Separation of transuranium elements on anion-exchanger Dowex-1 X 8 by consecutive elution with different aqueous-organic solutions; column 3.2 X 25 cm.

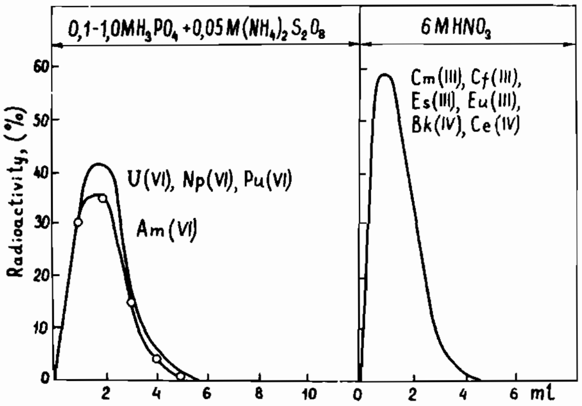

Fig. 4. Separation of transuranium elements in different oxidation states on cation-exchanger Dowex-50 X 8; column 0.3 X 6 cm.

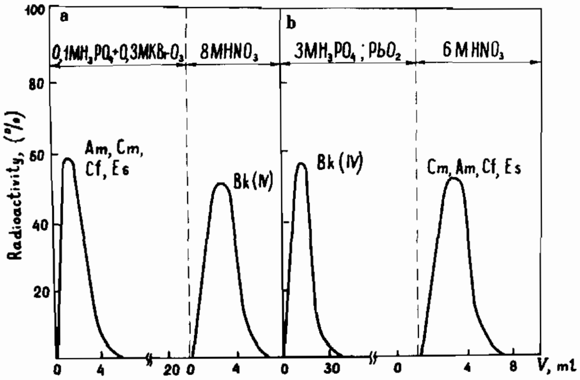

Fig. 5. Separation of ^{249}Bk(IV) from trivalent transplutonium elements by H_3PO_4 solutions on anion-exchanger Dowex-1 X 8 (a) and cation-exchanger Dowex-50 X 8 (b) in the presence of an oxidant; column 0.3 X 4 cm.

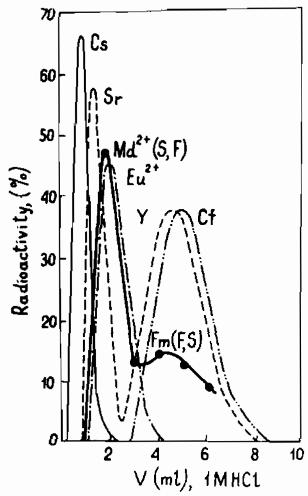

Fig. 6. Separation of ^{256}Md(II) from Cf(III) and Fm(III) on cation-exchanger Dowex-50 X 5 in the presence of Zn(Hg).

ing their hexavalent states, and their adducts with phosphoro-organic compounds have been obtained. In our institute the volatile β-diketonate complexes of californium, einsteinium and fermium have been synthesized with the use of the extraction method and the reactions of interaction of chlorides and the non-volatile β-diketonates with the β-diketone vapours. Their thermal stability and volatility have been studied (Fig. 7) [22].

Practical applications of the β-diketonates became possible after a number of complicated problems had been solved. The conditions for and ways of synthesis of the compounds, as well as the factors affecting

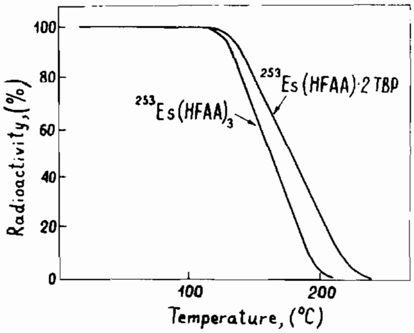

Fig. 7. Thermoradiograms of einsteinium hexafluoroacetylacetonate and its adduct with tributyl phosphate.

their stability in the gaseous phase, had previously been studied. With the help of a specially created technique their main physicochemical characteristics, such as temperature dependence of the saturated vapour pressure, sublimation heats, activation energy, thermal and radiation stability, had been determined [23]. The study of hetero and gaseous phase reactions of the β-diketone vapours with various metal compounds has given most interesting results. The ligand exchange in such reactions with the participation of the β-diketones in the gaseous phase had not been known before [24].

Methods of Determination

The whole variety of modern analytical methods, including chemical, physicochemical and physical methods, are widely used for determination of the radioactive elements in various materials with the aim of controlling the existing radiochemical processes, to analyse environmental materials, and to prepare standard samples, etc. Various radiometric methods are naturally most widely used. Thanks to the considerable improvement in measuring equipment and to the verification of the half-life periods of radioisotopes, the radiometric methods of TUE determination achieve high precision. This is particularly the case with the α-activity methods, in which the best measurement characteristics (low detection limit, high efficiency and small errors) are obtained with the use of silicon semiconductor detectors. Recently, for the determination of the α-activity of TUE directly in solution, 'immersed' Si(Li) detectors have been widely used [25]. Such detectors have a low counting efficiency and are convenient for measuring high levels of α-activities.

The parameters of radiometric methods of analysis have been enhanced with the application of modern spectrometers with built-in computers and the new mathematical methods of processing the obtained results. Out of all the modern methods of determination of radioactive elements we shall discuss the methods which provide for the determination of separate elements with the maximum sensitivity and precision.

Together with radiometry and neutron activation analysis, luminescence methods give high sensitivity. These methods are based on measurement of the intensity of the luminescence of crystallophosphorus (the inorganic compounds), the crystalline lattice of which contains the elements to be determined. The crystallophosphorus samples are prepared by introducing the solution to be analysed into the matrix, drying and calcination. The bands of crystallophosphorus luminescence on the basis of lead molybdate, activated by neptunium, plutonium and uranium, are far from one another (Fig. 8). This makes it possible

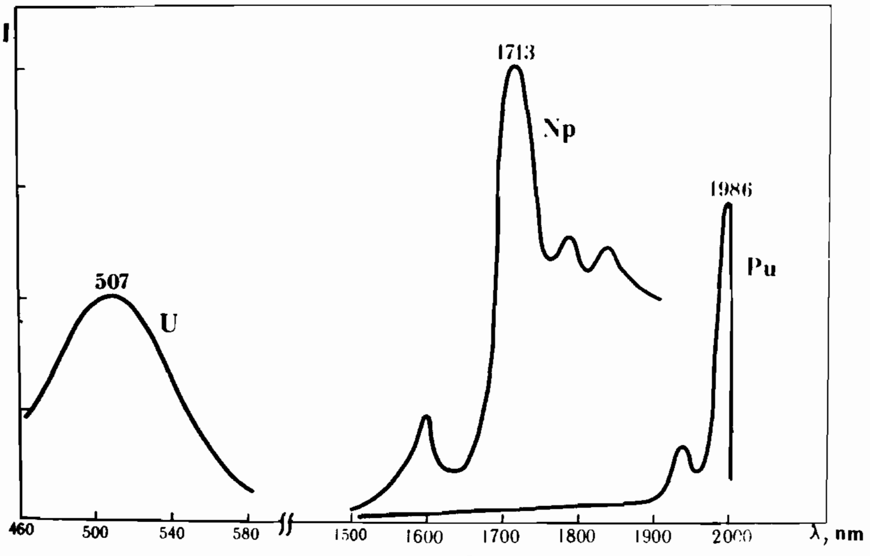

Fig. 8. Luminescence spectra of uranium ($1 \times 10^{-5}\%$), neptunium ($1 \times 10^{-7}\%$) and plutonium ($1 \times 10^{-7}\%$) at lead molybdate.

to determine these elements from one sample [26]. Since it is based on the use of crystallophosphorus, in most cases the method does not require preliminary concentration and isolation, nor transfer of the determined elements into the required oxidation state.

To enhance the rate of luminiscent determination, methods based on the luminiscent properties of the actinide complexes in frozen solutions have been developed. As for the neptunium determination sensitivity, this method is found to be inferior to methods based on crystallophosphorus luminescence, although it is on the same level as the radiometric methods. The selectivity is determined by the extraction methods of neptunium isolation.

Without doubt laser resonance spectroscopy is the most promising for the determination of actinide traces. Thus, according to the reference literature, this method provides for the detection of ionized plutonium atoms in a vacuum at the level of 10^{-17} g. In our first experiments, when neptunium solution was introduced into the acetylene–nitrous oxide flame, the action of the laser light with the frequency caused the selective multiple ionization of the NpO^+ molecules, giving a sensitivity of 10^{-9} g. The sensitivity might be increased if a non-flame atomization were used.

Coulometry is one of the most accurate methods. The major advantage of this method is that it does not require standards. The amount of material is determined by the absolute quantity of the electricity, obtained from the electrolysis current integration. Two main trends in this method are known: coulometry at a constant current density; and coulometry with a controlled potential. Lately the second trend (potentiometric coulometry and its variation), coulometry with potential scanning, has been more intensively developed.

The method is mainly used for precise determination of the bulk component, particularly uranium and plutonium, in standard solutions metals and oxides, as well as for determination of the stoichiometric composition of various materials. Precision of the order of several hundredths of a percent is achieved [27].

Coulometry with a controlled potential is also used for the determination of small amounts of americium from both Am(VI)/Am(V) and Am(IV)/Am(III) couples (Table II). The same method is applied to the precise determination of trace amounts of berkelium [28].

Coulometry with the potential scan is successfully used for the determination of trace amounts of neptunium and plutonium in samples of various composition. The sensitivity of the method is $\sim 10^{-9}$ M. Relative precision depends on the amount of the material in the sample to be analysed, and it is 1.0–2.0% for 10–100 μg.

Together with luminescence and coulometry, the X-ray fluorescence method has lately been widely used for the determination of neptunium and plutonium in solution and solid materials [29]. The method makes it possible to determine the content of the elements in the materials to be analysed with a sufficient precision (~0.5%), approximation and sensitivity without preliminary chemical preparation of the samples. Also the chemical state of the determined element does not affect the results of the analysis.

As for samples which are analysed by radioanalysts today, the environmental material is certainly the prior task [30]. The intensive development of atomic energetics, and the extensive use of isotopes, including the actinide ones, in medicine, space technique and scientific research, and the existing laws on environmental control, all require development of the methods of determination of many radionuclides, including plutonium, uranium and americium isotopes, with a very high sensitivity and reliability [31].

The existing methods of plutonium determination, for example, are still too complicated, and time- and labour-consuming [32].

Conclusions

Some prospects for further progress in the analytical chemistry of radioactive elements are as follows.

TABLE II. Selective Coulometric Determination of Americium

Couple	Composition of the solutions	Oxidation time (h)	Detection limits (μg)	Relative standard deviation (%)
Am(VI)/Am(V)	2 M H_3PO_4 + 0.1 M $HClO_4$	1.5	5	1.5
Am(IV)/Am(III)	0.5 M H_3PO_4 in CH_3CN	0.5	5	2
Am(IV)/Am(III)	0.1 M $HClO_4$ + 6 × 10^{-3} M PW[a]	0.25	6–70	3
Am(IV)/Am(III)	1–2 M Na_2CO_3 (pH 10)	0.5	5	2.5

[a] PW = potassium phosphotungstate.

One of the major tasks is the study of the chemical properties of transplutonium elements in the yet unknown oxidation states and the development on this basis of new efficient methods for their isolation and highly sensitive methods for their determination.

In the field of extraction, the use of the multiphase systems, particularly aqueous systems, salt and metal melts, as well as the study of kinetics, interphase equilibria and new ways of accomplishing the process, are worth thorough attention.

Further development of the sorption methods requires a wider application of modelling, synthesis and study of the properties of new sorbents.

The use of gaseous phase reactions, especially new ways for the preparation of the volatile compounds of radioactive nuclides, open up great opportunities.

The development of the methods of determination of radioactive elements should be based first of all on the remote analysis methods, the methods with a high productivity (e.g., flow injection methods), and on a wide use of computers.

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