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PROGRESS IN THE ANALYTICAL CHEMISTRY OF URANIUM, THORIUM, AND PLUTONIUM

V.I.Kuznetsov, S.B.Savvin, and V.A.Mikhailov

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INTRODUCTION

Extensive use of atomic energy had led to the rapid development of the analytical chemistry of uranium, thorium, and plutonium, and several thousand papers on this subject have been published.

Numerous data on the chemistry of uranium, thorium, and plutonium are collected in the monograph by Seaborg and Katz (Russian translation edited by Nikolaev[1]) and in a recent monograph by Seaborg[2]. The most complete review of the literature on the analytical chemistry of thorium is given in a monograph by Ryabchikov and Gol'braikh[3]. The analytical chemistry of uranium is dealt with in monographs and review articles by Rodden[4-6], Palei[7], and others[8-23]; interesting data on the chemistry of thorium are given in reviews by Rodden[4,24], Schoeller[25], Moeller[26], Bonardi[27], and others[8,11,31,28,29], and on the chemistry of plutonium in reviews by Metz and Harvey[30-34].

This article reviews modern chemical methods for determining uranium, thorium, and plutonium, and discusses the analytical reactions of these elements. Physical, physicochemical, and other instrumental methods, which

have been reviewed elsewhere[1,4,20,35-40], are dealt with in less detail.

The analytical chemistry of neptunium has been reviewed by Alimarin and Zolotov[41], but little has yet been published on the analytical chemistry of the other transuranic elements.

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A characteristic feature of the analytical reactions of uranium, thorium, and plutonium is the close similarity between ions of the same type, which results from the similarity in the electronic structures of the elements. The pH values at which hydrolysis of the cations of the elements begins are almost identical, which means that the conditions under which the corresponding analytical reactions take place are closely similar. Differences are shown in some specific reactions, e.g. the luminescence of uranyl compounds.

The reactions of the U^{4+}, Th^{4+}, and Pu^{4+} cations also resemble those of Zr^{4+}, Ti^{4+}, and Ce^{4+}. The pH values required for reactions related to the ease of hydrolysis of these cations lie in the same order as those of the "start of precipitation of the hydroxides": i.e. Ti^{4+} (< 2), Zr^{4+} (2), Pu^{4+} (3), U^{4+} (3.2), and Th^{4+} (4).[1] Reactions of this class include many colour reactions with organic reagents, precipitation reactions, etc.

Similarities are also shown by the UO_2^{2+} and PuO_2^{2+} ions, and by the Pu^{3+} and U^{3+} ions. Considerable differences are shown, however, by the redox reactions. Although thorium(III) is known, Th^{3+} ions in aqueous solution are so unstable that the redox reactions cannot be used for analytical purposes[1].

Uranium is most stable as the cation UO_2^{2+}. Uranium(IV) is oxidised in air as readily as, or even more readily than ferrous salts. Uranium(III) is a powerful reducing agent, and its salts are oxidised in air as readily as titanium(III) salts.

Plutonium(IV) is stable in air. Plutonium(III), unlike uranium(III), is not a powerful reducing agent, and Pu^{3+} salts are oxidised in air at approximately the same rate as uranium(IV) or ferrous salts. The PuO_2^{2+} ion is a weak oxidising agent, of approximately the same strength as elemental iodine.

The most important chemical methods for separating uranium, thorium, and plutonium from other elements depend on the variable oxidation state of plutonium and uranium and the ability of Pu(VI), Pu(IV), U(VI), U(IV), and Th nitrates to be extracted from aqueous solution by oxygen--containing organic solvents immiscible with water. Methods involving the precipitation of Pu(IV), U(IV), and Th from strongly acidic solution, and some chromatographic methods, are also fairly selective.

The chief chemical methods for the determination of the elements are: redox volumetric for U and Pu; volumetric complexometric for Th, Pu(IV) and U(IV); gravimetric for all three; and photometric for U(IV), U(VI), Pu(IV), and Th.

The colour reactions of the +4 cations are carried out in solutions acidified with inorganic acids, and in a number of cases this forms the basis for their direct determination in the presence of +1, +2, and +3 foreign cations whose colour reactions occur in less strongly acidic solutions.

1. REACTIONS OF URANIUM, THORIUM, AND PLUTONIUM IONS IN AQUEOUS SOLUTION

A. Reactions of Uranium

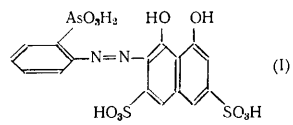
In acid solutions free from sulphate, phosphate, fluoride, and other complex-forming substances, uranium(VI) is present as solvated UO_2^{2+} ions, which give the solutions a yellow-green colour. When the pH is increased to ~4.5, the UO_2^{2+} ions are hydrolysed and converted to the soluble $UO_2(OH)^+$ cations[1,4], or, according to other data, to the polycations $U_2O_5^{2+}$ and $U_3O_8^{2+}$.[42,43] Subsequently, insoluble uranates $M_2U_2O_7$ or more complex salts are formed[44-47]. Some authors doubt the existence of UO_2^{2+} ions and consider that basic salts of $U(OH)_4^{2+}$ or $U^{6+}.2UO_3$ are formed[48-54].

At high concentrations of nitrate or chloride in acid solutions, uranyl ions form unstable complexes $UO_2NO_3^+$, $UO_2.(NO_3)_2$, UO_2Cl^+, UO_2Cl_2, $UO_2Cl_4^{2-}$, etc.[1,6]. The sulphato complexes are more stable, so that in solutions containing sulphate, many reactions of uranyl ions (colour reactions, some precipitation reactions, etc.) are not complete. Phosphato complexes are much more stable than the sulphato complexes, and in weakly acidic and neutral solutions, phosphate masks all the reactions of uranyl ions. Phosphato complexes are not formed in strongly acidic solutions[55].

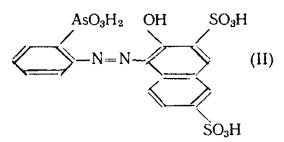
From pH 4–5 upwards, i.e. in the range where UO_2^{2+} cations start to undergo hydrolysis, uranium(VI) forms compounds with various organic ligands[56-59]. Tartaric, citric, and other hydroxycarboxylic acids form hydrophilic anions which are difficult to precipitate and cannot be extracted. These ligands suppress the reactions of UO_2^{2+}, except in strongly acidic media.

At pH 4–7, Trilon B (sodium ethylenediaminetetraacetate, versene, Complexone III, EDTA) and its analogues form comparatively weak complexes with UO_2^{2+}, but have a marked masking action on many other elements[60-69]; this distinguishing feature of uranium(VI) is widely used in analytical chemistry[70-78].

Uranium(IV) is present as solvated U^{4+} cations, in sufficiently acidic solutions, and these have a comparatively intense green colour. These cations are hydrolysed at pH 2.0–3.0, and react with organic reagents of general formula R–OH, such as arsenazo(I) and thoron(II) in more strongly acidic media than U(VI) [79], so that the reactions of U^{4+} are more selective than those of UO_2^{2+}:



(I)



(II)

Nitrato, chloro, sulphato, phosphato, and fluoro complexes of U(IV) are much more stable than those of U(VI).[79-81] The nitrato, chloro, and sulphato complexes exist in both strongly acidic and weakly acidic solutions, but the phosphato and fluoro complexes only in weakly acidic solutions. Chlorides and nitrates do not mask the colour reactions of U^{4+}, but sulphates, and more particularly, phosphates and fluorides, have a strong masking action. The complexes of U(IV) with organic ligands are similar to the correspond-

ing U(VI) complexes. Unlike uranium(VI), uranium(IV) forms stable complexes with Trilon B.

The reactions and conditions for the existence of uranium(IV) ions resemble those of thorium ions.

In acidic and neutral solutions, uranium(III) is present as solvated U^{3+} cations, whose red-violet solutions are rapidly oxidised by atmospheric oxygen, so that reactions involving oxidation of U^{3+} are difficult to study in open vessels.. Complex U(III) ions appear to be analogous to the corresponding complexes of the rare-earth elements.

Uranium(V) exist in solution as UO_2^{2+} ions, which are fairly stable only at pH 2–4.[1,50,82] Uranium(V) disproportionates to U^{4+} and UO_2^{2+} ions with change in the pH or on prolonged standing. The analytical reactions of U(V) have been little studied.

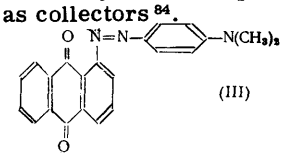
The precipitation reactions of the various uranium ions are not the same. The best known are the precipitation reactions of UO_2^{2+} ions, which form sparingly soluble uranyl salts or hydrolysis products. Most precipitants which have been described contain oxygen and react with uranyl ions to form a U(VI)–O bond, so that they are effective only at those pH values at which this bond can exist, i.e. at pH 4–5 and above. Precipitation by oxygen-containing inorganic (NaOH, NH_4OH, Na_2HPO_4, etc.) and organic (8-hydroxyquinoline, quinaldinic acid) precipitants is not selective, and the same is true of sulphur-containing reagents.

Potassium ferrocyanide precipitates uranyl ions from solutions which are not too strongly acidic[82], and since the precipitate has a bright reddish-brown colour the reaction is usually employed not as a precipitation reaction but as a colour reaction for uranium.

Methods for the precipitation of uranium(IV) are more selective. The precipitation is carried out from fairly strongly acidic solutions, so that elements present as ions which are difficult to hydrolyse remain in solution. Uranium(IV) appears to be precipitated by all precipitants used for thorium and zirconium, although not all such reactions are described in the literature. The ease of oxidation of uranium(IV) salts makes their analysis difficult, but this can be overcome if the precipitation is carried out from solutions containing a powerful reducing agent.

Precipitation of U(IV) by ammonia or alkalis in the absence of reducing agents is not specific, since U(IV) is oxidised very rapidly in alkaline solutions. Phosphate precipitates U(IV) in inorganic-acid solution, and this is used to coprecipitate uranium phosphate with thorium or zirconium phosphate, with subsequent oxidimetric determination of the U(IV) (Volkov's method)[7].

Like the cations of thorium and other elements in oxidation state IV, U(IV) ions are precipitated by many organic anions of high ionic weight; a characteristic example is the precipitation by various arylarsonic acids $Ar-AsO_3H_2$.[83] Uranium(IV) should be precipitated from strongly acidic solutions in the form of complex anions such as UCl_6^{2-} and similar ions, but such reactions have not been described. Uranium(VI) in the form of its anionic thiocyanato complex can be precipitated from extremely dilute solutions (0.1–1 γ/litre) with the heavy organic cations of anthrazo(III) or methyl violet, whose thiocyanates are precipitated simultaneously and act as collectors[84].



(III)

Some theoretical problems related to the coprecipitation of elements with organic collectors have been discussed by Kuznetsov[85,86].

The extraction of uranium may be carried out in a number of ways: by "physical distribution" of uranyl nitrate between aqueous and non-aqueous phases, as oxonium salts from strongly acidic solutions, and as the hydrophobic complexes formed by uranyl ions with various organic compounds[87–101].

The extraction of uranium in the form of its nitrato compounds is of great practical importance. Uranyl nitrate can be extracted from aqueous solutions by many oxygen-containing organic solvents immiscible with water, the most efficient of which are compounds containing carbonyl oxygen, and esters containing one oxygen atom per 4–7 carbon atoms. Ethers are much less efficient. The compounds extracted are complexes of general formula $UO_2(NO_3)_2 . mH_2O . nS$, where S is the solvent, m = 2–4, and n most frequently equals 2. The presence of the two water molecules possibly indicates the existence of $U(OH)_4^{2+}$ (= $UO_2 . 2H_2O^{2+}$).[48]

Fairly efficient extraction is usually achieved from solutions with high concentrations of nitrates, which act as "salting-out agents", and in the absence of complex-forming masking agents capable of combining with uranium to form hydrophilic complexes which are not extracted[88]. The most efficient salting-out agents are nitrates which crystallise with water of crystallisation, i.e. those of aluminium, iron, zinc, and calcium. Ammonium and alkali-metal nitrates, which crystallise without water of crystallisation, are less efficient[88]. The salting-out activity of a number of substances has been compared by Vdovenko and others[102–106].

Extraction is possible from weakly acidic solutions and also from concentrated nitric-acid solutions[107]; in the latter case the extraction proceeds even in the presence of phosphate and other complex-forming substances, which do not combine with uranium under these conditions. Certain experimental data indicate that uranium is extracted from strongly acidic solutions not only in the form of nitrato compounds, but also as salts of complex nitratouranate anions and oxonium cations formed by solvent molecules with hydrogen ions[88,108]. The extraction of uranium in the form of its nitrato compounds is the best method for separating uranium from other elements, since the only other metals extracted are thorium, plutonium, and, to some extent, scandium and gold. Other elements are extracted to a very small extent[88].

The extraction of uranium as the thiocyanate is complete, but is less selective than the extraction of nitrato compounds[109]. Uranium can also be extracted from hydrochloric-acid solutions in the form of salts of chlorouranate anions and heavy hydrophobic organic cations such as the tribenzylammonium cation[88,100]. Many elements form complex chloro anions in hydrochloric-acid solution so this method is less selective than extraction of nitrato compounds.

Like many other elements, uranium is extracted in the form of the salts formed with a number of organic precipitants, whose molecules do not contain the hydrophilic SO_3H, COOH, or NH_2 groups. Extraction is possible at pH values at which the salts exist, i.e. pH 3.5 and above, and this method can be used not only with oxygen-containing organic solvents, but also with others such as chloroform[111–116]. This type of extraction, like the corresponding precipitation reactions, is not selective.

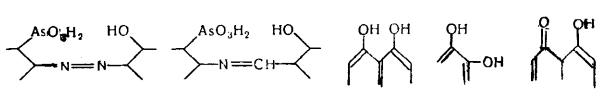
Many workers have developed interesting methods of extraction in the presence of Trilon B, which greatly increases the selectivity of the separation of uranium [109].

Like uranium(VI), uranium(IV) is extracted both as the nitrate and as the chelate compounds formed with the organic reagents 8-hydroxyquinoline, cupferron, and many others. These compounds are formed in more strongly acidic solutions in the case of U(IV) than in the case of U(VI), so that the extraction of the former is more selective, and U(IV) and U(VI) can be separated at low pH values. The advantages of extraction methods for the separation of uranium are the high selectivity, speed, and convenience, and the possibility of working with very small quantities of the element. The thermodynamics of the extraction equilibria for uranyl nitrate have been discussed by Rozen [87]. Various other theoretical problems related to the extraction of elements have been examined by Irving, Nikolaev, and others [88-90,117-119].

The normal redox potential of the U^{4+}/UO_2^{2+} system is -0.32 V, [1,120,121] but UO_2^{2+} ions are reduced rapidly only by very powerful reducing agents, and not by weak reducing agents which might have been expected to do so from their redox potentials. This may be due either to the fact that in actual solutions uranium is not likely to be present as non-hydrated uranyl cations, or to the slow rate of the processes taking place. Slow rates of reduction are characteristic of uranyl ions. In photochemical reactions, uranyl ions are reduced even by such weak reducing agents as ethyl alcohol or diethyl ether, but the reduction is again slow. Uranium(IV) is rapidly oxidised, however, even by weak oxidising agents, and this is used for the volumetric determination of uranium. A number of theoretical problems related to redox methods for determining uranium have been discussed by various authors [1,122,123].

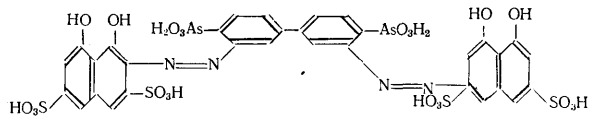
General colour reactions based on the reducing action of U(IV) and U(III) may exist, but none have been described.

Colour reactions for uranium are of two main types, the first based on the chromophoric action of U–O and U–S groups [124-127], produced by interaction between uranium and the colourless or weakly coloured reagents hydrogen peroxide, salicylic acid, nitroso-R salt, thiocyanate, and alkyldithiocarbamates. The formation of coloured simple inorganic compounds, such as perchlorates, phosphates, and others, is also used, but these methods, particularly for U(VI), are not sensitive. In all cases, U(VI) gives a yellow or brown colour, while U(IV) gives green colours. Colour reactions of the second type involve the formation of complexes by uranyl ions and coloured reagents containing various atomic groupings capable of combining with uranium, such as



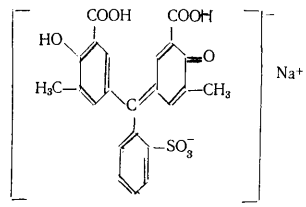
and others [128]. The changes which take place in the reagent molecules as a result of complex-formation lead to a marked deepening of the colour. The mechanism of colour reactions of this type has been discussed [128-147].

Since the colour of the uranyl ion is produced only at intermediate pH values, not less than 4–5, at which other elements also react with these groupings, the selectivity of the reactions is not high. The colour reactions of uranium(IV) take place in more strongly acidic solution and are more selective. The sensitivity of reactions of this type is very high. Interesting reagents of this type include arsenazo I, mentioned above, arsenazo II(IV):



(IV)

quinizarinsulphonic acid, and Eriochrome Cyanine R(V):



(V)

A solid-phase colour reaction [148] has been described for the chlorouranate(VI) anion with anthrazo reagent [149]. Since the complex anion is stable only at high acidity, the reaction is carried out in 6–12 *N* HCl.

The chromophoric action of the uranium(IV) grouping U(IV)...O is less marked than that of U(VI)...O, so that the corresponding colour reactions of U(IV) with colourless inorganic or organic reagents are more sensitive, can be carried out at lower pH values (1–4), and are therefore more selective.

The colour reactions of uranium(IV) with coloured organic reagents are also of interest. They are slightly less sensitive than the corresponding reactions of uranyl ions, but are carried out in much more strongly acidic solutions and are much more selective. Colour reactions of this type for U(IV) resemble those for Th and are carried out under approximately the same conditions with the same reagents, particularly arsenazo and thoron [7,150,151].

Sensitivity and selectivity of the analytical reactions of uranium. Values of the "gamma sensitivity indices" for some reactions of uranium are given in the Table.

Gamma sensitivity index* for some reactions of uranium(VI).

Reagent	Reaction conditions			Gamma sensitivity index
	pH	reagent concentration, M	wavelength, mμ	
Arsenazo	6.0	0.00003	572	0,083
Potassium ferrocyanide	5.0	0.008	458	0,017
Sulphosalicylic acid	9.5	0.08	458	0,003
$H_2O_2 + Na_2CO_3$	9.5	0.018	458	0,0014
Actual colour of uranyl salts	1.8	—	413	0,000025

* The gamma sensitivity index is the difference between the light absorption of the reaction product and that of the same quantity of reagent as is combined in the reaction product. The difference is related to a solution with an element concentration equal to 1 γ/ml and a layer thickness of 10 mm. [152]

The most sensitive and selective reaction is the fluorescent reaction in fused NaF, which permits the determination of 0.001 γ of uranium. The next most sensitive reaction is that with arsenazo, which permits the determination of 0.1 γ of uranium per ml, but which has a low selectivity. Potassium ferricyanide permits the determination of 0.5 γ/ml and sulphosalicylic acid and other organic reagents 1–2 γ/ml.[7]

The spectral method can be used for specimens containing 0.005% uranium, the polarographic method for concentrations of 10^{-3}–10^{-5}% uranium, and oscillographic polarography for concentrations of about 10^{-6}%.[20]

The low stability of UO_2^{2+}–Trilon B complexes and the ability of organic solvents to extract uranyl nitrate are used in various combinations to achieve high selectivity in the determination of uranium[7,153], particularly by the sensitive, but insufficiently selective, reaction with arsenazo[147]. The reactions of tetravalent uranium are much more selective.

B. Reactions of Thorium

Thorium does not form chromophoric atomic groupings and shows an almost invariable oxidation state, so that the element has fewer analytical reactions than uranium or plutonium. No direct redox reactions are known, and colour reactions are possible only with coloured organic reagents[128,129].

In acid solutions thorium forms solvated Th^{4+} cations which undergo stepwise hydrolysis, from pH 2–3 upwards, to form complex solvated +3, +2, and +1 cations. The degree of hydrolysis depends on the pH, thorium concentration, solution composition, and other factors. When the solutions are acidified, the hydrolysed thorium ions are readily reconverted to the reactive Th^{4+} ions. This ready breakdown of the polynuclear ions or oxoions differs from the behaviour of Zr, Ti, and other heavy metals, but resembles the behaviour of uranium, and is particularly favourable for colour reactions.

Thorium ions have a marked tendency to form complexes,, and in the presence of large quantities of nitrate and chloride they form the complex anions $Th(NO_3)_6^{2-}$ and $ThCl_6^{2-}$, which exist in strongly acidic solutions[110]. Both anions are unstable, so that the presence of nitrate or chloride does not interfere with the usual reactions of Th^{4+}. Sulphate, and more particularly fluoride and phosphate, however, form fairly stable complexes with thorium and mask its reactions so completely that a method for the determination of fluorides[154], and extremely sensitive colour reactions for sulphate and phosphate[155], have been based on this property.

In weakly acidic or neutral solutions, Trilon B, oxalic acid, many hydroxycarboxylic acids, and other organic complex-forming substances form stable complexes with thorium[156]. Direct analytical reactions of the colourless hydrophilic anions formed by thorium with these substances have not yet been described.

Precipitation reactions. At approximately the same pH values at which hydrolysis starts (pH 2.5–4) thorium cations are precipitated by many organic reagents which form chelate compounds containing the Th–O bond. These methods can be used to separate thorium from the rare-earth and many other elements which are precipitated only in less strongly acidic solutions.

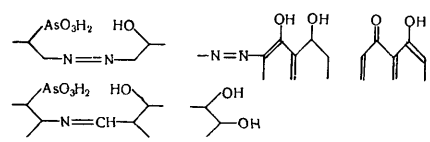
Thorium cations can also be precipitated from strongly acidic solutions as normal salts with various inorganic and heavy hydrophobic organic anions. Thorium forms a sparingly soluble fluoride, phosphate, iodate, molybdate, and tungstate. Organic precipitants include oxalic acid and various monobasic acids with molecular weights greater than 150–200. Arsonic, seleninic, and sulphinic acids are efficient and fairly selective precipitants.

Extraction methods. Although thorium nitrate, $Th(NO_3)_4 \cdot 4H_2O$, dissolves in many organic oxygen- and nitrogen-containing solvents, it can be extracted by only a few of these, and then only in the presence of salting-out agents. It is readily extracted by compounds with carbonyl oxygen — ketones and esters — and less readily by ethers[157–160]. Various metal nitrates are used as salting-out agents, and their efficiency is approximately the same as in the extraction of uranyl nitrate[88,105]. Tributyl phosphate extracts thorium nitrate efficiently without a salting-out agent[3,4].

Mesityl oxide, which is most frequently recommended for the extraction of thorium nitrate, permits separation from the rare-earth and many other elements. Extraction is possible from weakly acidic solutions in the presence of salting-out agents and also from solutions in concentrated HNO_3;[88] in the latter case most of inorganic and organic complex-forming substances do not interfere. Sulphates appear to prevent the extraction of thorium, even from strongly acidic solutions. Thorium is extracted from solutions in concentrated acids to a considerable extent by an oxonium mechanism in the form of a salt of the complex anion, such as $Th(NO_3)_6^{2-}$, and an appropriate oxonium cation[88].

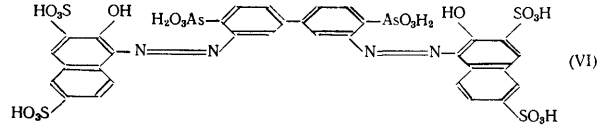
Thorium can be extracted according to general rules as the chelate and simple salts formed by organic complex-forming substances with a heavy hydrophilic molecule[88]. Extraction is possible in the absence of masking agents, at the pH values at which these salts exist. The separation is made selective by keeping the pH as low as possible. Extraction methods appear to be the most selective for the separation of thorium from other elements, and separation from zirconium, titanium, and the rare-earth elements can be achieved in one operation.

Colour reactions. A large number of these have been described. The great majority of reagents used are azo dyes — intensely coloured substances which can act as pH indicators[128–131]. A necessary condition for a colour reaction is the presence of a complex-forming atomic grouping characteristic for thorium, such as



etc.

The most interesting reagents are those containing the arsono group: arsenazo I, arsenazo II, thoron I, and thoron II(VI):[147]



(VI)

These and other reagents containing sulphonate groups form soluble compounds with thorium, but in the presence

of excess thorium suspensions may be formed which appear to consist of simple salts of thorium and a complex anion formed by thorium with the reagents. These suspensions dissolve when sufficient reagent is added.

The colour reactions of thorium with thoron II can be carried out in 4–6 *N* HCl[147], with arsenazo II and thoron I in 0.1–0.5 *N* HCl, and with others at pH above 1.5–2.[136] Since other elements (except Zr, Ti, and Hf) do not give colour reactions with the reagents at such high acidity, the selectivity of the determination of thorium is high. Masking agents such as sulphate, fluoride, phosphate, organic hydroxyacids, etc., interfere with the colour reactions of thorium and as a rule should be removed before the determination.

Arsenazo II and thoron II form very stable complexes with thorium, so that the element can be determined directly in solutions containing large quantities of sulphate, phosphate, and other compounds[147].

The most sensitive reactions for thorium appear to be the luminescent reaction with morin and the colour reaction with aminothoron, which permit the determination of 0.003 γ/ml and 0.1 γ/ml Th respectively. No photometric method for the determination of thorium has been described, but a qualitative test has been reported[147]. Arsenazo and thoron permit the determination of 0.2 γ/ml and 0.5 γ/ml Th respectively. Volumetric methods involving titration with Trilon B, fluoride, and oxalate are suitable for the determination of 0.2–20 mg of thorium.

The selectivity of methods for determining thorium is satisfactory, that of the photometric determination with coloured organic reagents being very high. The selectivity of other methods can be increased by preliminary extraction of thorium nitrate. The precipitation of thorium as the oxalate is fairly selective, and precipitation as the iodate slightly less so. In the presence of complex-forming compounds, thorium is selectively precipitated by phytic acid, a derivative of pyrophosphoric acid[162].

C. Reactions of Plutonium

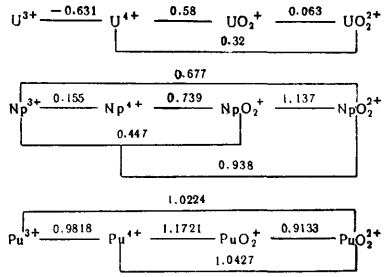
Plutonium exhibits several oxidation states and readily forms various complex ions and chromophoric atomic groupings, so that the analytical chemistry of plutonium is as varied and complex as that of uranium, although comparatively little has been published. Because of the high specific activity of all plutonium isotopes (Pu^{242}, Pu^{240}, Pu^{239}, Pu^{238}, Pu^{236}, etc.), radiometric methods are the most important.

Plutonium ions exist in aqueous solution in oxidation states III, IV, V, and VI, the most stable being the IV state. Many of the properties of Pu^{4+} ions, except the redox properties, are intermediate between the corresponding properties of thorium and zirconium. Like thorium, Pu(IV) has a marked tendency to form complexes. In the presence of nitrate, Pu(IV) forms the extremely characteristic complex nitrato anions, $Pu(NO_3)_6^{2-}$, and possibly other species, including the neutral nitrate[163]. In nitrate solutions with concentrations > 1 M or in 1–4 M HNO_3, almost all the Pu(IV) is present as undissociated $Pu(NO_3)_4$. At higher HNO_3 concentrations, part of the Pu(IV) forms anions of $HPu(NO_3)_5$ and $H_2Pu(NO_3)_6$: in 11 M HNO_3, anions of $H_2Pu(NO_3)_6$ alone are formed[164]. Nitrato complexes of Pu(IV) are fairly stable and for this reason nitric-acid solutions of plutonium have a different colour from its hydrochloric- or perchloric-acid solutions[163].

At high hydrochloric-acid concentrations Pu(IV) forms the complex anion $PuCl_6^{2-}$.[1] In less strongly acidic solutions, from pH 2.5–3.5 upwards, Pu(IV) forms soluble coloured carbonato compounds[165,166], and also oxalato[167,168], tartrato, citrato, ethylenediaminetetraacetato, and other complexes[1,169].

Oxidising agents of intermediate strength, such as elemental bromine, slowly oxidise Pu(IV) to Pu(VI), but oxidation by powerful oxidising agents such as Ce(IV) or MnO_4^- is more rapid. Powerful reducing agents, and hydrogen in the presence of platinum, rapidly reduce Pu(IV) to Pu(III).[1]

The normal redox potentials of systems formed by various oxidation states of Pu, U, and Np are given below*:



When hydrochloric- or nitric-acid solutions of plutonium, free from complex-forming substances, are evaporated to dryness on a water bath with several drops of HBr and then with HNO_3, all other oxidation states are converted to Pu(IV).

Plutonium(VI) forms ions analogous in composition and properties to those of uranium(VI). The colour of PuO_2^{2+} salt solutions varies between yellow and orange-pink[2], but plutonyl nitrate solutions have a greenish colour. Reducing agents such as iodide ions slowly reduce plutonium to the +4 state. Plutonyl-salt solutions are quite stable in air.

Plutonium(III) exists in aqueous solutions as Pu^{3+} ions, which are comparatively stable in air and whose chemical properties resemble those of the rare-earth elements. The colour of the solutions varies between blue and violet[2].

Plutonium(V) exists in aqueous solutions as PuO_2^+ ions. Solutions of Pu(V) in 0.2 M HNO_3 are obtained by the reaction Pu(III) + Pu(VI) $\rightleftharpoons$ Pu(IV) + Pu(V) when solutions of Pu(III) and Pu(VI) are mixed. The reaction is rapid when the mixture is shaken with a solution of tributyl phosphate in benzene to extract the Pu(IV). Pure Pu(V) remains in the aqueous phase, but gradually disproportionates to form Pu(IV) and Pu(VI) ions[1,170–173]. The chemical properties of Pu(V) have been insufficiently studied.

The sensitivity and selectivity of the analytical reactions of plutonium. Radiometric methods using commercial α-counters permit the determination of 0.001–0.01 γ of plutonium. The reactions with arsenazo or thoron permit the determination of 0.1–0.5 γ/ml Pu. Analysis of the peaks in the absorption spectra of plutonium-salt solutions is convenient for the determination of the various oxidation states of plutonium in their mixtures[1,174,175]. Several milligrams of plutonium are required for the determination.

* These values refer to 1 M perchloric-acid solutions (Ed. of Translation).

A feature which distinguishes Pu(IV) from many elements and from other oxidation states of plutonium itself is its ability to form the fairly stable complex anion $Pu(NO_3)_6^{2-}$. The precipitation of Pu(IV) in the form of salts of this anion is extremely selective. Extraction with ketones is fairly selective: Pu(IV) nitrate is readily extracted while Pu(III) is not. If plutonium is present as Pu(III), U(IV) and Th can be separated by extraction, and after conversion of the Pu(III) to Pu(IV), the latter can readily be separated from all other elements. Other schemes for the selective extraction of plutonium are possible.

2. METHODS FOR THE SEPARATION OF URANIUM, THORIUM, AND PLUTONIUM FROM OTHER ELEMENTS

A. Methods for the Separation of Uranium

Precipitation. Separation methods based on the precipitation of uranium(VI) are not very selective. Hydrolytic precipitation of uranium with ammonia [75,176-178], pyridine, urotropine [179-182], ethylenediamine [183], ammonium benzoate [184,185], and tannin [186-189] achieve separation only from the alkali metals and a number of divalent metals including the alkaline-earth metals, and also give partial separation from the rare-earth elements. The selectivity is not greatly increased by using reagents which precipitate uranium in the form of chelate compounds: cupferron [190-193], 8-hydroxyquinoline [75,194,195], quinaldinic acid [196], α-nitroso-β-naphthol [197,198], arsanilic acid [199], cacodylic acid [200,201], and others [202-208].

The precipitation of uranium as peroxouranic acid $UO_4 \cdot 2H_2O$ with hydrogen peroxide is slightly more selective [4,209,210] and achieves separation from Ag, Al, Zn, and the alkaline-earth elements. Slight coprecipitation of Cu and Pb, and marked coprecipitation of Fe, P, and Si, take place [209]. The precipitation is carried out using 0.01–0.2 M uranium solution and a slight excess of H_2O_2 at pH 2–2.5 and 40–50°. [209,210] According to other data, the solution should be cooled and filtered at 2°. [4] In alkaline medium, H_2O_2 reacts with uranyl ions to form a stable soluble complex which is not broken down by ethylenediaminetetraacetic acid, and this is used in the complexometric determination of the alkaline-earth metals in the presence of uranium [211].

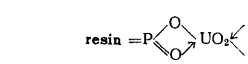
A characteristic and in most cases efficient method of purification uses sodium carbonate with which uranium forms a soluble carbonato complex, $Na_4[UO_2(CO_3)_3]$, while many other elements are precipitated as hydroxides or carbonates [4,7,212,213].

The precipitation of U(VI) by oxoacids of phosphorus [214-219], arsenious acid, potassium ferricyanide, ammonium vanadate, and other precipitants [220-227] is not selective and permits separation only from the alkaline-earth, rare-earth, and a few other elements.

The selectivity of the precipitation of uranium from solutions containing Trilon B is much higher. Methods have been described for precipitation with ammonia [70,75], cupferron [76], 8-hydroxyquinoline [71,73,75,78], and ammonium phosphate [74,216,225], and for electrolytic deposition [226]. Complete separation is achieved from Fe, Cr, Cu, Ni, V, Ce, Bi, and many other elements.

Uranium(IV) may be precipitated using phosphate, fluoride, or cupferron [227,228]. Small quantities are readily coprecipitated with $BaSO_4$, and uranium(VI) and thorium are not coprecipitated [229]. Uranium may be coprecipitated from extremely dilute solutions with Al and Fe hydroxides or phosphates [218 230], with double sulphates of the rare-earth elements [231], or with organic coprecipitants. Kuznetsov and Akimova have developed a method for the coprecipitation of uranium from sea water with the insoluble methyl violet thiocyanate. The limiting dilution of uranium is $1:10^{10}$ (0.1 γ/litre U). The uranium is determined fluorimetrically after separation [84].

Chromatographic methods. Ion exchange is widely used for the technical and analytical separation of uranium. A distinguishing feature of these methods is that even extremely small amounts of uranium may be separated completely from large amounts of impurities [232-254]. The use of complex-forming resins containing phosphinic-acid groups for the separation of very small quantities of uranium from heavy metals is of particular interest. It is reported that complexes of the type



are 10^6 times more stable than the corresponding monomers [254]. Partition chromatography using columns of cellulose [225-258] or silica gel [76] is, in effect, continuous extraction. Paper chromatography is used for work with small amounts of material [259-266]. General problems related to the chromatographic separation of elements have been discussed in reviews by Ryabchikov, Senyavin, and others [232-237].

Extraction methods for the separation of uranium as nitrato compounds are well known. Diethyl ether is most frequently recommended as extracting agent [111,267-274], rather unjustifiably, since the uranium distribution constant for this solvent is small compared with that for other solvents [88], and the inflammability of ether makes its use dangerous. More convenient extracting agents include ethyl acetate [218,275-286], mixtures of tributyl phosphate with other solvents [120,287-295], methyl isobutyl ketone ("hexone") [116,296,297], and many other oxygen-containing solvents [298-302]. After extraction of uranyl nitrate with tributyl phosphate, the uranium can be re-extracted by washing with 5–10% solutions of $(NH_4)_2CO_3$, Na_2CO_3, $H_2C_2O_4$, $(NH_4)_2SO_4$, or CH_3COONH_4. [295]. It is reported that solutions of the organophosphorus compounds $(RO)_3PO$, $(RO)_2RPO$, $(RO)R_2PO$, and R_3PO in CCl_4 are efficient extracting agents for uranium and plutonium [303].

Nitrates of ammonia [273], sodium [304], aluminium [276-280,285], iron, and other elements [102-106] are used as salting-out agents. Extraction from concentrated nitric acid [291] and extraction using triisooctylamine [312] have been much recommended.

A variety of reagents are used for the extraction of uranium in the form of hydrophobic chelate compounds, including cupferron [111,114,228,305], 8-hydroxyquinoline [113,116,306], dibenzoylmethane [307,308], acetylacetone [309-311] dibutyl hydrogen phosphate [207], and sodium diethyldithiocarbamate [112,115,313,314]. Uranium is readily extracted from carbonate solutions as the 8-hydroxyquinolinate by methyl isobutyl ketone [315], and the same solvent also extracts the diethyldithiocarbamate. Methods have been described for the separation of U^{233} from Bi^{212} and Pb^{212}. [300]

Uranium is extracted as anilinium triacetatodioxouranate $C_6H_5NH_3[UO_2(CH_3COO)_3]$ by alcohols and ketones [316]. Apparatus used for the extraction of uranium and other elements is described in monographs by Morrison and Freiser [317], and others [318-320].

B. Methods for the Separation of Thorium

Precipitation. Thorium can be separated from the alkali, alkaline-earth and rare-earth metals, Mn, Ni, Co, Cu, Zn, Cd, and other elements by precipitation with ammonia, pyridine, urotropine, or ammonium thiosulphate [321-328], and from Zr, Nb, Ta, and Sn by precipitation with fluoride [329-332].

The classical oxalate method permits separation of thorium from most elements except Ca, Sr, Ba, and the rare-earth elements [333-341]. A convenient precipitation of thorium is achieved with methyl oxalate, which first dissolves to form a clear homogeneous solution, and is hydrolysed, and precipitates the thorium [339]. According to some authors, however, the precipitation of thorium with oxalate is not always sufficiently complete [335].

Satisfactory separation from many elements, except Zr, Ti, and Ce, is achieved by precipitating thorium with iodate [331,333], various oxoacids of phosphorus [331,342-355] and selenium [356], and many other reagents [357-359]. A scheme has been described for the separation of thorium from other elements using H_2S. [360].

Very small quantities of thorium can be removed from dilute solution by coprecipitation with calcium oxalate [361], basic titanium phosphate [331,354], zirconium phosphate [331,355], bismuth hypophosphate [329,353], yttrium oxalate, and lanthanum fluoride [329,355]. A method has been described for the coprecipitation of thorium with zirconium using the colorimetric reagent for thorium — thoron [362].

Many methods have been described for separating thorium with organic acids and other organic reagents of high molecular weight [363-429], including sebacic [363-365], picric [336], aryloxyacetic [367-371], 4-aminosalicylic [372-374], benzoic [375,376], *p*- and *o*-aminobenzoic [377], chlorobenzoic [371], nitrobenzoic [379,380], hydroxynaphthoic [381,382], 5,6-benzoquinaldinic [383,384], hydroxytoluic [385], and picrolonic acids [386,387], dibenzoylmethane [388], cupferron [321,389], 8-hydroxyquinoline and its derivatives [386,390-393], phenylarsonic [394-395], phenylphosphinic [396], 7-hydroxycoumarin-4-acetic [397], and phenylglycerol-*o*-carboxylic acids [398,399], cinnamic acid and its analogues [400-402], ferron [363,403], mercaptobenzothiazole, phytic acid, and other reagents [363-407,410,414,426,427].

Thorium is separated completely from the rare-earth elements by precipitation with 8-hydroxyquinoline in the presence of Complexone III. [428] It is precipitated from $< 1.0\,N$ HCl solutions with sodium benzenesulphinate, and Be, Al, Ti, Fe, and the rare-earth and other elements do not interfere [429].

Most of the methods differ little as far as selectivity, convenience, or other factors are concerned, since the precipitation conditions depend chiefly on the properties of the Th^{4+} ion and only slightly on the nature of the reagents used.

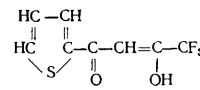
Although methods for the precipitation of thorium are more selective than those for uranium, complete separation of thorium from other elements, particularly those with readily hydrolysable cations, cannot be achieved in one operation, so that it is always necessary to carry out a double precipitation with oxalate, iodate, or, occasionally, phosphate, fluoride, or an organic coprecipitant.

Chromatographic methods can be used to remove thorium from very dilute solutions [430-442], and this is particularly convenient for the analysis of waste liquors, natural waters, and other materials of low thorium content.

A large number of papers are devoted to the separation of thorium from the rare-earth elements [436-442]. A number of cation exchangers absorb thorium and the rare-earth elements completely from almost neutral solutions. The rare-earth elements are then eluted with 1 *N* HCl and the thorium with 3 *N* HCl. [439] Methods involving paper and cellulose column chromatography, which are forms of extraction, have been described.

Extraction of thorium in the form of nitrato compounds is most frequently carried out using mesityl oxide [330,331,340,354] and, less frequently, with other oxygen-containing solvents, such as diethyl ether [443,444], ethyl acetate [157,445], tributyl phosphate [446,447], methyl isobutyl ketone [448] and mixtures of this ketone with tributyl phosphate [912], cyclohexanone [449,450], and other ketones [451-458]. Extremely small quantities of thorium are extracted completely by a mixture of butyl phosphate and methyl acetate [354].

Methods for the extraction of thorium as chelate compounds formed with organic reagents are much less selective. The reagents include thenoyltrifluoroacetone (TTA) (VII), [434,459,460] α-nitroso-β-naphthol [448], acetylacetone, 8-hydroxyquinoline, quinalizarin [488], cupferron [461], quercetin [462], and others [4,88,445,463].



(VII)

C. Methods for the Separation of Plutonium

Precipitation methods have been described chiefly for microquantities of plutonium. Separation from large quantities of other elements is achieved by means of redox cycles [1,464] in which the impurities accompanying plutonium in one or other of its oxidation states show different behaviour when the plutonium is converted to a different oxidation state. Carriers used for the coprecipitation of plutonium include lanthanum fluoride [1,30,464-471], which coprecipitates Pu(III) and Pu(IV) [472], potassium lanthanum sulphate [471,473], certain oxalates and iodates [1], calcium oxalate [467], and bismuth phosphate [470,474-478].

A standard method has been described for the determination of plutonium by coprecipitation with lanthanum fluoride [479]. From strongly acidic solutions, carriers such as zirconium phenylarsonate [465,480] and phosphate [464,477,478] precipitate only Pu(IV), while sodium uranyl acetate precipitates only Pu(VI). [1] Tracer quantities of lanthanides are separated from Pu(VI) by coprecipitation with calcium fluoride [481]. A list of carriers for plutonium in its various oxidation states is given by Hyde [464].

Macro-quantities of plutonium can be separated from some elements by precipitation as hydroxides [470,480,482,483]. The pH values at the start of precipitation of the hydroxides of plutonium cations at concentrations of $\sim 10^{-3}$ M are: Pu(III) ~ 7, Pu(IV) ~ 3, Pu(V) ~ 9, and Pu(VI) ~ 4.5. [282-284] The precipitation of Pu(IV) as the iodate is more selective [467,470,480]. Plutonium can be separated from indium [485], bismuth [486], cadmium [487], and titanium [30] by precipitation as the peroxide at pH 3-4 [480,488], and Pu(III) and Pu(IV) can be separated from uranium, iron, and certain other elements by precipitation of the oxalates in acid medium [1,30,489,490]. Pu(VI) is precipitated as the double carbonates with alkali metals [491,492]. Precipitation of plutonium with 8-hydroxyquinoline gives satisfactory separation from other elements [480,493], and plutonium(III) is precipitated by salicylate at pH 3.5. [30]

The precipitation of plutonium(IV) as various basic salicylates is ~95% complete at pH 4.5 and 99.3–99.9% complete on heating at pH 8. In this case plutonium(IV) is precipitated from solutions containing the element in different oxidation states. Plutonium can also be precipitated with other organic reagents[480].

Chromatographic methods for the separation of plutonium are based on the adsorption of Pu(IV) in its anionic form $PuCl_n^{(n-4)-}$ on Dowex-1 anion-exchange resin at a hydrochloric-acid concentration not less than 2.5 M[464,494,495]. Other actinides in the +4 state are sorbed only at higher HCl concentrations, and separation from uranium is also achieved. Up to 100 mg of plutonium(IV), obtained by oxidising plutonium(III) with a 25-fold excess of sodium nitrite, can be separated completely from most fission products, americium, and other higher actinides, by adsorption on an anion-exchange resin from 7 M nitric acid[496]. Iron appears to be the only common element adsorbed with plutonium[30].

Plutonium can also be separated from other elements with cation exchangers and elution with hydrochloric acid[464] or complex-forming substances such as oxalic acid[169,464,497]. Paper chromatography with n-butanol or ethyl acetate saturated with hydrogen chloride is recommended for the separation of plutonium(III), (IV), and (VI).[490]

Extraction. Like uranium and thorium, plutonium is extracted by many solvents as compounds formed with organic reagents, and from nitrate solutions as salts of a complex nitrato anion by oxygen-containing solvents including diethyl ether, various ketones, and other compounds[30,464,463,498–501]. Separation from most of the lighter elements is achieved in this way. According to Chaikhorskii and Kondratov, extraction with ether separates plutonium completely from neptunium[471]. Dibutyl carbitol extracts Pu(IV) from dilute nitric acid as $HPuO_2(NO_3)_3$.[501] Methods have also been described for the extraction of plutonium(IV) and (VI) with tributyl phosphate[30,464,502–505], solutions of acetylacetone in benzene and other solvents, and TTA.[424,433,460,463–466,464,475,478,506–509]. The extraction of elements with TTA obeys the general laws governing the influence of pH on the tendency of elements to react with reagents of general formula R–OH.[88,510,511] Other readily hydrolysable ions [Pa(V), Fe(III), U(IV), Zr, etc.] are extracted with Pu(IV) from acid solutions. For separation from these elements, the plutonium is reduced to the +3 state, the impurities extracted with TTA, the plutonium in the aqueous layer reoxidised to the +4 state and extracted with a fresh portion of TTA solution in hexone[30].

Plutonium is separated from many radioactive and non-radioactive elements in 1 M nitric acid by extraction with 0.5 M TTA solution in xylene.[479] Further separation from radioactive zirconium, protoactinium, and iron is achieved by re-extracting the plutonium into 10 M nitric acid. Tantalum (0.01–2%) can be separated from plutonium and uranium by extraction with hexone from 6 M sulphuric acid — — 0.4 M fluoride solution[512]. Plutonium can be separated by extraction as amine derivatives[514] and with mixtures of dibutyl ether and carbon tetrachloride[513]. Separation from uranium is achieved by distribution between an organic solvent and a stationary aqueous phase on silica gel[515].

Any extracting agent, for example chloroform, can be used to extract plutonium as the compounds formed with cupferron or neocupferron[464,478,516,517]: in acid medium separation is achieved from uranium, americium, and some other elements. The extraction of plutonium with many other organic reagents has been studied qualitatively[480,518,519].

Other methods. Plutonium is separated from iron and other metals by electrolysis at a mercury cathode[1,30,520]. The element can be deposited electrolytically from weakly acidic, oxalate, or alkaline solutions[470,520–524]. Methods for the deposition of stable coatings of plutonium, neptunium, uranium, and americium have been described[525]. Plutonium is separated from biological materials by mineralisation with hot $HNO_3 + H_2O_2$ mixture[478] and subsequent separation of the plutonium by various methods[478,517]. Filtration of air samples is used to trap plutonium aerosols[526]. The apparatus and necessary precautions for carrying out analytical and other work with plutonium have been described[527,528].

3. METHODS FOR THE DETERMINATION OF URANIUM, THORIUM, AND PLUTONIUM

A. Methods for the Determination of Uranium

Gravimetric methods for the determination of uranium are exactly analogous to the preparation methods described above. Uranium is precipitated by carbonate-free ammonia[176–178], pyridine[180–182], urotropine[179], other organic reagents described above[184,186–208], hydrogen peroxide[4,209,210], ammonium sulphide, and other precipitants[202–208,529]. The precipitates obtained do not as a rule have a definite composition and are ignited at 800°–1050° to U_3O_8.

Careful drying of uranium 8-hydroxyquinolinate at 120°–140° gives a product with composition $UO_2(C_9H_6NO)_2 \cdot C_9H_7NO$. According to Ray and others, precipitation of phosphate or ammonium uranyl phosphate yields the stoichiometric product $U_2(P_2O_7)_3 \cdot 4UO_3$.[217]

Uranium can be precipitated with $K_4Fe(CN)_6$ from 1 M KNO_3 solution at pH 2–4 and 50°–60° as $K_4(UO_2)_4[Fe(CN)_6]_3$,[223] and can also be determined accurately in KNO_3 solutions by amperometric titration with $K_4Fe(CN)_6$.[223]. Uranium(VI) is also precipitated by cacodylic acid $(CH_3)_2AsOOH$: a stoichiometric product is obtained by careful heating at 200°.[200,201]

These methods are not selective, and most elements must be removed beforehand. Little is known of more selective methods for precipitating uranium(IV).

Volumetric methods for the determination of uranium include direct titration of U(VI) with powerful reducing agents, and of U(IV) by oxidising agents, indirect redox methods, titration of uranyl solutions with precipitants, complexometric titration, and others[530–583].

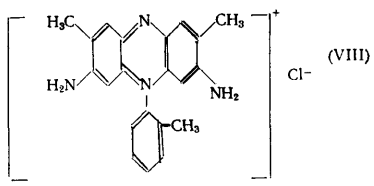
Preliminary reduction of uranium is unnecessary for the direct titration of uranyl solutions with reducing agents, but the instability of the powerful reducing agents used — titanium(III),[530] stannous tin[531], and chromous sulphate[532,533] — reduces the accuracy of the determination.

Methods for the titration of U(IV) are more important. Almost any common oxidising agent can be used, and the choice of titrant and indicator presents no difficulty. Preliminary reduction of the uranium is carried out with powerful reducing agents — titanous chloride[191], stannous tin[531], chromous sulphate[533–536], and solid or liquid amalgams of zinc[538,539], cadmium[540,541], magnesium, aluminium, lead, bismuth, copper, silver, and other metals[533,542–544]. The

reduction is accompanied by contamination of the solutions with ions of the metals used. This does not interfere with the subsequent titration of U(IV), but may be undesirable in some cases. The reduction sometimes yields a mixture of U(IV) and U(III), so that subsequent conversion of U(III) to U(IV) becomes necessary.

An interesting method of reduction involves the use of alcohol. An ethyl-alcohol solution of the uranyl salt, approximately 1 *N* with respect to H_2SO_4, is exposed to direct sunlight or under a mercury lamp for 20–30 min[544]. The uranium is reduced to U(IV) and no foreign ions are introduced. Diethyl ether can be used instead of alcohol[545].

Ceric sulphate[191,434,546-551], potassium dichromate[543,552-554], potassium permanganate[539,553,555-557], ferric sulphate, sodium vanadate[544,545,558,580], and other oxidising agents[4] can be used. Titration with $KMnO_4$ requires no indicator and in other cases the equivalence point may be detected electrometrically[557-559] or with internal indicators such as diphenylamine, diphenylbenzidine, diphenylaminesulphonic acids, etc.[545,548,552,556]. Safranine T(VIII) is used for the oxidation of excess reducing agent by atmospheric oxygen[11]:



(VIII)

The titration of U(IV) is usually fairly easy. When $Fe_2(SO_4)_3$ is used, the titration is carried out in an atmosphere of CO_2 at ~70°. The equivalence point is determined potentiometrically with platinum or calomel electrodes[1,30]. Uranium(IV) may be titrated potentiometrically in the presence of U(VI) with $Ti_2(SO_4)_3$,[582] and a special burette for the titration of uranium(IV) in an inert atmosphere has been described[583].

Other volumetric methods for the determination of uranium have been described, including coulometric titration with electrolytically generated Ti(III) ions[571,572], Ce(IV), bromide ions[573], and other reagents[542,574], precipitation of uranium with 8-hydroxyquinoline followed by titration of the precipitate with bromide-bromate mixture[560-562], titration of uranyl salt solutions with sodium phosphate[564,565] or caustic soda[566,568], complexometric titration with Trilon B[509,510], precipitation of uranium with hydrogen peroxide and subsequent titration of the precipitate with $KMnO_4$,[563] and other methods[538,575-577].

Volumetric methods, particularly redox methods, permit the determination of 5–100 γ of uranium[549,558]. The accuracy of the determination equals, and in some cases exceeds, that of gravimetric methods[558,560,578]. The titration of uranium with Trilon B and quercetin indicator permits the determination of 12 γ of uranium with an error of up to 5%.[510]

Physical and physicochemical methods, although less accurate than gravimetric and volumetric methods, have the advantages of high selectivity and rapidity[20]. These methods include polarographic[262,293,584-601], radiometric[35,230,620-615], neutron-activation[602,616], spectral[36,617], X-ray spectral[618-620], the rapid fluorescent X-ray spectral[34,621-626], the highly sensitive microradiographic[627-629], and other methods[630-635].

Polarographic methods are extremely sensitive and only slightly less accurate than chemical methods, and 0.1–10 γ of U can be determined with an error of not more than 3–5%.[20] The use of oscillographic polarography increases the sensitivity by a factor of ~30.[20] The selectivity of the polarographic methods is satisfactory, but preliminary separation of other substances is necessary in the determination of uranium in minerals, ores[585,588,589,601], and other materials[586-588]. The selectivity of the methods is greatly increased by using Trilon B to mask other elements[591,592] including Ni, Co, Zn, Mn, Al, Cr, Be, and Ti: interference is caused by Pb and Cu.[592] Problems related to the polarographic determination of uranium have been discussed in detail[4,20].

Fluorescence methods. The almost exclusive ability of the UO_2^{2+}–NaF system to give a bright yellow-green fluorescence under the influence of ultra-violet light was first studied by Nichols and Slattery[636-638], and subsequently Hernegger, Fioletova, Leonova and others developed extremely sensitive methods for the determination of uranium[639-669]. The determination is carried out either visually or with photoelectric fluorimeters[640,641] which increase the sensitivity to 0.001–0.005 γ of uranium in 0.5–2 g of melt, with a mean error of not more than 5–7%.[640,642]

The flux may be pure NaF,[4] or a mixture of 9% NaF, 45.5% Na_2CO_3, and 45.5% K_2CO_3,[4] in which case the melts obtained are stable, homogeneous, and non-hygroscopic; these are important factors in the photometric measurements, while the sensitivity is the same as for pure NaF fluxes. Fluorides of other alkali or alkaline-earth metals have been used as fluxes but do not appear to have any advantages over fluxes based on NaF.[4]

The fluorescence of uranyl compounds in liquids such as concentrated H_3PO_4 and H_2SO_4 solutions[643,644], and organic solvents[645], can also be used for the quantitative determination of uranium.

Fluorescence in uranyl compounds is produced not only by ultra-violet radiation, but also by β-, α-, and cathode rays, protons, and probably neutrons[646]; this may be used to determine uranium and also to measure ionising and neutron radiation.

Interference is caused by strong quenching agents such as Fe, Cr, Co, Pb, Mn, Ni, Pt, Ag, La, Ce, and Ca,[640,647-649] and also by elements which on fusion with NaF exhibit fluorescence, such as Ce, V, Nb, Ta, and Sb.[640,649] Detailed references to elements which interfere in the direct determination of uranium have been given[640,649].

The degree of quenching depends only on the concentration of impurity in the flux and is independent of the UO_2^{2+} : : quenching agent ratio. For fluxes containing up to 1 γ of strong quenching agents (Fe, Cr, Ag) and 10–1000 γ of other quenching agents per g of flux, the decrease in the fluorescence of uranium is almost insignificant[640], so that if a small sample is used, in which the ratio of UO_2^{2+} to other elements is 1 : 10^3–10^6 (uranium content 0.1–0.0001%), direct determination of uranium is still possible without preliminary separation of other elements. In this case decomposition of the specimen is also unnecessary and micro-specimens of minerals are fused directly with the flux[650-652].

The analysis of specimens of complex composition and low uranium content requires preliminary separation of most of the other elements present[279,653-655], or concentration of the uranium, e.g. in sea water with organic coprecipitants, followed by fluorimetric determination[84].

The X-ray-fluorescence method permits the determination

of 0.2–2.0 mg/ml U in 1 ml M of $NaNO_3$ solution[627]. According to Galkin and Feofilov, uranium(III) also exhibits fluorescence in single crystals of CaF_2, SrF_2, and BaF_2.[656]

Many uranium minerals exhibit luminescence, which can be used for their detection and identification[657].

Fluorimetric methods for the determination of uranium have been described in a number of papers[4,22,640], and theoretical problems related to the fluorescence of uranyl compounds have been discussed by Vavilov, Levshin, and others[646,658-661]. The problem of the nature of the luminescent centre in the $NaF-UO_2^{2+}$ system has been discussed by Dobrolyubskaya and others[662-664].

Colorimetric methods. Uranium may be determined from the colour of its simple inorganic salts, e.g. uranium(IV) phosphate in 40% H_3PO_4 (λ, 660 mμ),[670] uranyl perchlorate in $HClO_4$ (λ, 415–420 mμ),[671-673] uranyl sulphate in H_2SO_4 (λ, 420–430 mμ),[674-676] and others[540]. A method has been described for the direct spectrophotometric determination of uranium in sulphate or carbonate solutions, based on the characteristic absorption of the complexes in the ultra-violet range. Methods using the colour of uranium salts are highly selective but the sensitivity is insufficient (up to 0.5–5 mg/ml U).

A number of methods have been described for the photometric determination of uranium with oxygen- and sulphur-containing reagents. The simple and convenient method using H_2O_2 in Na_2CO_3 solution is not very sensitive[678-689]. The selectivity is satisfactory, and can be increased further by using a medium of H_2O_2 + NaOH + Na_2CO_3 at lower H_2O_2 concentrations[687]. Interference from V, Mo, and W is almost completely eliminated, but some interference is caused by bicarbonate[679]. A number of authors have studied the composition of the coloured peroxo compounds of uranium[683,684].

In some cases hydrogen peroxide is also used to mask UO_2^{2+} in the complexometric titration at pH 10[211] and in the photometric determination with arsenazo reagents[146].

Uranium may also be determined with KSCN in aqueous solution[690-696], and in mixtures containing acetone, alcohol, or ethylene glycol monobutyl ether[289,697,698]. The selectivity of the method is much higher in the presence of acetone or alcohol; relatively large quantities of Th, Zr, Sn, Hg, Mn as well as sulphate, phosphate and acetate do not interfere; Fe(III) is reduced with ascorbic acid[698] or stannous chloride[697].

An advantage of the thiocyanate method is that the determination can be carried out over a wide range of acidity (0.1–2.0 *N* HCl or HNO_3). The method is insufficiently sensitive (~20–40 γ/ml U). The use of $K_4Fe(CN)_6$ provides a more sensitive method[111,218,699-701], and permits the determination of 10–100 γ of U in 25 ml with an error of 3–5%, but it is insufficiently selective and it is almost always necessary to remove other elements, as well as acetates, oxalates, citrates, and other complex-forming substances[702]. Methods have been described for the determination of uranium in ores, minerals[218], and other materials[111]. At high uranium concentrations, or in the presence of KNO_3, $K_4Fe(CN)_6$ gives a precipitate, which makes the colorimetric determination impossible[703,704]. The composition of the products formed by uranyl ions with potassium ferro- and ferricyanide has been discussed[223,705].

The chromophoric action of uranium also forms the basis of analytical methods using weakly-coloured or colourless organic reagents[124,706-765], including salicylic, aminosalicylic, and sulphosalicylic acids[124,128,706-712], salicylamide[713], salicylaldoxime[714,715], salicylhydroxamic acid[716], Tiron [1,2,4,6-$C_6H_2(OH)_2(SO_3H)_2$]* [717], gallic[732], and chromotropic acids[716-720], dibenzoylmethane and its analogues in aqueous or organic media[285,726-731], 2-acetoacetylpyridine[734,735], R-salt and nitroso-R-salt[721], 8-hydroxyquinoline[116,607,722], ammonium thioglycollate[723-725], reductone[733], isatin-β-oxime[737-743], sodium azide[746], various naphthosulphonic acids[202,747], tannin[182,732], morin[748-751], morellin[752,753], ferron[765], and others[754-765].

All these methods using organic reagents have approximately the same sensitivity (~5 γ/ml). The selectivity is not high, but may be increased using Trilon B to mask interfering elements. According to Pribil, Almashi, and others, Cu, Fe, Ni, Co, Mn, Bi, Al, Th and some other elements do not interfere in the determination of uranium with dibenzoylmethane, morin, or salicylaldoxime in the presence of Trilon B.[70-77]

Methods using coloured organic reagents have recently become more important. Arsenazo, the most widely used[128,143,145,146,540,766,767], is suitable for the determination of both U(VI) and U(IV). The determination of U(VI) is not selective: Th, Zr, Ti, Al, Fe, and the rare-earth and other elements interfere and must be removed. The interference of small amounts of other elements can be eliminated by adding Trilon B or by using the fact that the UO_2^{2+} : : arsenazo complex is decomposed by adding H_2O_2.[146]

The determination of U(IV) is more selective, and only the readily hydrolysable elements Zr, T, and Th interfere. Phosphate, fluoride, and oxalate mask the uranium in both cases. The sensitivity of the reaction is 0.05–0.1 γ/ml U.

U(VI) can be determined with urotropine at pH 4.5–6. The light absorption is measured with a spectrophotometer at 620 mμ or with a photocolorimeter and a red filter. U(IV) is determined in more strongly acidic medium — 0.05–0.2 *N* HCl.[146] Banks and Fritz have described methods for the determination of small amounts of rare-earth elements in uranium[768,769]. Thoron, an analogue of arsenazo, has been proposed for the determination of U(IV).[128] The reaction is also carried out in acid medium (0.1–0.3 *N* HCl) and only Zr, Ti, and Th interfere. The sensitivity of the method is 0.1–0.2 γ/ml U. Arsenazo II has been suggested for the determination of UO_2^{2+}. The sensitivity does not exceed that of the reaction with arsenazo I, but the selectivity is much higher[147].

A number of other coloured reagents have been recommended, including alizarin S and its analogues[770,771], aluminon[772], 1-(2-pyridylazo)-2-naphthol[294,773], and other azo-dyes[128,774]. Nitrilotriacetic acid, KCN, or Trilon B have been used to mask Cu, Zn, Ni, Hg, Fe, Zr, and Th in the determination of uranium with 1-(2-pyridylazo)-2-naphthol[773]. The determination can also be carried out directly after extraction in tributyl phosphate–$CHCl_3$ with the addition of the reagent and pyridine[775,776].

There is an indirect colorimetric method for the determination of uranium, consisting in the oxidation of U(IV) to U(VI) with Fe(III) salts and subsequent determination of the Fe(II) formed with *o*-phenanthroline or dimethylglyoxime[775,776].

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* Tiron is actually the name given to the disodium salt of 1,2-dihydroxybenzene-3,5-disulphonic acid (Ed. of Translation).

B. Methods for the Determination of Thorium

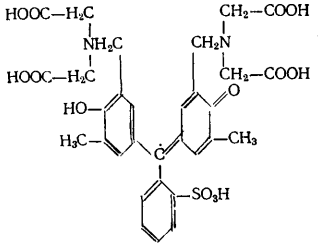
Gravimetric methods for the determination of thorium are based on the precipitation reactions already described. In the hydrolytic precipitation of thorium with bases[321-328] or organic acids[363-426], the metals Zr, Ti, Fe, U and large quantities of rare-earth elements must be absent.

In the absence of rare-earth elements and calcium, thorium is precipitated by oxalate as $Th(C_2O_4)_2 . 6H_2O$[333-341], or by fluoride as $ThF_4 . 8H_2O$.[329] In the absence of Ti and Zr, thorium is precipitated as the iodate[331,333]. In most cases the precipitates obtained are ignited to ThO_2. Precipitation of thorium with phytic acid and ignition of the precipitate yields stoichiometric $Th(PO_3)_4$.[162].

After precipitation of thorium with hydroxyquinoline or mercaptobenzothiazole, the precipitates may be dried at 105°–120° and weighed as $Th(C_9H_6NO)_4 . C_9H_6NOH$ [4,426] and $Th(C_7H_4NS_2)_4$ [777] respectively. Precipitation of thorium with sodium benzenesulphinate and drying at 110° gives stoichiometric $Th(C_6H_5SO_2)_4$.[429] Reviews of gravimetric methods for the determination of thorium have been given[4,29].

Volumetric methods for the determination of thorium are more rapid and convenient. The indirect iodate–thiosulphate method developed by Chernikhov and others is of particular interest[222,355,778,779]. It is based on the precipitation of potassium thorium iodate $4Th(IO_3)_4 . KIO_3 . 18H_2O$ from nitric-acid solution[222,331,333] with subsequent dissolution of the precipitate in acidified potassium iodide solution and titration of the liberated iodine with sodium thiosulphate. The method is fairly selective: Zr, Ti, and large quantities of Bi and the rare-earth elements must be absent.

Recently-developed complexometric methods for the determination of thorium are very rapid and convenient. Trilon B,[780-804] KF,[790,805] or $Na_2C_2O_4$,[790] can be used and the indicators are reagents which give distinct colour changes with thorium: alizarin S,[128-130,780,798] various azo-dyes, particularly those based on chromotropic acid or R-acid[784], 2,7-dinitrosochromotropic acid[785], Eriochrome Cyanine R,[128] xylenol orange (IX),[799,801] ferroin[803], pyrocatechol violet[787,789,800,803], and others[786,788,796]. Physicochemical methods are also used to detect the equivalence point[791,792].



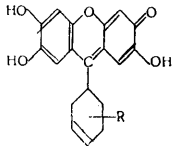
(IX)

A method has been suggested for the determination of micro-quantities of thorium in metallic uranium by coprecipitation with lanthanum fluoride followed by titration with EDTA and Eriochrome Cyanine RC indicator[806]. All elements which react with the complexone, or give colour reactions with the indicators at the experimental pH, interfere. A critical review of complexometric methods for the determination of thorium has been given by Přibil[807].

Other volumetric methods include amperometric titration of thorium with ammonium molybdate[808-810], titration of thorium hydroxyquinolinate with bromine[393], potassium permanganate titration of thorium oxalate[811,812] or the excess oxalic acid after precipitation of thorium[812,813] and others[810,814].

Physical and physicochemical methods include spectral[38,815-820], X-ray spectral[38,619], X-ray fluorescent spectral[623,821], radiochemical[37,612-614,822-824], neutron-activation[825,826], indirect polarographic[827-831], and other methods[4,832]. One indirect polarographic method involves treatment of a standard solution of the ethylenediaminetetraacetato complex of lead with the test thorium solution, followed by polarographic determination of the Pb, which is displaced from the complex by the thorium[831]. An indirect radiochemical method involves precipitation of the thorium with pyrophosphate labelled with radioactive phosphorus and measurement of the activity of the excess pyrophosphate in the filtrate[349].

Thorium can be determined from the fluorescence with various flavones, morin, or, in alcoholic solution, with 1-amino-4-hydroxyanthraquinone[833,834]. Many elements do not interfere, but the reaction is not so selective as the fluorescent reaction of uranyl ions in fused NaF. The complex formed by thorium with 2,3,7-trihydroxy-6-fluorone is decomposed with sulphate in an indirect fluorescence method for the determination of thorium: the liberated reagent exhibits fluorescence[835].



Photometric and colorimetric methods. Most appear to be based on the use of thoron (APANS),[329,330,361,435,459,766,836-849,912] first suggested in 1941 by Kuznetsov[143,145]. The error in these methods is 1.5–2%. The determination is carried out at pH 0.7–1.2: UO_2^{2+}, Fe(II), Al, Pb, and other elements do not interfere, but Zr, Ti, and anions which mask thorium cause difficulty.

Thorium may also be determined with arsenazo[449,450,540,766,850,851]. The reaction is more sensitive, but the influence of other elements is more marked. Zaikovskii and Gerkhard have described a method for determining thorium with arsenazo in the presence of Zr and Ti, which are masked with tartaric acid[852].

Other reagents proposed for the determination of thorium include alizarin[853-856], quinalizarin[857], methyl red[858,859], quercetin[434,462,860,861], morellin[862], nitroso-R-salt[863], pyrocatecholsulphonephthalein[864], morin[865], Carmine Red[866], 2,7-dinitrosochromotropic acid[867], hematein[868], 2,5-dihydroxyquinone in methanol[869], various azo-dyes, mostly based on chromotropic acid[870-874], and other reagents[875-877]. The sensitivity of these methods is 1–10 γ/ml Th, and interference is shown by the same elements as in the case of arsenazo and thoron, i.e. Zr, Ti, Fe(III), large quantities of Al, UO_2^{2+}, and rare-earth elements, and by anions such as sulphate, phosphate, and fluoride which mask thorium.

Indirect colorimetric and nephelometric methods[331,345,878-880] have no advantages over direct colorimetric methods and are not widely used.

C. Methods for the Determination of Plutonium

Gravimetric methods. Plutonium is generally determined gravimetrically as the dioxide[480], obtained by igniting plutonium hydroxide[480], or the precipitates obtained with organic compounds[480,489,490,493] or H_2O_2.[480,483]. Thermogravimetric study of many plutonium compounds has shown that plutonium dioxide is formed above 600°–650°.[881] The properties of plutonium dioxide have been studied[882,883], and a recent detailed study has shown that the weight of the product obtained at 900° differs from the theoretical by between +0.1% and +0.5%.[884] The weight of plutonium dioxide obtained by ignition at a given temperature is constant. The weight corresponds closely to the stoichiometric composition PuO_2 after ignition at 1100°–1200° in air. At higher temperatures lower oxides are formed[885,886]. In some cases, plutonium dioxide may be oxidised to $PuO_{2.1}$. Plutonium(IV) may also be determined gravimetrically as the sulphate or iodate[480].

Volumetric methods. These are based chiefly on redox reactions. Pu(III) is titrated in sulphuric-acid medium with cerium(IV), potentiometrically or with ferroin indicator[887]. The error in the determination of 2–10 mg of plutonium is 0.1–0.25%. It is reported that plutonium may be determined by titration with potassium permanganate, or by thiosulphate titration of the iodine liberated from iodide by plutonium(IV) in sulphuric acid[480].

Plutonium may be oxidised to the +6 state by perchloric acid followed by reduction with excess Fe(II), which is then titrated potentiometrically with standard Ce(IV) sulphate solution[30,34]. This method permits the determination of 400–500 mg of plutonium with an error of 0.03%. Iron does not interfere, but Cr, V, and Mo should be absent.

Plutonium in the +3, +4, and +6 states forms stable complexes with ethylenediaminetetraacetic acid[30,34,888]. The instability constant of the Pu(IV) complex is $\sim 10^{26}$ and that for Pu(III) $\sim 10^{16}$. A volumetric method has been recommended for the determination of Pu(III), based on the back titration of excess complexone with thorium at pH 2.5 in the presence of sodium alizarinsulphonate and methylene blue (internal light filter)[889]. The error in the determination of 10 mg of Pu is ~1%.

Physical and physicochemical methods. The radiochemical method appears to be most widely used[30, 34,509,890-893]. Various methods for preparing specimens for α-counting have been described[893-895] including electrolysis[492,520-523]. When less than 0.01 g/litre Pu and high concentrations of other salts are present, preliminary separation of the plutonium is necessary. In other cases the solution is diluted and an aliquot portion evaporated on the target[30,34]. A radiometric method for the determination of Np^{239} and Pu^{239} in a homogeneous reactor has been described[896]. The results of the radiochemical analysis depend on the isotopic composition of the plutonium[30-34], which can be found by measuring the α-particle energy or by mass-spectrometry[30,34,897].

An improved method for the determination of micro-quantities of plutonium in water[898], and a method for the radiometric determination of plutonium after separation by coprecipitation or extraction[479] have been described. Autoradiography may also be used[470].

The oxidation state of plutonium in micro-quantities can be determined by coprecipitation of the different states with carriers[1], or, in milligram quantities, from the spectra of the solutions[899].

According to Allison, the Pu(III) bands at 602 mμ can be used for the spectrophotometric determination of plutonium with an error of 1%[30,34]. According to other data, 8–14 mg of plutonium(III) can be detected with an error of 0.1% by differential spectrophotometry at 565 mμ.[174] The plutonium is reduced by 5% hydroxylamine hydrochloride in 1 M HCl. No interference is caused by a tenfold excess of thorium, uranium, calcium or cerium, by an equal quantity of ferrous iron (in the presence of stannous chloride), or by less than equal quantities of aluminium and zirconium. Connick et al. recommend the use of the following main analytical absorption bands (the molar extinction coefficients are given in brackets): Pu(III) 560 mμ (36.1), 600 (35.3), 900 (19.3); Pu(IV) 470 mμ (49.6), 655 (34.4), 730 (14.6), 815 (19.6); Pu(VI) 833 mμ (~300), 953 (19.9), 983 (8.9).[899] Analogous spectra are characteristic of crystals of plutonium salts[900] The molar extinction coefficients of plutonium in solution depend on the temperatue, pH, and the nature and concentration of anions present[34,899]; the above data relate to solutions in 0.5 M HCl or HNO_3.

For the coulometric determination of plutonium, Dreeben has recommended electrolytic reduction to the +3 state followed by oxidation with electrolytically generated ceric ions in nitric acid–sulphanilic acid medium[901].

Brunstad, Carson, and others have proposed a method for the oxidation of plutonium to Pu(VI) with potassium permanganate, excess of which is removed by heating with formic acid. The Pu(VI) formed is then reduced in sulphuric acid–phosphoric acid medium by electrolytically generated Fe^{2+} ions[902,903]. Plutonium can be detected coulometrically in 0.5–10 mg amounts with an error of 0.5–2%, and 3 γ of plutonium with an error of 5%[31,34]. Milligram quantities of Pu(IV) are determined by reduction with zinc amalgam followed by titration with Ce(IV) sulphate[904]. Plutonium can be determined with an error of 0.1–1% in 0.05–2 mg amounts by coulometry at controlled potential with a mercury or platinum electrode[905,906]. Direct determination of plutonium is possible in the solutions obtained by treatment of nuclear fuel.

The quantitative polarographic determination of plutonium can be carried out using the clearly-defined wave for the reversible reaction in oxalate solutions at pH 3.5–6.[907] Plutonium(III) in hydrochloric-acid solution containing hydroxylamine does not interfere in the polarographic determination of uranium, vanadium, and titanium[30,34]. Methods have been described for the polarographic reduction of Pu(IV) at an Hg cathode in the presence of complex-forming substances[908].

The concentration of plutonium in pure concentrated solutions may be found from the X-ray absorption[30,34], with an error not exceeding 0.15%.

Plutonium can be determined spectrographically using the lines 2972.54 Å, 2994.07 Å, 3000.53 Å, and 3989.78 Å.[891,909]

Colorimetric methods. Only a few have been described. Plutonium(IV) forms brown and red peroxo complexes in acid solutions and reacts with ferron at pH 2.5–8[519]: Pu(IV) can also be determined from the red colour obtained with sodium alizarinsulphonate in formic-acid buffer at pH ~3.[910] Most other elements are removed by electrolysis at a mercury cathode: oxalate, tartrate, and other anions interfere.

Plutonium(IV) in 5–100 γ amounts can be determined with thoron in 0.05–0.25 N HNO_3 or HCl. Interference is caused by U(IV), Th, Zr, to a lesser extent by Ce(III), Fe(III),

U(VI), and by anions which form complexes with Pu. The influence of F^- ions can be eliminated by adding aluminium nitrate. The error in the determination of 100 γ of Pu is ±1% [911].

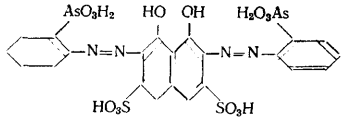
It should be possible, by analogy with U(IV) and Th, to determine Pu(IV) with reagents such as arsenazo I, arsenazo II, and thoron II, but no such methods have been described.

NOTE ADDED IN PROOF

Since this article was prepared, a number of new papers have appeared, and we shall mention the most important of these. A monograph by Lipilina [913] is devoted to the chemistry of uranyl compounds. A review by Ryabchikov and Golbraikh gives data on the chemistry of thorium and its compounds [914], and a fluorimetric method has been described for the determination of uranium in zircon, permitting the determination of 0.0005% of U. [915].

A number of new photometric methods have been published. Uranium can be determined directly from the colour of its compounds in the organic phase after extraction with TTA [916] or a mixture of tributyl phosphate and CCl_4. [917] Everest and Martin have described a method based on the extraction of thorium with tributyl phosphate in isobutyl ketone followed by determination with thoron [918]. The selectivity of the determination of thorium with thoron II is increased by carrying out the determination in strongly acidic solution containing oxalic acid to combine with Zr. [919] A rapid method has been developed for the determination of thorium in monazite using arsenazo II. [920] Zirconium is masked with phosphate, and other elements have almost no effect. Methods have been described for the determination of uranium with arsenazo I [921] and arsenazo II [922]: in the latter method the uranium is separated beforehand by extraction with so-called "fusible extracting agents" — a melt of paraffin and cyclohexanone. The syntheses of arsenazo II and thoron II have been described [925].

The reagent arsenazo III, proposed by Savvin, can be used for the determination of a number of elements, including thorium and uranium [923], with which the reagent forms very stable intensely coloured green complexes.



Solutions of the reagent are pink. The reagent reacts with thorium over a wide range of acidity: 0.01–10 M HCl, HNO_3, or H_2SO_4; with UO_2^{2+} the colour is produced at pH 1–3. Thorium can be determined conveniently in 3–6 *N* HCl in the presence of oxalic acid to mask zirconium. Other elements do not interfere [913]. A method for the determination of thorium in zircon with arsenazo III has been described [924].

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