Radioprotection 1996
Vol. 31, n° 2, pages 229 à 245

Article

Analytical determination of actinides in biological samples

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(Manuscrit reçu le 26 juin 1995, révisé le 12 décembre 1995)

Détermination analytique des actinides dans les milieux biologiques

RÉSUMÉ **Dans le cadre de la surveillance radiotoxicologique du personnel après suspicion de contamination interne par les radioémetteurs alpha, il est essentiel de rechercher et de doser les actinides de manière spécifique dans les milieux biologiques. La technique proposée permet, sur un même échantillon, de mesurer l'activité des isotopes de l'uranium et du plutonium, de l'américium et du curium, en s'affranchissant de l'activité due au thorium et à ses descendants. Cette technique utilise d'une part le pouvoir extractant de nouveaux composés organiques fixés sur des résines inertes et d'autre part les potentiels de fixation et d'élution des divers actinides en fonction de la composition ionique et du pH des solutions utilisées.**

ABSTRACT **When radiation toxicological monitoring of personnel is carried out following suspected internal contamination with alpha particle emitters, it is imperative that specific techniques be used to detect and measure the actinides present in the biological samples. The technique proposed here makes it possible to measure the activity of the uranium, plutonium, americium and curium isotopes on a single sample, without interference caused by the activity which is produced by the thorium and its descendants. This technique uses both the extracting power of new organic compounds fixed on inert resins, as well as the fixation and elution potential of the various actinides depending on the ionic composition and pH of the solutions used.**

1. Introduction

Measurement of the amount of actinides present in the biological media is particularly significant with regard to the monitoring of internal contamination with alpha particle emitters. Following ingestion, these elements are partially absorbed into the bone and liver tissue. Following inhalation they remain in the alveoli of the lungs, clearance varies depending on the particle size. Owing to the shortness of alpha particle tracks in tissue, slight internal contamination cannot be detected *in vivo*, with the exception of americium which can be detected using X-ray spectometry (X-ray at 59.6 keV). Therefore, analysis of excreta remains the key element in measuring the level of internal contamination with actinides.

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For analysis purposes, uranium can be measured using a gravimetric method. However, when uranium of unknown isotopic composition, plutonium or transplutonium elements are present, it becomes imperative to measure the activities of the different isotopes using a radiochemistry technique.

Different radiochemistry techniques have been described using varied substances, usually urine and faeces. They can be used to measure the total alpha activity of a sample but not to establish the activity of each isotope (Eakins and Gomm 1967; Harduin and Peleau, 1993) or specifically measure each radionuclide. To do this, a separate sample must be taken and a different method used for each analysis (Essling *et al.* 1991; Horwitz *et al.*, 1992; Horwitz and Kalina, 1984).

This article describes a laboratory technique which uses a single sample of any substance to measure the volume of each radionuclide and to calculate the activity of each isotope (with the exception of the plutonium 239 and 240 isotopes which are not separate) without interference caused by the activity of thorium and its descendants.

2. Principle

In the case of faecal ash, the solution obtained following reduction to mineral form and the various preparatory chemical treatments takes the form of a 4M hydrochloric acid solution which is yellow in colour due to the presence of Fe^{3+} ions.

In the case of urine, the mineral solution obtained is also a 4M hydrochloric acid solution, to which ferric ions must be added. The iron phosphate precipitate containing the actinides is obtained at pH 4.0 by addition of ammonia and is then dissolved in 8M nitric acid medium. This solution constitutes the working liquor for radiochemical separation. The principle of this separation is based on the use of three columns containing different resins.

– In 8M nitric acid medium, BIORAD AG 1 x 8 anionic resin quantitatively retains the tetravalent plutonium and a portion of the thorium (Ballada *et al.*, 1983; CETAMA, 1966). Specific elution enables the plutonium to be obtained => *source N. 1* (Fig. 1). Then the TEVA.Spec resin made up of a functional grouping (ODDMAX quaternary ammonium salts: octyl-di-decyl-methyl-ammonium) bound to an inert substrate (XAD-7 Amberlite resin) is able to retain the tetravalent radioactive elements in 8M nitric acid (Horwitz *et al.*, in press). Thus it will fix the residual thorium.

– The functional grouping of the TRU.Spec resin used subsequently, is an organophosphorous derivative [(CMPO: octyl (phenyl) -N, N-diisobutyl carbamoyl methylphosphine oxyde)] dissolved in tributylphoshate and bound to an inert substrate (Amberlite XAD-7). In nitric acid medium, this resin is able to extract the actinides of valencies 3, 4 and 6 (Dietz *et al.*, 1993; Horwitz *et al.*, 1990, 1993; Nelson and Fairman, 1990) but in the presence of Fe^{3+} ions, the americium and curium are not extracted (Barney and Cowan, 1994). Plutonium and thorium having been retained earlier, the TRU.Spec resin will thus extract only the hexavalent uranium => *source N. 2* (Fig. 2).

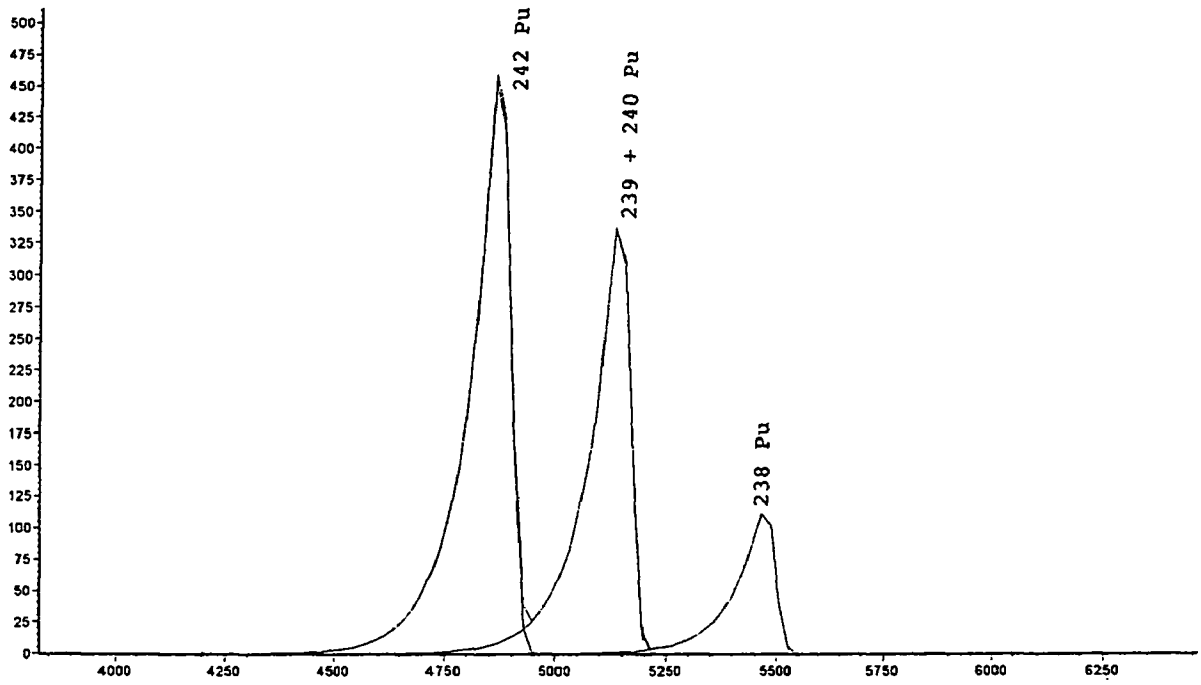


Fig. 1 – Dosage du plutonium, spectre obtenu après déconvolution. Technique : fixation du plutonium sur résine anionique (BIORAD AG 1 x 8).

Plutonium assay, spectrum obtained following deconvolution. Technique: fixing plutonium to anionic resin (BIORAD AG 1 x 8).

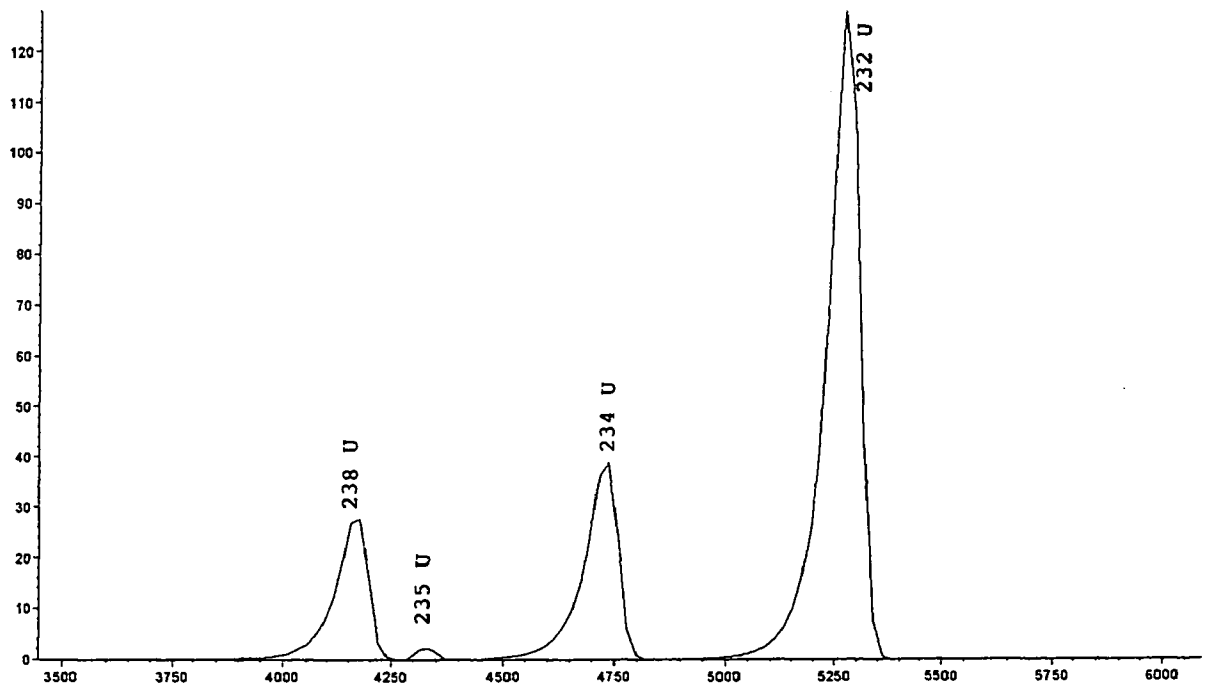


Fig. 2 – Dosage de l'uranium, spectre obtenu après déconvolution. Technique : élimination du thorium et descendants sur résine TEVA puis extraction par CMPO (résine TRU.Spec).

Uranium assay, spectrum obtained following deconvolution. Technique: elimination of thorium and its descendants in TEVA resin, then extraction using CMPO (TRU.Spec resin).

– The trivalent americium and curium are finally extracted using the same TRU.Spec resin following regeneration, but under different analytical conditions. These are: a 2M nitric acid medium, containing EDTA and aluminium nitrate (Barney and Cowan, 1994) => *source N. 3* (Fig. 3).

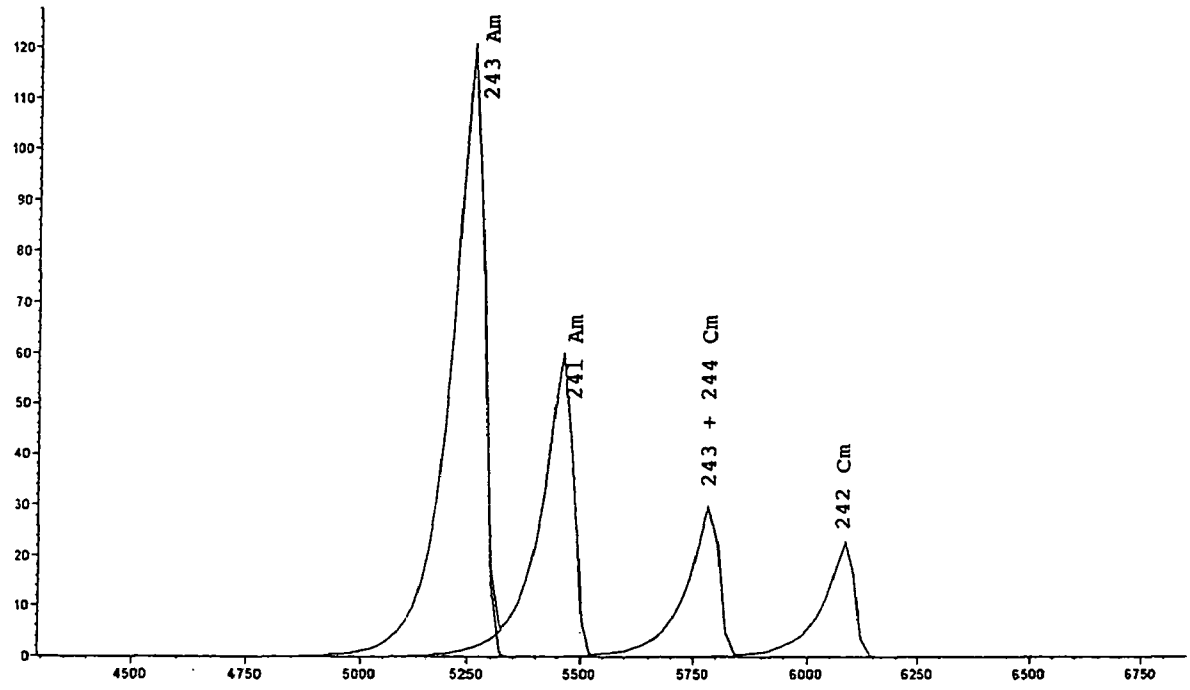


Fig. 3 – Dosage de l'américium et du curium, spectre obtenu après déconvolution. Technique : extraction par CMPO après élimination des autres actinides (résine TRU. Spec).

Americium and curium assay, spectrum obtained following deconvolution. Technique: extraction using CMPO following elimination of the other actinides (TRU.Spec resin).

3. Materials and methods

3.1. Reagents

The TEVA and TRU.Spec resins are supplied by EICHROM Industries[1], and take the form of ready-to-use mini columns. The DOWEX-type anionic resin comes from the company BIORAD[2]. This is a 200-400 mesh BIORAD AG 1 x 8 resin, in the Cl^- form. It must be appropriately prepared prior to use.

The reagents in solution are prepared from extremely pure chemical products, and the water is supplied by a MILLIPORE brand Milli-Q system.

1. EICHROM Industries, 8205 S. Cass Avenue, Suite 107 Darien, IL 60561, USA.
2. BIORAD, 94-96 rue Victor Hugo, 94200 Ivry-sur-Seine, France.

The internal tracers used, ^{242}Pu, ^{232}U and ^{243}Am, are supplied by AEA Technology, Harwell U.K. The dilutions to be added are prepared in an 8M nitric acid medium using a quality assurance protocol and are tested by alpha counting after electrodeposition.

3.2. Equipment

• Electrodeposition set, type EDP 7000 (supplier: SDEC[1]):
– TACUSSEL-type electrolysis electrode, PT XM 150 platinum disk with screw-on head; regulated direct electrolysis current, adjustable from 0.1 A to 5 A; fixed connector (cathode): a polished steel disk, 20 mm in diameter.

• Alpha spectrometry:
– Equipment used: ALADIN-type semi-conductor alpha spectrometer; implanted and passivated junction silicon detectors. (Supplier: Eurisys Mesures[2], Area: 450 mm^2, thickness: 100 µm.)
– Settings: distance between source and detector: 5 mm. Count efficiency ~ 24%, number of channels: 512.
=> Spectra deconvolution programme: INTERALPHA. (Supplier: Eurisys Mesures.)

• Counting time: dependent on the activity.

• Resolutions obtained: 60 keV with co-precipitation, 50 keV with electrodepositing (The short distance between source and detector is the "limiting factor" which prevents better resolution).

• Detection limit: around 1 mBq/actinide for a 4,000 min count time.

3.3. Analytical procedure

See appended technical sheet describing the operation method.

4. Results and discussion

4.1. Results

In comparison with conventional radiochemical techniques, this new technique has enabled some significant improvements to be made:

• detection limits have been reduced because each radionuclide is analysed over the whole sample. In conventional techniques, several aliquots need to be used to measure each of the radionuclides. Indeed, the alpha spectrum obtained by a technique of measuring total actinides does not enable the ^{238}Pu to be separated from the ^{241}Am. In addition, in the case of faecal ash analysis, the

1. SDEC, 19 rue Edouard Vaillant, 37000 Tours, France.
2. Eurisys Mesures, ZIRST 4403, 23 chemin des près, 38944 Meylan Cedex, France.

presence of numerous natural alpha emitters gives a complex spectrum (Fig. 4). This spectrum cannot be exploited when artificial alpha radiation emitters and added internal tracers are present,

- analysis is carried out stage-by-stage by one single technician,
- all the operations involving passing through columns can be carried out using gravity,
- the resin columns used are ready to use or are very simple to prepare and are disposable,
- with the exception of the final phase of extracting the americium, the solution can be passed through the first three resin columns in series,
- the alpha spectra obtained are easy to exploit, free from interference from the thorium,
- different matrices can be tested. Although this technique is commonly used with urine and faeces, it has been successfully tested on bone ash reduced to mineral form which contains a considerable quantity of calcium salts. It is conceivable that it will be applied to other substances, and, in particular, environmental ones,
- the chemical yield depends on three factors: the extraction power of the resins, the elution capacity of the resins, and the performance of the radiochemical technique.

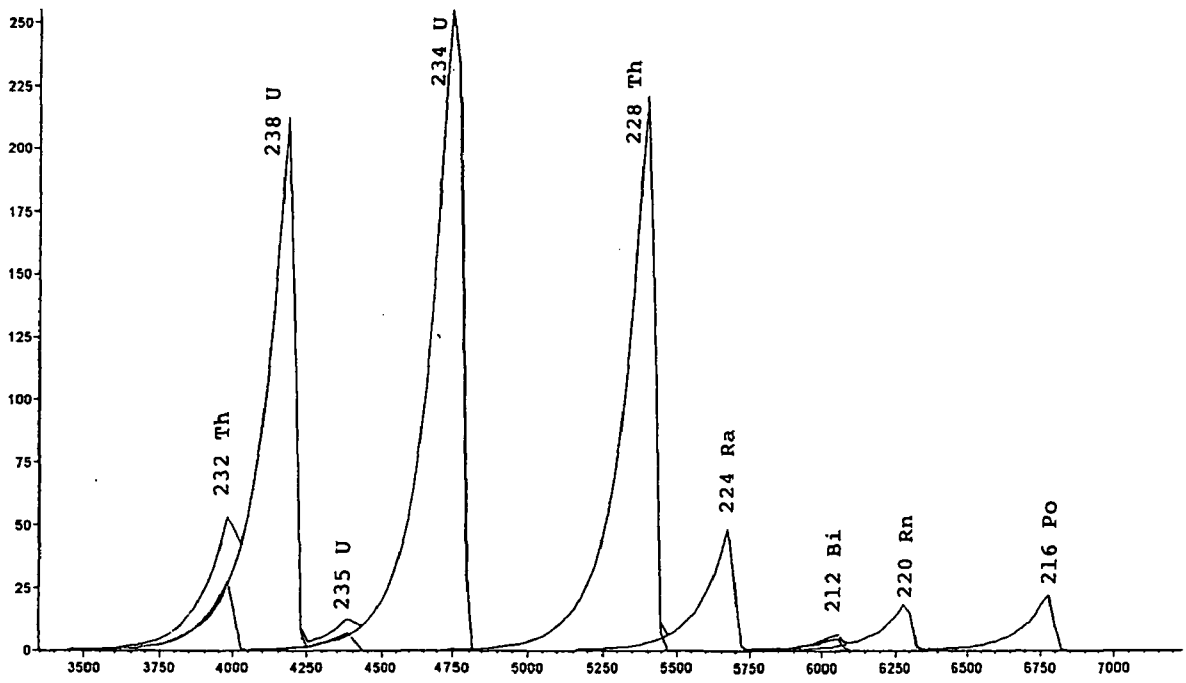


Fig. 4 – Emetteurs alpha naturels d'une selle standard, spectre obtenu après déconvolution. Natural alpha emitters in a standard faecal ash sample, spectrum obtained following deconvolution. Technique: extraction of actinides using CMPO (TRU.Spec resin).

Allowing for a minimal yield loss at the extraction and elution stages, the chemical yield depends mainly on the quality of the radiochemical processes. In the course of this technique, the sample must pass through four columns of resin, which is unusual in conventional techniques and requires particular care to be taken. However, other radiochemical processes are simple. If the operating conditions are strictly observed, i.e. volume and normality of solutions both in loading and rinsing, use of disposable materials etc., then the chemical yield that may be expected should be particularly high and constant. In practice, yield is around 80 to 90% for plutonium and uranium and 70 to 80% for americium.

4.2. Comments and Discussion

4.2.1. Technical discussion

The presence of Fe^{3+} ions

The Fe^{3+} ions present in faecal ash are an essential feature of the radiochemical process of this technique. Indeed, Horwitz (US Int. Cont. 1985) showed that in an 8M nitric acid medium and in the presence of Fe^{3+} ions, the uranium was extracted by the CMPO from the TRU.Spec resin, but not the americium or the curium. However, the addition of aluminium nitrate to a nitric acid medium led to the extraction of americium and curium from the same resin. This phenomenon is enhanced by the addition of EDTA.

Americium and Curium Measuring Phase

Before being passed through the TRU.Spec column, the solution must be evaporated to dryness, then the resulting residue must be dissolved in an aluminium nitrate solution in a nitric acid medium. Usually, a cloudiness can be seen after being put in solution. However, this does not disturb the flow of the solution through the column. Nevertheless, it is preferable to clarify the solution by filtering. Elution of the americium and curium is carried out using 2M hydrochloric acid which, at this level, possesses greater elution power than the ammonium bioxalate solution.

Assay of Plutonium and Transplutonium Elements in the Faecal Ash

Uranium assay is often of no real use. In the case where only the plutonium and transplutonium assays are required, it suffices, in the operating technique, to add the ^{242}Pu and ^{243}Am tracers, to pass the solution through the BIORAD resin to retain the plutonium and then following retention of the residual thorium to the TEVA column, to pass the solution directly through the TRU.Spec column. In this way *sources N. 1 and 3* are obtained.

Specific Assay of the Uranium in the Faecal Ash

In the case where only activity produced by uranium is required, it is possible to add only the ^{232}U tracer to the initial solution, to retain the thorium on a TEVA resin column, then to extract the uranium with a TRU.Spec resin to obtain *source N. 2.*

Assay of Plutonium and Transplutonium Elements in Urine

Add the ^{242}Pu and ^{243}Am tracers to the urine sample. Following reduction to mineral form and dissolution in the nitric acid medium, *do not add iron,* but 40ml of concentrated ammonia. Cover the beaker with a watchglass and let the precipitate settle over several hours. Decant the supernatant, spin the precipitate in a centrifuge and dissolve it in 40 ml of 8M nitric acid. Following addition of sodium nitrite, pass the solution through the Dowex resin column and elute the plutonium => *source N. 1.*

Then, load the solution on a TEVA resin column which will extract any thorium which may be present. Evaporate the solution, dissolve it into 30 ml of the aluminium nitrate solution in a nitric acid medium and load it onto the TRU.Spec resin column. Elute the americium and the curium using 10 ml of 2M hydrochloric acid => *source N. 3.*

Specific Assay of Uranium in Urine

Add the ^{232}U tracer to the urine sample. Reduce to mineral form in accordance with the method for precipitating phosphates. Put the residue into an 8M nitric acid solution. Load this solution on a TEVA resin to extract the thorium and its descendants, then on a TRU.Spec resin to extract the uranium => *source N. 2.*

Assessment of the Thorium in the Faecal Ash

The technique proposed here can also include thorium determination. Indeed, it is sufficient to recover the hydrochloric solution which was passed through the BIORAD column before elution of the plutonium, and to add to it the eluate of the TEVA resin (20ml of 2M hydrochloric acid) then to prepare a *source N. 4* (Fig. 5) by co-precipitating the thorium using lanthanum fluoride or using the electrodeposition technique. The thorium activity can only be estimated in this way because no internal tracer was added, so it is not possible to make any calculations of the chemical yield.

Note 1: *It should be noted that in using this technique, the thorium naturally found in the faecal ash is measured, but so is the thorium contained in the 232U internal tracer.*

Note 2: *229Th may be used as an internal tracer.*

Preparation of the source

The sources for the alpha counters can be prepared by following two different processes: co-precipitation with lanthanum fluoride (Harduin and Montels, 1968) or electrodeposition. From a practical point of view, the two techniques are simple and can be automated. The co-precipitation is a faster and less onerous task than electrodeposition. However, although it must be recognised that the electrodeposition technique results in higher resolution than that obtained after co-precipitation, the spectra obtained by the latter

method remain easy to use. The process adopted in the laboratory is to use co-precipitation of the lanthanum fluoride for routine analyses and electrodeposition for the control analyses.

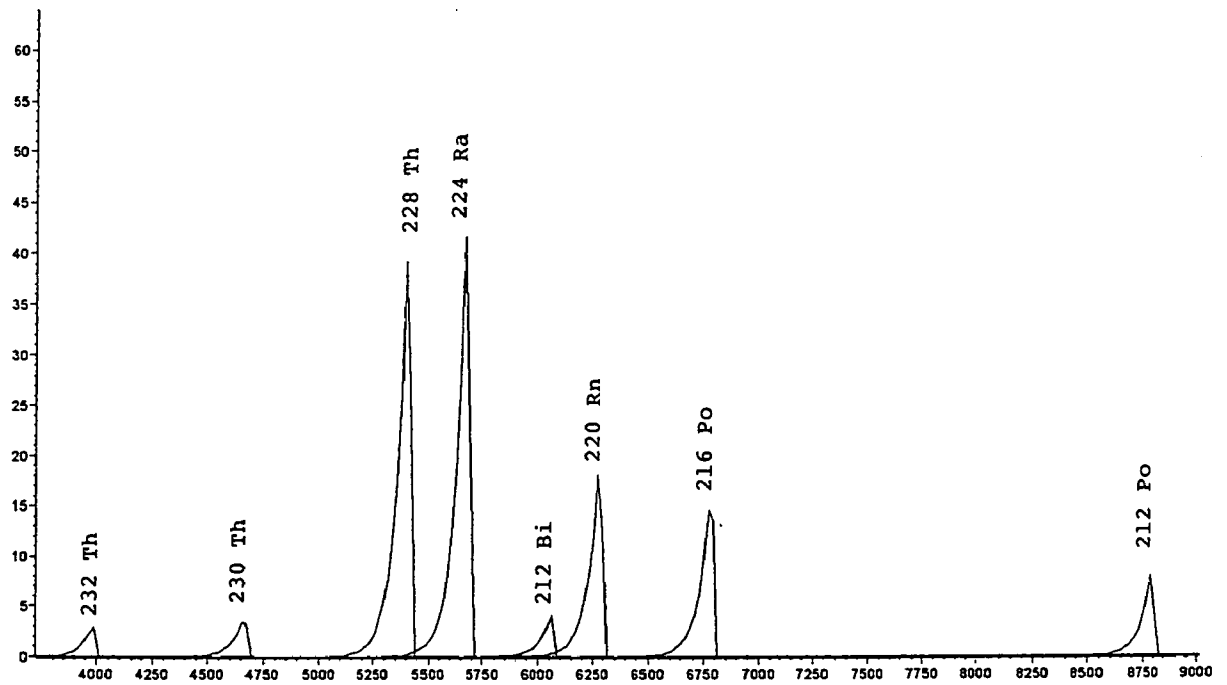


Fig. 5 – Spectre thorium et descendants obtenus à partir d'un échantillon de cendres de matières fécales, spectre obtenu après déconvolution (mesure effectuée après plusieurs semaines).

Spectrum of thorium and its descendants obtained from a sample of faecal ash, obtained following deconvolution (assay carried out after several weeks).

4.2.2. Development

The main development should be to automate the process of passing the solutions through the different resins using robotic equipment.

The quantitative search for thorium in the faecal ash could be of analytical interest and needs to be developed further, in particular with regard to the use of the ^{229}Th tracer.

Appendix. Operating method

The chemical separation technique proposed here can be applied to many substances. The only difference lies in the preparation and reduction to mineral form of the samples to be analysed. The processes for reduction to mineral form described below are applicable to urine and faeces which are the most commonly analysed biological samples.

A.1. Reduction to mineral form

A.1.1. Urine

Pour the sample of volume *V* into a pyrex beaker

Add ^{242}Pu, ^{232}U and ^{243}Am tracers

Note: to fully exploit the alpha spectrum, it is advisable for the activity of the tracer to be of the same sort as the activity of the isotope being sought. In practice, the latter value being unknown, the activity of each of the tracers added may be around 10 to 30 mBq. It should also be noted that in the case of 232U, a monoenergetic peak is obtained at 5.32 keV, the thorium present and its descendants having been fixed by the TEVA resin (Fig. 2).

Then add: *V*/10 ml of 16M nitric acid; *V*/100 ml of 110 vol. hydrogen peroxide; 2 ml of the calcium chloride solution (R1).

Put the beaker on a hotplate set at 300 °C until only a dry residue remains. When cold, add 20 ml of 16M nitric acid. Put the beaker back on the hotplate for 2 hours at 300 °C. Leave it for 5 hours at 500 °C. After cooling, add 100 ml of 2M nitric acid to the perfectly white mineral residue. Cover with a watch-glass and boil for 30 minutes, avoiding significant evaporation. At this stage, a persistent cloudiness can be observed following cooling.

Filter through a 0.45 µm porosity cellulose acetate filter. Pour it all into a 600 ml beaker. Add deionised water to obtain a volume of 500 ml. While stirring magnetically, add 40 ml of concentrated ammonia. Leave the precipitate to settle for several hours and spin in a centrifuge for 10 minutes at 2,500 rpm. Discard the supernatant. Re-dissolve with 50 ml of 4M hydrochloric acid and add 0.2 ml of the ferric chloride solution (R2).

A.1.2. Faeces

Ash the sample for 12 hours in the muffle furnace, gradually increasing the temperature to 700 °C. Weigh the ashes after cooling. Dissolve the mineral residue with a minimum of 16M nitric acid. Evaporate under an infrared lamp. Ash for 4 hours at 700 °C. After cooling, add 15 ml of 4M hydrochloric acid per gram of ash. Heat on hot plate at 200-250 °C for 15 minutes. Spin for 10 minutes in the centrifuge at 3,000 rpm. Collect the supernatant. Dissolve the residue in 20 ml of 6M hydrochloric acid then heat in a sand bath. Spin for 10 min. in the centrifuge at 3,000 rpm and collect the supernatant. Transfer the residue to a platinum cucible. Evaporate until totally dry in a sand bath or under an infrared lamp. Add 10 ml of 40 % hydrofluoric and dry under infrared lamp. Repeat the operation. Re-dissolve with 20 ml of 4M hydrochloric acid. Transfer to a 100 ml centrifuge tube. Spin for 10 minutes at 3,000 rpm and collect the supernatant. Mix together the three lots of hydrochloric acid supernatant collected. Add the ^{242}Pu, ^{232}U and ^{243}Am tracers.

A.2. Co-precipitation of the actinides using an iron phosphate precipitate

Bring the sample, made up to 100 ml with deionised water, to pH 4.0 by adding ammonia. (If the sample is too weak the precipitate will not form. In this case add 0.5 ml of 1M orthophosphoric acid and 0.1 ml of the iron perchloride solution (R2) then bring it to pH 4.0). Spin in the centrifuge, discard the supernatant and dissolve the residue in 40 ml of 8M nitric acid.

A.3. Plutonium assay

A.3.1. Loading on the resin

Add approximately 100 mg of sodium nitrite to the sample. Shake for several minutes, and allow to settle for 3 hours. Introduce 1.6 to 1.8 ml of pre-prepared BIORAD resin (R3) into a disposable mini column (height 40 mm, diameter 10 mm). Load the solution onto the column, then add 10 ml of 8M nitric acid, keep all the eluate from the load and rinse steps, which contains the uranium, americium and the curium as well as some of the thorium (Solution A).

Note: it can be beneficial to carry out the loading and the rinsing under vacuum at a flow rate not exceeding 2 ml/min.

Pass two 10 ml rinses of 8M hydrochloric acid though the column. Discard this hydrochloric acid solution which contains the fixed thorium. Elute the plutonium with two 10 ml rinses of a hydroxylamine hydrochloride solution (R4). Collect the eluate in a 100 ml beaker.

A.3.2. Preparation of the Pu source

A.3.2.1. Using electrodeposition

Evaporate the eluate completely. Re-dissolve it with 5 ml of 0.1M ammonium bioxalate (R5). Warm slightly then transfer it into the prepared electrodeposition cell. Repeat this operation until reaching a final volume of 10 ml. Add 2 ml of a 2M ammonia sulphate solution (R6).

Electrodeposition conditions: current set at 1 ampere; time: maximum 3 hours.

Add 1 ml of ammonia, leave for 1 minute. Stop the electrodeposition and empty the cell. Submerge the disk in deionised water then in methanol. Dry the disk under an infrared lamp.

A.3.2.2. Using co-precipitation with lanthanum fluoride

Make the eluate up to 50 ml with deionised water. Bring to pH 1.3 with ammonia. Add 1 ml of the lanthanum chloride solution (R7) and 1 ml of 40%

hydrofluoric acid while stirring magnetically. Leave to settle for 10 minutes. Filter under vacuum through a 0.22 µm porosity cellulose ester filter. Rinse the beaker and the teflon funnel twice with 5 ml of 2M hydrofluoric acid. Dry the filter under an infrared lamp and lay it on a stable substrate.

A.4. Elimination of residual thorium

Load solution A onto a pre-packed TEVA column. Rinse with 5 ml of 8M nitric acid. The solution obtained contains uranium, americium and curium (Solution B).

A.5. Uranium assay

A.5.1. Extraction

Use a pre-packed TRU.Spec column. First load Solution B onto the column then add 5 ml of 8M nitric acid. The solution obtained contains the americium and curium (solution C). Wash the column with 10 ml of 1M nitric acid. Discard this solution. Elute the uranium using 10 ml of an ammonium bioxalate solution (R5).

A.5.2. Preparation of the Uranium source

A.5.2.1. Using electrodeposition

In the electrodeposition cell, add 2 ml of 2M ammonium sulphate (R6) to the eluate. Electroplating conditions: see § 3.2.1.

A.5.2.2. Using co-precipitation of lanthanum fluoride

Make the eluate up to 50 ml with deionised water. While stirring, add 0.1 ml of the titanium trichloride solution (R8) to reduce the uranium to valency 3. Leave to settle for 1 minute. Then add 1 ml of the lanthanum chloride solution (R7) and 1 ml of 40 % hydrofluoric acid while stirring magnetically. See § 3.2.2.

A.6. Americium and curium assay

A.6.1. Extraction

Use a new TRU.Spec column or regenerate the TRU.Spec column having extracted the uranium using 10 ml of 0.1M ammonium bioxalate (R5) then 15 ml of 2M nitric acid. Evaporate Solution C containing the americium and the curium. Re-dissolve it with 30 ml of aluminium nitrate solution in nitric acid (R9). Place a watchglass over it and heat gently on a hotplate. Leave it to cool after the residue has dissolved. Filter the solution through a 0.45 µm poro-

sity cellulose acetate filter. Load the clarified solution onto the TRU.Spec resin column. Wash with 5 ml of aluminium nitrate solution in nitric acid (R9). Flush the column with 10 ml of 1M nitric acid. Elute the americium and the curium with 10 ml of 2M hydrochloric acid.

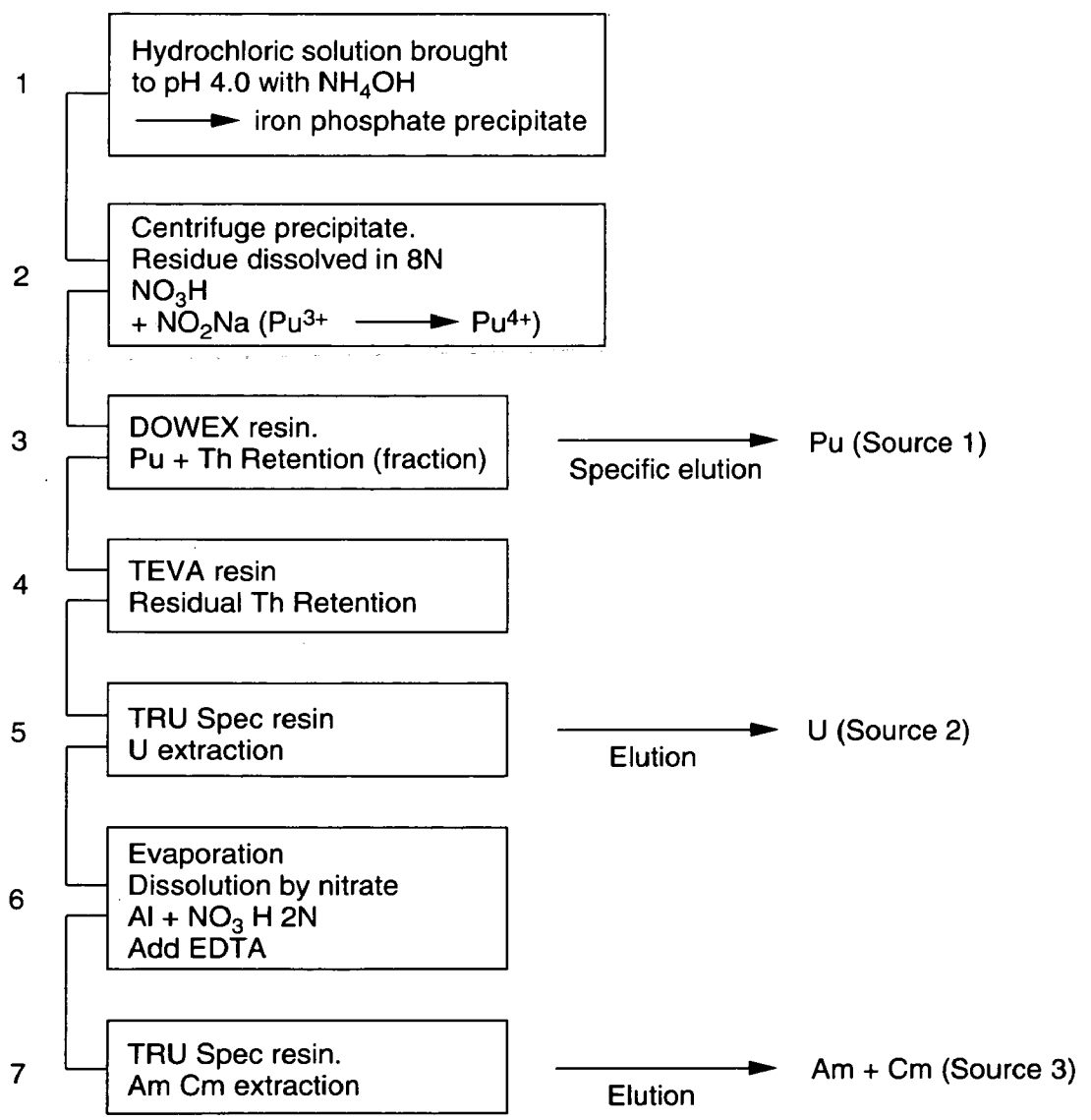
A.6.2. Preparation of Am and Cm Source

Using Electrodeposition: See Pu technique, § 3.2.1.

Using co-precipitation with lanthanum fluoride: See Pu technique, § 3.2.2.

A.7. Alpha spectometry: See equipment section.

A.8. Operational summary diagram



A.9. Reagents

R1
Calcium chloride solution of 88 g/l
- calcium chloride, $2H_2O$ ..... 88 g
- deionised water .....enough for 1 000 ml

R2
Ferric chloride solution, commercial grade, S.G.: 1.26

R3
Ready-to-use BIORAD resin:
Put 100 g of resin in a 1 litre beaker with 800 ml of deionised water.
Stir occasionally over a 24 hour period.
Decant. Add 800 ml of 1M nitric acid.
Stir occasionally over a 4 hour period.
Decant. Add 800 ml of 4M nitric acid.
Stir occasionally over a 4 hour period.
Decant. Add enough 8M nitric acid to cover the resin
Keep the resin in the 8M nitric medium in the dark if not used immediately.

R4
Hydroxylamine hydrochloride solution of 7 g/l
- hydroxylamine hydrochloride ..... 7 g
- 0.2N HCl .....enough for 1 000 ml

R5
Ammonium bioxalate solution (0.1M $NH_4HC_2O_4$)
- oxalic acid, $2H_2O$ ..... 12.61 g
- ammonium oxalate ..... 14.21 g
- deionised water .....enough for around 1 900 ml
Stir to dissolve
- deionised water .....enough for 2 000 ml
Filter through Whatman No. 2 paper

R6
2M ammonium sulphate solution
- ammonium sulphate ..... 264 g
- deionised water .....enough for 1 000 ml

R7
Lanthanum chloride solution at 100 mg of lanthanum/ml
- lanthanum oxide free from alpha activity..... 0.117 g
- 0.2N HCl .....enough for 1 000 ml

R8
Titanium trichloride solution of 15 %
Commercial solution to be used within 1 month of opening bottle only. Keep in the dark.

R9
2M nitric acid solution of 0.5M aluminium nitrate.

1. Parent solution
This solution is prepared from a 2M purified aluminium nitrate solution:
- aluminium nitrate, $9H_2O$ ..... 750.3 g
- deionised water .....enough for around 950 ml
Stir to dissolve
- deionised water .....enough for 1 000 ml
Purify this solution by passing it through a 100-200 mesh BIORAD AG 1 x 8 resin column

2. Working solution
- 2M purified aluminium nitrate solution ..... 500 ml
- concentrated NO_3H ..... 250 ml
- deionised water .....enough for 2 000 ml

A.10. Duration of analytical operations

A.10.1. Reduction to Mineral Form

24 hour old urine (V = 1.4 to 2.2 l) or	28 h
24 to 72 hour old faecal samples (1 to 10 g ash weight)	28 h

A.10.2. Radiochemistry

Co-precipitation	1 h
Plutonium assay	
- LaF_3 technique	from 7 to 12 h
- electrodeposition technique	from 10 to 15 h
Thorium elimination	1 h
Uranium assay	
- LaF_3 technique	3 h
- electrodeposition technique	6 h
Evaporation	2 h
Americium and curium assay	
- LaF_3 technique	5 h
- electrodeposition technique	8 h

Note: as several operations may be carried out simultaneously, the radiochemical technique, including preparation of the source using co-precipitation in LaF_3, can be completed in 17 to 24 hours and in 20 to 27 hours if the source is prepared using electrodeposition.

A.10.3. Counting

The counting time varies considerably depending on the activity level of the source and on the detection limit desired. When activity levels are low, (around 1 mBq on the source), the counting time is at least 72 hours.

A.10.4. Summary

In practice, the isotopic composition and the alpha activity of an analytical sample can be determined within 5 working days. This time can be notably increased if the activity levels looked for are very low. ■

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