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Determination of actinides in environmental and biological samples using high-performance chelation ion chromatography coupled to sector-field inductively coupled plasma mass spectrometry

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Abstract

High-performance chelation ion chromatography, using a neutral polystyrene substrate dynamically loaded with 0.1 m*M* dipicolinic acid, coupled with sector-field inductively coupled plasma mass spectrometry has been successfully used for the separation of the actinides thorium, uranium, americium, neptunium and plutonium. Using this column it was possible to separate the various actinides from each other and from a complex sample matrix. In particular, it was possible to separate plutonium and uranium to facilitate the detection of the former free of spectral interference. The column also exhibited some selectivity for different oxidation states of Np, Pu and U. Two oxidation states each for plutonium and neptunium were found, tentatively identified as Np(V) and Pu(III) eluting at the solvent front, and Np(IV) and Pu(IV) eluting much later. Detection limits were 12, 8, and 4 fg for ^{237}Np, ^{239}Pu, and ^{241}Am, respectively, for a 0.5 ml injection. The system was successfully used for the determination of ^{239}Pu in NIST 4251 Human Lung and 4353 Rocky Flats Soil, with results of 570±29 and 2939±226 fg g^{-1}, respectively, compared with a certified range of 227–951 fg g^{-1} for the former and a value of 3307± 248 fg g^{-1} for the latter.

Keywords: Sector-field inductively coupled plasma mass spectrometry; Thorium; Uranium; Americium; Neptunium; Plutonium

1. Introduction

Investigation into relevant literature [1–3], has shown that studies into the application of chelation chromatography utilising a substrate dynamically modified with 2,6-pyridinedicarboxylic acid has special separation properties for the determination of uranium and thorium in real samples. These dynamic systems have been successfully used to separate U(VI) from Fe(III), La(III) and Th(IV), illustrating the potential for real sample separations to be performed for the determination of uranium and thorium. A recent paper [4] described the quantification of uranium in certified waters and stream sediments at ng ml^{-1} levels using UV–Vis spectroscopy with post-column reaction (PCR) detection systems utilising asenazo (III). These separations were undertaken using 0.1 m*M* dipicolinic acid in 0.5 *M* HNO_3, and 1 *M* KNO_3 to prevent ion-

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exchange with the column substrate. The retention time and column efficiency could be adjusted by changing the acid concentration. Under these conditions the column substrate was dynamically coated with the chelating reagent (i.e., dipicolinic acid). Dynamic coating of the substrate is thought to occur through a combination of hydrophobic and $\pi-\pi$ interactions between the aromatic group on the dipicolinic acid and the benzene groups on the resin [5]. Eventually, a state of equilibrium is set up between the sorbed layer of dipicolinic acid on the substrate, and in the mobile phase, called dynamic modification.

The aim of this study was to use the dipicolinic dynamic system, with an appropriate substrate, to separate thorium, uranium, neptunium, plutonium and americium from matrix ions, in order to facilitate their determination in environmental samples. Virtually all $+2$ and $+3$ metal ions, such as the lanthanides, aluminium and ferric iron, exhibit minimum retention on the high-performance chelation ion chromatography (HPCIC) system [5]. Hence, it is possible to separate these elements from the actinide elements such that the former elute in, or close to, the solvent front. This is an advantage for the analysis of soils and sediments, which can contain high concentrations of transition metals. The potential of this system to separate analytes under highly acidic conditions is also a useful characteristic for this work, because samples are normally prepared by leaching with strong acid.

Inductively coupled plasma mass spectrometry (ICP-MS) is a detection system ideally suited for the determination of the concentration and isotopic composition of the actinide elements. The principal advantages of advanced ICP-MS instrumentation are speed and sensitivity, with the capability of determining all the actinide elements within a min, at sub fg ml^{-1} levels without preconcentration. However, previous work [6] on polyatomic ion interferences on the determinations of actinide elements has highlighted that, if ^{239}Pu is to be analysed in the presence of a large amount of ^{238}U, then an interference due to $^{238}U^{1}H^{+}$ is likely. Unfortunately, sector-field (SF) ICP-MS is not capable of resolving this interference, for which a resolution of >50000 is required (the sector-field instrument used in this work has a maximum resolution of approximately 10000). Hence, the use of a coupled chromatographic technique for uranium separation is an attractive option. A number of resins have been used for the pre-concentration and separation of the actinides and, recently, a number of very specific chelating resins have become available which are particularly suited to this task. Some extraction procedures and applications of these resins have been addressed by Horwitz [7], and Wyse and Fisher [8] who have reported a potential 3 fg absolute detection limit for plutonium using ICP-MS and TRU-Spec resin (Eichrom). They concluded that results for the determination of ^{239}Pu in urine were comparable to those obtained using alpha-spectrometry. Perna et al. [9] used UTEVA resin (Eichrom) for actinide determinations with detection limits down to low pg g^{-1} levels, and compared the method with earlier studies [10] using CS10 (Dionex) column separations. Twin column extractions have also been used recently [11] combining a Diphonix and either Tru-Spec, UTEVA or TEVA resins (Eichrom) for the final separation. Considering the complexities of sample preparation and separation procedures for the majority of methods currently available, it is considered that application of the system described in this work would be another means of determining the actinide elements with a greater degree of simplicity.

2. Experimental

2.1. Instrumentation

All analyses were performed using a sector-field inductively coupled plasma mass spectrometer (ELEMENT 1, Finnigan-MAT, Germany) interfaced with a high-performance liquid chromatography (Varian 9010 HPLC pump, Surrey, UK). For this work, the SF-ICP-MS was operated in low-resolution mode in which it is capable of very low limits of detection for the actinide elements. The sample injection system used a Rheodyne Model 9010 injection valve (Rheodyne, Cotati, CA, USA). PTFE tubing was used mainly for the sample loop, as this was preferable to polyether ether ketone (PEEK), which is less appropriate for prolonged exposure to high acid concentrations. A second valve after the injection valve was configured to allow intermittent changing of the

mobile phase to 1% nitric acid between sample runs this helped to maintain instrument sensitivity and stability throughout long periods of operation. Data were acquired in transient peak hopping mode, which allows time resolved monitoring of multiple isotopes. Operating conditions are shown in Table 1.

2.2. Analytical column

Columns were prepared using PRP-1 polystyrene–divinylbenzene (PS–DVB) substrate (7 μm, Hamilton, Reno, NV, USA) and PLRP-S (PS–DVB) substrate (5 μm, Polymer Labs., UK), packed into PEEK columns of 100 mm×4.6 mm I.D.

2.3. Reagents

All solutions were prepared using analytical-grade reagents and distilled–deionised water (DDW, Ultra Pure Water, Elgastat Maxima, Elga, Bucks., UK). Nitric acid of Aristar grade (BDH, Poole, UK) was used throughout. It should be noted that uranium was present as a contaminant in the reagents. Acids were purified as far as possible by pre-application of extraction resins as described earlier [6,12,13], which considerably reduced the amount of uranium present as a contaminant. Dipicolinic acid (Aldridge, Dorset, UK) was made up in DDW to 25 m*M*, subsequent dilutions to 0.1 m*M* were made up with diluted nitric acid. Off-column reducing solution was prepared from 0.1 *M* sodium formaldehyde sulfoxylate (Rongalite). A mixed standard solution of individual stock solutions ^{237}Np, ^{239}Pu, ^{241}Am and ^{243}Am (Amersham International, Bucks., UK), was prepared by boiling to dryness in nitric acid and made up in the mobile phase.

Table 1
Operating conditions for SF-ICP-MS

	Finnigan MAT ELEMENT 1
ICP	
Forward power (W)	1100
Plasma gas (l min^{-1})	14.0
Auxiliary gas (l min^{-1})	0.9
Nebulizer gas (l min^{-1})	1.1
Sample flow (ml min^{-1})	0.5–1
Torch	Fassel (quartz)
Nebulizer	Concentric MicroMist (quartz)
Spray chamber	Jacketed cyclonic
Interface	
Sampler	Ni
Skimmer	Ni
Mass spectrometer	
Resolution	400 (low resolution)
Ion masses (m/z)	^{230}Th, ^{232}Th, ^{234}U, ^{235}U, ^{237}Np, ^{238}U, ^{238}Pu, ^{239}Pu, ^{241}Am, ^{243}Am
Data acquisition	
Points per peak	25
Dwell time (ms)	30

2.4. Sample preparation

The certified reference material (CRM) NIST 4351 Human Lung (National Institute of Science and Technology, Gaithersburg, MD, USA) was subjected to a dry and wet ashing procedure as described previously [6,12,13]. It was necessary to digest the whole sample of human lung (approx. 45 g), as required by the certificate, due to inhomogeneity caused by the presence of ''hot particles''.

A microwave leach was performed on 1 g of NIST 4353 Rocky Flats Soil No. 1 (National Institute of Science and Technology). The samples were weighed into microwave bombs, 5 ml of concentrated HNO_3 acid was added, and the bombs were irradiated in the microwave digester (Perkin-Elmer PAAR Physica Multiwave Sample Preparation System), for 6 min at 700 W and 15 min at 1000 W power. Samples were then quantitatively transferred to clean vials and made up to a known mass with approximately 7 g of 2 *M* HNO_3. Reagent blanks were also prepared.

2.5. Calibration

Standard solutions were introduced by flow injection through a 500-μl injection loop on a six-port valve (Model 9010, Rheodyne), into a carrier stream of 0.1 m*M* dipicolinic acid solution in between 0.5 and 1.75 *M* of HNO_3 at a flow-rate of approximately 1 ml min^{-1} and the analyte masses monitored.

2.6. Analysis of samples

The samples were diluted further by mixing

approximately 3 g of sample plus 2 g of 0.1 m*M* dipicolinic acid solution giving a total of 5 g. Samples were then injected onto the column via the 500-μl loop and the analysis was performed.

3. Results and discussion

3.1. Choice of substrate

Initially, two different substrates were tested to ascertain which yielded the best separations, namely Hamilton PRP-1 (7 μm) and Polymer Labs PLRP-S (5 μm). Two separate columns were packed with the substrate, and a 100 fg solution each of ^{237}Np, ^{239}Pu, ^{241}Am and ^{243}Am were injected into a mobile phase of 0.1 m*M* dipicolinic acid in 1.75 *M* HNO_3 at a flow-rate of 0.5 ml min^{-1}. Chromatograms for the Hamilton and Polymer Labs columns are shown in Fig. 1 and Fig. 2, respectively. It is evident from Fig. 1 that uranium (which was present as a contaminant at pg ml^{-1} concentration in the mobile phase,

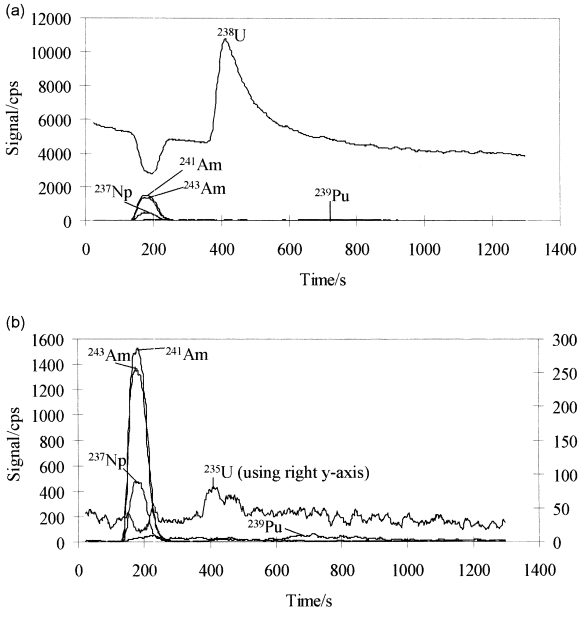

Fig. 1. Chromatogram obtained using the Hamilton column for an approximate 100 fg injection of each actinide with 0.1 m*M* dipicolinic acid plus 1.75 *M* HNO_3 mobile phase at a flow-rate of 0.5 ml min^{-1}: (a) full scale; (b) expanded scale.

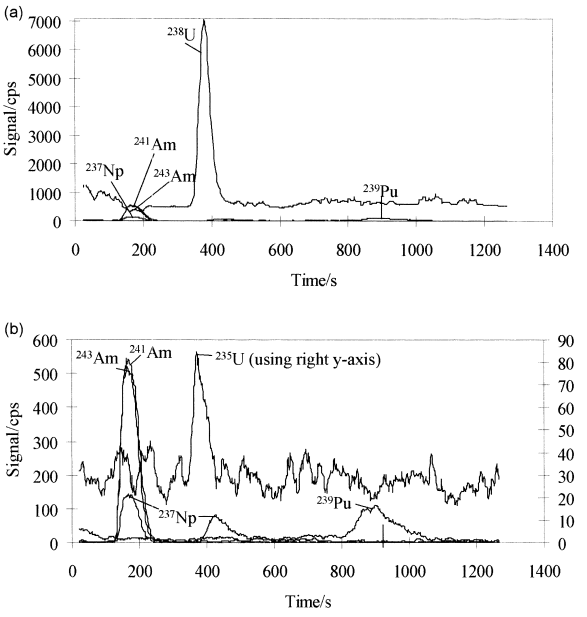

Fig. 2. Chromatogram obtained using the Polymer Laboratories column for an approximate 100 fg injection of each actinide with 0.1 m*M* dipicolinic acid plus 1.75 *M* HNO_3 mobile phase at a flow-rate of 0.5 ml min^{-1}: (a) full scale; (b) expanded scale.

standards and reagents) and plutonium exhibited considerable broadening using the Hamilton column, whereas the Polymer Labs column yielded much improved peak shapes (Fig. 2). The peak shapes for neptunium and americium were much the same for the two columns because these species eluted close to the solvent front in both cases. The improvement in peak shape observed with the Polymer Labs column was thought to be due to the smaller particle size of this substrate. Two separate peaks were observed for neptunium using the Polymer Labs column, probably due to the presence of different oxidation states of neptunium. The presence of two separate peaks of plutonium were also observed, however, these were more visible when the concentration of un-oxidised plutonium was increased (Fig. 3), again, the two peaks are possibly attributed to different oxidation states. In the oxidised standards the peak close to the solvent front disappeared, indicating that this phenomena was not concentration dependent. All further studies were performed using the Polymer Labs column.

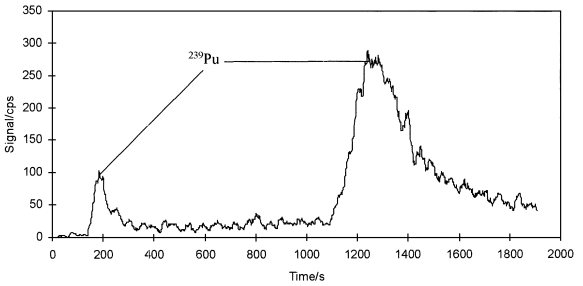

Fig. 3. Injection of approximately 500 fg unoxidised ^{239}Pu with 0.1 m*M* dipicolinic acid plus 1 *M* HNO_3 mobile phase at a flow-rate of 0.5 ml min^{-1}.

3.2. Retention factors (k′) of Th, U, Np and Pu

Fig. 4 shows the log *k′* values for plutonium, uranium, neptunium and thorium as a function of HNO_3 concentration. The dipicolinic acid concentration was constant at 0.1 m*M* throughout the experiment. The curved plots are very similar to those found in previous work on high-valency metal separations [5] and reflect the rather complex relationship between complexation in the eluent and complexation on the substrate.

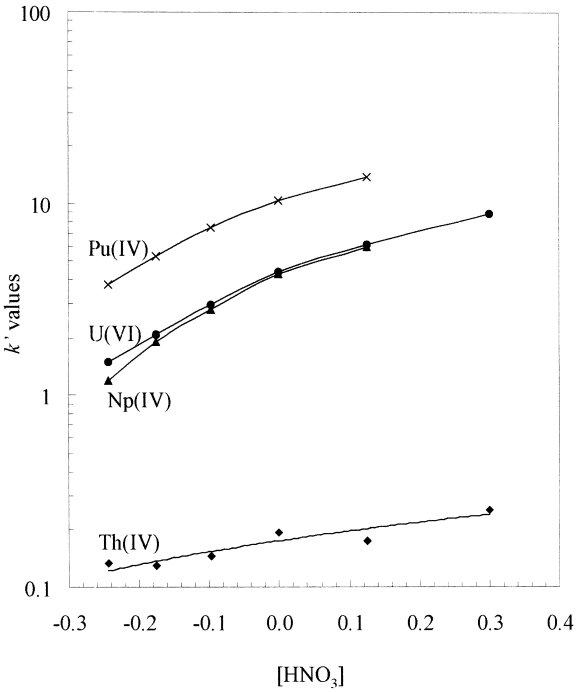

Fig. 4. Log *k′* values versus [HNO_3], for Th, U, Np and Pu using the Polymer Laboratories column with 0.1 m*M* dipicolinic acid mobile phase.

U(VI) and the suggested (IV) oxidation state for neptunium (later eluting peak in Fig. 1b) had very similar retention factors over the range of acid concentrations studied. The second plutonium peak in Fig. 3 [possibly Pu(IV)] had the highest retention factor, indicating that the column could potentially be used to separate ^{238}U from ^{239}Pu to overcome the serious interferences of $^{238}U^1H^+$ on ^{239}Pu commonly observed in ICP-MS when the natural uranium concentration is much higher than plutonium, as is often the case for samples of soil or sediment.

3.3. Limits of detection

Instrumental detection limits for ^{237}Np, ^{239}Pu, ^{241}Am and ^{243}Am are shown in Table 2, with absolute detection limits of between 4 and 12 fg obtained using SF-ICP-MS and a jacketed cyclonic spray chamber. Solutions containing approximately 30 fg of the actinides were used to determine the instrumental detection limits for the method.

3.4. Analysis of reference materials

Initially, NIST 4351 Human Lung was analysed for Pu with a mobile phase of 0.1 m*M* dipicolinic acid plus 1.75 *M* HNO_3, and a flow-rate of 1 ml min^{-1} (unfortunately, ^{241}Am was not analysed, as levels were below the method limits of detection).

Table 2
Instrumental detection limits for the actinide elements, on-column in 0.1 m*M* dipicolinic acid+1.75 *M* HNO_3 (500 μl injections) using SF-ICP-MS with jacketed cyclonic spray chamber

Element	Detection limit	
	Relative (fg g^{-1})	Absolute (fg)
^{237}Np	24	12
^{239}Pu	15	8
^{241}Am	7	4
^{243}Am	8	4

Poor peak resolution was observed, with plutonium barely separated from the uranium hydride peak. The high acidity due to the digestion procedure was thought to be affecting the separation, so the acid concentration in the mobile phase was reduced to 0.75 *M* HNO_3, with a consequent improvement in the separation. A chromatogram for the Human Lung digest is given in Fig. 5 showing the excellent separation of ^{239}Pu from the $^{238}U^1H^+$ interference. It should be noted that the plutonium eluted much sooner than normal due to the high acidity of the sample compared to the mobile phase. The sample was not fully matrix matched because further dilution would have resulted in a sample solution that was below the method limit of detection. However, the change in elution time is not a problem when using an element selection detection method like ICP-MS as long as there is a definite separation of ^{239}Pu from the $^{238}U^1H^+$.

A NIST 4353 Rocky Flats Soil (No. 1) extract was also analysed using these conditions and, as can be seen from Fig. 6 the separation method was still able to resolve the ^{239}Pu and $^{238}U^1H^+$ peaks, despite the elevated concentration of uranium (2.4 μg g^{-1} of ^{238}U) in the sample and a much higher acidity in the digested sample. Results for the determination of ^{239}Pu in NIST 4351 Human Lung and NIST 4353 Rocky Flats Soil (No. 1) are given in Table 3. In the case of the human lung the found concentration for ^{239}Pu fell within the certified range, however, the mean recoveries for the Rocky Flats soil were approximately 11% less than the certified value. In the latter case, the low recoveries could have been

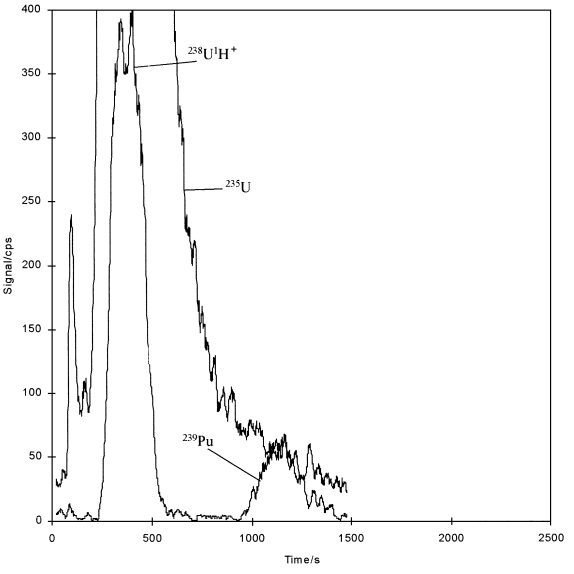

Fig. 5. Separation of ^{239}Pu from uranium, and consequently the $^{238}U^1H^+$ interference in NIST 4351 Human Lung reference material. Polymer Laboratories column with 0.1 m*M* dipicolinic acid plus 0.75 *M* HNO_3 in the mobile phase (^{239}Pu elutes earlier than usual due to a higher acidity of the sample to that of the mobile phase).

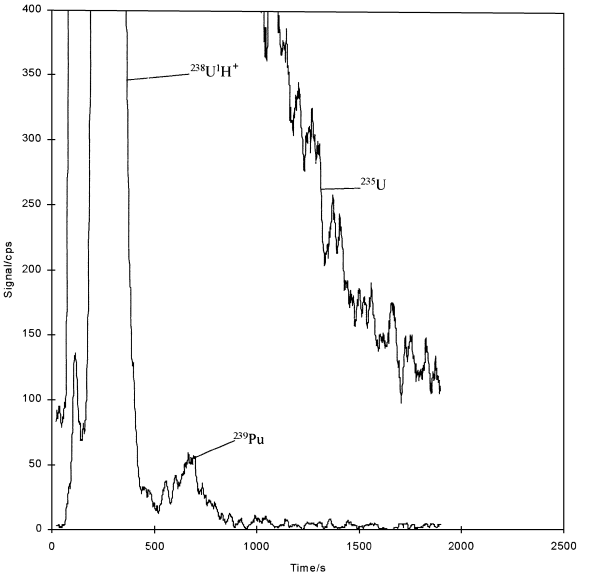

Fig. 6. Separation of ^{239}Pu from uranium and subsequently the $^{238}U^1H^+$ interference for NIST 4353 Rocky Flats Soil (No. 1) reference material. Polymer Laboratories column with 0.1 m*M* dipicolinic acid plus 0.75 *M* HNO_3 for the mobile phase (^{239}Pu elutes earlier than usual due to a higher acidity of the sample to that of the mobile phase).

Table 3
Results for the determination of ^{239}Pu in certified reference materials with HPCIC analyte separation

Material	Certified value[a] (fg g^{-1})	Found[a] (fg g^{-1})
NIST 4351 Human Lung	453 (227–951)[b]	570±29[c]
NIST 4353 Rocky Flats Soil	3307±248[d]	2939±226[e]

[a] Assuming that 6% of the activity was due to ^{240}Pu.
[b] Certificate states 453 with an uncertainty of +110% to −50%.
[c] 95% confidence, $n=1$, three injections.
[d] Certificate states 7.5% uncertainty.
[e] 95% confidence, $n=3$, one injection.

due to incomplete leaching. The certificate states that approximately 8% of the Pu resists HNO_3 leaching, so this is the most plausible explanation. These were excellent results bearing in mind the nature of the sample and the concentration of the analyte that was determined.

3.5. Separation of different oxidation states

It is apparent from the chromatograms that two different oxidation states of neptunium (Fig. 2) and plutonium (Fig. 3) were separated by the column. The chemistry of neptunium and plutonium is somewhat complicated, particularly that of plutonium, as it can coexist in the +3, +4, +5 and +6 oxidation states [14], though, the +5 oxidation state is very unstable in strong acid [15] and tends to disproportionate to the +3, +4 and +6 states [16].

In general, lower oxidation states are more stable in acidic solutions while basic solutions favour the higher oxidation states [17]. Similarly, neptunium can coexist in the +4, +5 and +6 oxidation states in 2–6 *M* nitric acid at approximately 100°C. However, the +5 state should be predominant [18].

A series of experiments using reducing and oxidation agents were performed in order to ascertain the oxidation states of the ions, and it was tentatively concluded that Np(IV) eluted at the solvent front, with Np(V) later, and near U(VI). Plutonium was also thought to be present as Pu(III), eluting near the solvent front, with Pu(IV) eluting much later and, after U(VI). During the experiments it was also noted that uranium probably underwent reduction when sodium formaldehyde sulfoxalate (rongalite) was used as a reducing reagent, this effect being substantiated by other studies in which the reagent was used for reducing U(VI) to U(IV) [19]. These differing oxidation states for plutonium and neptunium have also been observed by other workers using UTEVA resin [9] and TRU-Spec [6,20], with uranium separations being performed by a CG10 (Dionex) column [21].

As can be seen from Fig. 7, without any reducing solution present, the uranium was present as U(VI) and eluted at approximately 550 s. After treatment for 30 min with rongalite a peak appeared at approximately 400 s, probably due to U(IV). It should be pointed out that the mixed oxidation states

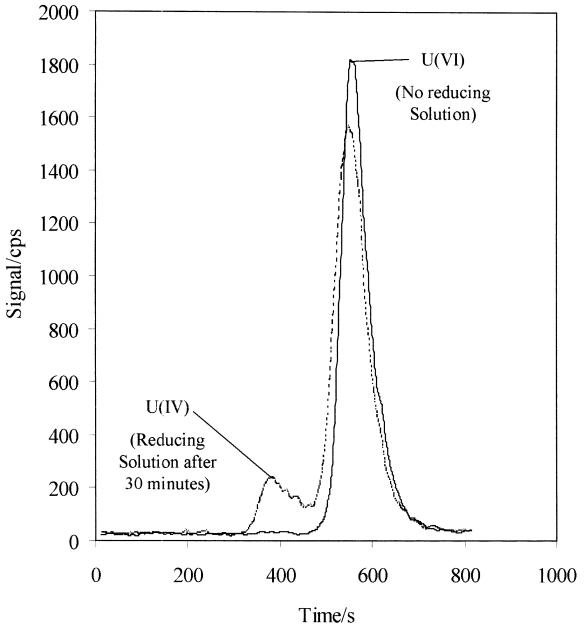

Fig. 7. 0.5 ng injections of uranium (^{238}U) with and without Rongalite reducing solution using the Polymer Laboratories column with a mobile phase of 0.1 m*M* dipicolinic acid plus 0.75 *M* HNO_3 at 1 ml min^{-1}. (N.B. Acquisition performed using less sensitive PQ3 quadrupole-ICP-MS, VG Elemental UK, using standard operating conditions.)

of neptunium and plutonium were found in the standard solution. There was no indication of oxidation state on the containers and it was not possible to find out the standard preparation method. It is expected that the strong oxidising conditions used in preparing the soil and lung samples resulted in only one oxidation state being present. Nevertheless, the preliminary redox studies show that mixed oxidation states could be present depending on the sample type and preparation methods and that workers should be aware of this when developing hybrid chromatographic techniques for the determination of the actinides. Further studies are being carried out on the factors influencing the presence of mixed oxidation states of uranium, plutonium and neptunium.

4. Conclusions

A novel method for actinide determination has been developed based on the coupling of HPLC and

SF-ICP-MS. HPCIC, using a Polymer Laboratories PS–DVB substrate dynamically loaded with 0.1 m*M* dipicolinic acid has been successfully used for the separation of the actinides thorium, uranium, americium, neptunium and plutonium.

When using the column mixed oxidation states were observed for neptunium and plutonium, with redox studies giving some indication of the retention times of the specific oxidation states. Neptunium was thought to be Np(IV) eluting at the solvent front and the Np(V) peak eluting later. Plutonium was thought to be present as Pu(III) eluting near the solvent front with Pu(IV) elutes after U(VI). Rongalite was also found to reduce U(VI) to U(IV) in agreement with other studies [19]. The separation of ^{239}Pu from the interference due to $^{238}U^{1}H^{+}$ at m/z 239 was successfully removed and ^{239}Pu was determined in NIST 4351 Human Lung and NIST 4353 Rocky Flats Soil (No. 1). Results were in agreement with the certified values obtained using the dry and wet ashing and microwave procedures, respectively. The concentration of plutonium in NIST 4353 Rocky Flats soil was found to be 11% lower than the certified value, however, the low result was attributed to the nitric acid digest, as CRM literature suggests an 8% loss of plutonium when using nitric acid extractions.

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