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Determination of lanthanides and actinides in uranium materials by high-performance liquid chromatography with inductively coupled plasma mass spectrometric detection

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Abstract

The determination of Nd, U and Pu by isotope dilution analysis is well known as the classical method for the calculation of the burn-up of a nuclear fuel. Numerous isobaric overlaps restrict the direct determination of fission product and actinide isotopes by mass spectrometry and therefore an extensive chemical separation is required. As a first step towards the development of a more advanced method for the determination of fission product isotopes in irradiated uranium fuel, high-performance liquid chromatographic (HPLC) and inductively coupled plasma mass spectrometric (ICP-MS) systems were installed in glove-boxes and all the lanthanides were separated by HPLC and detected on-line by ICP-MS. As high U and Pu concentrations strongly suppress the signals of trace elements in ICP-MS, a separation method to elute U and Pu first was developed. Thus it was possible to determine the isotopic composition of Nd in a high U and Pu matrix. With the same equipment, a method was developed to prevent isobaric overlaps in the mass spectrum by separating U, Am and Pu.

Keywords: Lanthanides; Actinides; Uranium materials; Inorganic cations

1. Introduction

The demand for the analysis of high-purity materials is growing rapidly because of the wide use of such materials in different industrial applications. The low detection limits for a large number of elements and the possibility of measuring isotopic ratios have established ICP-MS as a powerful multi-element technique in diverse analytical applications for the characterization of nuclear materials [1–3]. One important application is the determination of fission prod-

ucts and actinides in spent U–Pu mixed oxide fuel. The resolution of today's quadrupole mass spectrometer is not sufficient to separate different nuclides with the same mass. Numerous isobaric overlaps due to β -decay and neutron capture restrict the direct determination of fission product and actinide isotopes by mass spectrometry and therefore an extensive chemical separation is required [4]. The lanthanides, with 40%, are the major part of the stable and long-lived fission products of ²³⁵U and ²³⁹Pu irradiated in a light water reactor.

The determination of Nd, U and Pu by isotope dilution analysis is well known as the classical

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method for the calculation of the burn-up of a nuclear fuel [5]. The separation steps to isolate the fission product monitor Nd, Pu and U involve slow classical column techniques, resulting in a great risk for contamination of the sample. Further, the chemist is exposed to a high radioactive dose during the separation steps, which take several days.

As a first step towards the development of a more advanced method for the determination of fission product isotopes in irradiated uranium fuel, all the lanthanides were separated by chelation ion chromatography with a Dionex CS10 cation-exchange column and detected on-line by inductively coupled plasma mass spectrometry (ICP-MS) [6]. High U and Pu concentrations strongly suppressed the signals of trace elements in ICP-MS. For example, the signals of a 20 ng/ml lanthanide solution in a 0.1% U matrix were suppressed by up to 30% of the signals obtained in 1% HNO₃. In addition, deposition of material on the sample and skimmer cone caused serious drift problems [7]. Therefore, an even larger dilution factor from the solid into solution is necessary and this in turn has a negative influence on the detection limits that are achievable. With the separation method described here it was possible to elute U and Pu first. By the introduction of a postcolumn valve, the U and Pu fraction was directly passed to waste and the ICP-MS system was not exposed to large amounts of dissolved solids. As it was possible to inject solutions with U and Pu concentrations up to 1%, very low detection limits for the lanthanides in U–PuO₂ were reached. With the same chromatographic equipment, a method to prevent isobaric overlaps in the mass spectrum by separating U, Am and Pu was investigated.

2. Experimental

2.1. ICP-MS system

Modifications of a commercial ICP-MS instrument (PlasmaQuad PQ2+, Fisons, Winsford, UK) were carried out in order to handle and measure highly toxic radioactive samples in a

glove-box. Connection of ICP-MS with HPLC and flow-injection (FI) systems was established. The interface between the mass spectrometer and the torch-box was connected to the electropolished stainless-steel plate of the glove-box. The sample interface, spray chamber, nebulizer, torch-box, peristaltic pump and waste container were installed in the glove-box. All gas inlets into the glove-box, the vacuum line between the interface and the rotary pump and the ventilation inlet/outlet were equipped with absolute filters in order to prevent possible radioactive contamination. No absolute filters have previously been installed between the quadrupole chamber and the diffusion pumps. Experience over the last 2 years has shown that contamination of the photomultiplier holder assembly can be neglected. The glove-box was connected to the laboratory ventilation system, providing cooling of the torch-box and an exhaust for the plasma gases at the same time. The waste from the spray chamber was pumped into the waste container inside the glove-box. Two independent cooling systems were used, one for the diffusion pumps and the interface (18°C) and the other for the spray chamber (2°C). No significant influence on the performance of the modified instrument by installation in the glove-box was observed. However, at first some degradation of the long-term stability was observed as a result of increasing temperature inside the glove-box up to 32°C. As a consequence, a modification was made to the cooling air inlet. The response was about 5000–10 000 counts per second for 1 ng/ml ¹¹⁵In. The instrumental parameters and measurement conditions are listed in Table 1.

2.2. HPLC system

For all separations a Dionex (Sunnyvale, CA, USA) DX300 HPLC system was used. The advanced gradient pump for pressures up to 5000 p.s.i. was connected to an IonPac CG10 guard column (50 × 4 mm I.D.) and an IonPac CS10 analytical column (250 × 4 mm I.D.). The agglomerated cation-exchange resin of the CS10 column consists of internal core particles of

Table 1
Instrumental parameters and measurement conditions

<i>Instrument parameters</i>				
R.f. power	1350 W			
Outer gas flow-rate	13 l/min			
Intermediate gas flow-rate	0.7 l/min			
Aerosol carrier gas flow-rate	1.0 l/min			
Sample uptake rate	1 ml/min			
<i>Measurement conditions</i>				
Method	A	B	C	D
Measuring mode	Scan	Scan	Peak jumping	Scan
Masses	139–240	139–176	142–146, 148, 150	234–245
Dwell time per channel	0.32 ms	0.32 ms	10.24 ms	0.32 ms
Channels per u	24	24		24
Points per peak			5	
Time slices	6 s	6 s	6 s	6 s

ethyldivinylbenzene cross-linked with 55% divinylbenzene. To the 8.5 μm internal core particle a layer of small-diameter latex particles is attached. Provided that the outer layer carrying the functional sulfonic acid groups is very thin, the agglomerated resin exhibits excellent chromatographic performance owing to the very short diffusion paths available to solute ions during the ion-exchange process [8]. The flow-rate was 1 ml/min. For preliminary optimizations of the chromatographic conditions, postcolumn reaction with 0.3 mM Arsenazo III and photometric detection at 650 nm were used instead of ICP-MS to detect peaks. The flow-rate of the postcolumn reagent was set to 0.5 ml/min.

To couple the HPLC and ICP-MS systems, the HPLC eluate was pumped through a postcolumn valve (Dionex) directly into the ICP-MS system. To handle highly toxic radioactive material, the chromatographic columns, the postcolumn valve and the photometric cell were installed in a glove-box. Furthermore, a Model 401 diluter, a Minipuls 3 peristaltic pump and a Model 232 autosampler (all from Gilson Medical Electronics, Villiers-le-Bel, France) were installed in the same glove-box. The autosampler was equipped with a Rheodyne (Cotati, CA, USA) Model 7010 valve. Fig. 1 shows a schematic view of the coupled system.

2.3. Reagents

Distilled, deionized water (DDW) from a Milli-Q system (Millipore, Milford, MA, USA), 65% HNO_3 (Suprapur) and 48% HF (Ultrapur) (Merck, Darmstadt, Germany) were used for all dissolutions and dilutions. A Spex 1 10 $\mu\text{g/ml}$ multi-element standard solution (Spex Industries, Edison, NJ, USA) and a 1000 $\mu\text{g/ml}$ Nd atomic absorption standard solution (Aldrich, Milwaukee, WI, USA) were diluted with 1% HNO_3 . Uranyl nitrate 6-hydrate (zur Analyse) (Merck) was used to prepare U matrix solutions. DDW, α -hydroxyisobutyric acid (HIBA) (purum) (Fluka, Buchs, Switzerland) buffered to pH 4.5 with 30% ammonia solution (Baker Instra-Analyzed) (J.T. Baker, Philippsburg, NJ, USA) and 30% HCl (suprapur) (Merck) were used as eluents. Arsenazo III (Fluka) and acetic acid (puriss, p.a.) (Fluka) were used as post-column reagents. KMnO_4 (Siegfried, Zofingen, Switzerland) was used to oxidize Pu.

2.4. Sample preparation

Uranyl nitrate was dissolved in 1% nitric acid. Nuclear fuel samples were dissolved in 7 M HNO_3 -HF under pressure at 160°C.

Pu can be found simultaneously in four differ-

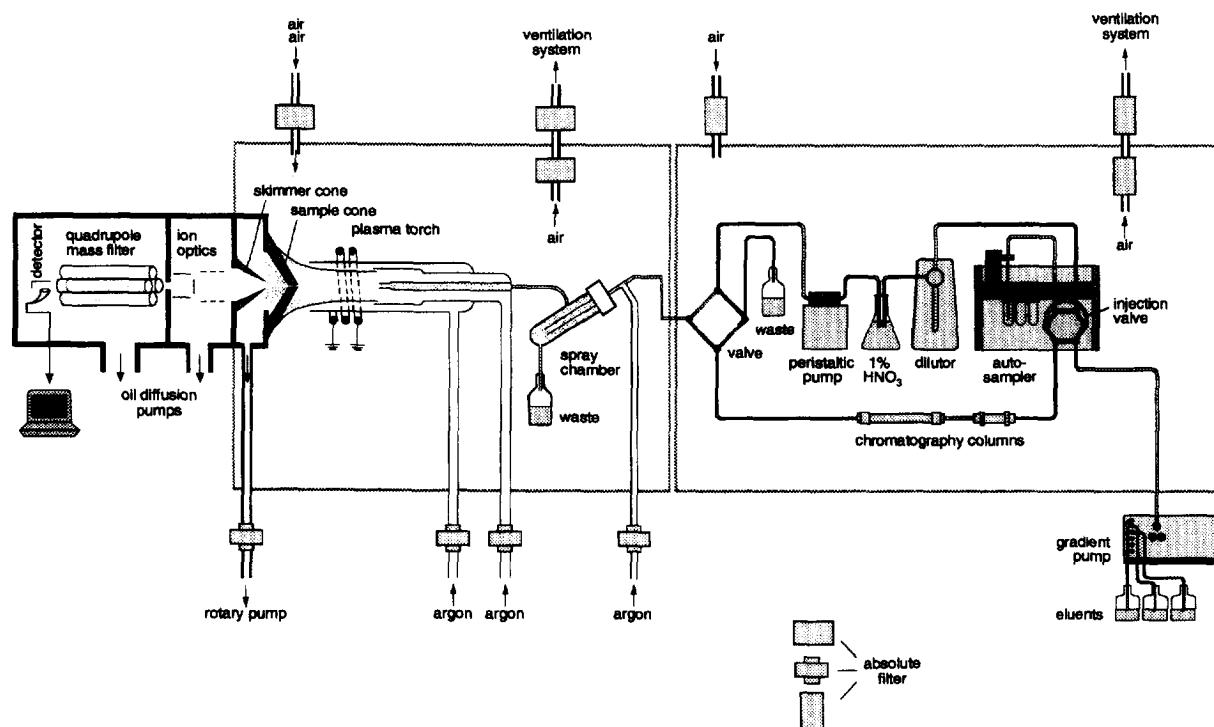


Fig. 1. Schematic view of the coupling of the HPLC and ICP-MS systems. In order to analyse highly toxic radioactive samples, all parts of the system which have contact with the samples are installed in glove-boxes.

ent oxidation states, of which the most stable is Pu^{4+} . As Pu^{4+} elutes as a peak with tailing, Pu was oxidized to PuO_2^{2+} with 0.001 M KMnO_4 – 1 M HNO_3 [9]. The next day the solutions could be diluted without changing the oxidation state of Pu.

2.5. Chromatographic conditions

The chromatographic conditions for the separation of the lanthanides are listed in Table 2. U

Table 2
Chromatographic conditions for the separation of the lanthanides in a U–Pu matrix

Time (min)	DDW (%)	1 M HCl (%)	0.4 M HIBA (%)
0.0	0	100	0
0.1	90	0	10
8.0	90	0	10
26	35	0	65

is eluted with 1 M HCl . The lanthanides are separated with a linear 18-min gradient from 0.04 to 0.26 M of HIBA.

The chromatographic conditions for the separation of the actinides are listed in Table 3. U and Pu are separated with 0.4 M HNO_3 and Am is eluted with 0.26 M HIBA .

3. Results and discussion

3.1. Separation of lanthanides

In aqueous solution, the lanthanides are present as strongly hydrated trivalent cations. Because of their similar ionic properties, they cannot be separated easily by cation exchange as trivalent cations. However, the selectivity of the separation can be increased with the use of appropriate chelating agents such as HIBA. Lanthanides form complexes with HIBA that lower the affinity of the lanthanide for the

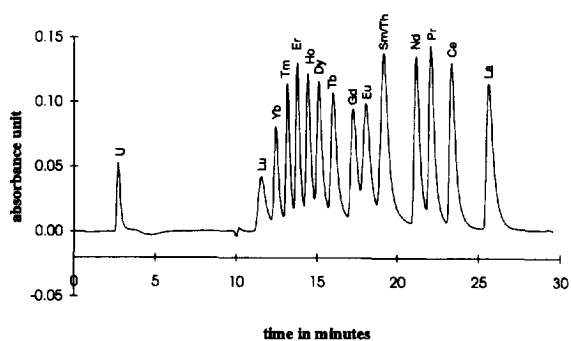


Fig. 2. Chromatographic separation of U, Th and all the lanthanides according to the conditions in Table 2 and photometric detection at 650 nm after postcolumn reaction with 0.3 mM Arsenazo III. Injection of 50 μ l of 1% nitric acid containing 10 ppm of U, Th and of each lanthanide.

cation-exchange resin. Therefore, lanthanides that form the most stable complexes with HIBA, such as Lu, will elute first. Lanthanides such as La, which forms a weaker complex with HIBA, elute later [10,11].

The chromatographic method for the separation of lanthanides was further developed in order to elute uranium first. Using the chromatographic conditions reported in Table 2 and photometric detection at 650 nm after post-

column reaction with 0.3 mM Arsenazo III, the chromatogram in Fig. 2 was obtained by injection of 50 μ l of 1% nitric acid containing 10 μ g/ml of U, Th and each lanthanide.

In aqueous solution U is present as the uranyl cation (UO_2^{2+}) and forms strong complexes with chloride ions. These complexes are not retained on a cation-exchange resin and therefore uranium can be eluted with hydrochloric acid [12–14]. In order to co-elute with UO_2^{2+} , the different oxidation states of Pu are oxidized to PuO_2^{2+} [15,16].

By detecting peaks with ICP-MS, it was possible to determine much lower concentrations than with photometric detection. Using the chromatographic conditions in Table 2, the chromatogram in Fig. 3 was obtained by injection of 1 ml of 1% nitric acid containing 20 ng/ml of U, Th and each lanthanide. For detection, method A in Table 1 was used. Although Sm and Th are eluted together from the column, they were separated by MS because they have different masses. False interpretation because of hidden peaks or several peaks of the same element in different oxidation states could now easily be discovered. This, together with the very low detection limits for a large number of elements,

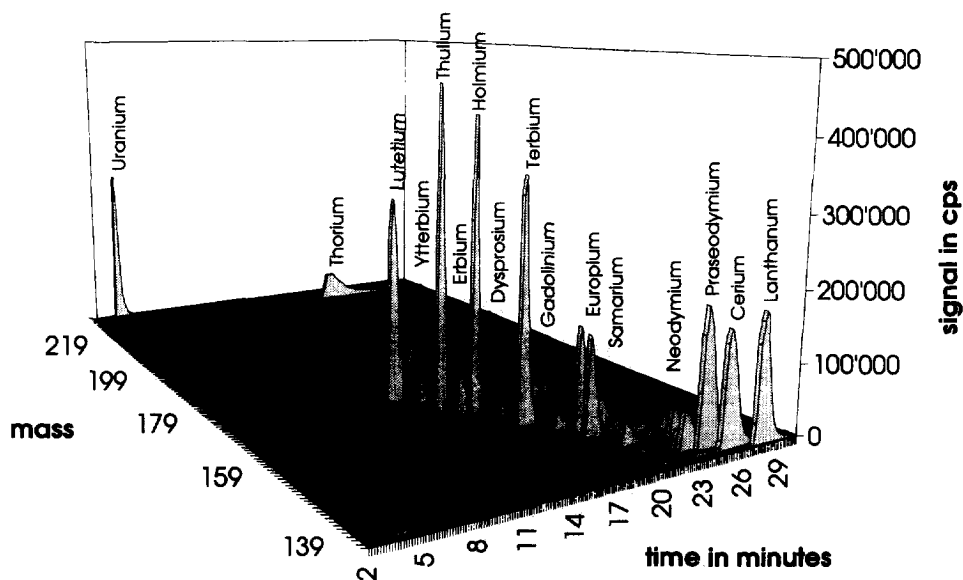


Fig. 3. Analysis of 1 ml of 1% nitric acid containing 20 ng/ml of U, Th and of each lanthanide by HPLC-ICP-MS. Chromatographic conditions as in Table 2 and ICP-MS method A in Table 1.

makes ICP-MS an extremely powerful detection method in the chromatographic analysis of cations.

The determination of traces of lanthanides in irradiated uranium fuel by ICP-MS is hindered by numerous isobaric overlaps and also by the large matrix effect of high U concentrations. In irradiated samples no mathematical correction procedure can be applied owing to the unknown isotopic compositions of the elements. An element-specific separation prevents isobaric overlaps of different nuclides with the same mass and overlaps due to oxide and doubly charged peaks. Signals of a 20 ng/ml lanthanide solution in a 1% uranium matrix were suppressed to about 90% of the signals obtained in 1% HNO₃ and deposition of material on the sample and skimmer cone causes serious drift problems. Therefore, an even larger dilution factor from the solid into solution was necessary and this in turn has a negative influence on the detection limits that are achievable.

Fig. 4 shows the separation of 1 ml of a 20 ng/ml lanthanide solution in a 1% U matrix. With the use of a postcolumn valve, the U fraction was passed directly to waste and so the ICP-MS system was not exposed to large

amounts of dissolved solids. Therefore, as the lanthanide signals were not suppressed by a huge U matrix, very low detection limits were reached [17]. Fig. 4 shows a very good chemical separation of all the lanthanides. As Pm is a radioactive element with a short half-life, the lanthanide solution does not contain any Pm, resulting in a gap between Sm and Nd in the chromatogram.

To obtain better results, only the masses 142–146, 148 and 150 were measured with ICP-MS in the peak-jumping mode (method C in Table 1). Fig. 5 shows the perfect chemical separation between Sm, Nd and Ce. Without chemical separation large isobaric overlaps would disturb the analysis.

3.2. Separation of actinides

Numerous isobaric overlaps due to neutron capture restrict the direct mass spectrometric determination of U and Pu in spent U–PuO₂ fuel. A separation of U and Pu is necessary. Further, the isobaric overlaps between Am and Pu have to be separated. A separation between ²³⁷Pu and ²³⁷Np is not required because of the low concentration of ²³⁷Pu resulting from the short half-life of ²³⁷Pu (45.3 days). To oxidize all

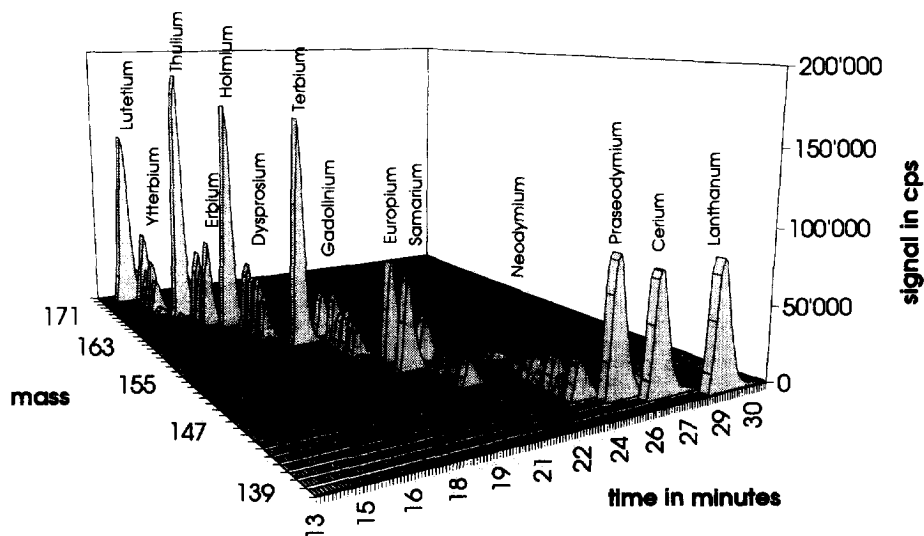


Fig. 4. Analysis of 1 ml of 1% U solution containing 20 ng/ml of each lanthanide by HPLC–ICP-MS. Chromatographic conditions as in Table 2 and ICP-MS method B in Table 1.

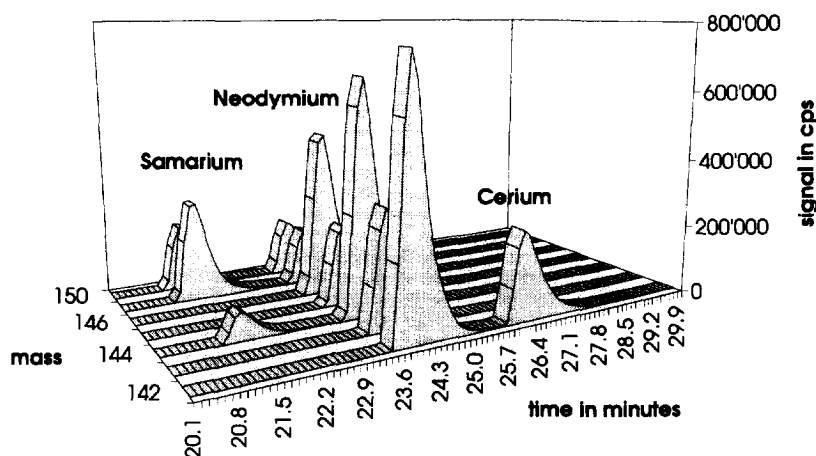


Fig. 5. Separation of Sm, Nd and Ce in 1 ml of 1% U solution containing 200 ng/ml of each lanthanide by HPLC-ICP-MS. Chromatographic conditions as in Table 2 and ICP-MS method C in Table 1.

Pu^{4+} to PuO_2^{2+} a 10 $\mu\text{g/g}$ Pu solution containing 0.001 M KMnO_4 and 1 M HNO_3 was left overnight. The next day the solution was mixed with U and Am standards to produce a solution containing 100 ng/ml Pu, U and Am. Owing to α -decay of ^{241}Am there is a small amount of ^{237}Np in the Am standard. With the separation procedure in Table 3 the interfering nuclides were well separated, as can be seen in Fig. 6.

3.3. Burn-up determination

Burn-up is an important quantity for the design and operation of nuclear power reactors. The classical burn-up method includes a very

Table 3
Chromatographic conditions for the separation of the actinides

Time (min)	DDW (%)	2 M HNO_3 (%)	1 M HCl (%)	0.4 M HIBA (%)
0.0	80	20	0	0
0.1	80	20	0	0
1.5	80	20	0	0
1.6	0	0	100	0
4.0	0	0	100	0
4.1	35	0	0	65
15.0	35	0	0	65

time-consuming separation of the fission product monitor Nd and the actinides U and Pu. As the concentrations of these elements are determined by isotopic dilution analysis, the separation has to be done twice, once for the sample and once for the spiked sample. The isotopic ratios of the isotopes are then measured by a thermal ionization mass spectrometer (TIMS). Owing to the many separation steps, there is a great risk of contamination of the sample, especially because the sample is measured only after the last separation step.

In the DOMO (Dodewaard Mixed Oxide) programme, classical burn-up analysis of mixed oxide fuel including pellets from Japan (J type) and Belgium (X type) with around 6% Pu had to be performed. Pieces of the segments X37 and J12 of test rods which had been irradiated for four cycles in the boiling water reactor Dodewaard NL between 1988 and 1992 were received. Samples of each fuel (about 2 g) were transferred into the "hot cell" and dissolved in 7 M HNO_3 -HF under pressure at 160°C. The classical determination of the burn-up by isotopic dilution analysis failed owing to contamination with natural Nd in the classical separation procedure. In order to find out in which step the sample became contaminated and to have a first impression of the accuracy of the new method,

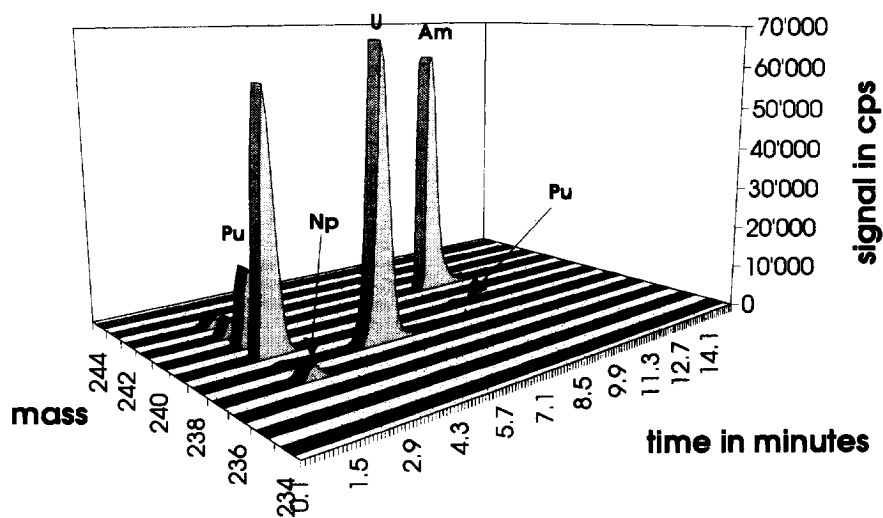


Fig. 6. Separation of U, Pu and Am in 200 μl of 1% HNO_3 solution containing 100 ng/ml U, Pu and Am by HPLC–ICP–MS. Chromatographic conditions as in Table 3 and ICP–MS method D in Table 1.

samples were taken from the classical separation procedure at different stages and analysed by HPLC–ICP–MS.

To obtain a survey of the lanthanides formed in spent fuel, 1 ml of the J12 fuel solution containing about 0.5 mg/ml fuel was mixed with 2 ml of 0.002 M KMnO_4 solution. The next day the solution was diluted tenfold to about 16

$\mu\text{g}/\text{ml}$ fuel. A 200- μl volume of this solution was separated by HPLC–ICP–MS using the chromatographic conditions in Table 2. As can be seen in Fig. 7, only the lanthanides Gd–La were formed in concentrations high enough to be detected by ICP–MS.

Fig. 8 shows the separation of the fission product monitor Nd in the J12 sample after the

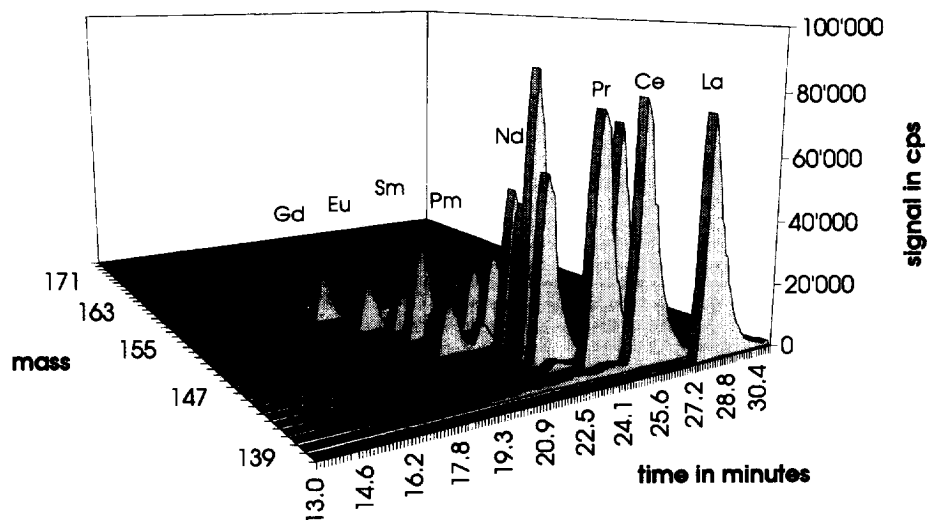


Fig. 7. Separation of 200 μl of about 16 $\mu\text{g}/\text{ml}$ spent U–PuO₂ (J12) by HPLC–ICP–MS. Chromatographic conditions as in Table 2 and ICP–MS method B in Table 1.

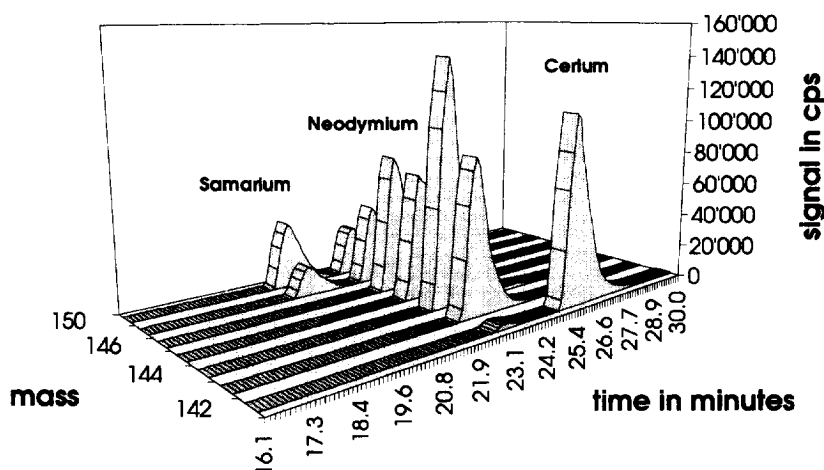


Fig. 8. Separation of the fission product monitor Nd in 200 μl of J12 sample after the first classical separation by HPLC–ICP-MS. Chromatographic conditions as in Table 2 and ICP-MS method B in Table 1.

first classical separation (preseparation of U). The separation was performed using the chromatographic conditions in Table 2 and ICP-MS method B in Table 1. All Nd isotopes are well separated from the interfering Sm and Ce isotopes.

To calculate isotopic composition of Nd, mass discrimination has to be corrected. Many workers use models of mass discrimination for a thermal ionization mass spectrometer [18,19]. Experience over the last 4 years has shown, however, that with ICP-MS measurements the mass discrimination does not depend linearly on the mass differences of measured nuclides, but

depends erratically on the mass or mass calibration. To calibrate for this effect, a solution of natural Nd was measured and the isotopic gain of every measured isotope was calibrated.

The fuel was spiked with ^{150}Nd . To determine the number of ^{148}Nd isotopes by isotopic dilution analysis, the $^{150}\text{Nd}/^{148}\text{Nd}$ isotopic ratio had to be measured in the spiked and unspiked samples. Table 4 shows the precision of the isotopic composition measurement by HPLC–ICP-MS of the X37 sample after the second classical separation. The measurement was repeated four times. A relative standard deviation of less than 1% for the $^{150}\text{Nd}/^{148}\text{Nd}$ isotopic ratio was ob-

Table 4
Precision of the isotopic composition measurement by HPLC–ICP-MS of the X37 sample after the second classical separation

Sample	^{142}Nd (%)	^{143}Nd (%)	^{144}Nd (%)	^{145}Nd (%)	^{146}Nd (%)	^{148}Nd (%)	^{150}Nd (%)	$^{150}\text{Nd}/^{148}\text{Nd}$
<i>X37</i>								
Mean	0.78	19.61	29.69	16.41	17.46	10.27	6.07	0.591
SD	0.15	0.03	0.15	0.13	0.09	0.14	0.10	0.003
<i>X37, spiked</i>								
Mean	0.89	18.77	28.38	15.78	16.69	9.83	9.65	0.982
SD	0.14	0.09	0.11	0.17	0.17	0.03	0.10	0.009

The Nd isotopic composition of 200 μl of X37 sample after the second classical separation was determined four times using the chromatographic conditions in Table 2 and ICP-MS method C in Table 1. The mean and the SD for the unspiked and spiked samples are given.

Table 5

Accuracy of the Nd isotopic composition measurement of the J12 sample after the first classical separation using HPLC–ICP-MS compared with the classical determination

Sample	¹⁴² Nd (%)	¹⁴³ Nd (%)	¹⁴⁴ Nd (%)	¹⁴⁵ Nd (%)	¹⁴⁶ Nd (%)	¹⁴⁸ Nd (%)	¹⁵⁰ Nd (%)	¹⁵⁰ Nd/ ¹⁴⁸ Nd
<i>J12</i>								
TIMS	0.60	19.43	30.06	16.51	17.31	10.14	5.95	0.587
HPLC–ICP-MS	0.61	19.88	30.18	16.31	17.81	10.11	5.94	0.587
<i>J12, spiked</i>								
TIMS	0.51	19.14	29.67	16.29	17.05	9.98	7.36	0.737
HPLC–ICP-MS	0.64	19.48	29.86	16.17	17.12	10.08	7.41	0.735

The Nd isotopic composition of the J12 sample determined using HPLC–ICP-MS is compared with the classical determination using TIMS for the spiked and unspiked samples. A 200- μ l volume of the J12 sample after the first classical separation was analysed using the chromatographic conditions in Table 2 and ICP-MS method C in Table 1.

tained. As the classical determination failed owing to natural Nd contamination, the results could not be compared. It cannot be presumed that the precision of the classical determination is better because it takes at least 2 weeks to obtain a result for one measurement and only one repeat is usually made.

Sample J12 was measured after the first classical separation. As the contamination with natural Nd was avoided for sample J12, the isotopic composition of Nd could be measured classically and by HPLC–ICP-MS. Very good agreement between the two methods was found for the ¹⁵⁰Nd/¹⁴⁸Nd isotopic ratio (Table 5), which is relevant for isotopic dilution analysis.

4. Conclusions

For burn-up calculation according to the ¹⁴⁸Nd method the ¹⁴⁸Nd and the U and Pu isotopic concentrations in a spent fuel aliquot have to be determined. As isobaric overlaps hinder the direct mass spectrometric determination of these elements, the interfering elements have first to be separated. With a Dionex CS 10 column it was possible to separate all interfering elements. Nd isotopic composition measurements showed that the loss of accuracy of ICP-MS compared with TIMS was compensated for by the very reliable and efficient separation. It can be assumed that

burn-up values from HPLC–ICP-MS determination are comparable to those using standard techniques. An important feature of the HPLC–ICP-MS method is the saving in terms of analysis time and analyst exposure to radiation relative to the classical technique. Moreover, it is possible to determine other lanthanides with the same equipment.

The new, much faster and much more reliable burn-up analysis method will be tested in an inter-laboratory program.

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