



Effective separation of Am(III) and Cm(III) using a DGA resin via the selective oxidation of Am(III) to Am(V)

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Abstract

A new method was developed to effectively separate Am(III) from Cm(III). Am(III) was selectively oxidized to Am(V) using a mixture of Na₂S₂O₈, Ag(I), and NaOCl in 0.01 M HNO₃. Cm(III) was selectively retained on a DGA resin, while Am(V) had no retention. A separation factor of 110 ± 20 was usually obtained from a single separation. The new separation method was applied to determine Cm isotopes 244, 245 and 246 by accelerator mass spectrometry (AMS) in spent nuclear fuel samples.

Keywords Curium · Americium · Separation · DGA resin · Accelerator mass spectrometry (AMS) · Nuclear fuel

Introduction

The measurement of Am and Cm in irradiated nuclear materials is of interest for nuclear waste management [1], spent nuclear fuel recycling [2–7], nuclear fuel analysis, and nuclear forensics [8]. The measurement of some isotopes of Am or Cm by alpha or mass spectrometry can be challenging. For example, the isotopes ²⁴³Cm and ²⁴⁴Cm have energies too similar to be resolved by alpha spectrometry (5883 and 5902 keV, respectively for the main peaks [9]), therefore mass spectrometry is required to measure the individual isotopes. However, ²⁴³Am cannot be resolved from

²⁴³Cm with mass spectrometry techniques such as thermal ionization mass spectrometry (TIMS), inductively coupled plasma mass spectrometry (ICP-MS), or compact accelerator mass spectrometry (AMS). Thus, to properly quantify Am and Cm isotopes in the same sample, the optimal solution is to effectively separate these 2 elements. Also, in irradiated nuclear materials, there is often significantly more Am than Cm, which could result in undesirable contamination of the ion source of the mass spectrometer with Am when measuring Cm isotopes. It could make it more difficult subsequently to measure trace amounts of Am isotopes if desired. In the cases mentioned, it would be advantageous to separate Am from Cm; but, the separation of Am(III) and Cm(III) is extremely challenging due to their very similar chemistry in aqueous solution. Current methods to separate Am and Cm are tedious and usually result in low separation factors (SF) [5, 10]. More efficient separation methods are needed.

The main Am isotopes (²⁴¹Am and ²⁴³Am) are commonly measured by alpha spectrometry as they have relatively short radioactive half-lives ($t_{1/2}$): 432.6 ± 0.6 a [9] and 7367 ± 23 a [9] for ²⁴¹Am and ²⁴³Am, respectively. Cm isotopes are measured by radiometric or mass spectrometry methods. Alpha spectrometry can be used to measure ²⁴³Cm ($t_{1/2}$ = 28.9 ± 0.4 a [9]) plus ²⁴⁴Cm ($t_{1/2}$ = 18.11 ± 0.03 a [9]) at trace levels, but it is not usually sufficiently sensitive to measure the lower abundant high mass Cm isotopes such as ²⁴⁵Cm ($t_{1/2}$ = 8250 ± 70 a [9]), ²⁴⁶Cm ($t_{1/2}$ = 4723 ± 27 a [9]), and ²⁴⁷Cm ($t_{1/2}$ = 1.56 × 10⁷ a [9]). Long-lived Cm isotopes are usually measured by mass spectrometry techniques such as TIMS [11], ICP-MS [12], and more recently by low energy compact AMS [13, 14].

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The chemistries of tri-valent Am and Cm in aqueous solutions are strikingly similar making them difficult to separate [15, 16]. Normally, Am in acidic aqueous solution is of oxidation state (III) [17], but under rare select conditions in aqueous solutions, Am(III) can be oxidized to Am(V) or Am(VI) [18]. However, Cm(III) is much more difficult to oxidize. After selectively oxidizing Am(III) to higher oxidation states, Am can be separated from Cm(III) by precipitation or ion exchange chromatography [19, 20]. The preferred strategy used to separate Am from Cm is through the selective oxidation of Am(III) to Am(V). Am(III) is first oxidized to a higher oxidation state (mixture of Am(V) and Am(VI)) using ammonium persulfate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$) in dilute nitric acid (HNO_3). Then, Am(VI) is reduced to Am(V) using sodium hypochlorite (NaClO) [1, 5, 7, 10, 18, 21]. Another strategy employed is to selectively oxidize Am(III) to Am(VI) using $(\text{NH}_4)_2\text{S}_2\text{O}_8$ and silver nitrate (AgNO_3) in a dilute HNO_3 solution [2]. However, Am(VI) is unstable in nitric acid solutions and tends to reduce to Am(V) (and gradually to Am(III)) [18].

The optimal conditions to oxidize Am(III) to Am(V) or Am(VI) have been studied, but have not led to a simple and effective method to separate Am and Cm. Burns et al. [5] first suggested in 2012 that a column separation would be possible. Then, they demonstrated a partial separation using ion exchange chromatography, but low SF were obtained [20]. Mincher et al. [10] tried to separate Am(V) from Cm(III) in 2015 using a commercial TRU resin, but they observed that a significant amount of Am(V) was reduced to Am(III) by the resin (about 50%). They then tested inorganic materials to see if they could improve the separation. They calculated the distribution coefficient (K_d) of Am(V) and Cm(III) on these inorganic materials and predicted a SF of up to 142 after 24 h equilibrium.

An effective and simple method to separate Am and Cm is needed. This work describes such a method. Am(III) was oxidized to Am(VI) with persulfate and silver and then Am(VI) was reduced and stabilized to Am(V) using hypochlorite [22]. The Am(V) was stable for an extended period of time (> 3 days). Cm(III) was not oxidized in these conditions. Cm(III) was selectively retained on a DGA (Eichrom) resin but not Am(V). The separation between Am and Cm was optimized and tested on irradiated nuclear fuels to demonstrate the applicability of the developed method.

Experimental

Reagents and standards

Radiochemical isotope standard solutions of ^{241}Am , ^{243}Am , ^{244}Cm were purchased from the National Institute of Standards and Technology (NIST), (Gaithersburg, MD, USA) and

^{248}Cm from Oak Ridge National Laboratory (Oak Ridge, TN, USA). Trace metal grade nitric acid (HNO_3) and hydrochloric acid (HCl) were purchased from Fisher Scientific (Fair Lawn, NJ, USA). Extraction chromatography (EXC) resins UTEVA and DGA normal (50–100 μm) were purchased pre-packed in 2 mL cartridges from Eichrom Technologies, Inc. (Lisle, IL, USA). The main extracting agents of the UTEVA and DGA (normal) resins are dipentyl pentylphosphonate and N,N,N',N' -tetra-*n*-octyldiglycolamide, respectively. De-ionized water used for this work was obtained from a Millipore Direct-Q5 purification system (Billerica, MA, USA). Sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$), silver nitrate (AgNO_3), and 10–15% sodium hypochlorite (NaClO) solution were obtained from Sigma-Aldrich (Oakville, ON, Canada).

Equipment

An Octete Plus[®] Alpha Spectrometer with eight 450 mm² ULTRA-AS ion-implanted silicon detectors (AMETEK/ORTEC Inc., Oak Ridge, TN, USA) was used for method development tests. A coaxial high purity germanium (HPGe) detector with 10 cm of lead shielding (AMETEK/ORTEC Inc., Oak Ridge, TN, USA) was used to identify the fission products present in the fuel samples and to verify that Am was properly removed before preparing the AMS targets. The compact 0.6 MV AMS system TANDY (ETH Zurich, Zurich, Switzerland) was used to measure separated Cm isotopes [23].

Separation procedure

The procedure has two parts: Am(III) oxidation and DGA column separation as shown in Fig. 1.

The sample, containing Am(III) and Cm(III), was evaporated to dryness in a 20 mL glass vial. Note, if the sample is initially in hydrochloric acid solution, convert to nitrate form by evaporation twice with concentrated nitric acid. A volume of 10 mL of 0.16 M $\text{Na}_2\text{S}_2\text{O}_8$ + 0.005 M AgNO_3 in 0.01 M HNO_3 was added to the residue and the vial was heated for 20 min at 80 °C using a water bath (Fig. 1, steps 1 and 2). Note that a suspension of Ag_2O is formed in the reagent solution and the solution shall be shaken before being used. The sample was taken out of the water bath and 1 mL of 33 mM NaOCl reagent was immediately added to the solution (Fig. 1, step 3). A white precipitate of AgCl was formed. Immediately after the addition of NaOCl , the solution acidity was adjusted to ~1 M HNO_3 by adding 1.5 ml of 8 M HNO_3 (Fig. 1, step 4). The solution was left to cool to room temperature, transferred to a centrifugation container, and centrifuged (Fig. 1, step 5).

The DGA resins were mounted on top of a 12-hole vacuum box. The 2 mL pre-packed columns were conditioned

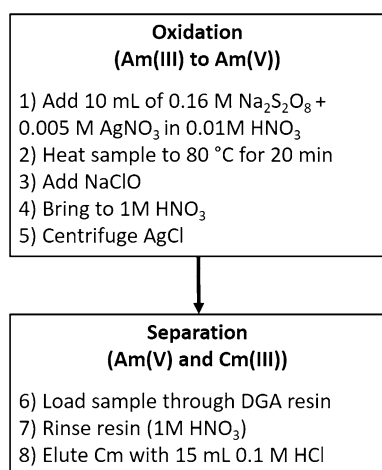


Fig. 1 Flow chart of the separation method

by passing 10 ml of 1 M HNO₃. The supernatant was passed through the DGA resin at a flow rate of about 1 mL min⁻¹ to selectively extract Cm(III) (Fig. 1, step 6). The resin was rinsed with 10 ml of 1 M HNO₃ (Fig. 1, step 7). The load solution plus rinse were collected for Am analysis if necessary as the Am(V) is not retained by the resin under these conditions [24]. Cm(III) was eluted from the resin using 15 mL of 0.1 M HCl (Fig. 1, step 8).

Method development

The formation of Am(V) was optimized by studying the effect of the concentration of three reagents: Na₂S₂O₈, HNO₃ and AgNO₃. The concentration of Na₂S₂O₈ was varied from 0.08 to 0.42 M in 0.01 M HNO₃ without the use of AgNO₃. The Na₂S₂O₈ concentration was then held at 0.16 M and two concentrations of AgNO₃, which were in large excess from Am, were evaluated (0.005 and 0.01 M). Finally, nitric acid concentrations were varied from 0.01 to 2 M, with 0.16 M Na₂S₂O₈ and 0.005 M AgNO₃. An excess of NaClO was used, thus was not further optimized.

The stability of Am(V) was evaluated utilizing ²⁴³Am, with measurement by alpha spectrometry. A solution was prepared as per the procedure section. Aliquots of the solution were taken periodically up to 14 days and Am(V) and (III) were separated using a DGA resin. All alpha sources were prepared using the CeF₃ micro-precipitation described by Dai [25].

The SF of the method was determined by using about 100 mBq of ²⁴¹Am or ²⁴⁴Cm. The test samples were passed through all the steps of the method described in the separation procedure above. The sample activities were measured by alpha spectrometry and then the SF was calculated.

The SF is defined for this work as the activity (or mass) ratio of a given element before and after chemical separation

from another element in a given fraction or sample. For example, if a sample initially contains 100 mBq of Am and 200 mBq of Cm and after separation, 1 mBq of Am is measured in this fraction, the SF in this example would be 100 (100 mBq/1 mBq).

The typical activities of ²⁴¹Am or ²⁴⁴Cm used for the method development tests were between 15 and 30 mBq.

Application of the method to nuclear fuel samples

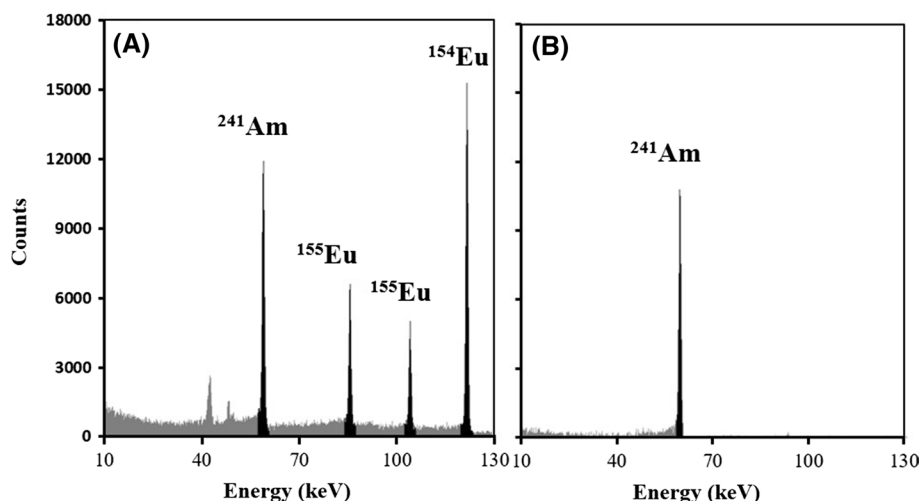
This method was tested on irradiated nuclear fuels originating at the Chalk River Laboratories (Chalk River, ON, Canada). These fuel samples had high levels of Am relative to Cm, making the separation desirable before measurement by AMS. These samples are highly radioactive due to the presence of fission and activation products. To assist with safety in handling, an initial separation was undertaken to remove the majority of the uranium, plutonium and fission products.

The fuel was dissolved in a nitric acid solution in a hot-cell. A small fraction of the resultant solution was removed from the hotcell and made to 8 M HNO₃. A known amount of ²⁴⁸Cm tracer (~ 10 pg) was added to the samples for Cm recovery calculations. The solutions were passed through UTEVA and DGA stacked columns. After separation, the columns were dismantled and Am, Cm and Ln were eluted from the DGA column using 20 mL of 0.1 M HCl. An aliquot of 2 mL of the eluate was evaporated to dryness in a small Teflon beaker. The residue was dissolved with 5 mL of 16 M HNO₃ and evaporated again to dryness to ensure removal of HCl in the sample. The spent nuclear fuel samples were then processed according to the Am/Cm separation procedure described above. If the procedure needs to be repeated to obtain a higher SF, the elution fraction from the DGA resin (15 ml of 0.1 M HCl, as given in Fig. 1, step 8) was evaporated to dryness. The residue obtained was then converted to nitrate form by evaporation twice with concentrated nitric acid. Then, the residue was re-dissolved with 10 mL of 0.16 M Na₂S₂O₈ + 0.005 M AgNO₃ in 0.01 M HNO₃, which enabled to repeat the Am/Cm separation procedure. The purified Cm was prepared for AMS measurement according to Dai et al. [13]. Americium-241 was measured by gamma spectroscopy before and after separation to estimate the method SF.

Radioactivity reduction

Following the preliminary separation steps described above (UTEVA + DGA resins), significant radioactivity remained in some high burnup samples as shown in Fig. 2a. Gamma spectroscopy analyses showed the radioactivity originated from Eu isotopes in the solution. To reduce the radioactivity, it was chosen to separate lanthanides (III) from actinides

Fig. 2 Gamma spectrum of the purified fuel solution. **a** Before and **b** after removal of the lanthanides



(III) on a TEVA resin using Eichrom method [26]. Briefly, starting with evaporation of the DGA resin eluate (Fig. 1, step 8), dissolve the actinides and lanthanides in a solution of 4 M KSCN + 0.1 M formic acid. Pass the resultant solution through a TEVA resin column, which selectively retains the actinides in these conditions but not the lanthanides. Finally, elute the actinides from the TEVA resin using a 1 M HCl solution. After Ln removal, no Eu was detected by gamma spectroscopy and the Am/Cm was fully recovered (Fig. 2b).

Results and discussion

Optimization of the formation of Am(V)

By adding a small amount (0.08 M) of persulfate to a 0.01 M nitric acid solution containing initially Am(III) and Cm(III), most of the Am was not retained on the DGA column (~80%) while almost all of the Cm was retained, as shown in Table 1. Increasing the amount of persulfate from 0.08 to 0.42 M did not significantly improve the oxidation from Am(III) to Am(V) (Table 1). Since the pre-loading step in this column work included the addition of hypochlorite, which is known to reduce Am(VI) to Am(V) [22], it suggests that a portion of the Am was not converted to Am(V) or Am(VI). A concentration of 0.16 M $\text{Na}_2\text{S}_2\text{O}_8$ was chosen for subsequent experiments.

A concentration of silver nitrate of 0.005 and 0.01 M, in addition to the persulfate, significantly reduced the retention of Am on the DGA resin to 1.1 ± 0.7 and $1.2 \pm 0.4\%$, respectively (see Table 2) compared to about 15% without AgNO_3 . The presence of silver promoted the oxidation of Am to higher oxidation states. As the results were similar for both concentrations tested, 0.005 M of silver was chosen for subsequent work.

Table 1 Extraction percentage of Am and Cm on the DGA resin as a function of the molar concentration of $\text{Na}_2\text{S}_2\text{O}_8$

$[\text{Na}_2\text{S}_2\text{O}_8]$ (M)	Am (%)	Cm (%)
0	90 ± 5	98 ± 2
0.08	17 ± 3	101 ± 5
0.16	14 ± 4	100 ± 4
0.28	16 ± 2	100 ± 1
0.42	20 ± 4	101 ± 2

Table 2 Extraction percentage of Am on the DGA resin as a function of the molar concentration of AgNO_3 in the presence of 0.16 M $\text{Na}_2\text{S}_2\text{O}_8$

$[\text{AgNO}_3]$ (M)	Am (%)
0	14 ± 4
0.005	1.1 ± 0.7
0.01	1.2 ± 0.4

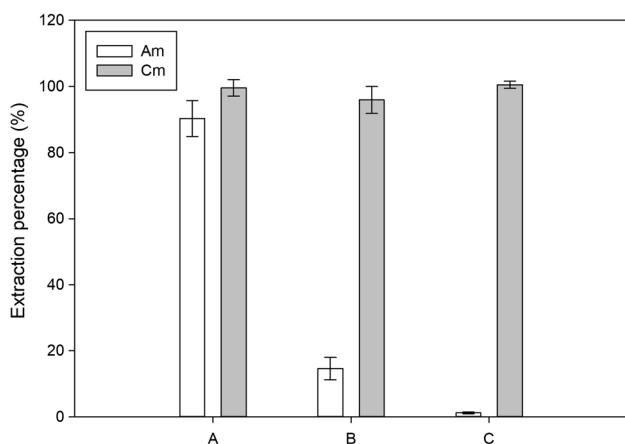
The final reagent concentration optimized for this work was nitric acid. By increasing the concentration of nitric acid from 0.01 to 2 M, Am was less retained on the DGA resin as shown in Table 3. This is in agreement with published results [7, 10, 22]. Am(V) is less favored when the nitric acid concentration increases. As per the experimental section, 0.01 M HNO_3 was chosen as the optimum concentration.

The overall effect of the reagents is summarized in Fig. 3 illustrating the successful separation of Am and Cm.

The elution fraction of the DGA resin (Fig. 1 step 8) should contain less Am after separation, which was the case since a SF of 110 ± 20 for Am was obtained (Table 4). The absence of Cm in the load and rinse solution fractions eluted from the DGA column (Fig. 1 steps 6 and 7) was also verified by obtaining a SF of $\geq 3300 \pm 200$ for Cm (Table 4). This is expected given that Cm(III) is known to be strongly retained on the DGA resin. A separation factor of about 110 for Am in the resin elution fraction is comparable to similar

Table 3 Extraction percentage of Am and Cm on the DGA resin as a function of the molar concentration of HNO₃ in the presence of 0.16 M Na₂S₂O₈ and 0.005 M AgNO₃

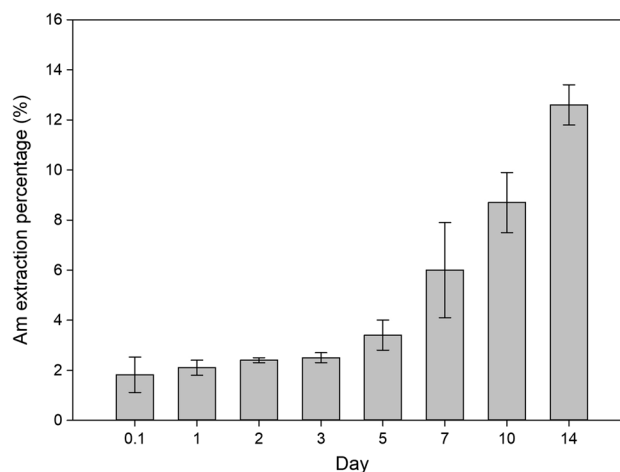
[HNO ₃] (M)	Am (%)	Cm (%)
0.01	1.1 ± 0.3	101 ± 5
0.1	1.3 ± 0.4	100 ± 5
1.0	1.7 ± 0.4	102 ± 7
2.0	43 ± 2	100 ± 7

**Fig. 3** Optimization summary of the Am and Cm separation on DGA resin in various conditions: **a** 0.01 M HNO₃, **b** 0.16 M Na₂S₂O₈ in 0.01 M HNO₃, and **c** Mix of 0.16 M Na₂S₂O₈+0.005 M AgNO₃ in 0.01 M HNO₃

methods [10]. The separation method is rapid, therefore the Cm fraction can be purified several times if needed to obtain a higher SF. This is the strategy used to purify Cm from Am in the application section described below.

Stability of Am(V) species

It is crucial to maintain Am as Am(V) in solution long enough to be able to separate it from Cm(III) using a DGA resin. The stability of Am(V) in solution was tested for 14 days (Fig. 4). Americium(V) was slowly reduced over

**Fig. 4** Percentage of Am extracted on the DGA resin as a function of time to test Am(V) stability

time as demonstrated by increased retention of Am on the DGA resin (Fig. 4). Importantly, once converted to Am(V), the oxidation state remained almost unchanged for at least 3 days, (variation of Am extraction < 2%). Subsequently, Am(V) very slowly reduced to Am(III) (Fig. 4). This is comparable to previously published work [5]. However, Am(V) was not significantly reduced by the DGA resin as it has been observed with the TRU resin [10].

Application to irradiated nuclear fuel

The measurement results for selected irradiated fuel samples are shown in Table 5. The Am SF was around 90 ± 20 for samples 1–5 for one separation (Table 5). For sample 6, a higher SF was desired to reduce the amount of Am lower or close to 1 pg for AMS measurement; therefore, a second separation was performed. A much higher SF was obtained ($> 1030 \pm 70$). This Am/Cm separation factor is significantly higher than previously published work [5, 10]. The time for a single separation is less than 2 h, thus this is a rapid and convenient method. Should a high SF be required, it is quick

Table 4 Separation factor (SF) of the method

Fraction description	Replicate	Activity added (mBq)	Activity measured (mBq)	SF	Average SF	SD
Am in the elution fraction of the DGA resin (Fig. 1, step 8)	1	107 ± 3	1.04 ± 0.07	103 ± 7	110	20
	2	107 ± 3	0.79 ± 0.06	140 ± 10		
	3	107 ± 3	1.13 ± 0.08	94 ± 7		
Cm in the load + rinse fraction of the DGA resin (Fig. 1, step 6 and 7)	1	88 ± 3	0.025 ± 0.002	3500 ± 300	3300	200
	2	88 ± 3	0.028 ± 0.002	3200 ± 200		

Table 5 Determination of Am and Cm in spent nuclear fuels

Sample ID	²⁴¹ Am before separation ^a (pg)	²⁴¹ Am after separation ^a (pg)	SF	Cm isotopes ^b (fg)		
				²⁴⁴ Cm	²⁴⁵ Cm	²⁴⁶ Cm
1	29 ± 1	< 0.4 ± 0.4	> 73 ± 5	420 ± 10	8.8 ± 0.5	3 ± 1
2	34 ± 2	< 0.4 ± 0.4	> 86 ± 6	340 ± 10	9.1 ± 0.4	3 ± 2
3	230 ± 10	2.5 ± 0.4	90 ± 6	340 ± 10	6.8 ± 0.4	ND
4	48 ± 2	< 0.4 ± 0.4	> 122 ± 9	300 ± 10	7.1 ± 0.4	2 ± 1
5	140 ± 3	2 ± 1	77 ± 5	560 ± 20	8.4 ± 0.5	2 ± 1
6*	485 ± 6	< 0.5 ± 0.5	> 1030 ± 70	12,200 ± 300	710 ± 20	213 ± 3

SF separation factor, ND not detected

*Separation repeated twice, ^aMeasurement by gamma spectrometry, ^bMeasurement by AMS

to repeat. For interest, the amount of ²⁴⁴Cm, ²⁴⁵Cm, and ²⁴⁶Cm measured in the samples are presented in Table 5. To the best of our knowledge, this is the first measurement of Cm isotopes in spent nuclear fuel sample by AMS.

For samples 1, 2, 4, and 6, the ²⁴¹Am activity measured by gamma spectrometry after separation was equal to the background. For these samples the activity measured after separation was estimated as the minimum activity that could be detected and the value was reported with the sign less than (see Table 5). The SF calculated from these values are reported with the sign greater than since the real SF value is superior to what is reported, but cannot be calculated more precisely (see Table 5). It explains why after a double separation a SF > 1030 ± 70 was obtained when a value of approximately 8100 would be expected (90 × 90).

Conclusion

A new effective method was developed to selectively separate Am from Cm. An effective separation was obtained by optimizing the conversion of Am(III) to Am(V) and maintaining this oxidation state for a long time (> 3 days). Am(V) was not reduced by the DGA resin. A sample batch can be processed in just a few hours providing rapid turnaround. A single separation gave a SF of 110 ± 20. This rapid and easy separation method can be repeated to obtain much higher SF if desired. The new developed method has potential applications in nuclear fuel analysis, radiochronometry, forensics and environmental studies.

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