KfK 4346
Januar 1988

Determination of Am-241, Cm-242 and Cm-244 in Environmental Samples

M. Afsar, H. Schüttelkopf
Hauptabteilung Sicherheit

Kernforschungszentrum Karlsruhe

KERNFORSCHUNGSZENTRUM KARLSRUHE
Hauptabteilung Sicherheit

KfK 4346

Determination of Am-241, Cm-242 and Cm-244 in Environmental Samples

M. Afsar, H. Schüttelkopf

Kernforschungszentrum Karlsruhe GmbH, Karlsruhe

Kernforschungszentrum Karlsruhe GmbH
Postfach 3640, 7500 Karlsruhe 1

ISSN 0303-4003

Abstract

An analytical procedure for the determination of Am and Cm in environmental, liquid and gaseous effluent samples was developed. The mean value of the chemical yield is about 90 %. A detection limit of 7 µBq/g is achieved. The decontamination factors for important α emitters are $> 10^4$. Four analyses/week can be performed by one technician.

Die Bestimmung von Am-241, Cm-242 und Cm-244 in Umgebungsproben

Zusammenfassung

Eine analytische Methode zur Bestimmung von Am und Cm in Umwelt-, Abwasser- und Abluftproben wurde entwickelt. Die mittlere chemische Ausbeute liegt bei ca. 90 %. Die Nachweisgrenze liegt bei 7 µBq/g. Die Dekontaminationsfaktoren für wichtige α-Strahler sind $> 10^4$. Vier Analysen können von einem Techniker in einer Woche ausgeführt werden.

Content

Page

1. Introduction 1
2. Literature Survey 1
3. The Matrix Separation 2
3.1. Chemicals 2
3.2. Apparatus 4
3.3. General Concept of the Analytical Procedure 4
3.4. Pretreatment of the Sample 5
3.5. The Extraction of Am and Cm 5
3.6. Elution of Americium and Curium 14
4. Purification of Americium and Curium 17
4.1. General Description 17
4.2. The Separation of the Rare Earths 17
4.3. The Electrodeposition of Am and Cm 19
4.4. Losses During the Particular Chemical Steps of the Procedure 21
5. Results 22
5.1. The Radiochemical Procedure 22
5.2. The Decontamination Factors 23
5.3. Applications 23
6. Literature 25

1. Introduction

Spent nuclear fuel contains Am-241 and low activities of Am-243, the two α emitters of americium, as well as the significant α emitters of curium, Cm-242 and Cm-244. The half-lives and the content in the fuel for a burnup of 45,300 MWd/t and a cooling time of one year are presented in Table 1.

Table 1: Half-lives and the Specific Activity in Nuclear Fuel for Am-241, Am-243, Cm-242 and Cm-244. Burnup: 45,300 MWd/t. Cooling Time: 1 y.

Nuclide	Half-live, y	Specific activity Bq/t heavy metal
Am-241	433	$1.06 \cdot 10^{13}$
Am-243	7650	$1.11 \cdot 10^{12}$
Cm-242	0.45	$3.64 \cdot 10^{14}$
Cm-244	18.1	$1.76 \cdot 10^{14}$

The Am-241 activity increases after 7 decades up to a specific activity of about $1.28 \cdot 10^{14}$ Bq/t due to the decay of Pu-241. According the very short half-life, Cm-242 is without significance after some years.

To perform the environmental surveillance around the research facilities of the Karlsruhe Nuclear Research Center (KfK), especially the reprocessing plant, a sensitive and fast procedure for the determination of Am and Cm had to be developed.

2. Literature Survey

Many techniques for the analysis of Am and Cm in environmental samples have been reported in literature up till 1982, being not only difficult but also time consuming, sometimes without a good resolution of Am and Cm α spectra. The procedures were tested in our laboratories but no satisfactory results were obtained. In each case the sample weights were too small. Therefore, we had to develop an analytical technique on our own that guarantees a good separation of Am and Cm from interfering α emitters and trivalent lanthanides and allows the analysis of 100 g samples. The optimized analytical technique is described in detail in chapters 3 and 4 [1 - 6].

3. The Matrix Separation

3.1. Chemicals

All the chemicals used were of reagent grade. The important chemicals used for a complete analysis are the following:

- TOPO/Kieselgur Mixture (TOPO-Tri-n-octylphosphineoxide). Dissolve 30 g of TOPO in 100 ml of Diethylbenzol. To this solution, add 60 g of Kieselgur and stir till a homogeneous mixture is obtained.Add acetone to this mixture till the level of acetone is a little higher than the level of TOPO/Kieselgur in the beaker and cover it with a watchglass. Continue occasional stirring for about 48 hours. Evaporate the remaining aceton by use of a water pump. Heat the TOPO/Kieselgur mixture with 0.1 $\underline{M}$ HNO_3, 200 - 400 ml, in a 500 ml beaker at 80 °C for 4 hours. Cool and filter the HNO_3. Again heat the TOPO/Kieselgur mixture with 2 x 400 ml H_2O at 80 °C for 4 hours. Filter. Transfer the TOPO/Kieselgur mixture in an open dish of 500 ml capacity and take the mixture to almost complete dryness in an electric oven at 80 - 90 °C overnight. Make a sludge of this mixture with H_2O or diluted HNO_3 before using it in a column. The level of H_2O or HNO_3 should always remain a little higher than the level of TOPO/Kieselgur in the column. Wash the column after use one time with 200 ml of 0.1 $\underline{M}$ HNO_3 and use it for the next determination.

- Nitric acid CH_3OH Mixture. Mix 35 ml of conc. HNO_3 with 465 ml of pure methanol.

- HCl, 0.1 $\underline{M}$, NH_4SCN, 0.5 $\underline{M}$, CH_3OH, mixture. Mix 50 ml of 1 $\underline{M}$ HCl and 50 ml of 5 $\underline{M}$ NH_4SCN with 400 ml of pure methanol.

- Hydrochloric acid, 32 %, Methanol mixture. Mix 65 ml of 32 % HCl with 430 ml of pure methanol and dilute to 500 ml with distilled water.

- Methyl Red, 1 %.

- NH_4SCN, 5 M, 38.06 g NH_4SCN with distilled water up to 1 l.

- 9 M HCl, 891 ml HCl, 32 %, with distilled water up to 1 l.

- Cleaning of the $HCl/NH_4SCN/CH_3OH$-Solution: 1 l is purified with anion exchanger, Dowex 1 x 4, Cl^--form, 100 - 200 mesh. Column: 2 cm inner diameter, 40 ml ion exchanger, 2·5 ml/min. The first 250 ml are discarded.

- Dissolve 1 g of Methyl red in 150 ml of ethanol.

- 1 M Aluminium nitrate. Dissolve 375 g of $Al(NO_3)_3 \cdot 9H_2O$ in 500 ml of distilled water. Add 350 ml of conc. HNO_3 and bring the volume up to one liter with distilled water.

- Anion-Exchange Resin. Dowex: 1 x 8, 100 - 200 mesh, Cl^--form. Dowex: 1 x 4, 100 - 200 mesh, Cl^--form.

- Cation-Exchange Resin: Dowex: 50 W x 8, 200 - 400 mesh, H^+-form.

- 0.9 M HF/8 M HNO_3: 560 ml HNO_3, conc., and 40 ml HF, 40 %, make up the volume with distilled water up to 1 l.

- 6 M NaOH (for neutralization) 240 g NaOH dissolved in distilled water and brought to 1 l.

- 0.1 M HNO_3: 7 ml HNO_3, conc., distilled water up to 1 l.

- 2 M HNO_3: 140 ml HNO_3, conc., distilled water up to 1 l.

- 4 M HCl: 396 ml HCl, 32 %ig, distilled water up to 1 l.

- $(NH_4)_2C_2O_4$; 4 %: 4.6 g $(NH_4)_2C_2O_4$ dissolved in 95.4 g H_2O.

- Am-243-standard solution, 0.1 - 0.3 Bq/ml.

3.2. Apparatus

- Ion exchange columns having the following dimensions: 15 cm of length and 0.7 cm inner diameter. 20 cm of lenght and 2 cm inner diameter.

- Electrolytic cells with platinum electrodes.

- Stainless steel planchets, 2·5 cm diameter, for electroplating.

- Proportional counter for the measurement of gross α activity.

- Silicon surface barrier: detector ORTEC BPY-55-350 SQ/R, Ortec GmbH, Frankfurt/Main, Federal Republic of Germany.

3.3. General Concept of the Analytical Procedure

The leaching procedure has been tested and optimized for large sample volumes above all for plutonium [8] and has been applied successfully for Am and Cm. It consists of a double leaching with an mixture of HF/HNO_3 and $HNO_3/Al(NO_3)_3$ which can be performed within one hour.

The first separation of Am and Cm is achieved by extraction with trioctylphosphinoxide adsorbed on Kieselgur. Most of the matrix elements can be separated and a fairly clean Am and Cm fraction is achieved. The radiochemical separation of lanthanides follows the procedure described by E. Holm et al. [9] with partial modification. It consists of a cation/anion-exchange in conc. HCl, an adsorption on anion exchanger in CH_3OH media and a special cleaning step for lanthanides using NH_4SCN in CH_3OH/HCl.

The preparation of the pure Am and Cm fraction is done by electroplating from oxalic acid/HCl. Counting is performed by use of a surface barrier detector and α spectrometry.

3.4. Pretreatment of the Samples

The samples collected in the environment around the Karlsruhe Nuclear Research Center were dried at 110 °C and then ashed at 550 °C in an electric furnace overnight. 100 g of the sample were leached with 290 ml of 8 M HNO_3/0.9 M HF for half an hour, centrifuged and the supernatent solution separated from the residue. The residue was again leached with 250 ml of 5 M HNO_3/1 M $Al(NO_3)_3 \cdot 9H_2O$ for half an hour. The supernatents were united. The residue was discarded.

The residues of soil samples, containing 100 Bq Am-241, were checked for unleached Americium activity. Four experiments showed that 0.2, 1.25, 1.8 and 1.9 % remained in the residue. This means that the leaching process is practically quantitative.

3.5. The Extraction of Am and Cm

HDEHP: Applying the in the literature chiefly reported method the extraction of Americium and Curium was checked with 0.2 M HDEHP in n-Heptane out of various concentrations of HNO_3. The 500 ml solutions received in 3.4 were extracted twice with 25 ml of 0.2 M HDEHP. It was found that besides many experimental difficulties, the distribution coefficient of Americium, extracted from 0.1 M HNO_3, did not exceed 1.5. With the increase in concentration of HNO_3, the distribution coefficient decreased. The solubility of HDEHP in nitric acid was so high that measurements of Am-241-tracer in the aqueous phase were very difficult. Therefore we had to look for another extractant for Americium and Curium.

TOPO: A solution of TOPO, 0.2 M/Cyclohexan was then checked for the extraction of Am and Cm from various concentrations of HNO_3 in 500 ml solutions. Americium and Curium yielded a distribution coefficient of about 1.0 - 1.5 from 0.1 M HNO_3 concentration. As this extraction coefficient was not higher than with HDEHP, we had to look for another alternative. Additionally it was proved that Americium and Curium extraction from higher concentrations of HNO_3 gave lower extraction coefficients. Results for the extractions of Am-241-tracer with 25 ml of 0.2 M and 0.5 M TOPO/Cyclohexan from 500 ml of 0.1 M HNO_3 are presented in Fig. 1 and Fig. 2.

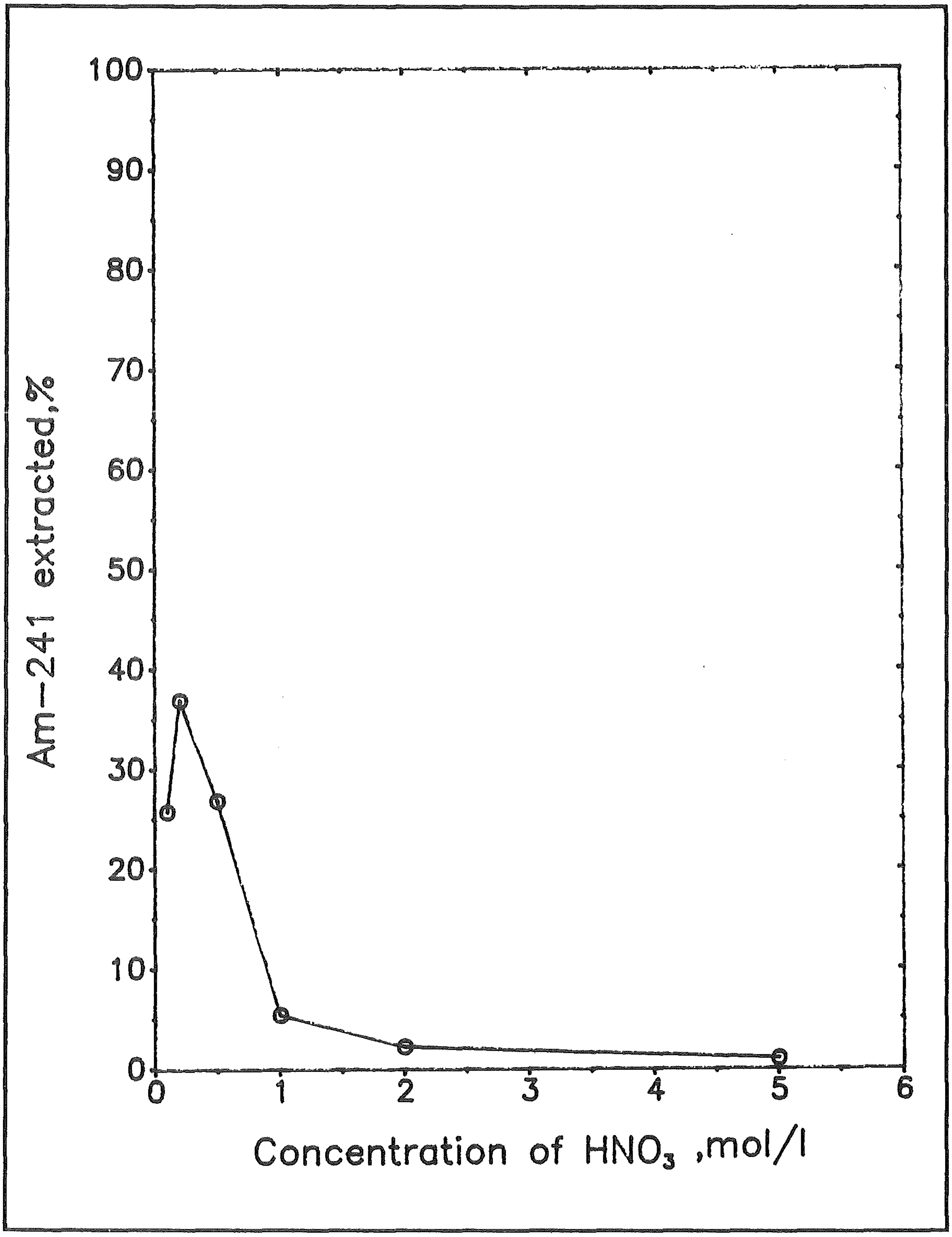

Fig. 1: The Fraction of Am Extracted by 25 ml 0.2 M TOPO/Cyclohexane Depending on the HNO_3 Concentration of 500 ml Solution. Extraction: 15 min.

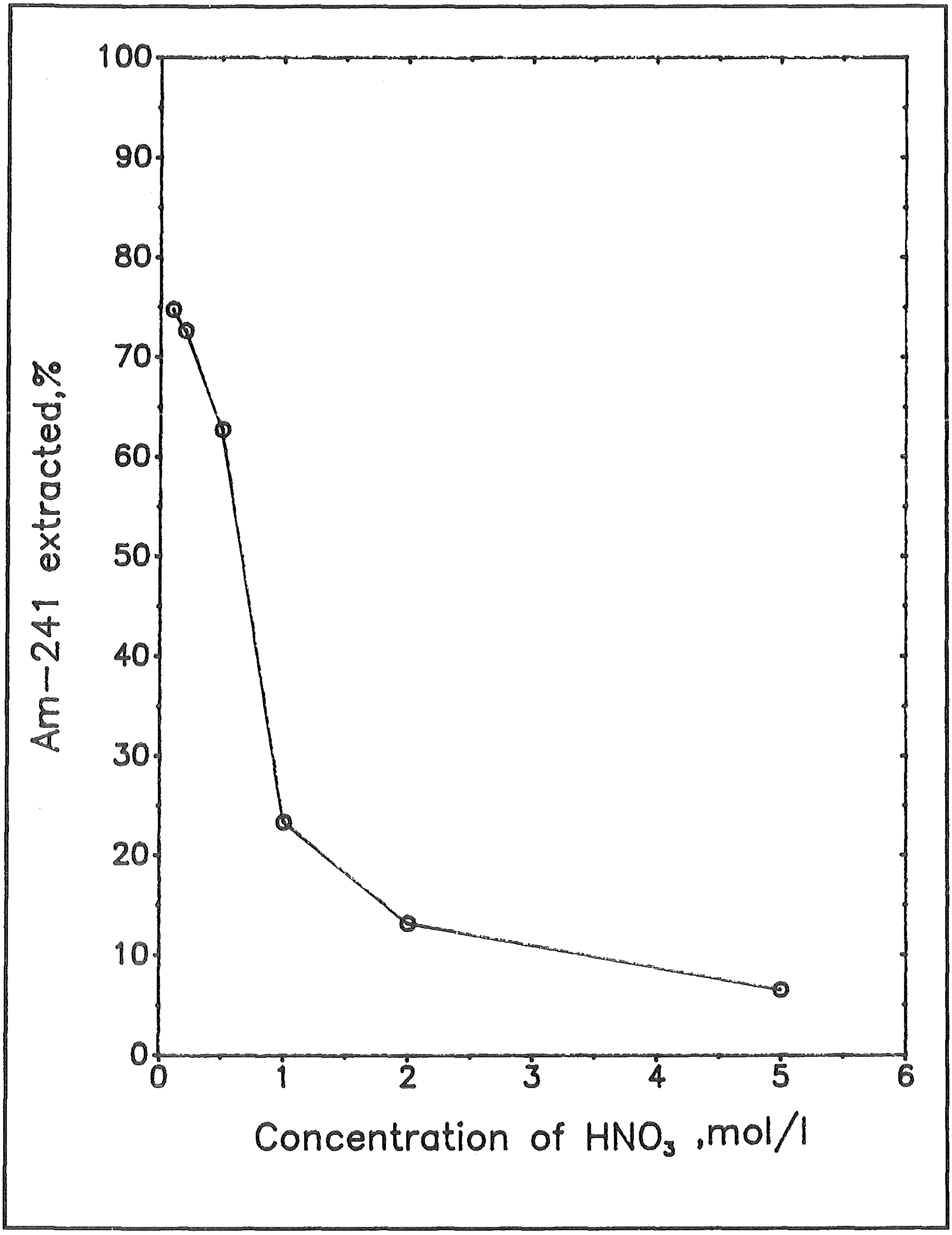

Fig. 2: The Fraction of Am Extracted by 25 ml 0.5 M TOPO/Cyclohexane Depending on the HNO_3 Concentration of 500 ml Solution. Extraction: 15 min.

The way of TOPO extraction was then changed to column chromatography. TOPO 30 % in diethylbenzene was adsorbed on the surface of the silicious material Kieselgur (see 3.1). 15 - 20 ml of this mixture were used in an ion-exchange column of 20 cm length and 2 cm inner diameter. The extraction of Americium and Curium in various concentrations of HNO_3 was then checked by using this column. It was found that from 0.2 M HNO_3 concentration about 95 % of Americium and Curium were extracted into the TOPO/Kieselgur mixture (Fig. 3). The extraction or retention of Am and Cm on column decreased with the increase in concentration of HNO_3. We concluded that a 0.1 M HNO_3 concentration should be the most proper for getting high extraction yields for these nuclides. Therefore we reduced the HNO_3 concentraiton of the leached solution to 0.1 M by neutralization with NaOH in advance. Almost 100 % of Am and Cm remained on column.

A similar experiment with HDEHP/Kieselgur gave very good results too (Fig. 4). Nevertheless HDEHP and other phosphor compounds partly passed the column during the absorption and washing process. These compounds were difficult to separate and finally disturbed the electroplating procedure. Because of these serious losses of HDEHP from the column, further experiments were performed only with TOPO.

Washing of TOPO/Kieselgur Column: To get rid of most of the matrix and added salts and of some rare earths adsorbed on the TOPO/Kieselgur column, it was neccessary to wash the column with 0.1 M HNO_3. After passing about 150 ml 0.1 M HNO_3 through the column, much of the unwanted matrices were leached out and almost nil residue was found in an extra wash with 150 ml solution (Fig. 5). Am and Cm remained fairly unleached (Fig. 6 and Fig. 7). To purify Americium and Curium from remaining rare earths, a washing of the column with various concentrations of HCl was also checked. It showed that Am and Cm were continuously leached by the washing with HCl. The use of 0.075 M HCl as reported in [3] also gave errorneous results (Table 2). Therefore only HNO_3 was used for the washing.

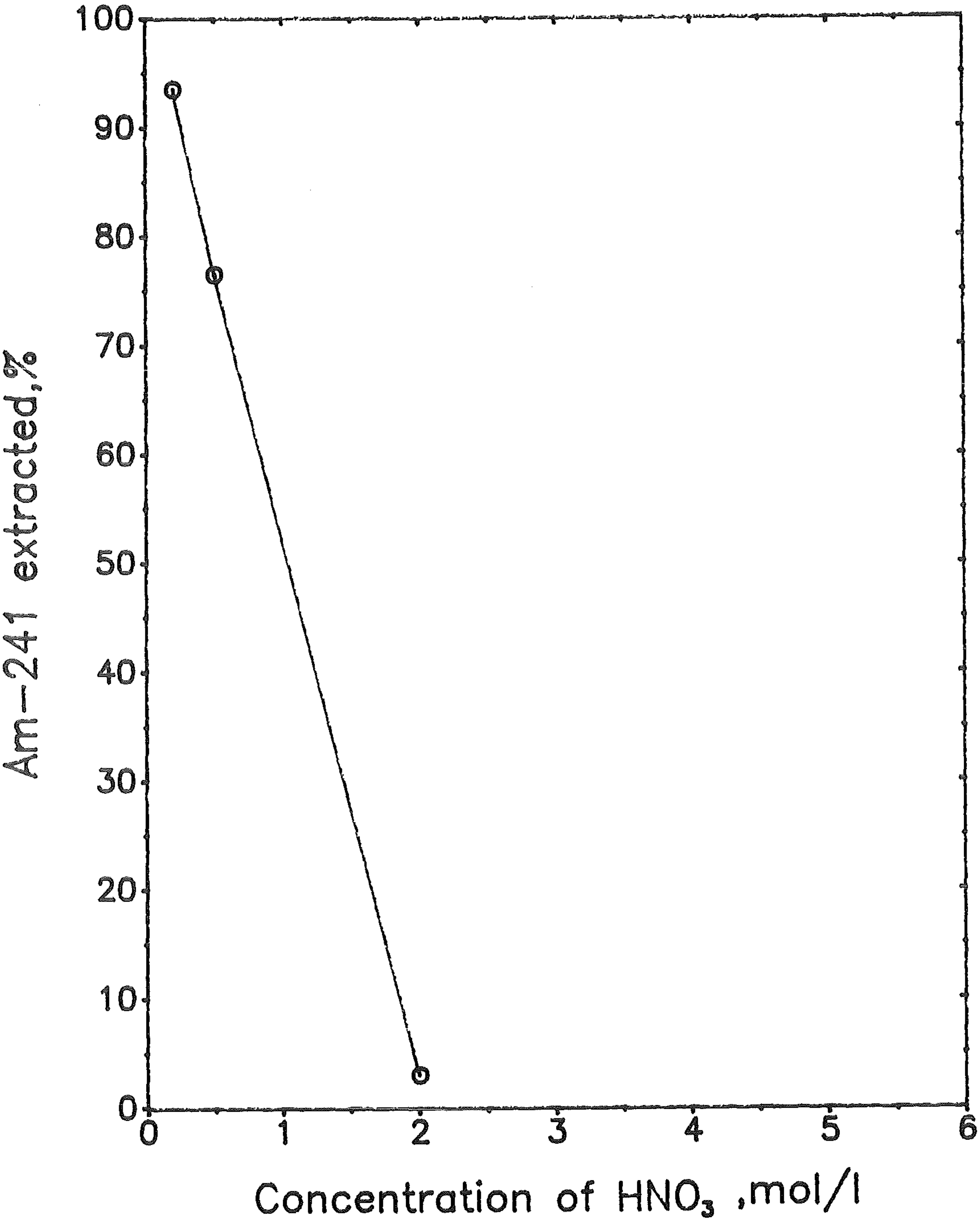

Fig. 3: The Fraction of Am Extracted with a TOPO/Kieselgur Column Depending on the HNO_3 Concentration of 500 ml Solution.

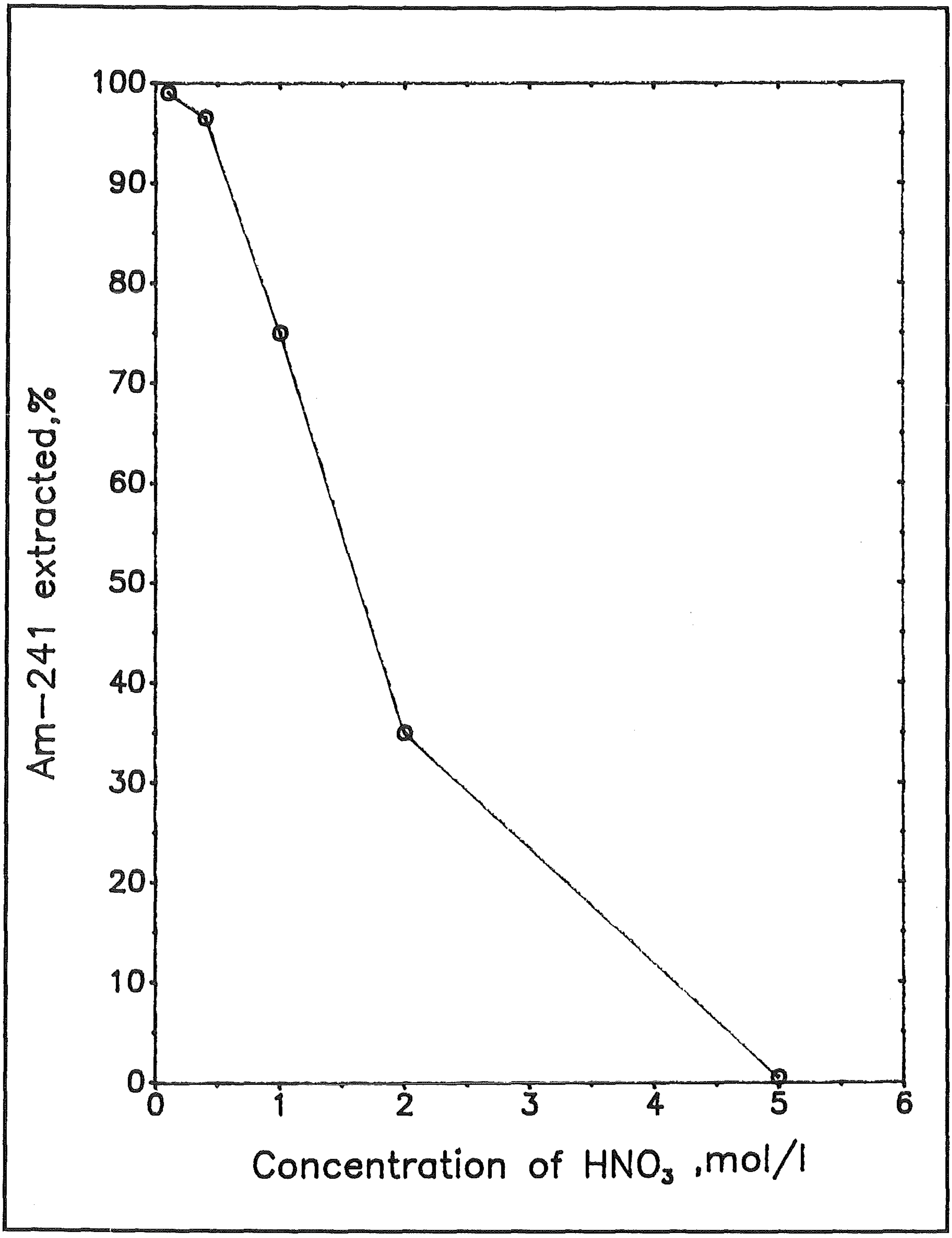

Fig. 4: The Fraction of Am Extracted with a HDEHP/Kieselgur Column Depending on the HNO_3 Concentration of 500 ml Solution.

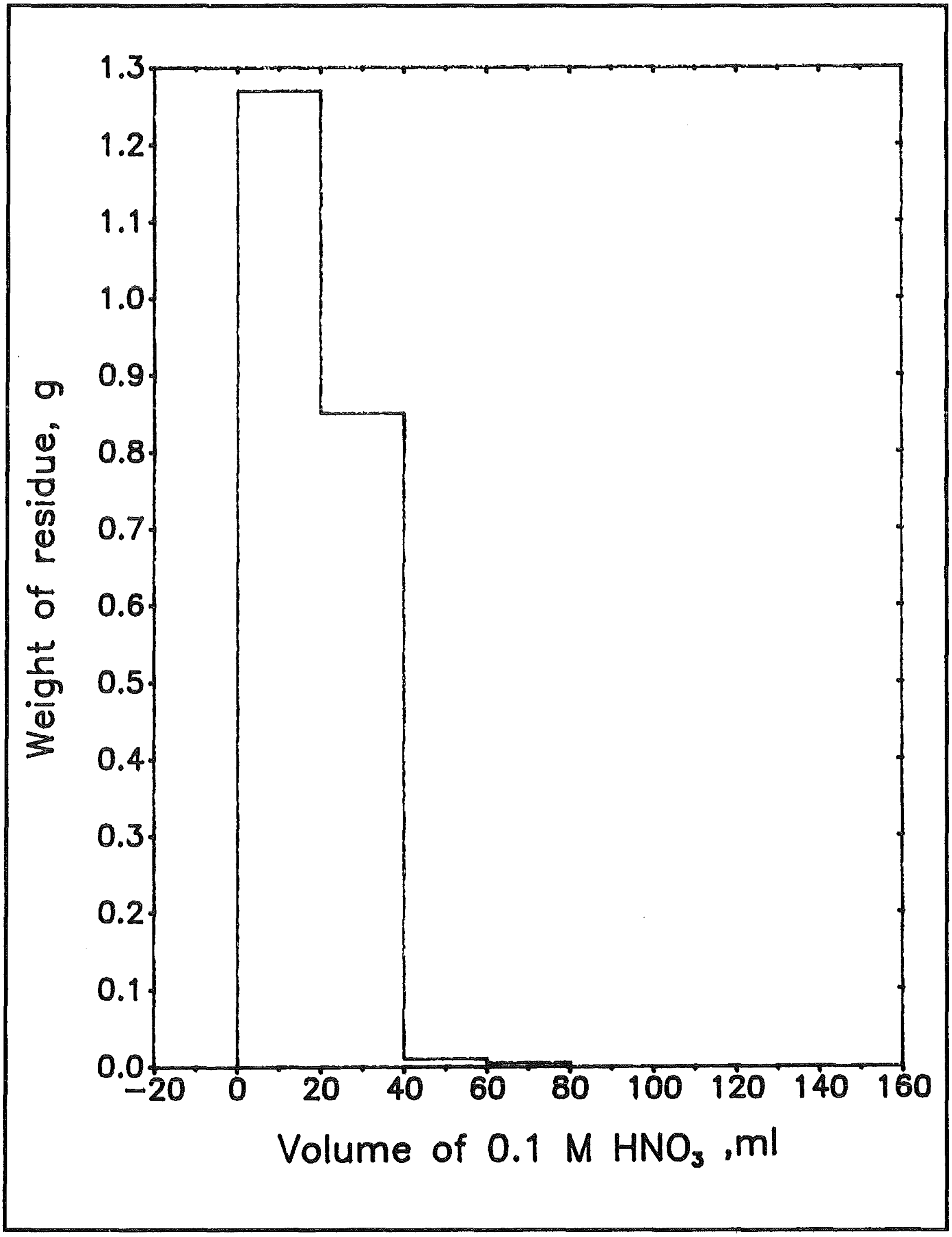

Fig. 5: Dry Weight of Matrix Material Washed Out from the TOPO/Kieselgur Column Using 0.1 M HNO_3 as Washing Solution.

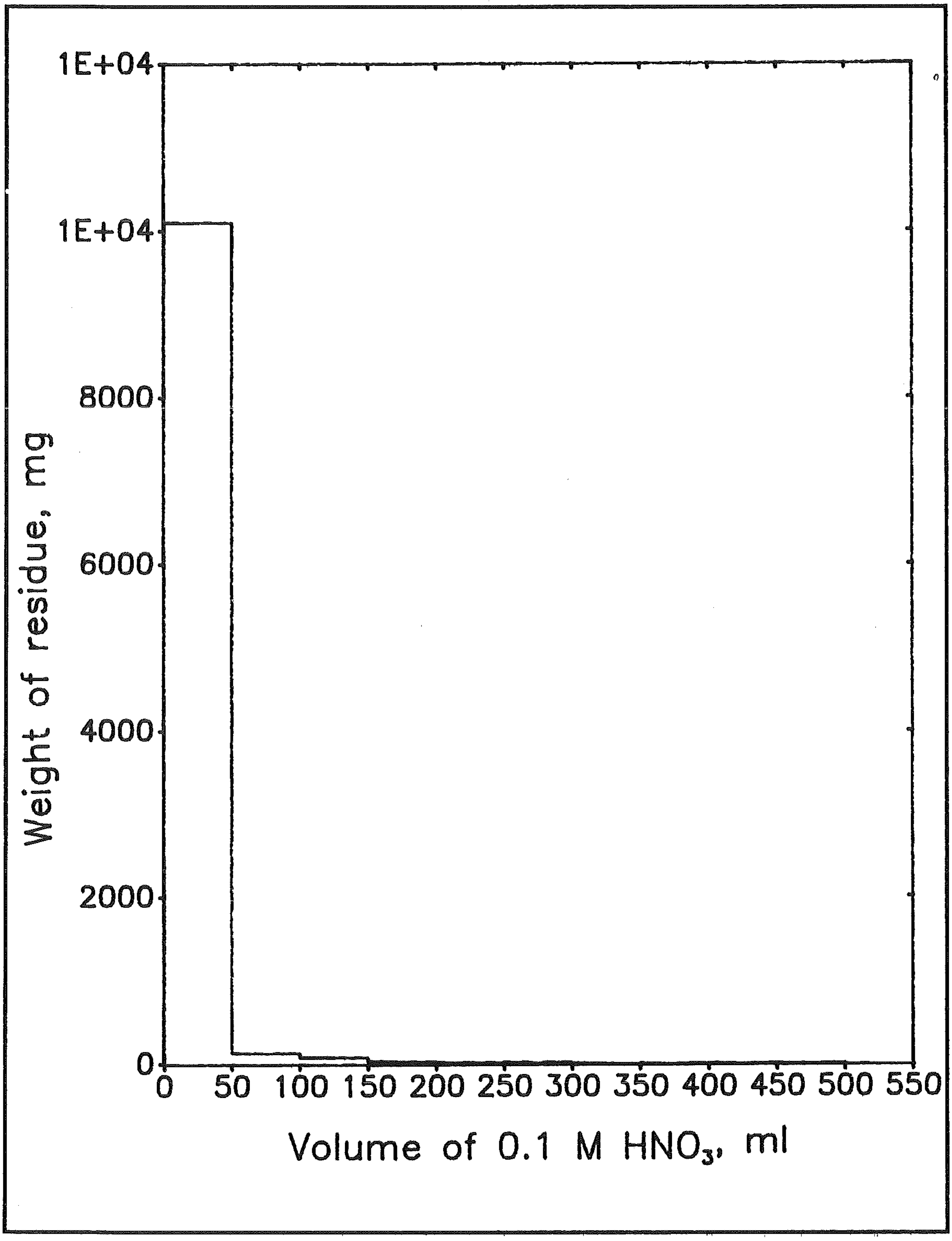

Fig. 6: Dry Weight of Matrix Material Washed Out from the TOPO/Kieselgur Column Using 0.1 M HNO_3 as Washing Solution.

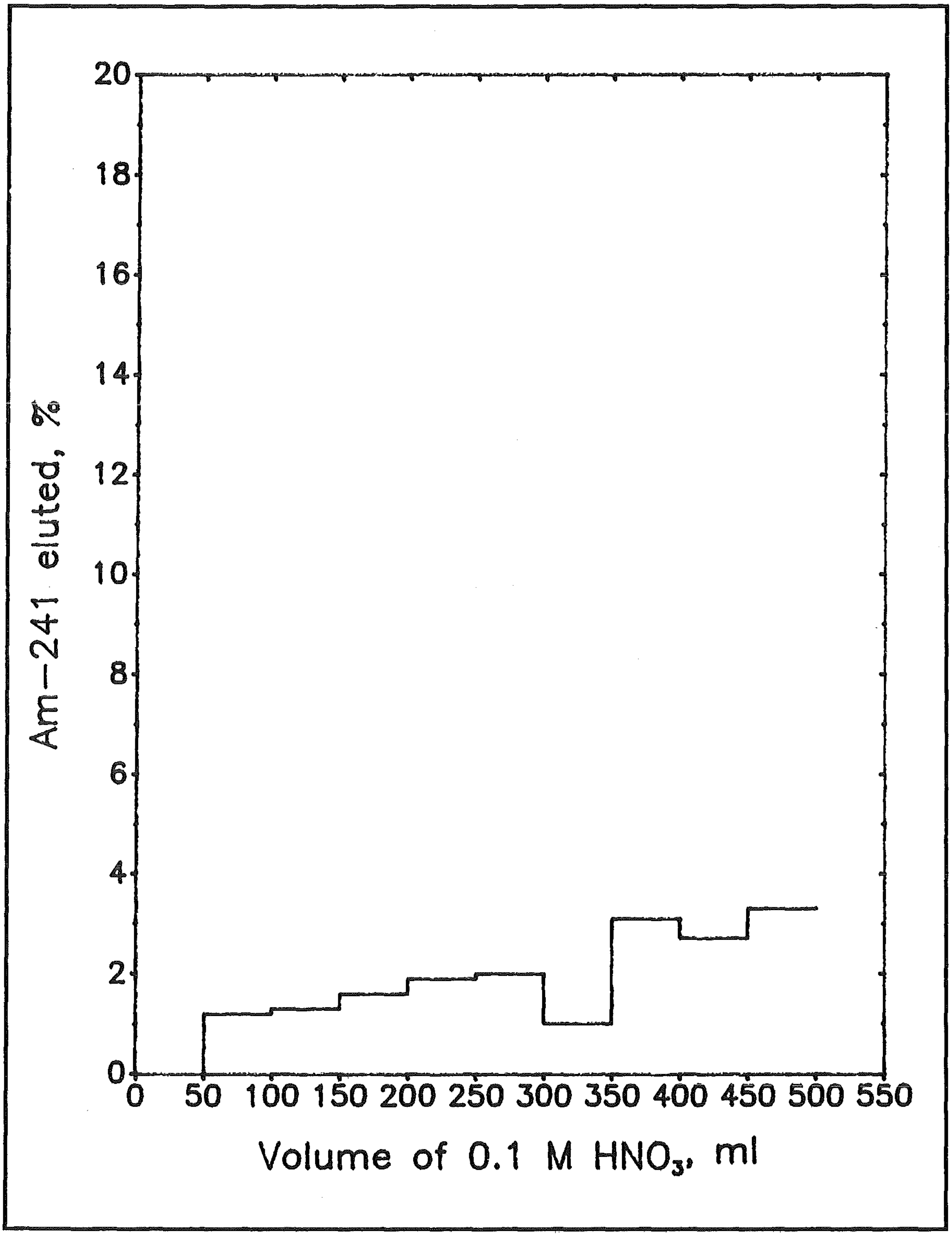

Fig. 7: Am-241 Washed Out from the TOPO/Kieselgur Column Using 0.1 M HNO_3 as Washing Solution.

Table 2: Am-241 Eluted by Various Concentrations of HCl Washing Solutions.

HCl concentration mol/l (300 ml of HCl solution)	Am-241 eluted in %
0.050	2.4
0.075	17.6
0.100	49.2

3.6. Elution of Americium and Curium

Americium and Curium were leached with 8 M HNO_3 from TOPO/Kieselgur column. Many experiments were made in optimizing the total volume of 8 M HNO_3 required for complete elution of Am and Cm. 150 - 200 ml of 8 M HNO_3 yielded the best results (Fig. 8 and Fig. 9).

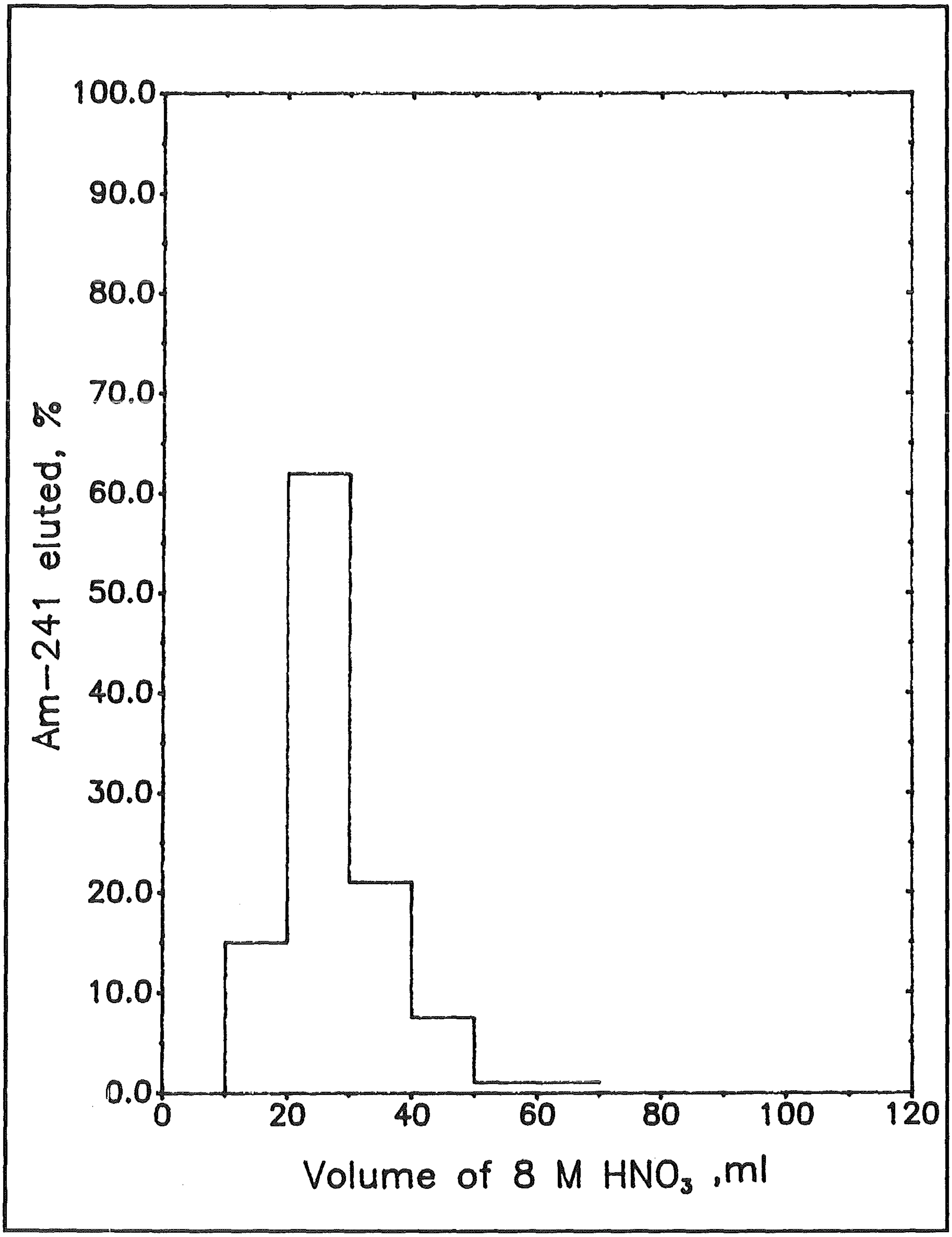

Fig. 8: Elution Curve for Am-241 Using 8 M HNO_3.

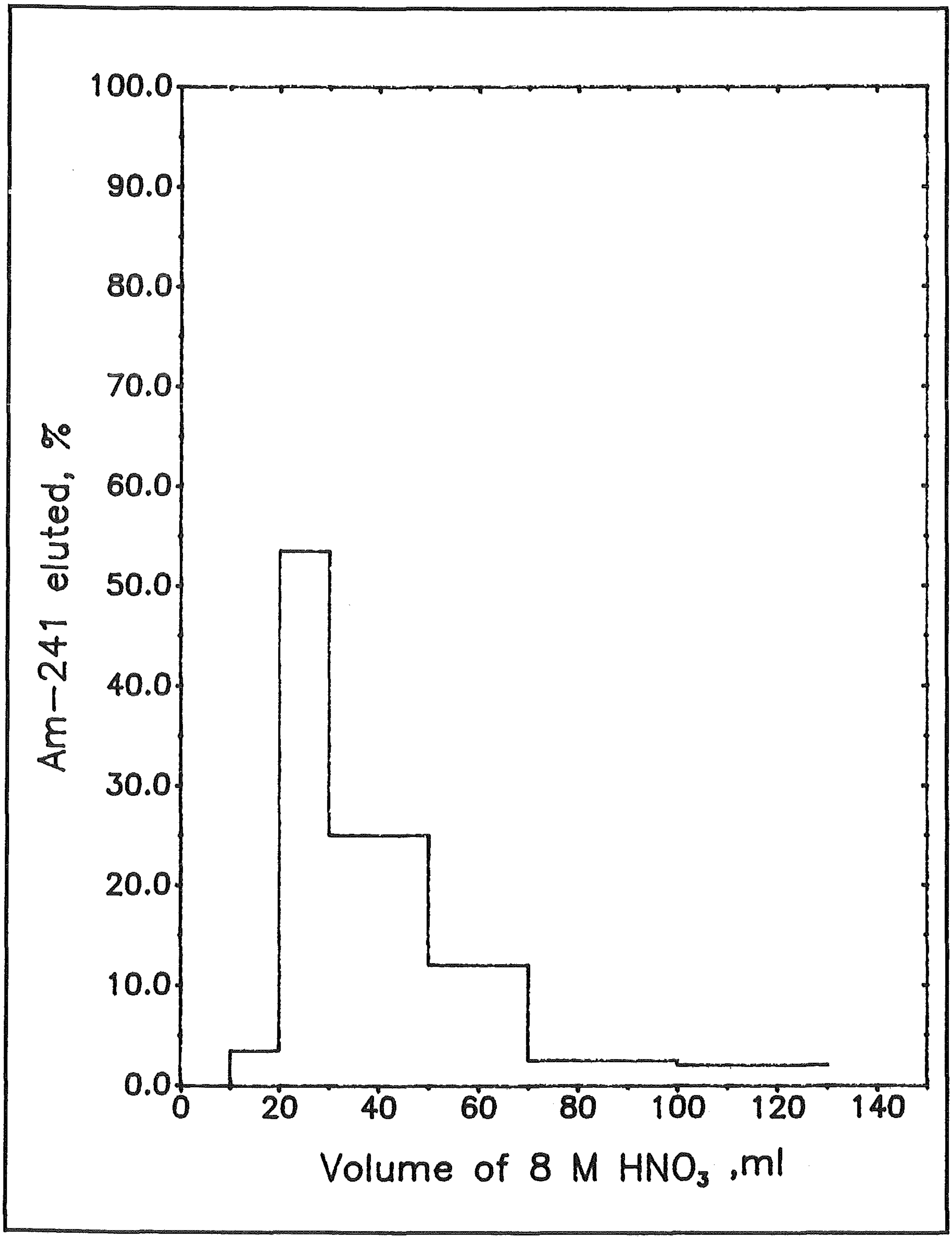

Fig. 9: Elution Curve for Am-241 Using 8 M HNO$_3$. Real Sample Processed.

4. Purification of Americium and Curium

4.1. General Description

Before looking for the requirement of additional purification of Am and Cm from other interferring nuclides, the 8 M HNO_3 eluate was evaporated directly and the possibility of electroplating Am and Cm onto stainless steel planchet from Ammonium-oxalate media was checked. It proved to be impossible due to milligram quantities of residue, left after 8 M HNO_3 evaporation, which when dissolved in HCl for carrying out electrolysis produced a type of suspension rather than a clear solution. Also at the end of electrolysis, many salts were deposited on the surface of the planchets which resulted in degraded α spectra and much lower chemical yields of about 0.5 - 10 %. The reason for these observations was the presence of trivalent rare earths that get electroplated along with Americium and Curium forming non-soluble oxalates. Therefore, there was no alternative than to look for an additional purification technique that yields a good separation of Am and Cm from interfering nuclides. Americium and Curium were additionally purified by using a combination of an anion and cation exchange procedure. The 8 M HNO_3 eluate of Am and Cm was evaporated to complete dryness and the residue was dissolved in 9 M HCl. This solution was then passed over a double layer column, lower layer an anion exchange resin, Dowex 1 x 8, 100 - 200 mesh, and upper layer a cation exchange resin, Dowex 50 W x 8, 200 - 400 mesh, in a 15 cm long and 0.7 cm inner diameter column at a flow rate of 0.5 ml/min. Remaining traces of Fe, Po, Th, U and Pu were sorbed on the column [9].

4.2. The Separation of the Rare Earths

The effluent of the column is evaporated to dryness and the residue is taken up in 10 - 20 ml of 2 M HNO_3/CH_3OH, 93 %, mixture. This solution is then passed through a second column containing anion exchange resin, Dowex 1 x 4, 100 - 200 mesh, at a flow rate of 0.5 ml/min. The column is washed with additional 30 ml of 1 M HNO_3/CH_3OH, 93 %. Americium and Curium are adsorbed on the column together with traces of rare earths, Pb and U etc. while any remaining traces of iron pass through.

The rare earths together with remaining traces of Uranium are eluted from the column with 60 ml of the HCl/NH_4SCN/CH_3OH mixture, Am and Cm remain on the column.

Finally, Am and Cm are eluted from the column with 30 ml of 1.5 M HCl/CH_3OH, 86 %, solution at a flow rate of 0.5 ml/min (Table 3) [9].

Table 3: The Separation of Lanthanides and Am by Anion Exchange Procedure, 50 mg La^{3+} and 5100 Bq Am-241 were used.

Part of the separation process	La and Am in the solution in %	
	La	Am
Adsorption from 1 M HNO_3/CH_3OH, 93 %		
1st 10 ml	< 0.2	0.004
2nd 10 ml	< 0.2	< 0.0002
Washing with 1 M HNO3/CH_3OH, 93 %		
1st 10 ml	< 0.2	< 0.0002
2nd 10 ml	< 0.2	< 0.0004
3rd 10 ml	< 0.2	0.0003
Separation of lanthanides by 1 M HCl/0,5 M NH_4SCN/CH_3OH, 80 %		
1st 10 ml	1.5	0.001
2nd 10 ml	31.4	0.002
3rd 10 ml	59.3	0.003
4th 10 ml	6.3	0.004
5th 10 ml	1.4	0.007
6th 10 ml	< 0.2	0.004
Elution with 1.5 M HCl/CH_3OH, 83 %		
1st 10 ml	< 0.2	0.01
2nd 10 ml	< 0.2	72
3rd 10 ml	< 0.2	6

4.3. The Electrodeposition of Am and Cm

At first, the system for the electrodeposition of Americium and Curium given by [10] was adopted. This system is based on dissolving the final residue of leached solution in H_2SO_4 and then adjusting the pH of the resulting 8 $\underline{M}$ H_2SO_4 solution to pH 3. The pH was adjusted using Methyl red as indicator. Electrolysis was carried out at 1.1 A for 2 hours. The quality of both the planchet and α spectra was quite satisfactory. The chemical yield was about 70 % which is confirmed in literature by other workers. Using two 100 g soil samples chemical yields of 19 and 99 % were measured. These nonuniform results were always observed if no wet ashing before electrolysis with H_2SO_4/HNO_3 was performed. Since this wet ashing is very time consuming we checked another procedure using an oxalate/HCl medium [5]. With pure tracer solution the following results were achieved.

Table 4: Chemical Yields of Am and Cm Electroplating form Oxalate/HCl Medium.

Sample No.	Tracer used	Chemical yield in %
1 2 3	Am-241	90.5 100.2 101.4
Mean value		97 ± 6
1 2 3 4	Cm-244	90.0 81.7 84.7 99.0
Mean value		89 ± 7

Since the chemical yields were higher than the values obtained by the H_2SO_4 procedure and the α spectra were also pretty well, we applied this method to analyses on real and simulated samples. The chemical yields were acceptable. The results are given in Table 5.

Table 5: Chemical Yields of Oxalate/HCl Electroplating of Am and Cm. Further Explanation, see Text.

Experiment No.	Tracer used	Chemical yield in %
1		71
2		100
3	Am-241	71
4		35
5		73
Mean value		70 ± 23
6/1		100
6/2	Am-241	82
6/3		85
7	Am-241 + Cm-244	81
8	Am-241	92
9	Cm-244	78
Mean value		84 ± 7

Using the full analyses for blank, plant or soil samples (experiments 1 - 5) a mean value of 70 ± 23 % for the electroplating of Am-241 tracer was obtained. The very seldom but also during later analyses oberservable low values are to accept since chemical yield is individually determined for each analysis.

Nevertheless we observed a yellow to redbrown ring in the anion exchanger column which was used for lanthanides separation during leaching of Am and Cm. It was proved that the ring was caused by trace content of iron in the $(NH_4)SCN$ solution and that Am-241 (as very likely Cm) was eluated together with this ring.

In experiment 6 we divided the eluation volumes in one part which was collected before the ring reached the end of the column, 6/1, and in parts containing the yellow ring and the later solution, 6/2 and 6/3.

So we decided to use another anion exchanger column to clean the $(NH_4)SCN$ solution before application to analysis (see 2.1). The result was an increased chemical yield of electrodeposited Am and Cm and a decrease of standard deviation as could be demonstrated with the experiments 7 - 9.

4.4. Losses During the Particular Chemical Steps of the Procedure

Using Am-241 as a tracer, the α activity of each solution and solid collected during the analysis was determined and compared with 100 % activities in the same solutions and solids. The results are presented in Table 6.

Table 6: Losses of Am During Single Radiochemical Steps of Am + Cm Analysis.

Analytical step which was controlled. Content of Am in:		Losses of Am-241 in %
Residue of 100 g soil		0.2 - 1.9
H_4SiO_4 precipitate after pH adjustment		0.08 - 3.9
HNO_3 after extraction of Am		< 0.2 - 0.6
Wash solution:	100 ml 0.1 M HNO_3	0.08 - 1.4
	150 ml 0.1 M HNO_3	1.5
	200 ml 0.1 M HNO_3	0.5 - 4.2
	100 ml 0.8 M HNO_3	4.0 - 6.6
Kieselgur/TOPO, after elution		0.2 - 0.3
Regeneration of Kieselgur/TOPO for further analyses:		
100 ml 8 M HNO_3	1st 50 ml	1.0
	2nd 50 ml	0.07
150 ml 8 M HNO_3		1.9
200 ml 4 M HNO_3	1st 50 ml	0.03
	2nd 50 ml	0.007
	3rd 50 ml	0.006
	4th 50 ml	0.007
10 ml 9 M HCl for cleaning the anion and cation exchanger		1.2
Anion and cation exchanger after cleaning		0.1
Solution after passing the anion exchanger		< 0.001 - 4.1
Wash solution of 30 ml 1 M HNO_3/CH_3OH, 83 %		< 0.001 - 1.5
60 ml of 1 M $HCl/CH_3OH/NH_4SCN$ after passing through anion exchanger		0.02 - 0.05
Anion exchanger		0.14 - 2.0

5. Results

5.1. The Radiochemical Procedure

1) The ashed sample material is covered with 290 ml 0.9 M HF/8 M HNO_3 and boiled for 30 min. Cooling and centrifugation.
2) The solution is separated and the residue again is covered by 250 ml 5 M HNO_3/1 M $Al(NO_3)_3$ and boiled for 30 min. Cooling and centrifugation. Combining of the two solutions and discard the residue.
3) The pH of the solution is adjusted to 1.0 - 1.3 using NH_3, 25 %.
4) Transfer the solution to a column with TOPO/diethylbenzene/Kieselgur. The flow is 10 ml/min. At the same flow rate it is washed with 150 ml 0.1 M HNO_3.
5) Am and Cm are eluated with 150 ml 2 M HNO_3, flow rate 5 ml/min.
6) The eluat is twice extracted using $CHCl_3$. Discard the $CHCl_3$.
7) Evaporation to dryness. Dissolution in 10 ml 9 M HCl.
8) These 10 ml 9 M HCl are transfered to an ion-exchanger column. It contains an anion and a cation exchanger. Flow rate 0.5 ml/min. Two times washing with 10 ml 9 M HCl. Evaporation to dryness.
9) The residue is dissolved with 10 - 20 ml 1 M HNO_3/CH_3OH, 93 %. Transfer to an anion exchanger column. Flow rate 0.5 ml/min. The solution is discarded.
10) Wash with 50 ml 1 M HNO_3/CH_3OH, 93 %, using five 10 ml portions. Flow rate 0.5 ml/min. Wash with 60 ml 0.1 M HCl/0.5 M NH_4SCN/CH_3OH. Flow rate 0.5 ml/min.
11) Elution of Am and Cm with 60 ml 1.5 M HCl/CH_3OH, 86 %. Flow rate 0.5 ml/min. Evaporation to dryness. Dissolution in 10 ml HNO_3, 65 %, and evaporation to dryness. Three times evaporation with 1 ml 9 M HCl each.
12) 0.4 ml 4 M HCl is used to dissolve the almost invisible residue. The beaker is washed three times with 1 ml $(NH_4)_2C_2O_4$, 4 %. Rinse with 0.6 ml H_2O. Each solution is transfered into the electroplating cell.
13) Electroplating at 300 mA for 2 hours. After 2 hours 1 ml NH_3, 25 %, is added, 1 min electroplating is continued then the current is switched off.
14) The solution is discarded. The stainless steel platelet is washed with H_2O and ethanol and heated in a gas flame before α spectrometry.

5.2. The Decontamination Factors

The decontamination factors for important α emitters were determined adding a convenient radiotracer to a sample and performing the full analysis. The activity measured on the stainless steel platelet was compared with the added activity. The results are presented in Table 7.

Table 7: The Decontamination Factor.

Decontamination of	Used tracer activity nuclide/Bq	Decontamination faktor
Plutonium	Pu-239/4100	$> 7.3 \cdot 10^5$
Neptunium	Np-237/3900	$8.9 \cdot 10^3$
Uranium	U-232/3700	$> 6.4 \cdot 10^5$
Thorium	Th-228/3700	$> 2.1 \cdot 10^5$
Radium	Ra-226/2900	$> 4.3 \cdot 10^5$
Polonium	Po-210/2900	$> 5.9 \cdot 10^4$

5.3. Applications

The radiochemical procedure was applied to many different kinds of samples. For chemical yield determination about 0.07 Bq Am-242 was added to environmental samples and about the 10 fold amount for aerosol filters contaminated in gaseous effluents. In Table 8 the results of the analysis of environmental samples are presented and in Table 9, results are given for Am- and Cm-analysis of gaseous effluents.

Table 8: Am-241 and Cm-244 Concentrations in Environmental Samples.

Sample	Concentration in mBq/kg	
	Am-241	Cm-244
Corn	< 0.5	< 0.4
Potato	< 0.	< 0.
Cabbage	0.2	< 0.2
Carrot	< 0.	< 0.
Onion	< 0.2	< 0.2
Tomato	< 0.2	< 0.2
Beans, dry	< 1.7	< 2.3
Ground water	< 0.3	< 0.2
Sediments	30	< 11
Soil	30	< 11

Table 9: Am-241 and Cm-244 Concentrations in Gaseous Effluents of Different Nuclear Installations.

Installation	Concentration in mBq/m^3	
	Am-241	Cm-244
Nuclear fuel reprocessing plant		
1	12	0.9
2	8.3	2.1
3	3.7	< 0.7
Incineration facility		
1	0.5	< 0.01
2	0.1	< 0.01
3	0.8	< 0.01
Pressurized water reactor		
1	0.007	< 0.002
2	0.004	< 0.001
3	0.007	0.003

6. Literature

[1] Terry D. Filer, "Separation of the Trivalent Actinides from the Lanthanides by Extraction Chromatography", Anal. Chem., Vol. 46, pp. 608 - 610 (1974)

[2] C.W. Sill, K.W. Puphal, and F.D. Hindman, "Simultaneous Determination of Alpha-Emitting Nuclides of Radium Through Californium in Soil", Anal. Chem., Vol. 46, No. 12, pp. 1725 - 1737 (October 1974).

[3] C.N. Murray and H. Statham, "Application of a Solvent Extraction Procedure Using Di-2-Ethyl-Hexyl Phosphoric Acid (HDEHP) for the Separation of Transuranic Elements in Environmental Samples", Deutsche Hydrographische Zeitschrift, Hamburg, Jahrgang 29, Heft 2, p. 69 - 75 (1976).

[4] Michael H. Hiatt et al., "Simultaneous Determination of Americium and Curium in Soil", Anal. Chem., Vol. 51, No. 2, pp. 295 - 298 (1979)

[5] Darl Knab et al., "Determination of Americium in Small Environmental Samples", Anal. Chem., Vol. 51 , pp. 1095 - 1097 (1979)

[6] John C. Sheppard et al., "Retention of Neptunium, Americium and Curium by Diffusible Soil Particles", US Environmental Science & Technology, Vol. 13, No. 6 (1979).

[7] Kenneth W. Puphal et al., "Electrodeposition of Alpha-Emitting Nuclides from a mixed Oxalate-Chloride Electrolyte", Anal. Chem., Vol. 44, No. 2 p. 284 -289 (February 1972)

[8] H. Schüttelkopf, "Entwicklung einer Analysenmethode für Plutonium im Femtogramm/Gramm-Bereich und ihre Anwendung auf Umweltproben", KfK 3035, September 1981.

[9] E. Holm, S. Ballestra, R. Fukai, "A Method for Ion-Exchange Separation of Low Levels of Americium in Environmental Materials", Talanta, Vol. 26, pp. 791 - 794 (1979)

[10] N.A. Talvitie, "Electrodeposition of Actinides for Alpha Spectrometry Determination", Anal. Chem., Vol. 44, No. 2, p. 280 (February 1977).