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PROCESSING OF IRRADIATED ^{241}Am TARGETS BY ION EXCHANGE AND EXTRACTION

by

W. MÜLLER, F. MAINO and J.Cl. TOUSSAINT

1970

Joint Nuclear Research Center
Karlsruhe Establishment - Germany

European Transuranium Institute

Paper presented at the 5th Radiochemical Conference,
Zvikov-Castle, Czechoslovakia
19-24 May 1969

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The separation of americium and curium from each other is possible by selective oxidation and precipitation of the americium or by extraction chromatography with quaternary ammonium salts from lithium nitrate solutions.

Several of these methods have been combined to process two sets of ^{241}Am targets, exposed to integrated fluxes of 10^{21} and 1.7 x 10^{22} n/cm^2 respectively. A mixture of ^{241}Am and ^{242}Cm recovered from the first set of targets and containing about 450 mg ^{242}Cm was welded into a platinum capsule. This heat source served to power a radionuclide battery.

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ABSTRACT

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KEYWORDS

AMERICIUM 241
TARGETS
IRRADIATION
NEUTRON BEAMS
SEPARATION PROCESSES
PLUTONIUM 238
PLUTONIUM 242
CURIUM 242
FISSION PRODUCTS
ION EXCHANGE
SOLVENT EXTRACTION
AMMONIUM COMPOUNDS
SALTS
OXIDATION
PRECIPITATION
RECOVERY

CONTENTS

PROCESSING OF IRRADIATED ^{241}Am TARGETS BY ION EXCHANGE AND EXTRACTION(*)

1. The irradiation of ^{241}Am

Transplutonium nuclides can be produced by neutron irradiation of ^{241}Am.

1.1. Nuclear reactions

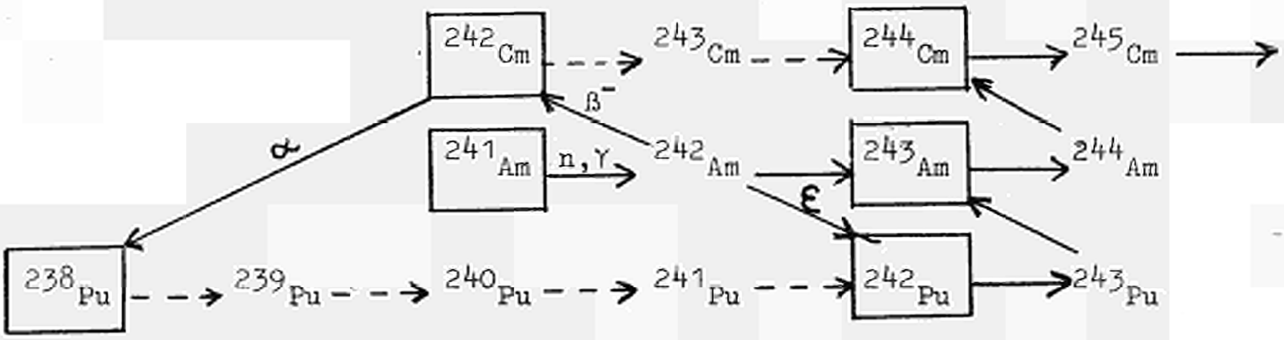

Fig. 1 : Formation of transplutonium nuclides by neutron irradiation of ^{241}Am. Full lines indicate the main build-up path.

Fig. 1 shows in a simplified way the nuclear reactions leading to the formation of curium isotopes. After short time irradiation, part of the starting nuclide still remains; besides fission products, ^{242}Cm, ^{242}Pu and ^{238}Pu are formed, the latter by α-decay of ^{242}Cm. After complete burn up of ^{241}Am, ^{242}Pu, ^{243}Am and ^{244}Cm represent the major part of the heavy nuclides.

1.2. Targets

The targets contain the starting nuclide as alloy or as oxide. The oxide is diluted with MgO or aluminium powder, the mixture is compressed to Cermet pellets. The pellets are, in general, welded into stainless steel or aluminium cladding. Aluminium which is preferred because of its thermal conductivity and crystal plasticity may however become brittle under the influence of fast neutrons.1)

(*) Manuscript received on 18 September 1969.

1.3. Conditions

The first nuclides formed during the irradiation of ^{241}Am are ^{242}Am and ^{242m}Am; both have appreciable fission cross sections which results in a high release of fission heat at the beginning of the irradiation. Therefore it is common practice to begin the irradiation at a moderate flux which is increased corresponding to the decrease of fissile material in the targets (table 1).

Number of cycles*)	Average flux (n/cm^2 . sec)	Integrated flux (n/cm^2)
8	$1{,}5 \cdot 10^{14}$	$1 \cdot 10^{21}$
20	$3 \cdot 10^{14}$	$5 \cdot 10^{21}$
14	$9 \cdot 10^{14}$	$11 \cdot 10^{21}$

Table 1 : Typical increase of neutron flux during ^{241}Am irradiation.

*) 1 cycle : 10 days irradiation, 4 days interruption

1.4. Products

It is difficult to calculate precisely the amount of higher nuclides formed during the irradiation: the real neutron flux, its distribution, its decrease due to selfabsorption, and the dependence of the cross sections of the heavy nuclides on the neutron energy are not known sufficiently.

Targets which had been irradiated under nominally equal conditions contained different quantities of products. Table 2 shows the distribution of the thermal power and the $\beta\gamma$ dose rate for several targets exposed in the same irradiation channel to an integrated flux of 1×10^{21} n/cm^2 and allowed to cool for 250 days. The results reflect the flux distribution in the reactor core.

nb. of capsule according to position	thermal power (W)	$\beta\gamma$ dose rate (10^3 rem/h)
1	17	2
2	18,4	4
3	18,9	6
4	16,1	-
5	-	-
6	15,1	-
7	13,2	2

Table 2 : Power and dose rate distribution of irradiated ^{241}Am targets. Integrated flux: 1 x 10^{21} n/cm^2. Cooling period : 250 d.

The thermal power decayed with a half life of about 160 d, corresponding to the α-decay of ^{242}Cm. After a cooling period of 250 days, the dose rate resulted mainly from the fission products ^{137}Cs-Ba, ^{95}Zr-Nb, ^{106}Ru-Rh and ^{144}Ce-Pr.

2. Processing of irradiated americium targets

2.1. Separation procedures

The recovery of transplutonium elements from irradiated ^{241}Am targets in aluminium cladding comprises in general

- the removal of matrix and cladding aluminium
- the removal of fission products
- the isolation of the plutonium fraction (^{238}Pu, ^{242}Pu)
- the isolation of the transplutonium elements and their separation from each other.

Whereas plutonium may easily be isolated as tetravalent ion from hydrochloric or nitric acid by anion exchange or extraction methods (TBP*), substituted ammonium salts), it is more difficult to separate the trivalent transplutonium elements from lanthanides or from each other.

2.1.1. Actinide - lanthanide separations

Group separations may be performed by anion exchange2) or extraction with organic solutions of tertiary ammonium salts3), the aqueous phase being in general slightly acid, highly concentrated LiCl solutions.
With trilaurylammonium chloride in toluence solutions as the organic phase, extraction coefficients E_a^o (= ratio of the element concentrations in organic and aqueous phase) for transplutonium elements and europium as a function of extractant concentration C are represented in fig. 2.4)
In general, the slope of the curve $\log E^o{}_a = f (\log [C])$ is assumed to equal the ratio of extractant to metal in the complex. On the basis of chemical and spectroscopic evidence the slope 2 was ascribed to the formation of an ion quadrupole5)

$$\begin{array}{ll} TLAH^+ & Cl^- \\ MCl_4{}^- & TLAH^+ \end{array}$$

Quite recently, the existence of tetrachloro complexes in organic phases has been confirmed by RAMAN spectroscopy6). From the extraction coefficients, actinide - lanthanide separation factors of 10^2 may be calculated.

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*) tributylphosphate

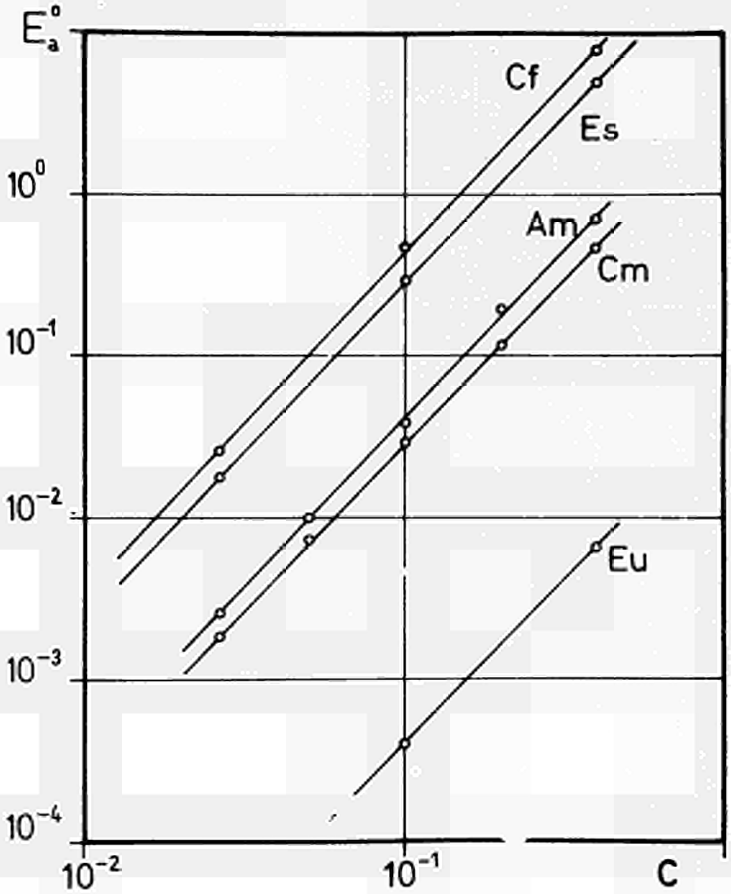

Fig. 2 : Extraction coefficients of transplutonium elements and europium as a function of extractant concentration. Organic phase: trilaurylammonium chloride in toluene. Aqueous phase: 6.3 M LiCl; 2.7 M $CaCl_2$; 0.01 M HCl.

Similar lanthanide - actinide group separations may also be achieved by the TALSPEAK[7)]-process or variations thereof, where the organic phase is a diluted solution of HDEHP[xx)] in the presence of an aqueous phase containing complexing agents like lactic acid and substituted aminopolyacetic acids (DTPA)[xxx)] (Fig. 3).

[xx)] di-(2 ethyl-hexyl)phosphoric acid

[xxx)] diethylenetriaminopentaacetic acid

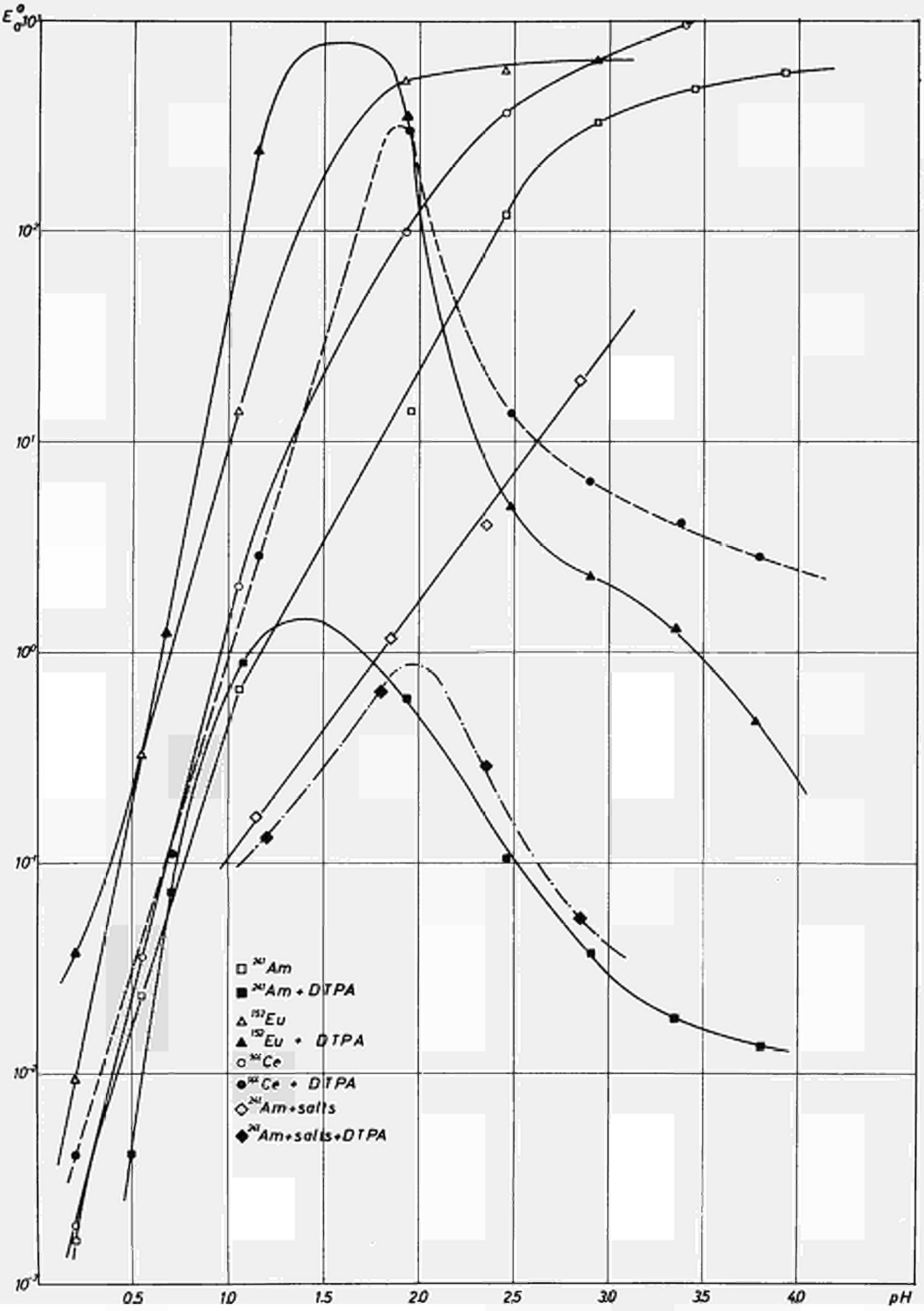

Fig. 3 Extraction coefficients of ^{241}Am, ^{152}Eu, ^{144}Ce as a function of p_H. Organic phase: 0.5 M HDEHP, in Solvesso. Aqueous phase: 1 M lactic acid (0.05 M DTPA).

2.1.2. Americium - Curium separations

These elements can be separated as trivalent ions by anion exchange $^{8,9,10)}$, extraction or extraction chromatography$^{11,12)}$ from nitric acid or lithium nitrate solutions; other separation methods make use of the ability of americium to be oxidized to oxidation numbers higher than 3 $^{13)}$.

Besides the separation of americium from curium by precipitation of $K_3AmO_2(CO_3)_2$ after peroxidisulphate oxidation, we routinely carry out americium - curium separations by extraction chromatography on kieselguhr columns impregnated with quaternary ammonium nitrate (Aliquat). With aqueous phases of 3.5 to 4.0 M $LiNO_3$ and working at 10°C, separation factors of 3.5 have been obtained.

2.2. Flow sheet

Because of the high α, β, γ and neutron activity of the irradiated targets, the processing necessitates remote handling in hot cells. Therefore, only very simple procedures are adequate and the following flowsheet, based on anion exchange, was adopted (Fig. 4) :

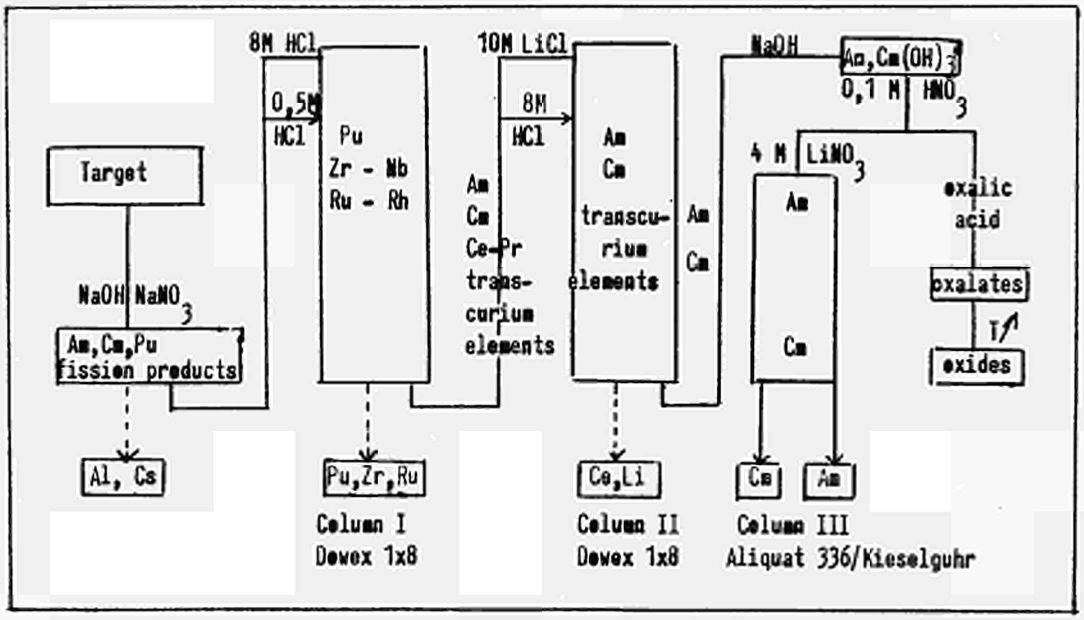

Fig. 4 : Flow sheet for the processing of irradiated ^{241}Am targets

2.3. Problems

During the processing, problems arise which are characteristic for samples containing nuclides of high specific activity like ^{242}Cm :

- α-radiolysis

  Hydrochloric acid solutions containing ^{242}Cm decompose and form H_2, Cl_2, O_2, H_2O_2, O_3 ...; after decomposition of the acid, especially in concentrated chloride solutions, the hydroxides of trivalent actinides or lanthanides precipitate. The products of radiolysis oxidize cerium, in neutral solution also americium to the tetravalent ion.

- Decay heat

  The thermal power of 1 g ^{242}Cm is 120 W. The resin and the aqueous solutions are decomposed by radiation and heat in the bands in which ^{242}Cm is concentrated on the exchanger column. Despite external cooling, solid samples of ^{242}Cm may become red hot.

- Corrosion and erosion

  The products of radiolysis and the Cm containing concentrated solutions readily attack most metal, rubber and plastic parts of the equipment. Under the influence of the recoil of α-decaying atoms, glass particles have been observed to flake off the walls of glass vessels 7)

In order to overcome such and other problems, the equipment had to be carefully studied.

The resin beds were compressed between porous polyethylene disks in order to prevent the formation of bubbles of radiolysis gases plugging the column. The elution speed was increased - and thus attack of the resin reduced - by applying a gas pressure. It is interesting to note that at ORNL where the TRAMEX process (extraction by tertiary ammonium salts) is the common separation procedure, the final purification is made by ion exchange in high pressure columns$^{14)}$.

3. Results

2 sets of irradiated Am targets were available

- 3 capsules prepared at Livermore and irradiated in BR2 up to an integrated flux of $1.7 \times 10^{22} n/cm^2$,
- 9 capsules prepared at Karlsruhe and irradiated in BR2 up to an integrated flux of $1 \times 10^{21} n/cm^2$.

3.1. High integrated flux

The processing of the first of the 3 capsules served to check the methods, equipment and hot cell facilities (fig. 5).

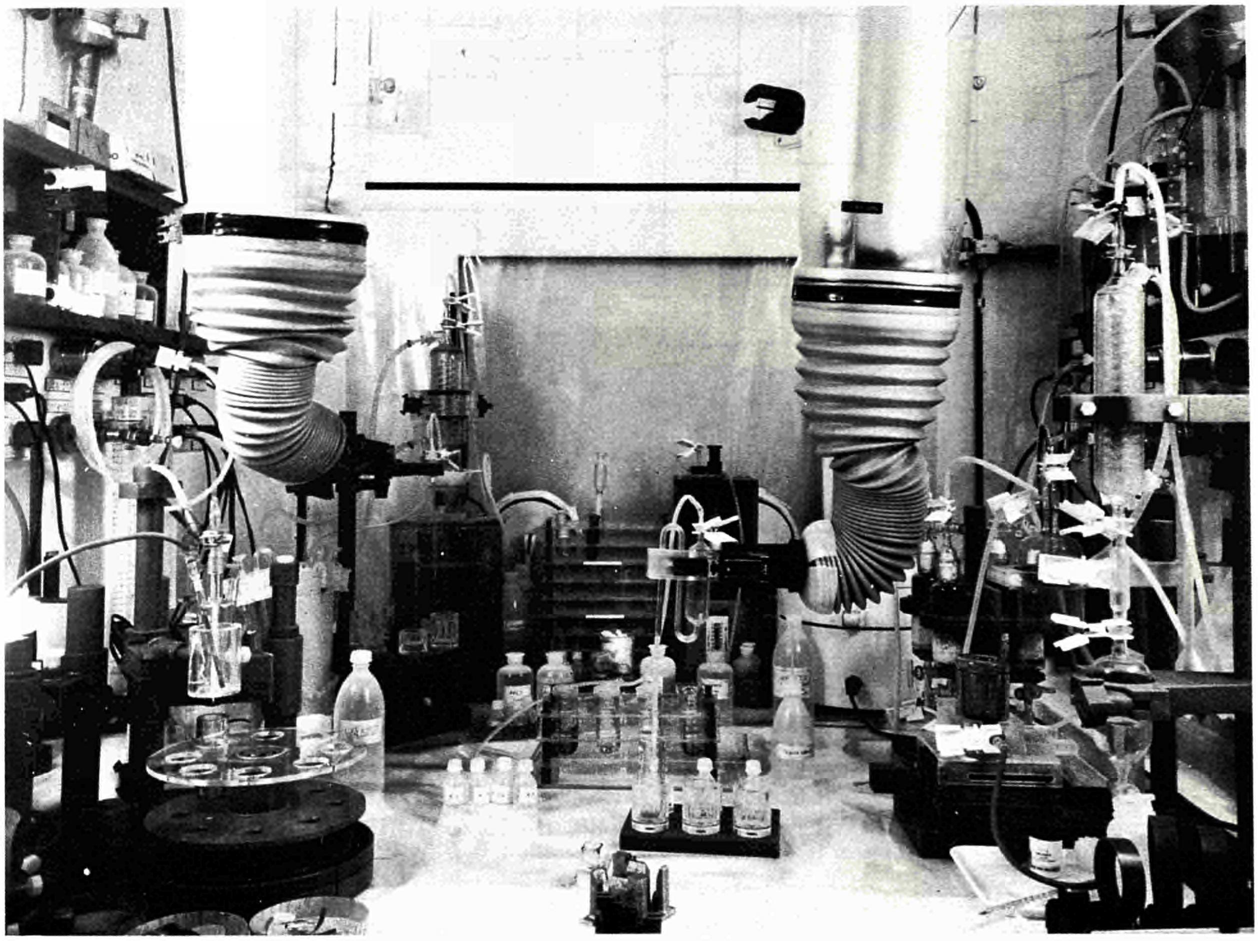

Fig. 5 : Interior of a hot cell during the processing

The presence of ^{238}Pu in the plutonium fraction (^{238}Pu/^{242}Pu=0.45) made it easy to follow its removal by α-counting (Fig. 6). The fractional elution of both lanthanides and actinides by 10 M LiCl,

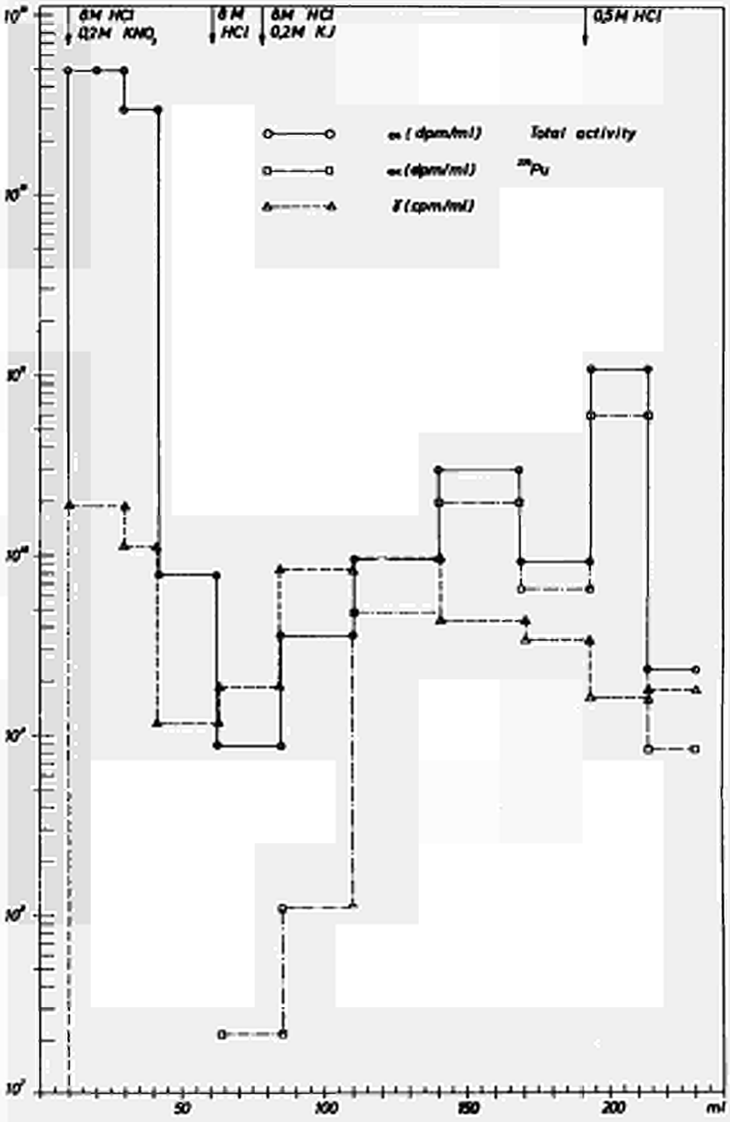

Fig. 6 Removal of the plutonium fraction and some of the fission products. Dowex 1 x 8.

0.01 M HCl, 0.1 M NH_2OH HCl, 5% CH_3OH leads to a transplutonium fraction in a large volume of concentrated salt solution (Fig. 7). If, however, this procedure is modified in such a

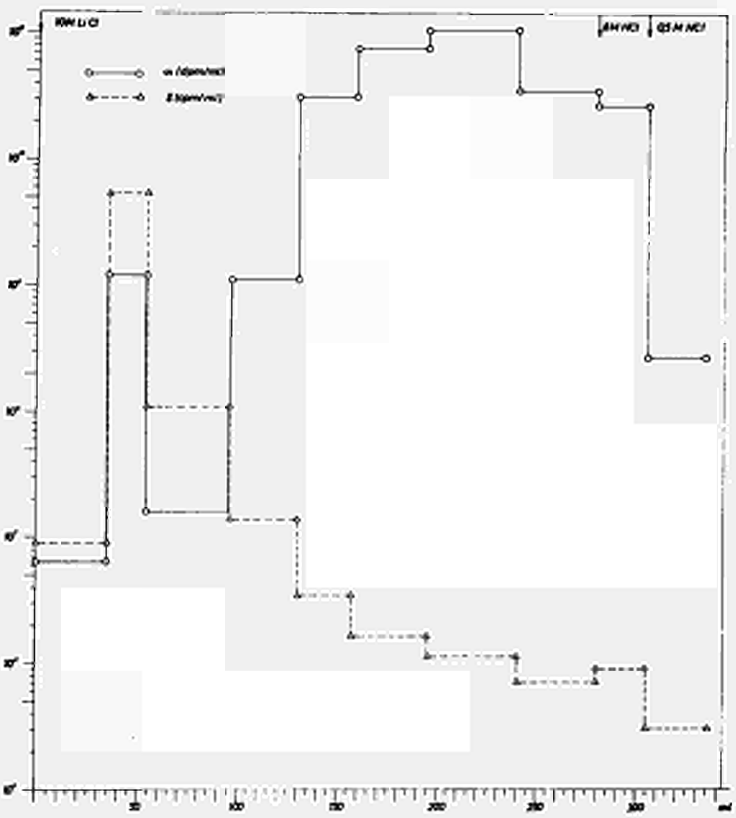

Fig. 7 Separation of trivalent lanthanides and transplutonium elements on Dowex 1 x 8 by fractional elution with 10 M LiCl, 0.01 M HCl, 0.1 M NH_2OHHCl, 5% CH_3OH.

manner that the americium-curium fraction is eluted by 6 M HCl, the transplutonium elements are collected in a smaller volume which is only about 2 M in LiCl and, thus, facilitates the recovery of the trivalent elements (Fig. 8).

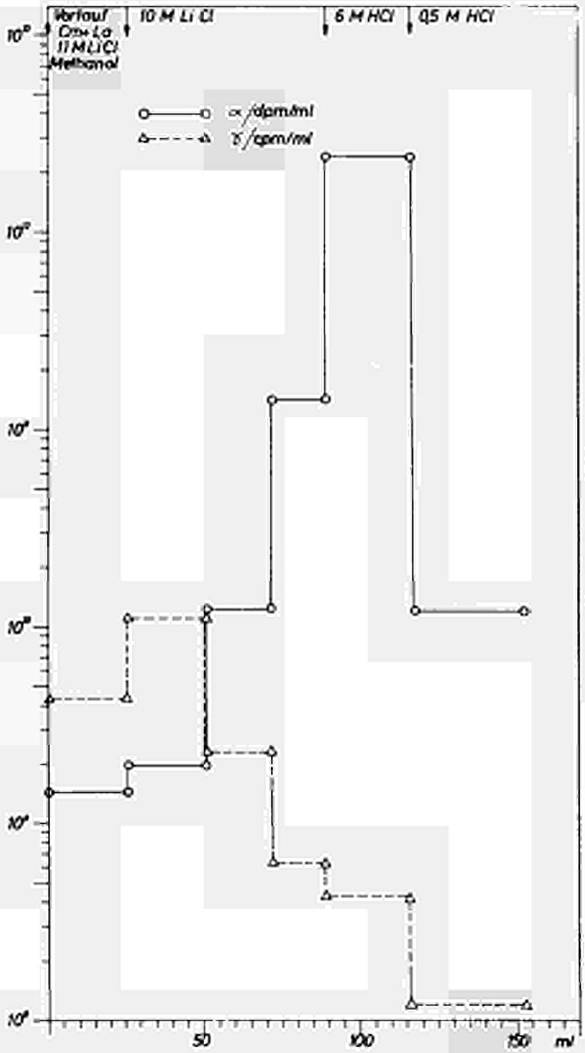

Fig. 8 Separation of trivalent lanthanides and transplutonium elements. Elution of the lanthanides by 10 M LiCl; elution of the Am-Cm fraction by 6 M HCl.

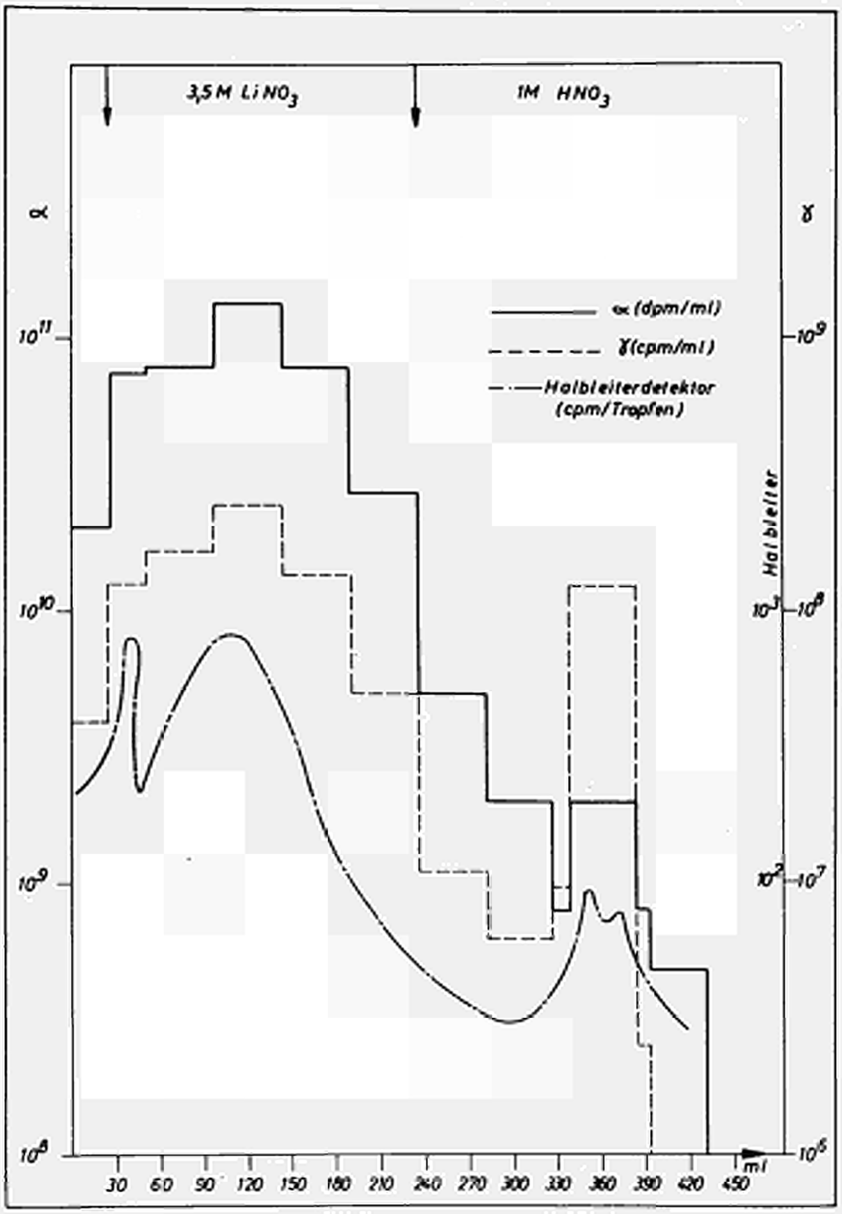

Fig 9 : Separation of ^{244}Cm and ^{243}Am by extraction chromatography on quaternary ammonium (Aliquat-336) nitrate/kieselgur.

——— Total α activity (dpm/ml)
------ Total γ activity (cpm/ml)
—.—. α-sensitive solid state detector (cpm/drop)

Elution of the major part of the ^{244}Cm by 3.5 M LiNO$_3$; elution of the ^{243}Am fraction by 1 M HNO$_3$.

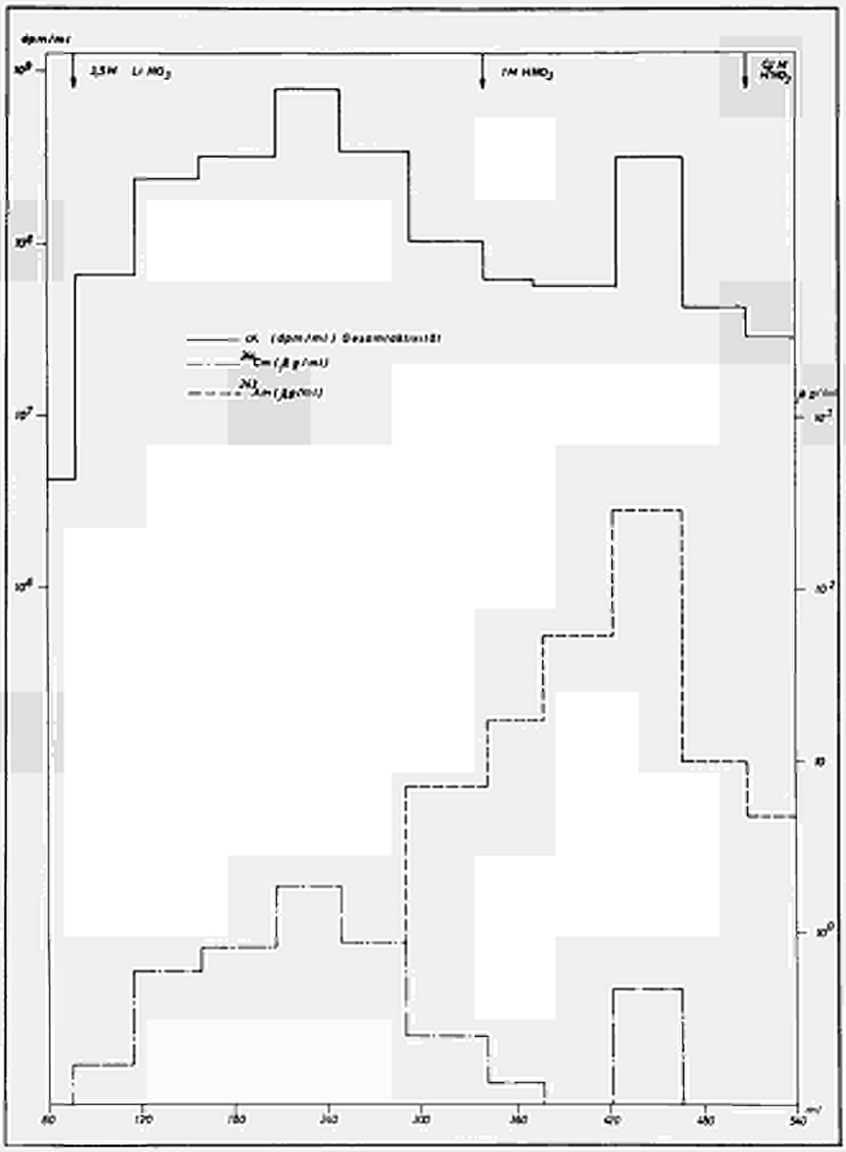

Fig. 10 : Removal of ^{244}Cm from ^{243}Am by extraction chromatography on quaternary ammonium (Aliquat-336) nitrate/kieselgur.

——— Total α activity
—·—· μg/ml ^{244}Cm
----- μg/ml ^{243}Am

Elution of the Cm fraction by 3.5 M LiNO$_3$; elution of the Am fraction by 1 M HNO$_3$.

After 2 successive Am-Cm separations by extraction chromatography (Fig. 9, 10), ^{243}Am with less than 200 ppm Cm was obtained. The curium fraction had the isotopic composition (measured and converted to the values for the end of the irradiation):

nuclide	mg/g starting nuclide
^{242}Cm	64
^{243}Cm	2
^{244}Cm	62
^{245}Cm	1
^{246}Cm	2

Table 3 : Isotopic composition of the Curium fraction.

3.2. Low integrated flux

From part of the 9 capsules containing several grams of ^{238}Pu, ^{242}Pu, ^{241}Am and more than 1 g of ^{242}Cm, the heavy nuclides were recovered[15]. It was decided to fuel a radionuclide battery with the remaining ^{242}Cm in order to demonstrate the potential use of transplutonium nuclides for power production [16][17].

The separations on the columns were controlled by solid state α and neutron counting. After the removal and desorption of the plutonium fraction, the ion exchange column was dark coloured, possibly because of the presence of ruthenium complexes. The removal of the lanthanides could be followed from the radioluminescence bands of the actinides and lanthanides on the column (Fig. 11).

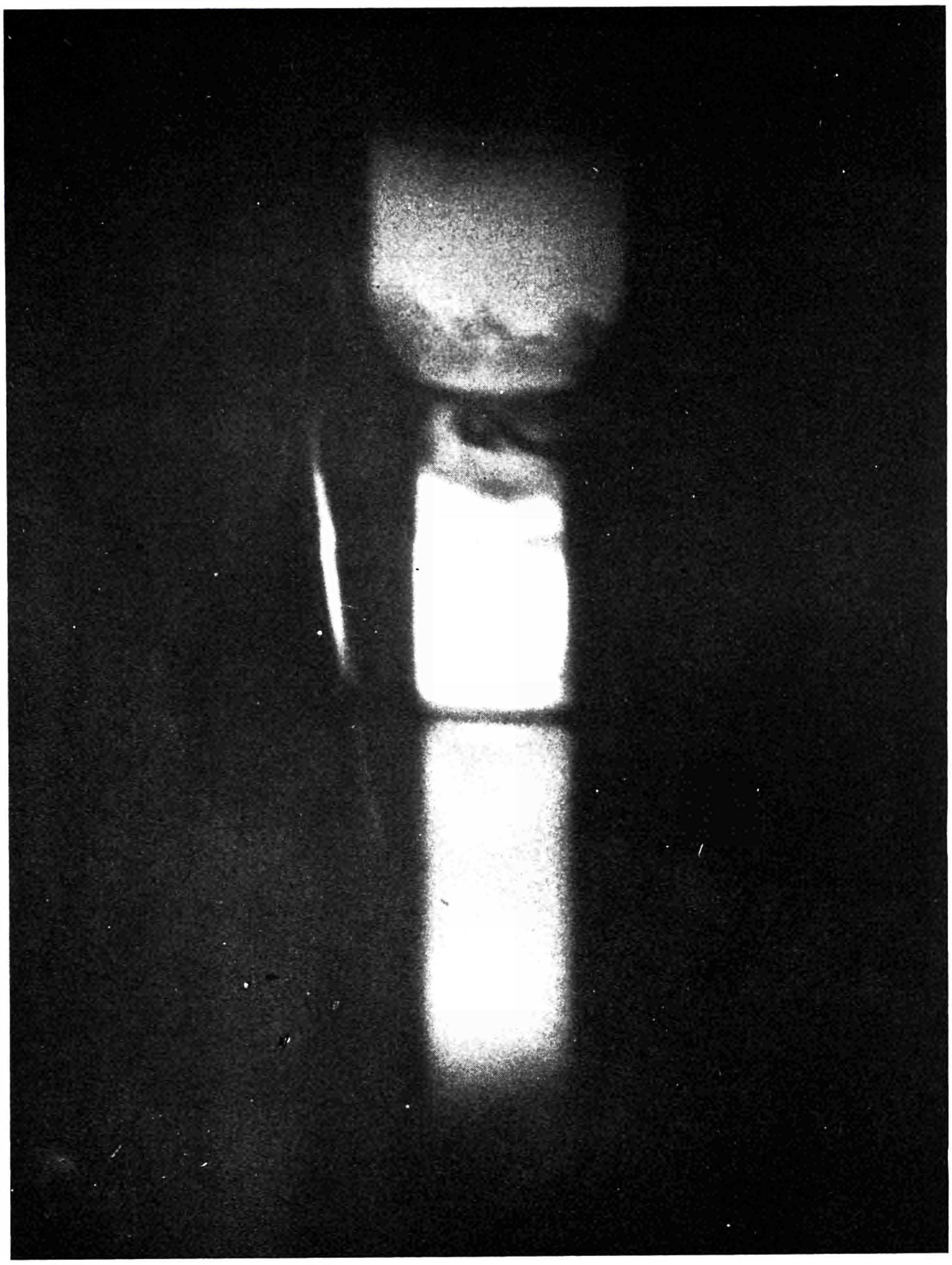

Fig. 11 Radioluminescence of the actinide (Cm:orange red) and lanthanide bands on the ion exchange column.

A comparison of the gamma spectra before and after the separations (Fig. 12) showed that a decontamination factor of approximately 10^3 is achieved for the most important fission products.

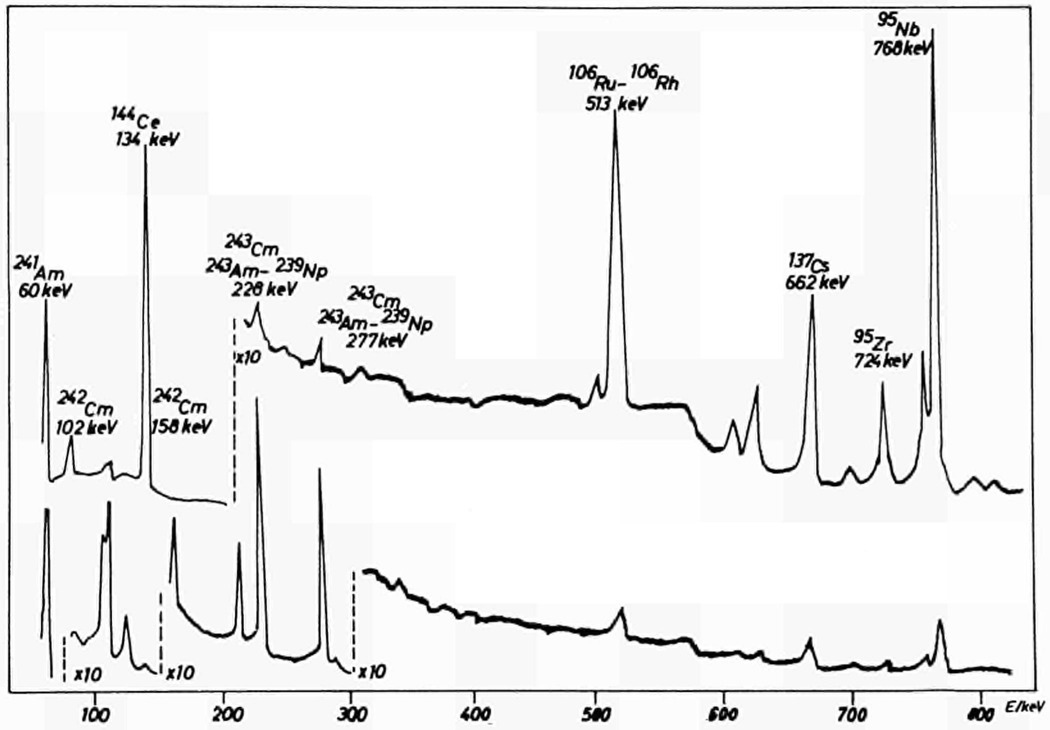

Fig. 12 Gamma spectra of the feed solution before the plutonium removal and of the ^{241}Am-^{242}Cm fraction after removal of the lanthanides.

A mixture of americium and curium was precipitated by oxalic acid. As soon as the oxalates were drying on the glass filter, they were transformed into black oxides by the α-decay heat. Despite intensive cooling of the platinum crucible which contained the americium and curium, the oxide mixture glowed.

About 5 g of the oxide mixture containing more than 450 mg ^{242}Cm were welded into a double-walled platinum capsule. The capsule was connected to a cooling water supply and transferred into a line of 3 glove-boxes shielded against neutron radiation by 25 cm of water.

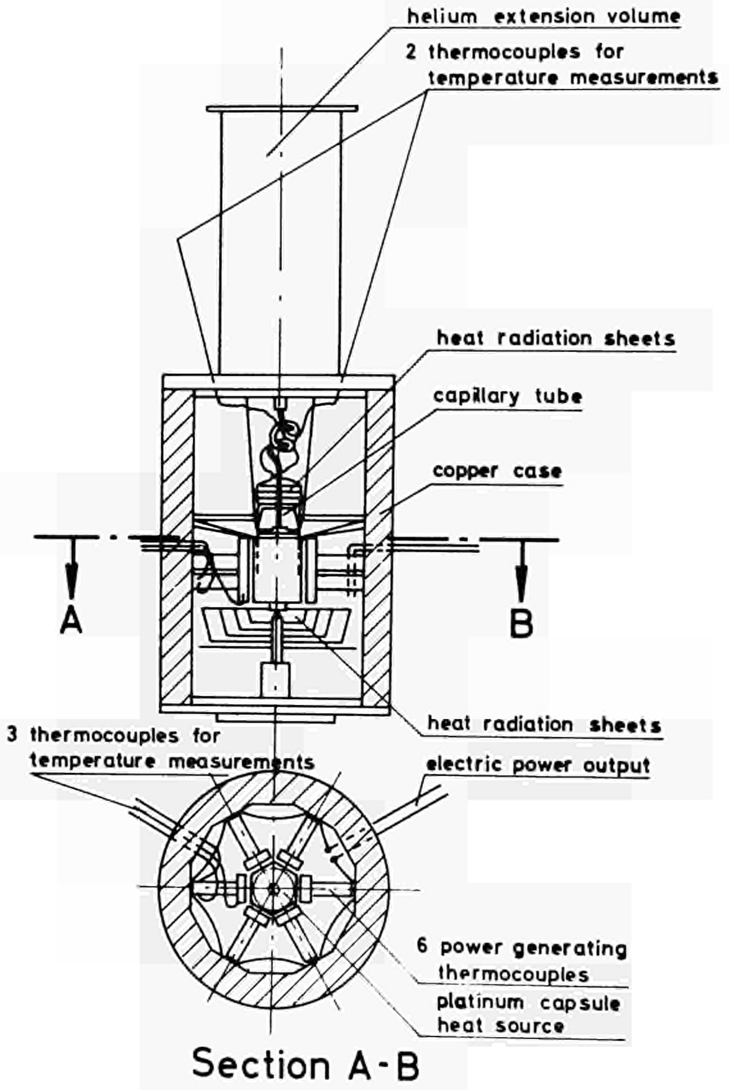

Fig. 13 Scheme of the radionuclide battery.

In these glove-boxes, the platinum capsule was decontaminted, welded a second time into a hexagonal capsule and inserted into a thermoelectric converter containing 6 power thermocouples in a hexagonal arrangement (Fig. 13). In vacuum, the surface temperature of the platinum capsule exceeded 750°C.

Fig. 14 shows the radionuclide battery in operation.

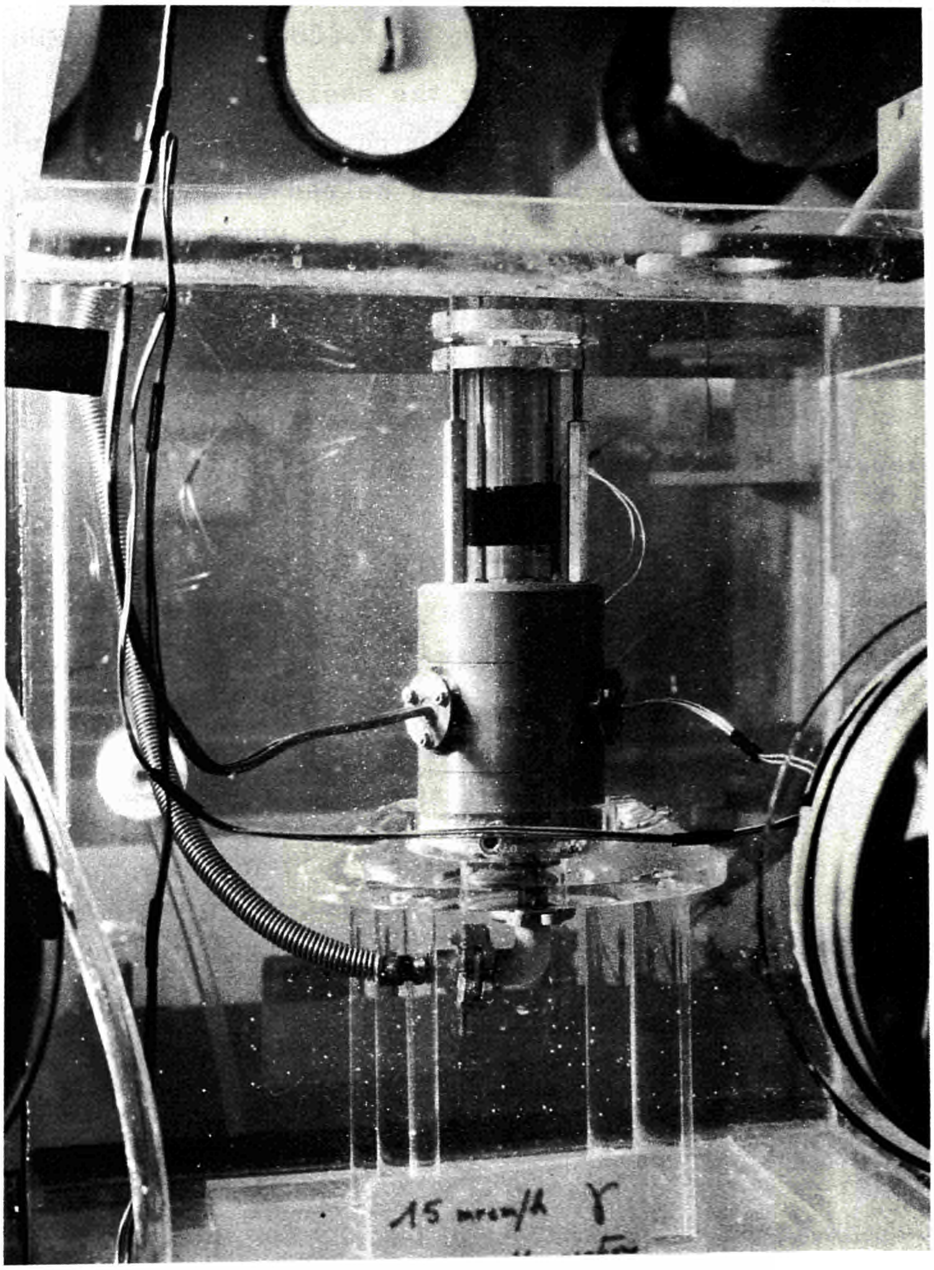

Fig. 14 Radionuclide battery in operation.

After the processing of metallographic samples and waste solutions, about 10 mg ^{242}Cm had to be separated from a tenfold amount of ^{241}Am. The precipitation of $K_3AmO_2(CO_3)_2$ reduced the americium amount by 95 %. The final separation was carried out by extraction chromatography with quaternary ammonium nitrate.

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Alfred Nobel

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