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MASTER

AEC RESEARCH AND DEVELOPMENT REPORT

CURIUM PROCESS DEVELOPMENT

I. GENERAL PROCESS DESCRIPTION

I. D. EUBANKS and G. A. BURNEY

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Savannah River Laboratory

Aiken, South Carolina

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Chemical Separations Processes
for Plutonium and Uranium
(TID-4500)

CURIUM PROCESS DEVELOPMENT

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I. GENERAL PROCESS DESCRIPTION

by

I. Dwaine Eubanks
Glenn A. Burney

Approved by

L. H. Meyer, Research Manager
Separations Chemistry Division

April 1966

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ABSTRACT

Chemical processing steps to purify ^{244}Cm produced in Savannah River reactors are described, and specifications on radioactive impurities in the product are stipulated.

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FIG. 1 PRODUCTION OF ^{244}Cm BY SUCCESSIVE NEUTRON CAPTURES

FIG. 2 CURIUM PRODUCTION PROGRAM

CURIUM PROCESS DEVELOPMENT

I. GENERAL PROCESS DESCRIPTION

INTRODUCTION

^{244}Cm is produced from ^{239}Pu in successive reactor irradiations at Savannah River.⁽¹⁾ The sequence of nuclear reactions to produce ^{244}Cm is shown in Figure 1; the Savannah River program for producing ^{244}Cm in several irradiation stages is depicted graphically in Figure 2. Initially ~3 kg of ^{244}Cm will be produced for evaluation as a source of isotopic power.

In the first irradiation, ^{239}Pu either fissions or captures neutrons to produce heavier actinide isotopes. The mixture of plutonium isotopes that is produced is recovered and fabricated into targets for a second irradiation, in which a portion of these plutonium isotopes is converted to ^{242}Pu , ^{243}Am , and ^{244}Cm . From the targets of this second irradiation the ^{244}Cm is separated and purified; the ^{242}Pu and ^{243}Am are recovered and can be used for subsequent irradiations to produce additional ^{244}Cm .

This report is the first in a series that will describe the development of separations processes for purifying ^{244}Cm produced in Savannah River reactors. The series will be issued under the general title Curium Process Development. Following the general title, a roman numeral will designate the subject area of the report, and an arabic numeral, the series report number in that subject area. A subtitle will describe the content of each report. Subject areas foreseen for this series are:

- I. General Process Description
- II. Chemical Processing Steps
- III. Analytical Chemistry Support

SUMMARY

Chemical processing steps have been selected to purify curium produced from plutonium targets irradiated in Savannah River reactors. Plutonium isotopes are removed by solvent extraction with 3.5% tri-n-butyl phosphate (TBP) in kerosene. Aluminum and some of the nonlanthanide fission products are then rejected during a batch extraction of the trivalent actinides into 50% TBP in kerosene. Trivalent lanthanides are subsequently rejected during extraction of the trivalent actinides with a mixture of tertiary amine hydrochlorides in diethylbenzene. Californium may be removed in this step by back-extraction of americium and curium

into lithium chloride solution, or it may be removed by ion exchange at a later stage of the process. Americium and curium are separated by precipitating Am(V) in a carbonate solution, and then are converted to the oxalates, which are calcined to the oxides.

Purity specifications for the curium oxide product are stipulated to insure that impurities will contribute gamma and neutron dose rates that are less than 10% of the respective rates for pure curium oxide.

DISCUSSION

DESCRIPTION OF PROCESS

Chemical processing to purify curium requires steps to separate the curium from other actinides, aluminum alloyed with and cladding the target elements, and fission products. In addition, plutonium and americium must be recovered for subsequent irradiations. The most abundant radioactive fission products present in the plutonium targets after six months cooling are expected to be ^{106}Ru - ^{106}Rh , ^{144}Ce - ^{144}Pr , ^{95}Zr - ^{95}Nb , ^{134}Cs , ^{137}Cs , ^{91}Y , ^{90}Sr - ^{90}Y , ^{147}Pm , ^{103}Ru , and ^{127}Te .

The curium purification process to be used at Savannah River is shown in Figure 3, and is discussed in the following sections.

Removal of Plutonium, Aluminum, and Nonlanthanide Fission Products

Irradiated plutonium targets are dissolved in nitric acid,⁽²⁾ and the plutonium is removed by solvent extraction with 3.5% TBP in kerosene⁽³⁾ as shown in Figure 4. Aluminum, trivalent actinides, and fission products remain in the aqueous stream in the first solvent extraction cycle.

The trivalent actinides and lanthanides are removed from the aqueous stream by batch extraction into 50% TBP in kerosene.⁽⁴⁾ Aluminum and the bulk of the nonlanthanide fission products are not extracted. The actinide-lanthanide mixture is quantitatively back-extracted into 0.2M HNO_3 and concentrated by evaporation.

Both of the above processing steps have been demonstrated in the Savannah River separations plant.

FIG. 3 PROCESSING STEPS TO PURIFY CURIUM

FIG. 4 PLUTONIUM AND ALUMINUM REMOVAL

Removal of Lanthanides

New processing steps are required for the remainder of the purification. The most difficult of these steps is the separation of trivalent curium and americium from chemically similar lanthanide fission products. A new solvent extraction method, originally conceived at Oak Ridge National Laboratory,^(5,6) was selected for the lanthanide removal step.

The nitrate solution from the aluminum removal step must be converted to a chloride solution to provide suitable feed for the lanthanide removal step. The nitrate-to-chloride conversion step selected for development is based on another Oak Ridge process.⁽⁷⁾ As shown in Figure 5, a mixture of tertiary amine nitrates in diethylbenzene is used to extract both actinides and lanthanides. The actinides and lanthanides are then back-extracted into concentrated hydrochloric acid, while the nitrate ion is retained by the solvent.

In the lanthanide removal step (shown in Figure 6), a mixture of tertiary amine hydrochlorides in diethylbenzene preferentially extracts trivalent actinides from trivalent lanthanides in concentrated lithium chloride. The actinides are subsequently back-extracted into concentrated hydrochloric acid.

Development of the tertiary amine solvent extraction steps for purifying large quantities of curium at Savannah River includes:

- Adapting the process to the specific feed material involved in a large-scale production program, and to the specific purification requirements for ^{244}Cm isotopic heat sources.
- Determining differences in process performance caused by radiation-induced chemical changes in the solutions, and modifying the process as necessary to compensate for these changes.
- Demonstrating the process with simulated feed solutions.
- Developing solvent washing procedures to permit solvent recycle.

Removal of Residual Nonlanthanide Fission Products

Nonlanthanide fission products remaining after the removal of the plutonium and aluminum are also removed in the tertiary amine solvent extraction steps. Of the nonlanthanides, only ^{106}Ru - ^{106}Rh and ^{95}Zr - ^{95}Nb are present in significant concentrations in the curium stream after the aluminum removal step. Ruthenium and zirconium are removed in both solvent extraction steps.

FIG. 5 NITRATE - TO - CHLORIDE CONVERSION

FIG. 6 LANTHANIDE REMOVAL

Removal of Americium

Americium is separated from curium by oxidation to AmO_2^+ in K_2CO_3 solution and precipitation as the double carbonate, $\text{K}_5\text{AmO}_2(\text{CO}_3)_3$, which is filtered from the soluble Cm(III) complex. Selection of this method was based on solubility information,⁽⁸⁾ small-scale tests by others,^(9,10,11) and studies of the stability of the precipitate. The steps in the process for separation of americium from curium by this method are shown in Figure 7.

Development of the carbonate precipitation at Savannah River includes:

- Selecting and demonstrating a method for converting the high-acid, dilute Am-Cm product from the amine extraction to a low-acid, concentrated Am-Cm stock usable for precipitation of the americium.
- Determining effects on process performance of radiation-induced chemical reactions at high curium concentrations.
- Selecting and demonstrating a method for recovering the curium from the K_2CO_3 solution.
- Evaluating the usefulness of the method as a routine production operation.

Removal of Californium

Two alternatives have been evaluated for removing californium from the curium stream.

- The back-extraction operation of the lanthanide removal step may be modified to remove californium.
- Californium may be removed by ion exchange chromatography of the curium stream after removal of americium.

The processing step selected for californium removal will be determined by the results of laboratory demonstrations of these alternatives.

The solvent extraction process for lanthanide removal modified for removal of californium is illustrated in Figure 8. The modified process functions because tertiary amine solutions extract californium more strongly than curium from concentrated lithium chloride.⁽⁵⁾ By substituting lithium chloride for hydrochloric acid in the back-extraction solution, the salting strength can be

FIG. 7 REMOVAL OF AMERICIUM AND PREPARATION OF OXIDES

FIG. 8 CALIFORNIUM REMOVAL BY SOLVENT EXTRACTION

adjusted so that curium will back-extract into the aqueous phase, and californium will be retained in the solvent.

Curium can also be separated from higher actinides including californium by absorption on cation resin followed by elution with a chelating organic acid anion. Several such acids—citric, glycolic, lactic, and α -hydroxyisobutyric—have been used successfully; the latter acid gives the largest separation factors and is generally preferred.^(12,13) Californium has been separated from several grams of curium with an ammonium α -hydroxyisobutyrate elutriant. Even though ion exchange gives good separation, the solvent extraction technique is favored at this time because of the number of batch operations required to process production quantities of curium by ion exchange.

Preparation of Curium Oxide and Americium Oxide

Curium is recovered from the concentrated carbonate solution by acidification followed by addition of potassium hydroxide to precipitate $\text{Cm}(\text{OH})_3$. The $\text{Cm}(\text{OH})_3$ is dissolved in dilute nitric acid, and curium(III) oxalate is precipitated with oxalic acid. The light-green oxalate is removed by filtration, washed, dried with air, and calcined in air to curium dioxide.

To produce americium dioxide, the $\text{K}_5\text{AmO}_2(\text{CO}_3)_3$ is first dissolved in dilute nitric acid containing a reductant to yield a solution of americium(III). Americium(III) oxalate is precipitated with oxalic acid. The tan precipitate is washed, dried with air, and calcined in air to black americium dioxide. The steps in the processes for preparation of curium oxide and americium oxide are shown in Figure 7.

Development of the steps for preparation of large quantities of curium oxide and americium oxide at Savannah River includes:

- Selecting and demonstrating the methods for recovery of curium from the concentrated carbonate solution.
- Evaluating the usefulness of the methods for routine production operation.

PURIFICATION REQUIREMENTS

The initial curium produced in the Savannah River program will be used for evaluation of ^{244}Cm in isotopic power sources.⁽¹⁾ The product must have the following characteristics to be suitable for encapsulation in power sources:

- The dose rate due to gamma photons and neutrons from the purified product must not be significantly increased by emissions from impurities.⁽¹⁴⁾
- No impurity may be present in high enough concentration to affect the thermal stability of possible fuel forms.

Limits for Radioactive Impurities

Increases in the gamma and neutron dose rates due to impurities are considered insignificant so long as they remain $\leq 10\%$ of the respective rates from pure curium oxide.

The contribution of impurities to the gamma dose rate of the encapsulated product varies with the amount of shielding incorporated in the power source assembly because the effectiveness of the shielding varies with the energy of photons being emitted, and because the impurities do not have the same gamma energy distribution as ^{244}Cm . The shielding requirements for the proposed curium power sources have not been firmly stipulated; hence, to assure acceptable product quality in all cases, the purity criteria must be met for any shielding configuration.

Required decontamination factors have been calculated for curium purification in the 3-kg pilot production program. These decontamination factors are based on the anticipated composition of irradiated mixed plutonium isotopes shown in Table I. The decontamination required from any single gamma-emitting impurity depends on the decontamination achieved from other gamma emitters. The interrelationships of required decontamination factors for the major gamma-emitting impurities, ^{144}Ce - ^{144}Pr , ^{106}Ru - ^{106}Rh , and ^{93}Zr - ^{95}Nb , are shown in Figure 9. Details of the calculations are given in the Appendix.

Purification requirements set for fission product removal during processing in the 3-kg pilot program will also assure acceptable curium when ^{242}Pu or ^{243}Am targets are irradiated, because fewer fission products are produced in these targets than in the mixed plutonium isotopes of the 3-kg irradiation.

FIG. 9 REQUIRED DECONTAMINATION FROM MAJOR FISSION PRODUCT IMPURITIES

FIG. 10 SPONTANEOUS FISSION NEUTRON SPECTRA

^{252}Cf is the only important neutron-emitting impurity in the curium product. Figure 10 shows that the energy spectra of spontaneous fission neutrons are similar for ^{252}Cf and ^{244}Cm ; ⁽¹⁵⁾ hence, the relative contributions of ^{244}Cm and ^{252}Cf neutrons to the total neutron dose rate are proportional to their individual emission rates. The purity criterion for ^{252}Cf in the curium product is that its neutron emission rate must be $\leq 10\%$ the neutron emission rate from pure curium oxide. From Table I, $< 5 \times 10^{-8}$ g of ^{252}Cf is expected to be formed per gram of ^{244}Cm in the initial 3 kg of curium produced. ^{252}Cf emits 2.4×10^{12} neutrons/(sec)(g), while ^{244}Cm emits only 1.09×10^7 neutrons/(sec)(g). ⁽¹⁴⁾ A required decontamination factor of 10 or less is calculated for removal of ^{252}Cf from the 3-kg product. Because more transcurium isotopes would be produced during irradiation of ^{242}Pu and ^{243}Am targets, higher californium decontamination factors would be required for curium produced in these targets.

TABLE I

Anticipated Composition of Targets of Mixed
Plutonium Isotopes Irradiated for Curium Production

<u>Component</u>	<u>Quantity, g/g ^{244}Cm produced</u>
^{242}Pu	4.7
^{243}Am	1.0
^{252}Cf	$< 5 \times 10^{-8}$
Lanthanide fission products	2.4
Other fission products	7.9
	<u>curies/g of ^{244}Cm produced (a)</u>
^{144}Ce - ^{144}Pr	7.5×10^2
^{95}Zr - ^{95}Nb	2.8×10^2
^{106}Ru - ^{106}Rh	8.7×10^2

(a) At 180 days after reactor discharge.

Limits for Other Impurities

Limits for nonradioactive impurities in the curium oxide product are indefinite because little is known about the effects of impurities on thermal stability of possible fuel forms. Processing approximately 0.2 g of curium through all steps (Figure 3) yielded an oxide product with the composition shown in Table II; physical properties of compacts made from this oxide are being determined.

TABLE II

Impurity Content of Curium Oxide

<u>Impurity</u>	<u>Amount of Each, wt %</u>
Am	0.4
Al, Ce	0.2
Ca, Fe, P	0.1
Ba, Bi, Co, Cr, La, Li, Mg, Mo, Na, Nb, Ni, Pb, Sb, Si, Sn, Ti, Zn, Zr	<0.05
Ag, B, Be, Cu, Mn	<0.005

APPENDIX

MAXIMUM QUANTITIES OF GAMMA EMITTERS IN CURIUM PRODUCED FOR ISOTOPIC POWER SOURCES

The gamma dose rate from a radionuclide is approximately proportional to the sum of the products of the number of photons and their energies from 60 kev to 7 Mev.^(1e) For any arbitrarily selected shielding configuration the maximum quantities of residual impurities allowed in the curium product are determined by their photon emission rates and energies according to the formula:

$$\sum_{\text{imp}} \left[\sum_i [N_{(\text{imp})}]_i [E_{(\text{imp})}]_i \right] \leq 0.1 \sum_i [N_{(\text{Cm})}]_i [E_{(\text{Cm})}]_i \quad (1)$$

where $N_{(\text{imp})}_i$ = number of gamma photons penetrating the shielding from each impurity at energy $E_{(\text{imp})}_i$

$N_{(\text{Cm})}_i$ = number of gamma photons penetrating the shielding from ^{244}Cm at energy $E_{(\text{Cm})}_i$

The summation is made from a selected value for i_0 (which corresponds to a particular shielding configuration) to the value of i for which $E_i = 6.5$ Mev. The calculations were made for $E_{i_0} = 0, 0.5, 1.0, 2.0, 3.0, 4.0,$ and 5.0 Mev.

Gamma radiations from ^{244}Cm are listed in Table A-I. Maximum allowable contributions from impurities to the product dose rate, as calculated with Equation 1, are given in Table A-II for each shielding variation.

The significant gamma-emitting impurities in the curium product are ^{144}Ce - ^{144}Pr , ^{106}Ru - ^{106}Rh , and ^{95}Zr - ^{95}Nb . The maximum permissible amount of each impurity was calculated for each shielding configuration. The gamma rays and energies used for these calculations are listed in Table A-III, and the results of the calculations are listed in Table A-IV. Since all these impurities may be present in significant concentrations in the final product, the permissible concentration of any one depends on the concentration of the others. These relationships are illustrated in Figure A-1. Required decontamination factors (Figure 9) in the pilot production program are obtained by comparing these permissible maximum quantities with the quantities present in the target elements (Table I).

TABLE A-I

Gamma Rays from ^{244}Cm From Alpha Decay⁽¹⁷⁾

<u>Energy, Mev</u>	<u>Abundance, photons/(sec)(g of ^{244}Cm)</u>
0.043	6.3×10^8
0.100	4.5×10^7
0.150	3.9×10^7
0.262	3.5×10^8
0.570 }	4.2×10^8
0.610 }	
0.825	2.0×10^8

From Spontaneous Fission⁽¹⁴⁾

<u>Energy, Mev</u>	<u>Abundance, photons/(sec)(g of ^{244}Cm)</u>
0-0.5	1.2×10^7
0.5-1.0	7.4×10^6
1.0-1.5	3.3×10^6
1.5-2.0	2.1×10^6
2.0-2.5	1.1×10^6
2.5-3.0	5.8×10^5
3.0-3.5	2.4×10^5
3.5-4.0	2.5×10^5
4.0-4.5	9.3×10^4
4.5-5.0	7.4×10^4
5.0-5.5	6.6×10^4
5.5-6.0	2.7×10^4
6.0-6.5	1.6×10^4

From Fission Products of Spontaneous Fission⁽¹⁸⁾

<u>Energy, Mev</u>	<u>Abundance, photons/(sec)(g of ^{244}Cm)</u>
0.1-0.4	6.3×10^8
0.4-0.9	1.9×10^7
0.9-1.35	1.9×10^8
1.35-1.8	2.4×10^8
1.8-2.2	1.2×10^8
2.2-2.6	4.5×10^5
2.6-3.0	4.4×10^4

TABLE A-II

Maximum Allowable Contribution of Impurities to
the Gamma Dose Rate from the Curium Product

Energy Range of Unshielded Photons, Mev	Dose Rate Index ^(a)
0-6.5	8×10^6
0.5-6.5	2.9×10^6
1.0-6.5	1.9×10^6
2.0-6.5	7.6×10^5
3.0-6.5	2.8×10^5
4.0-6.5	1.2×10^5
5.0-6.5	4.9×10^4

$$(a) \text{ Dose Rate Index} = \left[0.1 \sum_i [N_{(Cm)}]_i [E_{(Cm)}]_i \right]$$

TABLE A-III

Gamma Rays from Fission Products

Fission Products	Gamma Ray Energy, Mev	Abundance, photons/disintegration
$^{144}\text{Ce}-^{144}\text{Pr}(19, 20)$	0.033	9×10^{-3}
	0.041	8×10^{-3}
	0.054	2.5×10^{-2}
	0.058	1×10^{-2}
	0.080	3.6×10^{-2}
	0.095	5.5×10^{-2}
	0.134	1×10^{-1}
	0.145	3×10^{-3}
	0.695	1.6×10^{-2}
	1.490	2.5×10^{-3}
	2.185	8×10^{-3}
$^{106}\text{Ru}-^{106}\text{Rh}(19)$	0.513	2.85×10^{-1}
	0.624	1.52×10^{-1}
	0.873	1.1×10^{-2}
	1.045	2.2×10^{-2}
	1.14	3×10^{-3}
	1.56	6×10^{-3}
	1.77	3×10^{-3}
	2.28	2×10^{-3}
	2.43	3×10^{-3}
$^{95}\text{Zr}-^{95}\text{Nb}(19)$	0.74	0.98
	0.77	1.00

TABLE A-IV

Maximum Allowable Concentrations
of Fission Product Impurities

(In the absence of other radioactive impurities)

<u>Fission Product</u>	<u>Maximum Concentrations, curies/g ^{244}Cm</u>
^{144}Ce - ^{144}Pr	1.1×10^{-3}
^{106}Ru - ^{106}Rh	2.6×10^{-4}
^{95}Zr - ^{95}Nb	4.8×10^{-5}

FIG. A-1 ALLOWABLE CONCENTRATIONS OF
FISSION PRODUCT IMPURITIES

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