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ISOTOPIC POWER FUELS MONTHLY STATUS REPORT FOR MARCH 1974

C. L. Ottinger

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ISOTOPES DEVELOPMENT CENTER

ISOTOPIC POWER FUELS MONTHLY STATUS REPORT FOR MARCH 1974

C. L. Ottinger

Isotopes Division

MAY 1974

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ISOTOPIC POWER FUELS MONTHLY STATUS REPORT FOR MARCH 1974

C. L. Ottinger

CURIUM-244 FUEL DEVELOPMENT

(Division of Space Nuclear Systems Program LR 30 01 03 3)

$^{244}Cm_2O_3$ Compatibility Program

Compatibility Couple Tests *(J. R. DiStefano)*

Compatibility exposures from Set Nos. 1, 2, 7, 8, 9, and 10 (see Table 1) have been completed and examinations are in progress. The Ir, Pt, Pt—20% Rh, and Pt—26% Rh—8% W (Pt-2608) samples from Set Nos. 1 and 2 (900°C tests) were selected for examination on a first priority basis. Top disk samples were removed for chemical analysis and the results are shown below:

Metal or Alloy	Oxygen Concentration (ppm) Set 1	Set 2
Iridium	<10	<10
Platinum	<10	<10
Pt—26% Rh	<10	<10
Pt-2608	24	17

Table 1. $^{244}Cm_2O_3$ Compatibility Test Conditions

Set Number	Test Materials and Conditions
1	900°C; 5000 hr; helium atmosphere; Ir, C, Pt, Hf—1% Pt—0.5% Pd, Hastelloy C-276, Haynes alloy 25, Haynes alloy 188, ThO_2, Pt—20% Rh, and Pt—26% Rh—8% W
2	900°C; 5000 hr; graphite, helium atmosphere; Ir, Pt, Hf—1% Pt—0.5% Pd, Hastelloy C-276, Haynes alloy 25, Haynes alloy 188, ThO_2, Pt—20% Rh, and Pt—26% Rh—8% W
7	1400°C; 5000 hr; graphite, helium atmosphere; Ir, C, Mo, Mo—46% Re, Ta, T-111, W, and W—26% Re
8	1400°C; 5000 hr; dynamic vacuum; Ir, Mo, Mo—46% Re, Ta, T-111, W, and W—26% Re
9	1400°C; 5000 hr; helium atmosphere; Ir, Mo, Mo—46% Re, Ta, T-111, W, and W—26% Re
10	1400°C; 5000 hr; graphite, dynamic vacuum; Ir, C, Mo, Mo—46% Re, Ta, T-111, W, and W—26% Re

Only the alloy Pt-2608 picked up oxygen. We had previously found that only Pt-2608M (containing 0.5% Ti) picked up oxygen (from 10 to 90 ppm) at 1100°C.

At 1100°C unalloyed platinum reacted with $^{244}Cm_2O_3$ to form a Pt-Cm compound, but little or no interaction occurred with the other materials (Pt—20% Rh was not tested at 1100°C). Preliminary examination of the 900°C tests indicates that unalloyed platinum has slightly reacted with $^{244}Cm_2O_3$, but the other materials appear to be unaffected.

Metallographic preparation of the other materials from Set Nos. 1 and 2 has been initiated.

FY 1974 Compatibility Matrix *(J. R. DiStefano and C. L. Ottinger)*

All components for the mechanical properties tests have been received. Tests of Si_3N_4 disks will be conducted in a Si_3N_4 inner liner and a molybdenum outer container. Components for this test system are now being fabricated. Additional test pressings of Gd_2O_3 in the slot-type die gave good results, yielding flat uniform bars with good strength. The technique will be tested on $^{244}Cm_2O_3$ next month. Equipment for the in-cell assembly of the tensile strength/compatibility couples is being designed.

Thermodynamic Calculations — Reactions of $^{244}Cm_2O_3$ Fuel With Si_3N_4 and Pt-2608M *(E. E. Ketchen)*

Reactions of $^{244}Cm_2O_3$ fuel (95% Cm_2O_3—5% PuO_2) with the components of Pt—26% Rh—8% W—0.5% Ti (Pt-2608M) alloy and with silicon nitride have been investigated from a thermodynamic viewpoint. Both of these materials have been considered for use as encapsulants for $^{244}Cm_2O_3$. Samples of Pt-2608M were included in the FY 1973 test matrix, and the experimental results have been reported previously. Silicon nitride is scheduled for testing in the FY 1974 matrix.

Pt-2608M — The composition of Pt-2608M in atom percent is: Pt, 52.27; Rh, 39.33; W, 6.77; and Ti, 1.63. The partial molar free energies of mixing for Pt, Rh, W, and Ti were calculated using the method previously described.[1] The results are shown in Table 2. It should be noted that these calculations assume that there is no compound formation between Pt, Rh, W, and Ti.

Table 2. Partial Molar Free Energies of Mixing of Components of Pt—26% Rh—8% W—0.5% Ti

Temperature (°C)	$-\Delta\overline{G}_m$ (cal/mole)			
	Pt	Rh	W	Ti
600	1040	1570	4420	6,980
800	1300	1940	5490	8,620
1000	1560	2320	6560	10,260
1200	1820	2690	7630	11,890
1300	1950	2870	8160	12,710

[1]Eugene Lamb, *Isotopic Power Fuels Monthly Status Report for July 1973*, ORNL-TM-4339, Oak Ridge National Laboratory.

The free energies of formation of Pt_3O_4, Rh_2O, WO_2, TiO, Pu_2O_3, and PuO_2 were calculated from a data compilation by Glassner[2] and a review article by Oetting.[3] The results are shown in Table 3. Platinum will not react with oxygen at 600-1300°C, but rhodium, tungsten, and titanium will react with oxygen at 600-1300°C.

Table 3. Gibbs Free Energy of Formation of Pt_3O_4, Rh_2O, WO_2, TiO, Pu_2O_3, and PuO_2

Temperature (°C)	ΔG_f (cal/mole)					
	Pt_3O_4	Rh_2O	WO_2	TiO	Pu_2O_3[a]	PuO_2
600	10,300	-11,350	-101,160	-103,780	-352,120	-215,550
800	26,400	-9,090	-93,850	-99,380	-339,320	-207,050
1000	42,080	-6,940	-86,780	-95,080	-326,940	-198,700
1200	57,330	-4,900	-79,960	-90,870	-314,980	-190,530
1300	64,790	-3,910	-76,800	-88,810	-309,150	-186,500

[a] Pu_2O_3 data are used as a stand-in for Cm_2O_3.

The free energies from Tables 2 and 3 were used to calculate the free energies for Eqs. 1-6. Results are shown in Table 4.

$$6Rh(\text{in Pt-2608M alloy})(s) + Cm_2O_3(s) \rightarrow 3Rh_2O(s) + 2Cm(s) \quad (1)$$

$$2Rh(\text{in Pt-2608M alloy})(s) + 2PuO_2(s) \rightarrow Rh_2O(s) + Pu_2O_3(s) \quad (2)$$

$$3W(\text{in Pt-2608M alloy})(s) + 2Cm_2O_3(s) \rightarrow 3WO_2(s) + 4Cm(s) \quad (3)$$

$$W(\text{in Pt-2608M alloy})(s) + 4PuO_2(s) \rightarrow WO_2(s) + 2Pu_2O_3(s) \quad (4)$$

$$3Ti(\text{in Pt-2608M alloy})(s) + Cm_2O_3(s) \rightarrow 3TiO(s) + 2Cm(s) \quad (5)$$

$$Ti(\text{in Pt-2608M alloy})(s) + 2PuO_2(s) \rightarrow TiO(s) + Pu_2O_3(s) \quad (6)$$

Table 4. Gibbs Free Energy of Reaction for Eqs. 1-6

Temperature (°C)	ΔG_r (kcal/mole of Cm_2O_3 or PuO_2)					
	Eq. 1	Eq. 2	Eq. 3	Eq. 4	Eq. 5	Eq. 6
600	327.49	35.38	207.01	15.30	61.72	-8.92
800	323.69	34.78	206.78	15.30	67.04	-8.00
1000	320.04	34.09	206.61	15.18	72.48	-7.18
1200	316.42	33.28	206.49	14.96	78.04	-6.45
1300	315.07	32.85	206.19	14.77	80.85	-6.12

[2] Alvin Glassner, *The Thermochemical Properties of the Oxides, Fluorides, and Chlorides to 2500°K*, ANL-5750, Argonne National Laboratory (1957).
[3] F. L. Oetting, "The Chemical Thermodynamic Properties of Plutonium Compounds," *Chem. Rev.* 67, 261-97 (June 1967).

Since the free energies are positive for Eqs. 1, 3, and 5, Cm_2O_3 is not expected to react with the alloy. However, the reaction of titanium with PuO_2 has a negative free energy, hence the PuO_2 of the curium product is expected to react with the titanium of the alloy.

In conclusion, it appears from thermodynamic considerations that the Pt–26% Rh–8% W–0.5% Ti alloy will react with the Cm_2O_3 fuel containing 5% PuO_2 at 600-1300°C. The calculations were made on the assumption that there is no compound formation between Pt, Rh, W, and Ti or Cm metal and the alloy. The oxides most favorable to be produced were considered. No assumptions were made as to the solubility of oxygen in the alloy. Experimental verification of the calculations is needed.

Silicon Nitride — Several possible reactions of Si_3N_4 with the curium heat source product are conceivable. Some of the conceivable products are CmN, N_2, SiO_2, CmSi, Cm_2Si_3, and $CmSi_2$. The thermodynamic data needed for the calculating of the free energies of reaction to produce curium silicides are not available. Hence, the free energy calculations were made on the CmN, N_2, and SiO_2 only. Since the actinide silicides are probably among the weaker compounds, they may not be important anyway.

The free energies of formation of Si_3N_4, CeN, SiO_2, Pu_2O_3, and PuO_2 were calculated from a data calculation by O. Kubaschewski *et al.*,[4] a compilation by Glassner,[5] a survey by K. A. Gschneidner,[6] and a review article by Oetting.[7] The results are shown in Table 5.

Table 5. Gibbs Free Energy of Formation of Si_3N_4, CeN, SiO_2, Pu_2O_3, and PuO_2

Temperature (°C)	$-\Delta G_f$ (kcal/mole)				
	Si_3N_4	CeN	SiO_2	Pu_2O_3	PuO_2
600	240.79	56.18	172.92	352.12	215.55
800	260.99	51.18	164.60	339.32	207.05
1000	282.05	46.18	156.33	326.94	198.70
1200	303.97	41.18	148.10	314.98	190.53
1300	315.14	38.68	144.00	309.15	186.50

The data from Table 5 were used to calculate the free energies of Eqs. 7 and 8. The CeN was used as a stand-in for CmN, and Pu_2O_3 was used as a stand-in for Cm_2O_3. Results are shown in Table 6.

[4]O. Kubaschewski, E. L. Evans, and C. B. Alcock, *Metallurgical Thermochemistry*, Pergamon Press, New York, 1967, pp. 421-29.
[5]Glassner, ANL-5750.
[6]K. A. Gschneidner, *Rare Earth Alloys*, D. Van Nostrand Company, Inc., Princeton, N. J., 1961, p. 237.
[7]Oetting, *Chem. Rev.* 67, 261-97 (1967).

$$2Cm_2O_3(s) + Si_3N_4(s) \rightarrow 4CmN(s) + 3SiO_2(s) \quad (7)$$

$$12PuO_2(s) + Si_3N_4(s) \rightarrow 6Pu_2O_3(s) + 3SiO_2(s) + 2N_2(g) \quad (8)$$

Table 6. Gibbs Free Energy for Eqs. 7 and 8

Temperature (°C)	ΔG_r (cal/mole of Cm_2O_3 or PuO_2) Eq. 7	Eq. 8
600	156,960	16,320
800	171,740	17,980
1000	187,300	19,660
1200	203,640	21,350
1300	212,040	22,190

Since the free energies of Eqs. 7 and 8 are positive, the Si_3N_4 should not react with the curium oxide heat source product to produce CmN, N_2, or SiO_2. Nothing definite can be said about the reactions of Si_3N_4 with the curium oxide product to give a silicide but this is not expected since the silicides are probably weaker compounds than the nitrides. From the standpoint of thermodynamic calculations, the Si_3N_4 should not react with the curium oxide product. Compatibility studies are needed to verify the conclusions.

$^{244}Cm_2O_3$ Property Characterization

Helium Release *(P. Angelini)*

Four isochronal helium release experiments were performed using the remainder of the $^{244}Cm_2O_3$ fuel from helium release experiment C (650°C). These were performed to provide additional information as to the types of helium release mechanisms operating in the 500 and 800°C temperature range.

The experimental conditions of the four experiments are presented in Table 7.

Table 7. Experimental Conditions of the Isochronal Anneal Experiments for $^{244}Cm_2O_3$

Experiment Number	Initial Annealing Temperature (°C)	Time Period at Ambient Temperature (days)
1	800	3
2	800	20
3	1100	20
4	1100	4

In these experiments the initial annealing temperature was held for approximately 1 hr. The sample was then cooled over a 3-hr period to ambient temperature with the sample held at ambient temperature for the desired time period as shown in Table 7. The concentation of helium in the sample increased at ambient temperature during the desired time period. During each of the isochronal anneal experiments, the sample was maintained at the desired temperature for one half hour with the sample's temperature increased in 100°C intervals.

The data obtained during the experiments are presented in Fig. 1. In observing the data from experiment 1, the onset of a definite change in helium release characteristics of the sample occurred near the 600°C temperature range. In experiment 2 the sample was maintained at ambient temperature for 20 days (approximately seven times longer than experiment 1). Again the onset of the measurable change of the helium release characteristics occurred near the 600°C temperature. Thus the initial helium concentration in the sample within the concentration range obtained in the present experiments does not significantly change the temperature at which there is a definite change in the helium release mechanism.

The experimental conditions were changed such that the initial annealing temperature was increased from 800 to 1100°C. The data from experiment 3 (20-day period at ambient temperature) indicates that the onset of measurable change in the helium release characteristics again occurred near the 600°C temperature. In this experiment, burst-type helium release was observed to begin at 300°C, to peak at 800°C, and to continue to 1100°C. In experiment 4 (4-day period at ambient temperature) the onset of the change in the helium release again occurred near 600°C. In addition there was no burst-type helium release observed during the experiment. Thus changing the initial annealing temperature from 800 to 1100°C in the present experiments does not significantly change the temperature at which the onset of the helium release mechanism operative above the 600°C temperature range is initiated. The 600°C temperature is indicated to be the minimum temperature at which the helium release mechanism operating in the 600-1300°C temperature range is measurably observed in the present experiments.

The burst-type helium release in experiment 3 occurred over the 300 to 1100°C temperature range. This type of helium release decreased as time progressed in the 800°C steady-state helium release experiment; it increased with increasing time in the 500 and 650°C steady-state experiments. The results from these experiments show that the burst-type helium release is related to the helium concentration in the sample over the 300 to 1100°C temperature range and is the major helium release mechanism operating below the 650°C temperature range.

As shown by the gradual disappearance of the burst-type helium release in the 800°C experiment, this type of release becomes secondary during steady-state experiments above the 650°C temperature range.

The gradual increase in the burst-type helium release at temperatures below 650°C indicates that as the concentration of helium increases in a radiation damaged fuel form the single helium atoms form clusters. As time progresses

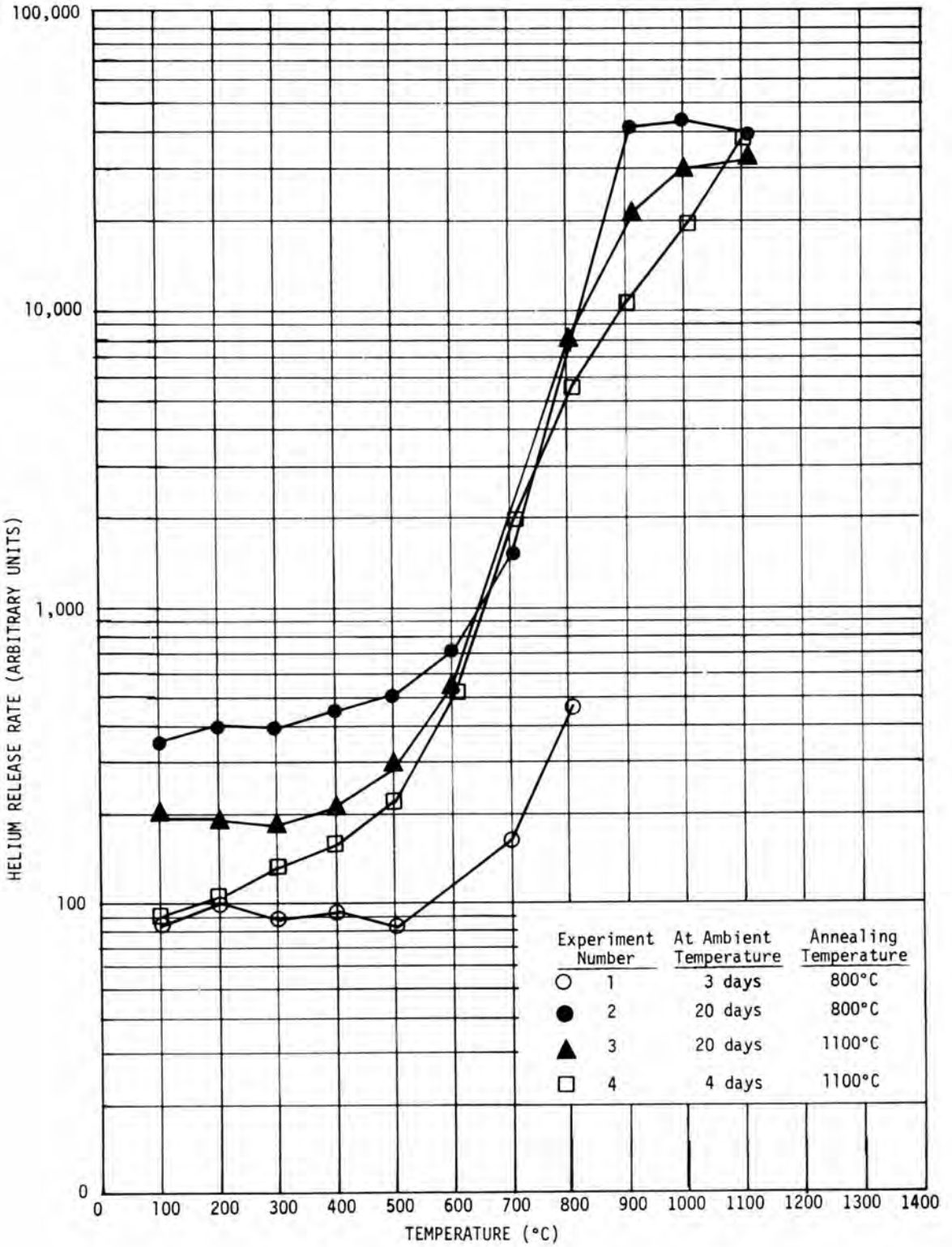

Fig. 1. Isochronal Anneal Experiments for $^{244}Cm_2O_3$.

the concentration of the clusters increases such that they interact in providing an interconnected microstructure through which the clusters may be released. Above the 650°C temperature range, the experiments indicate that the clusters or dislocation loops formed by radiation damage become significantly more mobile and migrate to releasing areas. The rate of annealing for this process at temperatures greater than 650°C is such that coalescence of large numbers of helium-filled clusters or dislocation loops is effectively reduced. The helium release rate becomes more uniform with the burst-type helium release almost eliminated as the sample's temperature is maintained for long periods of time at temperatures above 650°C.

Vapor Pressure *(P. Angelini and J. C. Posey)*

In preparation for a re-run on several data points of the vapor pressure, new target holders were fabricated. The use of a fresh target holder for each cycle of measurements should reduce the likelihood of incidental contamination on the targets; this is especially important at the low-temperature end of the range since the amount of ^{244}Cm collected is so small. In combination with the improved temperature control reported last month, this technique should provide better data.

Effects of Plutonium In-Growth *(C. L. Ottinger)*

Current experimental work with ^{244}Cm utilizes for the most part "stock" ^{244}Cm oxide resulting from a processing campaign carried out at SRL during the period 1968-1971. A small fraction of ^{244}Cm was recovered from waste residues at ORNL in 1973 to provide some low-plutonium material for experiments; but, except for this fraction, materials available contain plutonium concentrations ranging upwards from ∿10%. Although these are referred to as "production grade," this term is not intended to imply that they are identical to the ^{244}Cm products that would be expected from a by-product ^{244}Cm recovery plant.

Among the as yet unanswered questions in the development of ^{244}Cm for heat source use are those related to the effects of impurities. The impurities which will be present in practical ^{244}Cm fuels are of two types — process contaminants and plutonium. Process contaminants are those which are not totally removed during the ^{244}Cm separations process or which are introduced as a result of the process; these may include any of a large variety of impurities at various levels. Since the development of process flowsheets is still only in the study stage, evaluation of the likely process impurities can be only an educated guess at present. Based on previous processing experience, it can be inferred that americium will be the primary process contaminant, and that it will be present in concentrations of about 1-2 wt %. For study purposes, an initial ratio of process impurities to curium of 0.03 has been assumed, but no assumed distribution of impurities has been established. Possible influences of process impurities include effects on fabricability of fuels, compatibility, vaporization characteristics, thermophysical properties, and others in addition to the obvious effect on power density. It will be necessary to evaluate these

effects both by simulation and eventually by practical demonstration when typical fuels are available.

While the types and amounts of process impurities cannot be accurately predicted at present, the amount of plutonium can be since it varies only with the time since separation of the ^{244}Cm. (It can be confidently assumed that the plutonium content will be insignificant as of separation time.) In any processing/fuel fabrication/source encapsulation system which includes a few months of storage and/or shipment time for the products, plutonium will be the major contaminant in the fuel as of time of source fabrication. Also, the "curium" materials used in experimental work are, of course, mixtures of curium and plutonium (at least), and the interpretation of experimental results must take this into account. Not only the concentration of plutonium but also its chemical form can influence the characteristics of the fuel.

It has been generally assumed that a ^{244}Cm recovery system would process the ^{244}Cm to the CmO_2 form for storage and that it would be converted to the Cm_2O_3 form immediately prior to encapsulation. During the period between chemical separation and encapsulation, any plutonium resulting from decay would thus be in the dioxide form. The procedures normally used for conversion of the CmO_2 to Cm_2O_3 (typically calcination at 1000°C in an oxygen-free atmosphere) will not convert the PuO_2 to the sesquioxide form. As of the time of encapsulation, the fuel will be composed of Cm_2O_3 and PuO_2 plus the oxides of the process impurities. After encapsulation there will be no free oxygen available for oxidation of the decay-produced plutonium, and the mixed oxide system will become more complex.

One approach to dealing with the question of the chemical form of the decay-produced plutonium would be to establish curium process systems to provide Cm_2O_3 as the product form. The Cm_2O_3 could be stored under oxygen-free conditions and all subsequent operations carried out in inert-atmosphere enclosures. Both the curium and the decay-produced plutonium oxides would then remain in the most reduced state. While the Pu_2O_3 may exert significant influences on the characteristics of the fuel, the types and magnitudes of such influences could be determined systematically. Also, thermodynamic calculations indicate that PuO_2 may react with components of several potential encapsulants, so elimination of PuO_2 from the fuel may be attractive in this regard.

The recent re-orientation of the ^{244}Cm development program requires that most of the ^{244}Cm now in experimental facilities be encapsulated for long-term storage. These materials include batches ranging in plutonium content from less than 5% to nearly 20%. Selected fractions will be converted to the Cm_2O_3 form prior to encapsulation so that samples will be available in the future for examination.

General Operations *(C. L. Ottinger)*

The furnace and associated in-cell equipment which had been used for ceramic compatibility work were disassembled and discarded. Cell services

were removed and decontamination was started in order to release the cell for other operations. A considerable quantity of solid waste was removed from the Curium Souce Fabrication Facility cells and the equipment was checked out in preparation for $^{244}Cm_2O_3$ pelletization work.

Curium-244 Recovery Development

(C. L. Ottinger and J. C. Posey)

A program has been initiated to investigate various processes having potential applicability for the recovery of ^{244}Cm from the wastes resulting from processing of power reactor fuels. The ultimate goal of this program is the demonstration of one or more flowsheets adaptable to production-scale processing. If ^{244}Cm is to be utilized in significant quantities for power generation or other applications, a system must be developed which is attractive when compared to alternative sources of power. Comparisons between ^{244}Cm power sources and other potential sources will be based on several criteria, including relative safety, applicability, system weights (for space applications), availability, and, of course, cost. The cost of recovery and purification will be a significant factor in any analysis, and process development information will be required in order to predict these costs.

Since any ^{244}Cm recovery process must eventually be incorporated into a system which includes not only the separations process itself, but also receipt of feed material and preparation of solid wastes for storage, certain limitations are imposed on the ^{244}Cm process. In addition to meeting the normal criteria of technical feasibility, safety, and economics, the process must be designed to limit the volume of solid waste to the lowest practical value. The process should also be as versatile as possible in order to accomodate changes in feed composition. Future feed supplies (i.e., reactor fuel wastes) are expected to fall into two general categories: 1) feeds containing essentially fission products with relatively small amounts of other contaminants; and 2) feeds in which a large fraction of the elemental content results from process chemicals added during fuel reprocessing operations. Two reference fuel compositions will be defined to represent these two categories, and the necessary modifications to processes related to this variation will be investigated.

Processing of reactor fuel wastes for recovery of ^{244}Cm will involve four distinct but inter-related phases:

1. Feed Conditioning: Modification of the stored reactor fuel waste to provide a solution ready for ^{244}Cm processing.

2. LN/AN Separation: Processing to provide a ^{244}Cm stream which is composed essentially of those lanthanides (LN) and actinides (AN) that are analogs of curium in classical processes.

3. AN Separation: Fractionation of the LN/AN stream to provide a ^{244}Cm stream containing americium as the primary contaminant.

4. Purification: Final separation of the ^{244}Cm from the americium and the remaining other contaminants.

Each of these phases may incorporate several unit processes or operations, but this division provides a convenient description of the required group operations. The initial area of concentrated effort in this program will be Phase 3, although some work will be done on other phases concurrently. Solvent extraction, in particular differential extraction, appears to offer several advantages as applied to the LN/AN separation, and this process will be evaluated.

APPENDIX

Curium-244 Inventory

	^{244}Cm Status (g)		Element[a]
	3-1-74	4-1-74	(g)
Receipts From SRL			
Allocated to LR Heat Source Program	1,902	1,902	2,019
Allocated to NT Target Program	302	302	321
Total Receipts From SRL	2,204	2,204	2,340
Withdrawals From LR Program			
Analytical samples	2	2	2
Shipped to Lovelace	9	9	10
Shipped to SRL	2	2	2
Discarded to ORNL waste	6	6	6
Unaccounted for	80	80	85
Total Withdrawals From LR Program	99	99	105
Balance Remaining in LR Program			
Stored residues	233	233	247
In-use material	223	223	237
Stored raw material	1,347	1,347	1,430
Total Balance in LR Program	1,803	1,803	1,914
Withdrawals From NT Program			
Transferred to TRU	158	158	168
Shipped to other sites	47	51	54
Transferred to Target Group	8	8	8
Total Withdrawals From NT Program	213	217	230
Balance Remaining in NT Program	89	85	91

[a]All values corrected for decay to May 1, 1973.

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